Experimental Approaches for Assessing Time and Temperature Dependent Performances of Fractured Laminated Safety Glass

Experimentele onderzoeksstrategieën voor het beoordelen van de tijds- en temperatuursafhankelijke prestaties van gebroken gelamineerd veiligheidsglas

Didier Delincé

Promotoren: prof. dr. ir. -architect J. Belis, ereprof. dr. ir. R. Van Impe
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prof. dr. ir. Luc Taerwe (chairman)	Ghent University
Dr Fabrice Bernard	INSA Rennes
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prof. dr. ir. Wim Van Paepegem (secretary)	Ghent University
prof. dr. ir.-arch. Jan Belis (supervisor)	Ghent University
prof. dr. ir. Rudy Van Impe (supervisor)	Ghent University

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Laboratory for Research on Structural Models (LMO)
Department of Structural Engineering (EA14)
Faculty of Engineering and Architecture (FEA)
Ghent University

www.LMO.UGent.be

“Not many engineers are conscious of the subjectivity of their analysis.”
“Engineers however are not comfortable with quantifying beliefs.”
Prof. Dr. Guido Morgenthal, Chair German Group of IABSE,
extracts from the editorial of Structural Engineering International (SEI) 3/2013
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Didier Delincé
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Summary

Laminated safety glass has been introduced in the building industry in the 1980's as safety glazing, to improve the safety of persons in case of accidental impact. The basic product configuration made of two flat glass sheets around and bonded to a polymer film which acts as interlayer slightly evolved in the 1990's with the development of larger glazing façades, and in particular of new fixations systems with the so-called "structural glazing" applications, mainly the structural sealant glass systems (SSGS) and the point-fixing glazing systems. Laminated glass units began also progressively to be used in non-glazing applications, as elements of balustrades, glass fins, and finally up to load-bearing plates in glass staircases and glass floors. With the XXIth century and the emergence of structural applications in laminated glass, the product configurations evolved in larger proportions, towards multi-layered laminated glass products and with the integration of metallic inserts and the apparition of new interlayer products.

The design rules and the assessment methods of laminated glass products and glass works evolved in parallel, but not always with much apparent consistency. Besides the evolutions on the glass market for the building industry, other important changes were initiated in Europe in the field of standardization, with the introduction in 1989 of the Construction Products Directive (CPD) for the assessment of construction products and their performances in construction works. Its particularity is to introduce a new philosophy for development of standardization, based on a performance based approach. This led to the development of a series of standards and guidelines issued by two European institutes, the CEN (European Committee for Standardization) and the EOTA (European Organization for Technical Assessment) respectively, intended to support and implement the harmonization purposes of the CPD. However, due to the parallel processes and the various involved stakeholders, the so-developed European standardization framework also has a number of apparent inconsistencies, at least for parties non-closely involved in the developments.

These two evolutions are analysed in parallel. The definitions of safety performances and associated test methods in the product standards and other technical guidelines are compared and discussed, more particularly with regard to the assessment of the post-fracture performances of laminated glass products. Indeed, the evaluation of the residual load-bearing capacities of fractured elements progressively gained importance with the evolution towards non-conventional structural applications. Nonetheless, the safety performances and assessment methods as prescribed in current product standards for laminated safety glass products still implicitly mainly consider the traditional glazing applications. The experimental characterization of general post-fracture performances of laminated glass products with regard to various, vague and evolving application scopes is thus complicated. With regard to the fragmentation of design and assessment processes in product-oriented and project-oriented development, there exists a

serious risk of an uncontrolled increase of required tests. Consequently, most of the time relatively little attention is dedicated to the characterization of product properties ruling the post-fracture resistance of laminated glass elements, and in particular with regard to the time-temperature dependent mechanical properties of the interlayer components. The comparison of the concepts of "product family" (scope of product configurations considered for the assessment of the performances) and of "intended field of use" (applications scope) led to introduce an analysis grid distinguishing different *Application Fields*, which combined with each other's, help to detail the important parameters related to safety performances.

The various issues related to post-fracture performances can be better understood by means of structural analysis of fractured systems within a design approach based on failure scenarios. An inventory of the parameters ruling the damage sensitivity (probability of breakage of glass component) and the damage tolerance (consequence of damage on the residual resistance) of laminated glass elements is made. It is shown that the bridging function ensured by interlayers in fractured laminated systems can be reduced to two main load-transfer mechanisms. The behaviour in ultimate fractured states is ruled for all element and loading configurations by the same critical load transfer mechanism, the ligament function between glass fragments. This fulfils a bridging function depending on two complementary mechanisms : the delamination of the interlayer from the glass substrates, and its stretching. Assumptions and conditions are identified to dissociate the assessment of the post-fracture performances in quasi-static design conditions from the dynamic actions and the dynamic response of the element. This leads to the assumption that if an element survives to the successive accidents, the interlayer ligaments are not damaged by the fragmentation processes. As a consequence, the description of physical damage of fractured elements must be completed by initial delamination lengths near the crack tips at the interface between interlayers and glass fragments.

The assumptions made at the structural level allow to focus further on the material properties of interlayers intervening in the ligament response. An overview of the main features and characteristics of polymers used as interlayers shows that they belong to two families of polymer products, the thermoplastics and the elastomers. The former exhibit a glass-rubber transition in the range of service temperatures; the latter behave more closely to ideal rubbers. Their respective typical behaviour seems in both cases largely ruled by the secondary intermolecular bonds and the consequent mobility of the molecular chains. As the first ones correspond to the most used type of interlayers and exhibit more significant time-temperature dependent properties, the corresponding viscoplastic models in the large strain domain are considered in more details. The thermorheological simple or complex nature of the response is described, and a complementary phenomenon of importance for the properties in the solid phase is identified, namely *physical ageing*. This phenomenon appears related to

thermally reversible conformational changes at the molecular level. It has a rather important influence on the resistance to long-duration creep of polymer products. Then, particularities of polymers used as adhesives with regard to mechanical behaviour are summarized. It leads to assume that physical ageing affects the interfacial and bulk properties in a differentiated way, and to conclude that the end properties of products depend in an undefined and variable proportion on the lamination process and on the service conditions. Besides, with respect to the possible ranges of temperatures in service conditions for laminated glass products, ageing and rejuvenating effects are likely to modify the physical ageing state during their lifetime.

Two families of interlayer products are considered closer, polyvinyl butyral products in general (PVB) and a stiffer material, SentryGlas® (SG). The experimental aspects are considered, and the eligible configurations of test specimens for performing the assessment discussed. In this context, tests on laminated specimens seem the most appropriate approach. It leads to conceive the assessment of interlayer properties rather from the perspective of a *component* than a material, and to identify the determination of the most appropriate test configurations with regard to different purposes as an important part of the assessment problem.

Tests on fractured laminated glass elements can be performed at different experimental scales. The scale concept refers not only to the size of the tested elements, but more generally to different sets of test conditions. Because of the sensitivity of the response of polymer components to combinations of stress and temperature effects, it imposes particular constraints for the development of test infrastructures and test methods. A series of experimental campaigns performed during this research at different scales is analysed by means of a proposed analysis grid detailing different *Experimental Fields of Investigation*. The interest of this decomposition is its use for distinguishing different technical limits and sources of systematic deviations possibly arising during the conception of test configurations. In fact, it is mainly when the experimental fields of investigation are extended to larger ranges that new systematic deviations and errors are likely to appear, and this with regard to three categories of border effects. Border effects of the two first categories are essentially related to practical and experimental issues, in relation to processing methods of test specimens for the first, and in relation to tolerances and measurement uncertainties peculiar to the test configuration and ranges of investigation for the second. These types of border effects appear as potentially more important for tests on specimens of small dimensions. The third category of border effect accounts for sources of deviation due to analysis and processing methods of results, and propagation of uncertainties. The overview also helps to identify typical technical issues arising to reduce the uncertainties on a combination of experimental fields, in particular with regard to the combined use of optical measurement methods and of a climatic chamber.

The Through Crack Tensile test (TCT-test) is further considered as a reference test configuration for investigating the effect of different combinations of test temperature and loading level on the response of fractured laminates made with a SG-interlayer, for two loading modes. A single sample of about 60 specimens laminated glass with a unique configuration (type of interlayer product, thickness and processing method) is used to develop an incremental experimental strategy. This is based on series of tests of relative short-duration carried out at constant displacement rate (cdr-loading mode) and in creep loading mode in quasi-static loading conditions. The incremental approach allows to get an overview of the response on a relative large range of test conditions, for test temperatures varying between –20 and +60°C. The experimental aspects for the tests performed inside a climatic chamber and the related uncertainties are then analysed critically. In particular, limits on the accuracy of measurement of deformations by optical methods are evaluated, in regard with other related experimental aspects.

Two main deformation patterns are observed during the tests in the different test conditions : a regular delamination pattern and a crack propagation pattern through the ligament thickness. The failure mode appears to vary significantly according to the test conditions. The cdr-loading mode appears not so useful for the considered design problem, as no complete correspondence could be found with the creep load mode. This is mainly due to the variable contribution of the two deformation mechanisms to the overall ductility of the TCT-specimens. The campaign allowed also to point out the effect of storage duration on the behaviour of test specimens, attributed to an effect of physical ageing. This significant effect observed at experimental scales highlights an issue for interpreting test results in a quantitatively relevant way. The macroscopic response as analysed by means of the TCT-test results corresponds with an apparent thermorheological complex behaviour of the viscoplastic response, with significant differences of response on the experimental investigation scope.

The main outcomes of this thesis highlight the need for adapting the experimental assessment approaches for interlayer components of laminated glass products, in comparison with other construction materials, and the difficulties for obtaining quantitatively meaningful results for design practice. With the reported experimental works, a sensitivity analysis could be performed for a few experimental parameters seldom accounted for, which prove however to have a noticeable influence on test results. It also underlines the usefulness of tests at intermediate scale and TCT-tests in particular for isolating problems related to the time-temperature response of the interlayer from aspects related to the strength of the glass components. Finally, the intermediate experimental scales are also analysed from a sectorial perspective and reflexion trails are given with regard to development of “harmonized” calculation models and to implementation issues of characterization methods in design practice.

Samenvatting

Gelamineerd veiligheidsglas werd geïntroduceerd in de bouwsector in de jaren 1980 om personen te beschermen in geval van accidentele impact. De basis-configuratie bestaat uit twee vlakke glasplaten verbonden door middel van een polymeerfilm die ageert als tussenlaag. Deze configuratie evolueerde lichtjes in de jaren 1990 door de ontwikkeling van grotere glasgevels en in het bijzonder door de ontwikkeling van nieuwe bevestigingssystemen, voornamelijk verlijmde glazen systemen (eng.: Structural Sealant Glass Systems, SSGS) en verankerde puntverbindingen, binnen de zogenaamde "structurele beglazing" toepassingen. Gelamineerd glas begon geleidelijk aan ook gebruikt te worden in andere toepassingen dan beglazingen, bijvoorbeeld in balustrades, als glazen vinnen en zelfs als dragende platen voor traptreden en vloeren. Met de XXI^{ste} eeuw en de opkomst van meer structurele toepassingen met gelamineerd glas, ontwikkelden de productconfiguraties zich in grotere mate in de richting van multi-gelaagde gelamineerde glasproducten, met onder meer de integratie van metalen tussenstukken en de opkomst van nieuwe producten voor de tussenlaag.

De ontwerpregels en de beoordelingsmethoden van gelamineerde glasproducten en glaswerken ontwikkelden zich parallel, maar met weinig schijnbare consistentie. Naast de evoluties op de markt van glasproducten voor de bouw, werden in Europa andere belangrijke veranderingen geïnitieerd op het gebied van normalisatie met de introductie in 1989 van de Bouwproductenrichtlijn (eng.: Construction Products Directive, CPD) voor de beoordeling van bouwproducten en hun prestaties in bouwwerkzaamheden. De eigenheid van deze richtlijn bestaat erin een nieuwe filosofie te introduceren voor de ontwikkeling van normen op basis van een prestatiegerichte aanpak. Dit leidde tot de ontwikkeling van een reeks normen en richtlijnen als middel voor de uitvoering van de harmonisatiedoeleinden van de CPD, door twee Europese instellingen, namelijk het Europees Comité voor Normalisatie CEN (eng.: European Committee for Standardization) en de Europese Organisatie voor Technische Goedkeuringen EOTA (eng.: European Organization for Technical Assessment). Wegens de parallelle processen en de verschillende betrokken partijen ontstaan er echter een aantal duidelijke inconsistenties in het ontwikkeling zijnde Europese normalisatiekader, althans voor partijen die niet nauw betrokken zijn bij de ontwikkeling.

Deze twee evoluties worden in parallel geanalyseerd. De definities van veiligheidsprestaties en de bijbehorende testmethoden in de productnormen samen met andere technische richtlijnen worden vergeleken en besproken. In het bijzonder wordt ingegaan op de prestatiebeoordeling van gelamineerd glas na breuk. Immers, de beoordeling van de resterende draagcapaciteit van gebroken onderdelen werd steeds belangrijker naarmate niet-conventionele structurele toepassingen werden ontwikkeld. Niettemin houden de veiligheidsprestaties en de beoordelingsmethoden, zoals voorgeschreven in de huidige productnormen

voor gelamineerd veiligheidsglas, impliciet voornamelijk rekening met de traditionele beglazingstoepassingen. De experimentele karakterisering van algemene prestaties na breuk van gelamineerde glasproducten met betrekking tot de verschillende, vage en evoluerende toepassingsgebieden is dus ingewikkeld. Fragmentatie van ontwerp- en goedkeuringsprocessen in product- en project-gerichte ontwikkelingen kan echter leiden tot een ongecontroleerde toename van vereiste testen. Bijgevolg wordt er in het algemeen relatief weinig aandacht besteed aan de karakterisering van producteigenschappen die de mechanische weerstand na breuk van gelamineerde glaselementen bepalen, daarbij in het bijzonder de tijds- en temperatuurafhankelijke mechanische eigenschappen van de tussenlaag. De vergelijking van de begrippen "productfamilies" (de beschouwde omvang van de productconfiguraties voor de beoordeling van de prestaties) en het "beoogde gebruiksgebied" (toepassingsgebied) leidt tot een analyserooster dat een onderscheid maakt tussen verschillende *Toepassingsvelden* (eng.: Application Fields). Onderlinge combinaties helpen om belangrijke componenten met betrekking tot veiligheidsprestaties te detailleren.

Een beter inzicht in de verschillende aspecten van de prestaties na breuk wordt verkregen met behulp van structurele analyse van gebroken systemen, binnen een ontwerpaanpak op basis van bezwijkscenario's. Een inventaris wordt opgemaakt van de parameters die de schadegevoeligheid (kans op breuk van een glas-component) en de schadetolerantie (invloed van schade op de resterende weerstand) van gelamineerde glaselementen bepalen. Er wordt aangetoond dat de overbruggingsfunctie, die gerealiseerd wordt door de tussenlagen in gebroken gelamineerde systemen, kan worden herleid tot twee hoofdmechanismen voor de krachtsoverdracht. Het gedrag in uiterste gebroken toestand wordt voor alle elementen en belastingsconfiguraties gedomineerd door eenzelfde kritiek krachts-overdrachtmechanism, namelijk de ligamentfunctie tussen glasfragmenten. Deze vervult een overbruggingsfunctie dat afhankelijk is van twee aanvullende mechanismen : de delaminatie tussen de tussenlaag en de glassubstraten, en het uitrekken van de tussenlaag. Aannames en voorwaarden worden geïdentificeerd om de beoordeling van de prestaties na breuk in quasi-statische ontwerp-toestanden te onderscheiden van dynamische acties en van de dynamische reactie van het element. Dit leidt tot de veronderstelling dat als een element opeen-volgende accidenten overleeft, de tussenlaagligamenten niet beschadigd zijn. Hierom dient de beschrijving van de fysieke schade van gebroken elementen vervolledigd te worden met initiële delaminatielengtes in de buurt van de scheuruiteinden ter plaatse van het grensvlak tussen de tussenlagen en de gebroken glasfragmenten.

Deze op het structurele niveau gemaakte veronderstellingen laten toe om verder te focussen op de materiaaleigenschappen van de tussenlagen die meewerken in de ligamentreactie. Een overzicht van de belangrijkste kenmerken en eigenschappen van polymeren die gebruikt worden als tussenlaag, laat zien dat ze behoren tot twee families van polymeerproducten, de thermoplasten en de elastomeren. De

eerstgenoemden vertonen in het gebied van de werkingstemperaturen een glas-rubberovergang, terwijl de laatstgenoemden zich meer als ideale rubbers gedragen. In beide gevallen lijkt hun gedrag grotendeels gedomineerd te worden door de secundaire intermoleculaire bindingen en de daaruit voortvloeiende mobiliteit van de moleculaire ketens. Doordat de thermoplasten het meest gebruikt worden in tussenlagen en hun eigenschappen een grotere tijds- en temperatuurafhankelijkheid vertonen, worden de bijbehorende visco-plastische modellen in het domein van grote rekken meer in detail beschouwd. De thermorheologisch eenvoudige of ingewikkelde natuur van het gedrag wordt beschreven en een aanvullend fenomeen wordt geïdentificeerd dat van belang is voor de eigenschappen in de vaste fase, namelijk de *fysische veroudering*. Dit fenomeen blijkt in verband te staan met thermische omkeerbare vorm-veranderingen op moleculair niveau. Het heeft een eerder belangrijke invloed op de weerstand van polymeerproducten tegen kruip van lange duur. Vervolgens worden bijzonderheden betreffende het mechanische gedrag van polymeren die gebruikt worden als adhesieven opgesomd. Er wordt bijgevolg verondersteld dat fysische veroudering de interfaciale- en bulkeigenschappen op een gedifferen-tieerde manier beïnvloedt, en er wordt geconcludeerd dat de uiteindelijke producteigenschappen afhangen van het laminatieproces en de bedrijfs-omstandigheden in onbepaalde en veranderlijke verhouding. Trouwens, met betrekking tot het mogelijke bereik van werkingstemperaturen voor gelamineerde glasproducten, wordt de fysieke verouderinstoestand in werkomstandigheden waarschijnlijk gewijzigd door verouderende en verjongende effecten.

Twee families van tussenlaagproducten worden van dichterbij beschouwd : polyvinylbutyral producten in het algemeen (PVB) en SentryGlas® (SG), een stijver materiaal. De experimentele aspecten en eventueel geschikte proefstuk-configuraties voor het maken van de beoordeling worden besproken. In deze context lijken testen op gelamineerde proefstukken de meest geschikte aanpak. De beoordeling van de tussenlaageigenschappen wordt bijgevolg eerder vanuit het perspectief van een *onderdeel* dan vanuit het perspectief van een materiaal beschouwd. Het bepalen van de meest geschikte testconfiguraties met betrekking tot verschillende doelstellingen wordt dan ook een belangrijk deel van het beoordelingsprobleem.

Proeven op gebroken gelamineerde glaselementen kunnen uitgevoerd worden op verschillende experimentele schalen. Het schaalconcept verwijst niet enkel naar de grootte van de proefstukken, maar meer algemeen naar de verschillende testomstandigheden. Vanwege de gevoeligheid van de polymeercomponent voor combinaties van spanningen en temperatuurseffecten, worden bepaalde beperkingen opgelegd voor de ontwikkeling van testinfrastructuur en test-methoden. Een reeks experimentele campagnen, uitgevoerd op verschillende schalen, wordt geanalyseerd met behulp van een voorgestelde analyserooster met verschillende *Experimentele Onderzoeksvelden* (eng.: Experimental Fields of Investigation). Het belang van deze ontleding is het gebruik ervan voor de

onderscheiding van verschillende technische limieten en oorsprongen van systematische afwijkingen die mogelijk ontstaan tijdens het ontwerp van testopstellingen. Het is echter vooral wanneer het experimentele onderzoeksgebied wordt uitgebreid tot een grotere omvang dat nieuwe systematische afwijkingen en fouten waarschijnlijker worden, en dit met betrekking tot drie categorieën van randeffecten. Randeffecten van de eerste twee categorieën hangen hoofdzakelijk af van praktische en experimentele aspecten. De eerste categorie wordt verbonden aan verwerkingsmethoden van proefstukken, de tweede categorie aan toleranties en meetonzekerheden eigen aan de testconfiguratie en het onderzoeksdomein. Dit soort randeffecten lijkt potentieel een meer significante effect te hebben op proefstukken van kleine afmetingen. De derde categorie van randeffecten houdt rekening met afwijkingen als gevolg van de analyse en de verwerkingsmethoden van de resultaten, en van de propagatie van onzekerheden. Het overzicht helpt ook bij de identificatie van typische technische kwesties die voortkomen uit de vermindering van de onzekerheden bij een combinatie van onderzoeksvelden, in het bijzonder wat betreft het gebruik van optische meetmethoden in combinatie met een klimaatkamer.

De TCT-test (eng. Through Crack Tensile test) wordt verder beschouwd als referentie testconfiguratie om de combinatie van effecten van testtemperatuur en belastingsniveau op het gedrag van gelamineerde proefstukken met een SG-tussenlaag, bij twee belastingsmodi, te onderzoeken. Een uniek monster van ongeveer 60 proefstukken gelamineerd glas met unieke configuratie (soort tussenlaagsproduct, dikte en verwerkingsmethode) wordt gebruikt om een incrementele experimentele strategie te ontwikkelen. Deze berust op reeksen van proeven van relatief korte duur, uitgevoerd met een constante verplaatsingssnelheid (eng. constant displacement rate – cdr) en een kruipbelasting met quasi-statische belastingsvoorwaarden. De incrementele aanpak maakt het mogelijk om een overzicht te krijgen van het gedrag binnen een relatief groot bereik van testomstandigheden, namelijk voor testtemperaturen begrepen tussen –20 en +60°C. De experimentele aspecten van de uitgevoerde proeven in een klimaatkamer en de bijbehorende onzekerheden worden vervolgens kritisch geanalyseerd. In het bijzonder worden de limieten van de meetnauwkeurigheid van optische vervormingsmeetmethoden geëvalueerd in vergelijking met andere experimentele aspecten.

Twee belangrijke vervormingspatronen werden waargenomen tijdens de proeven bij verschillende testomstandigheden, met name een regelmatig delaminatiepatroon en een scheurgroeipatroon doorheen de ligamentdikte. De bewzijkmode blijkt zeer afhankelijk te zijn van de testomstandigheden. De cdr-belastingsmode lijkt niet zo nuttig voor het beschouwde ontwerpprobleem, aangezien er geen volledige overeenkomst met de kruipbelastingsmode kan worden gevonden. Dit is voornamelijk te wijten aan de variabele bijdrage van de twee vervormingsmechanismen aan de vervormbaarheid van de TCT-proefstukken. Het proefprogramma liet ook toe om de invloed van de bewaartijd van de proefstukken op

hun mechanisch gedrag, die aan een effect van fysische veroudering wordt toegeschreven, aan te tonen. Dit significante effect, waargenomen op experimentele tijdschaal, benadrukt een probleem voor de interpretatie van testresultaten op een kwantitatief relevante manier. Het waargenomen macroscopische gedrag bij de TCT-testresultaten komt overeen met een thermorheologisch complex gedrag van de visco-plastische reactie, met significante verschillen in de respons binnen het experimentele onderzoeksgebied.

De belangrijkste resultaten van deze thesis onderstrepen de noodzaak om de experimentele benaderingen voor de beoordeling van tussenlaagonderdelen van gelamineerde glasproducten in vergelijking met andere bouwmaterialen aan te passen. Ook de moeilijkheden voor het verkrijgen van kwantitatief zinvolle resultaten voor de ontwerppraktijk, komt in de resultaten duidelijk naar voor. Met de gerapporteerde experimentele werken kon een gevoeligheidsanalyse uitgevoerd worden op een aantal experimentele parameters die zelden in rekening worden gebracht. Deze parameters blijken echter een merkbare invloed te hebben op de testresultaten. Proeven op intermediaire schaal en TCT-testen in het bijzonder blijken nuttig te zijn om vraagstukken, verbonden aan het tijds- en temperatuurafhankelijke gedrag van de tussenlaag, te isoleren ten opzichte van de sterkte van glazen onderdelen. Ten slotte worden ook de intermediaire experimentele schalen geanalyseerd vanuit een sectoraal perspectief en worden reflectiepaden aangeboden met betrekking tot de ontwikkeling van "geharmoniseerde" rekenmodellen en met betrekking tot implementatiekwesties van karakteriseringmethoden in de praktijk.

Résumé

Les années 1980 voient l'introduction du verre feuilleté dans le secteur de la construction en tant que vitrage de sécurité pour la protection des personnes en cas d'impact accidentel. La composition de base, faite de deux feuilles de verre assemblées autour d'un intercalaire en polymère, évolue dans le courant des années 1990 parallèlement au développement d'applications de plus grandes dimensions, dits "vitrages structuraux". Ceux-ci se caractérisent par l'apparition de nouveaux système de fixation, principalement sous forme de vitrages extérieurs collés (VEC) et de vitrages extérieurs attachés (VEA). Progressivement le verre feuilleté commence à être aussi utilisé pour des applications autres que des vitrages et de simples éléments de remplissage, comme élément de garde-corps, comme raidisseur en verre et finalement comme élément porteur dans des escaliers et des planchers en verre. Avec le début du XXI^{ème} siècle et l'émergence d'applications en verre structural, la configuration des produits en verre feuilleté évolue davantage vers des produits multi-feuilletés, avec entre autres l'intégration d'inserts métalliques et l'apparition de nouveaux produits pour les intercalaires.

Les règles de conception des ouvrages en verre et les méthodes d'évaluation des produits en verres feuilleté évoluent en parallèle, mais sans toujours beaucoup de cohérence apparente. A côté des évolutions sur le marché des produits en verre feuilleté pour le bâtiment, le secteur de la construction connait d'autres changements importants dans le domaine de la normalisation au niveau européen, avec l'introduction en 1989 de la Directive Produits de Construction (DPC ou CPD en anglais). Cette directive a pour particularité d'introduire une nouvelle philosophie pour l'expression et l'évaluation des performances des produits utilisés dans les ouvrages de construction, reposant sur une approche dite performantielle. Ceci a conduit à l'élaboration d'une série de normes et guides techniques émis respectivement par deux organismes européens, le CEN (Comité Européen de Normalisation) et l'EOTA (Organisation Européenne pour l'Agrément Technique), destinés à soutenir et implémenter les objectifs d'harmonisation formulés par la CPD. La mise en œuvre de nombreux processus de développement parallèles et l'implication d'un grand nombre d'intervenants ont toutefois conduit au développement d'un cadre normatif européen présentant certaines incohérences manifestes, au moins en apparence et pour des acteurs non impliqués de manière directe dans ces développements.

Ces deux évolutions, technologique et normative, sont analysées en parallèle. La formulation de performances de sécurité dans les normes produits et dans d'autres guides techniques sont comparées et analysées avec les méthodes d'essai destinées à les évaluer, et plus particulièrement les performances consécutives au bris des composants verriers. En effet, l'évaluation de la capacité portante d'éléments brisés devient un point particulièrement critique quand ils sont utilisés dans des applications structurales non-conventionelles. Pourtant, les

performances de sécurité et les méthodes d'évaluation telles que prescrites dans les normes produit actuelles pour les verres feuilletés de sécurité sont encore toujours implicitement principalement basées sur leur utilisation en tant que vitrage. Une formulation générale des performances résiduelles en cas de bris apparait donc comme un problème assez complexe, tout comme la mise au point de méthodes de caractérisation expérimentales correspondantes. Cette difficulté est notamment due à des domaines d'application des produits variés et évolutifs, et dès lors délimités de manière plutôt vague. Par ailleurs, un risque de croissance incontrôlée du nombre d'essais requis apparaît, dû à la division des processus de conception et d'évaluation, entre d'une part une approche projet de la conception des ouvrages, et d'autre part le développement de procédures d'évaluation davantage orientés produit. Une conséquence directe est que la caractérisation des propriétés des produits jouant un rôle dans la capacité portante résiduelle en cas de bris fait l'objet d'assez peu d'attention, et en particulier en ce qui concerne la variation en fonction de la température et du temps des propriétés mécaniques des composants d'intercalaire. Une comparaison des concepts de "famille de produits" (l'ensemble des configurations de produit visé pour l'établissement des performances) et de "domaine visé d'utilisation" (domaine d'application) a conduit à proposer une grille d'analyse constituée de *Champs d'Application* (Application Fields en anglais), visant à distinguer les différentes composantes qui, combinées entre elles, participent aux performances de sécurité.

L'analyse structurale de systèmes brisés au moyen de scénarios de défaillance permet de mieux cerner les différents aspects relatifs à leurs performances résiduelles. Un inventaire est fait des paramètres qui gouvernent la sensibilité aux dégâts d'éléments en verre feuilleté (soit le risque de rupture des composants verriers) et leur tolérance aux dégâts (soit les conséquences de tels bris). L'analyse montre que la fonction de liaison assuré par l'intercalaire au sein de systèmes feuilletés fracturés peut se réduire à deux mécanismes de transfert de charge principaux. Un de ces deux mécanismes consiste en la formation d'un ligament qui se développe entre différents morceaux de verre. Ce ligament apparaît comme le mécanisme de transfert de charge critique pour le comportement d'éléments en état de rupture avancé, et ce quels que soient les configurations géométriques et états de chargement considérés. La fonction de liaison assurée par le ligament d'intercalaire repose à son tour sur deux mécanismes complémentaires : une délamination entre l'intercalaire et les fragments de verre, et un étirement du ligament ainsi formé. Des hypothèses apparaissent nécessaires pour pouvoir dissocier l'évaluation des performances résiduelles en régime quasi-statique des situations de chargement ou de réaction en régime dynamique. Cela conduit à supposer que si un élément survit à l'augmentation des dégâts due à la fragmentation progressive des feuilles de verre, cela se fait sans endommager le cœur des ligaments. La conséquence de cette hypothèse est la nécessité de compléter la description du niveau de dégâts physique d'un élément feuilleté fracturé au moyen de longueurs initiales de

délamination à partir des extrémités des fissures dans les feuilles de verre, au niveau de l'interface entre intercalaire et fragments de verre.

Ces hypothèses faites au niveau structural permet de se concentrer ensuite plus particulièrement sur les propriétés des intercalaires gouvernant le comportement mécanique du ligament. L'analyse des caractéristiques des polymères utilisés comme intercalaire montre qu'on peut distinguer deux catégories de matériaux, les thermoplastiques et les élastomères. Les intercalaires du premier type présentent une température de transition vitreuse comprise à l'intérieur de l'intervalle de températures d'utilisation, tandis que ceux du deuxième type présentent un comportement plus proche d'un caoutchouc idéal. Dans les deux cas, le comportement mécanique semble dominé par les liaisons secondaires entre chaînes moléculaires et la mobilité des chaînes qui en résulte. Les matériaux intercalaires les plus courants appartiennent à la première catégorie et justifient de regarder de plus près les modèles visco-plastiques en régime de grande déformation, décrivant la dépendance du comportement aux effets de durée de chargement et de température. Les comportements thermorhéologiques simple et complexe sont décrits, et un phénomène important supplémentaire est identifié pour l'état solide vitreux, appelé *vieillissement physique*. Il s'agit d'un phénomène associé à des changements de conformation réversibles thermiquement, qui a une grande influence sur la résistance au fluage de longue durée des matériaux polymères. Finalement, un aperçu est donné des particularités du comportement mécanique des polymères utilisés comme adhésifs. Le vieillissement physique est dès lors supposé pouvoir affecter de manière différentielle les propriétés mécanique d'une couche d'adhésif dans sa masse et le long de ses interfaces. Par ailleurs, ses propriétés mécaniques peuvent varier en fonction des conditions de lamination et d'utilisation dans des proportions indéterminées. Enfin, au vu des intervalles de température d'utilisation typiques des produits en verre feuilleté, on peut s'attendre à ce que leur état de vieillissement physique puissent induire des processus de vieillissement et de rajeunissement au cours de leur durée d'utilisation.

Deux familles d'intercalaires sont ensuite considérés plus en détails, les intercalaires en polyvinyl butyral (PVB) en général, et le SentryGlas® (SG), un intercalaire plus rigide. Les aspects expérimentaux sont analysés selon le type et la configuration des éprouvettes d'essai, et les essais réalisés sur éprouvettes en verre feuilleté semblent plus appropriés dans le cadre de procédures d'évaluation des propriétés des produits finis. En d'autres mots, il parait préférable de réaliser l'évaluation des propriétés des intercalaires en les considérant davantage comme des *composants* que comme des matériaux. La détermination des configurations d'éprouvettes et des configurations d'essai les plus pertinentes par rapport à différents objectifs apparaît dès lors comme un aspect essentiel des processus d'évaluation.

Les essais sur éléments fracturés peuvent être réalisés à différentes échelles expérimentales. Cette notion d'échelle expérimentale ne se limite pas à des différences de dimensions des éléments testés, mais plus généralement à différents 'sets' de conditions d'essai. La sensibilité du comportement de ces polymères à la combinaison d'effets de température et de contrainte induit des contraintes importantes pour le développement d'infrastructures et de méthodes d'essai. Une série de campagnes expérimentales sont analysées au moyen d'une grille d'analyse distinguant différent *Champs d'Investigation Expérimentales* (Experimental Fields of Investigation en anglais), destinés à identifier différentes limites techniques et sources d'incertitudes systématiques susceptibles de survenir au cours de la conception de configurations d'essai. En effet, c'est surtout quand le domaine d'investigation expérimentale est étendu que de nouvelles sources d'incertitudes et d'erreurs systématiques risquent d'apparaître, qui peuvent être associées à trois catégories d'effets de bord. Les effets de bord des deux premières catégories sont associés essentiellement à des aspects expérimentaux et pratiques, relatifs aux méthodes de fabrication des éprouvettes d'essai pour la première catégorie, et aux tolérances expérimentales et incertitudes de mesure propres aux configurations d'essai et aux intervalles d'investigation pour la seconde. Ces effets de bord apparaissent comme potentiellement de plus grande ampleur pour les éprouvettes d'essai de plus petites dimensions. Les effets de bord de la troisième catégorie prennent en compte les sources de déviation dues aux méthodes d'analyse et aux propagations d'incertitudes de mesure. Une analyse globale permet la mise à jour de problèmes spécifiques pour réduire des incertitudes sur différents champs d'investigation simultanément. Ceci est illustré en particulier par le cas de l'utilisation de méthodes de mesure optique en combinaison avec l'utilisation d'une chambre climatique.

Les essais TCT (Through Crack Tensile tests) sont ensuite considérés comme configuration de référence pour investiguer expérimentalement la combinaison des effets de la température d'essai et du niveau de contrainte sur le comportement ligamentaire, pour un verre feuilleté constitué d'un intercalaire SG et pour deux modes de chargement différents. Une stratégie expérimentale incrémentale est développée sur base d'un seul échantillon, constitué d'une soixantaine d'éprouvettes en verre feuilleté, ayant toute la même configuration (en termes de type et épaisseur d'intercalaire, et de méthode de fabrication). Il s'agit d'essais de relativement courte durée en régime quasi-statique, comprenant des séries d'essais réalisés à vitesse de déplacement constante (essai cdr) et des séries d'essais de fluage. L'approche incrémentale permet d'obtenir un aperçu du comportement mécanique sur un domaine relativement étendu en termes de conditions d'essai, avec des températures comprises entre –20 et +60°C. Les aspects expérimentaux relatifs aux essais réalisés en chambre climatique et les incertitudes de mesure associées sont ensuite examinés de manière critique. En particulier, une estimation est faite des limites relatives à la justesse des mesures

de déformation au moyen de méthodes optiques, et celles-ci sont comparées à d'autres incertitudes expérimentales en présence.

Deux modes de déformation principaux ont été observés durant ces essais pour les différentes conditions d'essai considérées : un mode de délamination régulière et un mode de propagation de fissure à travers l'épaisseur du ligament. Il apparait clairement que le mode de rupture dépend significativement des conditions d'essai. A l'issue de cette campagne, les essais de type cdr apparaissent finalement d'intérêt plutôt limité par rapport aux situations de projet considérées, vu qu'une correspondance complète avec les essais de fluage n'a pu être établie, et ce principalement à cause d'un rapport variable des contributions des deux mécanismes de déformation (de délamination et d'étirement) à la ductilité globale du ligament en configuration d'essai TCT. Cette campagne d'essais a permis également de pointer l'influence significative, à l'échelle de temps expérimentale, de la durée de conservation des éprouvettes sur leur comportement, attribuée au phénomène de viellissement physique ; ceci met en évidence une problématique importante pour une interprétation quantitativement pertinente de résultats d'essais. Sur base du comportement tel qu'observé et analysé au moyen de ces essais TCT, le comportement visco-plastique semble correspondre à un comportement de type thermorhéologique complexe, analysé toutefois à un niveau macroscopique, et avec des différences de comportement significatives sur le domaine d'investigation considéré.

Les principaux résultats de cette thèse mettent en évidence la nécessité d'adapter les stratégies d'évaluation expérimentale pour les produits en verre feuilleté, en comparaison avec d'autres produits de construction, et des difficultés spécifiques pour obtenir des résultats quantitativement pertinents pour le dimensionnement d'applications en pratique. Les essais réalisés ont permis de faire une analyse de sensibilité pour quelques paramètre expérimentaux rarement pris en compte, et qui pourtant sont prouvés avoir une influence significative sur les résultats d'essais. Cette recherche a également souligné l'intérêt d'essais 'à échelle intermédiaire', et des essais TCT en particulier, en vue d'isoler l'étude du comportement thermorhéologique de l'intercalaire de questions liées à la résistance des composants verriers. Pour finir, l'utilité d'essais associés à des échelles expérimentales intermédiaires est également analysé d'un point de vue sectoriel, et quelques pistes de réflexion sont données par rapport au développement de méthodes de dimensionnement "harmonisées" et aux questions d'implémentation de méthodes de caractérisation en pratique.

Symbols

$a(\bullet)$	measurement range (defined by limiting values)
a	delamination length (interfacial crack) / regression parameter
$\dot{a}$	delamination rate (propagation velocity of crack front)
a_0	initial interfacial delamination length
a_s	short crack limit of interfacial delamination length
$a_T(T)$	temperature dependent shift function
$a_\sigma(\sigma)$	stress dependent shift function
b	width (specimen) / regression parameter
d	(pre-)crack opening / displacement
d_{cr}	creep (pre-)crack opening $\left(d_{cr}(t) = d_{opt}(t) - d_{ini}\right)$
d_i	value of (pre-)crack opening for isometric curve (TCT-test)
d_{ini}	initial (pre-)crack opening (initial loading step creep test)
d_{opt}	measured (pre-)crack opening by optical method
d_{tb}	displacement transversal beam testing device
$\dot{d}$	crack opening rate / displacement rate
h	height / half-thickness of interlayer (TCT-test)
k	Boltzman constant $\left(k = 1.381.10^{-23}\ \mathrm{J/K}\right)$
ℓ_{act}	length of activation of a load-transfer mechanism
$\ell_{\mathrm{ff\text{-}ic}}$	distance between a (initial) cracked section and the far stress field
$\ell_{\mathrm{gl,min}}$	lower threshold value for planar dimensions of glass fragments
r	(radius) / size of process zone (near a crack tip)
r_n	relative residual resistance (fractured state at damage level *n*)
r_p	size of the plastic (or inelastic) zone (near a crack tip)
t	thickness (of a layer / interlayer / glass sheet) / time

t_a	annealing duration / initial ageing time
t_f	time-to-failure (creep)
t_{eff}	effective ageing time
t_{gl}	thickness of a glass component
t_{int}	thickness of a interlayer component
$u(\bullet)$	measurement uncertainty
v	velocity / displacement rate
A	cross-section area (uniaxial tensile test)
A_0	initial cross-section area (uniaxial tensile test)
D_s	structural damage (parameter / concept)
D_φ	physical damage (parameter / concept)
E	(axial) elastic modulus
F	force / load
F_{cr}	applied force in creep test
$F_{\max}$	(measured) peak force (in cdr-test)
F_{ss}	steady-state force (constant value of force in steady-state response)
G	shear (elastic) modulus
G_r	strain hardening modulus
K_{Ic}	(static) fracture toughness (crack opening mode I)
K_{Id}	dynamic fracture toughness (crack opening mode I)
L	gauge length (uniaxial tensile test)
L_0	initial gauge length (uniaxial tensile test)
R	universal gas constant $\left(R = 8.314472\ \mathrm{J/(K.mol)}\right)$
R	(radius) / size of the stress crack tip field (near a crack tip)
R_n	residual resistance (fractured state at damage level *n*)

R_0	initial resistance (undamaged state)
S	(physical) ageing state
$S_a(t)$	initial (physical) ageing state function
$S_{a,B}(t)$	bulk ageing state function (concept)
$S_{a,I}(t)$	interfacial ageing state function (concept)
T	(test) temperature
T_0	reference test temperature
T_a	annealing temperature (temperature of thermal pre-treatment)
T_g	(primary) glass-rubber transition temperature
T_m	melt temperature
T_{ref}	reference temperature
$T_{\max}$	maximal temperature of use
V^*	activation volume
δ	half-crack opening (TCT-test)
ε	(axial) strain
ε_{cr}	critical plastic strain
ε_s	axial strain (short crack limit)
ε_u	strain at breakage
ε_x	axial nominal strain
$\dot{\varepsilon}$	(axial) strain rate
$\dot{\varepsilon}_{\min}$	secondary creep rate (minimum creep rate)
$\dot{\varepsilon}_f$	creep rate at failure (maximum creep rate)
$\dot{\varepsilon}_0$	process rate constant (pre-exponential coefficient)
λ	(axial) stretch
λ_x	nominal axial stretch

η	viscosity
η_0	zero-viscosity
ν	Poisson's coefficient
σ	(axial) (nominal / true) stress
σ_y	yield stress
σ_r	strain hardening stress
$\sigma_{rej,0}$	rejuvenated yield stress
σ_s	driving stress
σ_u	tensile strength
σ_x	axial nominal stress
σ_0	characteristic stress
Δd_0	initial clearance (testing device)
ΔF	step (increment) value of force
ΔU	activation energy
$\Delta\sigma_y$	yield drop (stress component)
Γ	strain energy release rate (crack extension force)
Γ_c	(static) crack resistance (fracture toughness)
Γ_d	dynamic crack resistance
Γ_0	interfacial (static) (fracture) toughness
I, II, III	fractured stage / damage level / creep mode / crack opening mode

Acronyms

ANB	Annexe Nationale – Nationale Bijlage (Belgian National Annex to Eurocodes)
AF	Application Field (concept in this thesis)
BBRI	Belgian Building Research Institute www.bbri.be
BP	breakage point (TCT-test)
cdr	constant displacement rate (loading mode)
CE	CE-marking (of products)
CEN	European Committee for Standardiation www.cen.eu
COST	European Cooperation in Science and Technology www.cost.eu
CP	crack propagation (deformation / failure pattern TCT-test)
CPD	Construction Products Directive (European Directive 89/106/EEC)
CPR	Construction Products Regulation (European Regulation 305/2011)
CSTC	Centre Scientifique et Technique de la Construction (= BBRI)
CST	Compressive Shear Test (test configuration)
EFI	Experimental Field of Investigation (concept in this thesis)
EN	European standard (letter code)
ENV	experimental European standard (letter code used among others for first generation Eurocodes)
EOTA	European Organization for Technical Assessment www.eota.eu
ESD	Element Safety Diagram (concept)
ETA	European technical agreement / European technical assessment
ETAG	Guideline for European technical agreement
EVA	ethylene vinyl acetate (a family of polymers used as interlayer products)
FEM	Finite Element Method (numerical calculation method)
FM-D	Dynamic Failure mode (concept in this thesis)
FM-QS	Quasi-static Failure mode (concept in this thesis)
FPC	Factory production control (concept)
hEN	harmonized product standard (European standard used as basis for CE-marking of construction products)
IABSE	International Association for Bridge and Structural Engineering www.iabse.org
ISO	International Organisation for Standardisation www.iso.org
ITT	Initial Type Testing (concept)

LEFM	Linear Elastic Fracture Mechanism (model theory)
LN^2	liquid nitrogen
LMO	Laboratory for Research on Structural Models (research unit UGent)
LS	Longitudinal Shear (mechanism configuration)
LTM	Load Transfer Mechanism (concept)
MMS	Mechanics of Materials and Structures (research unit UGent)
NBN	Bureau for Standardisation (Belgian standardisation body) www.nbn.be Belgian standard (letter code)
NDP	Nationally Determined Parameters (in Eurocodes)
NIT	Technical document (FR), published by the BBRI (sectorial 'standard')
NA	National Annex (to Eurocodes, generic acronym)
OCT	Offset Crack Tensile (test / specimen / mechanism configuration)
PC	polycarbonate (family of polymers)
prEN	draft European standard (letter code)
PMMA	polymethylmethacrylate (family of polymers)
PVB	polyvinyl butyral (a family of polymers used as interlayer products)
PVC	polyvinyl chloride (family of polymers)
RD	regular delamination (deformation / failure pattern TCT-test)
SG	SentryGlas® (brand name for a stiff thermoplastic interlayer of DuPont)
SSGS	Structural Sealant Glazing System
STS	Unified technical specifications (Belgian specifications for construction works), published by the FPS Economy
TC	Technical Committee
TP	test purpose (category)
TCT	Through Crack Tensile (test / specimen / mechanism configuration)
TST	Tensile Shear Test (test configuration)
TU/e	Eindhoven University of Technology www.tue.nl
TV	Technical document (NL), published by the BBRI (sectorial 'standard')
UGent	Ghent University www.ugent.be
uPVC	unplasticised poly(vinyl chloride) (family of polymers)
WTCB	Wetenschappelijk en Technish Centrum voor het Bouwbedrijf (= BBRI)

Table of contents

(detailed content of chapters on next page)

Content of chapters

Chapter I - Assessment of laminated safety glass products for structural applications

Chapter II - Performances and properties of fractured laminated glass elements

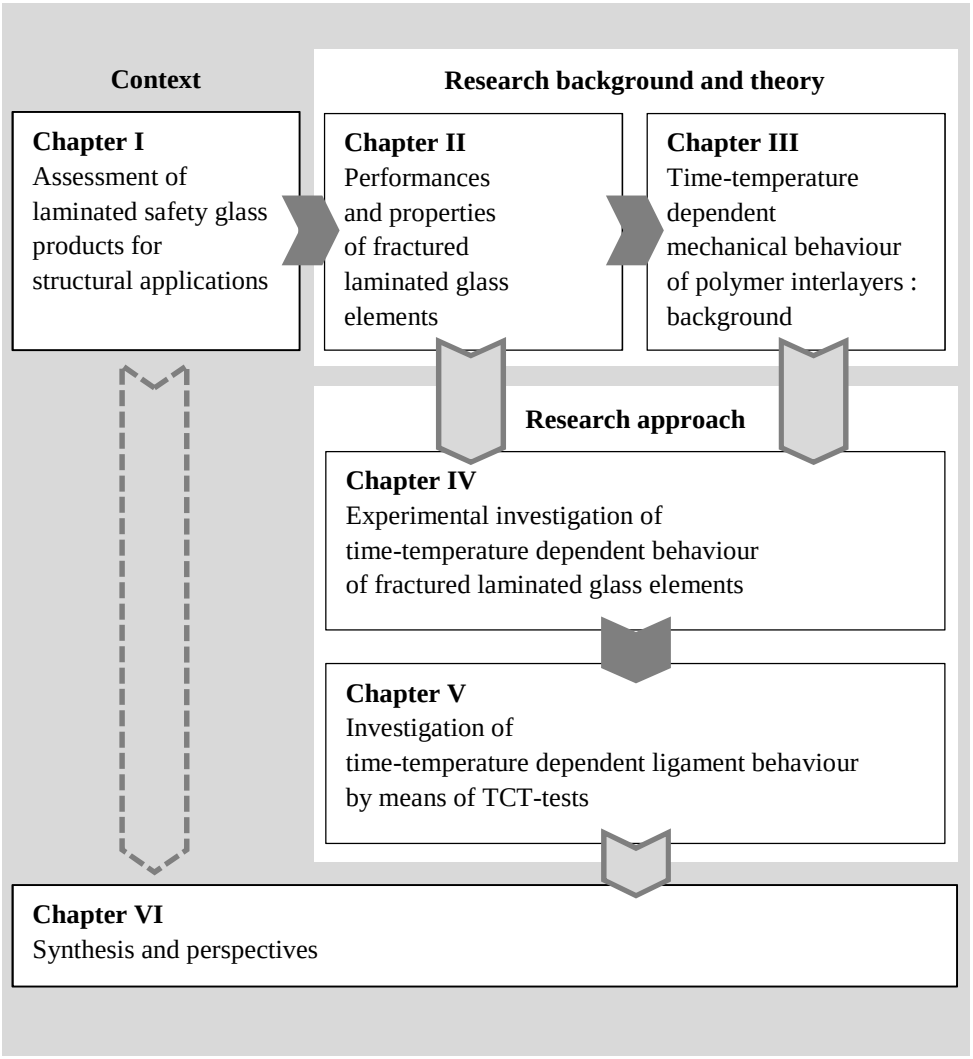
Chapter III - Time-temperature dependent mechanical behaviour of polymer interlayers : background

Chapter IV - Experimental investigation of time-temperature dependent behaviour of fractured laminated glass elements

Chapter V - Investigation of time-temperature dependent ligament behaviour by means of TCT-tests

Chapter VI - Synthesis and perspectives

Outline of the thesis



Chapter I

Assessment of laminated safety glass products for structural applications

"Europe will not be made all at once, or according to a single plan.
It will be built through concrete achievements which first create a de facto solidarity."
(Schuman Declaration of 9 May 1950)

I.1. Introduction

Laminated glass products are basically assemblies of two or more flat glass sheets around and by means of polymer interlayers, providing this category of glass products with interesting safety properties. Introduced in the automobile sector, laminated glass products conquered the construction industry for being used as safety glazing unit. Continuous technological developments, among others in polymer materials used as interlayers and in lamination processes, led to develop a variety of laminated glass products complying to various types of safety performances. Simultaneously, the ranges of use develop to different concepts of structural glazing systems up to impressive structural applications nowadays.

The technological evolutions were accompanied and supported by the development of safety concepts, and of assessment, design and calculation methods, in an attempt to follow the trends and ambitions of architects and designers. In parallel of these evolutions peculiar to laminated glass products in the building industry, the adoption at European level of the Construction Products Directive in 1989 shaped a new standardization philosophy. It introduced a performance-based approach for the assessment of construction products and construction works, and aims at developing 'harmonized' product standards and design codes. The development of a European design code for glass construction works is meanwhile a work item on the agenda of the CEN, the European Committee for Standardization.

However, in this process, inconsistencies appear between different types of co-existing standards. Challenges and difficulties are arising in accordance for developing new design codes and associated experimental assessment methods, in relation with a variety of closely inter-related technical, practical and conceptual issues. Current design methods and concepts still fail to address some important issues concerning the characterization of interlayer components for design purposes, in particular in relation with their time-temperature dependent behaviour. A particularly important aspect concerns the assessment of their contribution to residual load-bearing performances of non-conventional structural applications in laminated glass, in case of accidental breakage of the glass sheets.

Therefore a relative detailed analysis of the European standardization framework and an explanation of related concepts is proposed in this first chapter, in parallel of the recent evolutions in laminated glass products and applications. It leads to a proposition for describing the various components behind the concepts of 'family of products' and 'intended fields of use', in order to account for particularities of design processes in this particular field. The analysis is terminated with the formulation of a series of central questions : how to assess the contribution of interlayers to safety and post-fracture performances of laminated glass elements, and how to express these in terms of design values usable in combination with appropriate design and calculation methods ?

I.2. Short history of laminated glass products in construction works

Among the construction materials, glass occupies a quite special place. It is transparent, it exhibit chemical and physical properties quite stable in time, it is recyclable, but it is brittle, namely it breaks without warning signs. For a long time it had no competitor showing as many advantages together with a good transparency, and therefore is used in buildings since hundreds of years as glazing set in some frame. Many technological innovations improved the quality and the flatness of flat glass, especially in the XIXth and XXth centuries, to lead to the currently most used industrial process, the float process. However, these successive changes in the production process of flat glass panes did not change much to its brittleness.

A first improvement to reduce the risk of injury in case of glass breakage came in 1874 with the tempering process (Speelman and Savineau 2013). This enabled to produce thermally toughened glass [1] with two important improvements in comparison to 'usual', annealed float glass units : a higher strength and a safer breakage pattern in the form of very small splinters. It allowed to reduce the risk of fatal injuries in case of human impact against glazing, but with no retaining function in case of glass breakage.

Another innovation to compensate the unsafe brittle behaviour of glass followed shortly after, thanks to its association with another new transparent material, polymer, which appeared consecutively to the development of the oil industry[2]. In the early XXth century, the first "layered glass" product was invented[3], namely a piece of glass secured by a plastic layer to avoid splitting in case of glass breakage. Besides its ability to retain the glass fragments bonded to it in case of

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[1] The term "thermally toughened" is used for a category of glass products whose increased strength is obtained by means of a thermal treatment inducing compressive stresses along the outer faces. In comparison, "chemically toughened" glass products are strengthened by means of a chemical process, obtained by an exchange of K^+ ions of a smelted salt ($KN0_3$) with the smaller Na^+ ions of the glass pane. Chemically toughened glass products are not usual in building applications. Tempering processing of float glass products transform these into "heat-strengthened" or "thermally toughened" (safety) glass products, according to the grade of toughening, essentially due to difference in cooling rate of the tempering process. This involves also differences in typical fragmentation patterns; in that regard thermally toughened glass is judged safer than the two other toughened glass products, thanks to its splintering in much smaller glass fragments.

[2] Polymers were accordingly initially limited to organic materials. In the meantime, other non-organic polymer products were developed, as silicones. See Chapter III for more details about specificities of polymer materials.

[3] "Layered glass" or "Safety glass" is known to have been invented in 1903 by a chemist, Edouard Benedictus, who accidentally dropped a glass flask containing cellulose nitrate and discovered by this way the favourable post-fracture performances of a piece of glass secured by a plastic layer. A patent is introduced in 1909 for this finding.

breakage of the glass sheet(s), the concept appeared to provide also some containing capacity to a glazing unit, namely a capacity to prevent an impacting body to pass through it.

The latter was a decisive advantage upon toughened glass[4] to introduce and develop the concept further as front windscreen in cars during the 1930's, to improve the safety in case of accident : it helped not only to prevent critical injuries to the driver's or passenger's head in case of impact against the windscreen consequent to an accident, but also the ejection out of the vehicle of human's body through the windscreen[5]. The basic configuration of two flat glass panes adhesively bonded on either side of one plastic sheet (or interlayer) appeared as the most favourable one for such large planar glass elements, and led to the widely used term "laminated glass". In this configuration, the harder outer glass sheets protect the softer plastic interlayer, increasing the durability of the resulting product. Polyvinyl butyral (PVB) gained a dominant position as interlayer material in the laminated glass industry, among others thanks to its good processability (ease of processing) and the resulting toughness of laminated glass units.

Laminated glass found its way towards the building industry[6] for fulfilling similar functions, and became widely used as safety glazing during the 1980's. Other types of laminated glass products were then developed in order to achieve more demanding performances, as for burglar resistant (burglar retardant) glazing, bullet-resistant glazing, attack resistant glazing,... Other evolutions in composition of laminated glass units were related to other interesting qualities, as acoustic insulation, and the possibilities to integrate complementary components or features at the level of the interlayer useful to other safety and non-safety functionalities, related to fire resistance, energy, light control or aesthetics[7].

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[4] Besides, the increased strength of toughened glass appears in some cases as a potential source of injury : its higher strength can prevent its breakage during the impact, in which case an important force can be returned back to the impacting body during the elastic rebouncing. This is because the absorbed energy is not dissipated by the glass plate, but stored in the form of elastic deformation. For the same reason, a larger amount of energy is released at breakage. This is a potential issue when thermally toughened glass is part of a laminated glass unit (see also Chapter II paragraphs II.2 and II.3).

[5] It is useful to remind that laminated glass has been introduced for use in windscreens of cars in 1944, years before the invention of the seat belt (1958) and before the invention (in the 1970s) and wide introduction (late 1980s – early 1990s) of the airbag (Wikipedia: Volvo cars; airbag).

[6] Laminated glass began to be widely used as safety glazing in buildings during the 1980's (www.editions-ti.fr, Dossier N4404 "Intercalaires pour verres feuilletés", Gérard Savineau).

[7] For instance, fire-resistant glazing is obtained by means of an intumescent interlayer, electro-chromatic glazing by including liquid crystals in the interlayer (which orientation controlled by the application of an electric tension leads to transparent or translucent state), and some type of heating glazing by means of very thin wires included in the interlayer (working as electrical resistant element, for instance used for automatic de-icing of front windscreen in cars ...).

Extensions of product performances were provided by three kinds of modifications with respect to the basic composition[8] of 'simple' laminated glass unit :

1) adaptation of the overall layers composition, namely by changing the amount and thickness of the constitutive layers;
2) change of the type of interlayer material (grade or product)[9], and/or of lamination process;
3) change of the type of one or more glass layers, for instance by replacing one or more annealed float glass sheets by a toughened glass sheet or by another transparent plastic material.

These technological developments of laminated glass units were accompanied by an increase of their dimensions, altogether with evolutions in their use. In a first evolution step, traditional glazing setting in a closed frame evolved towards the development of the so-called 'structural glazing' applications in facades, corresponding mainly to a variety of alternative fixation systems. The requirements on safety and load-bearing capacity of the products increased progressively, in combination with primary performance requirements related to the glazing function[10]. The second evolution step is the use of laminated glass products in applications with no glazing function anymore, with a progressive increase of the "structural" role, from the use in various configurations of balustrades, glass fins in facades for reinforcing the stiffness against wind action, to "true" structural applications, glass floors and glass beams for instance. In the latter, mechanical and safety performances are overwhelming the design.

These parallel evolutions are illustrated in Figure I.1. They basically address two interlaced developments, on the one hand the development of laminated glass products, and the development of calculation, testing and construction methods.

The product development involved development of larger production facilities and evolutions in manufacturing methods. The control of the lamination process remains a central point of attention in all cases, as it will appear further in the text. However, production aspects will not be extensively discussed in the present work.

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[8] In this text, *composition* of laminated glass refers to the amount, order, thickness and characteristics of constitutive glass sheets and interlayers (or configuration "along the thickness"), whereas *configuration* is used in a more general sense, including also all other characteristics of the laminated glass unit (planar dimensions,…). Note that configuration is also used in the above restrictive sense of composition in some contexts. It will appear as an important concept for defining 'family of (laminated glass) product' (in sections I.4 and I.5).

[9] Grade refers to a variant of a same product, product refers to another type of product; see also Chapter III.

[10] The concept of glazing function is defined more precisely in paragraph I.5.1.

The corresponding innovations in designing new applications, in terms of configurations and functions, followed each other at an increased tempo during the last decade; however safety concepts and corresponding design and assessment methodologies seem to have progressed more slowly. Especially, the historically older "glazing" design methodology and the more recent "structural design" approach of glass elements still seem to conflict with each other. The reasons explaining these apparent contradictions are various, and deserve an attempt at being understood, accounting for technical and scientific improvements, but also for some economic and industrial aspects. It is necessary to understand some issues arising in ongoing standardization development.

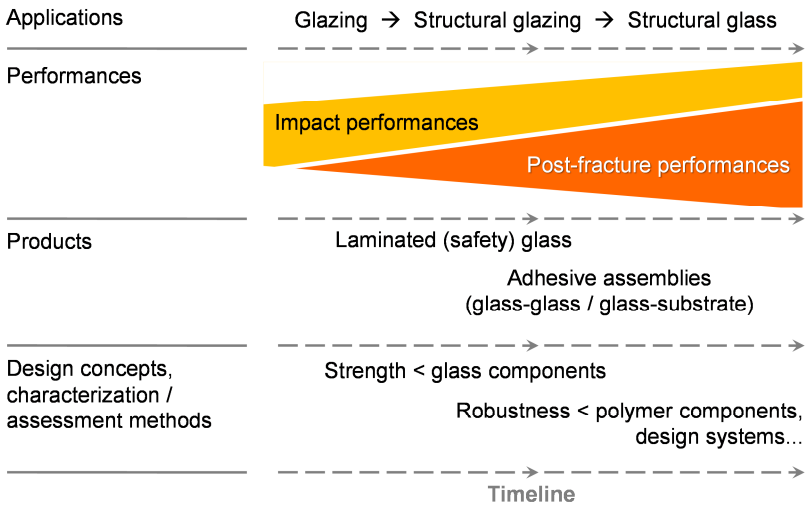

Figure I.1 – Evolution in use of glass products as building elements, from glazing to structural glass applications

From a technological point of view with regard to 'structural' and 'safety' aspects, it is worth noticing that the most decisive innovations in manufacturing flat glass products between the end of XIXth and begin of XXth century concerned mainly the production methods of glass sheets[11], while the more recent ones (approximately since the 90's) concerned rather the polymer and other synthetic, adhesive materials, transparent or not, used as interlayers, sealants and in other assemblies of glass products, than the glass product itself.

[11] The main evolutions in that field were the introduction of drawn glass sheet process in 1914 by the Belgian Foucault, of float glass process by Pilkington in 1959, and tempering processes already mentioned above. Other major fields of innovation in product development were related to the use of glass products as glazing units, with the various successive improvements of insulated units (glazing unit basically made of two glazing panes with an airtight space in between) and the use of coatings (thin metallic layer projected on a glass face) introduced to improve the energy control performances and the light transmission performance.

The parallel evolution in use of these flat glass products from glazing units to structural glass elements leads to state that design related questions of glass constructions are in fact more concerning these 'secondary' materials. Polymer components progressively played a more prominent role up to a leading one, and consequently questions about "structural use of glass", namely about resistance, robustness and durability of glass constructions, in fact appear as questions mainly addressing the behaviour of polymer components and their adhesion with glass (and possibly other substrate material).

In a similar way, another important and complementary aspect related to these design questions concerns the connection of the glass element with its support, and the importance of these to the overall performances. As it will be shown in next paragraphs, connections and boundary conditions of the glass element are playing an important role with regard to the safety performances, altogether, for windows and façade elements, to other ones in relation to their thermal, acoustic, water- and air-tightness behaviour. In fact, the latter dominated initially the design of the connections. However, effect of boundary conditions on impact and structural performances appears as very important, and probably relatively more important in comparison with, for instance, steel constructions. It is related to the lack of capacity of a glass pane to dissipate energy and its consequent much larger sensitivity to local, concentrated stress. The practical consequences on construction methods have not to be underestimated, nor misunderstood, in particular the way geometric tolerances are dealt with.

Besides the evolution fields sketched here above, it seems worth mentioning a few other parallel evolutions in the field of structural engineering :

1) The introduction and spreading out of computers and development of advanced calculation softwares, as finite elements models;

2) The development and spread of electronic devices, in particular measurement devices in testing laboratories in combination with computer acquisition and in steering systems, and similarly in control of production processes;

3) The raising and strengthening of the European standardization and regulation framework as a consequence of the building up of the European (trade) union, and in particular the evolution from a prescriptive based approach to a performance based one in technical specifications of building products.

This last point in particular induced important changes in the formulation of some design and assessment questions, and it is the main focus further in this first chapter.

The two other evolutions mentioned here above should be kept in mind when standardization issues will be considered closer and more practically : indeed, standards and technical guidelines are important tools for the building practice, and development of new standards participate to the building of a "common

reference framework" for all involved stakeholders. Standards in use, some of them about thirty years old, are generally considered as trustful and comprehensive documents, and even when they get outdated they still occupy an important place in the common reference framework which is to be built upon.

To summarize, the use of laminated glass products in building applications developed according to three complementary extension fields :

1) Technological developments of laminated glass products, from simple laminated glass units with limited dimensions to more complex products with larger dimensions, and with a larger amount of components (numbers and types of sheets, interlayers and inserts);
2) Extension of the application scope, in terms of configurations and structural role in various applications, from framed glazing unit to non-conventional load-bearing element;
3) Extension of the types and ranges of performance requirements, and of the corresponding test and calculations methods.

The different developments occurred in fact as a succession of small steps, appearing as a continuous evolution process. However, when comparing extreme application configurations, there are almost but only differences ! It seems however that, because the constitutive components remain of similar nature, the safety performances are expected to be ruled by the same mechanical properties, and accordingly to be determined by means of similar methods. In fact, such reasoning results from an accumulation of a series of presumed non-significant bias of different orders and of 'small' abusive extrapolations. Whereas some extension shifts probably rather deserve, or require, a more fundamental change of conceptual framework, and the development of a different assessment strategy.

In order to deal with this question, firstly an overview is given of the current European standardization context, and of its developing dynamic and underlying philosophy. Two important concepts are introduced and highlighted, the performance-based approach for the assessment of construction products and construction works, and the ongoing harmonization processes in standardization works. The particular situation in the field of glass products and glass works is summarized. In a second step, definitions of laminated safety glass products and related safety performances in product standards are considered, showing among others that these essentially address the (traditional) glazing applications. The third part looks at the level of applications, and identifies the similitudes and differences between the different categories of glass works, from the glazing application towards the structural applications. Finally, particularities of laminated safety glass products used in non-conventional, possibly structural applications are highlighted, and a framework is proposed to describe more accurately the application scopes, in relation with two important notions for assessment purposes, the 'family of products' and the 'intended fields of use'.

I.3. Standardization framework and harmonization processes

This section[12] does not intend to give an extensive inventory of existing standards and guidelines for glass works across Europe, but rather at giving an overview of recent developments and some comprehension keys about related concepts.

In fact, it appears difficult to pretend writing something objective and complete about the state of development of standardization nowadays. Getting a comprehensive overview of the current situation is further complicated for different reasons. Firstly, recently developed design codes about building glass products and glass works, in particular in the form of European standards and guidelines, still co-exist with older ones, whether the latter are still applicable, or were withdrawn but still used in practice[13]. As such, many reference technical guidelines still in use or recently replaced are more than 20 years old. It is worth acknowledging this when dealing with new standardization developments, as it determines what still belong to the "reference framework" of the stakeholders participating to new developments. Consequently some terms and concepts introduced and used in standards of different generations, with different purposes and application scopes, can cause confusion. Secondly, standardization involves a series of parallel processes conducted at different levels, more or less coordinated. These two reasons lead among other to different concepts covered in different documents by the same keywords or similar expressions. Consequently, some lack of consistency can appear, especially if standards and technical documents are considered individually out of their context.

A fine understanding of the standardization framework for construction activities in general, and for glass works and products in particular, is not only necessary at the time of drafting new specific standards, but also at earlier steps of the research process, as it both acts as the framework for the initial problem statement and for the implementation of new design and assessment methods. In other words, it is necessary to assimilate the 'dynamic' logic of development of standardization, and the corresponding limits and possibilities, in order to contribute to consistent (and still innovation friendly) developments[14].

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[12] The analysis in this section is largely inspired by a previous contribution (Delincé et al. 2010), yet with some updates, among others of Figure I.2.

[13] This is due to some "resiliency" of older standards and regulations, due to natural implementation delay or because withdrawn documents are still referred to by other ones still in force.

[14] It led to formulate the conclusion in a previous contribution (Delincé et al. 2010) as follows : "*It is also appearing as a trend that closer interaction between research and standardization activities are expected. It seems in particular important for researchers to have a good comprehension of the standardization context, because in many cases their contribution (included their publications) is expected to be finally valued for practical applications, and thus within the standardization framework. To achieve this, probably efforts have to be done from actors of both sides, in standardization and in research.*"

With the introduction of the Construction Products Directive (CPD)[15] in 1989, ambitious goals have been defined by the European Commission to ensure a harmonized development of design codes and assessment methods of products and works for the building industry in general. It however induced different parallel processes which interlock into each other, where the involved stakeholders also have different interests and expectations, leading to divergences of view about priorities, working methods and the outlook for developing practical guidelines and technical documents. However, this entails also many misunderstandings, in particular when general concepts and approaches are confronted with practical considerations, or when discussions are addressing different problem scales and different standardization levels.

A schematic representation of the standardization framework applicable to the design of glass works in Belgium is proposed in Figure I.2, showing the relations between older, existing and foreseen standards and technical guidelines, and the developments at European level in the meantime. It is completed by Table I.1 with a list of the corresponding titles, publication year and releasing institutes. The releasing institutes at Belgian level are the Belgian Building Research Institute (BBRI), the Federal Public Service Economy (FPS Economy)[16], and the Belgian office for Standardization (NBN)[17]; at European level standards for construction industry are released by the European Committee for Standardization (CEN) and other technical guidelines are released by the European Organization for Technical Assessment (EOTA)[18].

The structural Eurocodes constitute a particular category of European standards, in fact a series of European design (calculation) codes. The first generation Eurocodes has been issued in a series of experimental standards officially released by CEN under reference ENV 1991 to ENV 1999 between 1992 and 2003, most of them being endorsed in Belgium by the publication of a complementary National Application Document (NAD). The second and current generation of Eurocodes (EN 1990 to EN 1999) has the particularity that every standard belonging to this series must be completed by a National Annex in each member state, defining choices left open in the main text.

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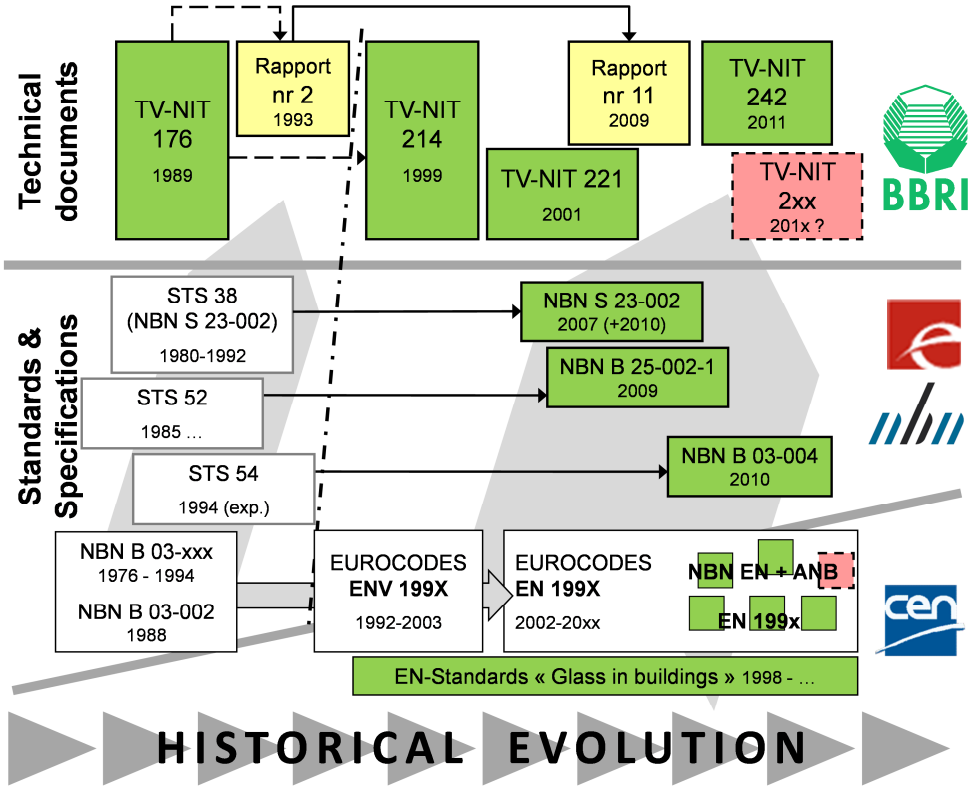
[15] Replaced in 2011 by the "Construction Product Regulation" (CPR, or Regulation 305/2011), which upgrades its legal force; among others, *essential requirements* were renamed into *basic requirements for construction works*, and a seventh one has been added which concerns "Sustainable use of natural resources" (CEN 2011)

[16] Previously Ministry of Economy (before 2000).

[17] Previously IBN-BIN; a deep reform, initiated in Belgium by a law of 2003, organized the decentralization of standardization activities, and led to the creation of the NBN (Standardization office) and the appointment of a series of Sectoral operators, ibid.

[18] Previously European Organization for Technical Agreements (founded in 1990), renamed consecutively to the replacement of the CPD by the CPR.



Legend :

- replaced by
- partially replaced by
- progressively replaced by (including implementation)
- withdrawn document
- active standard
- active technical report (standard “de facto”)
- foreseen document (in preparation...)
- limit between documents before and after CPD
- standards series

Figure I.2 – Schematic representation of evolution of standardization framework related to design of glass constructions in Belgium[19] (situation in December 2012)

[19] STS are Unified Technical Specifications published by the FPS Economy; NBN and ENV-EN are Belgian and European standards respectively, published by the NBN; TV-NIT are specific technical guidance documents published by the BBRI – see also Table I.1.

Table I.1 – Standards, specifications and technical guidelines for the design of glass works mentioned in Figure I.2.

Individual documents				
s	Ref.	Title [a]	Year	Published by
	STS 52.0	Carpentry works in façade. Generalities	1984	FPS Economy
	NBN S 23-002	Work in glass (STS 38 – 1980)	1989	NBN
	TV / NIT 176	Sloped glazing units	1989	BBRI
	Rapport no. 2	Calculation of thickness of glazing units in façade – Resistance to wind load	1993	BBRI
	STS 54	Guard rails	1994	FPS Economy
	TV / NIT 214	Glass products – Functions of glazing units	1999	BBRI
	TV / NIT 221	Rabbet setting of glazing units	2001	BBRI
	NBN S 23-002	Work in glass (+ AC:2010)	2007	NBN
	NBN B 25-002-1	External window work - Part 1 – Generalities (+ AC:2011)	2009	NBN
	NBN B 03-004	Railings of buildings	2010	NBN
	Rapport nr. 11	Application of Eurocodes to the design of external window work	2011	BBRI
	TV / NIT 242	Special glass works – Part 1 : structural applications	2011	BBRI

Standards series			
	Ref.	Description	Released by [b]
	NBN B 03-xxx [c]	Design rules for buildings : actions on constructions,…	NBN
	EN 1990 – EN 1999 (ENV 1990 – ENV 1999)	Structural Eurocodes (design standards for construction works)	CEN (TC250)
	EN-standards “Glass in building”	Harmonised product standards and support standards	CEN (TC129) (ISO/TC160)

[a] Freely translated in English from original title, except for NBN-standards for which English titles exist. See online catalogues of respective institutes for original titles in Dutch and in French.

[b] EN-standards are released by the European Committee for Standardization (CEN), and published, with possible translations, by national standardization bodies (NBN for Belgium). However, Eurocodes are also completed by a National Annex (see ibid. and in main text).

[c] This series of standards has largely been replaced by the Structural Eurocodes, firstly by the ENV (experimental) version between 1995 and 2002, and then by the EN version since 2002. Anno 2012, the two officially remaining standards of this series are the NBN B03-003 (deformation criteria in buildings; considered as a sub-part of the National Annex to EN 1990) and the NBN B03-004 (guard rails). However, other withdrawn parts are still referred to in still active technical documents (which implement the general design guidelines to more particular cases, like the ones for glass works), in particular the one regarding wind actions on structures (NBN B03-002-1) : this is a typical example of the mentioned “resiliency effect”.

To give a concrete example, the EN 1990 (basic part of the Eurocodes series) has been prepared and released by the CEN, and is published in every member state as a National standard, for instance in Belgium with the reference NBN EN 1990. The text cannot be modified, but may be translated under the responsibility of the national authorities, generally the national standardization body…

The main Eurocode document is completed by a National Annex (NA) published under a national reference, in Belgium as NBN EN 1990-ANB. The types of changes and additions allowed in National Annexes to Eurocodes are strictly framed, among others by means of a series of Nationally Determined Parameters (NDP) defined in each Eurocode part. These are essential concepts with regard to harmonization purposes. They allow national committees to modify mandatory and optional clauses, and to define different values of safety parameters, to implement differentiated levels of safety in calculation codes, according to related specificities in national regulations, climates and building traditions[20]. However, this is the theory; in practice, the amount and variety of NDP's also reflects the grade of harmonization and consensus that could be reached inside a sector of the construction industry at one specific moment, not only about specific methods or calculation rules, but also about more specific harmonization goals and implementation strategies.

More explanations about the standardization issues related to the Eurocodes can be found on dedicated information websites[21].

For understanding what "harmonization" effectively means in this context, it is useful to look at the developments at European level for the past decades along a timeline. The efforts in developing European standards for building glass products started at the end of the 1970's, as acknowledged by a report published by the CEC [22] dated of 1983 (Commission des Communautés Européennes 1983)[23]. This report presented the vision of the glass industry regarding the standardization developments at the time being, essentially thought from the perspective of glazing applications.

The standardization goals expressed in 1983 have meanwhile been largely achieved in the form of different European standards, mainly prepared by the technical committee CEN/TC129[24]. However, "structural glazing" and "structural use of glass" which aroused mainly during the 1990's were not really taken into

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[20] Traditions refer to technological preferences and quality control mechanisms.

[21] Among others on : www.normen.be/eurocodes and eurocodes.jrc.ec.europa.eu.

[22] Commission of European Communities (CEC) was the equivalent in the time of the EEC of the current European Commission. This report published by the CEC had been prepared by the GEPVP (acronym designing the previous name of the European federation of manufacturers of flat glass products, renamed meanwhile "Glass for Europe" www.glassforeurope.com).

[23] This report is named CEC-report or EUR 8069 here after.

[24] CEN/TC129 is the Technical Committee "Glass in buildings" in CEN's organization.

account in these developments, yet they began to be discussed in the context of the works related to the development of the "EN 13474" standards series[25], which took place approximately between 1998 and 2009. In 2007, a report is published by the European Commission (Zarnic et al. 2007)[26], which addresses issues and associated harmonization challenges for developing European guidelines for design of glass works, and in particular with regard to the non-conventional "structural" use of glass products, in the perspective of a possible Eurocode dedicated to glass constructions. In 2011, a workgroup dedicated to 'structural glass' is created within CEN/TC250 (Working Group 3). This one circulated in the beginning of 2013 a first 'SaT-report' (scientific and technical report; work document) as a first basis for developing a European design code, which has meanwhile been published by the European Commission (Feldmann and Kasper 2014)[27].

In parallel to CEN's works, the EOTA published in 2001 the ETAG002 "Guideline for European technical approval for Structural Sealant Glazing systems (SSGS)", and in 2002 the ETAG010 "Guideline for European Technical Approval for Self Supporting Translucent Roof Kits"[28].

Member states of the European Union are legally bonded to implement the EN-standards and ETAG's, since these are used as the technical basis to deliver CE-marking on construction products[29], which is principally aimed at promoting their free circulation within the European trade area. Simultaneously, member states authorities still hold the responsibility for ensuring that all construction works on their territory are designed and executed in a way that does not endanger the safety of persons, domestic animals and property, and that fulfils to other essential requirements in the interest of public wellness. Accordingly, they may prescribe different levels of protection, for instance to account for different climatic conditions. Development of regulations for determining how to assess that construction works are complying to these safety requirements are still the national competence of Member States.

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[25] The initial title of this series of standards "Glass in buildings – Design of glass panes" became "Determination of the strength of glass panes" between 2003 and 2005, but did not reach the first official draft stage; nevertheless, different draft versions circulated in that period, largely referred to and commented in scientific publications. Much more recently (end 2013…), two projects of standards related to the topic reached an official draft stage, the prEN 16612 ("Determination of the load resistance of glass panes by calculation and testing") and the prEN 16613 ("Laminated glass and laminated safety glass - Determination of interlayer mechanical properties").

[26] This report with reference EUR 22856 EN can be downloaded from the website of the JRC at next address : http://eurocodes.jrc.ec.europa.eu/doc/EUR22856EN.pdf

[27] This report with reference EUR 26439 EN is supposed to be made available on the dedicated website of the JRC http://eurocodes.jrc.ec.europa.eu; JRC-reports can also be recovered from the JRC Publications Repository : http://publications.jrc.ec.europa.eu.

[28] ETAG's and other guidance documents of the EOTA can be freely retrieved from www.eota.eu.

[29] In the wide sense of the concept, construction products also include 'kits' and 'systems'.

This distinction between harmonization of technical specifications on construction products and national competence for determining the safety level of construction works led among others to the introduction of the NDP's in the Eurocodes, which intended use are framed by the Guidance Paper L[30]. Whereas Eurocodes were initially conceived as calculation codes for structural systems and structural members [31] only, with the Guidance Paper L, their use is more generally encouraged with regard to safety requirements and assessment of all construction products falling in the scope of the CPD. Consequently, general parts of Eurocodes (EN 1990 for general framework and concepts, EN 1991 for actions on constructions, EN 1997 for geotechnical design and EN 1998 for seismic design) are supposed to be applicable to all construction works[32].

In the next sections, existing concepts and terms in standards about laminated glass products and their structural and safety performances are considered, seeking in particular for the corresponding test methods used for characterizing and assessing application performances and related product properties. In the last section of this chapter, the problem will be looked at in a wider perspective, in particular in regard to specificities of involved materials, glass and polymer interlayer, on the one side, and to formulation of design questions related to the use of this kind of products in structural applications on the other side.

I.4. Safety performances of laminated glass products

Application scope intended to be covered by product and design standards followed the technological developments sketched in section I.2. The current situation for laminated glass products is a little complicated, as standardization efforts in that field have delivered international and European standards. Safety performances for laminated glass products were logically mainly developed with regard to their use as safety glazing. In this section, it is examined in what extent the standardized concepts are robust or restrictive, namely whether they facilitate or restrict an extension of the field of use of laminated glass products to other, less conventional applications. Despite the advanced state of development of standardization in this field, many questions remain open.

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[30] The "Guidance Paper L : Application and use of Eurocodes", published by the European Commission in 2003, belongs to a series of guidance documents relative to the CPD.

[31] According to the terms defined in the Eurocode 0 (EN 1990). However, definitions of structural members and systems remain quite vague and open to interpretation, apparently by purpose when considered with regard to the development strategy of the European standardization framework. Structural element is further used below in a similar general and non-univocal acceptance.

[32] However, it does not imply that all application rules are appropriate nor all calculation rules applicable to glass products and glass works : between principles and applications rules, there is still a non-negligible field of interpretation, which by definition does not provide univocal guidelines.

I.4.1. Laminated glass products

Laminated glass products are covered by a European harmonised standard (hEN)[33], EN 14449, released in 2005 by the CEN, and referring largely to the previously endorsed standards series EN ISO 12543 (1998)[34]. According to these standards, a **laminated glass** product is defined as *"An assembly consisting of one sheet of glass with one or more sheets of glass and/or plastics glazing sheet material joined together with one or more interlayers."* (EN ISO 12543-1:1998)

This description seems clear, but it seems still necessary to emphasize that "product"[35] refers to the laminated glass unit as it is put on the market, say as it leaves the manufacturing plant[36].

A second definition is introduced for **laminated safety glass** product, which is defined as a *"laminated glass where in the case of breakage the interlayer serves to retain the glass fragments, limits the size of opening, offers residual resistance and reduces the risk of cutting or piercing injuries"* (EN ISO 12543-2:1998)

This definition refers thus clearly to "safety performances" of the product, but still rather formulated in terms of general targets. In that regard, it is necessary to note that, principally, performances of construction products in the European standardization framework are defined in relation to their contribution to performances of (part of) construction works. The way these safety performances are further expressed and evaluated is further discussed in next paragraph I.4.2.

Among the aspects treated in the EN ISO 12543 standards series, assessment procedures related to the quality control of the lamination process and to the durability of products are the most important ones. These are based on three different types of artificial ageing tests (Table I.2) typically performed on samples of small specimens laminated glass of 300x300 mm. Some adaptations to the specifications of these ageing tests are introduced in the harmonized product standard, for ITT-tests and FPC-tests and for the different categories of laminated

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[33] An harmonised European standard (hEN) basically determines 1) applicable methods and evaluation criteria for determining product performances corresponding to the essential requirements, which are used for the initial characterization, or Initial Type Testing (ITT), of the products, and 2) corresponding methods for the factory production control (FPC) depending on the specific conditions of the production process. It also gives technical details for setting up evaluation and conformity control system to assess the stability of considered performances.

[34] New versions of the six standards EN ISO 12543 have been released in 2011, but they are not automatically implemented in the European framework, because the references to this series of standards are *dated ones* in product standard EN 14449. The use of dated references can be seen as a sign of conflicting standardization strategies between different technical committees, in this case between attempts at developing international and European standards.

[35] According to the terminology introduced in the European standardization framework.

[36] In fact, the definition of "construction product" in the European framework distinguishes the product put on the market and the product as part of a built construction work.

glass products distinguished in the product standard (see below). Effects of artificial ageing tests are evaluated by means of visual observation of defects[37], and in some cases by a measure of the transparency before and after the ageing test.

Other properties of laminated glass products are evaluated by two means :

- Specific test methods on laminated glass products or applications, as for most of the 'safety properties' discussed in next paragraph;
- By assuming that the properties of constitutive glass sheets are unchanged by the lamination process, as for instance the strength of glass sheet[38].

Table I.2 – Assessment of durability of laminated glass products according to EN ISO 12543-4

Type of ageing test	Short description	Evaluation type (before and after ageing test)
High temperature test	2 hours at 100°C (in principle by immersion in boiling water)	Visual evaluation of defects
Humidity test - without condensation - with condensation	2 weeks at 50°C - at 80% relative humidity - at 100 % relative humidity	Visual evaluation of defects
Radiation test	2000 hours exposed to standardized UV-radiation	Visual evaluation of defects Luminous transmittance

Besides these guidelines for assessing the durability of products, this standard series essentially defines geometric tolerances of the products and a description of edge finishing (EN ISO 12543-5), and visual assessment procedures of the appearance quality of end products (EN ISO 12543-6).

In addition, the European product standard distinguishes three families of laminated safety glass products[39], namely according to the interlayer type as follows :

1) laminated glass with folio interlayer;
2) laminated glass with cast-in-place interlayer (resin);
3) laminated glass with intumescent interlayer.

This distinction appears not consistent at first sight, since the two first families seem to refer to two possible lamination processes, and the third to a specific behaviour of the interlayer at elevated temperature (in case of fire). As this

[37] The standard prescribes the conditions and criteria for performing this visual evaluation.

[38] See also Chapter II about strength of glass components in laminated glass unit.

[39] In its annexes dedicated to the specific procedures for evaluation of conformity of the products.

distinction is made in the context of defining production control measures, the main driving idea behind seems to adapt these to the specificities of each production process. However, when looking at the differences in recommended conformity tests between product families, differences are noticed also regarding the safety performances considered (what seems logical, as FPC-tests must ensure that product characteristics determined by the ITT are guaranteed).

The test methods in standards are not considered here with regard to their purpose of production control, but rather with regard to existing test configurations and methods for investigating product properties. There is in fact a very practical aspect not highlighted in the standards, namely whether the laminated glass product is considered as further processable by cutting operations or not, and in accordance whether it is deliverable in stock sizes or only in final cut sizes. There are different reasons why a laminated glass product cannot be further processed by cutting operations :

- the laminated glass unit may count at least one glass sheet of tempered glass[40];
- for other practical reasons related to the considered cutting tool or method, in function of specific behaviour of some component or the specific composition of the laminated glass unit, for instance due to a specific arrangement of the constitutive layers or a large total thickness;
- due to a risk of damaging some performance of the finished product, for instance if these are protected by a protective sealant placed along the edges of the plate (laminated glass unit with an intumescent interlayer).

This distinction between processable and not processable laminated glass products appears of major relevance for determining properties of the "finished product", but also when considering the possibilities and limitations in investigating their mechanical properties on smaller test specimens, in a research or quality control perspective. It does thus not only address the question of whether it is *possible* to produce small test specimens, but whether it is *relevant* with regard to their representativeness. This aspect is discussed in more details in Chapter IV in parallel to experimental methods.

Nowadays, the majority of laminated safety glass products is belonging to the family of folio interlayer laminated products. The two categories of products

[40] This means any non-annealed (float) glass sheet, namely of which surface strength has been increased by any thermal or chemical process (see also paragraph I.2). These are not mechanically processable after tempering, among others they cannot be cut anymore. The tempering process of a glass sheet always happens prior to its inclusion in a laminated product, the first process corresponding to a thermal treatment at much higher temperature than the second one : the maximal reached temperature is respectively about 600-675°C for a thermal tempering process (400°C for a chemical toughening process), and 140°C for a common autoclave process used for a folio-lamination (Haldimann et al. 2008). Other usual lamination processes are performed at similar or lower temperatures.

considered in this work belong to this category (see Chapter III paragraph III.3.1). However, cast-in-place interlayers are also used, especially for non-conventional laminated systems. The production of test specimens for the two categories of products addresses different production issues, which may influence significantly the measured properties.

I.4.2. Performances of laminated safety glass products

The definition of laminated safety glass according to the standard EN ISO 12543 given in previous paragraph is still rather vague about the determination of mechanical properties and of safety performances of laminated glass products. Among the different reasons explaining this, it seems that no compromise about the most suited method(s) could be found at international level. It will be shown that different misconceptions and different implicit assumptions are lying behind, which prevent convergence.

An important point of discussion is related to the details of the considered test for determining quantitatively whether a laminated glass product is a laminated *safety* glass product. The international standard acknowledges (in an addendum of 2004…) that different test methods are considered in the different countries, and refer to the EN 12600 for CEN-member states, and for instance to ANSI Z 97.1 (2004) for United-States. The essential point of discussion, at that time (Jacob et al. 2003), was apparently not about the principle use of a pendulum test configuration, but on the practical levels of impact obtained with the different test methods. Among others, the question addresses the choice of the impact body (or impactor) used in such tests[41], regarding two aspects essentially :

- its representativeness of an impact of a human body against the glazing, or in other words which amount of the impact energy is absorbed by the impactor and accordingly what is the amount of energy the glass pane must be in state to absorb;
- the reliability, in particular the reproducibility, of the test method, including calibration aspects of the test rig and verification of the impactor performance stability in time.

Behind the scientific considerations, there are of course economic considerations related to the practical consequences for the dimensioning of basic laminated safety glass configurations, but also with regard to the "competition" between thermally toughened safety glass products and laminated safety glass products in glazing applications.

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[41] The release of the EN 12600 by the CEN in 2003, which specifies the use of the "dual-tyre" impactor, is simultaneous to the publication of the technical report EOTA TR001 which specifies the use of the "ISO-sandbag" impactor for the evaluation of impact resistance of panel and panel assemblies. Ten years later this lack of consistency has not been solved.

This discussion raised another one, namely about the representativeness of the test configuration in relation to the scope of "intented use" which is supposed to be inherent to the definition of the product performance in the European framework. This aspect of representativeness of reference test configurations is further discussed in section I.5 about the performances of glass works.

The product standard EN 14449 does not define more specifically which resistance level a laminated glass product has to reach to comply to the definition of a laminated safety glass, only that it should show a breakage pattern "B"[42], what basically means that the glass pane cracks without falling out of the frame, and that the amount of released splinters or glass fragments remains but a small weight fraction of the (outer) glass panes. Because of its conceptual importance, the pendulum test method is detailed further below. Other performances related to "safety in use" [43] requirements are mentioned in the product standard, and for each a reference to a particular standard detailing the test method for assessing the corresponding product performance (Table I.3).

Table I.3 – Performances of laminated glass related to requirement of 'safety in use' according to product standard EN 14449 (2004)

Performance in EN 14449	Referred standard
Safety in use – Bullet resistance : shatter properties and resistance to attack	EN 1063 (1999)
Safety in use – Explosion resistance: impact behaviour and resistance to impact	EN 13541 (2001)
Safety in use – Burglar resistance : shatter properties and resistance to attack	EN 356 (1999)
Safety in use – Pendulum body impact resistance : shatter properties (safe breakability) and resistance to impact	EN 12600 (2002)
Safety in use – Mechanical resistance : resistance against sudden temperature changes and temperature differentials	
Safety in use – Mechanical resistance : resistance against wind, snow, permanent load and/or imposed loads of the glass unit	(prEN 13474)

Note: between brackets is indicated the release year of the first version of each standard by the CEN, which may differ from the publication year of the standard by National Standardization Body.

[42] See Annex C of EN 14449. The last version of EN ISO 12543 series (2011) has not changed the concepts nor the evaluation method and criteria required for a "laminated safety glass", and mentions that a resistance class "3B3" according to EN 12600 is required in CEN-member states. See also below about the meaning of the resistance class.

[43] Freely translated from the French version of EN 14449.

The different test methods used for each evaluation have some features in common : each is defining a particular type of dynamic loading condition, with specifications on the source of the dynamic action (as the impact body or the tools and restrictions on their use for prescribing attack modes), and for each performance different loading levels or grades of attacks are defined, generally in the form of categories. Each test method prescribes also the reference test configuration for performing the test at an element scale, involving one or a short series of test specimens. The reference configuration is characterized by prescriptions on the test frame (or test rig) and clamping method (how the specimen must be mounted and fixed in the test frame); these fix the geometry of the test specimens (planar dimensions) to arbitrary dimensions, and limit the possibility for its composition, by limiting the maximum thickness of the tested element.

Let us consider closer two of these test configurations. The first is the pendulum test (EN 12600) already mentioned here above, and the second is the hard body drop test (EN 356). There are different motivations for considering these two test configurations in particular. They define the basic compositions of laminated safety glass products for two different loading modes and they are probably the most known 'safety' standards for laminated glass products used in glazing applications. However, it is interesting to identify what *are* these safety performances exactly, and what a performance category for a laminated glass product according to these standards possibly tell about the post-fracture performances of the product in a more general way[44].

As summarized in Table I.4, the evaluation is performed on a prescribed reference configuration[45], with variable test conditions in terms of loading level or sequence specific to each performance, and fixed conditions in terms of ambient test temperature[46] and for the conditioning of the test specimens. In both cases, breakage of the constitutive glass sheets is allowed, but not supposed to lead to an overall failure or collapse of the tested element, at least not within specified time

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[44] Whereas the impact body used for the drop height test (EN 356) is considered as a "hard body", the double-tyre impactor is often described as a "soft impact body", and these tests are commonly often designed as "hard impact" and "soft impact" test, respectively. The concept of "hard impact" in this case means that the essential of the impact energy is transferred to the impacted element, with negligible deformations of the impact body. Noteworthy, this definition of "hard impact" is not in line with other ones, as for instance in the Eurocode part about accidental actions (EN 1991-1-7), where an impact "*is characterised as either hard impact, where the energy is mainly dissipated by the impacting body, or soft impact, where the structure is designed to deform in order to absorb the impact energy*".

[45] These two standards give more details about tolerances on the different test parameters and further prescriptions for controlling the clamping conditions of the test piece in a standardized frame. These are not presented in details here, however both are certainly relevant regarding the two discussed issues (reliability and representativeness of the tests).

[46] Specified ambient temperature for each test is slightly different.

limits between the moment of impact and the moment of evaluation[47]. As such, it seems implicitly assumed that if the integrity of the glass element is preserved after this time interval, risk of possible delayed failure due to further time-dependent deformation of the interlayer is discarded. The other specification on time durations concerns the conditioning period of the test specimens prior to the execution of the test[48].

The pendulum test method is not restrictive in its scope to the evaluation of laminated safety glass products, but to any other 'safety glazing' product, in particular thermally toughened safety glass[49]. This explains the two evaluation criteria (see Table I.4), in fact corresponding with different safety concepts (no injury for the person falling against the glazing on the one hand, containing capacity during the impact and retaining capacity after the impact on the other)[50]. Table I.5 gives a few examples of test scenarios in correspondence to different classes of performance.

Interpreted in terms of behaviour of the laminated safety glass unit, the pendulum test method of EN 12600 basically evaluates the capacity of the element to survive to one single impact (accidental action) and to keep its integrity, and the drop height test of EN 356 rather evaluates a containing capacity against a succession of impacts of similar intensity (attack). In terms of response to impact and corresponding post-fracture performance, it seems clear, from a *qualitative point of view* and based on typical product compositions complying to each resistance category (see examples in Table I.7), that the pendulum test accepts products with lower performances than the drop height test; however, these differences between performances assessed by different test methods cannot be easily compared, because they cannot be expressed on a equivalent *comprehensive quantitative way*, and this for different reasons.

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[47] The mentioned values are 3 minutes after impact for the pendulum test on a vertical element, and 5 seconds after impact for the hard body drop test on a horizontal element.

[48] The test specimens must be conditioned at least 12 hours at ambient (test) temperature.

[49] The expression 'safety glass' is consequently discarded because of its ambiguity between different safety criteria; the expression 'safety glazing' faces the same ambiguities and should be understood in the current context in its most general acceptance as 'glazing element complying to some safety requirement(s)'.

[50] An understanding of the resistance classes of the EN 12600 requires a fine reading of the test method and evaluation criteria. To summarize, two evaluation criteria are defined : the first (a) corresponds to the concept of a retaining function (typically associated to the performance defining a laminated safety glass product); the second (b) corresponds with the idea of a safe fragmentation pattern in case of breakage without retaining function (typically associated to the performance defining a thermally toughened safety glass product). The resulting category takes the form "α β φ", where α is the index number of the last drop height with no damage or a safe fracture or breakage mode as consequence of the impact (criterion a or b), β is a letter associated to the type of the observed 'safe fracture or breakage mode' (A: unsafe, B: safe with regard to criteria a and b, C: safe with regard to criterion b only), and φ is the index number of the last drop height with no damage or a safe fracture mode as consequence of the impact (criteria a).

The first reason is that the resistance to impact, and more generally to a dynamic action, is a performance which is fundamentally an element performance, and thus which can only be experimentally assessed by means of a test *at an element scale*[51], which involves the influence of boundary conditions, geometric and loading configurations, and possible size effects. Comparing the performances between the results of a pendulum test and of a drop height test is further complicated because the respective reference test configurations involve simultaneously different impact bodies, different geometric configurations and boundary conditions (clamping), and different evaluation criteria.

The second reason is related to the first : there is *no straightforward way for quantifying the effective level of energy absorbed by the element from the impacting body*, and of possible resulting damage for a family of similar test configurations, compared to a reference configuration. In fact, the proportion of absorbed or dissipated energy by the tested element and by the impact body varies with characteristics of the test specimen. Relating the element performance to intrinsic properties of the product and of its components require the use of advanced modelling tools (Brendler et al. 2004; De Pauw 2010). It is still common in publications and technical documentation to see the impact energy described by means of the kinetic energy of the impactor just before the impact. However, this parameter cannot be considered as a representative value of 'impact resistance' of the element (in terms of induced damage), as it does not tell what part of the energy is dissipated or transferred[52] by the glazing unit. Nevertheless, some values of impact energy for the two impact bodies considered above are given for a few drop heights (Table I.6); it represents thus the total mechanical energy present just before the impact (contact).

An impact is an interactive dynamic phenomenon between the impacting body and the impacted one. The kinetic energy available at impact will be dispersed between both according to their relative stiffness and their respective deformations during the impact duration. In the two impact configurations considered here, deformations playing a role are likely to occur at three level : the impactor, the impacted panel, and the supporting frame[53]. For the hard body drop test, the deformations of the steel ball can be considered as negligible with regard to the two others[54], but a similar assumption is certainly not valid for the pendulum test. Even if the element configuration (element + test frame) would be

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[51] The concept of element scale refers to the 'element experimental scale', see also Chapter IV.

[52] The energy transfer concerns the interactions with the impact body on the one hand, and with the test frame or supporting structure on the other hand.

[53] Deformations of the testing frame seem to be assumed as negligible, considering the specifications on it in the respective standards. The situation can be very different in the design configuration, see also paragraph I.5.1.

[54] This assumption that the energy absorbed by the impactor is negligible corresponds to the concept of "hard impact" (see here above).

the same for the two types of impact, the ratio between transferred and absorbed energy is likely to be quite different for the pendulum test and the hard impact body test, and different between drop heights for a given test configuration. Besides, in such impact problems, a part of the mechanical energy is likely to be dissipated in the deformation processes[55], so the conservation of mechanical energy during the impact is generally not a valid assumption. In next columns of Table I.6, other typical parameters used in the description of impact problems are given as indicative values and are not commented further by now (velocity and momentum[56] of the impactor just before impact).

An example of impact resistance levels reached for a series of typical compositions of laminated glass products with PVB-interlayer according to the two afore mentioned test methods are given in Table I.7, as mentioned by the manufacturer (Saint-Gobain Glass 2006). A quick comparison of impact characteristics in Table I.6 and typical configurations for each resistance class of Table I.7[57] confirms that not any of the parameters used for expressing the 'energy of impact' does give a comprehensive order of magnitude of 'safety level'.

In complement to the information collected in the Table I.7, the manufacturer added in its documentation that the mentioned performances are ensured for a temperature in the glazing component comprised between 10 and 45°C, and that it should not be kept exposed to a temperature larger than 60°C. However, no detail is available about according which criteria these limits are fixed[58]. In fact, data about the sensitivity of results of this type of impact tests to test temperature are scarce; an example is given in Figure I.3, again for a specific reference test configuration (Kuraray 2009).

Similarly, guidelines or specifications in design codes and standards about range of service temperature generally do not distinguish which performance is concerned by the specified limits, for instance between the ambient temperature for resistance to an accidental impact and temperature ranges with regard to the durability of the product (no degradation of the interlayer). Accordingly, specifications on range of service temperature and on safety performances tend to appear as distinguished, uncorrelated issues.

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[55] In particular by the interlayer of the laminated glass element.

[56] The momentum is the product of the mass of the impact body with its velocity at impact.

[57] A laminated glass '33.1' is considered as the thinnest composition for laminated glass products used in building applications, since it combines the thinnest film with the thinnest glass sheet. Mentioned performance levels assume that the two glazing sheets are annealed float glass sheets.

[58] These values can be compared with the ones given and discussed in Chapter III, section III.1.

Table I.4 – Comparison of impact tests according to EN 12600 and EN 356

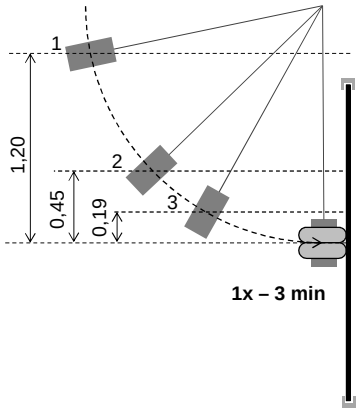
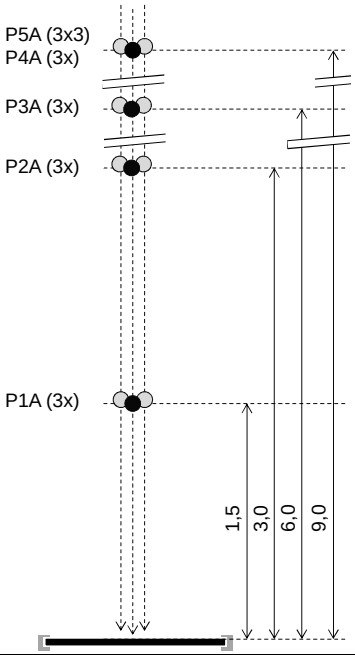
Test type	Pendulum test	Hard body drop test
Standard	EN 12600 (2003)	EN 356 (1999)
Testing configuration (sketch on scale)		
Impactor	Dual tyre (50 kg), diameter ~250 mm	Steel ball (4,1 kg), diameter 100 mm
Tested elements	Flat glass products, 4/8/12 test pieces 876 (width) x 1938 (height) mm set in a standardized *vertical* frame	Flat glass products, 3-4 test pieces 1100 x 900 mm set in a standardized *horizontal* frame
Test conditions	Test pieces conditioned at least 12h and tests carried out at (20±5°C)	Test pieces conditioned at least 12h and tests carried out at (23±2°C)
Test procedure	Impacts *at progressive height* (3, 2, 1) on sample of 4 test specimens; impact in the centre of the specimen; no second impact after a possible rebound is allowed	On each specimen, 3 impacts *for a chosen drop height* (3x3 for P5A); impacts on the corners of a equilateral triangle with c = 130 mm round the centre of the sample; after each impact, clean upper surface from loose glass fragments
Evaluation method and criteria	After each impact, two criteria are evaluated : *a) if no overall failure* within 3 min : do not let pass a sphere with diameter 76 mm under a force of 25 N through the glass; Amount of fragments < 10.000 mm² Largest fragment < 4.000 mm² *b) if overall failure* : 10 largest fragments < 6.500 mm²	After each impact, it is checked if : *a) slippage criterion* : edges of test piece do not move more than 5 mm in the clamping frame; *b) penetration criterion* : impactor does not pass through within 5 sec. Test successful if criterion b) still verified after last impact.
Test result	Category of resistance : α β φ (α, φ = 3, 2 or 1, and β = A, B or C)	Category of resistance : PxA (with x : drop height class)

Table I.5 – Examples and interpretation of resistance classes according to EN 12600

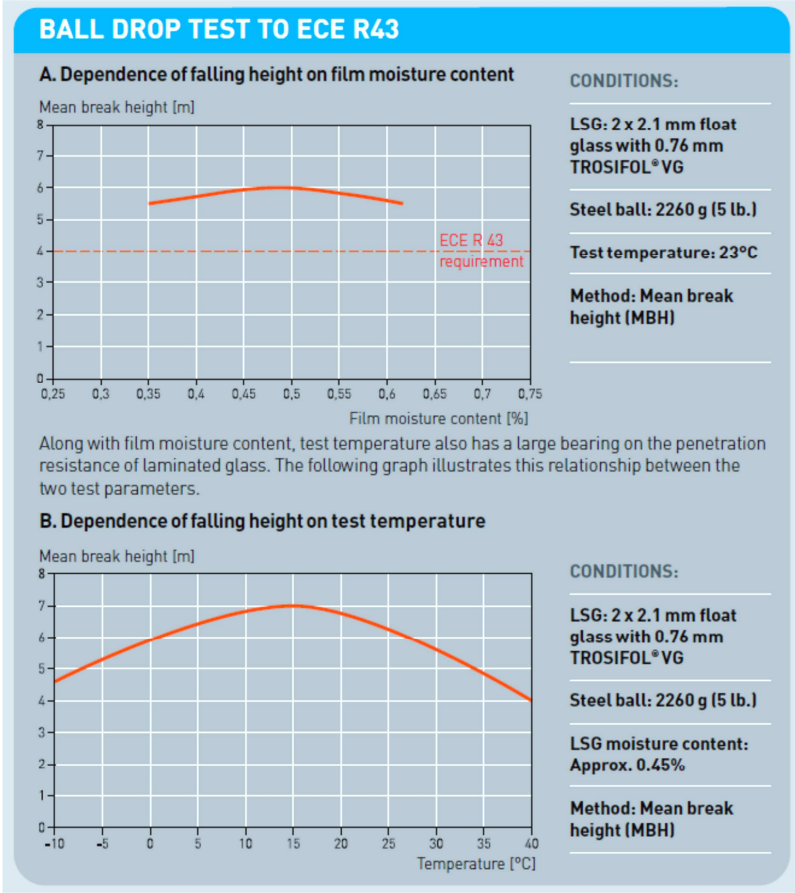
	Meaning of the assessed behaviour at the successive drop heights		
Resistance class	3 (190 mm)	2 (450 mm)	1 (1200 mm)
(3A0)	Breaks unsafely	Breaks unsafely	Breaks unsafely
1C0	Breaks safely (b)	Breaks safely (b)	Breaks safely (b)
1C2	Does not break/fracture (or fracture safely (a) *)	Breaks safely (b)	Breaks safely (b)
2B2	Does not fracture or fracture safely (a)	Does not fracture or fracture safely (a)	n.a. (**)
1B1	Does not fracture or fracture safely (a)	Does not fracture or fracture safely (a)	Does not fracture or fracture safely (a)
(a) and (b) refer to failure/breakage criteria (see also Table I.4) (*) the probability that this second behaviour occurs effectively for a thermally toughened glass unit seems very low (**) two interpretations are possible : either the laminated glass specimen breaks without complying to any of the two criteria a) and b), either it has not been evaluated at this drop height (this is allowed by the standard)			

Table I.6 – Comparison of impact energy (1 impact)

	Impactor mass [kg]	Drop height [m] (height class)	Impact energy [J] (of 1 impact)	Impact velocity [m/s]	Momentum [kg.m/s=N/m]
Pendulum test	50	0.190 (3)	93.2	1.93	96.5
		1.200 (1)	588.6	4.85	242.6
Hard body drop test	4.1	1.5 (P1A)	60.3	5.42	22.2
		3.0 (P2A)	120.6	7.67	31.5
		9.0 (P4A)	362	13.29	54.5

Table I.7 – Typical compositions of laminated safety glass with PVB-films for some impact resistance classes (Saint-Gobain Glass 2006)

SGG Stadip (Protect)	Thickness [mm]	Weight [kg/m²]	Resistance class
33.1	6,5	15	2B2
33.2	7	16	1B1 / P1A
44.2	9	21	P2A
44.3	9	21	P3A
44.4	10	22	P4A
Note: composition of laminated glass units is commonly described using a denomination with format *XX.Y*, where *X* refers to the (respective) thickness in mm of the (two) glass sheet(s), and *Y* to the thickness of the interlayer expressed as the amount of PVB-films with a thickness of 0.38 mm used, or the equivalent thickness obtained with a thicker PVB-film.			



Note : tests according to ECE R 43 and EN 356 are similar in their principle, but with different specifications

Figure I.3 – Temperature dependence of impact performance (Kuraray 2009)

The safety performances mentioned in Table I.3 are considered as product performances, and consequently each of the corresponding test methods corresponds to an ITT test, which in principle implies the identification and the description of a family of products on the one hand, and of intended field(s) of use on the other. *Family of products* and *intended field of use* are two complementary concepts constitutive of the description of application scope(s). Both can be pre-defined to or result of an assessment procedure.

It is a little surprising that the product standard EN 14449 gives so few concrete indications about how to precise these two concepts[59], and consequently about the reference compositions of the test specimens to consider for performing the experimental evaluation[60]. A refined framework for the description of application scopes is further proposed and discussed in paragraph I.6.

To terminate the overview of safety performances in product standards, let us mention that according to the EN 14449, the strength of an individual constitutive glass sheet included in a laminated glass product is considered as unchanged in comparison to its initial strength when considered isolated. This seems an obvious statement, provided that the question of the characterization of the glass strength of glass products, a non trivial topic on its own, is left aside. In fact, assessing the contribution of the strength of individual glass components to safety performances of a laminated glass element is another, distinctive issue, than assessing the resistance to breakage of an isolated glass sheet : this aspect is shortly discussed below in paragraph I.5 dedicated to performances of glass works, and is further addressed in Chapter II. This statement about the non-alteration of mechanical properties of glass components by the lamination process has no equivalent in the standards for the interlayer components. Such an assumption is indeed generally not applicable to the interlayer components, even not for folio interlayers. This is an important aspect regarding questions addressing characterization methods of *material* properties of an interlayer component, which are further developed in Chapter III.

In summary, different questions are arising about the *representativeness*[61] of results of standardized impact tests with regard to the final performances of laminated glass elements assembled in construction works. It is further noticeable that the four test standards mentioned in Table I.3 are referring in their title to "security glazing", which confirms the presence of a confusion between a product

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[59] The guidelines in the product standard EN 14449 about how to describe a family of laminated glass products are limited, but yet assign the task and responsibility of making this description and updating it to the manufacturer (meaning the one performing the lamination process).

[60] In other words, test configurations and test methods are standardized, but not the assessment methodology aiming at defining on which scope test results are assessing final performances of applications. The product standard mentions that each type of ITT should be performed on (or consider) configurations corresponding to the "minimal design specifications" of the family of products (which seems associated to a configuration where each component has the thinnest specified thickness), and identifies further which performance resulting of a specific ITT has to be re-evaluated for any change in the definition of the extent of the family of products, according to the type of change and the concerned component, but only on a qualitative basis. The corresponding analysis grid only addresses changes in composition, by listing properties of type and thickness of constitutive layers, and assembly order. Planar dimensions and configurations are not considered in the definition of products.

[61] The question of the representativeness is in fact covering different aspects and issues, which will be further address in Chapter IV.

and an application scale, or suggests that the "intended field of use" of laminated safety glass products in the product standard is limited to (vertical) glazing units set in a frame[62]. The product standards for laminated glass products are also silent about possible sensitivity of safety performances to ambient temperature. Moreover, each of the mentioned test methods is assessing some post-fracture resistance, but only in the form of a pass or fail criterion : they do not give a quantitative value of the post-fracture resistance usable for design purposes[63]. It appears thus clearly that considered test methods mainly assess safety performances *during* the dynamic event caused by the impact, and only to a limited extent the load-bearing performance *after* the impact. This suggests implicitly that if the laminated glass element survives the impact, no further failure is likely to happen in service conditions until the element has been replaced. It neglects thus possible further deformation or breakage of the fractured element due to significant time-temperature dependent behaviour of the interlayer polymer material, between the accident fracturing the element and the moment of first intervention for safeguarding the concerned part of the building. This seems again a fairly acceptable assumption for fractured elements in a framed vertical configuration, with their own weight transferred via compressive efforts between fragments.

I.5. Safety performances of laminated glass works

Previous sections have shown that the assessment of safety performances of laminated glass products as conceived in product standards do not deal explicitly with possible different intended fields of use. Neither are they dealing with the evaluation of the representativeness of the selected test configuration in comparison with a wider range of similar configurations, nor with regard to different environments or test conditions (ambient temperature, etc.). Expression of one type of safety performance does not provide a characteristic value usable for design (calculation) purposes. Finally, the assessment method does not evaluate the contribution of the individual components of the laminated glass element to its safety performance, and does not give guidance for that purpose.

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[62] The publication order of the different standards shows that it is difficult to fix a clear and unambiguous terminology before assessment methods and classification criteria are available; unfortunately, this does not prevent possible further confusions. In this context, the term "security glazing" has to be understood with the same general acceptance as "safety glazing" used earlier in this chapter.

[63] In the case of the pendulum test according to EN 12600, it is even not immediate from the expression of the test result whether the element would still be able to resist its own weight after the impact, if the same configuration (specimen and frame) would be set in a sloped or horizontal position instead of a vertical one. In the case of the hard body drop test according to EN 356, a successful test assesses that the fractured element can at least resist its own weight, for the tested configuration and test conditions (test temperature, duration between the moment of the last impact and the moment of the evaluation,…), without consideration for the amount of fractured glass components nor for the density of the fragmentation patterns.

However, with the evolutions in fields of use of laminated glass products, such questions became meanwhile much more relevant.

In this section an overview is given about safety performance requirements on construction works including (laminated) glass elements, and how their compliance is assessed according to the current practice.

Existing approaches in design codes or standards, technical guidelines and specifications are taken as a starting point, because these often are the first documents considered by designers in practice. It illustrates some particularities of the design process and their influence on the formulation of safety performance requirements for laminated glass elements, in particular with regard to possible time- and temperature dependent contribution of interlayer components and the stability of their properties during their lifetime. This will lead to identify application scopes for conducting the assessment of safety performances in practice, and in particular when these are dealing with the post-fracture behaviour.

I.5.1. Performances of glazing and structural glazing units

Glazing units refer here to flat glass product used in windows, as vertical façade glazing or as sloped overhead glazing, namely elements having a primary glazing function. A *glazing function* consists thus in separating an indoor space from an outdoor space, or more generally in isolating two spaces of each other's by means of a separation wall the glazing unit is part of. The particularity of the design process of glazing units consists in an integration of the constraints raised by the various associated performance requirements (thermal insulation, acoustic insulation, etc.), possibly leading to contradictory specifications on the most suited glass product to use.

Glazing applications are commonly not considered as load-bearing elements, in the sense that they do not play a role in the global stability of (part of) the construction. Current design codes still generally impose or recommend to not take into account the stiffness of the glass elements when calculating the necessary sections of the frame (millwork). Notwithstanding, the glazing unit has to transfer loads due to actions directly applied on it, as wind, snow, and impact actions. Besides, the setting of the glazing unit in a frame always involves some level of constraints (in plane compression efforts)[64], to contribute to other satisfactory performances, as water- and airtightness, and avoid undesired vibration movements of the glazing unit. This configuration of glazing in a set frame is quite favourable for the safety and post-fracture performances of the element.

[64] Setting constraints are usually not quantified (and probably not easily quantifiable), and seem generally implicitly assumed to be negligible.

The concept of 'structural glazing' appeared with the development of glazing elements of larger dimensions, but mainly in combination with the apparition of alternative fixing systems, with two typical configurations. The first are referred to as structural sealant glazing systems (SSGS) : the glazing unit, instead of being set into a frame, is attached on a frame by means of a peripheral adhesive joint. The second are point-fixing systems, with the glazing unit fixed to the structure by means of four or more mechanical fixations, involving the use of particular (laminated) glass products with holes through the whole thickness or only in one of the outer glass sheets. The assessment of load-bearing and safety performances gained importance compared to traditional glazing configurations, but did not led to fundamentally new design philosophy and assessment methods[65].

It is worth noticing that, with regard to the definition of "product family" according to EN 14449 (see section I.4), no extension of the "product" specifications is necessary for extending the intended field of use from framed glazing to other peripheral or adhesive fixing configurations. In comparison, development of point-fixing systems with mechanical fixations through foreseen holes in the laminated glass unit corresponds to an extension of the "product family", as the usual annealed float glass sheets for the glazing components were replaced by thermally toughened glass sheets, in order to reinforce the local strength near the holes. It is justified to consider the change of glass type as an extension of the product family : indeed, the lamination process of two thermally toughened glass sheets is more sensitive because of the larger flatness imperfections of the glass sheets caused by the tempering process (Domingos and Schimmelpenningh 2011; Jalkanen 2005). It confirms that the development of a concept of "laminated safety glass product" in product standards was clearly mainly driven by concerns about quality control measures of the lamination process, and not much by the intended field of use.

The switch from framed systems to point-fixing systems however leads to different design situations with regard to post-fracture performances. The first reason is related to the consequences of change in fixing configurations on the load-transfer mode when the element is fractured, the second to very different

[65] An European Technical Approval Guideline, ETAG 002 (1999), has been released for providing guidance for the assessment of SSGS works and systems; an updated version has been released in 2013. No equivalent European guidance document for point-fixing systems in façade has been established to this day, but there exist a few technical guidelines released by national standardization or assessment bodies (among others in France and Germany). According to Springborn (Springborn 2004), other types of EOTA guidance documents, CUAP (document resulting from a Common Understanding of Assessment Procedure), have been established for assessing some point-fixing systems and other 'particular' applications : this type of guidance documents is much less known than ETAG's, because they can be established by a single approval institute member of EOTA, whereas ETAG's have to be endorsed by an appointed European Working Group. It is thus the question whether provisions in guidance documents such as CUAP's fundamentally differ from national or project-specific ones.

fragmentation patterns of glazing components typically used in each system. If one compares a SSG system with a point-fixing system with similar configurations of product and of application, the safety performances of each system are rather different when comparing their respective initial resistance (non damaged configuration) and their post-fracture behaviour. Whereas the initial resistance is more influenced by the difference in glass component type rather than in fixing conditions, the post-fracture performances are influenced by both in undetermined proportions.

Expression of performances and assessment methods for various glazing systems is thus conceptually not straightforward. It is further complicated by fragmented expressions of performances, between different technical guidelines and standards, issued by CEN and EOTA respectively for what concerns the European level, therefore with relative little apparent consistency in the expression of performances and of related assessment methods.

Table I.8 – 'Safety in use' requirements on glass works involving impact performances and tests, as mentioned in harmonised standards (CEN)[66]

		Laminated glass products	Windows	Facades (curtain walling)
Performance "Safety in use"		EN 14449 (2005)	EN 14351-1 (2006)	EN 13830 (2003)
●	Pendulum	EN 12600 (2003)	EN 13049 (2003)	EN 14019 (2003)
▶	Burglar resistance	EN 356 (2000)	EN 1627 to EN 1630 (2011)[67]	not mentionned
▶	Bullet resistance	EN 1063 (2000)	EN 1522, EN 1523 (1999)	not mentionned
●	Explosion resistance	EN 13541 (2001)	EN 13123-1 (2001), EN 13123-2 (2004)	not mentionned
● : not perforating impactor type ▶ : perforating impactor type				

Expression of performance requirements on a glazing element in a particular project configuration to different types of actions is codified by "action models", allowing to determine a design value of each action corresponding to a specified probability of exposure of the element to this action. Many action models were harmonized at European level, so far as possible, in the first part of the

[66] Table I.8 and Figure I.4 are inspired from similar ones published in WTCB-Contact 2006/2.1 (see www.bbri.be), the classification into not-perforating and perforating type impactors is a personal addition - see discussion in the main text.

[67] This series of four standards has been firstly released in the form of pre-standards ENV (also referred to as "experimental standards") from 1999.

Eurocodes[68]. Specific performance requirements of glazing elements to impacts, attacks and explosions and corresponding assessment methods by testing are not dealt with in Eurocodes, but are part of other product standards released by the CEN since 1999 on the one hand, and of other guidance documents issued by EOTA on the other. An overview is given in Table I.8 about the 'safety in use' performances in CEN standards for windows and curtain walling facade, with a direct correspondence with performances mentioned in Table I.3.

While each safety performance is expressed at the level of the built application, the product standard for windows refers to other test method than the one for laminated glass products (Table I.8). Figure I.4 compares drop heights corresponding to the resistance classes resulting from an evaluation by means of a pendulum test for glazing and windows products. The test methods look very similar, but have in fact other assessment objectives. Test according to EN 12600 aims at characterizing the safe behaviour of the glazing component (the "glass product"), tests according to EN 13049 and EN 14019 rather intends to evaluate the contribution of the frame or supporting structure to the final performances of the assembly. The role of the used glazing panel in both tests is not the same, and accordingly the test protocol, the evaluation criteria and the amount of required test specimens necessary for performing the assessment in each case are different ! In a general way, pendulum test protocols according to EN 13049 and EN 14019 are less precise and let a larger margin of appreciation to the test operator, for instance in choosing position of impact point and in interpreting conformity criteria[69], in other words they rely much more on the experience and capacity of judgement of the laboratory.

Corresponding test methods for evaluating performances against burglar attack of windows and laminated glass products are less similar to each other, and this is justified by the local nature of the attack.

The specifications in respective standards for test methods on work configurations are not more precise about assessing the post-fracture performances and the choice of the test configuration in relation to an "intended field of use", and rely thus on the experience and development strategies of the stakeholders involved in the assessment process. Noteworthy, all test methods mentioned in Table I.8 are leading to an ITT performance mentioned in the documentation related to CE-marking for the corresponding 'construction product'.

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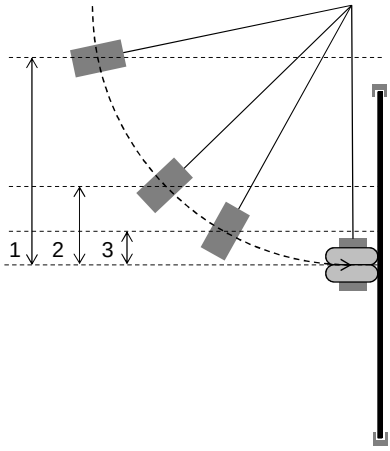
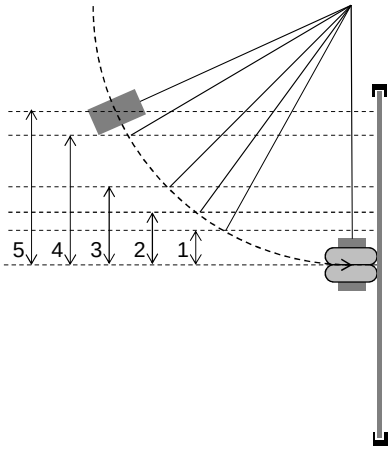
[68] For instance exposition to wind and snow actions can be evaluated according to provisions in EN 1991-1-4 and EN 1991-1-3 respectively. See also paragraph I.3.

[69] Classification of performance against pendulum impact distinguishes resistance classes according to impacted side (from outdoor or indoor side) for façade element, while similar classification for a window element does not make this distinction in terms of resistance class, probably due to the implicit assumption that the resistance class is more likely to be the same for the two impacting sides in the last case.



a) impact heights EN 12600



b) impact heights EN 13049/EN 14019

Tested element	Glass product	Frame (window), support structure
Standard	EN 12600 (2003)	EN 13049 (2003), EN 14019 (2003)
Drop height [mm]	Drop height level	Resistance class
(0)		0, E0/I0
190	3	
200		1, E1/I1
300		2, E2/I2
450	2	3, E3/I3
700		4, E4/I4
950		5, E5/I5
1200	1	

Figure I.4 – Schematic comparison of impact levels for pendulum test for (a) glass products and (b) glazing applications, according to EN-standards

However, the choice of the glass product used for performing the assessment of a windows frame and the choice made in the design of a project configuration are different issues. In the first case the responsible of the test should in principle select a glazing element for the test which is unfavourable for the performance of the frame, while in the second case, the resistance classes for the glazing component and for the frame component have to be selected in order for the design assembly to meet the overall performance requirement expressed on the construction part[70].

[70] Altogether complying to all other performance requirements specified by the design code or project specific specifications.

Above considerations highlight different issues. Firstly, the expression of safety performances against dynamic actions on construction parts, and on glass works in particular, appears as a quite complex problem. The apparent simplicity of standardized test methods used for experimental assessment is counterbalanced by the complexity in choosing the most representative test configuration and test conditions. Indeed, this choice requires that the "intended field of use", or application scope, is well identified beforehand[71], and may further vary according to the initial understanding one has of the specific mechanical characteristics of each component involved. Secondly, influence of (testing and design) ambient temperature and of possible ageing effects on the safety performances are not considered in the assessment process (ITT), suggesting these aspects are negligible or implicitly covered with respect to glazing applications. Finally, assessment of residual performances of the fractured element after the impact is limited to a conventional fail/pass criterion, which basically assesses the preservation of some retaining or containing capacity, in a reference configuration and for reference test conditions. No further guidance is given in comparison with the laminated glass product standards about for which design conditions the assessed performances are deemed to satisfy, and no methodology or reference is given how to address these questions.

The implementation of the design and assessment framework developed in European standards and guidelines in national design codes[72] has not been an easy work, in particular for defining in which design conditions new assessment tests are required. The conceptual and methodological gaps non identified in European standards and technical guidelines lead inevitably to new national standards and guidelines, which on their turn delay the harmonization purposes.

In summary, the assessment of safety performances of laminated glass products used in glazing applications appears as relatively complex. It is a multi-scale problem, but also – and this probably differentiates more the assessment problem with regard to other industrial sectors using laminated glass products – it involves multi-scale design processes, with many stakeholders involved, but at different steps and levels. In this process, concerns about the influence of time-temperature

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[71] The definition of the application scope in this context covers in fact two strongly inter-related aspects, in terms of configuration range on the one hand (ranges of configuration for the "assessed system") and of performance range on the other hand (covered by the different "performance/test" standards).. Accordingly, questions of "representativeness" of the test configurations are addressing these two aspects. However, from the moment on test methods are considered as fixed, it tends to define individual application scopes in terms of configuration ranges for each standardized performance independently of each other.

[72] Principally for the last versions of NBN S 23-002 (glass works), NBN B 25-002-1 (windows and facades) and NBN B 03-004 (guard rails), see Figure I.1 and Table I.1. Noticeably, a variety of national standards in other European countries are dealing with "overhead glazing" and "guard rails" applications, however with different related specifications and assessment requirements, among others in France, Germany and United Kingdom.

dependent properties of polymer components (interlayers) on the safety performances tend to land in a second row category.

This seems not a critical problem for framed or supported vertical glazing units, which, in absence of any permanent tensile effort in their plane after the impact, are not suspected to collapse all of a sudden. But for non-vertical glazing units and all other configurations with other supporting conditions, addressing the design issue of the post-fracture performance raises questions. How much time will the fractured element hold in place ? How can this behaviour be quantified ? And is it accordingly possible and relevant to take this result into account concretely in the design ?

This kind of questions lead to complementary performance requirements at application level, imposed at national level in different EU member states through national standards or technical guidelines, technical approval procedures or for individual projects, for instance for particular, "non-conventional" configurations. In many cases, complementary experimental investigations on different test configurations, with identical or different impact bodies, test protocols and evaluation criteria, are required by the national authority or can be requested by one involved party. These types of tests are also generally performed only at ambient temperature, and do not address question of representativeness on a different way as in the methods presented here above. Conceptually, these tests are rather validation tests than characterization tests, in the sense that they do not evaluate a quantitative performance of a product, configuration or system, but rather verify the compliance of a specific configuration with regard to pre-defined criteria for a specific project.

From a normative point of view, the *current* situation is that, *strictly considered*, the assessed performances of laminated (safety) glass products by means of standard tests as EN 12600 and EN 356 do not guarantee the final performances of the built element configuration, but rather assess that with the use of laminated safety glass products a range of application configurations has a 'reasonable probability' to reach a certain level of performance. They neither characterize properties of products usable for design purposes. On the other hand, test configurations and methods prescribed for assessing the corresponding performances on a larger application level (laminated glass element + frame or supporting structure) rather correspond to validation tests than characterization tests. Consequently, in practice the notion of "similar configurations" arises, but it is left to the designers and control authorities to determine which criteria are relevant to define the grade of similarity. Noticeably, most of the related issues involve evolutions or extensions of application configurations ("intended field of use"), generally without extension of the laminated glass product configurations ("product family") according to the definition in the harmonized product standard.

I.5.2. Performances of structural elements in laminated glass

While resistance to impact and consequent post-fracture behaviour of laminated glass units conceptually seem to address very similar issues, whether it is used as glazing, structural glazing or structural glass element[73], there are some important conceptual and practical differences.

Conceptual differences are mainly related to different safety concepts, design and assessment philosophies. This is a complex issue, investigated and discussed quite extensively by Bos (Bos 2009)[74]. The transposition and interpretation of design rules for products and systems initially intended for 'non-structural' use to more 'structural' ones is facing extrapolation barriers of different nature.

A major issue is related to the changes of 'standardization framework' introduced in paragraph I.3 above, in particular the assimilation of the performance-based approach philosophy for standardization, and the consequences it has on assessment methods and on implementation issues for new design codes. Another important aspect, which tends to be underestimated, is the relative lack of understanding structural engineers and architects still have about the behaviour of polymer components used in structural elements in general, and of interlayers and structural adhesives in particular[75]. More precisely, it concerns problems to identify which of the usually considered assumptions used for calculating resistance and deformation of structures and structural elements are not applicable anymore, and which ones are lying behind the usual experimental approaches and associated statistical analysis methods proposed in the various standards. In other words, limits to the use of "traditional" calculation models and experimental assessment methods for similar structural systems are not identified because some fundamental implicit assumptions behind the concepts of "material" as they are used and implemented in design codes neither are.

This tendency can lead to critical issues at the moment of setting up an initial experimental program, as it might be non univocal which guidelines and experimental methods are effectively relevant to consider.

A more detailed analysis about these issues has been proposed in (Delincé and Belis 2013). It is pointed out that there is still a gap to fill with respect to general

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[73] The conceptual limits of each "application" category remain relatively vague, and result among others of the structuration and harmonization philosophy in the European framework.

[74] Assessment method of safety performances of structural elements in laminated glass proposed by Bos is summarized in Chapter II, paragraph II.3. According to Bos, shortcomings in codes with regard to safety concepts result from "*the lack of an integrated safety approach*"; he noted also that "*even in the practical execution and assessment of codified experiments […], they appear to be many ambiguities that mainly stem from an incomplete or insufficiently detailed description of the experiments*" (Bos 2009).

[75] In fact, this does not address a capacity of understanding the scientific aspect, but rather the difficulties of *assimilation* and *integration* in their conceptual and methodological framework.

guidance documents available at European level about experimental assessment methods. Two important ones are on the one hand the core part of the Eurocodes, the EN 1990 (CEN, 2002), also referred as Eurocode 0, and more specifically its Annex D "Design assisted by testing", and on the other hand the EOTA GD 003 "Guidance document 003 : Assessment of working life of products" (EOTA, 1999). These two documents are rather intended as general guidelines for CEN and EOTA Technical Committees to draft more specific ones for different applications, systems, products and materials.

In principle, the Annex D of the Eurocode 0 can always be used[76], however it does not give specific guidelines with regard to load-bearing polymers, and more generally does not address issues for accounting with time-temperature-ageing dependent properties of products and systems. In fact, this annex seems to have been rather conceived for assessing alternative or new configurations of systems and connections made with known materials (among which concrete and steel).

The EOTA GD 003 rather addresses issues related to the assessment of new materials and products[77]. It provides a general framework and lists of ageing agents ("degradation factors"), with identification of the ones which different categories of products and materials are known to be potentially sensitive to. It also proposes a general methodological framework and describes the different categories of experimental approaches and related generic test methods.

In summary, these two general guidance documents rather list the eligible and recommended approaches and the main points of attention to deal with particular cases, and give general guidelines about test and analysis methods. These however appear not specific enough to set up relevant and economical experimental programs for specific polymer components and laminated systems they are part of.

Besides these two general guidelines, more specific test methods have been developed and implemented in standards and ETAG's, or other documents with similar statute. Two in particular are widely referred to in scientific literature reporting about the development of experimental campaigns addressing or involving the behaviour of polymer components (Delincé et al. 2007; Ensslen 2007; Louter et al. 2011). The first is the EN ISO 12543-4, already presented in paragraph I.4.1. The second is the ETAG002, already mentioned in paragraph I.5.1, which contents in particular specific guidelines and test methods to assess the mechanical properties of the structural (silicone) sealants of structural glazing

[76] The Annex D has an informative statute in the main (European) part of the text, which has been kept in the National Annex for Belgium.

[77] This guidance document mentions to be partially based on a draft international standard (ISO) entitled "Systematic Methodology for Service Life Predictions of Building Materials and Components". It is not clear however if the latter has evolved towards or has been included into an approved ISO-standard to this day.

systems, among others mechanical tests following artificial ageing tests, and mechanical tests performed at different test temperatures. These two documents and other similar standards/guidelines are generally already quite specific, as important assumptions have been made to specify the standardized experimental assessment methods, and associated test configurations and conditions. These assumptions include for instance the type of polymer component, the type of system configurations which the latter is part of, and knowledge about the associated typical response and sensitiveness to various ageing agents of the product configurations.

However, the nature of ageing phenomena, which are assumed to be present in the different ageing conditions considered, is not always made explicit. More specifically, it may be unclear whether the expected ageing effect is of chemical or physical nature or both, and whether it is reversible or irreversible. For instance, with regard to the effect of temperature and humidity, it is not clear whether their effects are assumed to be correlated or not. Similarly, sensitivity to temperature and creep load are assessed separately, while a component exposed to a permanent load in design conditions is likely to be exposed to combined effects due to temperature variation and creep. With regard to possible ageing effects on the adhesion characteristics, it seems also useful to distinguish if the ageing effect is directly exerted at the level of the interfacial plane between the polymer and its substrate, or if it acts primarily on the bulk properties of the polymer which affects its adhesion level (effective or apparent one) with the substrate. In some cases, for instance for laminated glass elements, ageing effects are only evaluated in terms of visual changes, not on the basis of corresponding changes involved at mechanical level.

Accordingly, it seems there is a *minimum of understanding to have about the nature of phenomena involved*. In what extent these phenomena differ between different polymer products and of other materials ? What kind of border effects are associated to each effect ? These are important questions to account for to be able to select the most appropriate experimental investigation methods and scales, and develop relevant assessment approaches.

Leaving temporarily the conceptual and methodological approaches aside, it is in fact possible to identify different types of concrete fields of extension[78] addressed by 'structural glass' works. Instead of trying to define boundaries between possible identified conceptual categories (Green 2013; Siebert and Seel 2011; Siebert 2006; Springborn 2004), it is proposed to firstly identify the different extension fields typically differentiating a structural glass application from other glass works, with the hope that it would give a more practical view of the

[78] The concept of field of extension is elaborated in section I.6 below and further developed and discussed in Chapter IV with regard to experimental investigation methods.

questions that general guidance for designing and assessing "structural glass applications" have to deal with.

1) Product configurations and family of products

Laminated glass products for 'glazing applications' are generally limited to 'simple' laminated glass units, made of two glass sheets (generally of the same type and same thickness) and one interlayer. They are often integrated as a component in an insulated glass unit, which is also a particular type of 'glass product', and is accordingly covered by a harmonised product standard. Safety performances of end applications integrating this category of products, as resistance to body impact, raise similar questions about test configurations and application scopes. In general, the family of laminated glass products considered for the assessment of a "structural glazing" system still remains relatively limited with regard to the production volumes covered by the scope of the assessment.

Laminated glass products intended for 'structural' use are almost always made of at least three glazing sheets and two interlayers, where the assembled configuration can be made of glazing sheets of different type and of different thickness. Complexity of laminated glass products increased with inclusion of various types of inserts and reinforcing components (Carvalho et al. 2011; Feirabend and Sobek 2009; Neugebauer 2006; Puller et al. 2011), in complement to components identified as 'glazing' and 'interlayer'. Besides, lamination of hybrid products, namely with different types of materials or surfaces in contact with the interlayer component, is possibly more sensitive because of (slightly) different adhesion mechanisms. Achievement of homogeneous adhesion grade can be complicated for more complex configurations with regard to used production processes, for instance in function of the sensitivity to (local) pressure applied during the one or other step of the lamination process.

Simultaneously with the increased complexity and variety of possible configurations on the basis of a limited set of basic components, the production volumes for this type of products are also much smaller than the ones of 'families of product' developed for the glazing market. This represents an important constraint on the amount of tests which can be reasonably imposed within an assessment procedure.

In many cases, the used polymer products used as interlayer are unchanged, although processing methods usually have to be adapted in some extent. The amount of 'particular' configurations within a 'family of products' which is potentially to be addressed by the assessment can increase dramatically, with potential consequences on the amount of requested tests.

The extension of product dimensions leads to reach critical proportions, which can rise new production issues, new construction issues and new design

questions, among others with regard to new critical failure modes (see also point 4) below).

2) Critical dimensioning performances

Consequences of the failure of a "structural" element in laminated safety glass are generally larger than the one of a "structural glazing" element. Requirements on residual performances in case of failure, even if not yet specified with much accuracy, are more and more likely to influence the result of the design significantly, simultaneously with the demise of (safety and non-safety) performance requirements specific to glazing applications. Similarly, the ratio between permanent loads and variable loads[79], or between loads of long and short duration, is increasing. 'Strength'-dominated design moves towards 'robustness'-oriented design, which require another type of material/product characterization (loading curve in place of single conventional value of strength or deformation,…).

3) Applicable methods for quality control of lamination process

Simultaneously with the increase in variety of configurations inside a 'product family' (point 1) here above), common destructive test methods used for controlling the adhesion level in quality control, such as the Pummel test and the CST test[80], reach limits of their application scope. A Pummel test requires a relatively large evaluation area (respective planar dimensions above ~10 centimetres), and CST tests can be confronted to a variety of possible border effects[81], disregarding the feasibility issues. Besides, with each test method only one of the two adhesion interface of an interlayer can be controlled.

4) Critical failure modes and design situations

More deformation and failure modes are likely to be considered in the design problem, and for each one the contribution of the interlayer component may address another part of its behaviour domain, possibly with different levels of expected or specified accuracy. For instance, the required level of reliability for characterizing the shear transfer properties of an interlayer component could vary whether they are used for assessing the stiffness of laminated glass products either against wind actions when used in framed glazing units, either with regard to the resistance to lateral buckling of a primary laminated glass beam element on which an important relative fraction of permanent load is applied in design conditions.

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[79] Permanent loads are due to the own-weight of the element and other static loads of long duration; variable loads are loads of shorter duration, as climatic loads and service loads.

[80] These two tests are shortly presented in Chapter II section II.5 and discussed further in Chapter III paragraph III.3.4.

[81] The border effects can be of different nature and magnitude, among others according to the size and possible production methods of the test specimens; see also Chapter IV.

There is one evidence so far : structural use of laminated glass products means in fact that the "intended field of use" (application scope) is larger, more vague, and potentially more difficult to delimit. Each new project is likely to correspond to a demand in 'extending' the application scope with regard to one or more fields. Among the extension fields, questions addressing the 'time-temperature' dependence of mechanical properties of the interlayer component and their influence on the behaviour and performances of a structural element in laminated glass gains in importance, *in combination with* the various other aspects of extension of the application scope. However, due to the particularities of polymer materials used as interlayers, design of experimental investigation methods for answering these questions and interpretation of test results is not straightforward !

A very schematic representation is proposed in Figure I.5, in an attempt to situate design questions and assessment approaches[82]. The curves represent qualitatively the expected variation, in function of ambient temperature, of two complementary performances, the resistance to impact and subsequent residual load-bearing capacity[83], of a structural element in laminated glass for a given configuration. In this schematic view, a vertical line represents a test on this reference configuration, executed in reference test conditions, at a reference temperature T_0. Diamond points are representing the corresponding measured performances from a reference test[84]. A well-designed laminated glass product is in fact likely to show optimal performances for the specified reference test conditions.

Consecutive questions arising about variation of performances in function of temperature from a design perspective will lead to formulate complementary requirements, which is equivalent to fix new limits participating to the definition of an application scope. Let us assume that the formulated performance requirements are represented in this case in Figure I.5 by the horizontal dotted lines : it seems then logical to complete the evaluation by means of complementary tests performed in corresponding test conditions. One specific problem for building applications is that the performance requirements on products and systems are not absolutely fixed, given data. Indeed, they may vary in function of project specific requirements : the variations are justified on the one hand by real different climatic environments for each construction project and different usage-related exposures of the considered product or system, and on the

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[82] In a recent contribution (Delincé and Belis 2013), a similar reflexion was presented in a more general perspective, namely by considering structural elements which performances are depending in some extent on adhesive polymer components.

[83] The qualitative shape of the curves stems in with to the one of Figure I.3, and will be further confronted in next chapters.

[84] For instance the performances as evaluated by the pendulum test or the hard body drop test presented above, if the test configuration (dimensions, frame…) is judged representative of the built configuration (construction work).

other hand by differences in legally required safety level (from national regulations, design codes, or project-specific specifications). Also the accepted assessment methods for justifying the design may vary in accordance. In other words, except in the case of full-scale tests designed to validate a built configuration for a specific project, the position of the horizontal lines is not fixed when the experimental program for assessing a structural concept or construction product is conceived and executed, but is fixed at the moment one desires to build it in practice for a specific project. The balance between requirement levels on the two complementary performances may also vary for an identical configuration according to the project specifications; consequently, the two horizontal lines representing the performance requirements can move independently of each other in a vertical direction. If other safety performance requirements are expressed on the same construction part, they can be represented by means of complementary figures similar to Figure I.5.

Figure I.6 looks similar, but represents now the question about the experimental fields of investigation to consider from the perspective of the assessment of a family of products or applications. It highlights two practical problems : the increase of experimental costs in function of the extension of the temperature range to be covered (dashed curve), and the uncertainties about the range to cover (in function of the range of possible positions for the horizontal lines in Figure I.5). The experimental costs include cost of the extra specimens necessary for performing the complementary tests in conditions corresponding to the identified boundaries of the application scope, fixed costs of the used test infrastructure, development time for assessing the intrinsic quality of the experimental method in case of new test configurations, consequences on the overall time necessary for assessing a particular concept, etc. The shape of the curve “experimental costs” accounts qualitatively for some physical limits in extending the investigated range of test temperature[85].

However, Figure I.5 and Figure I.6 still represent a simplification of the general problem to be dealt with : temperature is only one variable parameter of the identified intended field of use.

Complementary questions arising with regard to particularities of polymer materials are developed in next chapters. One particularity for polymer materials is to be sensitive to possible *combination of effects* between different ageing agents, and between an ageing agent and a simultaneously applied mechanical effort of longer duration (in particular in the form of creep and fatigue effects), in function of the respective intensity and level of the one and the other.

[85] Corresponding concrete experimental aspects and constraints will be highlighted in Chapter IV.

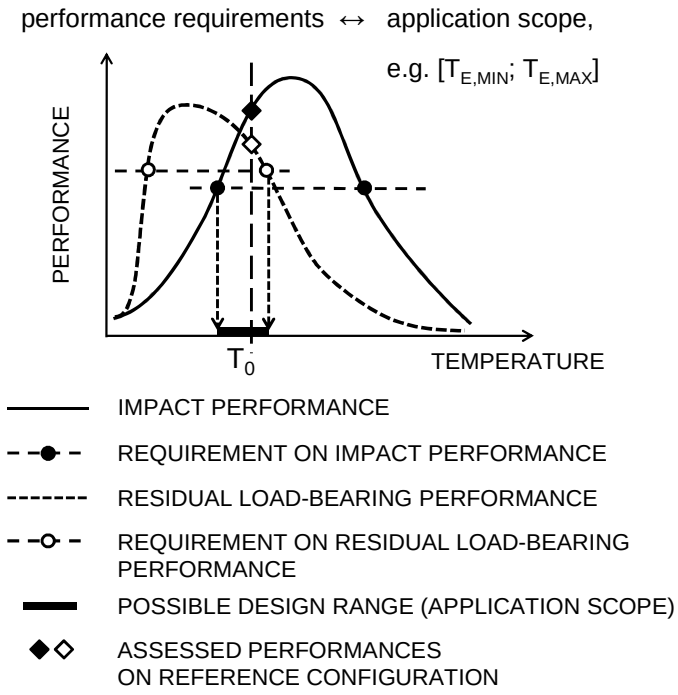

Figure I.5 – Schematic formulation of the assessment problem in regard to performance requirements (Delincé and Belis 2013)

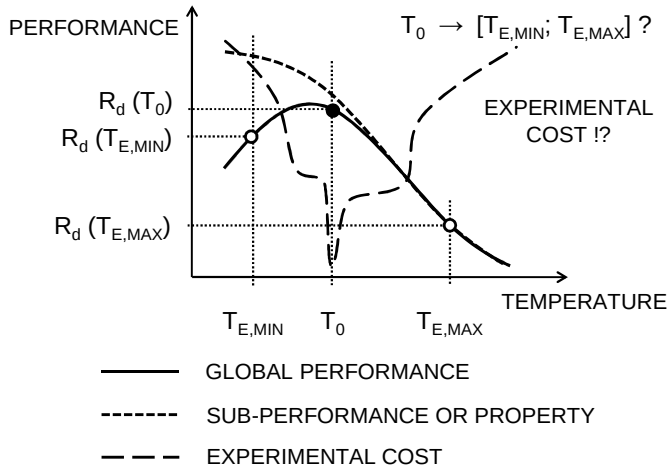

Figure I.6 – Schematic formulation of the assessment problem from an experimental perspective (Delincé and Belis 2013)

In summary, the assessment of the interlayer contribution to safety performances in general, and to post-fracture performances in particular, of laminated glass products used as structural element in non-conventional configurations, has to face a variety of conceptual and practical issues. Especially, the lack of *comprehensive* test methods or experimental approaches hinders to 'fill the (knowledge) gap' between providers and users of laminated glass products and systems. Consequently, the development of appropriate and comprehensive assessment methods and safety concepts is complicated.

I.6. Outcomes and problem statement

Expression of safety performances of laminated glass products in the particular context of construction works has been introduced in the present chapter, together with an overview of existing assessment methods and approaches. The relatively fast evolution in the development of laminated glass products on the one hand and their field of use on the other is accompanied by new questions. These concern the contribution of the interlayer components to the overall safety performances of built configurations, the involved product and material properties and test methods for characterizing element performances and material properties.

The overview of the current state of the standardization framework and ongoing developments at European level highlights some particularities of design processes in this field, and the associated concepts and difficulties for assessing laminated safety glass products and construction works in laminated glass, in particular with regard to safety and post-fracture performances in function of time-temperature dependent phenomena in polymer components.

A first identified issue concerns the description and limitation of different application scopes. An important question addresses the concepts of *family of products* with regard to vague and evolving *intended fields of use*. For each of the two concepts, different **Application fields (AF)** can be defined, in order to distinguish issues of different nature in questions addressing the possibilities and the limits for extending application scopes. Table I.9 proposes different categories of AF's, and for each an inventory of field descriptors, corresponding to parameters allowing a quantitative description. Accordingly, an application scope can be defined as a combination of ranges of application fields.

Extension of intended fields of use of laminated glass products can be translated into a range extension of one or more application fields. However, the various application fields are not evolving independently of each other, some being directly or indirectly related with each other.

Table I.9 – Description of application scopes (intended fields of use) and product families

Application Field (AF)		**Examples of AF descriptor**
	Product : Material	- type(s) of glazing sheet - type(s) of interlayer product - type(s) of embedded inserts and reinforcements
	Product : Geometry and configuration	- description of ranges of geometric configurations : composition, amount and thickness of layers; inserts; … - possibilities and limits for lamination sizes - possibilities and limits for cutting sizes
	Product : Processing	- production methods : lamination, cutting (incl. holes,…), edge finishing,…, possibilities and limits in function of considered configuration ranges - level of standardization of the various processing steps
	Product : Connections	identification of possibilities and limits for connecting the laminated glass product into a construction work : zones and features intended to be used / avoided for connecting the element; (in)compatibility with other materials and with service conditions
	Application : Design : Performance requirements	Expression of performance requirements : - resistance to impact(s) / source(s) of damage - loading cases : type, configuration and extent of individual action; combination rules (ULS, SLS,…) - exposure conditions : temperature, ageing agent,… due to climatic and service conditions (cleaning,…) - non-structural performance requirements affecting the design : acoustic, insulating, light control, etc.
	Application : Design : Geometry and Configuration	- Element dimensions : planar dimensions, (maximal value of) total thickness, functional constraints in function of performance requirements and design configuration (with regard to edge finishing, etc.) - Connections and fixing configuration and conditions, intermediate pieces (mechanical connections) or components (adhesive connections,…), possible consecutive requirement on edge or surface finishing,…
	Application : Execution : Processing and assembling methods	Identification of execution steps likely or intended to induce constraints (stress) into the laminated glass element or any of its component.
	Application : Service conditions	- measures to take in case of damage / failure : safeguarding and replacement of the damaged element, - measures to take in case of change/deviation of service conditions with regard to initial assumptions or specifications used for the design - control and monitoring in service conditions (optional)

The application fields can be used to compare different design configurations, and define the grade of similarity between them. Similarly, they also can be used for identifying or selecting characteristics of reference configurations with regard to assessment questions, and compares a design configuration with a test configuration used for assessing it. Finally, the proposed definition of categories distinguishes characteristics of products and of end applications.

The analysis grid proposed in Table I.9 must allow to situate all design questions in relation to each other, and to identify all related design and control parameters. It must be clear, however, that this table is not yet complete and must rather be seen as a 'work in progress' and a support for further developments. In particular, every 'field descriptor' should identify the relevant *quantitative* parameter(s) to consider, and which parameters are involved in which design (sub)questions. It is intended to be used for identifying assumptions and conditional data, namely types or values of some field descriptors which are respectively meaningful or valid for identified limited ranges of other AF's, and for identifying missing data and possibly missing reliable test or assessment methods behind the missing data. Determination of most of the parameters should refer to design codes, measurement or test methods, and for application fields associated to Product categories the *assessed* properties or characteristics and the other ones.

To conclude this chapter, developed analysis leads to following questions about design methods and assessment strategies :

1) How could and should existing test methods used for assessment of laminated safety glass products be completed to distinguish the contribution of individual components to the overall safety performances ? What are the *characteristic* properties of each component involved ?

2) Which (mechanical) properties of interlayer materials are involved in safety and post-fracture performances of laminated glass units and systems ? According to which methods can these be *characterized* for design purposes, in particular properties potentially significantly sensitive to time-temperature-ageing effects with regard to service conditions ?

3) To which extent are safety performances of a laminated glass element as a *construction work* (resistance to impact, etc.) depending on the *product* properties and on other characteristics of the installed *configuration* (in function of element configuration, type and configuration of connections / fixings) ?

4) Which characteristics of the laminated glass product or product family (from preliminary technical documentation) could be accounted for to select or develop a suited experimental investigation program for assessing their safety and/or post-fracture performances ?

5) How to conciliate application and product-oriented assessment procedures, in particular to keep the amount of requested tests within reasonable proportions

in regard to the identified application scopes ? How to integrate vague and evolving application scopes in assessment processes, in particular with regard to particularities of adhesive polymer materials in terms of mechanical behaviour and influence of production processes on their mechanical properties ? How to express and assess limits of possible fields of use of laminated safety glass products for structural applications ?

6) Which material and structural models are applicable for characterizing the contribution of interlayer materials to the response of fractured products and systems, and how can the corresponding design parameters be calibrated or validated ?

7) In summary, are the safety concepts, assessment approaches and calculation methods developed for laminated glass products used as glazing unit appropriate and transposable for the design and assessment of structural glass works ?

Expected research outputs are :

1) Recommendations about suited test methods and experimental investigation approaches for characterizing the contribution of interlayer materials to the post-fracture performances of laminated glass elements, in particular with regard to their time-temperature dependent behaviour;

2) Contribution to development of integrated safety concepts and associated assessment approaches, in particular for distinguishing assessment of products, of components and of final applications.

Chapter II

Performances and properties of fractured laminated glass elements

"Science never solves a problem without creating ten more"
(George Bernard Shaw, 1856-1950, Irish playwright)

II.1. Introduction

Standardized test methods developed to assess safety performances of laminated glass products used in building applications are performed on test configurations supposed to be representative of the application scope of the products, and mainly address the glazing applications. The development of non-conventional structural applications in laminated glass is characterized by a larger variety of boundary conditions and more demanding performance requirements with regard to a safe behaviour in case of accidental breakage of the glass components. Simultaneously, although the production volumes of individual configurations are diminishing and the complexity of product configurations in terms of number of layers and types of components is increasing, they are still assembled with the same or similar types of interlayer materials. Besides, new ones are appearing.

In this context, the assessment of the residual load-bearing capacity of fractured elements is becoming much more important in comparison with more traditional glazing applications. In particular, the time-temperature dependency of the response of the laminated products to accidental actions and failure scenarios, and in particular the mechanical behaviour of fractured units, cannot be disregarded anymore. It appeared however already difficult to account for time-dependent properties of interlayers and laminated glass units for 'standard' glazing applications, in particular regarding their post-fracture behaviour, it is by consequent unlikely to be an easier issue to deal with for products used in non-conventional structural applications.

This chapter proposes to address this issue by means of a walk through the different scales of the problem and the identification of related experimental methods. It will firstly addresses the question whether the assessment of performances of fractured systems can be dissociated from the preceding response to impact and other accidental actions leading to breakage of glass components. Issues with current experimental approaches for testing new structural configurations are considered critically. Concepts of robustness of structures are introduced in parallel of failure scenarios and associated experimental approaches. By considering the underlying mechanisms during and after accidental breakage of glass components, it leads to distinguish different fractured states and load-transfer mechanisms, and to identify the critical ones. In a second step, possible test configurations for investigating specific load-transfer mechanisms are reviewed. They are analysed in particular with regard to their potential fitness for purpose to characterize the related mechanical properties of the interlayer components, with a central question in the background : how to account for the time-temperature dependent properties of the interlayer ?

II.2. From dynamic to quasi-static safety performances

The evaluation of safety performances of laminated glass products and construction works in current design specifications and test standards mainly focus on their response to a dynamic action. The performance is assessed by means of a (simple) pass or fail criterion evaluated shortly after the end of the dynamic response, which basically assesses whether the element lost its integrity or not. If the element survived the test, it is implicitly assumed that it has the ability to remain in place long enough to allow a safeguarding intervention before the replacement of the damaged element by a new one[1] (Chapter I).

This implicit assumption seems in practice often acceptable for traditional configurations of framed glazing units : there is no significant dead load applied to the fractured element, and the framed configuration in rabbet setting contributes favourably to the residual resistance. However, this is not true anymore for any other configuration of structural element in laminated glass, because of the applied permanent loads and other boundary conditions. These reasons relative to the structural configuration combined with the viscoelastic nature of the interlayer components explain that the residual load-bearing capacity is in general a time-temperature sensitive property. The time-dependence of the response of fractured element depends also, in a lesser extent, on a possible phenomenon of static fatigue in the remaining glass sheets or fragments. Static fatigue in glass and viscoelastic deformations of interlayers, possibly combined with each other's, can lead to delayed further crack propagations in the glass pieces. By consequent, further fragmentation processes can be initiated without modification of the external loading conditions, simply because of the time-dependence of the response of fractured elements to a static loading case.

In general, there is no correlation between the level of solicitation of a dynamic action (due to an accidental event or an attack action) and the level of consecutive quasi-static efforts present in the fractured element. Indeed, the dynamic action[2] as specified in a safety requirement[3] represents an extern factor. The probability

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[1] The related requirements can thus also vary according to the considered or applicable design specifications. Different time periods can be distinguished : time-to-discovery, time-to-evacuate, time-to-support (concept a little more restrictive than safeguarding), and time-to-replace, with corresponding values varying between a few hours up to 6 months (Bos 2009). It is sufficient to note that, as a natural trend, corresponding specified values for each time period will increase with the 'structural grade' of a non-conventional application in comparison with a common 'glazing unit'. Of course, effective safety level is influenced by the engineering understanding (in specifications and for management of accidental situations…), but also by psychological effects related to the perception of safety by the users : these factors are however difficult to codify objectively…

[2] When a safety requirement expresses a risk of attack rather than an accidental impact, the action is a 'rapid' succession of a series of individual dynamic actions, or spectrum.

[3] In the sense defined by the performance-based approach introduced in Chapter I.

of exposure to this action for a construction element rather depends on its functional role in the construction than on the structural system it is part of. In comparison, the level and the distribution of internal efforts resulting of applied permanent load during and after the dynamic action rather depend on the configuration of the element, and are possibly modified in function of its response to the extern action[4].

The *damage sensitivity* of the element expresses its response to the applied dynamic action and rather evaluates the consequence of the exposition. The most noticeable form of damage is the cracking and fragmentation of the glass components. Fracture of the glass components is a clear and necessary step in the process leading to an overall failure. The brittle breakage of glass is characterized by a sudden very fast crack propagation once a *critical damage state* is reached[5], preceded by a slow sub-critical crack growth known as *stress corrosion* or *static fatigue* (Haldimann 2006; Haldimann et al. 2008). The latter occurs only along surface zones where tensile stresses develop under the effect of static load. While the sub-critical crack growth velocity largely depends on external factors such as grade and distribution of surface damage, stress level and environmental conditions, the critical crack growth velocity can be considered as a material constant and is about 1500 m/s in soda lime silica glasses (Haldimann et al. 2008). Glass breakage is thus also a dynamic event, with a velocity at least one order of magnitude larger than the one of considered dynamic actions due to impact, etc. In general, the initiation of critical crack growth in glass leads to fragmentation, namely the crack propagates through the sheet up to its boundaries : the element is then broken in a series of fragments. In a few particular cases however, as in case of crack propagation under quasi-static loading in beam configurations, propagation of cracks can be limited to the tensile zone of the element (Louter 2011)[6].

Besides, independently of the cause of initiation of glass breakage, cracks propagation is accompanied by a release of elastic strain energy[7] (Bos 2010a). This strain energy is stored in the material and two components can be distinguished : an intrinsic component is due to the residual stresses in the glass

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[4] A decrease of the applied static load on a structural element can follow from its deformation, as transferred loads are redistributed to the surrounding structure (extern redistribution of efforts), whereas the intern efforts in the element generally become more severe in some location as a consequence of the damaging of its constitutive components (intern redistribution of efforts).

[5] This critical damage state is due to microscopic defects, in the form of surface cracks or flaws, with typical depth values between a few and a few hundreds micrometres. The sub-critical progression rate is very slow (except in case of accidental or volunteer scratching of the surface…) and the surface damage is most of the time not visible with the naked eye.

[6] See also Chapter IV, paragraph IV.3.2.1 about crack propagation patterns in beam elements.

[7] The energy necessary to create the cracked surfaces uses a fraction of the released strain energy during the fragmentation process (Bos 2010a; c; Gross and Seelig 2011).

sheet resulting from the production process[8], and the extrinsic component corresponds to intern efforts caused by extern actions. Thanks to the perfect linear elastic behaviour of glass, the superposition principle can be applied to stress components. Consequently, advantages of thermally toughened glass in terms of strength and shattering behaviour are balanced by the larger quantity of elastic strain energy released at breakage. There is thus a clear drawback to the use of stronger glass components in laminated glass units, firstly during the impact event (more energy release during critical crack growth, which is probably not fully consumed by the creation of more cracks) and secondly after the impact event (due to the large density of fragmentation pattern). Therefore, a safe calculation of the effect of the energy release rate should consider an *upper characteristic value of the glass strength* instead of a lower one[9].

Above considerations illustrate that a same initial external cause of damage can involve a succession of different consequences, according to the response of a specific laminated glass product. Its response depends thus not only on the properties of its constitutive components, but also on its configuration. This matter of fact constitutes a challenge for developing experimental assessment approaches which are not punctual validation tests of particular project configurations.

This leads to define *quasi-static design situations* not only in absence of an external dynamic action, but also of any fragmentation process of the glass components possibly caused by non-dynamic loading cases. Figure II.1 summarizes the scenarios of damage progression in a laminated glass element in service conditions, and distinguishes accordingly two possible categories of failure modes. The first regroups the failure modes caused by a dynamic event (FM-D), and the second the ones corresponding to a quasi-static design situation

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[8] Residual stresses appear in the form of compressive stresses along the outer surfaces and tensile stresses in the bulk of the glass sheet. These residual stresses, minimized in annealed float glass products (slow cooling rate) and maximised in thermally toughened glass products (fast cooling rate), largely determine the characteristic differences between the two types of products (mechanical and thermal strength, damage sensitivity, shattering patterns).

[9] However, standardized assessment procedures for glass products only characterize a lower characteristic value in static conditions. Considering the large scattering in values of glass strength, this is certainly an important difference to make. For instance, for annealed float glass products, individual test results from double-ring tests according to test standard EN 1288-2 vary between about 30 MPa and 120 MPa (for one reference thickness); this corresponds to the test configuration used to determine current design values (of the surface strength) in calculation codes. The scattering of the "random" part of the glass strength for tempered glass products is about the same order of magnitude (in absolute value). Besides, the validity domain of proposed functions for expressing dependence of glass strength on duration of applied load in standards (for instance coefficient k_{mod} in prEN 13474) is limited to a quasi-static loading range (load duration typically larger than 5 sec.). Such function is thus not appropriate for dealing with that problem of upper glass strength in the dynamic range, even if it could be assumed that the same time-dependent function is applicable to lower and upper design values of glass strength.

(FM-QS)[10]. According to the assumption made above, this second type of failure mode cannot be caused by a breakage of a glass component or fragment, and thus necessarily involves a failure at the level of interlayer components[11].

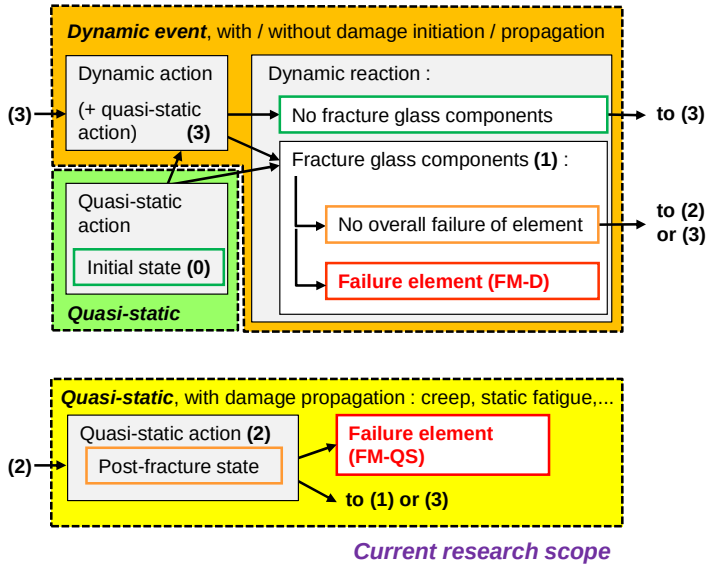

Figure II.1 – Schematic representation of failure scenarios for a structural element in laminated glass (FM-D: dynamic failure mode; FM-QS: quasi-static failure mode)

In summary, two behaviour ranges of fractured laminated glass elements are distinguished, and it seems accordingly possible to dissociate investigation of quasi-static post-fracture performances from questions related to glass strength and fragmentation processes. Conversely, the contribution of the interlayer is obviously not limited to the quasi-static performances in a post-fracture state : it is thus necessary to verify whether the separation into successive dynamic and quasi-static design situations can be justified from the perspective of the prediction of the behaviour of the interlayer.

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[10] The flowchart can be used for different types of failure scenarios, to move through successive accidental design situations (according to the terminology of Eurocodes). The possible number of steps (or 'loops' through the flowchart) depends on the succession of accidents in the failure scenario on the one hand and on the evolution in time of damage on the other. For instance, a successful pendulum test on a laminated glass unit according to EN 12600 can be associated to a scenario (0)-(3)-(1)-(2) : an extern impact (3) causes a breakage of one or more glass components (1) with no direct failure as consequence, and no further failure shortly after the impact when a static load is applied (2); the evaluation consists at assessing that the element did not fail *during* the impact (no FM-D failure after step (3) or (1)) nor *shortly after* the impact (no FM-QS failure after a step (2) with a specified duration). A successful drop height test according to EN 356 corresponds to an implicit scenario (0)-(3)-(1)-(3)-(1)-(3)-(1)-(2), namely each impact is supposed to increase the level of damage in one or more glass components.

[11] Provided that failure at the level of connections with the surrounding structure is excluded.

II.3. Fractured stages, damage and residual resistance

In previous section quasi-static design situations for fractured states of laminated glass elements have been defined in relation to a failure scenario and have been dissociated of glass fragmentation issues. In this section description of the corresponding fractured states and quantification of the damage level are considered.

Structural engineers defined different post-fracture states, generally identifying three distinguished fractured states. The first stage (I) is the *initial non-fractured configuration*, in which the possible role of interlayers is limited to a shear transfer between the different sheets. The definition of two next fractured stages is different according to authors and considered configurations, but can be associated to a general scheme, namely a *partially fractured stage* (II) and a totally or *ultimate fractured stage* (III). Partially and ultimate fractured stages appear to have to be understood in this context with regard to the capacity (stage II) or incapacity (stage III) of constitutive glass sheets to participate to significant further decrease of overall residual resistance by further cracking. In other words, when considering more specifically a dynamic loading range, the latter refers to the capacity of the laminated glass element to dissipate further energy by creating new cracks in its constitutive glass sheets.

Kott (Kott and Vogel 2003, 2004a; Kott 2006) introduced three *fractured stages** by considering mainly applications made of 'simple' laminated glass units[12] and loaded transversally, namely plate or slab configurations with bending efforts across the thickness as principal deformation mode. Stage I corresponds to an undamaged element, stage II to an element of which one of the two glass sheets is fragmented, while in the stage III all glass sheets are fragmented. The structural response involves rather different loading paths in the different stages, and accordingly a different mode and level of contribution of the interlayer to the overall load-bearing capacity of the element. In stages I and II, the efforts in the interlayers are essentially longitudinal in-plane shear stresses, while in stage III the interlayer is submitted to a dominant tensile effort in some localized places, namely where the interlayer is making a ligament that bridges the glass fragments across cracks in a same transversal section.

The initial definition of fractured stages at the element scale (Kott and Vogel 2003) neglected the possible existence of shear transfer in stage II between the non-fractured glass sheet and the glass fragments resulting from the breakage of the second sheet. In a second step (Kott and Vogel 2004a; Kott 2006), the concept of fractured stages has been refined by considering that a fractured laminated glass element (stage III) can be divided into different segments corresponding to

[12] Simple laminated glass units = 2-glass-ply laminates made of two glass sheets of the same type and with the same nominal thickness, and one interlayer.

one of the three defined fractured stages. The concept of fractured states is not applied at the level of the whole element anymore, but on different zones of smaller dimensions delimited between transversal sections. This division is thus depending on the density and the distribution of cracks in the different glass layers, or fragmentation patterns, and the consecutive stress (re)distributions in different cross-sections (Figure II.2).

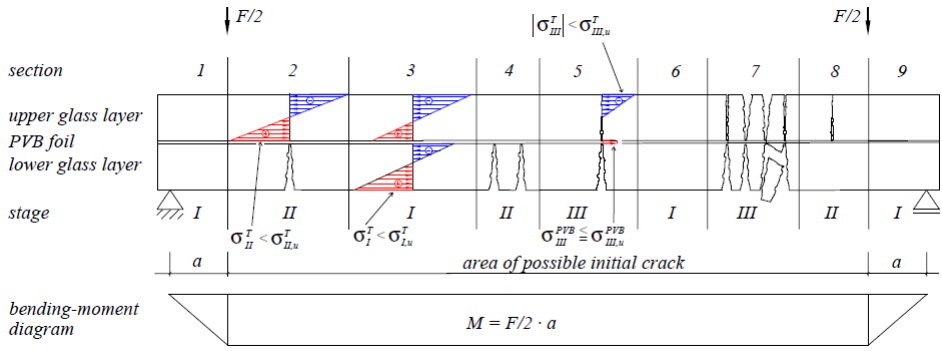

Figure II.2 – Fractured stages of a laminated glass plate under bending efforts (Kott and Vogel 2004a)

*** Note about used terminology :**
Kott used the term “broken stage”; another term used in other contributions is “post-breakage stage”. In fact, individual glass components are generally broken into different pieces or fragments, but still part of the element as they are hold together by keeping their adhesion with the interlayer : it leads to a fractured stage of the laminated glass element. In this context, a fracture is considered as a closed crack (distance between the two faces of the crack is considered negligible), and accordingly a “fractured state” is different from a “cracked state” (term used among others for reinforced concrete). A laminated glass element is considered as broken when at least one crack surface goes through the interlayers and the thickness of the whole element accordingly, what corresponds to a complete failure, and most of the time a collapse : it is a state beyond a ‘ultimate’ or ‘total’ fractured state (concept refined below). This explains why the terms “fractured stage” and “post-fracture behaviour” are preferred here (by lack of univocal reference terminology). Also, the terms “stages” and “states” are used, “fracture stage” inducing the idea of progression or succession between different states as a consequence of damage progression. This can be important to distinguish as some designs of products or elements have an initial configuration which can be assimilated to an initial fractured state, but not an initial fractured stage, as it has not been obtained by fragmenting the glass components after the lamination : it will appear in this chapter why it can be a relevant distinction to make.

This allows Kott to account in model development for possible stiffening effects of the bonded glass fragments of the fractured sheet when situated at the tensile side (illustrated as zone 3 type in Figure II.2), provided that their planar dimensions are large enough for the development of such stiffening effects (Kott and Vogel 2004a; Kott 2006). The achieved grade of stiffening is also dependent on the shear transfer capacity of the interlayer, and accordingly the contributing level along the different segments is supposed to vary with the (relative) stiffness of the interlayer. Expressed in another way, the effective length of the segments of the different types is varying with the stiffness of the interlayer; this reduces the practical usefulness of the concept.

The concept of fractured stages can be generalized for similar 'simple' laminated glass elements in other loading configurations. However, similar fragmentation patterns in different loading configurations have different consequences on the diminution of the load-bearing capacity. In particular, the structural response and the residual resistance between a plate and a beam configuration can be of different orders of magnitude between corresponding similar fractured stages. Such differences were observed experimentally among others by performing four-point bending tests on laminated glass units of similar dimensions, loaded about their weak and their strong axis respectively (Belis et al. 2009)[13].

Bos (Bos 2009, 2010b) adapted the definitions of fractured stages for beam applications made of non-'simple' laminated glass products, more particularly 3-glass-ply products (Figure II.4). The considered product configurations were essentially of two types, on the one hand 'standard' 3-glass-ply products[14], namely with the three glass sheets and the two interlayers respectively of the same type and with the same thickness[15], and on the other hand an innovative concept of hybrid reinforced glass beams integrating a steel rod at the lower side of the beam, developed and experimentally investigated by Louter (Louter 2011; Louter et al. 2012a; b)).

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[13] See also Chapter IV paragraph IV.3.2.1.

[14] 3-glass-ply laminated glass products composition is in practice commonly considered as a 'minimum' requirement for structural elements in construction projects. It is tricky to explain that such a compromise *de facto* is arising in formulation of technical prescriptions on 'structural' elements in laminated glass, but that simultaneously univocal criteria for distinguishing structural from non-structural applications do not exist. It is however also a typical example of technical specifications expressed by means of a prescriptive requirement (namely by imposing constraints on product configurations, what restricts the field of design solutions) in place of a performance requirement (which should express the benefits expected from the prescriptive rule, or the criteria used to establish it, allowing possible alternatives solutions complying to the same performance requirements). This comment has to be considered in parallel of the analysis developed in Chapter I.

[15] Three types of glass sheets (annealed, heat-strengthened or thermally toughened float glass products) and two types of interlayers (PVB and SG – see Chapter III paragraph III.3.1) were considered.

Table II.1 – Comparison of definitions of fractured stages for laminated glass element

	Element scale : # fragmented glass sheets		Segment / section scale
Author :	Kott	Bos	(Kott)[a]
Scope :	'simple' 2 ply lam. glass; out-of-plane bending	2 and 3 ply lam. glass; in-plane bending (beam)	'simple' 2 ply lam. glass; out-of-plane bending
Stage 0	n.a.	none (intact element)	n.a.
Stage I	none (intact element)	1 outer glass sheet	longitudinal shear
Stage II	1 of 2 glass sheets	(n-1) glass sheets	none
Stage III	2 of 2 glass sheets	all glass sheets	longitudinal tension
	amount of fragmented glass sheets		load-transfer mechanism in interlayer

[a] The definitions of fractured stages at segment scale are rather an interpretation based on the figures and models developments of Kott. Load-transfer mechanisms are illustrated in Figure II.2 and further developed in section II.4 below.

Bos introduced a stage 0 to refer to the non-fractured stage (equivalent thus to the stage I of Kott), and uses it as reference initial configuration to express the residual resistance of the element in the subsequent fracture stages. Stages I and II are defined as elements with one or both external sheets fractured respectively, and stage III indicates an element with all its constitutive glass sheets fractured[16]. Consequently, for 'simple' laminated glass units, stages II and III are equivalent concepts, while for non-'simple' laminated glass elements with three glazing layers or more, at least one glazing component is still the main load-bearing element : this definition seems in fact to better correspond to the more general concept of stage II as retained by Bos. The definitions of fractured stages according to respective authors are summarized in Table II.1.

The logic behind the definition of fractured stages by Bos has to be understood within the conceptual framework he developed within his research, with purpose

[16] In fact, the definitions of fractured stages given by Bos in (Bos 2009 chap. 6) are still more subtle, referring to an "extent of damage" : "*[I / II / III] : physical damage to the extent that [one glass layer / all outer glass layers / all glass layers] do(es) not transfer principal tensile stresses related to the governing load case anymore in at least one section of the element*". This indicates that the definition of damage level and fractured stages have been formulated mainly with regard to their consequences for specific configurations and loading cases.

of developing and applying concepts of robustness[17] to structural glass elements and more generally to glass constructions. It resulted in a proposition of graphical representation of safety properties of a laminated glass element. This representation tool, called **"Element Safety Diagram" (ESD)**, relates with each other and in a summarized form different parameters used to quantify the structural robustness of the element, namely : the damage sensitivity[18], the relative resistance, the redundancy and the fracture mode. An example of such a safety diagram with a short description of the different components is reproduced in Figure II.3.

In this context, Bos introduced two different damage parameters (Bos 2009) :

1) a *structural damage parameter* D_s, which is in fact an expression of residual load-carrying capacity, defined as the complementary part to the ratio between a residual resistance of a laminated glass element in a given fractured state R_n on its initial value in the undamaged state, R_0 :

$$D_s = 1 - \frac{R_n}{R_0} = 1 - r_n \qquad \text{with } n \text{ : damage stage (I, II, III)} \tag{II.1}$$

(Note: the second term r_n in above equation corresponds to the remaining load-carrying capacity for each fractured stage, as defined by Kott, or relative residual resistance[19])

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[17] Among the numerous references addressing the topic of "Robustness of structures", let us mention the general guideline "Design for robustness" published by IABSE (Knoll and Vogel 2009), the more recent outcomes of COST-Action TU0601 "Robustness of structures" (2007-2011, http://www.cost-tu0601.ethz.ch), and for the particular case of glass constructions the doctoral thesis of Freek Bos (Bos 2009). It is acknowledged by the different authors that concepts of robustness are not much developed in current design codes (Eurocodes,…). There is also no complete consistency (yet) between defined concepts and used terminology between these different references. Furthermore, it seems that the conceptual framework (Canisius 2011; Knoll and Vogel 2009; Sørensen 2011) does not account for time-dependent, delayed response of a structure nor for materials with time-dependent properties.

[18] This concept is equivalent to the definition of vulnerability (to damage) in (Sørensen 2011), relative to the resistance to failure initiation; a complementary concept is the damage tolerance, relative to the capacity of the system to survive the damage initiation (Sørensen 2011).

[19] The concepts used by Kott and Bos must not be confused with each other. Kott expresses the relative residual resistance r_n with regard to the initial (measured) resistance of the non-damaged element, whereas the relative resistance in the definition of Bos relates the residual resistance R_n in a fracture stage n to a design value corresponding to the applied effort for a loading case representative of a ultimate limit state, $S_{d,uls}$, considered for dimensioning the element in the non-fractured stage. This concept of relative resistance rather corresponds to a safety margin formulation, in comparison to the initial (calculated) safety margin in the non-fractured stage. The value of the relative resistance according to the concept of Bos is therefore depending on formulation of design states and on related used design values and resistance models, where the actions are determined on the basis of codified actions in design codes (Eurocodes,…).

and

2) a *physical damage parameter* D_φ, associated to physically measurable damage levels; in the analysis of performances of glass beams, this parameter has essentially been associated to the amount of broken glass sheets, and accordingly reduced to a few damage categories, corresponding to the fractured stages (0, I, II, III) introduced above.

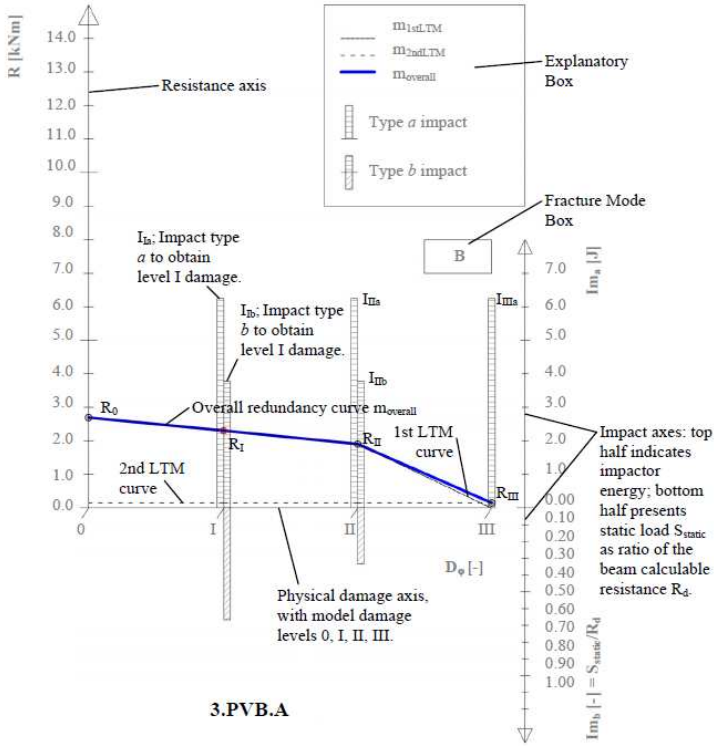


Figure II.3 – Example of "Element Safety Diagram" (ESD) for laminated glass beam element developed by Bos (Bos 2010b)

The parameter D_φ better complies with a concept of 'damage level' of the laminated glass element, as a state variable supposed to depend only on the element configuration, and not on the loading configuration considered for determining the post-fracture performances[20]. However, the damage level D_φ is

[20] In terms of the application fields introduced in Chapter I, the physical damage parameter, as a state variable, would depend on parameters of the category 'AF-Application: Design: Geometry and Configuration', and would not (directly) involve parameter values of the category 'AF-Application: Design: Performance requirements'.

also a *consequence* of a dynamic event (impact,…), and is identified as a key parameter *conditioning* the residual load-bearing capacity, represented by D_s. The structural damage parameter D_s as defined by Bos is not an 'intrinsic' property of the fractured laminated glass element, but describes a property of the element for a specific loading configuration. Besides, the family of built configurations[21] the ESD is supposed to be representative of[22] is not identified.

The ESD (Figure II.3) represents on the one hand the damage sensitivity of an element configuration to attain each fractured stage (associated to qualitative damage levels I, II, III, represented on the horizontal axis) under the effect of successive impacts (represented by vertical bars and quantified by a parameter Im_a on the right vertical axis), and on the other the consequence of each damage level on the residual load-bearing capacity (represented on the left vertical axis).

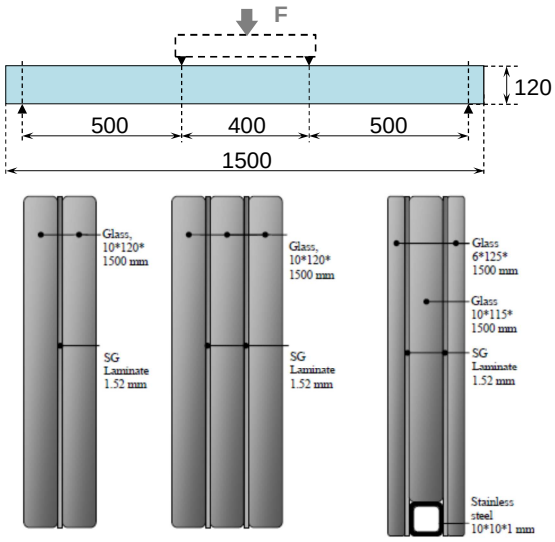
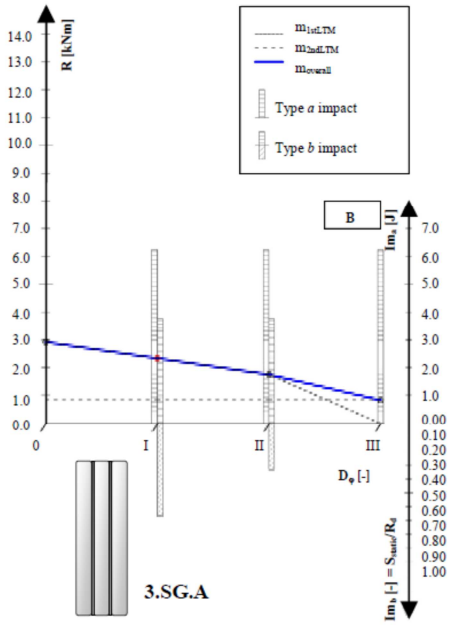
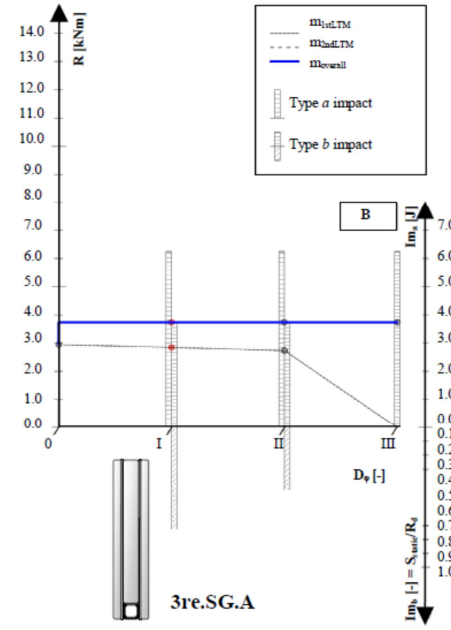


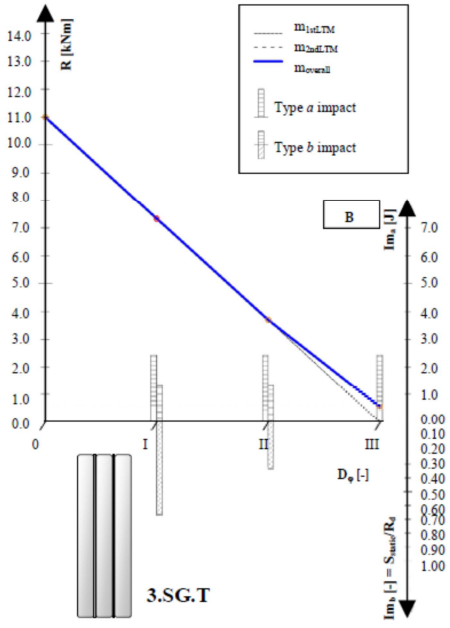
Figure II.4 – Test configurations of beam elements (dimensions in mm) investigated experimentally by Bos. From left to right: 'simple' 2-ply laminated glass beam; 3-ply laminated glass beam; hybrid laminated glass beam concept by Louter

[21] The family of built configurations in this context is equivalent to the concept of 'intended field of use' and related Application fields introduced in Chapter I.

[22] The question about the "representativeness" of the reference test configuration is further considered in Chapter IV section IV.2. In this regard, it must be highlighted that test configurations considered by Bos (and Louter) are four-point bending configurations with in-plane loading, and with lateral supports preventing possible failure mode by lateral buckling.







Legend of beam compositions :

3.SG.A :
- glazing layers :
  annealed float glass 10 mm
- interlayers : SentryGlas 1.52 mm

3.SG.T :
- glazing layers :
  thermally toughened float glass 10 mm
- interlayers : SentryGlas 1.52 mm

3re.SG.A :
- glazing layers :
  annealed float glass 6 / 10 / 6 mm
- interlayers : SentryGlas 1.52 mm
- reinforcement bar :
  steel hollow section 10x10x1 mm

Figure II.5 – Application of the Element Safety Diagram concept to different configurations of beam element with SG-interlayer - selection from (Bos 2010b)

For constructing the ESD for a series of configurations of laminated glass beams, Bos conducted series of 4-point bending tests (Figure II.4) for each configuration. The applied test protocols correspond to two types of damage scenarios, essentially distinguishing elements carrying only their own-weight and elements on which an extra static load is applied. The impact type *a* and the impact type *b* in fact consider the same hard impact body[23], but different loading conditions of the element, whether a complementary permanent static load is applied on the element during the impact (type *b*) or not (type *a*). The value of the applied static load considered by Bos (load *F* in Figure II.4) at each fractured stage is determined on the basis of the expected residual load-bearing capacity of the element at the next damage level, and calculated as a fraction of the initial resistance measured by means of four-point bending tests performed on a series of intact elements. For a sound understanding of the test results, it is necessary to mention that in both cases the impact was applied along the lower flange, in the middle of the beam, in a zone where the tensile effort caused by the permanent loads is the largest in magnitude in the impacted glass component.

Figure II.5 reproduces the developed ESD's for a few configurations of SG-laminates[24]. These show that the required level of impact for breaking the glass is lower when a complementary static load is applied (impact type *b*), but that the complementary applied static load during the impact has no consequence on the residual resistance. In other words, a larger value of applied static load would increase the damage sensitivity but not the damage tolerance. However, the test protocol used by Bos may be misleading in that regard, because it did not consider experimental situations for which the ultimate fractured stage (stage III) is reached under a combination of static load and impact, and because the considered loading configuration is relatively insensitive to effects of time and temperature as long as at least one glass component is remaining intact[25].

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[23] The impact body is a steel punch with a sharp head, thus with a very small contact area at impact, mounted on a spring-loaded device aimed at controlling the energy of impact, expressed by Im_a. This type of impact rather corresponds with an attack action or a perforating impact body. Note that the impact level necessary to cause the glass breakage *in this configuration* and with this impactor, expressed in terms of impact energy (impact energy ~2.5 .. 6 J), is one to two orders of magnitude smaller than with standardized impact bodies (for instance, respectively ~60 .. 360 J for classes of EN 356 and ~100 .. 600 J for classes of EN 12600 – see Chapter I table I.6). This shows that 1) impact energy is not giving a comprehensive basis for comparing different impact problems, and 2) is not representative for expressing the damage sensitivity and damage tolerance of an element; among others, the presence of the static load modifies significantly the damage sensitivity.

[24] SG-laminate refers to any type of laminated glass product made with SentryGlas® (SG) as interlayer; see also in Chapter III, paragraph III.3.1 about the particularities of this product.

[25] Besides, as the complementary static load is applied by means of a hydraulic loading device, it is possible that the deformation consecutive to the damage caused by the impact leads to a drop of the applied load. Also, the presentation form of the ESD does not give a comprehensive order of magnitude of the ratio between the two components of permanent load, namely between values of the own-weight of the beam and of the complementary applied static load.

Conversely, Bos and Kott both observe that the measured residual resistance of a pre-damaged element[26] is in some cases larger than the one observed when an element in the same configuration is loaded statically from an undamaged state to a final failure or collapse[27].

The comparison of the ESD's for the same element configurations with annealed (3.SG.A) and toughened glass sheets (3.SG.T) reveals that the second configuration increases the initial resistance but simultaneously the damage sensitivity in almost equal proportions (about a factor 3), with an ultimate load-bearing resistance (in stage III) slightly smaller. However, this comparison does not account for probable much larger effect on the corresponding values of deflection : this could also explain the difference of values of ultimate resistance between the two configurations, as the corresponding results correspond to tests with an application of the static load at a constant displacement rate[28] !

Along the same lines, Kott observed that a different fragmentation pattern is obtained for each method used for cracking the (first) glass component (for laminates made with annealed float glass sheets) and resulted in a different measured value of residual resistance; it seems also that the effect of the fragmentation pattern on the residual resistance is larger for plates simply supported along their four edges than for plates supported only on two sides (Kott and Vogel 2004a; Kott 2006). However, this remains quite a qualitative statement based on a limited set of test configurations; any extrapolation to other configurations without further experimental investigations is probably abusive.

The reported interactions between effects of an impact and of a pre-existing static load in a laminated glass element are thus possibly of different nature. Before glass breakage, the impact force can accelerate the sub-critical crack growth in glass components due to the permanent static load. This increases also the amount of elastic strain energy stored in the element and released at glass breakage, and is thus a potential source of supplementary damage brought to the fractured element. In summary, the *combination* of a static permanent load and of an impact action leading to glass breakage is likely to reduce the post-fracture resistance of an element in comparison to the one corresponding to each effect assessed individually.

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[26] This notion of pre-damage encompasses in Kott and Bos their view damage in the form of fractured components caused by any kind of impact previous to the application of the static load (in test) or loading case (in calculations).

[27] It must be noticed that in the test configurations considered by Bos and Kott, the impact and the static load are applied in the same direction, namely their effects in critical tensile zones are likely to combine with each other.

[28] In the experimental works reported by Kott and Bos, the static load is applied at a fixed displacement rate : v=0,2 mm/min (Kott and Vogel 2004a) and v=2 to 5 mm/min (Bos 2009). The same loading configuration with an effective constant value of applied force would produce less favourable outcomes.

Bos considers further a monotonic decrease of D_s in function of D_φ, namely that damaging of the element (increase of D_φ value with damage evolution *n*) can only lead to a diminution of the residual resistance (increase of D_s value from 0 to 1). This assumption appeared to be not verified for the tests performed on the hybrid beam concept, where the load bearing capacity of the fractured beam is larger than the initial one, leading consequently to a negative value of D_s and a relative residual load-bearing capacity larger than one (Figure II.5, case '3re.SG.A'). However, this does not account for the relative large level of deformation necessary to attain the mentioned value of resistance in the ultimate fractured stage[29]. This highlights the difficulty of the conceptual level of application of the ESD in its current format.

The major shortcoming in the analysis of Bos is not that the influence of time-temperature effects on post-fracture performances has not been investigated experimentally, but that related questions finally *disappeared from its scope of concerns*, and this mainly because of the experimental issues for investigating these. Bos writes here about (Bos 2009 chap. 5) : "*Additional temperature requirements deviating from normal room temperature, e.g. demanding that a certain degree of residual strength has to be proven over a temperature range of, say, –20 to +60°C, is felt to make experimental proof testing unnecessarily complicated*". This analysis however only reflects a still widespread opinion among practising design engineers and in the manufacturing industry. However, is seems necessary, with regard to safety issues, to express this on a slightly different way : the expected costs of experimental investigation of temperature dependence of the post-fracture performances by means of tests on specimens at element scale[30] is judged economically non affordable[31], and thus related issues should better be assessed by means of alternative (experimental) methods and assessment approaches… still to be developed.

In fact, experimental programs reported in literature developed for assessing design of innovative structural applications (project-oriented assessment) hardly ever include tests at different temperatures, but also this aspect of the problem is not or not much discussed (Beer 2005; Smith and Dodd 2003). In literature, the test protocols are often dissociated of the failure scenarios established beforehand

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[29] For the hybrid configuration represented in the ESD of Figure II.5, the average value of the vertical displacement measured experimentally in stage III is 2.6 cm, corresponding with a deflection of $2.6/140 \approx 1/56$.

[30] The concept of 'element scale' is refined in Chapter IV section IV.2. It is sufficient at this step to consider that it corresponds to tests on specimens of large dimensions, in experimental configurations which are 'sufficiently representative' of the built configuration.

[31] This statement must probably be nuanced : it is rather the ratio between the allocation of means with regard to expected practical benefits at an instant x which is problematic… It seems thus rather addressing the lack of available appropriate assessment strategies and the corresponding repartition of costs among involved parties than excessive costs in absolute terms.

or of the observed failure patterns during the tests : this makes it difficult to rebuild the reasoning behind the development of this type of experimental campaigns[32]. A few cases of assessment tests performed at different test temperatures on final configurations are reported, as for instance tests performed on point-fixing systems (Nugue, Fouillen, et al. 2003).

In general, experimental approaches are relatively close to the ones reported above, and based on failure scenarios considering the breakage of one up to $(n-1)$ of the n constitutive glass plies (corresponding to fractures states I or II), with requirement on loading case(s) to consider for assessing the residual load-bearing capacity in the consecutive fractured stages. The loading case(s) are basically accounting for the own-weight of the element possibly with a complementary static load expressed as a fraction of the design variable load (or combination of variable loads) or an absolute value (fixed arbitrarily); or similarly with other types of loading.

Shortcomings in literature about experimental investigation of temperature dependent performances at application scale have different reasons. The first is the relatively small number of this type of tests, which require specific (and expensive) test installations (this aspect is further discussed in Chapter IV). The second is the problem of re-interpreting project specific results for different configurations, with possible abusive utilization of selected results out of their context. This makes involved parties reluctant to publish detailed test reports; also some may consider that such publications are potentially more harmful than beneficial for their own business, but also for others because of possible abusive utilization of selected results out of their context. Finally, little detailed critical analyses are available on the relevancy of the specific selected experimental scale or protocol in the one and the other cases.

In fact, the questions to address are : which test configurations at element scale are necessary to validate a specific design configuration (project-oriented assessment), and which approach could be suitable when the assessment rather addresses a wider “intended field of use” ? To address these issues, a new paradigm shift (Davies and Bennison 2003) is probably necessary. To this point, this need will be summarized as a need to pay more attention to design of experimental approaches and experimental investigation programs, and to structuration of test reporting in general.

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[32] In fact, performing such a review work according to some systematic method is estimated to be a much time-demanding task, in regard to effective benefits to be expected. Most of the time, the grade of detailing of test protocols and test results in publications is too low for being reused in quantitatively relevant comparisons; and it is often not straightforward to have a clear idea about the effective measured data and the derived or calculated ones (see also Chapter IV about unidentified border effects).

In summary, the proposed concept of Element Safety Diagram mainly expresses a well-identified need for representation tools providing a more comprehensive overview of concepts and properties related to the description of post-fracture performances in general, to facilitate comparisons of safety performances of laminated glass products and applications, in particular for non-conventional ones. The identified shortcomings in the ESD can be related to problems of two natures : the lack of a robust conceptual framework, and issues for performing extensive experimental programs with regard to the specificities of the investigated performances on the one hand, and of the application context on the other.

Essential reasons behind some shortcomings are thus identified, and some suggestions can be summarized to address them :

1) The fragmentation response and post-fracture behaviour of laminated glass are essentially application or *element performances* not easily summarized in terms of product or material properties. It is therefore proposed to abandon the term and concept of structural damage as introduced by Bos, and to shift the expression of any 'residual load-bearing capacity' firstly at the level of an element performance rather than of a cross-section property[33]. In fact, impact resistance is not a performance that can be defined at the level of a cross-section of an element. Any structural post-fracture performance is necessarily associated to clearly identified *element* and *loading* configurations. The definition and representation of post-fracture performances must also account for a possible *change of the critical failure mode* caused by the damage progression, towards a failure caused by excessive deformation, possibly in combination with a buckling phenomenon; or in function of performance requirements peculiar to the design configuration[34].

2) The physical damage parameter D_{φ} is renamed as an *element damage parameter*, with the purpose that it accounts for any type of physical change of intrinsic properties of the laminated glass element affecting its response in service conditions. Therefore, the constitutive parameters of D_{φ} must all be state variables. Three generic components can be further identified, the damage to glass sheets ($D_{\varphi,gl,i}$, with i = 1 .. n), the damage to interlayer(s) ($D_{\varphi,int,j} = 1..(n-1)$), and the damage to interface(s) ($D_{\varphi,ij}$), with n the

[33] In other words, it comes down to express the element resistance as a primary experimental parameter (here the applied force at the level of the testing device), rather than a derived one (an intern effort) : on this way, the "cursor" between results of the experimental assessment and model development is set at the "best place" to avoid polluting experimental results with modelling uncertainties. This is one of the aspects aimed to be covered by the concept of border effects introduced and further discussed in Chapter IV.

[34] A criterion of excessive deformation is generally imposed by the function of the element or in relation to the risk of damage to the surrounding elements of the structure caused by the deformation.

number of constitutive glass (or glazing) sheets. In a general way, a possible variation of the value of D_{φ} must be assumed also in quasi-static design situations of post-fracture stages (corresponding to a step of the type (2) in Figure II.1). It will be shown in Chapter IV and V that this kind of uncertainty on the time-dependency of a property can involve important constraints for designing experimental investigation programs.

3) The *combination of a permanent static load and a dynamic action* on a laminated glass element is generally producing less favourable effects than each considered individually, and such combinations are thus necessary to account for in assessment, from a conceptual but also from an experimental point of view. Combination of static load and dynamic action affects both the damage sensitivity and damage tolerance[35]. The evaluation of the damage tolerance addresses mainly the risk of progressive collapse[36] in function of the level of applied static load present at the moment of initiation of critical crack growth, and whether or not this load is modified by the deformation of the element consecutive to damaging[37]. However, the ESD in Figure II.5 mainly reflects this combination of effects on the damage sensitivity (reduction of impact energy necessary to initiate glass breakage) and not on the damage tolerance, in particular between fractured stages of type II and III[38]. In other words, it does not seem relevant to express risk of accidental breakage and corresponding values of residual resistance, whether with regard to a failure mode of the type FM-D or FM-QS, in isolation of simultaneous and consecutive loading case(s)[39].

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[35] Note that for the damage sensitivity, the static load includes also residual stresses in the glass sheet.

[36] Progressive collapse refers to a sequence of accidental and consecutive damaging events leading to collapse (Canisius 2011), and is generally rather conceived at the level of a hyperstatic structure; progressive collapse is generally used to refer to an insufficient damage tolerance. The concept however does not seem to integrate a time component for assessing the risk of this failure mode.

[37] It is also necessary to consider the possible variation in experimental conditions of the intended applied value of the complementary permanent load, when this one is applied by means of an hydraulic jack steered with an actuator, or more generally by means of a machine with active load control. The readability of the ESD could be improved accordingly by representing these two components at a same scale, namely by showing the absolute value of the parameter 'S_{static}' in regard to the one of '$R_{d,\varphi}$', corresponding respectively to the value of applied permanent static load *during* the glass breakage (crack initiation), and *shortly after* (namely at a time distinguishing the dynamic event from the consecutive quasi-static design situation – in relation with failure steps in Figure II.1).

[38] The ultimate residual loading capacity (in stage III) is expected to vary significantly because of the sensitiveness to creep of interlayer materials (see Chapter III for generalities and Chapter V for SG-laminates in particular).

[39] The two corresponding loading cases could be defined by means of accidental loading combinations according to Eurocodes (EN 1990 / EN 1991-1-7), possibly with a necessary reinterpretation of the combination rules for different configurations (application).

It is further assumed that it is possible to determine univocal parameters and criteria for distinguishing 'dynamic event' and 'quasi-static post-fracture design situation' for accidental scenarios and associated design situations. Some of these parameters are further identified in next sections.

II.4. Fragmentation patterns and load-transfer mechanisms

Different issues have been identified for characterizing properties of laminated glass products related to safety performances on the basis of experimental investigations at an 'element' scale, and more particularly with regard to the time-temperature dependence of the post-fracture load-bearing performances. The question is thus which alternative, 'intermediate' experimental configurations could be used for this purpose[40].

Bos expressed his awareness that the definition of the damage parameter D_{φ} (as introduced in previous paragraph) and the corresponding practical way of quantifying it should be completed and refined (Bos 2009), and this mainly with regard to two complementary questions : how to measure it during tests, and how to evaluate the probability of occurrence in design conditions ? Indeed, both questions highlight the need for a refined description of fractured states, which also accounts for the influence of the fragmentation process history and consecutive possible locally different loading paths.

The most noticeable damage which can be measured experimentally at element scale is the amount of created cracks between two subsequent fracture stages. It is however not a measure of the total dissipated energy during the fragmentation process, and not a measure of the post-fracture residual load-bearing capacity; let us consider these two aspects successively.

Predicting results of fragmentation process in the glass components consecutive to a dynamic event, whether its 'damaging capacity' is dominated by the impact energy or by the elastic strain energy release consecutive to initiation of critical crack growth is a complex issue, especially because the ruling parameters or properties vary in importance according to the element and loading configurations. It is not easy to propose a *general* expression or intuitive representation form for catching the relative contributions and mechanisms of energy transfer and energy dissipation, nevertheless it is possible to make a few qualitative observations.

Initiation of critical crack growth in glass (elastic brittle material) is expressed by a fracture criterion resulting from an energy balance known as the Griffith energy balance, applicable to a quasi-static, stable crack growth. It expresses that the

[40] 'Element' and 'intermediate' experimental scales are defined more accurately in Chapter IV section IV.2.

crack propagation results from a local energy transfer, where the energy necessary to create a crack surface comes from a release of elastic strain energy in the vicinity of the crack tip of an initial flaw. Therefore, the resistance to crack propagation in a material for a given crack opening mode[41] is expressed by means of the *fracture toughness* (generally expressed in [MPa.m$^{1/2}$]), for instance K_{Ic} for glass where crack propagation is dominated by mode I. This property expresses the resistance to crack growth for a given geometry and a determined "far field" loading state (or stress field calculated by neglecting the presence of surface defects), as the geometry of the initial defect modifies the stress distribution around the crack tips.

The stress intensity or concentration around the crack tip is expressed accordingly by means of K-factors, one for each of the three pure crack opening modes. These are expressing the relation between the local acting force applied on a small *process zone* of size r around the crack tip (on which assumptions of continuum mechanics are not fulfilled anymore) in function of the stress distribution on a zone of larger dimensions R in the vicinity of the crack tip ($R >> r$), called the *crack-tip field* and which controls the crack propagation process. The crack-tip field also corresponds to a zone on which the stress distribution pattern is significantly modified by the presence of the crack, in comparison with an identical geometric and loading configurations but without the presence of crack (Gross and Seelig 2011). Accordingly, the stress field generated by the loading state outside the crack-tip field can also be referred to as the *far stress field*. It is further assumed that outside the crack-tip field only elastic deformations occurs in surrounding materials; in particular, the dimensions of the material volumes on which energy is dissipated by plastic deformations (or another irreversible dissipation mechanism) is smaller than the size of the crack-tip field, what is formalized by a generic equation $r_p \leq R$. The orders of magnitude of the size of both zones are shortly discussed below.

Linear elastic fracture mechanics theory allows to derive the stress field around the crack tip (Gross and Seelig 2011) from the far stress field in case that the dimension of the crack-tip field is small in comparison to the other dimensions of the problem. A crack propagation problem can be considered as independent of the geometry when all dimension parameters, including the dimension of the initial crack, are larger than the size of the ruling crack-tip field. In such case, the crack propagation problem corresponds to a displacement of the crack-tip field

[41] The crack opening mode depends on the relative displacement of the cracked faces compared to the position of the crack tip or front, and distinguishes thus the different components of the displacement vector *a* describing the movement of the crack front. Corresponding K-factors relate components of stress tensor near the crack tip or front with each component of the crack displacement (I for normal crack opening, II for in-plane shear and III for anti-plane shear). 'Pure' modes represent thus idealized situations where one crack propagation direction interacts only with one component of the nominal (continuous) stress field.

without modification of its shape, namely without modification of the stress distribution pattern in a close vicinity of the crack tip, when considered from a point belonging to the (advancing) crack tip.

For more complex loading configurations, and for interfacial crack propagation problems in particular, the resistance of a material or a structure to crack propagation is rather expressed in regard to a more general formulation of the acting force, in the form of a *strain energy release rate* Γ (also named *crack extension force*) which expresses the amount of strain energy consumed for extending the crack surface; the corresponding resistance property is named *crack resistance* Γ_c (also referred to as fracture toughness because of the equivalency of concepts in case of pure crack opening mode[42]), typically expressed in [J/m²] and corresponding to the amount of energy required to create a unit of crack surface[43]. This quantity expresses also the amount of energy dissipated in case of stable crack growth.

As indicated in section II.2, critical crack growth in glass involves high crack propagation velocity, and the energy balance requires the addition of a term of kinetic energy (equation of Mott) (Haldimann et al. 2008; Overend et al. 2007). This is the gateway into dynamic fracture theories, in which the kinetic energy appears as a complementary source of potential damage besides the release of elastic strain energy (at least in elastic brittle materials). The critical crack growth is unstable[44] as it propagates without increase of the extern applied force (Gross and Seelig 2011) and the crack propagation direction becomes unstable as well, with branching as possible outcome (Haldimann et al. 2008; Overend et al. 2007).

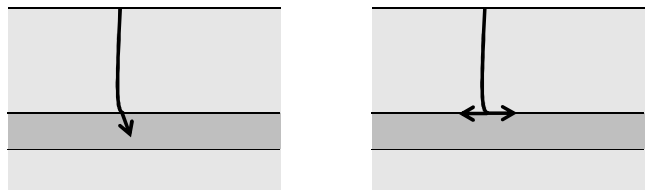

Figure II.6 – Crack propagation across glass-interlayer interface

[42] For pure mode I in perfect homogeneous elastic material, there is a direct relationship between the two concepts and the elastic properties of the material (Gross and Seelig 2011).

[43] There is thus not a direct idea of time-dependence in the definition of strain energy release rate.

[44] This distinction between a stable and an unstable crack growth is based on the behaviour in perfectly elastic brittle materials.

In case of dynamic crack propagation, the resistance is rather expressed as a *dynamic fracture toughness* $K_{Id}(\dot{a}) \leq K_{Ic}$, or $\Gamma_d(\dot{a})$, expressing that the resistance to crack growth is a function of the crack-tip speed, or advancing velocity of the crack front. However, simultaneously, in dynamic crack growth conditions, the acting force also decreases with the crack speed; whereas, conversely, for a stationary crack (no crack propagation, or $\dot{a} = 0$) loaded dynamically, the stress intensity factor and the energy release rate are larger than in the equivalent static case (Gross and Seelig 2011).

In summary, stable or unstable crack growth is governed not solely by material properties, but also significantly by geometry (distribution and configuration of flaws and of loading) and type of loading (time-dependence of the applied load : static or dynamic,…). It seems also that the *formulation of the fracture problem changes according to the design problem*, namely whether the crack initiation or propagation is seen as a problem or as solution, namely a phenomenon to avoid or to promote respectively, and the level of expression varies accordingly.

The propagation of a crack in the glass sheet perpendicular to the glass-interlayer interface can principally follow two different paths (Figure II.6) : either it propagates through the interface and the crack extends inside the interlayer (crack penetration), either it is deviated perpendicularly and extends further along the interfacial plan (crack deflection). The latter crack propagation mode is obviously preferred for a safer behaviour, and can lead to the formulation of a crack penetration-deflection criterion (He and Hutchinson 1989; Parmigiani and Thouless 2006). The propagation path followed by the crack when reaching the interface between two elastic bodies is ruled by the geometry (mainly the thickness of the surrounding layers), by the elastic mismatch (difference in elastic properties of the two materials expressed by means of the two Dundurs coefficients (Muralidhar et al. 2000) or by means of the bimaterial constant (Gross and Seelig 2011)), and by the strength or toughness ratios (Parmigiani and Thouless 2006). However, this analysis of crack propagation at an interface remains in the field of the linear elastic fracture mechanic (LEFM), namely assuming deformations limited to small strain ranges (infinitesimal strains) and no energy dissipation by the materials, thus no (macroscopic) large strain deformations, no plastic deformation and no time-dependent properties.

With polymer materials typically used as interlayers (see Chapter III), the deviations from the assumptions of linear fracture mechanics are large; and the modelling of such a problem on a relatively large application scope in terms of temperature, loading rate (dynamic) or loading level (static) seems thus very complex. Treatment of crack propagation problems in elasto-plastic and visco-plastic materials in fracture mechanics theories can still be treated by means of LEFM when assumptions of small-scale yielding are fulfilled, roughly speaking when $r_p << R$. Otherwise, the crack propagation process is governed by *large-scale yielding* processes, which require the use of numerical models for

accounting for non-negligible dissipation mechanisms around the crack tip (Gross and Seelig 2011). As a trend, the size (radius) of the stress field involved in the local energy balance increases with its intensity. It seems thus clear that identifying the application scope on which crack propagation at interfaces of laminated glass products follows a safe pattern (a crack deflection) is not obvious : it is likely to involve different criteria.

However, principally, it can be expressed by means of a relative simple idea of a *weak interface* (Muralidhar et al. 2000)[45], which can be seen as a first principal requirement for a laminated safety glass to comply to[46].

In order to simplify the problem, it will temporarily be *assumed* that the dynamic event and associated fragmentation pattern complies with this principle, and further results in no significant damage in the bulk of the interlayer, whether in the form of crack penetration or plastification. Consequently, a crack in a glass component resulting from a fragmentation process necessarily involves small delamination surfaces along any crack tip in contact with a glass-interlayer interface. The description of the damage level at the beginning of the following quasi-static design situation requires thus to be completed by an *initial delamination length* a_0.

This assumption is comforted by some experimental observations realized during impact tests with advanced metrology system (Nourry and Nugue 2005), based on controlled impact tests on PVB-laminates in a configuration *close* to the EN 356 drop height test. In these conditions, the authors estimate that the impact energy dissipated by glass fragmentation and ejection of glass fragments accounts for a minor, negligible component in the balance of energy dissipation mechanisms. The impact energy is in first instance mainly dissipated by delamination and only later on by tearing of the interlayer (for larger penetration displacement of the impactor from the moment of impact). An order of magnitude of dissipated energy by damage in the glass and along the interface respectively can be gained by comparing typical values of fracture toughness of glass (2 .. 8 J/m²) and of interface of PVB-laminates (100 .. 900 J/m²)[47]. It is further assumed that these

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[45] The author's first name and name, Muralidhar Seshadri, seems to have been inverted in this publication; therefore, it is solely referred to the model of "Seshadri" further in this chapter.

[46] Seshadri formulated a criterion of weak interface for the stable crack growth on a quasi-static domain; see also section II.6 below.

[47] However, these values are not directly comparable with each other, as it corresponds to a fracture mode I for glass and to mixed fracture modes for interfacial toughness. The reported values of toughness of interface of PVB-laminates were derived from different experimental configurations and loading conditions, involving different fracture modes with interlayer modelled as linear elastic or non-linear elastic (hyperelastic) material (Muralidhar et al. 2000), thus the obtained value of crack resistance also accounts for energy dissipated by the bulk deformation of the interlayer. Values of fracture toughness derived by this type of method are affected by the three types of experimental border effects identified in Chapter IV.

initial delamination lengths remain relatively small compared to the thickness of the interlayer, thus $a_0 << t$. The validity domain of this assumption should however be further determined, with regard to the history leading to the considered fractured stage. However, the importance of estimating this parameter is also possibly different with regard to the modelling of the dynamic behaviour (failure mode FM-D), and the consecutive post-fracture performances (failure mode FM-QS).

The necessity of assuming that each crack in a glass fragment is causing initial interfacial delamination near the crack tips with the interlayer can also be argued another way around. In absence of any delamination of the interlayer from the crack tips, even an infinitesimal opening of the glass crack would lead to an infinite value of the axial strain in the central section of the ligament, and thus to initiation of crack penetration (Figure II.6). The evidence comes by considering the simple analytical expression of the axial strain in the ligament (Muralidhar et al. 2000) :

$$\varepsilon = \frac{\delta}{a} \tag{II.2}$$

with $\delta = d/2$ the half axial opening of the glass crack (mode I), and a the delamination length from the crack tips along the interface(s) with the interlayer.

All these considerations bring us back to the description of fractured stages, which has thus been enriched with a new parameter. However, attempt at correlating the post-fracture resistance of a given fractured configuration to the level of damage in relation to the extent of created crack surfaces remains not relevant, because of possible localization effects. Whereas the contribution of the interlayer to resistances and stiffness in stages 0 to II is essentially due to longitudinal shear-transfer, the residual resistance in an ultimate fracture stage (stage III) is associated by Bos and Kott to the development of another type of load transfer mechanism.

The tensile strength of the interlayer is playing a determinant role in taking over the internal flexural effort developed along 'stage III' fractured cross-sections (illustrated as zone 5 type in Figure II.2). For fractured laminated glass plates supported along their four edges, Kott identifies the development of similar yield line mechanisms[48]. The presence of a type III fractured section, with all glass layers cracked more or less in the same transversal plane, is critical for the residual load-bearing capacity and leads thus to a refined concept of a critical

[48] 'Yield line mechanism' in planar element (plate, slab) submitted to bi-axial bending deformations is a concept similar to plastic hinge in '1D' truss and beam element. It is used to describe localization of deformations in the behaviour of cracked reinforced concrete floor slabs.

ultimate fractured stage. This allows to simplify the risk analysis about glass fragmentation issues by considering the most unfavourable fractured configuration(s) for assessing the residual load-bearing capacity.

However, if the final failure is not ruled by a strength criterion but by excessive deformations, the identification of one critical fractured section is not sufficient. Indeed, the number of 'stage III' fractured sections affect the overall stiffness of the fractured element, yet to a different extent according to the specific configuration, in terms of load distribution and support conditions (Kott and Vogel 2004b). In comparison, the effect of different support conditions on the initial load-bearing resistance was noticeably related to the development of shear-transfer mechanism in the interlayer in each configuration (Gräf et al. 2003). For laminated glass beams with in-plane bending efforts, the contribution of the interlayer to the overall load-bearing performances in fractured stages are related to the same two main load-transfer mechanisms (Bos 2009), illustrated in Figure II.7 :

- a **ligament** bridging behaviour between glass fragments situated on respective sides of a cracked transversal section, or **through crack tensile load transfer mechanism** (**TCT-LTM**, called secondary Load Transfer Mechanism[49] by Bos). This configuration can in practice develop only if the glass pieces remained bonded to the interlayer.
- a **longitudinal shear transfer mechanism** (**LS-LTM**, called primary Load Transfer Mechanism[50] by Bos) between glass fragments. There are two different configurations of this mechanism, according to the fragmentation patterns : the first is referred to as an **offset crack tensile mechanism** (Nhamoinesu and Overend 2010) or **OCT-LTM** and occurs between fragments bonded to the opposite sides of the interlayer along a zone situated between cracks in the two related glass components in different transversal sections. Accordingly, the OCT-LTM fulfils a bridging function. The second configuration is closer to the shear-transfer mechanism in non-fractured configuration, and is activated only in case of transversal bending efforts in elements with relatively large fragments (as in zone 3 in Figure II.2), namely

[49] It is important to notice that this mechanism is not activated in non-fractured configurations, with regard to the specific time-temperature and time-stress dependence of the mechanical properties of polymer materials in the large strain range (see Chapter III). Is is also not activated in fractured zones with compression efforts, in non 'simple' laminated glass configurations. It is an important difference with other composite structure used in construction, as in reinforced concrete : the reinforcement (steel bars) is already contributing significantly to the transfer of tensile efforts in initial, non-fractured state, thanks to the higher stiffness of steel (about 200 - 210 GPa) compared to concrete (20 - 50 GPa), namely a stiffness ratio of 4 to 10.5. In comparison, the stiffness ratio between glass and interlayer is inversed, and in particular variable due to the time-temperature dependent properties of the interlayer material (see Chapter III); it is smaller than 1/10 or 0.1.

[50] The LS-LTM is activated in non-fractured laminated glass element once transversal bending efforts are applied, and it explains why Bos names it as the first LTM.

with a longitudinal dimension sufficiently larger than the thickness of the glass layer. This typically develops between a fractured and a non-fractured glass sheets (element stage II). Therefore, LS-LTM will be used for designing "non OCT" configurations.

These two mechanisms can be observed in any loading configuration of fractured laminated glass elements, whether the loading has a dominant component parallel or perpendicular to the plane of the element. As they are defined according to local fragmentation patterns, they are local configurations independent of boundary and support conditions of the element.

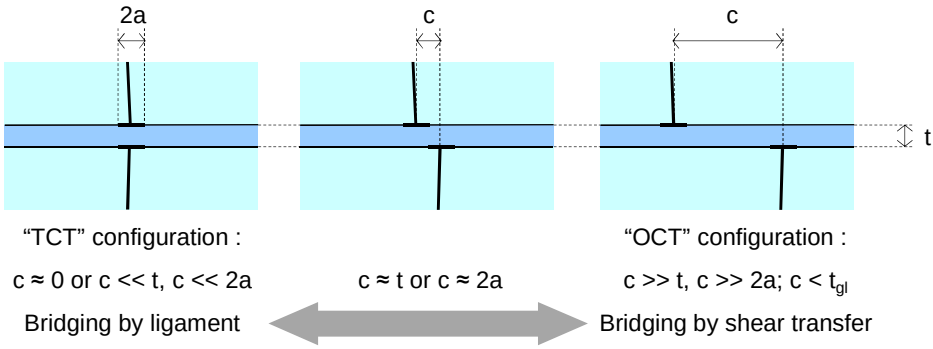
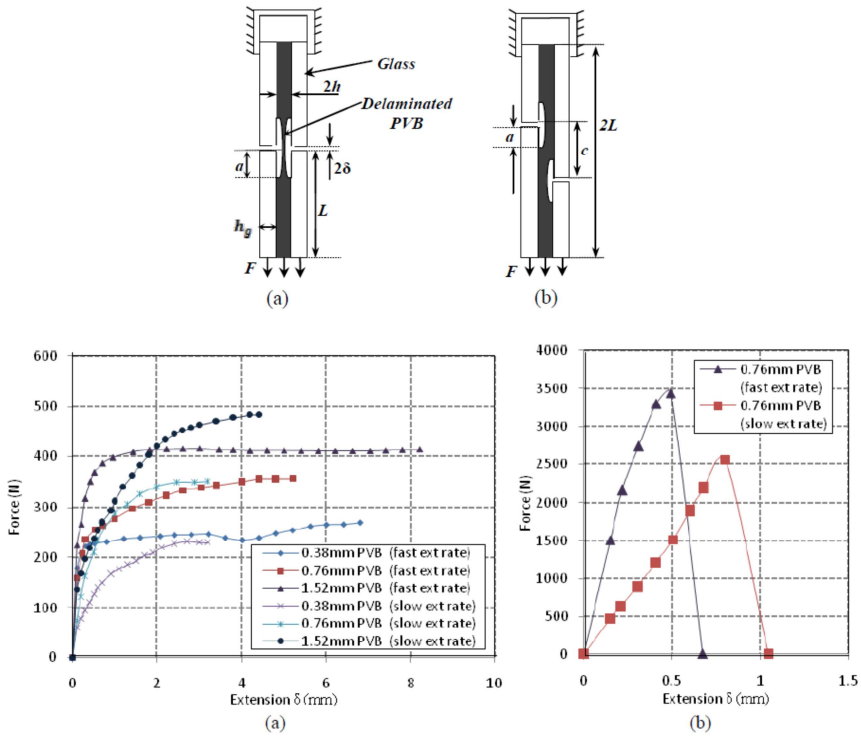


Figure II.7 – Transition between configurations corresponding to the same damage level but to different load-transfer mechanisms

A third mechanism could be identified in some loading configurations with significant in-plane efforts, namely a dominant transversal shear effort parallel to the crack plane. This one could be referred to as a *TCS-LTM, Through Crack Shear Load Transfer Mechanism*, and is also associated to a bridging function. However, this third mechanism is judged less likely to occur in practice due to the relatively large slenderness of laminated glass elements in building applications in general, what generally involves larger, more critical stress level due to bending efforts than to shear efforts, and is consequently not further considered.

If the two identified main load-transfer mechanisms are considered in isolation of the rest of the element (Figure II.7) and compared with each other, they correspond to an equal level of physical damage, they involve the same geometric parameters and to a large extent the same material properties. In order to summarize their contribution to the overall behaviour of a fractured laminated glass element, each one basically defines a relation between a tensile effort applied in the interlayer ligament (F) with an opening between the crack faces in the glass components (d). It has been shown in the previous section that the performance to determine for each LTM is not limited to its resistance (maximal force), but must also describe its deformation capacity, or ductility.

An engineering reasoning allows to assume that the in-plane axial deformation is larger across cracks (TCT-zones) than in between (OCT-zones), and that the risk of breakage by tearing of the ligament only concerns TCT-zones. The validity of the latter assumption is enforced by the various results of experimental tests on laminated glass elements reported in literature. Comparison of tensile tests on smaller specimens PVB-laminates, pre-cracked in TCT- and OCT-configurations (Figure II.8), gives an idea about the influence of cracks distribution in glass components on the axial tensile stiffness of a fractured element (Nhamoinesu and Overend 2010). For the geometries and test conditions considered in Figure II.8, the OCT-LTM appears as about 10 times stiffer than the TCT-LTM.



Dimensions specimens : thickness 6-(2h)-6 mm with 2h = 0.38 / 0.76 / 1.52 mm, width 50 mm, c = 20 mm (OCT)
Loading rates : 0,264 mm/s (fast ext rate), 0,0264 mm/s (slow ext rate)
Test conditions : room temperature (~20°C)

Note : in OCT-test (b), the maximum load is attained by further cracking of the second glass ply in one of the half-precracked transversal section

Figure II.8 – Comparison of axial stiffness of TCT (a) and OCT (b) load transfer mechanisms in PVB-laminates (Nhamoinesu and Overend 2010)

The TCT-LTM is thus clearly identified as a critical load-transfer mechanism with regard to safety properties and structural performances. Considered closer, the load transfer through the ligament is ensured at two levels, and accordingly the deformation capacity of the ligament, namely the macroscopic ductility of the load-transfer mechanism, appears to depend on a combination of two complementary mechanisms :

1) the *delamination* (debonding) between the interlayer and the glass pieces under shear (and eventually normal) stresses; and

2) the *stretching* of the interlayer under tensile (and/or shear) forces, eventually up to rupture by tearing.

In summary, the residual load-bearing performances in 'ultimate' fractured stage (element stage III) can be associated to two load-transfer mechanisms, identified as TCT-LTM and OCT-LTM, among which the former is considered as the dominating or critical load-transfer mechanism. Both are expected to show time-temperature dependent response, however the question is which experimental configurations are the most appropriate to characterize the involved 'product' properties.

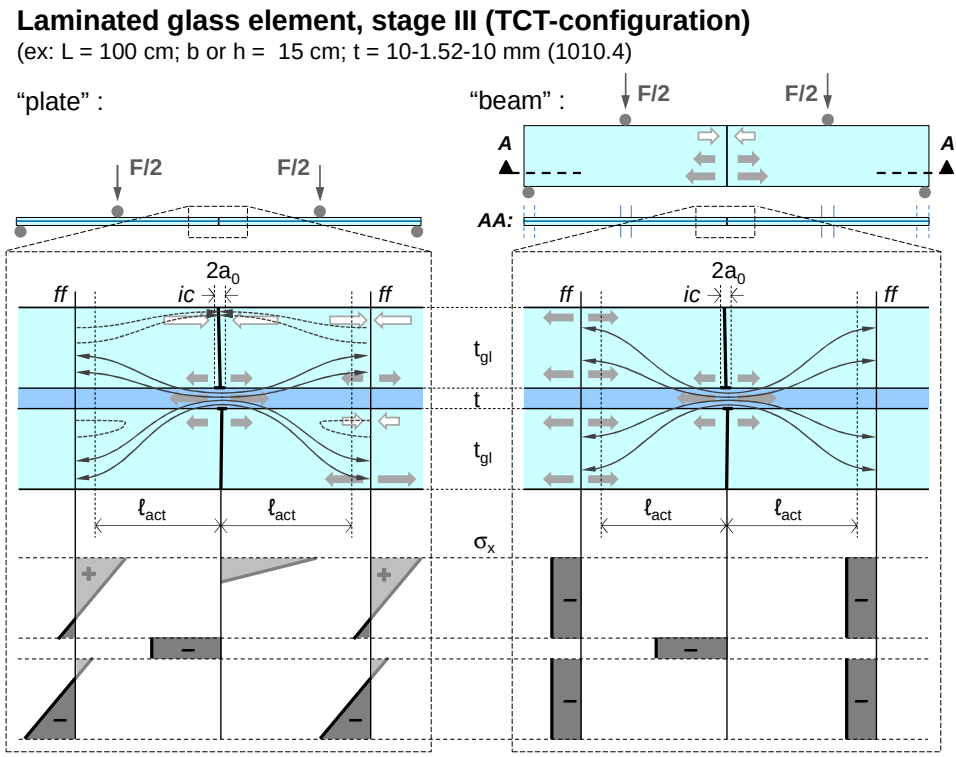


Figure II.9 – TCT-Load Transfer Mechanism in different loading configurations (example with two typical 4-point bending test configurations)

The selection of the most appropriate experimental investigation methods for *characterization* purposes is in fact not straightforward, for different inter-related reasons. As explained here above, a major one results from the fact that the initial state of a fractured configuration associated to a quasi-static design situation is influenced by the preceding glass fragmentation processes and the corresponding loading situations, during and after the initiation of cracks. Other aspects are related to interactions and overlaps between conceptual and modelling questions on the one hand and practical experimental aspects on the other with respect to specificities of mechanical behaviour of polymer interlayers; these will be considered more in details in next chapters. Last but not least, a third reason is related to an important approximation that the load-transfer mechanism in a TCT-configuration is similar for a variety of loading configurations.

Let us consider this last aspect closer on the basis of a relative simple example. A laminated glass element with a simple fragmentation pattern, namely with its two glass sheets fractured on such a way that it generates a TCT-configuration, is loaded in two similar 4-point bending configurations (Figure II.9) : in the first case, the load is applied perpendicular, and in the second case parallel to its main plane. The crack extension forces generated locally along the four interfacial delamination fronts in presence, in the vicinity of the initial cracked section (ic), can be related to the far stress field (ff) by means of an equation of energy balance typically used in fracture mechanics. It appears that the far stress fields obtained in both loading situations are different (if simplified to a two-dimensional, plane strain problem). Nevertheless, the assumption is made that the developed load transfer mechanism in these different loading situations can be reasonably approximated by the TCT-test configuration[51]. There is thus a zone of influence to consider for the TCT-LTM to be able to develop, with unknown dimensions. To acknowledge for this, a parameter is added to the description of the ligament function : ℓ_{act} is the necessary *activation length* of the load transfer mechanism, measured from the ridge of the TCT-section (ic). The use of this parameter in combination with the other ones involved in the crack propagation problem allow to formulate a relatively simple condition for the concept of TCT-configuration being usable at a structural level :

$$\ell_{act} = a + R \leq \ell_{ff-ic} \leq \ell_{gl,min} / 2 \qquad \text{(II.3)}$$

with ℓ_{ff-ic} the distance between the TCT-section and the "regular" far stress field, and $\ell_{gl,min}$ the minimum dimension of the glass fragments on each side of the TCT-section. The concept of activation length can then probably be extended to OCT-configuration as well. It needs then to be refined with regard to the various

[51] The identification of the conditions for which this approximation is valid is a question left open and submitted to developers of numerical models.

fracture mechanics models in order to be quantified; it seems however clear that related questions cannot be considered separately of the modelling of the "intrinsic behaviour" of the ligament made of "non-linear materials" (Gross and Seelig 2011). This last question appears to be a complex one for interlayer materials with regard to identified application scopes, as their mechanical behaviour can involve visco-elastic and visco-plastic effects (see Chapter III). The crack propagation problem in a TCT-configuration is therefore further considered here only qualitatively.

On the basis of the above reasoning and in the perspective of designing test configurations for investigating load-transfer mechanisms, it appears that the grade of dependency of a fracture problem on the geometry is mainly depending on the size of the crack-tip field and of the process zone, with characteristic values depending mainly on the characteristic size of the microstructure of the material and increasing with the material ductility. In materials showing a time-dependent behaviour, parameters used to describe the acting forces applied on the crack tips (K-factor, strain energy release rate,…) are time-dependent as well (Gross and Seelig 2011). The characteristic sizes of the parameters ruling the local energy balance ($R, r_p, ...$) can thus be expected to be at least one order of magnitude larger in the polymer interlayers compared to glass or metal components. Characteristic size of the cohesive zone in glassy polymers can have a length of several millimetres (Gross and Seelig 2011) – for the purpose of the current discussion, the length of the cohesive zone can be assimilated to the parameter r_p, namely the size of the zone along which inelastic (non-linear) deformations occur.

An original experimental method is reported by Siebert (Siebert 1999) to measure the activation length in PVB-laminates experimentally. The test set-up consists in a four point-bending test on a 'simple' laminated glass element in a plate configuration, with similar dimensions as the one sketched in Figure II.9a), and with solely the lower glass sheet pre-cracked in the central section of the element. The test specimen is further equipped with two series of strain gauges glued on the upper and lower surfaces of the glass sheets, in the close vicinity of the central pre-cracked section, which measure the axial strains along the outer faces of the bended element. The loading rates correspond to quasi-static loading ranges, with successive steps at different loading levels. For the reported test configurations[52], the length $\ell_{\text{ff}-\text{ic}}$ is shown to roughly vary between 30 and 50 mm, to slightly

[52] All test configurations have the same planar dimensions, and consist in 3 series with respective composition 6-X-10, 6-X-6 and 10-X-6, giving the thickness of the constitutive layers (in mm) and their order in the test setup, with X = 0.76, 1.52 and 2.28 mm the thickness of the PVB-interlayer. No measure of the corresponding crack opening or deflection is reported, however the latter can be estimated as not superior to 1.5 the total thickness of the laminated glass element; nonetheless, such deflection level is probably already larger than typical value to be accepted as a design criterion.

increase with the interlayer thickness and to be relatively independent of the thickness of the glass sheets. It is probably abusive to draw general conclusions on this basis; nevertheless, the derived order of magnitude for the size of the activation length indicates that this can be an order of magnitude larger than the thickness of the constitutive components. With respect to the corresponding value of the delamination length a, conditions are not met for working with assumptions of small-scale yielding or small-scale creep (Gross and Seelig 2011), in particular the condition $R << a$ is practically almost never fulfilled[53]. Among practical consequences, the use of models based on LEFM assumptions is probably not acceptable for modelling the mechanical response of fractured laminated glass units, because different size effects of possible significant importance are likely to be neglected. In particular, the crack-tip fields of the four crack fronts present in a TCT-configuration are overlapping or interacting, and accordingly the individual crack propagation processes cannot be considered as isolated of each other in the range of interest. At least, it seems relevant to account for these aspects in designing experimental investigation campaigns and in analysis methods.

Above analysis suggests that the experimental characterization of product properties ruling the TCT-LTM and their validation for use in design practice is likely to require the execution of tests at different experimental scales. In following section, further attention is dedicated to the raising but still vague concept of 'intermediate' experimental scale, and will consider two interlaced aspects : tests on specimens laminated glass of small dimensions, and tests designed for investigating specific load-transfer mechanisms. Finally, a few more specific comments will be done on the test configuration specifically designed for investigating the ligament response, the TCT-test.

II.5. Experimental investigation of load-transfer mechanisms

The first idea coming to mind for investigating load-transfer mechanisms consists in designing specific test configurations allowing to isolate the mechanism of interest. For that purpose, the use of specimens of small dimensions is often considered, on the one hand in function of limits imposed by the considered testing device, on the other hand for practical and economic reasons.

In this section, a rapid overview is given of a variety of experimental configurations of tests performed on laminated glass specimens, conceived or used for that kind of purpose.

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[53] No value of delamination length is reported by Siebert, but on the basis of observations made during similar tests (see among others Chapter IV paragraph IV.3.2), this seems a fairly correct assumption. Another argument is given by the observed delamination and deformation patterns in TCT-tests reported in Chapter V paragraph V.3.1.

Pummel test and Compressive Shear Test methods[54] were respectively developed and used for measuring the adhesion level in laminated glass units, in order to correlate the adhesion level with the performances against impact of glazing units[55]. The Pummel test has been developed specifically for testing laminated glass units, and is firstly mentioned in a U.S. patent[56] about processes for controlling the adhesion level of PVB-laminates (Keller and Mortelmans 1999; Tupý et al. 2013). The CST-test configuration, developed for testing broad ranges of adhesive and composite structures (Schneider et al. 2001), also seems to have been firstly developed for testing PVB-laminates. It is performed on different types of test specimens : small squared-shaped specimens cut out of larger laminated glass units, with a side length of ~2.5 cm (Jagota et al. 2000; Keller and Mortelmans 1999) or 5 cm (Froli and Lani 2011), and on small cylindrical specimens drilled out of laminated plates with a diameter of about 3 cm (Delincé, Belis, et al. 2007).

The same small drilled cylindrical specimens laminated glass were used for developing other test configurations, with evolving purposes : the CST-test evolved towards a TST-test more or less simultaneously with a change of the test purpose, namely for measuring the time-temperature dependence of the shear-transfer stiffness (Sobek et al. 1999; Weller et al. 2005), and the effect of artificial ageing on it (Delincé, Belis, et al. 2007; Ensslen 2007). In parallel works, the 45° compressive shear load of the CST-test is replaced by an axial rotation perpendicular to the plane of the interlayer, for measuring interfacial shear strength (Nugue, Nourry, et al. 2003) and similarly by a tensile load applied perpendicular to the interlayer plane for measuring the interfacial normal strength (Bati et al. 2009a). The applied rotation effort evolved further from a quasi-static force to cyclic oscillations, altogether with the visco-elastic response as a new test purpose (Bati et al. 2013).

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[54] These two test methods are mentioned in an informative appendix (Appendix C) of the product standard EN 14449 for laminated glass products, see Chapter I. Nowadays, the associated test conditions are adapted by each manufacturer to the particularities of the interlayer products; there is thus not really one single Pummel test method and one single CST-test method, but each is used with a series of slight variants. The Pummel test evaluates the adhesion level according to a conventional scale from 0 (low adhesion) to 10 (high adhesion) (see next footnote). The adhesion level obtained by a CST-test is expressed as a shear stress (typically in MPa).

[55] See also Chapter IV section IV.2.

[56] US Patent nr. 4144376 (1979) entitled “Process for the production of modified, partially acetalized polyvinyl alcohol films”. The Pummel test may seem to outsiders as a relative ‘dumb’ test. In fact, it requires to be performed on a rather rigorous way; on this way, interlayer manufacturers manage to use it as a reliable quality control method. The test is performed on a laminated glass plate of limited dimensions (width about 15 to 30 cm) which undergoes repeated hammering in controlled conditions (Tupý et al. 2013), pulverizing the upper glass sheet. The result is a conventional value expressed on a (non-linear) scale from 0 (low adhesion) to 10 (high adhesion) by evaluating visually the ratio of visible surface of the film interlayer. The reproducibility of the test method is estimated around ±1 Pummel unit.

With the successive "experimental shifts", different types of border effects[57] are expected to vary in different proportions, but seem still difficult to objectivize quantitatively, and accordingly quantitative comparison of tests of different sources seems in most cases impossible. Nonetheless, the influence of the drilling process on the 'state' of the interlayer component represents an important, probably even critical border effect; it appears to be more sensitive for harder interlayer materials such as SG-laminates (Delincé, Belis, et al. 2007) in comparison with softer PVB- and EVA-laminates (Weller et al. 2005). Similarly, the dispersion of test results seems affected by the stiffness of the interlayer according to the test conditions.

A second category of specimens of small dimensions are rectangular pre-cracked laminated glass specimens. In the basic configuration, the initial cracks in the two glass sheets are brought in a same transversal section, and this one is therefore referred to as TCT-specimen. TCT-specimens were firstly tested in a three-point bending configuration about the weak axis, mentioned as a "flexure adhesion test" in (Sha et al. 1997); in a second step, they are loaded with an effective tensile force applied perpendicular to the pre-cracked section, leading to a test configuration named "tension adhesion test" (Sha et al. 1997) and finally TCT-test (Muralidhar et al. 2000). Similar TCT-tests on specimens PVB-laminates have meanwhile also been reported by other authors (Bati et al. 2009a; Butchart and Overend 2012; Ferreti et al. 2012; Nhamoinesu and Overend 2010). These tests were executed at moderate loading rates (values of applied displacement rate below 1 mm/s); an alternative test set-up applying an axial impulse load of about 2.9 m/s is reported in (Keller 2005). A variant to the TCT-specimen and the TCT-test configuration is the OCT-test, already introduced in the previous section and in Figure II.8 (Nhamoinesu and Overend 2010).

Noticeably, test configurations similar to the OCT-test have been used in parallel researches but for investigating the pre-fractured composite behaviour, namely the longitudinal shear-transfer mechanism (LS-LTM). The test result is expressed as a shear modulus G (Schuler 2003; Schuler et al. 2004), and is compared with outcomes of parallel tests performed at element scale (four-point bending tests,…). The motivations behind development of related experimental programs and discussions about the experimental aspects (reliability, representativeness and robustness of the different test configurations and experimental scales…) are however not (yet) explained or commented with much detail. Is the use of small test specimens rather motivated by scientific considerations, as for instance the investigation of a 'pure' stress state or load-transfer mechanism, or rather by pragmatic, economic aspects, as for instance the possibility of performing more numerous tests or because of development costs of experimental infrastructures ?

[57] The concept of border effect is refined in Chapter IV, and it will be shown that there are three different types of border effects.

Only a few of these test configurations have been used for experimental investigation of the time-temperature dependence of the properties, and only with regard to the response to shear-transfer of non-fractured configurations, under quasi-static and oscillating loading conditions. The time-temperature dependence of the TCT-LTM has firstly been mainly addressed by means of standard uniaxial tensile tests, or similar non-standard configurations on specimens of interlayer film[58]. Tests on similar test configurations with specimens PVB of different shapes and with different loading conditions are also reported (Bati et al. 2009b), using an adapted loading protocol with purpose of determining a lower limit of resistance to creep of the material.

Finally, another category of tests performed on specimens of small dimensions is worth mentioning : the peel tests. Peel tests in fact refer to a relatively broad family of test configurations designed for investigating the interfacial properties between an interlayer and a glass substrate, or more generally between a (thin) layer adhesive polymer and a stiffer (and thicker) substrate. Test configurations can look very similar in terms of geometry, but be quite different in terms of behaviour and analysis method in function of the relative stiffness and strength of the different components part of the test specimen. Peel tests developed with regard to investigation of interfacial properties in PVB-laminates generally involve "half-laminate" configuration, namely one of the two glass sheets is replaced by a thin metal layer (in aluminium); however, this type of test configuration is judged not satisfactory for deriving quantitative relevant design values by Sha (Sha et al. 1997), for different reasons.

A first category of reasons refer to generated fracture processes in a peel test and related modelling issues; among others, the derived value of adhesive strength according to standardized test methods[59] does not account for the energy dissipation by inelastic deformations in the peel arm. Modelling of the peel test by means of more advanced numerical techniques, among other by using non-linear time-dependent model for the interlayer material, confirmed this analysis on a more quantitative way (Pelfrene et al. 2014).

However, there is a second series of reasons for disregarding peel tests for characterization purposes of properties of end laminated glass products. There exist apparently no means to assess that the reached adhesion grade in half-laminate specimens is representative of the adhesion grade in regular laminated glass units. In other words, there are question about the representativeness of specimen configuration used for peel tests, besides the representativeness of the

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[58] Use of standard test methods for determining mechanical properties of interlayer materials is discussed in more details in Chapter III paragraph III.3.2.

[59] Standard peel tests should therefore preferably be considered at a first glance as conventional tests, similarly to standard uniaxial tensile test performed on dog-bone specimens of (adhesive) polymer material (see Chapter III).

peel test configuration(s) with regard to deformation patterns in fractured states[60]. Besides, ageing effects are likely to be of a different extent in a half-laminate configuration than in a regular laminate configuration; in that regard the author shares the analysis summarized in (Sha et al. 1997). These second series reasons are the prior motivation for not dedicating more attention to peel tests in the current work, but do not constitute a definitive judgement about the usefulness of peel tests for other test purposes[61]. It also does not mean that there are less questions about size effects in TCT-tests or other similar test configurations on fractured systems than with peel tests, with respect to application scopes in the construction sector; it is yet tempting to accept the idea that they are of a lesser extent in the first cases than in the second.

In summary and on a general way, the reliability of tests using specimens of small dimensions for characterization purposes remains a sensitive point. This is also generally acknowledged by experimentalists, which state however that "*precision and accuracy of data on interlayer stiffness are not crucial when dealing with ordinary structural problems at room temperature*" (Bati et al. 2013). This led however, in a first draft version of the JRC-report EUR 26439 EN aimed to serve as a basis for developing a European design code for "structural glass" applications[62], to purely and simply disqualify all tests on "specimens of small dimensions" with regard to characterization purposes of mechanical properties of interlayer components. The statement about outcomes from tests on "small-size" specimens has been toned down in the published version of the report, and limited to the field of the determination of the visco-elastic properties with regard to their contribution to shear-transfer (LS-LTM) : "*With these existing "small-size"-tests the time and temperature dependent stiffness behaviour of interlayers can be determined. However they show some shortcomings in view of the size-effect*". Nonetheless, it remains undoubtedly a highly relevant point of attention. Besides, it is far from obvious that tests performed on specimens of larger dimensions are exempted of all questions about representativeness of the test results : this aspect is further developed in Chapter IV.

A variety of tests on laminated glass elements of larger dimensions have also been reported, which can be roughly sorted into three categories in function of their main purpose, namely 1) for characterizing the contribution of the shear-transfer mechanism (LTM-LS), 2) investigating the response to impact, or 3) focussing on the post-fracture performances[63]. Some experimental campaigns however

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[60] A finer analysis grid for distinguishing the different aspects involved in the definition of representativeness of test methods is proposed in Chapter IV section IV.2.

[61] See also Chapter III paragraph III.3.4.

[62] 'SaT-report' released begin 2013 and mentioned in Chapter I section I.3.

[63] The experimental works of Bos, Kott and Siebert reported in sections II.3 and II.4 above are associated to this latter category.

addressed more than one purpose. In the first category of experimental works, time-temperature response has been investigated with a variety of test configurations and loading conditions, mainly on 'simple' PVB-laminates, and by several researchers. Tests of the second category (impact tests) are mainly related to standard tests, and other similar configurations, but generally limited to tests at ambient temperature. Because of the addressed purpose (safety in use related to among others explosion and manual attacks), details about test methods and results are generally kept confidential. Experimental campaigns addressing mainly or at least to some extent the third field of interest (post-fracture performances in quasi-static conditions) with generation of experimental situations with TCT-LTM activated are mentioned among others[64] in (Belis et al. 2009; Bennison et al. 1999; Biolzi et al. 2010; Delincé, Belis, et al. 2007; Delincé, Zarmati, et al. 2007; Louter et al. 2010, 2012a; b; Pankhardt and Balázs 2010)[65], most of them by means of bending test configurations, with tests at different temperatures and loading modes, with specimens artificially aged, etc.

Almost all the tests executed during these experimental campaigns (of the third category, and to a lesser extent of the first) were conducted by considering one single loading configuration, applying a progressively increasing load at a constant displacement rate when controlled by an electronically controlled actuator, generally matching assumptions of quasi-static loading conditions. Glass components of the test specimens were generally damaged by the application of the loading, or pre-damaged. The second loading mode in terms of amount of tests is a loading under constant force. When alternative test conditions were considered, they were generally considered in isolation of other effects. As for the tests on small specimens, a quantitative comparison of test results of different sources seems relatively difficult, for a variety of reasons. Some are of similar nature as the ones evocated in section II.3 above in regard to project-oriented test campaigns, other ones are related to different experimental issues.

The variety of test configurations and experimental approaches is accompanied by a similar variety of modelling approaches. Again, these could be regrouped in a

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[64] This list is not exhaustive and do not include all the most recent contributions (after 2012). A tentative of making a broader collection work has been initiated within the COST-Action TU0906 and should result in a more complete and structured database of references (Savineau et al. 2013). For more references meanwhile, a large amount of conference proceedings dealing with these topics is available on www.glassfiles.com. See also section II.3 about project-oriented experimental programs.

[65] The tested hybrid beam concept in (Louter et al. 2010, 2012a; b) is the same or similar to the one presented above in Figure II.4 and Figure II.5. This is one of the few developments of innovative concepts which have been supported by a relatively extensive experimental program with tests performed at different temperatures, therefore mentioned in this part. However, the generated post-fracture stages in this case do not involve TCT-LTM as the critical load-transfer mechanism for the ultimate residual resistance (with regard to a failure mode FM-QS).

few categories, according to the approach considered for determining the properties of the constitutive components of the tested elements. No detailed review is made here, however a trend can be noticed to attempt to model most of the test configurations mentioned above by means of advanced numerical models, detailing the contribution of all the components and requiring accordingly a large number of parameters for each. These approaches do not seem to always account on an appropriate or realistic way for the various uncertainties and consequent border effects in presence. Questions about the representativeness of the test results and the application scopes of the models are consequently generally not addressed on a *comprehensive quantitative way*, at least for non-experts. In fact, no reference framework or specific reference methodologies seem to exist to address this type of issues.

In conclusion, it is observed that they are a variety of issues to address simultaneously when performing mechanical tests on laminated glass specimens. The complexity and the closely inter-related aspects make it difficult to assess the reliability of tests at 'intermediate' experimental scale considered as candidate configurations for assessing design properties of laminated glass products and interlayer components. The various involved aspects are addressed in more details in the next chapters. The TCT-test configuration is considered closer in next section, as it has been considered as a reference test configuration in the experimental campaigns reported in Chapter IV and Chapter V.

II.6. Modelling approaches and analysis of TCT-tests

As mentioned above, the response of the TCT-LTM of PVB-laminates has been investigated by means of TCT-tests or similar configurations. The corresponding test results have been used to develop advanced numerical models, but also simplified analytical models have been proposed (Iwasaki and Sato 2006; Muralidhar et al. 2000) and taken over by followers (Bati et al. 2009a). Corresponding model developments belong to a series of similar research approaches developed also with other test configurations, among other for the CST-test (Rahulkumar et al. 1999; Rahul-Kumar et al. 2000) and other types of tests on small specimens such as peel tests (Rahulkumar et al. 2000), focussed on the modelling of PVB-laminates products.

The analysis method developed by Seshadri (Muralidhar et al. 2000; Seshadri 1999) is based on the observed behaviour of PVB-laminates in TCT-tests at ambient temperature and moderate displacement rate, characterized by relative regular delamination patterns up to large delamination lengths. Above a 'short crack limit' ($a \geq a_s$), a *steady-state* in the loading curve develops, characterized by a constant value of the reaction force. In this 'long crack' regime, the overall crack opening is only alimented by the delamination process : the axial strain in the central cross-section of the ligament remains constant. This response

complies with an assumption of weak interface, formulated as follows in its most simple form :

$$\frac{\Gamma_0}{E^*.h} < \frac{\varepsilon_s^2}{2} \qquad \text{(II.4)}$$

with Γ_0 the interfacial fracture toughness, $h = t/2$ the half-thickness of the interlayer, E^* the "effective" elastic modulus (of the interlayer) and ε_s a value of axial strain in the interlayer ligament corresponding to a 'short crack limit'. This equation results from an energy balance in the framework of LEFM and expressing the crack penetration-deflection problem based on assumptions of glass as a rigid material and the interlayer ligament as a perfect elastic material. By means of FEM modelling of the investigated TCT-configuration, a short-crack limit range is determined for $a < a_s = 0.1.h$, or for the considered experimental configuration with a 0.76 mm thick interlayer, $a < 0.038$ mm.

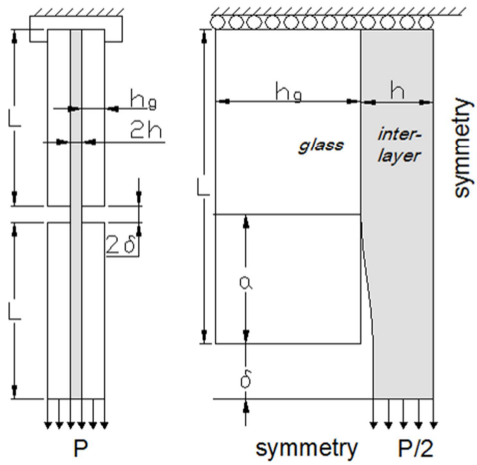

Figure II.10 – Schema of a TCT-test (lateral view), based on the representation by Seshadri (Muralidhar et al. 2000)

Previously, others (Sha et al. 1997) considered that in laminates with a relative thin interlayer ($t << t_{gl}$) and for relative small crack opening ($d < t$), the ligament transversal section is in (pure) tension, even in case of deviation from a pure TCT-configuration at a larger scale; however, they show also that the triaxiality of the stress is important near the crack tips (delamination fronts), and accordingly also in the central section of the ligament for small delamination lengths (compared to the thickness of the interlayer). Results of numerical modelling show that the assumption of uniaxial tensile stress in this central section is fairly well matched when the crack opening is larger than the interlayer thickness ($d \geq t$).

Disregarding further considerations about the theoretical developments and compliance of test results with the various underlying assumptions, the analysis method proposed by Seshadri imposes important constraints on the experimental response of a TCT-test for being processable. In fact, the method requires the measurement of the delamination lengths and the loading curve to reach a steady-state regime. Both conditions impose to reach a level of deformation in the response of the test specimen with an order of magnitude larger than the short-crack limit, imposed by the experimental scale for detecting the delamination fronts. This requires to reach larger values of crack opening than the effective range of interest in comparison with the corresponding application fields[66], and for usual thicknesses of interlayer also in a range for which $d > t$. Besides, the assumptions of time and temperature independent behaviour are really restrictive in terms of response range, and the calibration of an elastic or hyperelastic model on experimental data fits only in a limited range of behaviour. Aspects relative to assumptions about the mechanical response of the interlayer material is further developed in Chapter III.

A complementary experimental issue, resulting from the questions arising about appropriate mechanical models for the interlayer ligament, is related to the possibility of performing TCT-tests in other test conditions with regard to the identified application scope. Finally, regular delamination patterns are not always obtained on larger ranges of test conditions; however, it does not mean that the ligament has no resistance and no damage absorption capacity. These different aspects are illustrated in Chapter V by means of an experimental campaign performed on specimens SG-laminates. The various reasons announced in this paragraph led to disregard processing methods of test results proposed in literature, and to perform the analysis at a "lower level" in terms of experimental parameters.

As a consequence, the TCT-test configuration is further examined with regard to its potential to be used as an assessment method, by considering its potential to deliver quantitative relevant data in the context of structural use of laminated glass products in non-conventional configurations introduced in this chapter. The priority given to experimental issues led to push considerations about theoretical and model developments to a second order. Nevertheless, these are not totally forgotten, among others by acknowledging that experimental treatment of the test specimens can have an influence on the made assumptions for the initial fractured stage. This addresses in particular the made assumptions of a non-damaged ligament and of initial cracks in the glass sheets terminated by initial delamination lengths along the interfaces glass-interlayer.

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[66] This is however a rough general statement, with regard to possible failure modes at the element scale due to excessive deformations, which obviously largely depend on the amount of critical fractured sections (or TCT-sections).

II.7. Summary and outlooks

The problem of describing the post-fracture behaviour of laminated glass elements has been reviewed in the specific context of designing non-conventional load-bearing configurations in laminated glass in building applications. The constraints ruling the experimental assessment of products and applications has been analysed in parallel with the evolutions in the conceptual representation of the design problem by structural engineers, and the identification of the underlying mechanisms. It explains, without justifying, why investigation of time-temperature dependence of the post-fracture performances is in practice still generally left aside in current assessment methods, mainly in relation with practical difficulties to deal with it in experimental research. It is in fact not easy, from an experimental point of view, to disconnect the problem of the post-fracture performances from the questions about the damage sensitivity and fragmentation processes.

The concept of 'laminated safety glass' appears in fact to be mainly related to local crack penetration-deflection problems consecutive to breakage of glass components. A variety of situations and failure scenarios is identified which depend on the specific context of damage initiation, in relation with loading situations leading to a post-fracture stage. Failure scenarios tend to neglect the time-delayed response of a damaged structure after an accident. It is proposed accordingly *to complete failure scenarios with intermediate quasi-static design situations between dynamic events*, inducing or not further damage progression in the form of crack propagation in glass components. It provides a framework of very general applicability, but it requires some assumptions to dissociate the assessment of post-fracture performances of other phenomena occurring in dynamic ranges.

The damage sensitivity and the damage tolerance both appear depending on the presence of a permanent static load at the crack initiation and on the evolution of its value until the arrest of the crack propagation. The damage is clearly not limited to visible cracking of glass components, and this is acknowledged by completing the description of *physical damage* of fractured stages by means of *initial interfacial delamination lengths* near the crack tips of the cracks in the glass components. This is accompanied by an important assumption about the non-damaging of the interlayer in its bulk. The determination of the conditions, in terms of accidental situations and failure scenarios, for which these assumptions are valid is however left aside, and remains thus an open question for assessment processes.

This led to refine the description of failure scenarios and to distinguish two categories of failure modes (FM-D and FM-QS). In particular *intermediate post-fracture quasi-static design situations* have been defined :

1) by the absence of crack progression and fragmentation processes in the glass components; in contrast, stable interfacial delamination is not excluded;

2) by the absence of any other dynamic action *and* of any dynamic response;

3) and, as a consequence, are associated with a quasi-static failure mode (FM-QS) corresponding to a failure of the critical load-transfer mechanism at the level of the interlayer.

All other post-fracture design situations are associated with the occurrence of a dynamic event, whatever the cause and whatever the failure mode : all these design situations end with a failure mode associated to another category (FM-D).

Two main load-transfer mechanisms are identified to rule the 'ultimate' post-fracture performances in all element and loading configurations, the TCT-LTM and the OCT-LTM, according to the bridging configurations between glass fragments. In particular, the ligament function of the interlayer, describing the TCT-LTM, is identified as the critical one.

Within the complementary assumptions made for describing fractured stages, it is assumed possible to investigate the time-temperature dependence of the load-transfer mechanism by means of tests on smaller test specimens, and in particular by means of TCT-tests. The modelling of the material properties of the ligament with regard to a larger range of services conditions is identified as a probable difficulty. In a general way, the deformation capacity (ductility) of the ligament depends on the *ratio* between interfacial and bulk properties (fracture toughness and strength), in relation with two deformation mechanisms, the *delamination* from the glass substrates and the *stretching* of the interlayer under an axial tensile force.

Questions about time-temperature dependence of the post-fracture performances obviously address the two aspects, and possibly with regard to dynamic (FM-D) and quasi-static (FM-QS) failure modes; investigations carried out and reported in next chapters address essentially the second ones.

Chapter III

Time-temperature dependent mechanical behaviour of polymer interlayers : background

"*Fools ignore complexity. Pragmatists suffer it. Some can avoid it. Geniuses remove it.*"
(Alan Perlis, American computer scientist, 1922-1990)

III.1. Introduction

Laminated glass is a combination of superimposed glass and polymer sheets, adhesively bound with each other's. The first ones provide a laminated glass element its initial strength and stiffness, the second ones rather contribute to its good resistance to dynamic solicitations and its residual load-bearing capacities once some or all of the glass sheets are fractured. In ultimate fractured states, the polymer interlayer fulfils a ligament function, which results from a combination of delamination of the interlayer from the glass fragments and of its stretching over the volume released by the delamination process.

The ligament configuration has been described and analysed in previous chapter rather from structural and mechanical perspectives, assuming that the necessary properties of the individual components can be determined for characterizing the contribution of the interlayer to the overall performances of a fractured laminated glass element. In particular, influence of temperature and other time-dependent effects on the behaviour of the interlayer materials is still a matter of a variety of questions for designers and structural engineers, and in particular for designing non-conventional structural applications for which the assessment of post-fracture performances is gaining in importance.

This third chapter aims at getting a better comprehension of the main features ruling the mechanical behaviour of polymer materials, in the perspective of describing the behaviour of interlayer ligaments in fractured laminated glass unit, and of characterizing the involved material properties for design purpose.

A first section is dedicated to generalities about the mechanical behaviour of polymers and their specificities in comparison with other materials, and in particular their tensile behaviour in the large strain domain. Difference between intrinsic and macroscopic response is introduced, and particularities about the creep behaviour and associated failure modes are highlighted. Concepts and models of thermorheological simple and complex behaviour are introduced, and the importance of the phenomenon of physical ageing for some products in certain conditions is explained.

The second section analyses the consequences for assessing the mechanical properties of interlayer products, and explains some experimental issues. Two interlayer products are considered more particularly. The chapter terminates with some considerations about the choice of experimental approaches and of test configurations.

III.2. Characteristic features and behaviour of polymers

Despite polymers are already used in a variety of applications in the construction sector, as for instance in geotechnical products (membranes,…) and in millwork of windows and facades (PVC,…), their mechanical behaviour remains generally not well understood. The purpose of this section is to situate particularities of polymers in general, and interlayer products in particular, in comparison to more traditional construction materials.

This section starts therefore with general considerations about polymer materials and their different categories, and is followed by the presentation of models relative to their time-temperature dependent response. Intrinsic time-temperature behaviour is described, and two important categories of behaviour are introduced, the thermorheological simple and thermorheological complex models. Factors affecting the mechanical properties of products during their lifetime are identified, and among these physical ageing appears as an important phenomenon to account for. Outcomes follow for modelling the macroscopic response of polymer products in general, and particularities of polymers used as adhesive materials are finally pointed out.

III.2.1. Identification and classification of polymer materials

Polymers are a class of materials besides ceramics and metals, characterized by other dominant type of atomic bonds (Sharpe 2008).

Ceramics are mainly using the strongest interatomic bonds, the ionic bonds, providing these materials with superior chemical stability, but requiring higher processing temperature. Mineral glass is a typical example of ceramics, and similar chemical bonds are developing in concrete materials. Materials of this category are typically stiff and strong (high elastic modulus and high yield strength), but brittle and with a relatively low resistance to impact and surface defect. As it is the case for glass, the macroscopic strength is generally much lower than the intrinsic, interatomic bond strength, because of the sensitivity of strength to defects and stress concentrations, possibly in interaction with environment conditions (stress corrosion) (Haldimann et al. 2008). Their structure can vary from amorphous to (partially) crystallised forms, according to the regularity of the spatial organisation, which depends on chemical composition and processing conditions (cooling rate).

In contrast, metals and alloys mainly exhibit interatomic metallic bonds between strongly electropositive elements, which are less strong than ionic and covalent bonds. It provides metals with moderate resistance to environmental degradation and more variable mechanical properties according to their microstructure. Ductility and strength of metals largely depend on propagation of micro-defects (dislocations) in crystal lattices and between grain boundaries, and therefore largely depend on their grains size and shape, and in some cases on possible

different crystal structure (phase), both depending on processing conditions (Sharpe 2008).

Properties of ceramics and metals are thus mainly depending on short-range interatomic bonds. In comparison, polymers are more complex structures, made of long molecular chains, characterized by their chemical composition (individual molecular unit or *'mer'*), their structure (length and shape of polymer chains : linear, branched or cross-linked) but also their spatial organisation, or conformation (including possible entanglement between molecular chains which are not chemically connected). Mechanical properties of polymers largely depend on the secondary bonds between the macromolecular chains and the resulting segmental mobility : these intermolecular bonds can be of chemical nature (cross-linked networks involving covalent or weaker hydrogen bond) or of physical nature (electrical or Van der Waals forces) (Sharpe 2008; van der Vegt 2006).

Polymers are traditionally classified in three categories according to the nature and density of the cross-links (Louter 2011; van der Vegt 2006) :

1) **Thermoplastics** are characterized by long linear or branched chains, obtained by polymerisation (namely a process aimed at creating ionic or covalent bonds between chains ends), with physical bonds as secondary interaction forces between individual macromolecular chains (Van der Waals forces,…). The long molecular chains generally have coil shapes which are mutually entangled (Figure III.1), and this configuration largely explains their specific mechanical behaviour (van der Vegt 2006). Thermoplastics are softening up to melting and flow[1] when heated, and solidify again when cooled down : it makes this category of polymer products recyclable. According to the regularity and structure of the molecular chains and to the cooling rate, they may exhibit an *amorphous* or *semi-crystalline* structure.

2) **Thermosets** (also called thermosetting plastics) are heavily cross-linked and therefore rather exhibit a network structure, obtained by chemical reactions activated by environment (e.g. air or light activated hardening) or between different components (resins,…) during a so-called *curing* process carried out at or above room temperature. Thermosets generally exhibit an amorphous structure, show little or no softening when heated, and do not flow, but heating at larger temperature can lead to chemical degradation.

3) **Elastomers** are low cross-linked polymers, with the particularity of being able to deform elastically to large strain range at room temperature, what is referred to as a rubber-like or hyperelastic behaviour. The presence of the chemical cross-links between the molecular chains prevents the material to flow when heated.

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[1] Flow can refer to plastic flow, which rather corresponds to a material softening in a solid phase, or a viscous flow, which rather corresponds to a true melt state (liquid state) of the material.

This three categories classification system seems to rely on criteria of different nature, in relation with characteristics of processability, applicable production methods and end-product properties on the one hand, and with the nature/structure of secondary, intermolecular bonds on the other hand. It is not clear which is the decisive criterion for determining to which category a product belongs, and therefore it might appear as not always univocal or consistent[2].

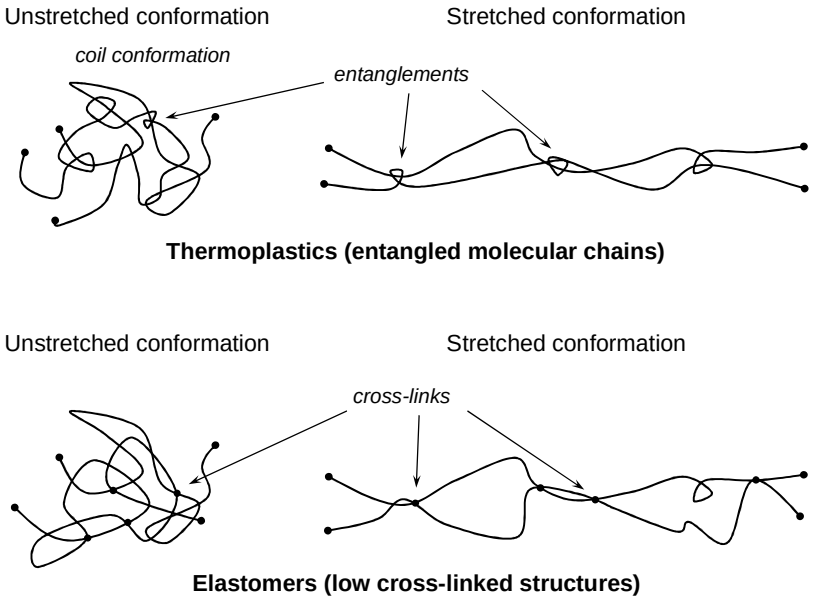

Figure III.1 – Schematic representation of secondary bonds for two families of polymers (amorphous thermoplastics and elastomers)

Nevertheless, this simple classification system highlights the importance of secondary bonds and conformation[3] of the molecular chains on the macroscopic mechanical response of plastic end-products, especially in the short deformation range on the one hand, and on the polymer processability on the other hand. Processability is an essential feature for making plastic products in general, and interlayers in particular, because it is going to have an influence on the conformation of the chains, and consequently on the mechanical properties. It will in fact appear as a non-negligible aspect with regard to the choice of representative test specimens.

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[2] For instance, a sub-category of elastomers is named "thermoplastic elastomers" (van der Vegt 2006), which are characterized by the fact that "without vulcanization, they behave as cross-link rubbers" (reference to a type of mechanical behaviour, associated with a certain chemical structure), and "can be processed as a thermoplast" (reference to processability).

[3] Conformation is the term used in chemistry to refer to structural, "spatial" arrangement at molecular level.

There are many other classification registers of polymer materials and products, referring to their main chemical component (carbon for organic polymers and silicon for silicones, the first being the most important group of polymers, and the second the most important group of inorganic polymers), the chemical composition of the molecular chains and their molecular structure (linear, branched, cross-linked,… ; amorphous or semi-crystalline;…), their source (natural, synthetic,…), their homogeneity or heterogeneity grade (polymer blend, copolymer, block polymer; use of additives, as plasticizers, etc., use of fillers, fibres, etc.), their stiffness, ductility and toughness at room temperature (rigid, semi-rigid, flexible) and their typical failure pattern (ductile, brittle…), etc. (Gooch 2011; van der Vegt 2006). The latter categories referring to mechanical properties are rather arbitrary fixed on the basis of conventional tests and criteria, executed and evaluated in reference conditions, and are not always sufficient[4].

It seems worth, finally, to give a complementary comment about the identification and naming of polymer products. Polymers (in fact, organic polymers) were historically named according to their main chain's molecular composition (monomer unit), but this method led, for more complex products developed in the meantime, to long and ambiguous designations. An alternative method, structure-based nomenclature, has been developed, which is based on the concept of "constitutional repeating unit" in polymer chains (Gooch 2011). In both cases, the nomenclature of polymer products relies on a descriptive approach of the constitutive molecular structure, which appears of limited interest with respect to a performance based approach for the evaluation of products (see Chapter I). For non-specialists, polymer names as polycarbonate (PC) and polyvinyl butyral (PVB) must rather be understood as referring to a family of products, most of the time produced by different manufacturers and/or available in different commercial grades. Indeed, products of a same family can exhibit quite different mechanical properties associated to a change of conformation (volume density, polymer-risation grade, density of entanglements or of cross-links, molecular chains orientation,…), according to secondary components (as plasticizers in PVB's, and other fillers or additives for other products) and/or to different production and processing conditions (Gooch 2011; van der Vegt 2006).

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[4] Let us consider an example to illustrate this : a rigid plastic is defined as "*a plastic that has a modulus of elasticity ether in flexure or in tension greater than 700 MPa (100 kpsi) at 23°C and 50% relative humidity when tested in accordance with ASTM methods D 747, D 790, D 638, or D 882 (ASTM D 883)*" (Gooch 2011). This definition is completed by this comment : "*This simple ASTM criterion has not always been adequate, especially with respect to vinyls whose impact strengths and other properties can vary widely while elastic modulus remains fairly constant. Vinyls are classified as rigid if their moduli are 1.4 GPa or higher, semirigid from 0.4 to 1.4 GPa, and flexible below 0.4 GPa.*" The issue is similar with the other mechanical properties and in other standardization frameworks, even if details of test configurations and evaluation criteria may vary. The issue is however probably not limited to the case of vinyls, but addresses more generally the question of the identification of the application scope on which a property value is meaningful.

It is not different for interlayer polymer materials commonly used in laminated glass products : for instance, PVB (polyvinyl butyral) and EVA (ethylene vinyl acetate) refers to a variety of commercial grades produced by different manufacturers, each providing enhanced feature or performance for a specific application or specific performance requirement. Each family of interlayer products however exhibits some common characteristic features, which are regularly presented, promoted or compared in literature. It appears among others that some grades of a same 'product family'[5] can be associated to different categories of the traditional classification system presented here above (Goebel 2013). Besides, final properties of interlayers in laminated glass units can depend on lamination conditions.

A more detailed discussion about the classification of interlayer products is following in next section III.3, and will consider more particularly two types of interlayer materials used in laminated safety glass products, which appear to belong to the category of the thermoplastics. However, as nowadays there exist also interlayer products belonging to the other categories, it seems useful in this section to not focus exclusively on thermoplastics for addressing polymers' mechanical behaviour, in relation with the more general framework considered in previous chapters.

III.2.2. Intrinsic mechanical behaviour of polymers

The mechanical response of polymers at constant temperature is firstly introduced. Secondly, the different characteristic temperatures used to describe a given polymer are explained and discussed. Finally, the influence of temperature on the evolution of the intrinsic response of a polymer material is sketched.

III.2.2.1. Intrinsic response at constant temperature

The onset for modelling the non-linear behaviour of solid amorphous thermoplastics is based on the distinction of contributions of the intermolecular connections in the short deformation range and of the network entanglement in larger deformation range, as illustrated in Figure III.2 (Meijer and Govaert 2005; van der Vegt and Govaert 2003). The intrinsic curve represents the local response of a polymer to a progressive load applied at a constant strain rate[6].

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[5] Note that 'product family' in this context is not complying with the concept defined in Chapter I.

[6] An intrinsic curve is typically determined by means of uniaxial compression tests, as, *performed in appropriate conditions*, it leads to homogeneous deformations (along the loading axis direction). Indeed, an uniaxial tensile test generally involves non-homogeneous deformations, and the obtained loading curve is then associated to a macroscopic response (Meijer and Govaert 2005; van der Vegt and Govaert 2003); uniaxial tensile test is therefore further discussed in paragraph III.2.4. Nonetheless, methods are reported for obtaining the intrinsic curve by means of tensile test configurations, which require the use of advanced measurement methods of the local deformations (G'Sell et al. 1992, 2002; Grytten et al. 2009).

A general expression of the intrinsic stress-strain curve represented in Figure III.2 takes the next form (Klompen 2005) :

$$\sigma(\dot{\varepsilon}, S, \varepsilon) = \sigma_s + \sigma_r = \sigma_{rej,0}(\dot{\varepsilon}) + \Delta\sigma_y(S) + \sigma_r(\varepsilon) \qquad \text{(III.1)}$$

with σ_s named the *driving stress*, separated into two components, a *rejuvenated yield stress* $\sigma_{rej,0}$ and the *yield drop* $\Delta\sigma_y$[7]; and σ_r the *strain hardening stress*. The components of the driving stress are further discussed below. According to this form, the rejuvenated yield stress depends mainly on the applied strain rate and the ambient temperature, the strain hardening stress depends mainly on the deformation level, and the yield drop height is essentially affected by the thermomechanical history (represented by the parameter *S*, further discussed in paragraph III.2.3 below).

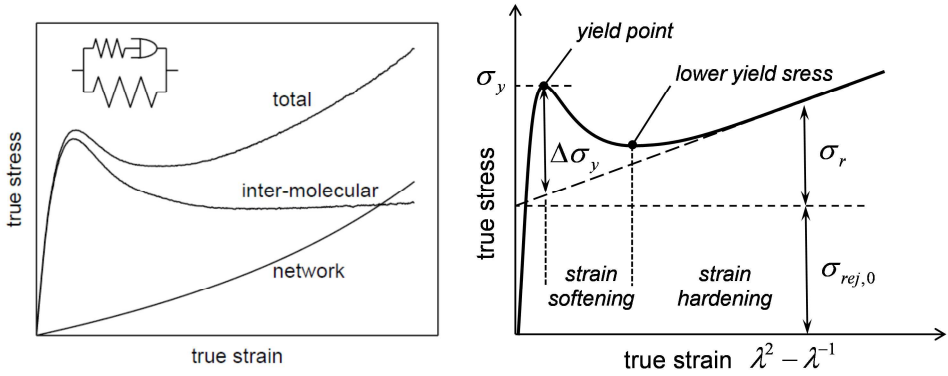

Figure III.2 – Stress decomposition of intrinsic response of amorphous thermoplastics
a) as proposed by Haward and Thackray in 1967 – from (Meijer and Govaert 2005)
b) decomposition in three components – based on (Klompen 2005)

In this view, the non-linear response and the presence of a *yield point* followed by *strain softening* in the small strain range are associated to breakage of secondary bonds and consequent segmental movements, leading to deformations described as irreversible and viscoplastic. In contrast, the large strain deformation is rather due to conformational changes of the molecular chains, involving stretching and orientation of the molecular coils (Figure III.1) and leading to *strain hardening*. These conformational changes are of viscoelastic nature : once unloaded, the molecular chains tend to come back, with a certain time delay, to coil conformation, which is a state of lower entropy. Therefore the large strain elastic

[7] The rejuvenated yield stress seems to correspond with the concept of overstress (Hooper et al. 2012) when the yield drop is equal to zero; in literature yield drop is also used to refer to the 'height difference' between the yield stress and the lower yield stress (Figure III.2).

behaviour is also named *entropic elasticity* (van der Vegt 2006), or rubber-elasticity.

The distinction between the viscoelastic or viscoplastic nature of the deformations[8] is however difficult to establish univocally and depends on the conditions of measurement (Sharpe 2008), and in particular on the considered test temperature and time scales[9].

III.2.2.2. Characteristic temperatures for polymers

Typical characteristic values of temperature defined for a polymer material, besides limit temperatures of use, are two transition temperatures : the glass transition temperature and the melt temperature. While the melt temperature affects the crystalline phase, the glass transition temperature affects the amorphous phase. Transition mechanisms are in fact depending on the exposure duration to a temperature, what explains that temperature and time dependence of polymer properties are inter-related.

Polymers present longer relaxation times than metals or ceramics that is explained by mechanisms of energy dissipation occurring at a larger scale of long-chain molecules rather than at the scale of atoms and ions (Sharpe 2008). Apparent plastic behaviour of polymers is thus due to different microscopic mechanisms than in metal alloys : whereas for the latter, plasticity is explained by the presence and the propagation of structural defects (dislocations) in crystalline structures (Sharpe 2008), in polymers it rather corresponds to a phase change of the amorphous structure from a (frozen) glassy state to a (mobile) rubber state. This specificity of polymer plasticity is related to the physical nature of their solid phase : below a certain temperature, the molecular mobility is getting too low to allow the material to attain a thermodynamic equilibrium, and this distinguishes the solid, glassy state of polymers (no thermodynamic equilibrium) from the rubber state (thermodynamic equilibrium).

The rubber state is a physical state specific to polymers, between solid and liquid, showing characteristics of both states : it is coherent and elastic as a solid, but it has a thermal coefficient (describing the temperature dependent volume variation) of the liquid state (Figure III.3a).

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[8] The concepts of anelastic and plastic strains used for instance by Visser (Visser 2010) seems to address the same question with regard to the distinction between reversible and irreversible deformations.

[9] Fortunately, a univocal distinction between viscoelastic and viscoplastic effects, namely the determination of the reversible or irreversible character of deformations, does not seem necessary for all design problems. It is the case for instance with regard to the quasi-static designed situation defined in Chapter II, and the response to creep load mode discussed below.

The *glass-rubber transition temperature* T_g (or simply glass transition temperature) characterizes this "state transition" between glassy and rubber states. It appears however to be not a real state transition, in the sense of a balance between two thermodynamic stable states, and because of its dependence on the cooling rate (van der Vegt 2006). The glass-rubber transition is not a process occurring in a narrow temperature range such as melting of water, but involves slower processes occurring at a temperature and time dependent rate in a temperature range around T_g ; accordingly, a co-existence of rubber and glassy phases in an amorphous polymer material can exist in this temperature range[10].

A second characteristic temperature is the *melt temperature*, or rubber-liquid transition temperature T_m, above which the crystalline fraction is reduced to zero and the amount of entanglements between the polymer chains is largely reduced : this explains why the melt temperature is also varying in function of the chain length of the polymer[11].

On a general way, the effective softening temperature is thus varying according to the phase state (of the amorphous phase) and to the crystalline fraction, that last one being also dependent on cooling rate between T_m and T_g . The values of these two characteristic temperatures are however not univocal for one polymer material, as they depend on the cooling rate[12] and the orientation of the molecular chains[13]; consequently, the mentioned reference values are in some extent conventional[14].

Some polymers show a secondary glass transition temperature below T_g , named β-transition, in comparison to the main glass-rubber transition named α-transition. The necessity to identify the presence of such a secondary transition mechanism in a polymer material seems again determined by the field of interest for which its mechanical behaviour has to be determined. Accordingly, the thermorheological simple (only α-transition present) or complex (α- and β-transitions) nature of a

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[10] This assumption of co-existing phases in structural adhesive is for instance explicitly used in terms of volume fractions by Bai (Bai and Keller 2011).

[11] The melt temperature is thus a non-defined parameter for amorphous polymers. The ability to crystallise varies according to the nature (structure) of the polymer.

[12] The cooling rate between the melt temperature and the glass transition temperature influences the crystallisation process, among others the regularity and size of crystallites. The rate of crystal growth is maximal at a few degrees below the melt temperature, and decreases to zero at the glass transition. Oriented chains (under stain) crystallize faster (van der Vegt 2006).

[13] The melt temperature can rise of a few dozens of degrees when highly stretched (van der Vegt 2006)

[14] The glass transition temperature can vary with 5 to 10°C between very rapid and very slow cooling rate; the melt temperature can vary in larger extent according to the degree of crystallization and the chain orientation induced in the large strain range (van der Vegt 2006). In others words, the determination of the characteristic values of temperature depends on the followed path.

polymer material seems also relative (Klompen 2005; van der Vegt 2006). Semi-crystalline structures can give rise to similar secondary relaxation mechanism but with a transition temperature *above* T_g (Klompen 2005). The secondary transition temperature is often associated to the toughness (thus among others the impact resistance) of a polymer product, especially for amorphous ones (van der Vegt 2006).

Figure III.3 summarizes the main changes with regard to temperature dependent changes of volume and of elastic modulus (van der Vegt 2006). It appears then that crystalline fraction on the one hand and cross-links density on the other hand both can reduce the height of the stiffness drop between the glassy and the rubber states, and displace the effective range of softening temperature above the glass transition temperature T_g [15]. The loss of stiffness represented by the elastic modulus is accompanied by a decrease of the apparent viscosity (defined as the ratio between the instantaneous stress state and the simultaneous rate of deformation, or strain rate)[16]. Only in a limited range of applied small stress the viscosity is a material constant, leading to a "Newtonian" behaviour and accordingly to linear viscoelasticity; in general it is a stress dependent value, the viscosity decreasing then with applied stress according to a power-law (van der Vegt 2006), bringing the response into a non-linear viscoelastic range (Klompen 2005).

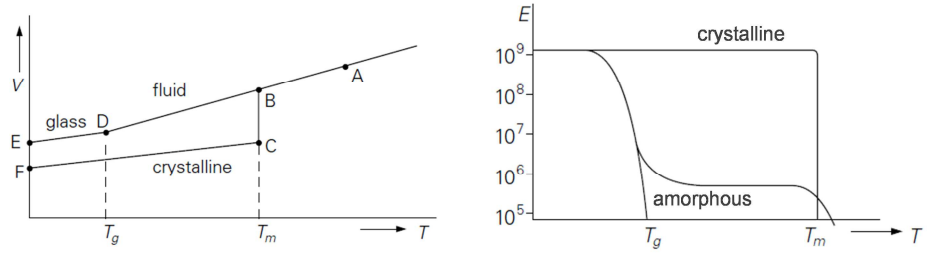

Figure III.3 – Change of phase states of polymers and associated characteristics a) volume variation in function of temperature, b) variation of elastic modulus in function of temperature - reproduced from (van der Vegt 2006)

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[15] A softening temperature is sometimes also defined on a conventional basis, for instance the standardized Vicat softening temperature (van der Vegt 2006); in a general way, the value of a softening temperature is comprised between T_g and T_m.

[16] It is further interesting to note that thermoplastics are much softer in the solid state than metals and glass (smaller initial elastic modulus, at ambient temperature), and simultaneously exhibit a much larger viscosity in the melt state. For instance, steel exhibit an elastic modulus of 210.000 MPa (at 20°C) and a viscosity about 0.006 Pa.s (at processing temperature of 1600°C), glass a modulus of 70.000 MPa (at 20°C) and a viscosity of 0.008 Pa.s (at processing temperature of 1600°C); in contrast, elastic modulus of polymers varies in a range about 1 to 15.000 MPa (in service conditions) and a viscosity in ranges of 100 to 10.000 Pa.s (at processing temperatures) (van der Vegt 2006). Comparison of characteristic temperatures between different classes of materials with regard to their mechanical response is thus not straightforward.

In contrast with thermoplastics, elastomers typically show a characteristic glass transition temperature below the ambient (service) temperature, and an elastic response close to an ideal rubber, namely with no time-dependent, viscous response (no flow).

For completing the picture about defined characteristic values of temperature for polymers, the definition of a *maximum temperature of use* is normally associated to the occurrence of (chemical) *degradation* mechanisms. However, such a value is also generally associated to exposure duration (Weller et al. 2010). This upper limit for the service temperature range can be improved by the chemical composition of the polymer (use of stabilizers,…). At the lower side of the temperature range, a *ductile-brittle transition temperature* (or *tough-brittle transition temperature*) can also be identified, but again, such a characteristic value is generally associated to a particular configuration (geometry and surface defects as notch,…) and loading case (for instance, resistance to impact). The lower limit defined by a ductile-brittle transition temperature lies generally below the other transition temperatures, and is higher in case of resistance to impact than in case of other, slower loading cases[17] (van der Vegt 2006).

III.2.2.3. Effect of temperature on the mechanical behaviour

The general equation (III.1) here above however does not represent explicitly the dependence of the response to the ambient temperature. The effect of temperature on the different identified stress components is reported in literature, but no clear integrated formulation with the temperature dependence of the different terms seems available.

The theoretical temperature dependence of the large strain rubber-elasticity (entropic elasticity) takes the following form, for a uniaxial tensile loading configuration and *deformation at constant volume* (*incompressible* solid)[18] :

$$\sigma_x = N.k.T.\lambda_x = N.k.T.\left(1+\varepsilon_x\right) \qquad \text{(III.2)}$$

with σ_x the nominal axial stress (related to the area of the initial cross-section), $\lambda_x = L - L_0$ the axial stretch and ε_x the corresponding nominal axial strain, N the amount of chain units between cross-links per volume unit, k the Boltzman constant and T the absolute temperature of the material (in Kelvin).

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[17] The ductile-brittle transition temperature in fact rather addresses the macroscopic behaviour and failure modes; see also paragraph III.2.4 below.

[18] It may looks strange to express a large strain response in terms of nominal stress and strain, only representative in short strain deformation range; this expression is chosen here as it correspond to the usual expression of results of a conventional uniaxial tensile test, see paragraph III.2.4.

Reworking this equation for expressing the relation between the true stress ($\sigma = \sigma_x / \lambda_x = A_0 / A$[19] with A_0 the initial cross-section's area and A the deformed cross-section's area) and the true strain ($= \lambda_x^2 - 1/\lambda_x$), these are related to each other by the same constant *N.k.T.* This relation however *only applies for an ideal rubber*, namely with no thermal expansion and with permanent cross-links delimiting the molecular segments which can move freely; some elastomers show a behaviour close to such an ideal rubber (far) above their glass transition temperature (van der Vegt 2006).

In thermoplastics, because the temporary entanglements can further be loosened under stress leading to a rather viscous response, the large strain stiffness is proportional to *N.k.T* only on a limited part of the characteristic stress-strain curve, in the (post-yield) large strain range and only in function of a fraction of the stress (right part of the curve of Figure III.2), and is then named the strain hardening modulus G_r (van der Vegt and Govaert 2003) :

$$\sigma = \sigma_{y,0} + \sigma_r = \sigma_{y,0} + G_r . \left(\lambda_x^2 - 1/\lambda_x \right) \tag{III.3}$$

Note the similitude of this equation with equation (III.1); accordingly, the driving force behind the large strain elastic response of thermoplastic (entangled chains) and of elastomers (cross-linked chains) seems of similar nature.

The temperature dependence of the yield stress, or viscoplastic response of the glassy phase, is often well caught by Eyring's plastic flow theory (Klompen 2005; van der Vegt 2006); it leads to the next relation expressing the dependence of the yield stress upon temperature and applied strain rate, for a *thermorheological simple model* and for a uniaxial loading situation as here above :

$$\sigma_y = \frac{k.T}{V^*} . \ln \left(\frac{2.\dot{\varepsilon}}{\dot{\varepsilon}_0^*(T)} \right), \; with \;\; \dot{\varepsilon}_0^*(T) = \dot{\varepsilon}_0 . \exp \left(- \frac{\Delta U}{R.T} \right) \tag{III.4}$$

$$and \;\; \sigma_y >> \frac{k.T}{V^*} = \sigma_0 \tag{III.5}$$

or to the more general form when condition (III.5) is not fulfilled :

$$\sigma_y = \frac{k.T}{V^*} . \operatorname{arsinh} \left(\frac{\dot{\varepsilon}}{\dot{\varepsilon}_0^*(T)} \right), \; with \;\; \dot{\varepsilon}_0^*(T) = \dot{\varepsilon}_0 . \exp \left(- \frac{\Delta U}{R.T} \right) \tag{III.6}$$

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[19] This relation explains also why the axial stretch λ_x is also named the *draw ratio*.(in reference to the second term, the ratio A_0/A of areas of undeformed on deformed cross-section).

In these equations, variables are the yield stress σ_y, the strain rate $\dot{\varepsilon}$ [20] and the ambient temperature T (absolute value of temperature in Kelvin). The three parameters (constants) of the model describing the *relaxation kinetics* are the activation volume V^* (in volume unit), the activation energy ΔU (in energy unit by mole) and the pre-exponential coefficient $\dot{\varepsilon}_0$ also named process rate constant; k and R are the Boltzmann and the universal gas constants, respectively[21]. The temperature dependence follows an Arrhenius function[22]. The simplified expression given by (III.4) is convenient[23], as it corresponds to a straight line on a semi-log plot of the yield stress against (the logarithm of) the strain rate at a given temperature, with a slope equal to the term kT/V^*.

The formulation of the viscoplastic criterion here above accounts thus for a single relaxation mechanism, namely the glass transition, or α-transition. The above equations relate algebraic values of stress and strain rate with each other (namely uniaxial tensile configuration or pure shear), but corresponding generalized tensorial expressions, necessary for use in numerical modelling methods with volumetric ("3D") elements, have also been developed (Klompen 2005; Meijer and Govaert 2005)[24]

The characteristic stress σ_0 defined by equation (III.5) is not a material constant, but it rather corresponds, for a given temperature T, to the stress level at which significant non-linear viscoelastic deformation appears[25]. A zero-viscosity at small stress is associated to this characteristic stress :

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[20] In fact, in the Eyring model this term is the plastic strain rate, which is equal at yield to the applied strain rate (in case of homogeneous deformations).

[21] The Boltzman constant $k = 1.381 \,.\, 10^{-23}$ J/K and the universal gas constant $R = 8.314472$ J/(K.mol) are related to each other by the number of particles in one mole, which is the Avogadro constant $N_A = 6.022 \,.\, 10^{23}$ mol^{-1}.

[22] The Arrhenius function is known to successfully describe the temperature dependence of rate of change of many thermally induced chemical reactions and solid-state processes (Sharpe 2008). It expresses that time and temperature have equivalent effects on a transition mechanism.

[23] $\ln(2.a) \approx \text{arsinh}(a)$, and conversely $\sinh(a) \approx 0.5 \,.\, \exp(a)$, for $a \geq e = 2.74$ (the choice of the limit value can depend on the desired precision…). On this condition, the hyperbolic functions sinh(a) and its reciprocal arsinh(a) (also noted $\sinh^{-1}(a)$) correspond to a straight line on a semi-logarithmic plot (respectively on a plot of a-log(sinh(a)) and of log(a)-arsinh(a)).

[24] These distinguish among others the influence of volumetric and deviatoric components in the response of the stress tensor. One such model is meanwhile referred to as the Eindhoven Glassy Polymer model, as in (Visser 2010).

[25] The characteristic stress value is only a few percent's of the yield stress value at a given temperature; to give an order of magnitude, in the models calibrated by Klompen for two different polymer materials (PMMA and PC), $\sigma_0 < 0.05 \,.\, \sigma_y$ (Klompen 2005). For an applied stress $\sigma < \sigma_0$, the behaviour is expected to comply fairly well with a linear viscoelastic model.

$$\eta_0(T) = \frac{\sigma_0(T)}{\dot{\varepsilon}_0^*(T)} = \frac{\sigma_0(T)}{\dot{\varepsilon}_0}.\exp\left(\frac{\Delta U}{R.T}\right) = \frac{k.T}{V^*.\dot{\varepsilon}_0}.\exp\left(\frac{\Delta U}{R.T}\right) \quad \text{(III.7)}$$

and so an expression is obtained for the (apparent) stress-dependent viscosity corresponding to the onset of the plastic flow (Klompen 2005); in other words this expression is only valid at the yield point :

$$\eta(\sigma,T) = \eta_0(T).\frac{\sigma/\sigma_0}{\sinh(\sigma/\sigma_0)} = \eta_0(T).a_\sigma(\sigma) \quad \text{(III.8)}$$

where $a_\sigma(\sigma)$, called the *stress dependent shift function*, is used to determine whether a time-stress equivalency is applicable (Klompen 2005). However, since the characteristic stress is defined for a given temperature, the function $a_\sigma(\sigma)$ also includes a temperature-dependent factor (reference temperature), and should then rather be written as $a_\sigma(\sigma,T)$ (Visser 2010). Similarly, in equation (III.7) the Arrhenius term defines a time-temperature equivalency, with a corresponding *temperature dependent shift function*[26]

$$a_T(T) = \exp\left(\frac{\Delta U}{R.T}\right) \quad \text{or} \quad \ln(a_T(T)) = \frac{\Delta U}{R.T} \quad \text{(III.9)}$$

It is practically often more convenient to express this function in relation to a reference temperature $T_{ref} \neq 0\,K$ so that $a_T(T_{ref}) = 1$, giving the alternative expression

$$a_T(T) = \exp\left(\frac{\Delta U}{R}.\left(\frac{1}{T} - \frac{1}{T_{ref}}\right)\right) \text{ or } \ln(a_T(T)) = \frac{\Delta U}{R}\left(\frac{1}{T} - \frac{1}{T_{ref}}\right) \quad \text{(III.10)}$$

with

- $a_T(T) < 1$ and $\ln(a_T(T)) < 0$ for $T > T_{ref}$, corresponding to a reduction of the yield stress and the plastic flow viscosity for a higher temperature;
- $a_T(T) > 1$ and $\ln(a_T(T)) > 0$ for $T < T_{ref}$, corresponding to a raise of the yield stress and the plastic flow viscosity for a lower temperature.

[26] This is conceptually similar to shift-functions defined on other domains, as for instance with the WLF-model for linear viscoelasticity already used in modelling the time-temperature dependence of the shear modulus of interlayers (Bennison et al. 1999; Callewaert 2011), but the expressions of the shift-functions are different.

Expressed in this second form, the sign of the coefficient $a_T(T)$ indicates a horizontal shift direction on some semi-log or double log plot (with a log time scale on the horizontal axis), and its value the magnitude of the shift, namely the distance between two curves or series of results at the two different values of temperature considered.

Polymers presenting two relaxation mechanisms (α- and β-transitions) are associated to a *thermorheological complex* behaviour. A generalized formulation of the yield stress is provided by the Ree-Eyring model (Klompen 2005; van der Vegt and Govaert 2003) :

$$\begin{aligned}\sigma_y = \sigma_\alpha + \sigma_\beta &= \frac{k.T}{V_\alpha^*}.\text{arsinh}\left(\frac{\dot{\varepsilon}}{\dot{\varepsilon}_{0,\alpha}^*(T)}\right) + \frac{k.T}{V_\beta^*}.\text{arsinh}\left(\frac{\dot{\varepsilon}}{\dot{\varepsilon}_{0,\beta}^*(T)}\right) \\ &= \sum_{x=\alpha,\beta} \sigma_{0,x}.\text{arsinh}\left(\frac{\dot{\varepsilon}}{\dot{\varepsilon}_{0,x}^*(T)}\right) \qquad \text{(III.11)}\end{aligned}$$

$$\text{with } \dot{\varepsilon}_{0,x}^*(T) = \dot{\varepsilon}_{0,x}.\exp\left(-\frac{\Delta U_x}{R.T}\right) \text{ and } \sigma_{0,x} = \frac{k.T}{V_x^*}, \text{ for } x = \alpha, \beta$$

The amount of model parameters compared to the simple Eyring model is doubled up to six, three for each relaxation mechanism; it introduces inflexion points (in fact, rather a transition zone) between straight segments on a semi-log plot (Figure III.4). This formulation appears to be quite robust for describing the yield behaviour of many different polymer materials – see the various materials and conditions for which it is successfully applied, for instance in (van Erp et al. 2012; Klompen 2005; van der Vegt and Govaert 2003).

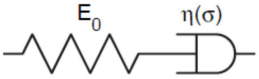
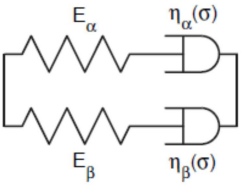
As mentioned here above, it seems that a polymer material does not intrinsically behave as a thermorheological simple or complex material, but rather that it complies with the typical response of the one or the other models on a defined application scope, in function of whether the influence of one or more mechanisms is significant (Klompen 2005; van der Vegt and Govaert 2003)[27].

There are a few other particular features associated with the thermorheological simple or complex models, which can be of practical use. Among these there are the so-called time-stress and time-temperature superposition principles. The latter basically state whether it is possible to derive some mechanical function (having a

[27] It is useful to note that the evaluation of whether a second mechanism has a significant influence on the mechanical response is not only related to an identified application scope, but can also vary according to the considered performance or property. For instance, no significant effect of a secondary mechanism might be noticed on the time-temperature dependence of the yield stress, altogether with a noticeable effect on the shape of (part of) the creep curve (Klompen 2005).

'smooth curved shape') at a given stress level (or temperature) by shifting the considered curve along the time axis (on a log scale) from a reference stress level (or reference temperature T_0 or T_{ref}), by means of a stress-shift function $a_\sigma(\sigma)$ for the time-stress superposition (equation (III.8)) (or a temperature-shift function $a_T(T)$ for the time-temperature superposition, equation (III.9)).

Table III.1 – Viscoplastic models for yield stress of polymers : summary

Thermorheological simple model	Thermorheological complex model
	
One non-linear Maxwell relaxation element, where the stress-dependent dashpot corresponds to a Eyring element	Two non-linear Maxwell relaxation elements in parallel, where the stress-dependent dashpots correspond to a Ree-Eyring equation
Eyring model : equations (III.4) to (III.9), 3 constitutive parameters	Ree-Eyring model : equations (III.11) and (III.12), 6 constitutive parameters
Time-temperature and time-stress superposition applicable	Time-temperature and time-stress superposition non applicable

Without going into the mathematical details of these superposition principles[28], in a general way some are applicable for thermorheological simple models but not for thermorheological complex models. Illustrative of this difference, analytical shift-functions $a_\sigma(\sigma)$ and $a_T(T)$ defined here above for the thermorheological simple model have no equivalent for thermorheological complex models. In the latter case, analytical expressions of stress dependent functions can only be obtained for each individual relaxation process, namely by replacing in above expression (III.8) the total stress by the partial stress (Klompen 2005) :

$$\eta_x(\sigma_x,T)=\eta_{0,x}(T).\frac{\sigma_x/\sigma_{0,x}}{\sinh(\sigma_x/\sigma_{0,x})}=\eta_{0,x}(T).a_{\sigma,x}(\sigma_x)\quad(x=\alpha,\beta)\qquad\text{(III.12)}$$

A consequence is that temperature-activated and stress-activated mechanisms are similar for a thermorheological simple behaviour, allowing to separate the stress

[28] Corresponding formulations can be found for instance in (Ferry 1980) for viscoelastic models in general and in (Klompen 2005) for yield stress, relaxation and creep functions of solid thermoplastics in particular.

and the temperature dependence into different terms in the expression of the yield stress. However, when a thermorheological complex behaviour is considered, there is not such an equivalency of effects of stress and temperature. This is for instance illustrated for creep compliance curves in Figure III.5 (Klompen 2005). More generally, this is one of the reasons why *extrapolation* of experimentally measured behaviour of polymers is delicate.

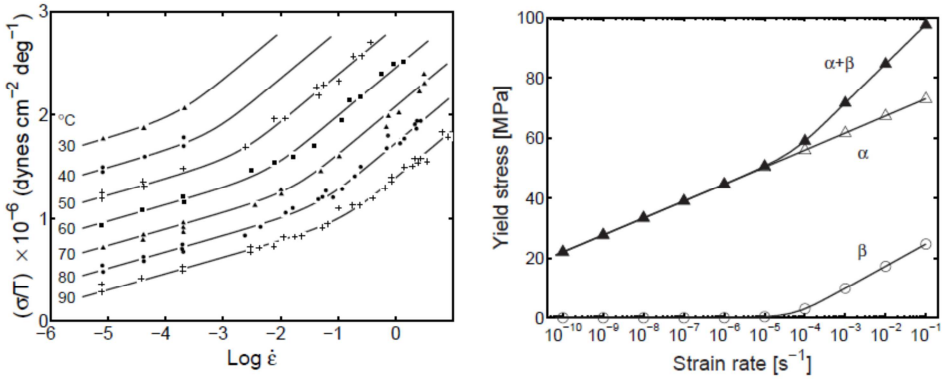


Figure III.4 – Time-temperature dependence of the yield stress (visco-plasticity) for a thermorheological complex behaviour a) experimental results for PMMA b) contribution of the two relaxation mechanisms to the strain rate dependence of yield stress according to equation (III.11) (Klompen 2005)

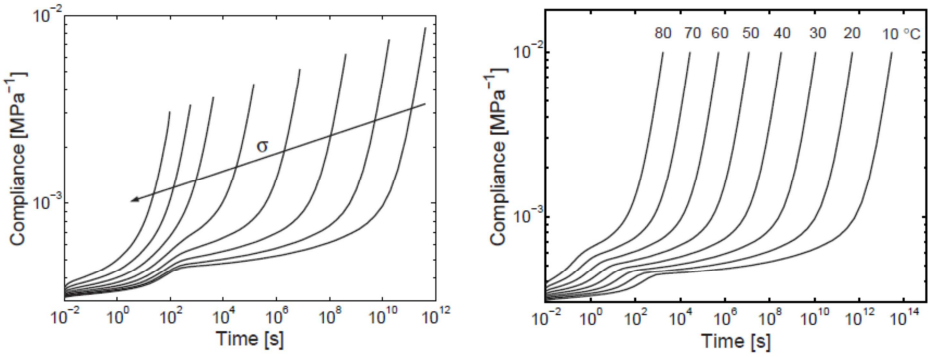


Figure III.5 – The effects of stress and temperature on creep compliance curves are not equivalent for thermorheological complex material – modelled creep curves for PMMA in uniaxial tensile loading configuration : a) for creep loads from 5 to 75 MPa at 20°C, b) for constant creep load of 5 MPa at different test temperatures (Klompen 2005)

The yield stress as determined by the plastic flow theory of Eyring (in equations (III.6) or (III.11)) predicts the *initiation* of strain softening along the intrinsic strain-stress curve sketched in Figure III.2, but does not predict the consecutive yield drop (diminution of the applied stress) due to the material softening nor the following strain hardening occurring at larger stretch level, namely *it does not account for the post-yield behaviour*.

However, the corresponding macroscopic behaviour of a polymer component, typically in response to a tensile load, is known to depend on the post-yield part of the intrinsic curve. In particular, whether the corresponding failure mode[29] is ductile or brittle depends on the ratio between strain softening and subsequent strain hardening (van der Vegt and Govaert 2003). Quantitative modelling of the (intrinsic) post-yield behaviour is investigated among others by Klompen (Klompen 2005).

The intrinsic behaviour discussed so far corresponds to the response of the material to a homogeneous deformation at constant strain rate; Table III.1 summarizes the main characteristics of thermorheological simple and complex models for the yield stress. What can we learn from it with regard to other loading modes, and in particular with regard to the response to creep (deformation under the effect of an applied force with constant value) ?

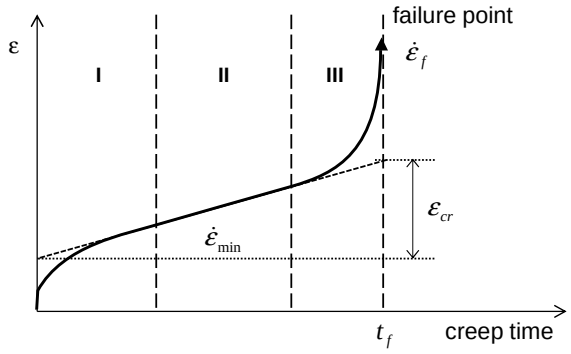


Figure III.6 – Schematic representation of typical three creep stages in creep curves for (thermoplastic) polymers and critical plastic strain. The failure point corresponds with the moment when the creep rate is maximal (initiation of failure).

[29] In this chapter and next ones, concepts of "breakage mode" and "failure pattern/mode" are used. A rigorous distinction is not always made between both terms in literature, especially when applied to multi-materials, composite products or structures. To get a more accurate picture of what these terms are exactly referring to, description of macroscopic failure has to be related to the amount of stretch (strain) before initiation of breakage (leading possibly to failure by excessive deformation) and to the crack propagation pattern, namely at which rate this occurs and whether this is stable or unstable.

Intrinsic creep curves (obtained for an homogeneous state of deformation from uniaxial compressive tests performed under constant true stress) show a similar shape as a macroscopic creep curve (for instance obtained by a uniaxial tensile configuration), with a succession of a low creep rate region (corresponding to a minimal value of creep rate), strain softening (increase of creep rate) and strain hardening (decrease of creep rate) (Klompen 2005). Visser and Klompen report that an equivalency of material state has been previously established between the region of low creep rate, or secondary creep, and the yield point on a corresponding intrinsic curve at constant strain rate. It implies that the equations of plastic flow apply to the secondary creep rate, predicting its stress and temperature dependence. This explains why the yield stress is assimilated as a measure of the resistance against plastic deformation, or resistance to creep.

This equivalency has been successfully applied to predict the resistance to creep (time to failure under creep load) of some thermorheological simple polymers (Klompen 2005; Visser 2010)[30], and relies on following relation (Visser 2010) :

$$\frac{t_f(\sigma_1)}{t_f(\sigma_2)} = \frac{\dot{\varepsilon}_f(\sigma_2)}{\dot{\varepsilon}_f(\sigma_1)} = \frac{\dot{\varepsilon}_{\min}(\sigma_2)}{\dot{\varepsilon}_{\min}(\sigma_1)} \tag{III.13}$$

where the subscript f refers to the failure point, defined as the point of the (intrinsic) creep curve where the creep rate reaches its maximal value, and $\dot{\varepsilon}_{\min}$ is the secondary creep rate. Typical order of magnitude of the ratio $\dot{\varepsilon}_f / \dot{\varepsilon}_{\min}$ for solid polymers ($T < T_g$) is in a range about 100 to 10000 (Klompen 2005; Visser 2010). The first and the second equalities arose from experimental observations[31].

A derived relation defines a constant result to the product between the time-to-failure and the secondary creep rate :

$$t_f(\sigma).\dot{\varepsilon}_{\min}(\sigma) = \varepsilon_{cr} \quad \text{or} \quad t_f(\sigma) = \frac{\varepsilon_{cr}}{\dot{\varepsilon}_{\min}(\sigma)} \tag{III.14}$$

where ε_{cr} is named the *critical plastic strain* (van der Vegt and Govaert 2003). The so-defined critical plastic strain appears effectively as relatively independent of temperature and, to a lesser extent, of the applied stress (van der Vegt and Govaert 2003).

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[30] This approach considers creep deformation as irreversible (assimilated with a propagation of micro-damage, as in plasticity of metals). It does not account for the observed capacity of large strain creep recovery after removal of the applied load (van der Vegt and Govaert 2003).

[31] However no clear indication has been found about the limits of validity of these two equalities, nor how to define these.

Visser calibrates its value by comparing results of tests conducted at constant strain rate with creep test results, performed at the same temperature (Visser 2010). However, the critical plastic strain is smaller than the effective tear strain (namely the total accumulated strain at the moment of breakage, or ultimate strain), and rather corresponds to the contribution of the secondary creep to the overall creep deformation (Figure III.6). Generalized to multi-axial deformation patterns, it is expressed in terms of a critical equivalent strain (Visser 2010).

An expression of the secondary creep rate of the form $\dot{\varepsilon}(\sigma,T)$ is obtained, either by deriving an analytical expression from equation (III.6) (Visser 2010), either by means of a numerical inversion of equation (III.11) (van Erp et al. 2012). Used in above equation (III.14), it gives an expression of the time-to-failure in function of the temperature and the applied constant value of stress.

For a thermorheological simple model, an analytical expression is obtained (by combination of equations (III.6) and (III.14)) :

$$t_f(\sigma,T) = \frac{\varepsilon_{cr}}{\dot{\varepsilon}(\sigma,T)} = \frac{\varepsilon_{cr}}{\dot{\varepsilon}_0}.\exp\left(\frac{\Delta U}{R.T}\right).\left(\sinh\left(\frac{\sigma.V^*}{k.T}\right)\right)^{-1} \quad \text{(III.15)}$$

$$= \frac{2.\varepsilon_{cr}}{\dot{\varepsilon}_0}.\exp\left(\frac{\Delta U}{R.T} - \frac{\sigma.V^*}{k.T}\right) \text{ if } \sigma >> \sigma_0 \quad \text{(III.16)}$$

The latter expression describes a straight line on a semi-log plot of the applied stress (creep stress) against (the logarithm of) the time-to-failure (with respect to the failure point as defined above), with a slope equal to $-k.T/V^*$, namely the opposite of the 'slope' in equation (III.4). Creep test results performed at different temperatures appear then as (almost) parallel straight lines.

Use of the obtained expressions in practice is limited by two aspects, which are the topic of the two next paragraphs : the first limitation is related to the phenomenon of physical ageing and mechanical rejuvenation, which affect polymer materials in their glassy state (thus when $T < T_g$); the second one is related to macroscopic failure modes.

III.2.3. Physical ageing and its effect on mechanical behaviour

A particularity of solid polymers, already mentioned here above, is that they are not in a thermodynamic equilibrium below their glass transition temperature. This induces a specific time-temperature dependent phenomenon in glassy state of amorphous phase, referred as *physical ageing*. The main characteristic of physical ageing is to be thermally *reversible*. An extensive review of this phenomenon and related experimental aspects is proposed in (Hutchinson 1995).

Looking back to Figure III.3, the amorphous glassy phase appears to occupy a larger volume than the crystalline one : the difference of volume between the two phases is called “free volume” (Hutchinson 1995; van der Vegt 2006). As a result of the lack of thermodynamic equilibrium below T_g , the free volume continues to decrease over time, at a rate depending on the segmental mobility, namely on the ambient temperature : this is referred as *volume retardation*. Whereas the corresponding variations of volume are very small, some mechanical properties appear highly sensitive to these, with the yield stress and thus the resistance to creep in first line. The ‘conformational changes’ involved in physical ageing are however not limited to volume retardation, and seem still not univocally identified (Hutchinson 1995), but are not related to any chemical degradation or transfer phenomenon. Accordingly, in a temperature range below the glass transition temperature, the state of a polymer material keeps changing in time, at a rate depending on the temperature and related material properties, named *ageing kinetics*. Below a certain temperature however, the segmental mobility of the molecular chains is getting too low to attain an equilibrium on a reasonable time scale : below $T_g - 25°\mathrm{C}$ is ageing likely to proceed over thousands of years (van der Vegt and Govaert 2003), and the thermodynamic equilibrium is in practice usually not attained for temperature smaller than $T_g - 15°\mathrm{C}$ (Hutchinson 1995).

When the temperature range in service conditions lies far enough below T_g , the initial ageing state of polymer products is mainly determined by the processing conditions, in function of the cooling rate from T_g . A “quenched” grade is obtained by fast cooling rate while an “annealed” one results of a slow cooling rate, or alternatively of an annealing treatment. The latter consists of an exposure to a temperature $T_a < T_g$ for a duration t_a , which accelerates the ageing process, thus increases the physical ageing state. The yield stress appears to increase with physical ageing; accordingly, the yield stress of a quenched material is expected to be lower than an annealed one. Conversely, an exposure to a temperature $T > T_g$ induces a *thermal rejuvenation* process.

Physical ageing seems thus a favourable effect as it increases the yield stress; however, there is also a potential counterpart in the form of a loss of ductility and a possible embrittlement at the macroscopic level as consequence. In fact, the rise of the yield stress is generally accompanied by a rise of the consecutive yield drop, and this corresponds to a reduction of the (macroscopic) ductility; in comparison, the large strain response seems not affected (van der Vegt and Govaert 2003).

The application of a constant stress is having a similar effect on the yield stress, namely by increasing the segmental mobility; therefore, *mechanical rejuvenation* is associated similarly to a change of physical ageing state. This effect is observed for both compressive and tensile stress states, but appears as less univocal than the effect of temperature (Hutchinson 1995; Meijer and Govaert 2005). It is not completely equivalent to thermal rejuvenation as mechanical

rejuvenation seems not always accompanied by a free volume expansion (Hutchinson 1995; van der Vegt and Govaert 2003); it seems however still a field of much scientific debate (Meijer and Govaert 2005). One explanation is that at yield, when segmental mobility is activated, the action of stresses leads to an orientation of the molecular chains, which activates the strain hardening mechanism (also called mechanical enhancement when used as a process treatment). The interaction between thermal and mechanical effects on yield stress is somehow visible in the expression (III.16) here above : the rejuvenation effect of applied stress only occurs when the applied stress is large enough. For a thermorheological complex material, the effect of stress on ageing state is less univocal, as the balance between temperature and stress effects on ageing process can be different for the two different relaxation mechanisms (Klompen 2005).

Due to the lack of completely established relation between thermodynamic variables and mechanical properties, the physical ageing state *S* is generally quantified by a measure of the yield stress from a well-adapted short-duration test (Hutchinson 1995; Klompen 2005). The value of the parameter *S* is depending on the choice of the reference value $\sigma_{rej,0}$ (equation (III.1)) at a given temperature, as it appears not possible to obtain experimentally a completely "fresh" (non-aged) material by thermal rejuvenation process. Information about the initial ageing state is implicitly included in the pre-exponential term $\dot{\varepsilon}_0$, or alternatively in the zero-shear viscosity η_0 (Klompen 2005; Meijer and Govaert 2005).

Equations of previous paragraph apply thus only to tests of short duration, meaning that no (significant) change of physical ageing state occurs (up to yield), namely that the pre-exponential term $\dot{\varepsilon}_0$ or η_0 remains constant for the considered loading duration. This condition is fulfilled when the initial ageing time t_a is much longer than the duration of the loading t (or an equivalent duration accounting for a test temperature T different from the ageing temperature T_a). When this condition is not met, *progressive physical ageing* occurs during the loading duration, increasing the resistance against plastic deformations. In particular, progressive ageing reduces the creep rate during creep tests of long duration, in comparison with the one measured with tests of short duration (for materials with the same initial ageing state)[32]. This leads to an apparent *endurance limit*[33] in creep test results : below a certain value of creep load, no failure is attained (on reasonable timescales), because the time-to-failure

[32] Besides possible occurrence of progressive physical ageing during long duration creep, a failure criterion due to excessive deformation combined with the non-linear response to loading above the characteristic stress is another reason why prediction of long-term creep based on a simple time-temperature shift function calibrated on results of short-duration tests is not reliable. This invalidates among others the use of the Burger model, a series combination of a Kevin-Voigt and a Maxwell linear elements, for modelling the creep response quantitatively (van der Vegt 2006).

[33] There is not an absolute value of endurance limit for a polymer material, neither a lower yield stress : such lower limits can only be identified in relation to a limited scope (Visser 2010).

increases towards much longer (thus safer) values than the values extrapolated from short-duration tests. However, a risk then exists, for long duration loading, of a transition in (macroscopic) failure modes, from ductile to brittle failure (Klompen 2005).

Influence of initial ageing state and progressive physical ageing is modelled by replacing the constant value of the pre-exponential factor in the viscoplastic criterion by a (ageing) time-dependent function. For instance, the constant η_0 in equation (III.8) is replaced by a function of the type (Klompen 2005) :

$$\eta_0\left(T_{ref}\right) \rightarrow \eta_0\left(T_{ref}, t\right) = \eta_{0,r}\left(T_{ref}\right).\exp\left(S_a\left(T_{ref}, t_a\right)\right) \tag{III.17}$$

where $\eta_{0,r}$ corresponds to the zero-shear viscosity of a fully rejuvenated material (at a reference temperature), and $S_a\left(t_a\right)$ is a state function determining the initial ageing state due to the thermomechanical history (for instance an annealing treatment). The same function is used for modelling the effect of progressive physical ageing during a long-duration test or in service conditions (Klompen 2005; Visser 2010), by splitting its time argument into an initial age and an "effective ageing time". This effective ageing time expresses the increase of physical ageing state *S* during a long-term loading (progressive physical ageing), for given conditions of temperature. Parameters involved in the ageing state function are therefore associated to *ageing kinetics*.

To get a more concrete idea about the trend predicted by such an ageing state function and an order of magnitude of the effect induced on the yield stress (corresponding to experimentally measured values), the next equivalent formulation (Klompen 2005) is probably more intuitive :

$$\sigma_y = \sigma_{y,0} + c.\log\left(\frac{t + t_a}{t_0}\right) \tag{III.18}$$

By comparing this expression with equation (III.1), the two terms can be recognized as the two components of the driving stress evaluated at the yield point (where the hardening stress is considered equal to zero).

Figure III.7 shows two situations where a progressive physical ageing effect is present. The first graph (on the left) shows a series of uniaxial tensile tests results performed in reference test conditions on specimens with different initial ageing states, which are used to determine ageing parameters (Klompen 2005); the second shows the effect of progressive physical ageing on results of creep tests

performed on two different grades of a thermoplastic product[34] (Visser 2010). According to the used experimental approach, the ageing state function is fitted by means of the two or three[35] parameters of above expressions.

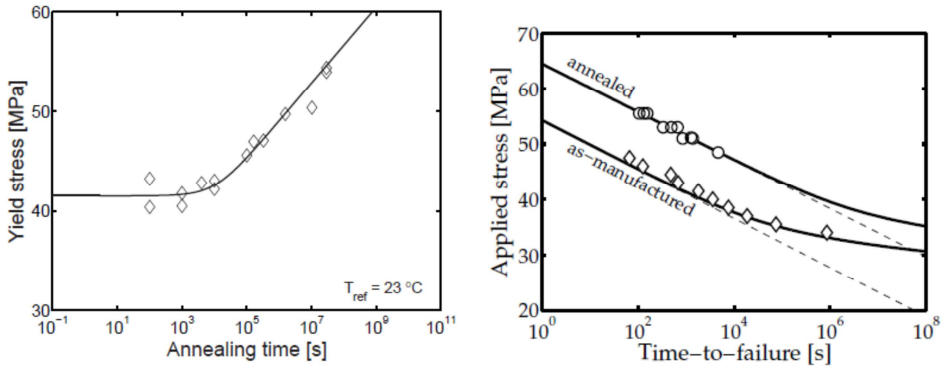

Figure III.7 – Application of the ageing function to two different situations a) effect of different initial ages (annealing times) on the measured yield stress in reference testing conditions (Klompen 2005) b) creep tests results compared to models without (dotted lines) and with (solid lines) progressive physical ageing (for annealed and as-manufacturers grades of uPVC tested at different creep load values) (Visser 2010)

Finally, it is suspected that the ageing kinetics can be altered by the confinement grade of the material for some configuration, say its deformation degree of freedom. This comes from an analogy with a given explanation for the thermorheological complex behaviour of semi-crystalline polymers. The presence of the crystalline phase would induce two different contributions of the amorphous phase, one due to the volume part free to deform, and the other one corresponding to zones constrained by the presence of neighbouring crystallites; leading to a different relaxation time for each (van der Vegt and Govaert 2003).

In summary, it appears that physical ageing is an effect potentially important to account for. In particular, it can represent a non-negligible constraint in designing experimental campaigns and in interpreting test results in some test conditions.

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[34] Consider a quenched and an annealed grade of the same polymer : the first has a 'lower level' of physical ageing state than the second, or a smaller initial age. If a creep load is applied on these two materials in the same conditions, progressive physical ageing will start earlier in the quenched grade than in the annealed one; in Figure III.7b, the as-manufactured grade corresponds with a quenched grade.

[35] The third parameter is the initial ageing state when this is unknown, represented for instance by the initial ageing time and the rejuvenated yield stress. However, practically, most of the time the initial age cannot be measured directly, and the values obtained by fitting experimental data include thus an arbitrary or virtual part. However, it allows to make quantitative predictions in regard to an arbitrary fixed reference state. There are tricks and issues how to perform such calibrations which are beyond the purpose of the current discussion…

III.2.4. From intrinsic to macroscopic behaviour

The specificities about the behaviour of polymer materials have been approached so far by considering generalities and the so-called intrinsic behaviour, associated to the response of a material in a homogeneous state of deformation and under a load applied at a constant strain rate. The macroscopic behaviour of a polymer material appears however to be slightly different. In this paragraph two aspects related to the macroscopic behaviour are reviewed and discussed. Firstly, some attention is dedicated to the conventional uniaxial tensile test configuration and arising issues for interpreting its results. In fact, this is still the reference test configuration often envisaged for determining the mechanical properties of a ductile material with capacity to carry loading in tension, it is the topic of a variety of standardized methods for metals and polymer materials, and it is a relative cheap test method. Among others, this test configuration is often considered for investigating the properties of interlayer films (see further in paragraph III.3.2). The second aspect addresses more particularly the macroscopic creep behaviour and the related specific failure modes, which did not appear above. In fact, quasi-static response under constant force has been identified in Chapter II section II.1 as an important loading mode for fractured laminated glass used in structural applications.

A conventional uniaxial tensile test on a polymer material is typically performed on a dog-bone specimen at a constant velocity (displacement rate) [36]. The typical response to such a test, expressed as a nominal stress-strain curve, exhibits a shape slightly different from the intrinsic curve. This is firstly due to the large deformation range, and secondly to often non-homogeneous deformations. The combination of these two particularities of the mechanical behaviour of polymers explains difficulties of interpretation of results of conventional uniaxial tensile tests (Meijer and Govaert 2005; Moore and Turner 2001; van der Vegt and Govaert 2003). In particular, results of conventional uniaxial tensile test on polymer materials do not allow to derive back an intrinsic stress-strain curve, whereas the latter allows to predict the response of an uniaxial tensile test (Meijer and Govaert 2005).

[36] Conventional uniaxial tensile tests typically measure the deformation as the variation of a prismatic gauge length, namely the length between two marked sections. Reference test methods for polymer materials are for instance the ones prescribed in international test standards ISO 527, and are generally performed on a dogbone specimen (see also paragraph III.3.2), this for avoiding secondary effects or breakage near the clamping areas. The qualifying of 'conventional' is used among others by Moore and Turner (Moore and Turner 2001).

This last statement essentially applies to specimens tested at a temperature below their glass-rubber transition : non-homogeneous response is essentially associated to the apparition of necking during the test (see Figure III.8)[37]. When tested at a temperature above their glass transition, the response is rubber-like, with little or no localization (no necking) and thus with (reasonably) homogeneous deformation along the gauge length. In that case, provided that the assumption of isochoric deformations[38] is valid, some hyperelastic models can be calibrated only by means of uniaxial tensile test results; when homogeneous non-isochoric deformations are involved, existing alternative calibration methods require advanced measurement methods (as the measurement of lateral contraction) or complementary tests on other configurations[39]. By definition, hyperelastic models cannot cope with a stress drop[40] in macroscopic loading curves[41].

An apparent softening in the form of a stress drop on a loading curve of an uniaxial tensile test (Figure III.8b) is not necessarily the consequence of an intrinsic material softening, or yield drop (Figure III.8a and Figure III.2); it can solely be due to a geometric softening caused by the apparition of the necking zone along the prismatic gauge length and the consequent reduction of the cross-section area. The neck initiation is however a direct consequence of the intrinsic yield stress, and can be followed by two types of response, or failure modes. The stress localization caused by the corresponding diminution of the cross-section's area can either be stopped by strain hardening in the necked zone, with as consequence a ductile behaviour caused by the extension ("flow") of the necking zone along the gauge length (as illustrated in Figure III.8); or it cannot, and it leads then to critical localization up to crack initiation and breakage of the cross-section, generally associated with a brittle failure (van der Vegt and Govaert 2003).

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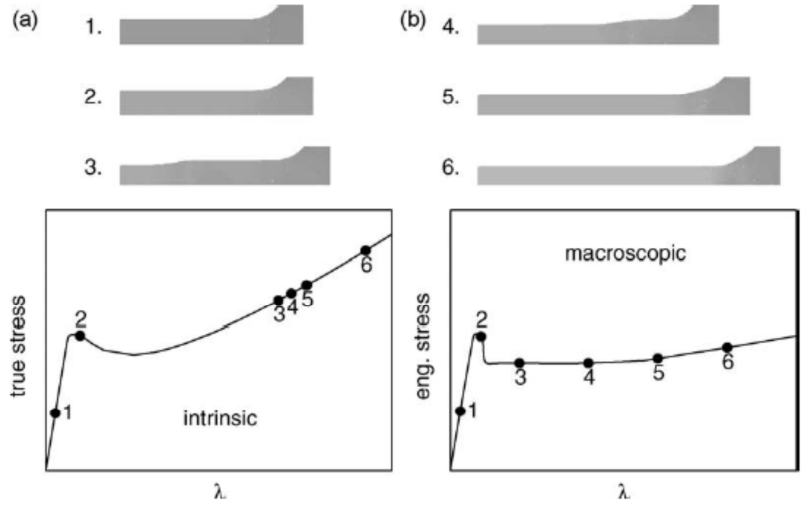
[37] The Poisson's coefficient, which expresses the volume dependence of the deformations, has a value around $\nu = 0.3 \,..\, 0.4$ for the elastic pre-yield behaviour of glassy polymers in general (corresponds to volume expansion in a uniaxial tensile test), and deformations at yield and in the post-yield range occur without noticeable volume change ($\nu \sim 0.5$), as for large strain rubber-elasticity (Moore and Turner 2001; van der Vegt and Govaert 2003). The Poisson's coefficient is originally defined in small strains theories (in the scope of linear elasticity in solid mechanics), it is therefore questionable whether it is not inducing confusion to use it for describing volume variations in the large strain range (in spite of a usual practice…).

[38] Isochoric deformations are deformations occurring without global change of volume.

[39] Such calibration methods for hyperelastic models are implemented in some Finite Element packages as Abaqus (Simulia).

[40] Stress drop is defined here as the difference between a upper value of (nominal) stress and a consecutive lower value on the loading curve of a conventional uniaxial tensile test.

[41] The Mullins effect, included in some hyperelastic models, is sometimes associated with a concept of visco-elastic strain softening : however, this rather accounts for differences between the loading and unloading paths for some types of rubbers, and is not related to yield drop.



Note: identified points on the intrinsic curve (left) correspond to the stress state in the central cross-section.

Figure III.8 – Comparison of intrinsic loading curve and macroscopic response (conventional uniaxial tensile test) of polycarbonate (PC) (Meijer and Govaert 2005)

However, the perception about the brittle or ductile nature of the failure may vary according to the loading mode and characteristics corresponding to each part of the two curves. The typical response of a material in a uniaxial tensile test configuration to two different loading modes is shown in Figure III.9 : a creep curve, obtained by the application of a constant force (constant *nominal* stress), is compared to the previously considered loading curve, obtained for a test carried at constant strain rate (namely at a constant *nominal* strain rate, or constant displacement rate). In fact, the effective (true) axial stress in any cross-section is rising with its area reduction consecutive to stretching.

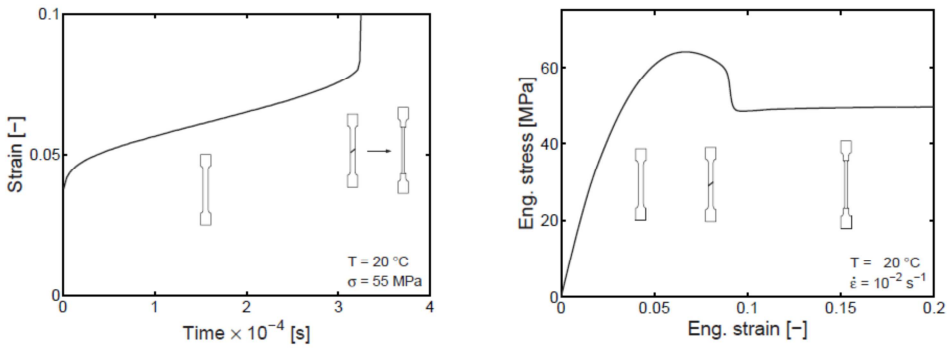


Figure III.9 – Uniaxial tensile test configuration : comparison of the response at room temperature of a specimen polycarbonate to a creep load (creep curve, left) and to a constant strain rate (nominal stress-strain curve, right) (Klompen 2005)

From the comparison of the two curves in Figure III.9, it seems that the apparent progressive ductile post-yield response during a test at constant strain rate rather corresponds to a relative sudden failure in the creep load mode : the neck initiation is followed by an unstable deformation mode, whether it is explained as a tertiary creep mode or a unstable crack propagation mode[42]. Accordingly, the ductile character of the failure seems different in the two loading modes.

Considering the creep loading mode, the limit between a stable (secondary) creep and an unstable (tertiary) creep regime is defined by a criterion comparing the accumulated plastic strain in the material with the critical equivalent strain (Visser 2010). When the first is getting larger than the latter in some point of the loaded volume, it initiates locally a plastic flow, which corresponds to a localization phenomenon, and for a creep load mode to a fatal failure. However, this localization phenomenon is not always visible at the macroscopic level; it can also take the form of localized, microscopic damages, as crazes, etc. (Moore and Turner 2001; van der Vegt 2006).

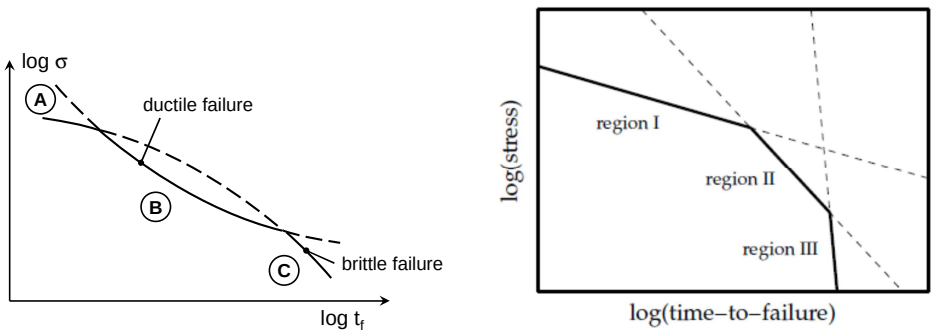


Figure III.10 – Typical failure modes of thermoplastic products under creep load
a) for (thermoplastic) polymers in general – based on (van der Vegt 2006)
b) particular cases of plastic pipes subjected to constant internal pressure (Visser 2010) : region I corresponds to a ductile failure mode (B), region II to a brittle failure mode due to local failure (hairline crack) (C), and region III to a brittle failure mode due to chemical degradation (multiple cracks)

When looking at the creep response on a larger scope of loading conditions, the general qualitative picture for solid thermoplastics[43] identifies a range of ductile failure mode, surrounded by two ranges of brittle failure modes (Figure III.10).

This general curve is obviously also temperature dependent, and this appears clearly by looking in parallel to the definitions of some characteristic temperatures given in paragraph III.2.2 here above. The ductile-to-brittle

[42] The notion of unstable propagation mode has been defined in Chapter II paragraph II.4.

[43] For services conditions in a temperature range sufficiently 'far' below the glass transition.

transition temperature refers to the point between segments A and B in Figure III.10a, the maximum temperature in service conditions refers to the region III in Figure III.10b. Different methods are reported to represent the measured stress-temperature dependent time-to-failure, and to extrapolate the results to longer creep load durations; an important point to notice is that such experimental investigation method is very time-consuming (van der Vegt 2006).

Besides all the above identified aspects making the interpretation of tests on polymer specimens difficult, in particular for thermoplastics, a complementary aspect is pointed out. The apparent ductility (or toughness) of a polymer material may seem larger when observed on smaller test specimens compared to larger elements. Alternatively, the probability of a brittle response increases with the size of the element. The phenomenon is explained by comparing the size of plastic zones (among others where crack propagation mechanism are present) to the dimensions of the specimen (Moore and Turner 2001).

In summary, many aspects are intervening for explaining why the apparent macroscopic ductility and resistance of a polymer component can be significantly dependent on the considered test configuration and test conditions. The main consequence is that any attempt at making an extensive characterization of all the mechanical properties of polymer components, taking into account all aspects of importance, inevitably tends to large, time consuming experimental programs, with possible unreasonable cost/benefit balance as consequence (Moore and Turner 2001; van der Vegt 2006). Characterization of the creep resistance of load-bearing components in particular is requiring many tests at different temperatures and creep load values, and is therefore only affordable for large scale applications with high safety requirements, as for polymer pipes used in gas distribution network (Visser 2010). For other structural applications, critical creep deformations is generally the design criterion (van der Vegt and Govaert 2003).

Another identified important related issue in the case of polymer components used as or in construction products, is to find appropriate way(s) to give a *comprehensive overview* of their mechanical performances to the user-designer.

III.2.5. Particularities of polymers used as adhesives

The structural role of an interlayer component in laminated glass has been described in Chapter II. It can be considered as a particular case of structural adhesive[44], with a particularity : an optimal contribution of the interlayer to the overall performances requires an appropriate balance between its intrinsic cohesive (bulk) properties and its adhesive properties. In particular, the

[44] According to the definition in standard EN 923 dedicated to the defined terms for adhesives : "*Adhesive : non-metallic substance capable of joining materials by surface bonding (adhesion), and the bond possessing adequate internal strength (cohesion)*"

determination and control of the adhesion level has to consider lower and upper limits. In fact, the ductility of the interlayer ligament in a TCT-configuration is not solely depending on its bulk properties, but depends largely on its delamination capacity of the glass substrates.

In what extent are the different particularities of polymers, and the related concepts and models presented above, applicable to adhesive configurations ?

It has been shown that the mechanical response of polymers is mainly ruled by secondary intermolecular bonds. Accordingly, it seems logical to firstly look at the corresponding mechanisms involved in the adhesive properties of polymer components.

Adhesion in general can rely on different mechanisms according to the nature of the adhesive and of the substrates, and to the characteristics of the surfaces assembled (Belis et al. 2011). It can involve mechanical interlocking, diffusion and adsorption mechanisms. These mechanisms are however generally complementary, and correspond also to different theories of adhesion. Mechanical adhesion is mainly due to the geometry of the assembled surfaces, characterized by their respective roughness. The associated feature of the adhesive is the viscosity, measuring its capacity to fill the gaps. Diffusion or adsorption mechanisms are associated to bonds at a smaller, molecular scale. The former is assuming some penetration of the molecular chains of the adhesive polymer into the substrates, whereas the latter is rather considering 'pure' interfacial interaction forces. In both cases the involved interaction forces are of a lower or a similar order of magnitude than the ones involved in the intermolecular bonds in the bulk of the adhesive component.

For explaining how temperature can affect on a differentiated way cohesive and adhesive interaction forces, a conceptual distinction between the various adhesion mechanisms should probably be related to a distinction between the nature of the interfacial forces at molecular level (in comparison with the cohesive intermolecular forces in the bulk) and their density along the contact surface (interface). If the nature of the adhesive bonds is close to the one of the cohesive bonds, it seems logic that their sensitivity to time-temperature effects will be close to each other's as well, and that the balance between stretching and delamination rates can be kept over a wider range of temperatures, even if the absolute value of the related performance varies. Besides, as explained in Chapter II section II.4, stress patterns along interfaces between two materials and consecutive acting forces ruling interfacial crack propagation processes are depending on the elastic properties of surrounding layers. Accordingly, if one of the two materials in contact exhibit a mechanical behaviour sensitive to time and temperature effects, interfacial behaviour and its contribution to structural behaviour of adhesive assemblies inevitably exhibits a dependence to similar effects.

Among the similar features for all adhesive products, it seems that they are always processed in an adhesive assembly at a temperature above their glass transition temperature. The main difference between (physically entangled) thermoplastic and (chemically cross-linked) elastomer adhesives is the position of their (primary) glass transition temperature compared to the services conditions. Thermoplastic adhesives have a glass transition temperature above or near the ambient temperature, and their lamination process necessarily requires larger temperatures for bringing them in a state of low viscosity. In contrast, elastomer adhesives have a glass transition temperature below the service range, and are processed at ambient temperature or at a slightly larger temperature but still inside the possible temperature range of their service conditions.

It is obvious that the industry of adhesive products has developed in the last years much knowledge about how to perform differentiated control of bulk and adhesive properties, and that a variety of means is used to achieve it, which vary for each product and application. However, current design issues are rather addressing the definition of limits of use, namely the identification of boundaries of possible application scope, and thus address their performances in a wider range of service conditions.

For a polymer bulk material, two types of limits have been identified : a loss of performances involving *physical and reversible* phenomena and a loss of performances due to *chemical degradation mechanisms*, namely involving irreversible damage of (primary) chemical bonds. Design criteria for thermoplastic products are mainly related to the first type, and for elastomers to the second type.

For adhesive assemblies in general, and for laminated products in particular, the issue is more complex, as the evaluated performances are related to preferred activation of failure mechanisms, among others related to preferred paths for crack propagation (which involves a post-yield behaviour). All the issues identified here above for the assessment of (thermoplastic) products are expected to be applicable for (thermoplastic) adhesive assemblies. Consequently, it can be expected that the characterization of the mechanical properties of an adhesive component has to account for possible differentiated effects of the processing conditions on its bulk and interfacial properties. Besides, the molecular mobility could be slightly different in the close vicinity of the interface compared to the one in the bulk of the adhesive layer. Finally, as it will appear in section III.3 below, it cannot be excluded in general that (de-)crystallisation processes are occurring in service conditions. Conceptually, all these effects can lead to some differences in relaxation and ageing kinetics in the bulk and along interfaces, namely with one or more different values of kinetic parameters. It is proposed to account for these possible differences by distinguishing conceptually two ageing state functions, $S_{a,I}(t)$ along the interfaces of the adhesive layer and $S_{a,B}(t)$ in

the bulk[45]. However, it seems logical to assume simultaneously that some correlation exist between these two functions.

Above considerations lead to formulate the design and the assessment problem by means of two complementary questions. The first addresses the assessment of the time-temperature dependent variation of the cohesive and adhesive properties, relative to each other and in absolute value, in a range of service conditions (temperature, loading rate or level,…). The second is related to the identification of for which conditions which of these values is leading to critical situations, and as a consequence which must be considered for the design (dimensioning). This distinction could correspond respectively to the assessment of product performances on the one hand, and to the evaluation of their compliance for design purpose on the other hand, and combine thus two assessment logics, the first product-oriented, the other application-oriented, where the performance requirements defined for an application (construction element) must be fulfilled in relation to the assessed performances of the construction product. Strong interactions are inevitably required between these two approaches, and thus also between the various involved stakeholders (Kooymans and Schneider 2009), to facilitate the use of this type of products in the construction sector. These interactions concern in first instance issues related to the choice of experimental approaches, the type of test configurations, scales and test conditions, the amount of tests and their execution order.

III.3. Consequences for the characterization of interlayers

Mechanical models and corresponding characteristic properties presented in previous sections here above have been mainly developed for solid thermoplastic products, namely products with a glass transition temperature significantly above temperatures in service conditions. For instance, Klompen considered mainly polycarbonate (PC) and polymethylmethacrylate (PMMA) products in a relative general, fundamental approach (Klompen 2005), while Visser considered mainly polycarbonate (PC) and unplasticised poly(vinyl chloride) (uPVC) as polymer products used for water and gas distribution pipes in the Netherlands, in a more practically, engineering oriented approach (Visser 2010).

The question is whether these models and associated experimental approaches are usable for characterizing interlayer products. In fact, two complementary questions can be distinguished. Firstly, are the models *a priori* robust enough for covering the intended application scope(s), in terms of type of materials, temperature and loading ranges, and in terms of service conditions, including

[45] For interlayer products rather of the elastomer type (cross-linked structure), as cast-in-place products processed in liquid form at lower or ambient temperature and hardened after lamination, these ageing states functions should be completed (or replaced) by similar state functions reflecting the time-temperature dependence of hardening processes.

ageing issues ? Secondly, assuming they are, are the proposed and developed experimental approaches applicable and relevant from a scientific point of view, but also are these viable from a practical (economic…) point of view ?

These questions are addressed in four steps in the following paragraphs, and will focus on two families of interlayer products principally. In a first step, their main characteristics are presented with regard to temperature ranges in service conditions for laminated glass products, and compared with other polymer materials used in structural applications. The second step discusses some interpretation issues of values of mechanical properties for interlayer materials, and in particular with regard to results of conventional uniaxial tensile tests. The third aspect addresses shortly some specificity of adhesion of interlayer materials with glass, and the related methods for measuring the adhesion level. Because of the identified pitfalls and issues with conventional test methods and experimental approaches for characterizing the mechanical behaviour and adhesive properties of interlayer materials, the fourth step looks for possible alternative approaches and test configurations.

III.3.1. Characteristic properties and service conditions

PVB refers to a broad family of products which act as reference material for laminated safety glass products used in the building industry. They are produced by different multi-national companies in different grades, which are adapted to comply with specific markets or applications. Let us mention a few ones among the most known brands for the building industry : the Butacite® produced by DuPont[46], the Saflex® by Solutia (Eastman group, previously Monsanto) and Trosifol® (Kuraray group). The second product is the SentryGlas (SG), which is the commercial name for a product described as an ionomer, and mainly known as a product "stiffer and stronger than PVB" (Stelzer 2010).

These two products belong to the category of thermoplastic polymers and are manufactured as folio interlayers, and produced in a few different standard thicknesses. The thickness of PVB-films follows an implicit international standard based on multiple values of 0.38 mm (15 mil). In laminated glass units for structural applications, thicknesses of 0.76 and 1.52 mm (30 and 60 mil respectively) are the most used. They are generally delivered in rolls of various widths. SG-interlayer was initially available in the form of thicker rigid sheets of 1.52 and 2.28 mm; more recently the assortment has been enriched with a thickness grade of 0.89 mm delivered in rolls. There exist nowadays however a variety of other interlayer products, and among these some are associated to the family of elastomers (typically cast-in-place type of interlayers).

[46] The division Glass Laminating Solutions (GLS) of DuPont has been bought in November 2013 by the Kuraray group.

Both PVB- and SG-laminates are processed by means of an autoclave process, characterized by a pressure-temperature controlled cycle of a few hours, during which the temperature is typically raised at a temperature of about 140°C and cooled down at controlled temperature and pressure decrease rates (Tupý et al. 2013).

A first comparison is made in Figure III.11, where characteristic values of transition and processing temperatures of different polymer products are compared with identified typical temperature ranges in service conditions. Three categories of products are considered :

1) *underground pipes* (for water and gas distribution networks), because it is one of the most important applications, in the civil engineering field, where polymer products are used as structural elements (van der Vegt and Govaert 2003), for which mechanical models presented earlier in this chapter are applied for dimensioning the elements and for controlling their effective residual lifetime duration during their design working life (Visser 2010). The product types are polycarbonate (PC) and unplasticised poly(vinyl chloride) (uPVC), and the corresponding characteristic values of transition temperature are the ones considered by Klompen and Visser in their respective work;

2) *glazing products* made of transparent solid plastics, used as isolated elements or as glazing sheets within laminated glass products (namely with a structural role similar to a glass component). Interestingly, most used polymer products used as glazing sheets are transparent grades of PC and of PMMA materials. It is thus assumed that their characteristic values of transition temperatures remain in the same order of magnitude as the ones used for pipes products; the validity of this assumption is supported by values found in literature, as for instance in (Bos and Veer 2007; Bos et al. 2006)[47];

3) *interlayer products*, here limited to PVB and SG products. PVB is referring to a family of products, provided by different manufacturers in various grades, whereas SG refers to one commercial product grade : this explains the wider range of values mentioned for the glass transition temperature of PVB in this figure. It corresponds to the range of values mentioned by Hooper (Hooper et al. 2012) and is thus relative to various grades and manufacturers : it must not be assimilated to an intrinsically larger scattering of this property for a specific PVB-product.

[47] Some authors also report on the use of PC as an interlayer component, namely when the adhesion with the glass sheets is not made by means of other, softer polymer interlayers (Veer et al. 2001). In spite of the few details given about the production process, probably the corresponding lamination process is performed at a temperature above the glass transition temperature of PC, namely above 150°C. The structural role of a PC layer is in such case slightly different, with possible consequences about eligible approaches for characterizing its mechanical properties.

The represented glass-rubber transition ranges for each family of products correspond to a temperature range equal to $\left]T_g - 25°C\,;\,T_g\right]$[48], namely the temperature range in which the physical ageing state of the polymer product is supposed to evolve at the fastest rate. The processing temperature for laminated glass components corresponds to the highest temperature reached during the lamination process. The value of the reference test temperature T_0 is equal to 20 or 23°C in usual standard test conditions.

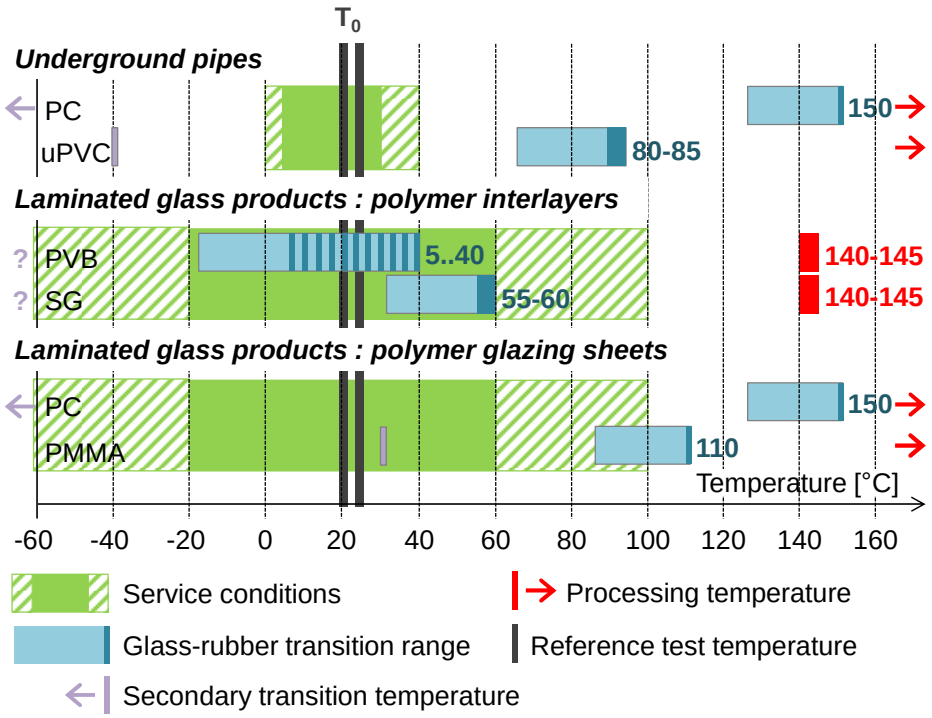


Figure III.11 – Comparison of typical temperature ranges for service conditions of end products with values of transition and processing temperatures of used polymer materials

Besides the characteristic values of transition temperatures for each product, temperature ranges corresponding to service conditions are represented, accounting for variable lower and upper limits. It is assumed that the range of service temperatures for underground pipes is smaller than for glazing products, because their underground position protects them of daily and seasonal cyclic variations of temperature. The service temperature considered for laminated glass applications corresponds to the temperature reached inside the considered component (interlayer or glazing); this one can be larger than the surrounding air

[48] In the graph, the typical value of T_g is mentioned on the right side of this interval. The origin of the value 25°C has been mentioned in paragraph III.2.2; it clearly remains a rather indicative value.

temperature due to daily and seasonal climatic variations the element is exposed to, the difference being due to absorption of solar radiation energy.

A few observations can be made on this basis. For considered service temperatures for underground plastic pipes made of considered polymer materials, thermal effects are expected to only increase the physical ageing state. In comparison, the physical ageing state of interlayer products seems to possibly be affected by thermal ageing *and* rejuvenating effect, because their glass transition temperature lies *within* the service temperature range.

Table III.2 – Indicative values of properties of interlayer products

Property	unit	PVB	SG
Volumetric weight (density)	kg/m³	1070	950
Elastic modulus*	N/mm²	18	300
Tensile strength* (σ_u)	N/mm²	> 20	34.5
Deformation at breakage* (ε_u)	%	> 250	400
Glass transition temperature T_g	°C	5 - 40°C	55..60°C
* Typical values obtained by means of a conventional uniaxial test on a dog-bone specimen, performed at moderate strain rate and room temperature; see also paragraph III.2.4 and Figure III.12.			

In literature, SG is generally described as a semi-crystalline thermoplastic (Louter 2011; Meissner and Sackmann 2006), while PVB is described as an amorphous (Bati, Fagone, & Ranocchiai, 2009; Meissner & Sackmann, 2006; Muralidhar, Jagota, Bennison, & Saigal, 2000) or semi-crystalline (Kott 2006; Weller et al. 2009) thermoplastic. However, this qualitative distinction does not seem a critical point to solve, for different reasons. On the one hand, the amorphous phase seems to play a dominant role on the time-dependent behaviour of semi-crystalline polymers; on the other hand, in regard to characteristic values of material transition temperatures, occurrence of crystallization processes could occur during lamination and/or service conditions. Besides, transparent polymers in general seem characterized by low crystallisation grade and small size of crystallized zones, as crystallization is known to be a cause of loss of transparency (van der Vegt 2006). The change of transparency of the material during the lamination process[49] could be thought of being due to change in crystallization

[49] Both PVB-films and SG-sheets are translucent in delivery conditions, and become transparent during the lamination process.

grade, but another advanced explanation is related to the surface roughness[50] of the interlayer sheets/films (Juang et al. 2001).

Thermal stability of some interlayer products against chemical degradation has been investigated (Weller et al. 2010), and the reported results indicate that with regard to identified typical temperature ranges in service conditions, PVB- and SG-laminates both seem to have a satisfying stability ($T_{\max} > 100°C$). PVB-interlayers seem more sensible than SG-interlayer to possible degradation processes during lamination, and this aspect is undoubtedly playing a role in the determination of the specifications for the autoclave cycle used in the lamination process.

III.3.2. Response of interlayers in uniaxial tensile tests

At ambient temperature, the observed behaviour of PVB and SG interlayer products appears as quite different of each other's : PVB exhibits a rather rubber-like response, while SG is described as having an 'elasto-plastic' behaviour by many authors. In particular, during a conventional uniaxial tensile test performed at room temperature, the deformations of a dog-bone specimen cut out of a PVB-film appear as relatively homogeneous, while the same test on a similar specimen from a SG-sheet clearly yields to the apparition and propagation of a necking along the specimen's gauge length (Figure III.12), which is the typical behaviour of a glassy polymer (similar to the one shown in Figure III.8).

Typical values of properties of these two types interlayers at ambient temperature are reproduced in Table III.2. Values of mechanical properties in this table come from uniaxial test results and should thus be merely considered as an indicative order of magnitude.

What about the influence of applied strain rate and temperature on results of such conventional uniaxial tensile test results ? Figure III.13 shows the temperature dependence of the response of PVB-specimens to conventional uniaxial tensile tests (fixed but unspecified specimen geometry and applied strain rate).

Other similar test results on PVB and SG-specimens, performed in different ranges of loading rates and of temperature, are reproduced in Figure III.15 to Figure III.18, with the corresponding specimen geometries in Figure III.14 (Belis et al. 2009; Hooper et al. 2012; Kott and Vogel 2003; Puller et al. 2011). Other similar test results on SG-specimens are reported in (Meissner and Sackmann 2006). The main characteristics of each test series are summarized in Table III.3, with a comparison of the corresponding loading ranges (the ratio of rates range is calculated on the basis of the largest and the smallest applied displacement rates).

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[50] Surface roughness of interlayer folio is known to be another important parameter for controlling the adhesion quality and the adhesion level, with regard to the evacuation of air bubbles.

A quantitative comparison of these different results is not straightforward, because of the different geometries of the test specimens. Whereas dispersion of test results within a series (Figure III.16 to Figure III.18) seems within reasonable limits, comparison of loading curves corresponding to in principle identical tests shows a noticeable difference in yield stress, with a still more noticeable difference between the post-yield part of the curves (between Figure III.17 and Figure III.18, tests on SG-specimens at 100 mm/min; difference of about a factor 1.3 between measured tensile strength). These differences can be explained by different experimental issues or differences in samples.

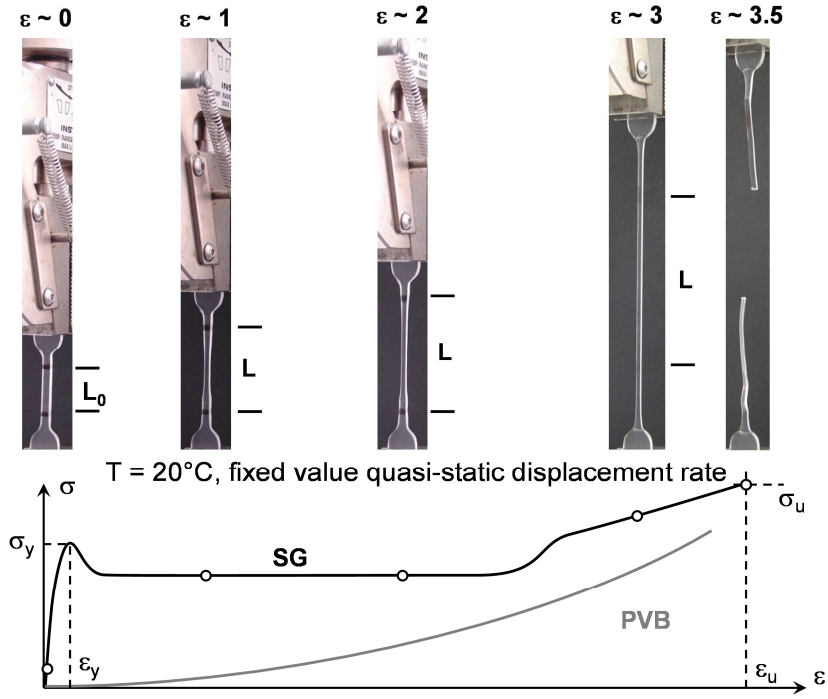

Figure III.12 – Typical nominal stress-strain curves obtained from conventional uniaxial tensile tests on dog-bone specimens cut out of PVB-films and SG-sheets. Above pictures show the deformation pattern along the loading curve for SG-specimen.

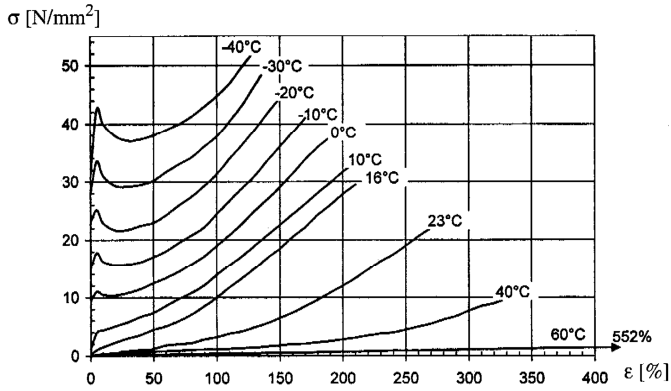

Figure III.13 – Influence of temperature on nominal stress-strain curves from uniaxial tensile tests on PVB-specimens (carried out at constant but unspecified displacement rate, and on unspecified geometry of the specimens). From technical data of Trosifol, as reproduced by Kott (Kott 2006)

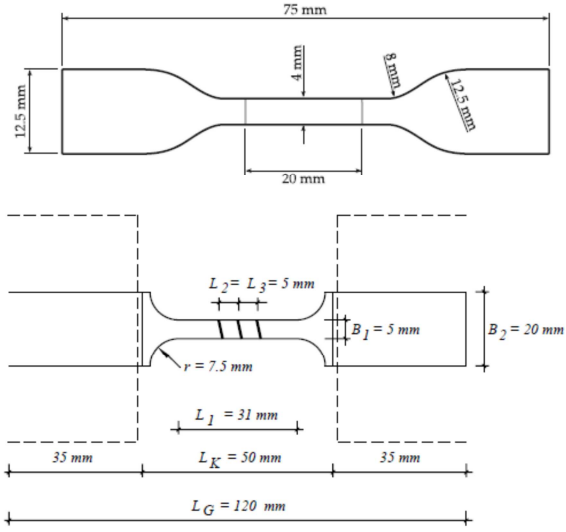

Figure III.14 – Dimensions of dog-bone specimens used for uniaxial tensile tests on interlayer film a) (above) according to EN ISO 527-2 (Belis et al. 2009; Hooper et al. 2012; Puller et al. 2011) b) (below) as used by Kott (Kott and Vogel 2003)

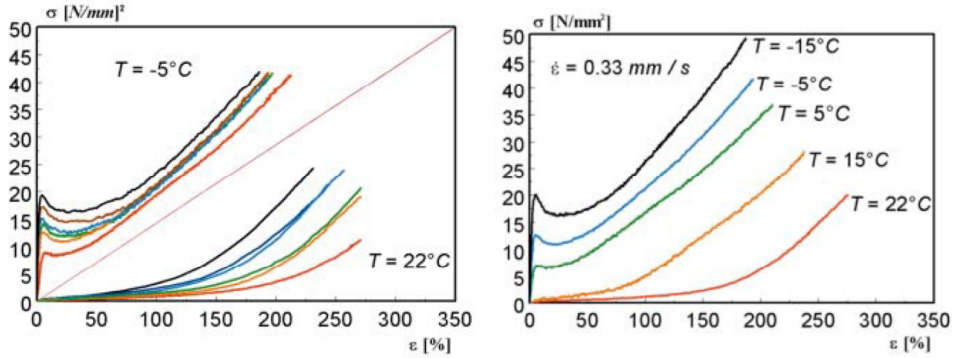

Figure III.15 – Results of uniaxial tensile tests on specimens PVB-interlayer, a) at 6 different constant displacement rates between 0.037 to 5.000 mm/s, b) at 0.33 mm/s for 5 different temperatures between -15 and +22°C (Kott and Vogel 2003)

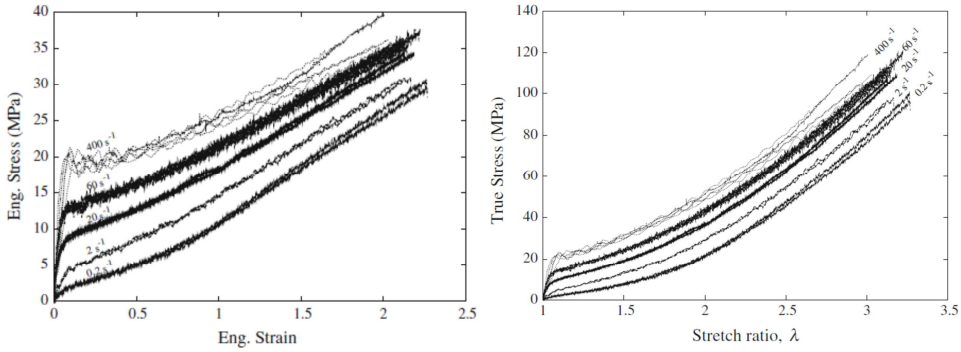

Figure III.16 – Results of conventional uniaxial tensile tests on specimens PVB-interlayer at ambient temperature and relatively large strain rates : (a) engineering strain-stress curves, (b) corresponding stretch-true stress curves, derived by assuming homogeneous and isochoric deformations (Hooper et al. 2012)

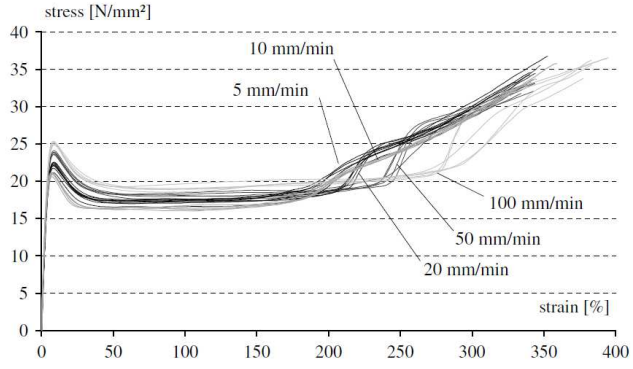

Figure III.17 – Results of conventional uniaxial tensile tests on specimens SG-interlayer at room temperature (engineering strain-stress curves) (Belis et al. 2009)

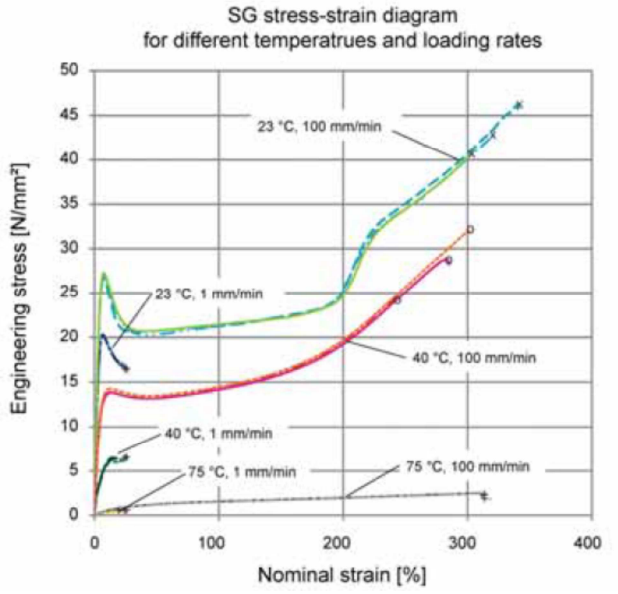

Figure III.18 – Results of conventional uniaxial tensile tests on specimens SG-interlayer at different temperatures above room temperature (engineering strain-stress curves) Note : the tests at 1 mm/min were stopped at an arbitrary strain of 26% (no breakage) (Puller et al. 2011)

Table III.3 – Overview of main characteristics of conventional uniaxial tensile tests corresponding to the results reproduced in Figure III.15 to Figure III.17.

Reference	Material	Specimen's geometry	Temperature range	Displacement rate range [a]	Ratio of rates range [b]
Kott (Figure III.15)	PVB	Fig. III.9 b)	-5 / 22 °C	0.037 – 5 mm/s	135
Hooper (Figure III.16)	PVB	Fig. III.9 a)	25 °C	0.01 – 15 m/s	1500
Belis (Figure III.17)	SG	Fig. III.9 a)	23 °C	5 – 100 mm/min	20
Puller (Figure III.18)	SG	Fig. III.9 a)	23 / 75°C	1 – 100 mm/min	100
[a] Values of applied displacement rate on specimen as given by the different authors [b] Ratio of the largest on the smallest applied displacement rate					

A qualitative comparison of these different results shows however similitudes in the observed response of PVB and SG-specimens. The yield stress vanishes to almost zero for a test temperature about T_g [51]. At smaller values of test temperature, the pre-yield stiffness and the post-yield, large strain rubber stiffness seem relatively little affected by a change in test temperature, in comparison to the effect on the yield stress and yield stress drop. Above T_g , the large strain rubber stiffness seems to get more sensitive to a change of temperature. Besides, the variation of the yield stress drop values for tests performed at different strain rates (in Figure III.13 for PVB and in Figure III.18 for SG) suggests a thermorheological complex behaviour for both interlayer types.

In conclusion, whereas the initial comparison of the respective materials based on tests at room temperature showed very different responses, when larger ranges of test conditions are considered with regard to possible service conditions, in terms of test temperature and applied loading rates, many similitudes appear. Among others, these results of uniaxial tensile tests show that both interlayer materials exhibit an apparent thermorheological complex behaviour in the investigated ranges. Questions and issues to deal with for the assessment of product performances appear further very similar whether they are made with PVB or SG interlayers.

III.3.3. Adhesion properties of interlayers with glass components

Adhesion of polymer interlayers with glass is dominated by an adsorption mechanism, corresponding to a binding force of similar nature than the physical or chemical intermolecular bonds in the bulk of the polymer. The adhesion mainly involves (weak) hydrogen bonds between free polar groups present in the polymers molecular chains on the one hand and in the SiO-group of the glass on the other hand, and possibly but in a lesser extent some stronger chemical bonds. This explains among others the importance of moisture and humidity in the control of the adhesion level (Juang et al. 2001; Keller and Mortelmans 1999; Savineau 1997; Tupý et al. 2013; Weller et al. 2005, 2009).

The secrets of controlling adhesion level (and understanding why it works) are however of another complexity grade, where parameters are often of similar nature but with different relative importance according to the used polymer materials. The largest amount of publications here about concern PVB-laminates, among others (Froli and Lani 2010; Juang et al. 2001; Keller and Mortelmans 1999; Savineau 1997; Tupý et al. 2013), some are also dealing with SG-laminates (Juang et al. 2001; Tupý et al. 2013; Weller et al. 2010).

[51] For the results of Figure III.15, Kott mentions a value of glass transition temperature for the PVB between 12 and 18°C.

However, optimizing the adhesion level in one thing, measuring it is another one. Current reference tests used in practice for controlling the adhesion level, the Pummel test and the Compression Shear test, have already been shortly reviewed in Chapter II section II.5. Other tests mentioned in literature are various configurations of peel tests. However, all these test configurations are also conventional. Whereas useful in quality control or in developing phase, they appear of little use for characterization purpose of properties and behaviour of end-products. Some complementary arguments for supporting this statement are further given in next paragraph and in Chapter IV.

III.3.4. Conventional test configurations and critical basic shapes

Different reasons have been identified here above why conventional uniaxial tensile tests on interlayer specimens do not appear suited for performing a quantitative characterization of its mechanical properties and behaviour within a laminated system. A series of reasons are common to all polymer materials with this type of test configuration, which appear more significant for materials with a glassy phase, having the particularity of being quite sensitive to non-homogeneous deformations and to time-temperature dependent effects. Another series of reasons is common to all polymers used as adhesive, addressing the influence of the lamination process on the end properties. Possible differences exist between bulk and interfacial physical state of the adhesive component due to the processing method, and due to physical ageing effects when it applies.

The alternative test configuration used to characterize solid thermoplastics (uniaxial compression tests) seems less appropriate for thin products as interlayers, and no information is gained about the adhesive behaviour. Finally, investigating separately the time-temperature dependence of the cohesive and adhesive properties is likely to increase the amount of required tests to achieve the characterization and to validate it with regard to an identified application scope. So, what are the possible alternative experimental approaches ?

Issues related to the development of an adapted evaluation strategy for determining mechanical properties of polymers in general, and thermoplastics in particular, are discussed in details in the book “Mechanical evaluation strategies for plastics” (Moore and Turner 2001). The authors explain the historical development of standardized test methods for plastic products and the problems rising by ‘classical’ experimental approaches, based on standardized, conventional test configurations and testing conditions. They draw the attention on the risk of an uncontrolled increase of the amount of tests for fulfilling characterization purposes, principally when the processing conditions are known or are expected to be critical for the mechanical properties of polymer end-products.

Especially when the available resources for making the experimental assessment are limited, it is proposed to adopt an adapted evaluation strategy based on

unconventional specimen shapes (and by extension, non-conventional test configurations), which are called critical basic shapes. The onset of this approach starts from the statement that the measured 'material' properties are in fact often rather 'structural' properties of the particular geometry of the test specimen (including the particular conformation of the material due to the processing method used for making the test specimens). In particular any anisotropy is also rather a characteristic of that structure, not of the material. In the view of Moore and Turner, a *critical basic shape* is resulting as a compromise between a research for a sufficiently general shape and a higher representativeness of the specimens with regard to the configurations and service conditions of end-products. Among other characteristics, critical basic shapes are supposed to have a more complicated shape than traditional ones, but simpler than the end-products aimed to be represented by these. They should simulate end products or parts of end-products. Moore and Turner mention that the concept of 'critical basic shapes' has in particular been used for many years in impact test programmes, but that they tended to be see in the limited scope of ad-hoc experimentation, and that a formal acknowledgement of their potential importance in a testing strategy has often been avoided.

This analysis seems to be largely transposable to the assessment of impact performances, and more generally safety performances, of laminated glass units. In fact, the standardized test configurations for impact tests (see Chapter I) can be considered as critical basic shapes. However, the concept remains relatively abstract, and must be adapted to the case of laminated systems.

The design or the selection of critical basic shapes have to account for the purposes of the experimental investigation. Different categories of test purposes are distinguished (Moore and Turner 2001) :

1) as criteria in quality control and quality assurance activities;
2) as a basis for the comparison and selection of materials;
3) as data for design calculations;
4) as a basis for predictions of service performances;
5) as an indicator in materials development programmes;
6) as a starting point for the formulation of theories in materials science.

With regard to assessment and characterization[52] of post-fracture performances of laminated glass products in relation with design methods, the four first purposes

[52] Characterization of properties in this context refers to 'the production of values of product properties which might be used in combination with appropriate models and conditions for designing real-case applications'. It must not be confused with the notion of "characteristic value" of property used in product standards and in the Eurocodes (see Chapter I), which is a more specific and quantified statistical concept.

are addressed, and should be related to the concepts of FPC and ITT (introduced in Chapter I)[53]. The two last categories seem rather of interest respectively for manufacturers in developing new products and for more fundamental research.

Table III.4 – Complementarity of tests at different experimental scales

Test configuration		Specimen configuration	Fitness for testing purpose				
			TP1 "Quality control"	TP2a "Comparison and selection of interlayer material"	TP2b "Comparison and selection of laminated glass product"	TP3 "Data for design calculation"	TP4 "Service conditions"
	Pummel test	small pieces laminated glass	v	?	?	o	o
	CST-test	cylindrical specimens drilled out of control laminated glass plates [a]	?	?	?	o	o
	TST-test		o	?	?	?	o
	TCT-test	small pieces laminated glass with pre-cracked sheets [a]	?	?	v	v	?
	OCT-test		o	?	v	v	?
	Tests on element	large pieces of laminated glass	?	?	v	v	v
[a] Limited to 'simple' laminated glass units with the two glass sheets in annealed glass (necessary condition for making pre-cracking and cutting operations feasible)							
Legend : o: test configuration less or not appropriate for test purpose; ?: test configuration possibly appropriate for test purpose; v: test configuration possibly the most appropriate for test purpose							

The question is which test configurations are useful for which purposes, and which ones can be considered as complying with the concept of 'critical basic shape'. Also, it must be determined how many test configurations are necessary for performing assessment of products with regard to the (un)identified application scopes (see Chapter I section I.5), and which is the best candidate for

[53] The FPC-tests (Factory Production Control) and possible other tests within control processes belong to the first category. The ITT-tests (Initial Type Testing) can be associated to the items 2 to 4, with possible other validation tests. See also Chapter I.

what purpose(s). A more precise identification of the fitness for purpose of different types of test configurations can be performed, in particular in terms of modelling and assessment strategies. Table III.4 is a possible format for summarizing the outcomes, here by means of three qualitative scores, comparing the fitness for purposes of different test configurations mentioned in Chapter II with each other.

This table has obviously no pretention to be neither complete nor corresponding to an already widely accepted scheme. Some test configurations discussed earlier in this chapter (conventional uniaxial tensile tests on specimens of interlayer material) and in Chapter II section II.5 (peel tests,…) are purposely not mentioned here, because they are not considered as potentially complying to the concept of 'critical basic shape' for adhesive polymer products used in laminated glass configurations.

The separation of the second test purpose category into two, TP2a and TP2b, corresponds more or less with the separation of the design process in two steps, the design of laminated glass products or systems, and the design of applications with these products or systems, because they are respectively managed by different stakeholders. However, for breakthrough innovative designs, these two aspects tend to be integrated into a single one, depending on the collaboration grade between manufacturers, designers, and possibly third parties (control or assessment bodies, test laboratories; and possibly contractors if they are not the product manufacturers).

Besides, control tests (FPC tests) only make sense with regard to unambiguous specifications or a well identified performance from an ITT. In addition to tests strictly performed within an ITT assessment strategy (tests leading to quantitative results transposable in design values, and associated FPC tests), some complementary tests are rather "validation tests" : they do not lead to quantitative values of performances or properties, but they validate their use on an extended application scope. The classification of a test as an ITT test (TP3…) or a validation test (TP4…) cannot always be made beforehand. This is the case when tests are performed with specifications corresponding to an extension of the (initial) application scope. If the results are in line with previously developed design models, it leads to simply extrapolate their application scope; if the deviation is large, there are two possible outcomes : if the failure mode is critical, it invalidates the extension tentative; if not, it can lead to an adaptation of the design rule (in place of a simple extrapolation), for instance in order to account for a reduced performance. Such a question leads to consider assessment strategies for laminated glass products and adhesive polymer components used in non-conventional applications in a progressive and iterative perspective, further developed in the two next chapters.

It seems clear that the fitness for purpose 'score' of any selected 'critical basic shape', in particular in the context of laminated safety glass products used in non-conventional applications, cannot result in an objective assessment of a test configuration on its own, but comprises also more subjective aspects. These elements of subjectivity are related to the balance between the identified application scope(s) and product range(s), and thus are also addressing the level of generality which is expected from the results. They also address another important aspect, namely the level of confidence that specialists of different disciplines can develop for the considered test configurations, and accordingly with all the aspects that might be of importance in interpreting or using the test results : in that regard, *intermediate* experimental characterization scales[54] can be seen as a necessary sub-category of 'critical basic shapes'. In this perspective, the determination of mechanical properties of the interlayer must be performed by considering it rather as a *component* than as a material.

In the context of this work, mainly the post-fracture performances of laminated glass products are addressed. For this purpose, TCT-tests seem an eligible configuration for characterizing the interlayer ligament's behaviour with regard to the identified critical load transfer mechanism[55]. The development of an experimental assessment strategy based on TCT-specimens of SG-laminates is developed and discussed in Chapter V. The potential and limitations of other test configurations is also discussed in Chapter IV.

[54] The concept of intermediate experimental scale is further developed in Chapter IV.

[55] The concept of 'critical basic shape' can be considered as slightly transformed, into a 'critical basic load-transfer mechanism', see also Chapter II section II.4.

III.4. Summary and outlooks

An overview has been given in the current chapter of the main features ruling the (complex) time-temperature dependent mechanical behaviour of polymer materials in general, and of interlayer products in particular. Interlayer materials belong to two main categories, thermoplastics and elastomers, differing by the nature of the secondary intermolecular bonds, which modify their processability and their typical time-temperature behaviour.

It has been shown that thermoplastic products used as structural, load-bearing elements generally exhibit a thermorheological simple or a thermorheological complex behaviour in relation to the amount of relaxation mechanisms, in particular with regard to their non-linear large strain behaviour. The simple or complex character is not an intrinsic characteristic of a polymer material, but rather depends on the considered range of testing and service conditions.

Physical ageing is identified as a complementary important phenomenon for polymers used below their primary glass transition temperature, and which explains the sensitivity of the yield stress and the resistance to creep to the thermomechanical history, dominated either by the processing or the service conditions. An ageing state function, $S_a(t)$, is used to account for this phenomenon on the mechanical properties.

The identified phenomena are likely to be similar for polymers used as an adhesive component, as interlayers. The time-temperature dependent and ageing effects are likely to affect differently the interfacial adhesive properties and the bulk cohesive properties of an interlayer component : this is formally accounted by considering that the interface and bulk properties can principally depend on two different ageing state functions, respectively $S_{a,I}(t)$ and $S_{a,B}(t)$.

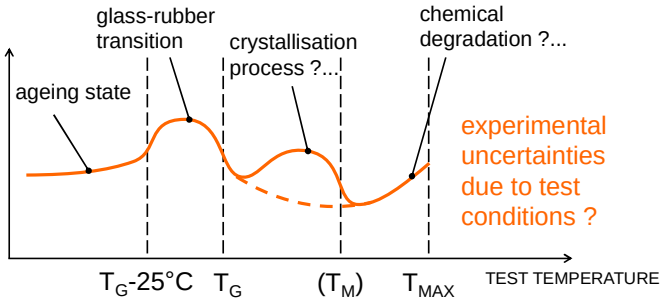

Figure III.19 – In what extent can the representativeness of test results vary in function of test conditions, with regard to possible 'phase changes' in polymer components ?

With regard to the ranges of service conditions, issues for assessing contribution to post-fracture performances of PVB and SG products seem quite similar, despite their quite different typical behaviour at room temperature. However, physical ageing phenomenon is expected to have effects on measured properties of another order of magnitude according to the investigated experimental range : a more significant influence is expected for tests carried out at room temperature on SG-laminates than on traditional PVB-laminates, in relation to their respective glass-rubber transition temperature.

In general, the identified factors affecting the behaviour of this category of interlayer materials represent a possible source of systematic deviation in interpreting test results, and a complementary constraint to take into account for conceiving experimental assessment programs. Figure III.19 summarizes schematically[56] the potential issues to address, with regard to their expected importance; however, the shape of this curve is rather expressing a qualitative question rather than giving a representative order of magnitude...

Because of the complex behaviour with regard to the identified service conditions, a risk exists that the required amount of tests for performing a satisfying characterization of interlayer properties in relation to post-fracture performances of laminated glass products increases on a non-reasonable way. Experimental approaches based on conventional tests on specimens interlayer, among others the uniaxial tensile tests, do not seem appropriate for a quantitative determination of the design values of properties of end-products.

Consequently, alternative approaches by means of test configurations using 'critical basic shape' were shortly presented and discussed. The concept, initially proposed for plastic products, need to be adapted for adhesive products and laminated systems. It will be examined in next chapters which test configurations comply with this concept, with regard to the assessment of the critical load transfer mechanism identified in previous chapter, namely the TCT-configuration.

[56] In fact, effect of temperature acts in combination with time and loading dependent effects, but it complicates a potential graphical representation of the addressed issues…

Chapter IV

Experimental investigation of time-temperature dependent behaviour of fractured laminated glass elements

"First remove the beam out of your own eye, and then you can see clearly to remove the speck out of your brother's eye" (Matthew 7:5, World English Bible)

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IV.1. Introduction

Characterization of product and material properties ruling the mechanical behaviour of fractured laminated glass elements appears to be relatively complex, in particular with regard to the sensitivity of the interlayer component to time-temperature-ageing effects. It has been shown in Chapter II that the load-bearing performances of fractured elements depend on the capacity of the interlayer(s) to keep fulfilling a bridging function between the glass fragments, and that in a variety of structural configurations this bridging function can reasonably be simplified to the same 'critical load-transfer mechanism', the TCT-configuration. This critical load-transfer mechanism is activated when all superimposed glass sheets are cracked in a same cross-section, perpendicular to the direction of the principal tensile effort. The interlayer can be considered as a particular case of polymer material used as structural adhesive. The macroscopic response of the interlayer ligament, its ductility, resistance and failure mode, depend directly on its bulk, cohesive properties and its adhesive properties with the glass fragments, and more particularly on the *balance* between them. It has been shown in Chapter III that interlayers are a particular case of structural adhesives, made with a variety of polymer materials belonging to two families, the thermoplastics and the elastomers. They share common features, as large strain behaviour, sensitivity of product properties on processing and/or service conditions, possible sensitivity to ageing and degradation mechanisms and complex time-temperature dependence of their mechanical properties. The latter is more significant for the behaviour of thermoplastics.

All these aspects induce particular constraints for performing mechanical tests in the perspective of *characterizing* design properties, with regard to the selection of the test specimens, test configurations and test methods. Besides, the particular assessment context on the one hand (Chapter I) and the complex interaction between questions about the post-fracture states with other aspects of the behaviour of laminated glass elements (Chapter II) and interlayer materials (Chapter III) on the other hand raise complementary constraints in terms of amount and scale of tests. The selection of the most suited experimental approaches, test configurations and test conditions is far from straightforward. Moreover, *extension* of the experimental investigation scope is facing a series of practical considerations and technical limits, with regard to the test specimens, the test facilities and the measurement methods.

In this chapter, an analysis grid is developed in order to compare and evaluate strengths and weaknesses, advantages and disadvantages of different test methods on laminated glass specimens performed at different scales and under different conditions, in particular with regard to their potential to be used as *characterization* methods. Two aspects are addressed : providing on the one hand representative results and relevant design values for designers of end-applications, and on the other hand assessing the *representativeness* and the *robustness* of test

methods for a variety of product configurations and applications scopes. However, the second aspect faces the difficulty that it addresses different categories of users, for different categories of applications, in particular with regard to non-conventional structural applications, and each raises specific questions and expectations. Consequently, at the time of conceiving and developing test methods, most of the time already different test purposes can be identified, which can influence the conception, and among others of the measurement methods, and the level of analysis and reporting. The analysis grid allows the further identification and the distinction of different sources of systematic deviations arising in experimental works.

In a second step, an overview of a series of successive experimental campaigns and experimental developments performed at different scales for investigating the post-fracture behaviour of laminated glass elements is given. The analysis grid is used to analyse retrospectively the development strategies of test configurations, test infrastructures and measurement methods. Technical issues are highlighted which finally appear to address compatibility issues between different approaches and different test methods.

Finally, questions related to systematic measurement uncertainties and their propagation in the processing and analysis of test results are shortly discussed.

IV.2. Experimental scales and Experimental Fields of Investigation

Because of the identified practical issues or limits for making quantitative relevant characterization of mechanical properties at a ‘structural’ or ‘element’ scale (see Chapter I and Chapter II) and at a ‘material’ scale (Chapter III), it followed relatively easily that we had to focus on experimental ‘intermediate’ scale(s) (Figure IV.1, above). However, this simple classification is not so univocal with regard to concrete experimental configurations and test conditions. In comparison, the distinction between tests on specimens cut out of folio interlayer or tests on specimens of laminated glass units seems not prone to confusion or misunderstanding (Figure IV.1, below).

A second representation (Figure IV.2) rather accounts for the investigation fields in terms of loading modes and loading ranges on the one hand, and of non-fractured and fractured systems on the other (Savineau et al. 2013) – but we lost the information about the experimental scales. Complementary fields of investigation and related parameters of importance specific for polymer interlayers, identified in Chapter III (temperature range; initial ageing state; configuration geometry; time…), still have to be added to our representation of experimental issues.

Research approach : multi-scale problem
Modelling approaches

scale	pre-breakage	post-breakage
material	small strains : visco-elastic	large strains : hyperelastic / visco-elasto-plastic
intermediate	(interfacial slip neglected)	adhesion delamination
element	long-term behaviour	breakage patterns propagation of cracks

Research approach : multi-scale problem
Experimental possibilities/limitations

scale	pre-breakage	post-breakage
material	tests on interlayer material samples	
intermediate	tests on laminated glass samples	
element		

Figure IV.1 – Initial definition of experimental scales :
the first system (above) is not univocal in terms of test configurations;
in comparison, a distinction between tests on specimens laminated glass and on specimens interlayer material (below) is more robust

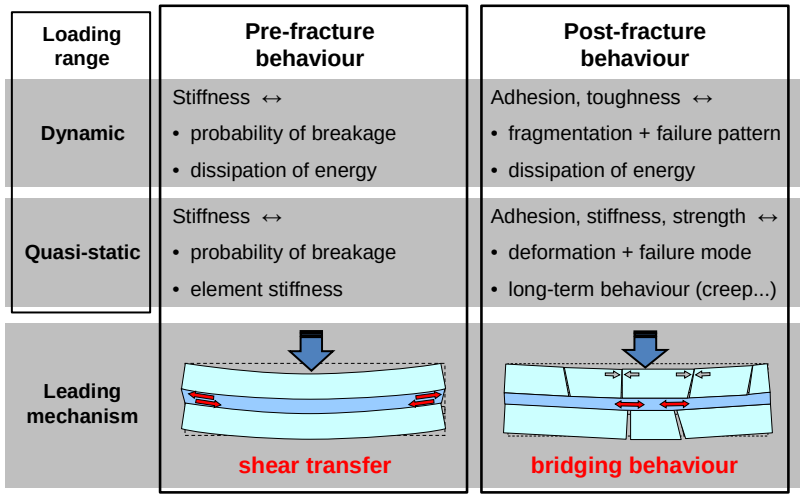

Figure IV.2 – Schematic representation of identified fields of investigation for mechanical performances of laminated glass elements, in terms of loading range, intact or fractured state and corresponding leading load-transfer mechanism (Savineau et al. 2013)

In general, test methods are developed to be performed at ambient temperature, for a reference configuration and for reference test conditions[1]. Because of the (un)known dependence of mechanical properties of the polymer component on time-temperature-ageing effects, demands for extending the scope of test conditions are addressed. For instance, issues regarding the extension of the test temperature range of a given experimental configuration are illustrated schematically in Figure IV.3. Adapting a test method or test configuration for extending the range of test temperatures is facing technical limits, which, if they have not been identified in an early stage, cannot be exceeded; it can then be necessary to restart the design of the test configuration from zero.

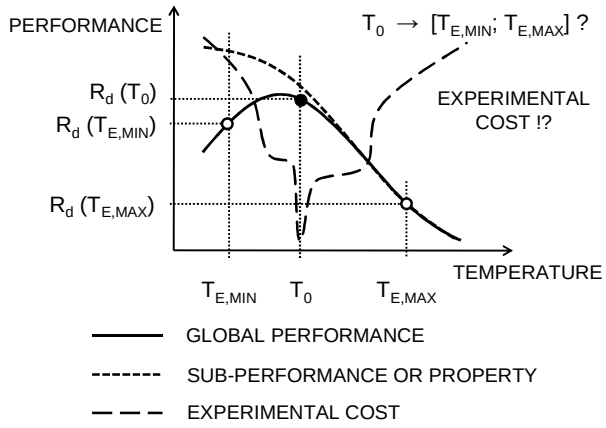

Figure IV.3 – Extending the range of execution temperature of mechanical tests is facing technical limits according to the experimental scale, test configuration and measurement methods (Delincé and Belis 2013)

The 'costs' related to the use of a climatic chamber are of different nature. Besides the primary cost of the device (the insulated box and heating/cooling systems), secondary costs take the form of constraints on usable measurement methods and devices, and possibly the sensitivity of some pieces of the test frame to variations of temperature (thermal movement, corrosion,…). With regard to measurement methods, constraints and 'technical barriers' are different for contact and non-contact methods[2]. The range of use of contact methods is limited

[1] See also Chapter I paragraph I.5.2 and Chapter II paragraph II.3

[2] "Contact methods" refers to the use of strain gages (bonded or integrated to the test specimen), extensometers, or any other technique requiring a contact with the test specimen; "non-contact methods" refer to the various sorts of optical methods and other waved-based measurement techniques (Sharpe 2008). With regard to tests performed in a climatic room or chamber, the concept of non-contact (measurement) method must be distinguished from the concept of non-contact test method, whether steps *during* the test are necessary which involve contact with or manipulation of the test specimen.

by absolute limits, related to the risk of damage of electronic devices due to condensation (lower, cold humid limit) or to corrosion or melting (upper, warm (humid) limit), and by relative limits, related to the loss of measurement precision in a range in between. Similar absolute and relative technical limits also apply to the use of non-contact methods, as their use must be compatible with the presence of walls of the insulated box on lighting and measurement paths (in case of continuous data acquisition), and whether components or devices necessary to the measurement method have to be placed inside or outside the climatic chamber.

Similar questions arise to extend the scope of tested products (family of products and ranges of test specimens' configurations). Not all test methods have the same potential with regard to the different extension fields (temperature range, loading range, range of specimen configurations), for practical reasons on the one hand (technical limits), and according to the identified purpose(s) within a larger assessment strategy on the other hand. Again, feasibility and relevancy must be distinguished.

In order to address these two aspects in a general way, different **Experimental Fields of Investigation (EFI)** can be identified to describe any experimental configuration and associated test specimen configuration (for mechanical tests on specimens laminated glass). An overview is given in Table IV.2, where the EFI's are regrouped into different categories. The individual fields are described by means of field descriptors, which are parameters discussed in more details below.

The 'rules' for selecting or defining each EFI-descriptor are similar to the ones for AF's. They should correspond to relevant *quantitative* variable(s), preferably primary experimental variables[3]; for some experimental problems, it can be necessary in order to avoid the introduction of unrelated systematic uncertainties. Determination of most of the parameters should be related to measurement or test methods. Contrary to the descriptors of the category AF-Product, the descriptors of the category EFI-Specimen can consider non-assessed properties.

There is obviously a parallel between the proposed categories of EFI's and the categories of Application Fields (AF's) introduced in Chapter I section I.5 to describe the possible application scopes of a family of products (Table IV.1 reproduces the corresponding analysis grid in parallel of Table IV.2). The categories EFI-Specimen and AF-Product are closely inter-related, with many identical field descriptors. They must however not be confused with each other, as the range of values of a particular EFI-descriptor can be different from the equivalent one used as AF-descriptor. It can for instance acknowledge for different processing methods available in production plants and in test labs.

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[3] Primary variables are for instance the applied force F, the displacement rate, the dimensions of the specimen, etc. Derived (calculated) variables are for instance the bending moment in a beam, a strain rate, etc.

Table IV.1 – Application Fields for laminated glass products and applications

Application Field (AF)		**Examples of AF descriptor**
	Product : Material	- type(s) of glazing sheet - type(s) of interlayer product - type(s) of embedded inserts and reinforcements
	Product : Geometry and configuration	- description of ranges of geometric configurations : composition, amount and thickness of layers; inserts; … - possibilities and limits for lamination sizes - possibilities and limits for cutting sizes
	Product : Processing	- production methods : lamination, cutting (incl. holes,…), edge finishing,…, possibilities and limits in function of considered configuration ranges - level of standardization of the various processing steps
	Product : Connections	identification of possibilities and limits for connecting the laminated glass product into a construction work : zones and features intended to be used / avoided for connecting the element; (in)compatibility with other materials and with service conditions
	Application : Design : Performance requirements	Expression of performance requirements : - resistance to impact(s) / source(s) of damage - loading cases : type, configuration and extent of individual action; combination rules (ULS, SLS,…) - exposure conditions : temperature, ageing agent,… due to climatic and service conditions (cleaning,…) - non-structural performance requirements affecting the design : acoustic, insulating, light control, etc.
	Application : Design : Geometry and Configuration	- Element dimensions : planar dimensions, (maximal value of) total thickness, functional constraints in function of performance requirements and design configuration (with regard to edge finishing, etc.) - Connections and fixing configuration and conditions, intermediate pieces (mechanical connections) or components (adhesive connections,…), possible consecutive requirement on edge or surface finishing,…
	Application : Execution : Processing and assembling methods	Identification of execution steps likely or intended to induce constraints (stress) into the laminated glass element or any of its component.
	Application : Service conditions	- measures to take in case of damage / failure : replacement of the damaged element, - measures to take in case of change/deviation of service conditions with regard to initial assumptions or specifications used for the design - control and monitoring in service conditions (optional)

Table IV.2 – Experimental Fields of Investigation for tests on laminated glass units

Experimental Field of Investigation		**Examples of EFI descriptor**
	Specimen : Material	- type(s) of glazing sheet - type(s) of interlayer product
	Specimen : Geometry	- possibilities and limits for lamination sizes - possibilities and limits for cutting sizes of specimens laminated glass
	Specimen : Processing	- Production method of test specimens versus production units, possibilities and limits
	Specimen : Pre-treatment, Conditioning	- Description of 'initial state' due to processing method - Description of state due to storage conditions - Complementary treatments (artificial ageing, exposure to agents,
	Test configuration : Basic device	- Basic testing machine, core part of testing infrastructure and complementary equipment
	Test configuration : Geometry	- Geometric configuration of test : fixed or variable limits,… - Possibilities and limits for dimensions of test specimen, and for fixing/grip methods
	Test configuration : Loading configuration	- Type of load(s) and area/point of application - Extent of spatial range of load application, fixed or variable position
	Test configuration : Control mode, Loading range	- Possibilities and limits for controlling the load application : field of control (displacement, force, strain, stress,…), type of control (continuous, discrete; constant value, regular cycle; automation grade)
	Test configuration : Measurement methods and measurement configuration	- Field of measurement and measurement device - Type of measurement devices - Acquisition system(s), acquisition type, limits of acquisition frequency - Limits of use in function of other EFI's
	Test conditions : temperature,…	- Distinction of active and controlled agents : temperature, relative humidity,… - Exposure / control range

Here are two examples to illustrate this statement. The description of the glazing components of the laminate (glass sheets) can contain a field descriptor "strength". In design oriented description (AF's), it is summarized as a characteristic lower value (for instance, 45 MPa for an annealed float glass product), namely corresponding to a loading level at which the element should not break. However, in the perspective of designing a test configuration where the glass components *have to* break (especially for tests at an 'element' scale), an estimation of the upper strength of the glass component of a test specimen should rather be considered (it would be a value about 120 MPa for quasi-static loading conditions; the reference value could vary with the size and loading configuration considered). This example however addresses probably one delicate field descriptor; in fact, the glass strength is not a primary experimental variable, but a derived one. As second example, the total thickness of a laminate product can be considered. As AF-descriptor, it is one of the parameter describing the possible production range of a family of product, where the upper value corresponds for instance to the maximal thickness for the calendaring process; as EFI-descriptor, the range is more likely to be limited by the dimensions of the test rig or of a test frame.

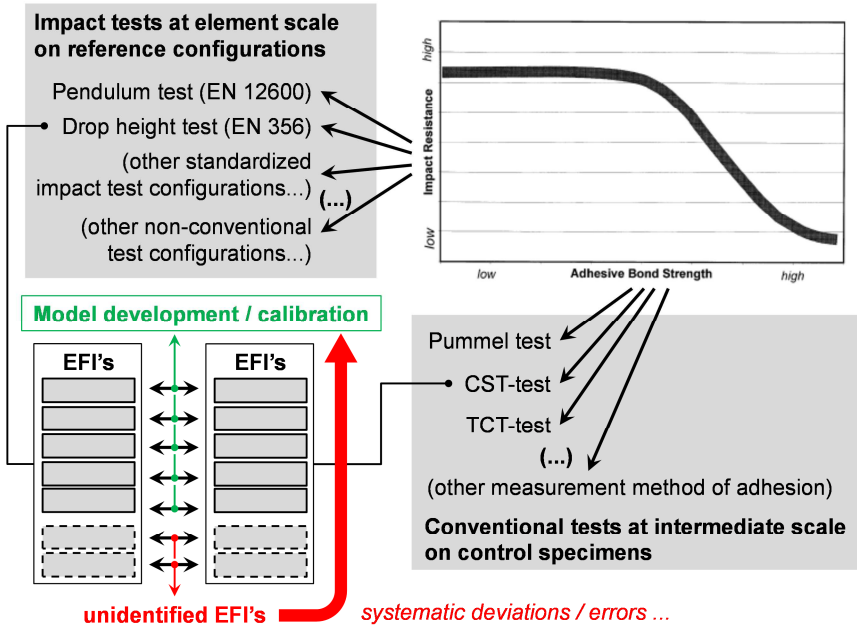

Figure IV.4 – Example of use of Experimental Fields of Investigation to identify sources of systematic deviations or errors in an empirical correlation between test results performed at different experimental scales, here between an impact performance (ITT-test) and a measurement of the adhesion level (FPC-test). (Qualitative correlation curve reproduced from (Keller and Mortelmans 1999)).

This second example illustrates also that the identified EFI's are more or less interconnected and inter-dependent with each other. Possibilities or limits for extending any individual EFI are related to compatibility issues with one or more other EFI. The different field categories are illustrated below and in next sections in this chapter by means of a series of examples, and some will be described in more details. They must be understood in first instance as a conceptual tool to explain some experienced compatibility issues and distinguish problems of different orders.

Let us consider firstly a more practical example, which "by the way" also illustrates the potential and the constraints for developing 'performance based approach' test methods. A common working method consists to compare results of tests performed at different scales[4], by means of an empirical correlation; when a qualitative correlation between two measurement scales appears, in a second step calculation models are used or developed, in order to explain the observed correlation on a quantitative way and evaluate the possibility of using these as predictive tools[5].

Such an empirical correlation is typically used for comparing the adhesion level of a laminated glass *product*, measured for instance by means of a CST-test, with a specific safety performance of the laminated glass *element* measured by means of the dedicated standardized impact test, for instance the drop height test according to EN 356 (Figure IV.4). The first types of tests are FPC-tests and the second ITT-tests[6]. The manufacturing industry typically uses this kind of approach to deal with the problem of adhesion control, and each manufacturer of interlayer or of laminated glass products developed a peculiar know-how with its own assortment of products. The empirical correlation in fact relates two measurement scales independent of each other's, therefore it will be qualified here as a *qualitative* correlation[7].

When attempting at modelling this kind of correlation, thus to establish a *quantitative relation* between properties and performances (or between a product performance and an application performance) by means of mathematical models,

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[4] Differences of experimental scales refer here not only to possible difference in geometry of test specimens, but to difference in any other of the identified EFI's.

[5] An example of such an approach applied to the problem of glass strength and strain energy release rate (see also Chapter II section II.4) is given in (Bos 2010).

[6] FPC-tests : Factory Production Control tests; ITT-tests : Initial Type Testing tests (see also Chapter I section I.4 and Chapter III paragraph III.3.4). The ITT-test methods mentioned on Figure IV.4 are described in Chapter I paragraph I.4.2; the FPC-test methods are shortly described and discussed in Chapter II and Chapter III.

[7] In early informal discussions with the manufacturer of the SG-interlayer (… between 2002 and 2004), a mentioned problem was that, when such an empirical correlation for tests on PVB-laminates was established, *similar* test configurations and methods performed on SG-laminates did not result in a qualitative meaningful correlation.

it leads to deal with various issues[8]. In order to assess the influence of the respective fields EFI-Test configuration of each test method on a meaningful way, it is implicitly assumed that there is *no mismatch of representativeness between the corresponding test specimens*, in the considered example between the small cylindrical specimens drilled out of a 300x300 mm laminated glass plate used for the CST-test, and the 1100 x 900 mm laminated glass unit used for the drop height test. Translated into EFI's, this assumption can be expressed in two ways :

1) All the field values of the category EFI-Specimen are equal between the two specimen scales (no difference of properties between specimens). With regard to the identified specificities of adhesive polymer materials in terms of (possible) sensitivity to time-ageing dependent effects (Chapter III), the verification of this assumption can require strict control of the respective conditioning and test conditions. For some types of specimens used in adhesion tests, the validation of the equality for some of the involved fields EFI-Specimen seems very complicated;

2) The differences between corresponding fields of the category EFI-Specimen of the two experimental scales can be identified qualitatively, and described quantitatively. For instance, there can be an issue due to different initial ageing states[9] between two specimens, caused by (significant) differences in storage duration and/or conditions, or by some processing steps (as the cutting of specimens of small size).

The differences of values between corresponding fields EFI-Specimen of two test specimens are a *first category of border effects*.

But let us temporarily assume that border effects of this first category, as different initial ageing states between specimens, are not significant. A *second category of border effects* is due to differences of values or correlation between corresponding field descriptors of the categories EFI-Specimen on the one hand, and EFI-Test configuration / EFI-Test conditions on the other hand, for each test configuration involved in the correlation research exercise. For instance, the strain rate reached in the interlayer during the test is of another order of magnitude between the CST-test (quasi-static constant displacement rate) and the drop height test (dynamic loading rate).

One can easily get convinced that problems are arising when some possible mismatches have not been identified between corresponding EFI's of two

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[8] This typically happens when a test method initially used as a FPC-test (test purpose TP1) is evaluated to become an ITT-test (test purpose TP3); categories of test purposes are described in Chapter III paragraph III.3.4.

[9] The concept of initial ageing state of polymer specimens has been introduced in Chapter III, paragraph III.2.3.

different experimental scales, or similarly between EFI's and AF's. A clear and detailed identification of the various EFI's beforehand can possibly help to track, in post-processing analysis[10], the possible sources of systematic errors or deviations between test results performed on different test scales. The probability of modeling successfully and robustly the considered initially empirical correlation can thus be harmed or complicated by the presence of a *combination of significant border effects* on the considered ranges of some EFI's[11], especially if these are related to unidentified but present EFI's.

Two types of unidentified EFI's can be distinguished :

1) An *unidentified field variation* : a field descriptor assumed to have a constant value during the conditioning period or during the testing period varies significantly, possibly with consequences on other EFI's.

   Examples :

   (EFI-Specimen: Conditioning) change of (initial) ageing state of specimen during storage duration (see Chapter V paragraph V.3.4);

   (EFI-Test conditions → EFI-Test configuration: Loading range) larger oscillations in the applied temperature during a test at cold temperature in a climatic chamber appeared to induce significant thermal movements along the loading string, and consequently oscillations appeared in the effective applied displacement rate on the TCT-test specimen (see Chapter V paragraph V.2.6);

   (EFI-Test conditions → EFI-Test configuration: Measurement method) during the same test, the cyclic coolant blows into the climatic chamber, which caused the oscillations in the applied temperature, appeared to cause also lighting disturbance in the form of fog (condensation of air humidity), resulting in correlated cyclic deviations in the optical measurements of the deformations (see Chapter V, paragraph V.2.6.);

2) An *unidentified field effect*, possibly in interaction with other identified EFI's.

   Examples :

   (EFI-Specimen: Processing or EFI-Specimen: Conditioning) edge effect, namely influence of processing of specimens on the damaging or ageing of the

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[10] In this context, post-processing analysis can also refer for instance to comparison of test results from different publications or test reports.

[11] Of course, similar systematic deviations can be inherent to the modelling approach and used models, but these can be overtaken by iterative work or by moving towards the use of more complex models. There is thus some overlap between experimental and numerical sources of systematic deviations, and a univocal allocation is, again, not always possible or straightforward, as it depends on the used models (and the confidence level the user has in these…). This is however already beyond the scope of the present discussion.

interlayer along the edges of the test specimen, possibly enhanced by the test configuration. This question addresses essentially all the tests performed on specimens of small dimensions cut out of larger pieces of laminated glass, as CST-test (Chapter II section II.5) and TCT-test (Chapter V paragraph V.2.2);

(EFI-Test conditions) possible *combination of effects of different ageing agents* during a radiation test : in what extent the effect of UV-radiation is enhanced by the concomitant value of ambient temperature during an ageing test ? (related issues between Chapter I and Chapter III);

(EFI-Test conditions ↔ EFI-Specimen: Material) *different effects of ageing agents* during a radiation test on two laminated glass specimens constituted of different interlayer materials, with regard to the nature of ageing at molecular level : in function of the material characteristics of the polymer component, the effective causes of the observed effect[12] can be different. For instance, in a first specimen the cause is a chemical degradation of molecular chains of the polymer component due to UV-radiation energy, while in another one the cause is a change of physical ageing state caused by the concomitant ambient temperature. In other words, the 'radiation ageing effect' appears to correspond to an effect of 'annealing treatment'… (related issues between Chapter I and Chapter III)

(EFI-Test conditions → EFI-Test configuration: loading range) variation of effectively applied displacement rate on specimens due to machine oscillations (Chapter V paragraph V.3.2.2).

From the above analysis, it appears that identified possible degradation problems for the representativeness or robustness of test methods in fact address two types of issues : effective technical problems or limits when an attempt is made in changing or extending the range of one or more EFI's (the test or the measurement cannot be performed), and problems of interpretation of test results, with regard to problematic or erroneous allocation of the sources of deviation. The detection of the second type of experimental degradations is obviously all the more difficult that they contain an interpretative component, which is again to be related to the identified possible multiple purposes of the tests (see Chapter III paragraph III.3.4) : this points out the importance of interactions between development of laminated glass products and applications on the one hand, and of test methods and assessment strategies on the other.

The EFI's allow to deal with different aspects of robustness and representativeness of test methods. The aspects of *robustness* are related to possibilities and

[12] The effect of a radiation ageing treatment can be assessed by means of different test methods, or evaluation criteria, for instance: the stiffness of the interlayer component; the transparency of the laminate;…

limits imposed by the individual test method, with regard to the relation between fields of the category EFI-Specimen and corresponding ones of EFI-Test configuration and EFI-Test conditions. Aspects of *representativeness* are related to the definition of applications scopes, that determine the possible ranges of use of end-products (EFI's vs. AF's) and to consecutive requirements on measurement accuracy and precision, and to possible mismatches between corresponding EFI's of two different experimental scales (whether in relation with difference between respective fields of the category EFI-Specimen or of the category EFI-Test configuration/conditions).

Here appears an important issue : it is not because a test can be performed that it is meaningful, and further, it is not because it is possible *and* meaningful when performed in some test conditions (for instance of temperature…) or on specimens with some type of interlayer, that it is even possible *and/or* meaningful to perform the same test on a specimen with another interlayer material or in other test conditions, all the other EFI's remaining unchanged between the two tests.

Concepts of robustness and representativeness of test methods and test configurations as defined here can thus be related to different types of *unidentified border effects*, which induce a degradation in the form of either a *loss of robustness* either a *loss of representativeness*, or both simultaneously. The extent of each type of degradation can be assessed, and possibly reduced, by identifying and validating the mismatch between corresponding EFI's, and between corresponding EFI's and AF's. However, it is often difficult to identify beforehand, and still not straightforward afterwards, which degradation risk is the more critical.

An important experimental shift between different test scales and test purposes concerns distinction between dynamic and quasi-static loading ranges : in terms of experimental infrastructures, measurement devices and test methods, there is an important "technical barrier" between both experimental investigation scales. It is however more convenient experimentally to make a distinction between a pre-fracture behaviour, a fragmentation process of the glass sheets, and a post-fracture behaviour in the quasi-static range than in the dynamic one (see also Chapter II section II.2). It is however accompanied by a "scientific" barrier, which is related to the differences of present phenomena in dynamic and quasi-static loading ranges, and in corresponding modelling approaches.

IV.3. Development of experimental approaches

In this section a succession of different experimental campaigns are analysed by means of the analysis grid proposed in the previous section, which have involved numerous tests at different experimental scales, in the context of the evaluation of the post-fracture performances. The experimental campaigns are presented by detailing the constitutive experimental block sessions in a chronological order, in order to highlight the chain of successive decisions, the related methodological issues and issues regarding the development of test infrastructures and test methods, in relation with different technical (and knowledge…) limits.

To be clear, the analysis grid presented in the previous section has finally emerged *after the execution* of the experimental campaigns, *at the end* of the process during this doctoral research. The analysis presented here is thus rather a retrospective one, certainly entailed of subjectivity. The purpose of this analysis is to highlight *a problem of research approach* with this kind of products and systems in the context developed in previous chapters, with the hope to give *some comprehension keys* to decision makers involved in assessment and research processes in relation to these questions.

This section distinguishes different series of experimental campaigns, regrouped as follows :

- A “preliminary phase” is related to a pre-standardization research project prepared at the initiative of the Belgian Building Research Institute between 2004 and 2006 and following discussions carried out inside its Technical Committee “Glass works”. A short overview is given of series of tests at different experimental scales and on different product configurations which have been performed in this context (paragraph IV.3.1);
- An “orientation phase” explains in more details the evolution in experimental approaches, in parallel of the technical aspects related to the development of test infrastructures and measurement methods during the first three years of a FWO-project carried out between 2006 and 2010 at Ghent University (paragraph IV.3.2);
- A “development phase” which consisted in an experimental campaign of TCT-tests during which a particular incremental strategy has been developed and executed between 2010 and 2013, and which is the topic of the Chapter V.

IV.3.1. Preliminary phase (2004-2006)

A pre-standardization research project had been started by the Belgian Building Research Institute (BBRI) in partnership with Ghent University and funded by the FPS Economy (department of the federal administration), which ends up in a research report (Delincé, Zarmati, et al. 2007) of which a summary has been presented in (Delincé, Belis, et al. 2007).

The experimental program consisted in fact in two parts conceived and developed relatively independently of each other. A first part was conducted in the context of a doctoral research on the lateral buckling resistance of laminated glass beams (Belis 2006), with a large amount of tests executed on beam specimens (element scale). The second part consisted of different series of compressive shear test (CST-tests) and tensile shear test (TST-tests) configurations performed on the same type of cylindrical specimens drilled out of laminated glass plates (about 250 specimens, 'intermediate' scale), and of 4-point bending tests performed on 1100x360 mm laminated glass plates (about 40 specimens, 'element' scale)[13]. The different tests are regrouped in three series, respectively "CST / TST tests", "Bending tests", "Buckling tests", and corresponded to tests performed in three different laboratories attached to different institutions. A summary is given in Table IV.3 structured with the proposed Experimental Fields of Investigation.

Common characteristics of these three experimental campaigns are the relatively large amount of tests specimens, limited amount of test configurations and loading modes, limited test conditions (mainly tests carried out at room temperature and constant relative humidity), and the investigation of two ageing effects according to the standardized procedures (see Chapter 1 paragraph I.2.2). In other words, large ranges of EFI-Specimen were investigated with narrow ranges of EFI-Test configurations/Test conditions.

Main outcomes were the gained insight about a series of particularities of the mechanical response of laminated glass structures and of related experimental issues. Among others, orders of magnitude were gained about the influence of a series of parameters, but a series of issues were also identified which addressed the representativeness and the robustness of test methods considered during this campaign. It led among others to question the usability of CST and TST test configurations with regard to characterization purposes (see also analysis in Chapter II section II.5). The analysis of the post-fracture performances and behaviour remained relatively qualitative, as most of the analysis efforts still remained mobilized around the shear-transfer contribution of the interlayer[14].

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[13] The test methods were slightly adapted in comparison with corresponding tests reported in other researches (see Chapter II section II.5), principally in terms of loading conditions.

[14] Corresponding to the Longitudinal Shear Load Transfer Mechanism (LS-LTM) defined in Chapter II section II.4.

Table IV.3 – Summary of tests at different experimental scales (FOD-project, 2004-2006)

Category EFI	**Field descriptors and investigated ranges**		
	CST / TST tests	Bending tests	Buckling tests
Specimen : Material	- large scope of laminated glass products, with different types of glass sheet (annealed, hardened and toughened float glass) and two types of interlayers (unidentified PVB, and SG)		
Specimen : Geometry	Laminated plates 300x300 mm, simple composition, 1 or 2 thickness interlayer	1100 x 360 mm, various composition (thicknesses layers)	Lengths 1 .. 3 m, various heights, various composition (thicknesses layers)
Specimen : Processing	As delivered	As delivered	As delivered
Specimen : Pre-treatment, Conditioning	2 types ageing tests + non-aged	2 types ageing tests + non-aged	None
	2 geometries cyl. specimens drilled out laminated plates diam: 30 / 60 mm		
Test configuration : Basic device	Universal electro-mechanical testing machine	Electronic controlled hydraulic loading system (actuator)	Manual controlled hydraulic jack
Test configuration : Geometry	2 geometries	1 fixed geometry	1 configuration
Test configuration : Loading configuration	CST : 2 geometries TST : 1 geometry	4-point bending tests (weak axis),	beam (strong axis) on 2 end-supports, load applied in central cross-section
Test configuration : Control mode, Loading range	Constant displ. rate, 4 loading modes, 2 displ. rates	1 main loading mode, constant displacement rate	1 main loading mode, ~ constant displacement rate
Test configuration : Measurement methods and measurement configuration	Force and displacement, continuous acquisition	Force and displacement, continuous acquisition	Force and (2) extensometers, continuous acquisition
Test conditions : temperature,…	Room temperature (unique conditions)	Room temperature [a] (unique conditions)	Room temperature [b] (unique conditions)

[a] A series of bending tests on 'unaged' specimens were performed between -10 and 50°C
[b] Orientation tests at higher test temperature with use of IR-radiants were performed

In a general way, issues related to degradation of *representativeness* of test results due to *propagation* of *measurement uncertainties* and of other *sources of systematic deviations/errors* were identified qualitatively, but could not be distinguished of each other and were still rather poorly *quantified*.

IV.3.2. Orientation phase (2006-2011)

Experimental investigations carried out during this research period have mainly been conducted in the context of a 4-year research project funded by the Research Foundation – Flanders, FWO-Vlaanderen (2007-2011).

The primary identified experimental purpose corresponded to the TP3 "[Generate] data for design calculations" (see Chapter III paragraph III.3.4). but it was not explicitly associated with the idea of developing ITT-procedures to characterize product properties. In terms of application scope, the focus was set on the quasi-static behaviour of structural elements, in pre-breakage and post-breakage stages (Figure IV.1).

The initial experimental goals can be rewritten in terms of the introduced EFI's :

1) EFI-Specimen: Material: interlayer SentryGlas (SG)
   (compared to reference material : PVB)
2) EFI-Specimen: Geometry + EFI-Test configuration: Geometry / Loading configuration / Loading range: 2 experimental scales: tests on a 'material' scale (tests on non-laminated specimens interlayers), and tests on 'element' scales, associated to 'pure' loading configurations (bending on weak axis and strong axis, torsion) and possible more complex loading configurations (buckling of laminated glass beams).
3) EFI-Specimen: Processing: (as delivered by the industry)
4) EFI-Specimen: Conditionning/Pre-treatment: (to fix for each individual test method / test configuration)

For the tests at element scale :

5) EFI-Test configuration: Basic device:
   - Test frames built for performing tests on elements in various loading configurations inside a climatic room;
   - Test on elements of larger dimensions to be executed on an existing frame, with use of IR-radiants for increasing the temperature;
6) EFI-Test configuration: Geometry: Limits imposed by the dimensions of the climatic room;
7) EFI-Test configurations: Loading configuration: 3- and 4-point bending tests, torsion tests;

8) EFI-Test configurations: Loading mode / loading ranges: relaxation and creep tests, control mode: imposed constant deformation (controlled by mechanical constraint) and applied constant force (controlled by fixed mass) respectively, including tests of long duration;

9) EFI-Test configurations: Measurements methods and devices: load cells, strain gages, extensometers with data acquisition system.

10) EFI-Test conditions: Tests at different temperature and relative humidity.

It appears quickly that the different EFI's here above are strongly interrelated in terms of development of test infrastructures and test methods. Also, as already mentioned in previous paragraph, the distinction between 'intermediate' and 'element' experimental scales was at an early step considered as a rather vague concept.

The feasibility and reliability of investigating the time-temperature response of structural elements of larger dimensions (bending tests on laminated glass beams of 3 m, with and without constraining a possible failure mode by lateral buckling) by means of radiant heating devices have been reported in (Belis et al. 2007).

The first part of the research purposes identified in the project[15] concerned the pre-breakage response of elements in different configurations. Experimental issues related to the investigation of the time-temperature dependence of the shear-transfer behaviour by means of tests on elements SG-laminates performed in a climatic chamber have been reported by Callewaert (Callewaert 2011; Callewaert et al. 2012)[16]. This research could perhaps retrospectively also be analysed with the proposed analysis grid; however, this tool was not yet developed at the time being, and consequently a similar analysis on the dedicated experimental campaigns has not been performed (so far). The corresponding campaign was constituted of an important series of relaxation and creep tests carried out on 'element' specimens SG-laminates, performed by means of imposed deformation level (by mechanical constraint) and fixed values of applied forces (by means of fixed mass bodies) respectively. Accordingly, the specifications of the climatic room built at that occasion had firstly considered the specifications peculiar to test configurations for which no steered loading device was necessary. Similarly, all the deformation measurements for these tests could be performed on a satisfying way by means of extensometers and strain gauges[17].

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[15] This FWO-project involved two doctoral researchers. The research efforts were more or less distributed according to the simple scheme "pre-fracture" and "post-fracture" behaviour between the two resulting doctoral theses. The first one has been defended in 2011 (Callewaert 2011).

[16] These experimental works have been conducted at the LMO between 2006 and 2011.

[17] These measurement devices belonged to the infrastructure and know-how of the lab already before the start of the research.

The experimental investigations related to the second research area (post-fracture performances) in the "orientation period" 2006-2010 are presented in more details. These consisted in a succession of experimental campaigns involving test configurations at different experimental scales, which are summarized on a time-line in Figure IV.5. The corresponding test results can be found in previous publications mentioned below, and are not reproduced extensively here, in order to focus the analysis on two complementary aspects : technical limits and issues for developing and extending test configurations for a *combination* of EFI's, and issues for interpreting test results in relation to the different border effects identified above.

The experimental developments are detailed below according to the three periods illustrated in Figure IV.5, and corresponding to a series of successive master theses. It is then followed by a complementary paragraph dedicated to a few complementary investigations and related considerations with regard to some developments of the TCT-test method.

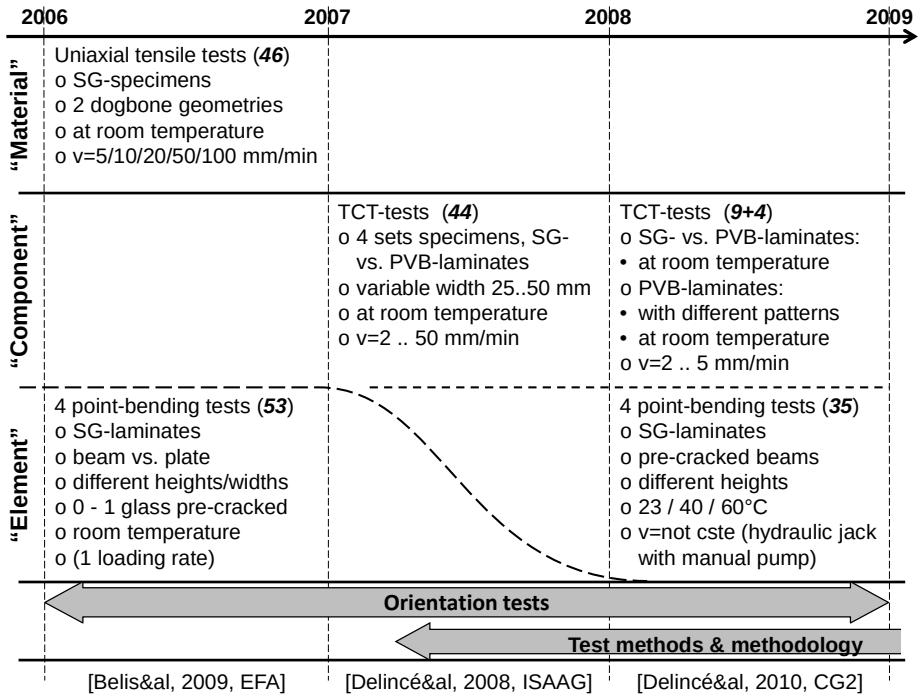

Figure IV.5 – Overview of experimental works conducted in the orientation phase for investigating post-fracture performances of SG-laminates used as structural elements. The 5 experimental campaigns have been performed on 4 different test infrastructures, using a variety of measurement devices and acquisition systems. The number in brackets indicates the amount of tests performed in each campaign.

IV.3.2.1. Experimental developments

- **Experimental campaigns during the first period** (2006-2007) :
  - 'material' scale was firstly investigated by means of uniaxial tensile tests on dog-bone specimens, cut out of sheets SG-interlayer; 2 geometries were considered (1 complying to the guidelines of the test standard regarding the geometry of the test specimens, 1 not). The tests were performed on an universal electro-mechanical testing machine equipped with a video-extensometer, at different loading rates (control: constant displacement rate) and ambient temperature (Belis et al. 2009).
    Issues and outcomes regarding the representativeness of this type of tests have largely been discussed in Chapter III paragraph III.3.2. There was however also a technical limit in extending the test temperature range for this type of tests : the used video-extensometer for measuring the axial stretch could not be used in combination with the climatic chamber (incompatibility between view angle of the optical measurement system and the geometry of the (mobile) climatic chamber); besides, this measurement device appeared to be a "closed black box" system rather conceived for standardized tests and not suited for full-field analysis, not even for measuring the transversal contraction of test specimen (whether necking would occur or not).
  - 'element' scale was investigated by means of a series of 4-point bending tests on specimens SG-laminates, about 1 m long and of different widths between 120 and 360 cm, made of two float glass sheets and one interlayer, loaded along their weak and strong axis (= 2 loading configurations), and two different initial states have been considered : elements with no or 1 pre-cracked glass sheet[18] (Belis et al. 2008, 2009; Delincé, Callewaert, et al. 2008).
    These tests have been performed at room temperature on a hydraulic testing machine (with fixed frame), and conducted at a moderate, relatively constant displacement rate.
    The pre-cracking step was made before mounting the specimen on the testing frame, and its description can thus be associated with the category EFI-Specimen: Pre-treatment/Conditioning[19].

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[18] These initial states correspond to the concepts of fractured states introduced in Chapter II.

[19] The execution method of the pre-cracking step during this first test campaign was *similar* to the one described in (Delincé et al. 2010) and detailed in Chapter V, paragraph V.2.2. This is an important difference with test protocols developed by other researchers, for instance by Bos, Louter and Kott (see Chapter II), where the cracking of the glazing sheets is part of the mechanical test (and where the fragmentation pattern is less controlled) : accordingly, field descriptors of EFI's associated to the cracking step can rather be associated to categories EFI-Test configuration / EFI-Test conditions.

- modeling : development of (relatively simple) analytical models showed that the same two mechanisms of interlayer stretching and delamination were present in fractured states of the two considered element configurations (plate and beam), which could be fitted on the experimental data. Fit parameters, namely model parameters which could not be determined experimentally, or not accurately enough, appeared as the height of the compression zone and the delamination length (x^*_{III} and a respectively for a plate configuration, see schema of Figure IV.6).
  These models appear useful for describing the experimentally observed trend in behaviour and show a correct quantitative order of magnitude of the influence of the various identified parameters on the overall response at the element scale; however, their prediction ability is still not assessed.

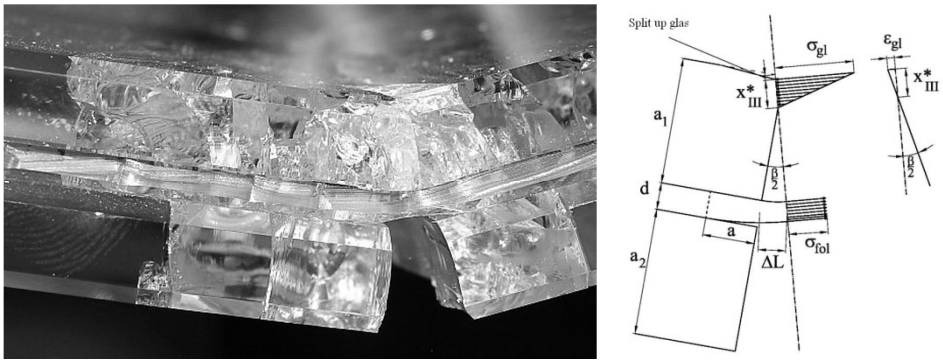


Figure IV.6 – Four-point bending test on a plate configuration (element scale) : experimental fractured pattern observed experimentally (left) and analytical model of the bridging behaviour in a cracked section (right) (Delincé, Callewaert, et al. 2008)

After this first experimental campaign, it was decided to not pursuit development efforts in further investigating effect of test temperature at 'material' scale by means of uniaxial tensile tests, with regard to identified difficulties for experimental developments, expected modelling issues and rising questions about the representativeness of test specimens (Chapter III paragraph III.3.2). The extension of the test temperature range appeared also as a constraint on the usable measurement methods, and the possibilities of using optical measurement methods in combination with a climatic chamber or room came into consideration.

In parallel, a complementary experimental goal was also defined for the next experimental campaigns : to avoid propagation of uncertainties related to hazardous crack propagation in the glass sheets during the loading step of the tests, in order to isolate the investigation of the ligament behaviour from these.

This led to work on 'stage III' pre-cracked elements in the next campaigns. Accordingly, qualitatively the frontier between experimental 'element' and 'intermediate'/'component' scales[20] is considered to have been crossed-over (represented by the curved dotted line in Figure IV.5 in the 'element' row).

- **Experimental campaigns during the second period** (2007-2008) :
  - the 'intermediate' scale began to be investigated by means of a series of TCT-tests, on four different test samples : specimens made with 2 interlayer materials, SG and PVB, and 2 processing methods for producing the small pieces of laminated glass. Some of the samples contained specimens of different widths; main characteristics of the samples are summarized in Table IV.4[21]. These tests were performed on an electromechanical testing machine, at moderate constant displacement rates, with use of a separated digital camera as vision system. The various experimental issues relative to this experimental campaign have been detailed in (Delincé, Sonck, et al. 2008).
    Among the various outcomes, it appeared that the basic assumption of weak interface was not observed for many specimens, and accordingly that breakage of the interlayer ligament began before a steady state could appear on the loading curve. Differences of response were not only observed between PVB- and SG-laminates, but also between specimens with similar geometry and same interlayer type of different samples, corresponding to different production and processing methods. An example is shown in Figure IV.7 with the comparison of deformation patterns of two different specimens SG-laminates. The first specimen (above in the figure) belongs to a sample obtained by dry cutting the specimens out of an 'older' laminated glass beam element with a height of 120 mm (namely from non-damaged part of an element used in previous experimental campaigns). The corresponding beam elements had been produced in industrial production conditions. The second (below in the figure) came from a sample prepared (lamination and further processing steps) by the quality control lab of the manufacturer of the interlayer product : the small specimens were sawn out[22] of laminated glass plates

[20] The possible ambiguity between the two concepts (intermediate/component scales) is purposely not suppressed here. The reason is that the concepts are likely to be implicitly interpreted or fulfilled otherwise when considering advanced numerical modelling development (Finite Elements Models with volumetric and interfacial cohesive elements). It is clearly a point of attention to further "interface" experimental and modelling issues.

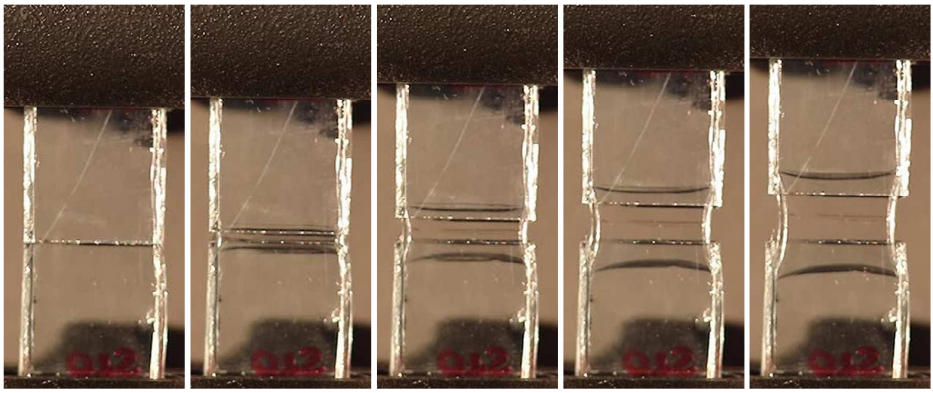
[21] The series "PVB-Sesh" referred to results of TCT-tests on PVB-laminates published in (Muralidhar et al. 2000).

[22] Sawing techniques for cutting pieces of glass or laminated glass required to be performed under steady flow of water, which is used as coolant.

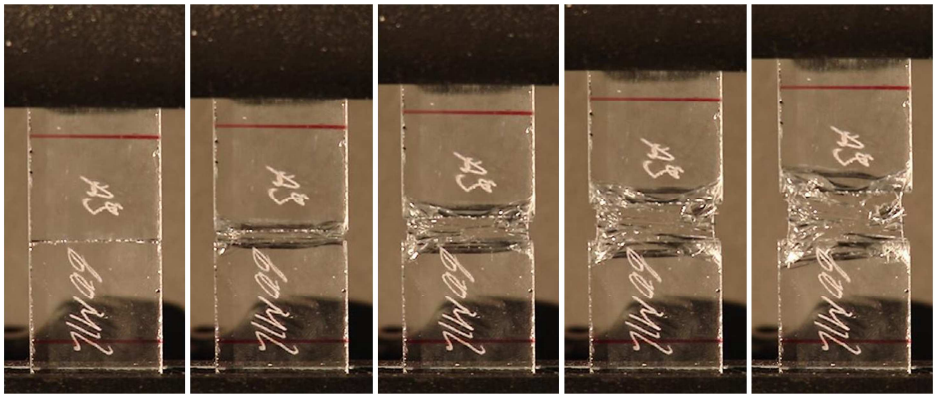
with dimensions 300x300 mm.
The difference in observed behaviour had been firstly allocated to a suspected difference of adhesion level between the two specimens, possibly influenced by complementary edge effects due to the respective cutting processes; an important unidentified EFI appeared meanwhile, which concerns a possible significant difference of physical ageing state (at least for the specimens with a SG-interlayer[23]). Similar significant differences were observed between the two samples PVB-specimens corresponding to the same differences in terms of preparation methods (see Table IV.4). It appeared however impossible to still refine the analysis of these experimental data *on a quantitative way* in order to review the previous conclusions.
The observations of different deformation patterns according to the material type and to the test conditions had also consequences on the further development of optical measurement methods. The measurement of delamination lengths appeared problematic for short crack opening range and in case of irregular deformation/delamination pattern.

- modelling : development in Simulia Abaqus of a 3D finite element model of the local bridging behaviour (TCT-configuration), mainly based on the work of Seshadri for PVB-laminates (see Chapter 2). This model uses cohesive elements with an assumption of mixed fracture modes, namely with same parameters for the three traction-separation laws corresponding to the three crack propagation modes applicable to interfacial delamination (‘mode I’ normal tensile force perpendicular to interfacial plane, ‘mode II’ and ‘mode III’ corresponding respectively to longitudinal and transversal shear stress with regard to the direction of the delamination front), and was tested with elastic, hyperelastic and elasto-plastic models for the interlayer material. Investigation of the robustness of the numerical model has been limited to the experimental geometry and loading configuration and mode.

[23] See Chapter III section III.3 for related theoretical background and Chapter V paragraph V.3.4 for further supporting experimental results.



a) TCT-specimen SG-laminate cut out of a laminated glass beam (“rough cut”)



b) TCT-specimen SG-laminate sawed out of a laminated glass plate (“fine cut”)

Figure IV.7 – Comparison of observed deformation patterns in a TCT-test for two specimens SG-laminates belonging to two different samples : a quite regular delamination pattern (above) and a more irregular delamination pattern with glass splinters remaining attached on the free part of the interlayer ligament (below) (Delincé, Sonck, et al. 2008)

Table IV.4 – Overview of TCT-test series by samples (Delincé, Sonck, et al. 2008)

Series	2h [mm]	b [3] [mm]	lat. edges	v [mm/min]	# tests [1]
PVB-LMO	1.52	25	“rough cut”	2	6/7
	1.52	50	“rough cut”	2	1/1
	1.52	30	“rough cut”	60	0/5
PVB-Dup	1.52	25	“fine cut”	2	0/4
SGP-LMO	1.52	25 … 50	“rough cut”	2 … 4	5/9
SGP-Dup	1.52	25	“fine cut”	2	5/6
	1.52	45	“fine cut”	2	1/3
PVB-Sesh	*0.76*	*25*	*n.a.* [2]	*60*	*8*

[1] amount of tests with steady state reached (before tearing of the interlayer) / total amount of tests in the series
[2] probably fine cut as well [3] nominal value of specimen’s width

Important outcomes at this step concerned the possibility of performing further TCT-tests inside a climatic chamber, and of further developing the optical measurement method in that perspective. Another sensitive experimental aspect had however been identified, which concerned the used clamping devices to fix the TCT-specimen on the testing machine (see also Chapter V paragraph V.2.5).

It led to change of basic testing infrastructure and of laboratory, because of these practical problems, but also in function of collaboration interests in developing the test and measurement methods, thus mainly in regard to the use of optical measurement methods in combination with a climatic chamber.

- **Experimental campaigns during the third period** (2008-2009) :
  - A first experimental campaign had as main purpose the realization of the switch from one test infrastructure to another one for the TCT-tests, and the adaptation of the optical measurement method to the refined experimental purposes. In this context, a variety of optical markers have been tested and evaluated with regard to the convenience of the method and the precision of the obtained measures.
    A difficulty for performing this step has been due to the fact that the climatic chamber was not yet installed on the test machine during this development step, and thus the forthcoming constraints had to be figured out. These were of two natures : constraints on the possible lighting conditions (usable light source and its position, thus its lighting angle; possible issues related to the presence of a window between the camera and the specimen; and other possible measures related to the control of the lighting conditions), and other constraints related to the future physical presence of the insulated box (for fixing the specimen on the machine, etc.).
    Another yet uninvestigated EFI during this research step concerned the robustness of the acquisition and measurement methods for different deformation rates.
  - The other parallel experimental campaign was based on a 4-point bending test configuration on pre-fractured[24] laminated glass beams (in-plane bending). The tests have been performed inside the larger climatic room at three different temperatures (23, 45 and 60°C) and on two different geometries, namely on specimens with two different beam heights (150 and 360 mm), all other characteristics remaining equal. For this purpose, a dedicated modular test frame has been conceived and built. The

[24] The execution method of the pre-cracking step has been described in (Delincé et al. 2010) and is also detailed in Chapter V, paragraph V.2.2.

application of the load on the element is achieved by means of a hydraulic jack, operated from outside the climatic room by means of a hand pump. Possible lateral displacements of the beam element due to lateral buckling were prevented by means of eight lateral supports covered with a low-friction plastic sheet, placed between the support and the loading rolls on each side of the beam. Deformations were measured by means of two complementary measurement methods : a series of extensometers on the one hand, and a optical vision system with as main hardware component one digital camera placed outside the climatic room. The description of the test configuration, some of the related experimental issues and test results have been presented in (Delincé et al. 2010). In summary, the deformations patterns were relatively different in function of the test temperature, altogether the resistance (maximum reached value in the applied load) varied significantly between the six test series (with a factor about 1/3 between tests carried out at 60 and 23°C).

This test configuration is very similar to the corresponding one considered for the "beam" tests performed at room temperature during the first period (and presented here above), however with a noticeable difference in the considered initial fracture states[25]. Contrary to the previously considered initial states, only TCT-configurations were considered, namely the two glass sheets of the element are pre-cracked along a same transversal cross-section previously to the mounting of the specimen on the test frame. This difference is sufficient to consider that we are facing a change of experimental scale, from 'element' to 'intermediate' scale. Indeed, with this new initial specimen configuration the problematic of crack propagation in the glass sheets during the test are expected to be eliminated, or at least strongly reduced. A schematic comparison of observed cracking patterns is given in Figure IV.8 : in case only one glass sheet is pre-cracked, three different crack paths are observed, whereas for a TCT-configuration, only one of these three patterns develop during the test, under the effect of compressive stresses between glass fragments at the upper side of the beam.

- modeling : the robustness of the finite element model developed for the TCT-test configuration has been investigated, by modifying the loading configuration and the boundaries conditions in the model, and it showed a satisfying response (capacity of convergence of the results).

[25] Concepts of fractured states have been introduced in Chapter II, section II.3..

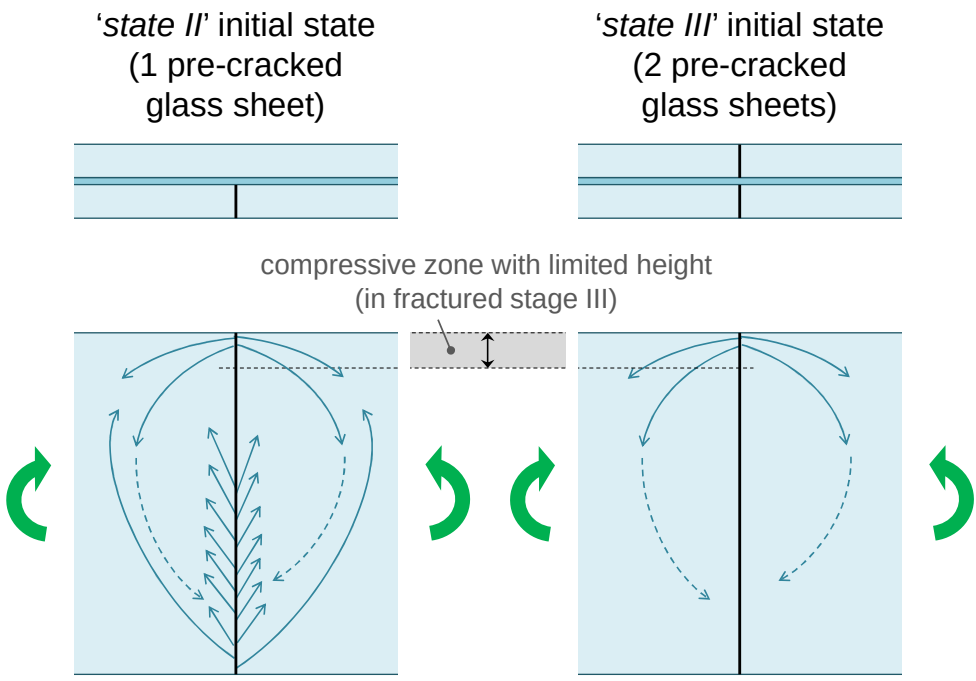


Figure IV.8 – Typical crack propagation patterns in glass sheets in function of the initial fractured state of a laminated glass beam element (4-point bending test) : initial configuration with 1 pre-cracked glass sheet (left, first period) and with 2 pre-cracked glass sheets (right, third period). The four thick arrows represent the applied bending moment; the light curves represent the observed crack propagation paths in glass sheets.

It is useful to mention that the choice of this 'element scale' test configuration for investigating temperature-dependent behaviour of fractured laminated glass elements has been influenced by considerations regarding the development of the optical measurement method[26]. In fact, in this constrained bending test configuration, the displacement of the two parts of the glass beam remain in a fixed vertical plane, what allows to use a single camera and a two-dimensional (2D) computer vision system. For test configurations with significant out-of-plane displacement (as it is the case for instance with a bending test on a plate configuration, or an unconstrained bending test on a beam element allowing failure by lateral buckling), it would require to move towards a three-dimensional

[26] The choice was also determined by an 'usual research approach' consisting in developing 'pure loading configurations', namely test configurations generating a 'pure effort' in a 'zone of interest' of the tested element, as a pure compression, pure bending, pure torsion, etc.

(3D) vision system for applying an optical measurement method[27] (Sutton et al. 2009).

One major weakness of the test configuration used for the bending tests on pre-cracked beams inside the climatic chamber (Galmart and Matthijs) was the poor control on the applied loading rate provided by the use of a hydraulic hand pump. An estimation a posteriori of the applied displacement rates (average rate of applied vertical displacement at the level of loading rolls up to peak force) showed variations of up to a factor 10 inside series, and up to 100 between different test series; the loading rate tended to be larger for elements with lower stiffness (namely test configurations with lower height of beam or higher test temperature). Investigation of possibilities to use a loading device equipped with an actuator inside the climatic chamber raised technical issues, followed by financial ones; this was one of the main limiting factor that led to interrupt this type of experimental investigations and to focus on the further development of the TCT-test method.

IV.3.2.2. Other experimental investigations with TCT-tests

Further experimental investigations concerned mainly the development of optical measurement methods for TCT-tests, and in a lesser extent tests on specimens with different interlayer materials. This experimental campaign has been characterized by a larger amount of more closely involved extern partners for its preparation, in comparison with the ones reported in previous paragraph (see Acknowledgements section at the begin of this chapter).

An attempt has been made to make a full-field measurement of the ligament deformations by using a DIC[28] method. For this purpose, special TCT-test specimens were prepared, with a speckle pattern printed "inside" the interlayer. In fact, the pattern was directly printed on a first PVB-film, which is covered by a second PVB-layer with the same thickness during the lamination. This way, the optical measurements should correspond to planar deformations of the interlayer at the level of its median plane.

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[27] Such a 3D vision system was not available nor affordable at the time being. However, it is not yet totally clear if the use of such a stereoscopic method is applicable 'as such' with view angles of the cameras passing through an insulated glass unit (the window of the climatic room); there could be still complementary experimental issues in investigating further in that direction…

[28] Digital Image Correlation (DIC) refers to optical measurement methods using an image matching algorithm, which involve the recognition of defined patterns on successive frames (pictures). An important sub-category of DIC-methods allows to make 'full-field' measurement of deformations, leading to a measurement result in the form of a strain field, thanks to the use of a 'speckle pattern' (pattern of randomly distributed black points or areas on a white background) printed or projected on the surface of the test specimen.

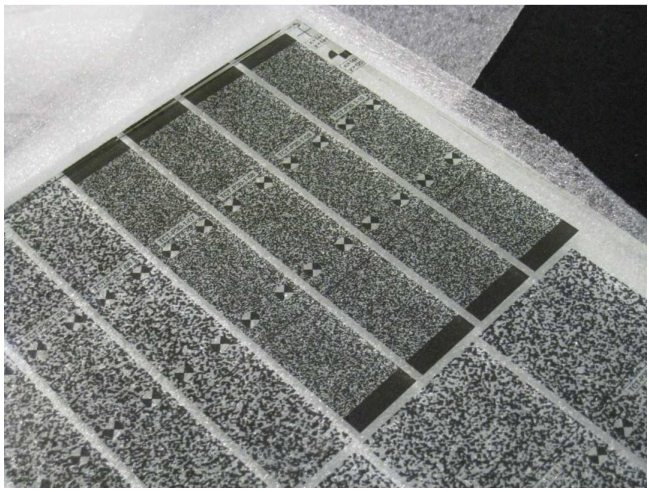

Figure IV.9 – Laminated glass plate with pattern for optical measurement, printed on the median plane of the interlayer, made of 2 superimposed PVB-films (the picture shows the plate before the cutting of the small specimens)

Two laminated glass plates were prepared by the manufacturer of the interlayer product with dimensions 300x300 mm and constituted of two 4 mm thick glass sheets and one interlayer made of 2 superimposed PVB-films of 0.76 mm, thus a total nominal thickness of 9.52 mm[29]. The printed patterns delimited 30 TCT-test specimens with two different widths (30 and 50 mm) and with two different scales of the speckle pattern (Figure IV.9). The small specimens for the TCT-tests were cut out of the laminated glass plates by combining sawing and manual cutting techniques[30]. Based on previous results of TCT-tests on PVB-laminates (see previous paragraph), an extra specification had been suggested to the manufacturer in charge of the lamination process, namely that the adhesion level should be "at the lower side" of the acceptable adhesion range (according to their intern standard method), to promote a regular delamination pattern[31]. Obtaining this type of deformation pattern was thought as a necessary condition for successful full-field optical measurements in the ligament area.

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[29] With the production tolerances on the thickness of the float glass sheets and the lamination tolerances, the effective thickness of a laminated glass plate is generally slightly thinner; in this case, the measured thickness of the small specimens after the cutting step is around 9.30 mm (see also comments about border effects due to measurement uncertainties in section IV.4).

[30] The printing of the speckle pattern on the PVB-interlayer has been performed by means of DuPont's 'SentryGlas Expression' patented technology (which, despite its commercial denomination, uses PVB-interlayer and is not applicable for SG-interlayer…).

[31] Failure modes in TCT-tests are described with more details in Chapter V paragraph V.3.1. The practical measures to meet this requirement were left to the discretion of the laminator (see also Chapter III paragraph III.3.3).

The testing of these 'customized' TCT-specimens was performed at room temperature and moderate displacement rate using the same testing device as previously. The computer vision system is separated into an acquisition system of the digital pictures during the test, which are used in a post-processing step for performing the optical measurements. The optical acquisition system is constituted of one PixeLink digital camera and its dedicated acquisition software (described with more details in Chapter V, paragraph V.2.6).

The different deformation and measurement zones are illustrated on a picture of the deformed TCT-test specimen as used by the vision system in Figure IV.10. The measurement of the crack opening by means of four target markers (two on each side of the initial pre-cracked section of the TCT-specimen) is performed successfully with a satisfying precision. According to the deformation pattern of the TCT-specimen, three zones of the ligament can be distinguished : the two delamination fronts, and the central, delaminated part of the interlayer ligament which appears between the glass fragments once the crack opening is getting large enough.

The processing of the pictures for performing the optical measurements has been realized separately by two different persons, on the one hand by means of a commercial "closed" vision software and on the other hand by means of an "open" analysis routine. The conclusions about the applicability of the optical measurement method seemed similar. Unfortunately, it appeared impossible to get reliable measurements of local deformations of the ligament by means of the speckle pattern. In the central zone, signs of irregular deformations appeared for relatively short crack opening, apparently due to initiation of crack propagation through the thickness of the interlayer. The two delamination fronts have a relatively regular shape; nevertheless, derivation of local strain measurements from the acquired digital pictures in the vicinity of the delamination fronts appeared as problematic, because of a low correlation coefficient[32] in these areas. The failure of the correlation in the ligament zone is attributed to severe distortions of the speckle patterns on the local subsets. It appears however difficult to estimate whether these shortcomings could be overcome by working on the DIC-algorithm and/or by adapting the patterns[33]. Nonetheless, a satisfying correlation was obtained between speckle patterns situated outside the ligament zone for measuring the effective crack opening.

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[32] The correlation coefficient (R^2) is a measure of the success grade of the image matching algorithm, and is calculated on 'subsets' of which size is an important adjustment variable in obtaining accurate measurement results. When this correlation coefficient is getting below a certain limit, no reliable measurement of the local deformations (strain) can be expected; and this condition has to be related to the required or expected accuracy level.

[33] Results by means of a similar method using an alternative pattern constituted of a regular dots grid has been reported in (Butchart and Overend 2012).

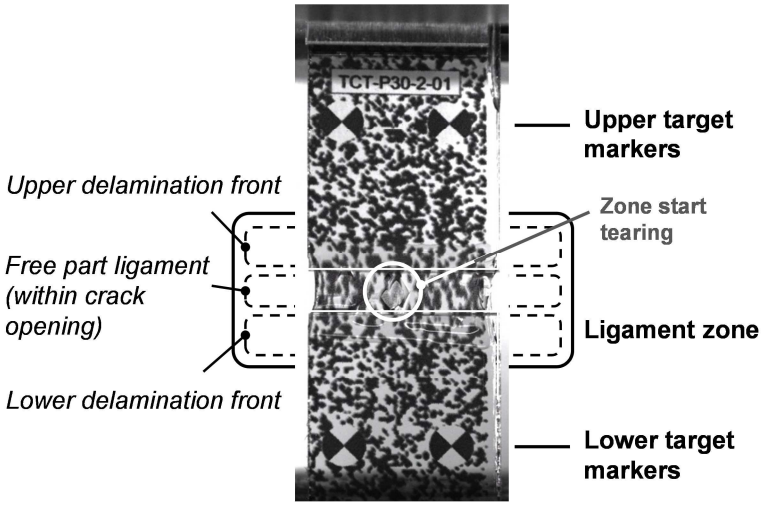

Figure IV.10 – TCT-test specimen (width b = 30 mm, thickness interlayer t = 1.52 mm) with speckle pattern printed 'in' the interlayer, and distinction of different zones for application of DIC method for the measurement of the deformations

There were different complementary motivations for not pursuing the investigations into the development potential of this type of full-field optical measurement method.

The first concerned priorities and related technical constraints in developing the experimental method : the primary objective was that the optical measurement method should be applicable in combination with a climatic chamber, for performing tests at different temperatures. The reliability and the precision of full-field measurements can depend on the used DIC-algorithm and analysis parameters (calibration of the vision system, size of the speckle subsets, etc.), but they depend at first on the initial quality of the 'raw' material, that is the digital pictures. However, the degradation grade to be expected due to the combined use with a climatic chamber was still unknown. Different sources of degradation of image quality effectively appeared later on : these are described in detail in Chapter V paragraph V.2.6.

The second reason rather addresses possible loss of representativeness of test specimens due to the application of the optical pattern on the interlayer. Indeed, the application of a speckle pattern *into* the polymer component of laminated glass specimens involves modifications of the production process[34], potentially inducing deviations in some fields of the category EFI-Specimen. Alternative methods could be imagined where the optical pattern would be applied to one of

[34] The used method for printing the pattern on the PVB-films is however believed to have been developed in order to reduce this type of effects, but it remains a qualitative assumption…

the surfaces of the interlayer film coming into contact with a glass sheet in the laminated unit. Still, these would probably also affect the resulting adhesive properties, and the pattern could behave poorly with the delamination process, potentially leading to even larger border effects. A more objective quantification of such border effects could be done by means of a complementary reference sample (another basic laminated glass plate with the same interlayer material without print), processed the same way (ideally simultaneously) and tested in the same conditions. Besides, such methods are only applicable to a limited family of interlayer products.

Any development of this nature involves thus firstly potential deviations in some investigation fields of the category EFI-Specimen, which raise issues of representativeness of the test specimens, before eventually allowing extension of the EFI-Test configuration: Measurement range (in combination with extension of EFI-Test conditions and EFI-Test configuration: Loading range). Measures should thus be foreseen to check that the balance remains favourable between the expected benefits, among others in terms of precision and accuracy of the measurement results, and possible induced degrading border effects. With regard to this problematic and the assessment perspective, the priority should be to avoid the introduction of border effects in fields of the category EFI-Specimen.

Consequently, in following campaigns TCT-tests, the use of DIC-method has been reduced to the detection of four target markers to measure the crack opening. Corresponding experimental issues, among others for determining precision and accuracy of the obtained measures, are further developed in the next chapter.

The use of a post-processing analysis scheme, with separated acquisition and analysis processes[35], is preferable for two reasons. Firstly, the acquired pictures can also be processed for qualitative observations (deformation and failure patterns, etc.) or even for developing alternative detection and measurement algorithms afterwards. Secondly, they can be used for controlling or optimizing the quality of the measurements obtained with the DIC-algorithm. There are also two counterparts to this working method : the optical measurement cannot be used as steering parameter (for instance for controlling the effective crack opening rate), and the achievable acquisition frequency can be limited by the registration process (according to the used acquisition system and hardware…). This last point can represent an issue for using such a method in a range of larger deformation rate.

[35] Post-processing analysis : the acquisition and registration of the digital pictures is performed during the test, and the application of the DIC-algorithm on the acquired series of pictures is made after the test.

IV.3.3. Development phase (2010-2013)

This last experimental phase consisted in an experimental campaign TCT-tests which is the topic of the Chapter V. In terms of EFI's, the investigated ranges can be summarized as follows : the ranges of EFI-Specimen were reduced to a minimum, namely one type of interlayer material (SG), one single sample in terms of production method and of configuration (thickness layers, type glass sheets,…), and investigation ranges were extended in the categories EFI-Test configuration: Loading modes and Loading ranges, EFI-Test conditions: Temperature, and related EFI-Load configuration: Measurement methods.

IV.4. Border effects due to measurement uncertainties

The analysis grid with EFI's can also be used for getting an overview of experimental uncertainties and other sources of systematic deviations for a particular experimental configuration. The field descriptors were firstly presented as experimental variables to identify and define, secondly as ranges of values for describing possibilities and limits of experimental investigations, in other words for defining the possible experimental investigation scope of a test method. In this sense, border effects were associated to differences between representative values of 'real' physical parameters, let say significant differences of values, or more specifically a difference of values at the level of one specific EFI-parameter with a significant effect on another EFI- or AF-parameter, among which the properties of the product or the application configurations respectively.

By changing the analysis level, a range of values for a parameter used as field descriptor can also be used to express uncertainties on a parameter for a specific test. The analysis level is changing, so does the range of values.

The majority of the parameters used as field descriptors are the result of a measurement, and the associated values are thus vitiated with *measurement uncertainties*. When the parameter is not resulting from a direct measurement, the associated uncertainty is resulting from a combination of measurement uncertainties, used to describe errors propagation.

For many fields, as measures of dimensions, distances or deformations, the measurement uncertainty is generally a constant and is not proportional to the measured parameter; accordingly, the relative systematic deviation becomes larger when the measured parameter is getting smaller[36].

[36] Concrete examples of such systematic sources of deviations or measurement errors on some parameters appearing in the TCT-test configuration for measuring the deformations are identified in Chapter V paragraph V.2.6.2. An order of magnitude of the systematic measurement error (accuracy) is estimated for identified parameters for the particular test configuration used.

Tests on laminated glass specimens of smaller dimensions (as for CST-tests and TCT-tests) are thus confronted with two different types of potentially larger border effects than tests on elements with larger dimensions. The first is associated with larger physical effects on test specimens due to their configuration and dimensions (including production tolerances, edge effects,…); the second is due to larger values of relative uncertainties on some measured parameters. Such larger border effects for specimens of small size are however also compensated by the elimination or reduction of other experimental uncertainties, as the ones associated to glass strength[37] and to random crack propagation patterns in glass sheets.

One such important border effect due to propagation of measurement uncertainties for laminated glass products and test specimens concerns the measurement of the thickness of the interlayer component. The thickness of the laminated interlayer results from an indirect measurement, following next expression for a 'simple', symmetric laminated glass unit :

$$t_{int} = t - 2.t_{gl} \tag{IV.1}$$

with t, t_{gl} and t_{int} respectively the total thickness of the laminate, and the thickness of the glass sheets and of the interlayer. The first two can be obtained by a direct measurement of the thickness of the constitutive glass sheets before lamination and of the total thickness after lamination. Counting with a typical measurement error range of $a(t) = \pm 0.02\ mm$ for each individual measure of thickness[38], the corresponding uncertainty, expressed as a standard deviation, is $u(t) = 0.02/\sqrt{3} = 0.0115\ mm$ for a rectangular distribution of the measurement error. A calculation of the combined measurement uncertainty on the interlayer thickness gives :

$$u(t_{int}) = \sqrt{u(t)^2 + 2.u(t_{gl})^2} \tag{IV.2}$$

and thus $u(t_{\text{int}}) = 0.02\ mm$, or expressed as an extended measurement uncertainty $U(t_{\text{int}}) = k.u(t_{\text{int}}) = 0.04\ mm$ (with k=2 for a 95% confidence interval). With regard to usual interlayer thicknesses for PVB-laminates, this

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[37] See for instance some definitions of residual resistance in Chapter II paragraph II.3 which consider, to determine the initial resistance R_0 of a laminated glass element, the characteristic strength of the constitutive glass components. The initial strength being already a derived variable, which is known to shows up with large scattering in values because of its intrinsic sensitivity to surface defect, the initial reference value is already a "vague" one…

[38] This value of measurement error corresponds to the typical one obtained with a micrometer calliper.

leads to relative uncertainties of about 10 % (0.38 mm), 5% (0.76 mm) and 2.5 % (1.52 mm)[39]. When modelling the experimental configurations involves the calculation of the flexural stiffness of glazing components, the uncertainties on the thickness of glass components propagate with a factor three (as the flexural stiffness of the glazing sheet depends on the third power of its thickness).

In practice, the effective thickness of the constitutive glass sheets is often only known with a lower level of accuracy: standardized production tolerances on laminated glass products (according to EN ISO 12543-5) are an order of magnitude larger. In order to compare with the previous numbers, standardized tolerance on thickness of a float glass sheet is about 5% (for instance : 4±0.2 mm, with an effective average value round 3.85 mm), and the tolerance on laminated glass products with folio interlayer is the sum of the tolerances on thickness of the constitutive glass sheets, with an extra tolerance of ±0.2 mm when the total thickness of interlayer components is larger than 2 mm[40].

This induces also that the expression of uncertainties on a parameter is likely to deliver another order of magnitude when it is used as AF-descriptor or EFI-descriptor, namely in function of whether tolerance or measurement uncertainty is addressed, respectively. Logically, the first one should always be (an order of magnitude) larger than the second one.

The analysis of experimental uncertainties for non-conventional test methods in literature is usually made by means of a "top-down" approach, in the form of a statistical analysis performed on test results of series considered as homogeneous (typically resumed to a calculation of averages and standard deviations, generally limited to a resistance or strength parameter). However, such a method does not allow detecting systematic deviations or bias in the measurements or in derived parameters. Inventory of data necessary for performing a "bottom-up" analysis (based on evaluation of individual uncertainties and calculation by combination of their propagation) is seldom performed, for different reasons. In particular, a series of (systematic) experimental uncertainties can often not be better than roughly estimated (they do not have a statistical meaning), and the execution of

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[39] This type of analysis complies with guidelines of the basic guidance document "GUM, Guide to the Expression of Uncertainty in Measurement (1994)", referred by many posterior documents, among others by the general standard ISO/IEC 17025 (2005) "General requirements for the competence of testing and calibration laboratories".

[40] The comparison of the order of magnitude of systematic measurement errors on thickness of glass sheets with, for instance, the precision level considered in literature and in discussions about the determination of characteristic values of glass strength delivers interesting information. Properties of interlayer components derived from mechanical tests on laminates appear still more sensitive to the influence of measurement uncertainties. Nevertheless, issues related to measurement accuracy (more particularly with regard to *systematic* errors) and to combination of uncertainties are generally not or poorly highlighted in test standards and in literature.

any kind of “bottom-up” analysis requires the choice or development of a mathematical model (analytical or numerical). Consequently, the calculated uncertainty is eventually affected by supplementary systematic deviations, which can be due to deviations in input parameters or due to numerical issues.

These reasons are however not justifying that inventory of experimental uncertainties would be disregarded. Analysis of uncertainties and of their propagation is certainly an interesting tool for estimating achievable precision and accuracy which can be expected from a test method, and for evaluating its extension potential, in terms of testing ranges or in terms of analysis level, especially when considered results are derived parameters calibrated by indirect measurement. This inventory cannot avoid errors in allocation of systematic deviations to each type of border effect, but a rigorous and clear distinction of the different analysis steps are certainly useful, or even necessary, to deal with these issues. Underestimation of effective measurement uncertainties and overlooking possible effects of propagation of uncertainties can be seen as a particular case of unidentified EFI and associated border effect. However, this type of border effect can generally only be addressed in a minor extent by the design of the experimental setup (test and measurement configurations) and other measures of experimental nature, and requires an equivalent investment level in the related analysis and reporting steps to effectively improve the global accuracy.

Border effects due to significant values of systematic deviations resulting from combination of uncertainties and of consecutive propagation of errors are considered as a *third category of border effects*. They can be the consequence of border effects of the two first categories, when significant deviations arise in derived parameters from a combination of non-significant deviations on primary experimental parameters. They can also be solely due to the selected model to make the derivation, in which case they are due to model uncertainties. The latter case can be illustrated by two typical situations already presented and discussed in earlier chapters. The first is the use of small-strain theories for deriving values of stress or strain when the range of deformation of the material clearly lies in a large strain domain (Chapter III paragraphs III.2.2 and III.3.2). The second is the negligence of size effects caused by an abusive acceptation of assumptions of small-scale yielding or small-scale creep (Chapter II section II.4).

The latter considerations have not only influenced the further development of the campaign TCT-tests on SG-laminates reported in Chapter V, they also influenced the selected ‘analysis level’ of the test results.

IV.5. Summary and main outcomes

In this chapter different *experimental scales* have been distinguished for test configurations on laminated glass elements. In particular, firstly a qualitative distinction has been introduced between 'element' and 'intermediate' experimental scale. *Robustness* and *representativeness* of test methods are proposed to be analysed by means of an analysis grid based on the identification of different *Experimental Fields of Investigation (EFI)*, regrouped into different categories and described by means of appropriate field descriptors. In particular, possibilities and limits to extend the applicable investigation range of test configurations are shown to depend on technical issues related to test devices and measurement methods on the one hand, and to test specimens on the other. The analysis grid allows also to identify and distinguish different sources of systematic deviations between EFI's or between EFI's and AF's, which can limit the representativeness and thus the relevancy of test configurations, and three categories of *border effects* are identified accordingly :

1) The *first category of border effects* addresses the deviations of properties related to the properties of products and test specimens, due to the production process, storage and pre-treatment conditions, and geometry of the test specimens, in particular in relation with specificities of the polymer component;

2) The *second category of border effects* is related to deviations due to experimental aspects associated to test configurations and test infrastructures, among others in terms of geometry of specimen and loading configuration, and the sensitivity of test configurations to dimensional and position tolerances;

3) The *third category of border effects* addresses issues arising from processing and analysis of test results, as consequences of measurement uncertainties (for direct and indirect measures) and propagation of errors for derived parameters (obtained by indirect measures). This type of border effect can be a consequence of other ones of the two first categories, but can also follow from significant deviations and effects resulting from the used analysis method and model.

Because of the high grade of interactions or interdependencies between some EFI's, the use of an analysis grid structured around related field descriptors can help in anticipating possible requests and related technical limits for extending the achievable investigation ranges of any test method. As illustrated in this chapter with a few examples, extension of test methods can face serious limitations of practical and experimental nature, in particular when the development of a test configuration addresses the extension of some EFI's *in combination* with other ones. This is especially the case when an extension of the test temperature range is required or is likely to be requested. This is generally true for developing experimental investigations methods, but it takes a new dimension when it

happens in the context of construction products or building applications, in relation to the different types of specificities explained in previous chapters.

It has also judged been useful to introduce the concept of 'unidentified EFI'. This acknowledge that some phenomena related to the nature of the tested interlayer component, or experimental aspects depending on processing methods of test specimens, are possibly overlooked or not identifiable *during* the development and execution of experimental works on laminated glass products.

These are typically addressing possible changes of the assumed state of test specimens through the various experimental steps, typically with regard to the parameters which have been added in previous chapters to describe physical ageing state and damage level. The interest of this concept is to provide a back-up opportunity for subsequent analyses, by associating beforehand different parameters to different suspected sources of potential border effects.

The different border effects can be dealt with in various ways. Some border effects of the first category can be detected by inspection of the test specimens or their influence can be observed in the dispersion of results of test series. Others cannot be detected by any of these two methods[41], when they are not visible (or not measurable by a state variable) and do not involve systematic deviations in results of test series. Border effects in general can be distinguished and estimated by adapting test programs and analysis methods. They can be reduced by adapting test configurations and test protocols, to avoid that too many EFI's are varying simultaneously between two considered experimental scales.

The proposed analysis grid of EFI's can still appear as rather conceptual in some extent, similarly to the corresponding one defining the categories of Application Fields (AF's). Its main purpose is to serve as a structured framework to better objectivize the evaluation of different test methods and experimental approaches and their comparison. It can among others be helpful to avoid unnecessary detailed sub-optimized precision on some investigation fields which tend to neglect other arising issues. In fact, modifying the investigation ranges of some EFI's can have consequences on other ones, and an expected reduction of border effects of one category can give rise to larger border effects in another one. Efforts in reducing identified border effects should thus be balanced with efforts in quantifying the various effects, with regard to the various types of uncertainties on the one hand, and with regard to the technical issues for extending some experimental fields of investigation on the other. Not every single border effect associated to a test method can be reduced or eliminated, but it is also probably not even necessary, with respect to the specified test purposes and analysis level.

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[41] Or their detection by inspection of test specimens would require the use or development of complementary investigation methods (for instance some non-contact measurement methods).

Efforts in identifying and quantifying the various border effects can help to orient the development of models, of test methods, and of strategies for dealing with the different types of uncertainties in design conditions.

A definition of unambiguous criteria to distinguish ‘intermediate’ and ‘element’ experimental scales appears not straightforward. However, EFI’s appear to be a useful expression tool for refining the analysis, and possibly in developing a more robust definition of ‘intermediate’ experimental scales.

The concept of ‘intermediate’ experimental scale addresses different experimental aspects, in relation with border effects associated to different categories of EFI’s :

1) Size of the test specimens : tests on specimens of smaller dimensions are confronted to possible larger border effects associated to the category EFI-Specimen, and to related effects in relation with test conditions (described by EFI’s of the other categories). For small test specimens cut out of larger units laminated glass, extent of border effects before, during and after the cutting step should be distinguished (for instance with regard to intended or unintended ageing processes);
2) Investigated field(s) in relation with the purpose(s) of the test : identification and measurement of a limited amount of mechanisms and/or of ‘material/product/element’ mechanical properties or performances. For each performance/property associated to a test configuration, achieved or achievable precision and accuracy of test results should be compared with desired ones… or conversely.

Determination of ‘intermediate’ experimental scales is thus clearly not only about size of the test specimen, and has to deal with different sorts of border effects.

In particular, the development of the TCT-test configuration through the successive test campaigns has been performed with regard to the different identified border effects. A more detailed analysis of the different aspects raised in this chapter is performed in Chapter V on the basis of an experimental campaign on a sample of specimens SG-laminates. A more general synthesis about the robustness and the representativeness of the TCT-test method for characterization purposes is made in Chapter VI section VI.3.3.

Chapter V

Investigation of time-temperature dependent ligament behaviour by means of TCT-tests

"The Devil is in the detail / God is in the detail"
(Anonymous / attributed to Ludwig Mies van der Rohe, architect, 1886-1969)

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V.1. Introduction

Experimental investigation of time-temperature-ageing dependent behaviour of fractured laminated glass elements appeared to address a variety of issues, on the one hand with regard to a specific context, the characterization of properties and residual performances for designing non-conventional building elements and systems (Chapter II), and on the other with regard to specific mechanical behaviour of polymer materials used for interlayers and related production processes of laminated glass units (Chapter III). Finally, general methodological aspects and practical issues related to different experimental configurations have been presented and discussed in previous chapter (Chapter IV). These various aspects led to select one specific test configuration, the Through-Crack-Tensile test (TCT-test), for investigating the time-temperature response of the corresponding load-transfer mechanism (TCT-LTM) on small-size pre-cracked specimens laminated glass (TCT-specimen).

This chapter reports on the conception, preparation and execution of an experimental campaign of TCT-tests on small specimens laminated glass made with a specific interlayer product, and designed as SG-laminates[1]. The purpose of the campaign is the investigation of the time-temperature dependent behaviour of the interlayer ligament and the characterization of the ruling product properties. The initially defined framework accounts for a limited amount of test pieces of about 60 TCT-specimens, with the aim of covering ranges of test conditions as large as possible, in terms of *combinations* of loading level and of test temperature. The two loading modes considered (constant displacement rate of crack opening, and constant force or creep) accounted for defined limitations to quasi-static loading ranges (displacement rates < 100 mm/min), and a temperature range between -20°C and +60°C is considered. The test specimens were limited to one batch laminated glass with a single interlayer thickness, identified as SG35.

These objectives led to develop an incremental experimental approach constituted of short test series in a dozen of successive steps, separated by intermediate analyses for refining the testing conditions of the following steps. With regard to the identified phenomenon of physical ageing in solid polymers below their glass-transition, short test series have been dedicated to the influence of different conditions of storage and conditioning of the test specimens.

The obtained test results provide a first global overview about combination of effects related to time-temperature-ageing dependent properties of SG-interlayer with regard to the ligament response in fractured laminated glass, and highlight the importance of some experimental aspects with this type of tests. Also more general comments are included about possible issues in using TCT-tests with other types of interlayer products.

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[1] Characteristics and features of this interlayer product have been presented in Chapter III.

V.2. Test method and experimental strategy

V.2.1. Design of the experimental campaign TCT-SG35

The initial objectives and constraints defined here above led to consider for this campaign TCT-tests tests of (relative) short duration for two loading modes, performed at constant test temperature within a initially defined range between -20 and +60°C :

- **cdr-tests** carried out at constant displacement rate $\dot{d}$ (of the initial crack opening), with an initial range of values comprised between 0.01 and 100 mm/min imposed by the used testing machine; and
- **creep tests** corresponding to a constant value of applied force F_{cr} .

Table V.1 – Overview of experimental program by test series (as executed)

test temperature	cdr-tests	creep tests	executed at
-20°C	s9 (0/3)	*	TU/e
0°C	s7 (3/3)	s8 (3/5)	
20°C	s1(a0) (4/6) s1b (3/3) s1(a1) (3/3) s1(a2) (2/2)	s2(a0) (4/5) s2(a3) (2/4) s2b(a3) (3/3)	UGent
40°C	s5 (5/6)	s6 (4/5)	
60°C	s3 (5/6)	s4 (2/2)	

Legend and notes :
- sX (b50) / sXb (b30) : wider / smaller specimens series (b = 50 / 30 mm);
- (A/B) : amount of successful tests (A) and total amount of tests (B) for the series ; the differences account for failed tests and rejected test results at the analysis (see details about criteria for rejection in the main text)
- sX(aY) : aY identifies sub-series of specimens with different storage age or with complementary conditioning treatment (see details in paragraph V.3.4)
- sX : X refers to the initial order in test series (see details in section V.2.3)
- * : no creep test has been performed at -20°C

The limitation to quasi-static loading conditions implicitly involves a lower limit for the test duration and an upper limit for the loading rate (to avoid dynamic effects), with a time-to-failure typically larger than 1 minute. The short duration character accounts for two aspects, namely the purpose of measuring the relative contributions to failure modes of two deformation mechanisms (stretching and delamination of the interlayer ligament) and with purpose to avoid progressive

physical ageing[2] during the test, resulting in a target upper value of at most a few days.

Complementary identified objectives for this experimental campaign are :

1) to develop an optical measurement method for measuring the crack opening and describing the deformation mechanisms for tests performed inside a climatic chamber;

2) to assess the accuracy and the precision of the test results, and their representativeness in the considered ranges of test conditions; for this purpose, a distinction between primary (measured) and secondary (derived) experimental parameters is considered[3];

3) to assess the representativeness of the test results in relation to their sensitivity to three types of border effects associated to different sources of systematic deviation[4], with regard to possible effects due to the sampling method, preparation method of test specimens and storage conditions, specificities of the test configuration, and analysis and processing methods of the test results;

4) to evaluate to what extent results of short duration tests allow to predict in some extent the long-term behaviour (with respect to ageing effects).

These various aspects fit with the more general purpose of assessing the TCT-test method in relation to experimental strategies for characterizing product properties or performances. Because this focus on the assessment of the test method in different test conditions already addresses a series of aspects and in order to assume a minimal influence of uncertainties due to difference between test specimens, a single production batch has been considered (related details are summarized in paragraph V.2.2 below).

The campaign has been conceived in an incremental way, namely as a succession of a dozen test series : the specific test conditions for a test series were determined on basis of the results from the previous ones, on basis of parameters identified as having a potential significant effect on the test results. A test series regroup tests of a same loading mode (cdr or creep) performed at a same test temperature. The complementary test parameters to determine before the launch of one test series were the applied displacement rate or the applied creep force according to the loading mode, which were completed by the storage duration and exposure

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[2] Concept of physical ageing has been introduced in Chapter III paragraph III.2.2, and concerns a priori the tests performed at a test temperature below the glass-transition temperature of the interlayer material, with a presumed value about 55-60°C for SG-laminates.

[3] Primary parameters are for instance the applied force F, the crack opening d, the displacement rate, the dimensions of the specimen dimensions,… and derived (calculated) parameters are for instance the force by unit of width, axial stress in the ligament, interfacial fracture toughness,…

[4] Related concepts and grid analysis have been presented and discussed in Chapter IV.

condition before the effective start of the test. It was also decided in a first step to perform each specific combination of test conditions on one unique specimen, leaving the investigation of reproducibility for a few sets to the last test series. However, this last objective finally could not be achieved within this campaign.

Table V.1 provides an overview of the executed program by test series. The determination or the order of the test series followed the general scheme presented in paragraph V.2.3; however, the effective execution order has been disturbed from intentions because of a variety of practical aspects, related among others to the development of the test configuration (in particular with regard to the use of the climatic chamber and of the cooling module). It led in particular to start the test series at colder temperature (s7-s9) later than initially wished. The reason for mentioning these practical aspects is related to the noticed influence of the storage duration between the results of the first and last test series at ambient temperature, initially expected to have a negligible effect with regard to the overall duration of the experimental campaign – which proved to be a wrong assumption thus (see more here about in paragraph V.3.4).

The developed experimental approach has been possible thanks to various contributions and collaborations, which are summarized in the credits section at the beginning of this chapter. The major part of the tests has been performed at the laboratory for Mechanics of Materials and Structures (MMS) of Ghent University (UGent); the three test series at colder temperatures have been performed at the laboratory of Polymer Technology at Eindhoven University of Technology (TU/e). The whole experimental campaign has been executed between March 2012 and April 2013.

V.2.2. Test specimens

A TCT-specimen basically consists in a piece of laminated glass of relative small dimensions, of which the glass sheets are pre-cracked before the test. The choice of the production method of the small specimens is framed by practical considerations specific to the used interlayer material, with regard to possibilities and limitations for the lamination and cutting processes, and is considered as having potential important effects on the test results, in function of identified potential border effects[5]. This section is detailed in consequence.

[5] These border effects are of the first type, in relation with deviations between experimental fields EFI-Specimen and application fields AF-Product (identified in Chapter IV and Chapter I respectively). Some technical issues for preparing and cutting SG-laminates have been pointed out in Chapter IV. On a general way, techniques used for cutting PVB-laminates often cannot be transposed as such for SG-laminates, mainly because of the higher stiffness of the interlayer at ambient temperature, and have to be at least slightly adapted in accordance. However, such adaptations appeared to be not immediate for any automatized cutting tool used in production plants…

The SG35 sample[6] consists in 63 small rectangular pieces laminated glass cut out of a larger plate with initial dimensions 1500 x 500 mm and with following *nominal* composition (corresponding to a 'simple' configuration with 2 glass layers and one interlayer) :
4 mm float glass – **0.89 mm** SG interlayer – **4 mm** float glass [7]

The small pieces were obtained by means of water-jet cutting technique, selected because of the minimal damage to the ridges of the glass sheets along their cut edges[8]. All the obtained pieces have a length of about 150 mm, 54 with a width of 50 mm and 9 smaller ones with a width of 30 mm (the cutting pattern with the position of the small specimens on the initial plate is given in Figure V.2; the specimens were numbered according to the chronological order of preparation into TCT-specimen and execution of the TCT-test, respectively from XX = 01 to 54 for the wider specimens and from XX = 55 to 63 for the narrow ones[9]). The small pieces were all cut at the same moment, and stored together until their preparation into TCT-specimens, in indoor conditions at ambient temperature and protected from light, but without more strict control on temperature and moisture environment.

On this way, the border effects on the properties between specimens of the sample are assumed to have been minimized. However the time interval between lamination and testing[10] (named further lamination-to-testing duration) is quite different between the first and last tests : this parameter proved to have a

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[6] The number 35 refers to the nominal commercial thickness of the SG-interlayer sheet before lamination (in thousandths of an inch : 35 mil = 0.96 mm); 0.89 mm corresponds to the nominal laminated thickness, used as commercial value and mentioned in technical documentation – for instance in documentation included in technical agreement DTA 6/12-2086 issued by the CSTB (France), assessing the "fitness for use" of SG-laminates in glazing applications.

[7] The effective average interlayer thickness for the SG35 sample is 0.86 mm, and corresponds with an average value obtained by measuring the total thickness of each individual TCT-specimen, and by accounting for a value of 3.85 mm for the average thickness of the glass sheets. In comparison with related measurement uncertainties, the difference with nominal thickness is not significant (the laminated thickness is a secondary or derived measure; see Chapter IV section IV.4 about related uncertainties and propagation of errors). See also paragraph V.2.6 for estimation of other experimental uncertainties.

[8] In comparison with sawed pieces. Besides, the water-jet cutting is assumed to involve less heating along the edges during the cutting process compared to sawing or traditional cutting technique (this last one inducing heating and stretching of the interlayer along the edges). Finally, such specimens are estimated to show less deviation of adhesion and interlayer properties compared to similar products produced in industrial conditions.

[9] A detailed overview of the all test specimens is given in the Appendix A.

[10] In fact, in this campaign, the differences of 'initial state' between test specimens of the sample are mainly due to different storage durations. However, in the case of "round tests" involving different laboratories and/or production plants, sources of systematic deviations could be more generally due to lamination-to-testing duration and conditions; if this factor remains an unidentified EFI (see Chapter IV section IV.2), it could lead to serious interpretation issues...

significant effect for some test results as explained further in section V.3.4. Test periods of the different test series are therefore added in Table V.2 below.

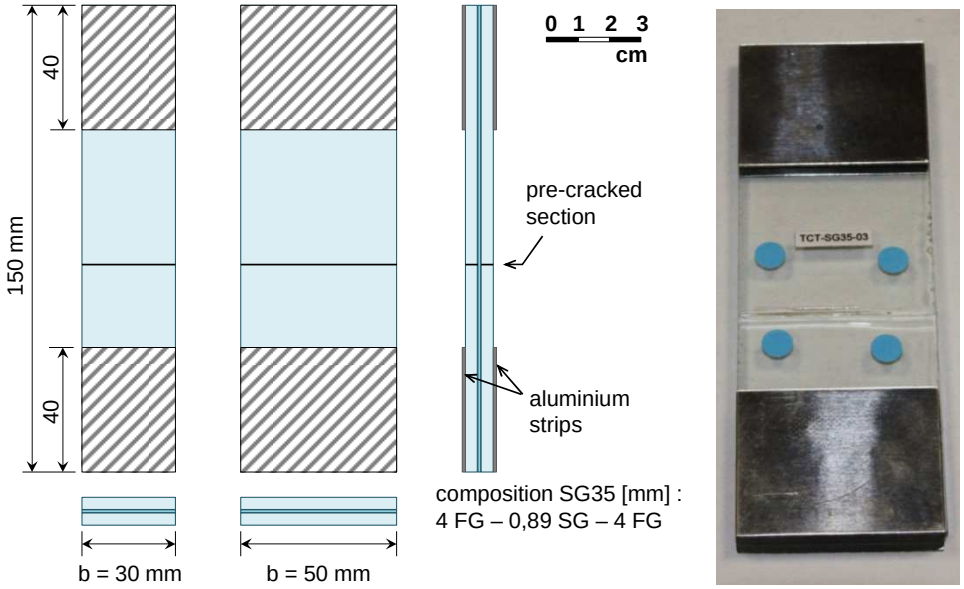

Figure V.1 – Geometry of TCT-specimens (sample SG35)

The final transition of each small piece laminated glass into a TCT-specimen is made shortly before the execution of the TCT-test and consists of three steps :

- the pre-cracking of the two glass sheets in a cross-section of the specimen[11], each obtained by making a straight notch on the glass surface with a glass cutter and by applying subsequently a bending effort on the specimen to generate the crack along the notch direction, in a plane perpendicular to the outer surface[12];

[11] The position of the pre-cracked cross-section was introduced at a lower level than the half-length of the specimen (Figure V.1) simply because of lighting issues (see also paragraph V.2.6)

[12] The method used for making the initial cracks in small-size laminated glass specimens (TCT-specimens) is similar to the one used for larger pieces of laminated glass (tests at element scale presented in Chapter IV) and described in (Delincé et al. 2010). Firstly, a straight notch is made by means of a classical glass cutter, equipped with a cutting (tungsten) carbide wheel and a small tank inside the handle containing the cutting oil. Then, a bending effort is applied on the specimen in order to generate a tensile effort perpendicular to the notch line, by means of a cut running plier typically used for thicker glass sheets (above 8 mm). The cut running plier (in fact a kind of small 4-point bending test device) is placed across the notch line along a free edge of the laminated glass plate, and the crack is generated from the notch by bending the specimen carefully until the crack starts to run. The process is repeated a second time for cracking the second glass sheet on the opposite side.

- the equipment of the specimen for the TCT-test : fixing of four aluminium strips (with thickness of 0.5 or 0.8 mm) at the extremities of the specimen by means of fast-bonding adhesive (Loctite 401), to avoid slip of the specimen during the test and to reduce the risk of glass breakage while fixing the specimen into the grips or during the test (see section V.2.5);
- the equipment of the specimen for the optical measurement (see paragraph V.2.6) : sticking of round markers on the front glass sheet of the specimen.

Figure V.1 figures out the dimensions of the two specimen's geometries used in this test campaign and shows a ready-to-test TCT-specimen. The width and the thickness of each ready-to-test TCT-specimen are then measured. The value of the effective width is an average of two measurements above and below the pre-cracked section; the value of the effective thickness is an average of four measurements along the two lateral sides and above and below the pre-cracked section (see here above about derived value of interlayer thickness)[13].

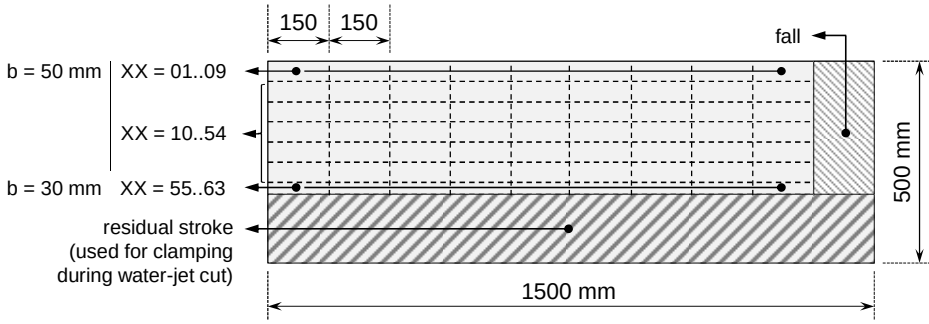

Figure V.2 – Cutting pattern of specimens of the SG35 sample (as executed)

V.2.3. Development of the experimental strategy

An overview of the experimental campaign has been given in Table V.1 with the different test series, which only gives a global picture about the types of test conditions. However, in order to understand the end results, it is necessary to explain the development process of the experimental approach. And probably this is at least as interesting and useful as the resulting numbers...

The execution order of the different test series followed roughly the descending order indicated in Table V.2; cdr-series got an odd number and creep series got an even one. The first step consisted in investigating the sensitivity of the response of TCT-tests to the applied loading rate, by means of a short series of TCT-tests performed at room temperature (20°C). The values of applied displacement rates

[13] A detailed overview of the average dimensions of each test specimen is given in Appendix A.

are selected in order to cover a range as large as possible within the identified limits, between 0.01 and 100 mm/min; the intermediate values are distributed equally along a logarithmic scale, corresponding to successive values multiple of each other's by a factor 10.

The corresponding test results were in a first step analysed on a relative rough way, and limited to the identification of the reached peak load values for each test of the series. The measured values of the maximal force are plotted against the corresponding value of the applied displacement rate, and appeared to fit fairly well with a straight line on a semi-logarithmic plot. The slope obtained by a simple regression analysis corresponded to a step in the peak force of $\Delta F = 177$ N for a displacement rate multiplied by ten (+1 unit on the log scale). These results of the first cdr-series are used for defining the values of applied creep forces for the corresponding creep test series : the value of the applied force for the first creep test has been chosen arbitrary in the lower range of the cdr-tests results (1000 N), and the subsequent tests of the series are performed at values of applied forces calculated from the slope of the regression line, namely incremented by step value equal to ΔF (in this case, the series s1(a0) was completed by tests performed at 1177, 1354 and 823 N). Figure V.3 summarizes the followed procedure, subsequently applied to the other couples of cdr-creep series for each test temperature[14].

Table V.2 – Experimental approach : periods of tests by test series (sample SG35)

Series nr.	Characteristics of test series	Execution period
s1 (a0)	b = 50 mm, T = 20°C, cdr	9 and 12/03/2012
s2 (a0)	b = 50 mm, T = 20°C, creep	30/03 .. 13/05/2012
s1b	b = 30 mm, T = 20°C, cdr	7/06/2012
s3	b = 50 mm, T = 60°C, cdr	21/05 + 31/07/2012
s4	b = 50 mm, T = 60°C, creep	21/05 + 29/05/2012
s5	b = 50 mm, T = 40°C, cdr	13/06 + 31/07/2012
s6	b = 50 mm, T = 40°C, creep	20/06 + 17/08/2012
s7	b = 50 mm, T = 0°C, cdr	3/09 .. 7/09/2012
s8	b = 50 mm, T = 0°C, creep	3/09 .. 7/09/2012
s9	b = 50 mm, T = -20°C, cdr	3/09 .. 7/09/2012
s1 (a1), s1 (a2)	b = 50 mm, T = 20°C, cdr	14/09 .. 10/12/2012
s2 (a3)	b = 50 mm, T = 20°C, creep	30/03 .. 15/04/13
s2b (a3)	b = 30 mm, T = 20°C, creep	30/03 .. 15/04/13

[14] The method can be understood on basis of the thermorheological simple model presented in Chapter III paragraph III.2.2, by assuming that the response of a TCT-test configuration is proportional to the intrinsic behaviour. The 'slope' of the regression line on the left plot of Figure V.3a) corresponds to the term k.T/V* in equation (III.4). The corresponding expression for a creep load mode in equation (III.15) describes a line with a negative slope of equal amplitude –k.T/V* on the right plot of Figure V.3. See also related analysis in paragraph V.3.3 and Figure V.16.

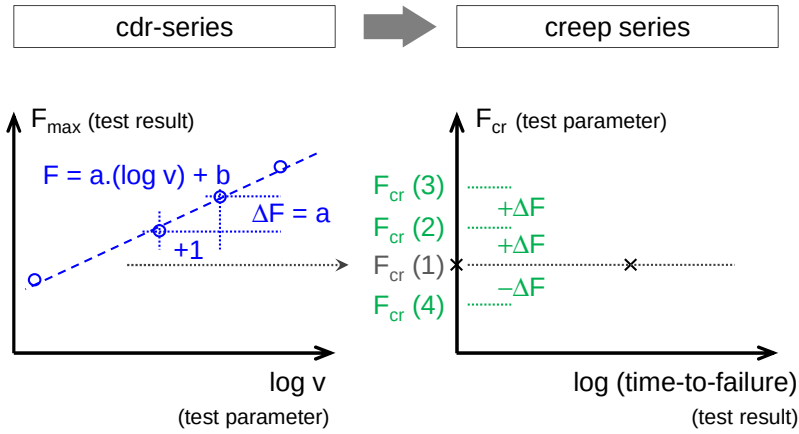

Figure V.3 – The loading values for a creep test series are determined on basis of the results of the corresponding cdr-test series

However, for some test series, the first results poorly matched a single straight line, suggesting the presence of an inflexion point; this was the case for the test series at 40 and 60°C (with initial series of three tests performed at rates between 0.01 and 10 mm/min), which showed an inflexion point in results at a value of crack opening rate about 1 mm/min. The results of the first corresponding creep tests showed a similar trend when the applied creep load force is plotted on a similar 'mirror' semi-logarithmic plot, against the logarithm of the measured time-to-failure (see corresponding series in Figure V.13 and Figure V.15). Completion of the test series with tests performed at intermediate values of the applied displacement rate confirmed the trend (in fact, this has been done only for the creep series at 40°C (s4); at 60°C (s6), because of the lower slope obtained with the results of the cdr-series, small differences in applied creep force lead to larger dispersion of the measured times-to-failure, compared to the other creep test series).

The test series at lower temperatures (series s7-s9) raised some practical issues with regard to the optical measurements which are further detailed below in section V.2.6. Whereas results of tests series at 0°C (s7 and s8) appeared in line with the results of the reference test series at 20°C (s1(a0) and s2(a0)), the results of the cdr-tests performed at -20°C (s9) appeared to be much less in line with the expected trend : a finer analysis of the data sets however showed that these deviations are in all likelihood rather due to experimental issues (detailed in section V.2.5) than corresponding to a representative trend in the intrinsic response of the interlayer ligament.

Finally, in a latter phase of the experimental campaign, a noticeable influence of the storage duration appeared when attempting to complete the initial reference series of cdr-tests performed at 20°C (series s1) by means of a test at intermediary

value of the applied loading rate. In order to confirm the trend and to get a more reliable order of magnitude of the supposed ageing effect on the response in cdr-loading mode, but also in creep mode, the last dozen test specimens were used for that purpose, and this led to add the test series s1(a1) and s1(a2) for the cdr-tests and s2(a3) for the creep tests. The addition of the extension '(aX)' to series numbers has thus been introduced for acknowledging the differences in initial ageing state between specimens tested in the same conditions of loading and temperature. This during the campaign arisen aspect forced us to reinterpret an earlier comparison between results of tests on specimens of different widths (namely between the series s1 and s1b), which led us to perform the last creep test series at ambient temperature on specimens with the same initial ageing state and with different widths (series s2(a3) and series s2b(a3), on specimens with width b equal to 50 and 30 mm respectively). These two experimental parameters correspond thus with two different, but possibly interacting, border effects; therefore, they are discussed in parallel in more details in paragraph V.3.4.

Other aspects having influenced the execution order of the tests are rather related to the daily life in research labs, among others related to the use of new test infrastructures and new combinations of test and measurement devices. Order of tests and details of test configuration were also influenced by previous knowledge gained from preliminary test results obtained during previous similar campaigns. A summary of these previous campaigns has been given in Chapter IV altogether with more general considerations here about, and therefore more specific details peculiar to this campaign TCT-tests "SG35" are not included in this chapter.

V.2.4. Test protocol (for individual tests)

The transformation of each small piece laminated glass into a ready-to-test TCT-specimen (pre-cracking of the transversal cross-section and subsequent preparation of the specimen, see paragraph V.2.2) is performed shortly before starting the test, namely between a couple of hours up to not more than a few days in advance (generally not more than 24h). In particular, the pre-cracking step of the constitutive glass sheets was realized in laboratory conditions (at a temperature between 18 and 24°C).

For all the tests performed inside a climatic chamber (see details in next paragraph), namely all tests excepted the ones performed at 20°C[15] (room temperature in the lab equals the storage temperature of the test specimens), the specimen was placed inside the chamber 30 minutes before starting the test, the mounting and clamping (tightening) of the specimen in the grips being performed during this conditioning period. The duration of this conditioning period also

[15] The tests at room temperature were performed between 18 and 24°C, with a smaller scattering of the test temperature within each test series; for the readability, tests at room temperate are given a nominal temperature of 20°C.

accounts for a stabilization time between the necessary opening moments of the chamber's door for fixing the specimen : the clamping of the specimen into the grips was in all cases achieved at least 10 minutes before the start of the test (see also related comment about the tightening step in paragraph V.2.5.2).

The value of the conditioning period is a slightly arbitrary choice : it aims at allowing the specimen to adapt to the test temperature (ambient temperature in the chamber) before the start of the TCT-test, and at the same time at avoiding the induction of an important annealing process[16] of the interlayer (expected to be especially noticeable at 40 and 60°C for SG-laminates), or of any other ageing phenomenon initiated or promoted by heating. In contrast, the cooling duration for the tests at colder temperature (0 and -20°C) was less strictly controlled, but remained comprised between 15 and 40 minutes. In fact, the control of the duration of the conditioning period for the 'colder' series has been disturbed in a few cases by other experimental issues (see among others in paragraph V.2.6).

V.2.5. Test configuration and data acquisition system

V.2.5.1. Devices and equipment used for the test configuration

The TCT-tests of this campaign were executed on two similar test infrastructures, composed of an electromechanical universal testing machine equipped with a climatic chamber. The tests series s1 to s6 (at test temperature of 20, 40 and 60°C) were realized on an Instron 'universal' electromechanical tensile machine 5800R (frame 4505 retrofitted with a digital controller 8800) having a maximum loading capacity of 100 kN and equipped with an Instron climatic chamber 3119-410 (Figure V.4); excepted a few first ones, the tests were performed with a load cell of 10 kN equipped. Test series s7 to s9 (at 0 and -20°C) were executed on a similar Zwick/Roell testing system (tensile machine Zwick 1475 equipped with a climatic chamber MTS 651). Both climatic chambers are insulated boxes combining a fan-assisted electric heating device and a cooling module fed by liquid nitrogen (LN^2) as coolant[17].

In the first case (s1-s6), Instron's Bluehill software was used for conducting and registering force and displacement data for cdr-tests, while creep tests were conducted using Instron FT-console and corresponding analogic signals were derived from the testing machine and registered via a Labview vi-routine; the precision of the measured force and displacement were slightly different in the

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[16] Annealing corresponds to a change of the initial ageing state of the specimen during the conditioning period towards a larger value of physical ageing state S_a in relation with the concept of initial ageing time t_a; concepts of annealing and quenching process for solid polymers with regard to phenomenon of physical ageing have been introduced in Chapter III paragraph III.2.2.

[17] The LN^2 coolant is released from a mobile tank of moderate capacity (the ones used had a capacity about 160 L). For the longest creep tests, more than one tank appeared to be necessary.

two cases due to different filtering techniques of the electric acquisition signals. For control and data registration of the latest series (s7-s9), the built-in software testXpert of Zwick/Roell was used for both cdr- and creep-tests.

The two tensile machines used are of the same type : the steering is performed by an electro-mechanical motor controlling the displacement of a transversal beam via two screwed spindles situated inside the two lateral columns. This type of tensile machine is the most efficient in applying a constant displacement rate in a range between 0.01 and 200 mm/min. The used climatic chambers typically allow holding a constant temperature within a precision range of about ±2°C around the set reference value. This range for the temperature stability was effective for all tests but the cdr-tests carried out at -20°C : temperature fluctuations as measured by the control unit of the cooling module has been observed between -18 and -25°C; the influence of these variations on the test results is further discussed in paragraph V.3.2.2 below.

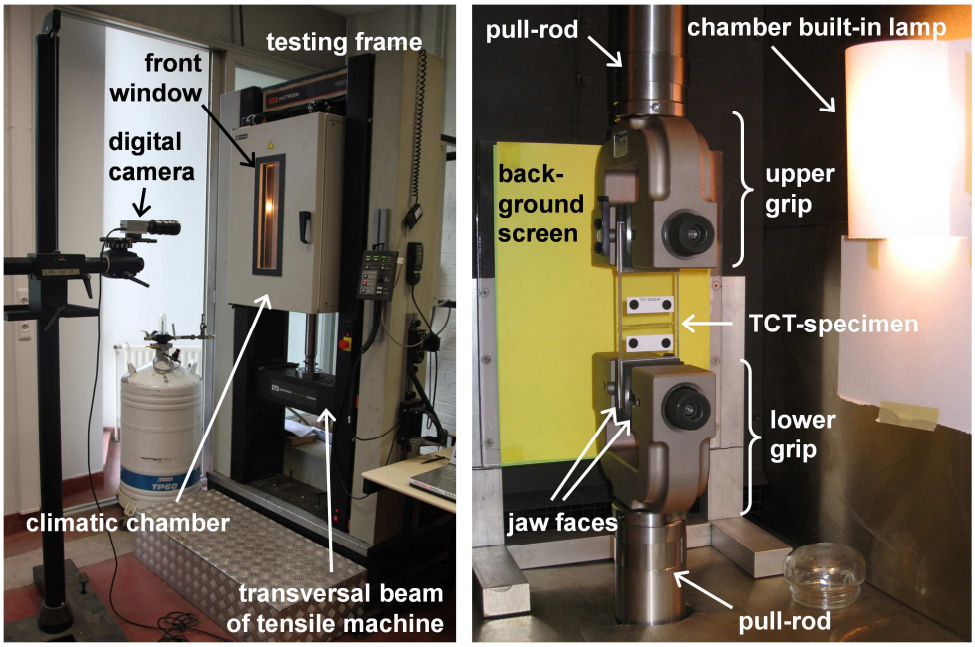

Figure V.4 – Test configuration for TCT-tests, used for test series s1 to s6 (left: global view; right: inside view in the climatic chamber)

TCT-tests performed on both testing systems were using the same advanced screw side-action grips (Instron 2710-116, with a maximal loading capacity of 10 kN) equipped with standard serrated jaw faces (Instron 2702-323). These grips are rigidly mounted inside the chamber to pull-rods going through the upper and lower walls, which are on their turn fixed rigidly to the frame. The different blocking rings between the different pieces being part of the loading string are

tightened while a pre-tension was applied by means of a stiff steel bar, to reduce plays due to initial clearance[18].

The acquisition of digital pictures for optical measurements of the crack opening was made by means of a separated acquisition system which is presented in paragraph V.2.6.

V.2.5.2. Comments about the used test configuration

Details of the test configuration resulted of different considerations which seem worth a supplementary comment. Points of attention were on the one hand the sensitivity of glass to breakage in case of local peak stress (and more generally its small elongation ability before breakage) and on the other hand the small deformation range corresponding to the field of interest[19].

The used test configuration for this experimental campaign was still slightly different from the ones used for previous experimental campaigns TCT-tests[20]. One noticeable modification concerned the use of advanced screw side-action grips in combination with a rigid mounting of these on the tensile machine, in place of previous configuration with wedge grips mounted with articulated connections.

The rigid coupling is apparently unusual for tensile test configurations in general. It had mainly been motivated with regard to the risk of damaging the specimen during its mounting and tightening in the grips : in fact, the rigid connections of the grips to the loading string is expected to reduce possible torsion effort applied to the specimen along the tensile axis when tightening it into the grips. However, this benefit can only be effective in absence of significant misalignment between the jaw faces of the lower and upper grips with respect to the sensitivity of the specimen to these experimental uncertainties[21]; TCT-specimens are anyway certainly more sensitive to misalignments than specimens in other materials than glass. Besides, such a rigid connected assembly is also judged favourable,

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[18] The choice of the type of grips *and* the respect of such guidelines from the manufacturer user's documentation, as a careful handling during the tightening of the specimen, are considered as important details, among others with regard to the particular sensitivity of TCT-specimens to misalignments and to deviations between displacement of the transversal beam and crack opening due to initial clearance along the loading string. See also next paragraph.

[19] It has been showed earlier in Chapter II section II.6 that two ranges of deformation can be distinguished for a TCT-specimen in relation with delamination lengths, a long and a short crack ranges. Arguments were then given to justify dedicating particular care to the experimental investigation of the behaviour in the short crack range, thus for $d < t$.

[20] A series of experimental issues and practical aspects related to the development of the TCT-test method have already been reported and discussed in Chapter IV section IV.3.

[21] Note that this type of misalignments, when they are not critical, can also induce non-negligible secondary transversal efforts in the specimen which are not detected by the load cell. Specimens with thinner or more rigid interlayer are expected to be more sensitive.

perhaps necessary, for successful steering of the creep load mode, certainly more delicate than for the constant displacement rate loading mode[22].

The preference for screw-side action grips upon wedge grips was also motivated by the relatively small deformation range of interest. In fact, a rigid assembly of the selected grips reduce initial movements between the intermediate pieces of the loading string and between the jaw faces and the specimen's ends. A comparison of the time-displacement curves of the transversal beam of the tensile machine d_{tb} and the effective crack opening d_{opt} (measured with the vision system, see paragraph V.2.6.2) in a cdr-test shows that the applied displacement rate (controlled at the level of the displacement of the transversal beam) is effectively attained when the peak force is reached (Figure V.5). The vertical distance between the two displacement curves corresponds to the initial clearance of the test configuration Δd_0. It can then be assumed that the further delamination of the interlayer ligament from the glass substrates starts at this point corresponding to the peak load force.

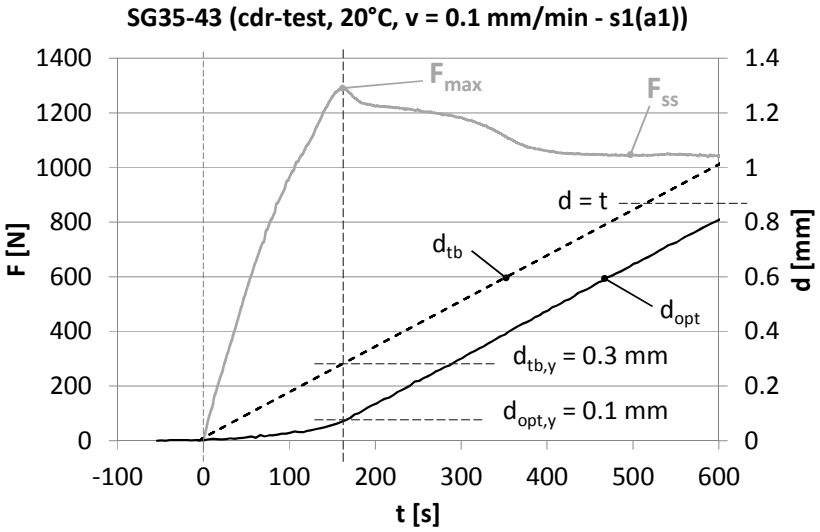

Figure V.5 – Difference between applied and effective displacement and displacement rates due to initial clearance of the test configuration

In summary, the success grade of TCT-tests and the overall accuracy of the test results are likely to vary according to the loading mode, the test temperature and the specificities of the test specimen (among others the stiffness of the interlayer material) possibly in relation with the ones of the tensile machine. Therefore,

[22] A few creep tests failed effectively because of issues with the steering.

assessing the reliability and accuracy of the TCT-test method for one set of reference test conditions is not sufficient; these have to be evaluated *on the whole range of test conditions* considered, or at least for a series of extreme ones with respect to the identified experimental investigation scope.

V.2.6. Image acquisition and optical measurements

Different methods for measuring the crack opening, the progression of the delamination fronts and the deformation of the interlayer in the ligament zone have been reported in literature and in previous chapters (respectively in Chapter II section II.6 and Chapter IV section IV.3). However, their transposition for tests performed inside a climatic chamber is either not possible either not straightforward. Optical measurement methods had to be adapted in consequence, and the measurement accuracy has to be re-evaluated for any extension of the experimental investigation scope (in terms of ranges of temperature and loading rate in combination with each other's).

As already mentioned, the identified issues and defined priorities for this experimental campaign led to focus on the small crack opening ranges. Within this perspective, the use of round markers (Figure V.1) was assumed to lead to the highest measurement accuracy for the crack opening : deviations in detection of the position of marker's edge, among others due to optical noise (blur,…) and varying lighting conditions between consecutive frames[23] were expected to be axisymmetric in relation to the marker's centre, with no or negligible consecutive measurement error.

V.2.6.1. Lighting configuration and computer vision system

The picture acquisition is performed by a digital camera placed in front of the climatic chamber and looking through the front window (Figure V.4). The first vision system (test series s1 to s6) uses a PixeLink camera equipped with a macro-lens (Schneider-Kreuznach Variagon 1,8/12,5 – 75 mm) controlled with the PixeLink® Capture OEM software, registering high-definition frames at a defined acquisition frequency, which varied between 1 frame/3 sec for the shortest tests up to 1 fr/30 min for the longest ones (respectively 0.33 to 0.00055 Hz). The second system (test series s7 to s9) used the same macro-lens mounted on another small digital camera controlled via a Matlab routine, with which the used acquisition frequency was comprised between 0.2 and 1 Hz.

The lighting conditions are an essential feature for using computer vision methods and image acquisition in general. The selection of the lighting configuration had

[23] Varying lighting conditions induce among others relative contrast change between marker and background, what is likely to modify the detected position of the edge of the marker by the detection algorithm.

to account for two different purposes and with practical constraints relative to the climatic chamber. Simultaneous objectives were high measurement accuracy with the pattern recognition of the round markers and the registration of high resolution and sharp pictures of the ligament zone, for detecting the deformation patterns of the ligament and the progression of the delamination fronts. Practical constraints concern the possibilities and difficulties to control the position and the orientation of the light source in order to get a uniform lighting field. Because of limits in view angle and reflection issues with the window of the climatic chamber, working with a lighting source situated inside the chamber is preferable.

With the two test devices used, the built-in lamp of the chamber has been used as intern lighting source. These provide a laterally oriented lighting with a horizontal angle between the lighting direction of the specimen and the optical axis of the camera (view axis perpendicular to the specimen front surface) between 60° and 85°, and a lighting direction in the vertical view field's plane situated in the upper quadrant with regard to an horizontal line through the centre of the specimen (see Figure V.4 for the test configuration and Figure V.6 for the lighting effects on acquired pictures). The contrast and homogeneity of the lighting field is improved by using a background screen with a lighter colour placed behind the test specimen, and its orientation and position is adjusted in order to reduce projected shadows on the specimen and on the background. Besides, the lighting direction renders the delamination fronts on a non-isotropic way according to their local orientation : this complicates their localization by means of automated edge detection algorithm in order to measure delamination lengths !

Lighting conditions were in general fairly constant, except with many of the tests performed at colder temperature. Besides a less favourable position of the built-in lamp, two sources of lighting disturbance have been noticed. The first is due to some localized condensation spot appearing on intern faces of the door's insulated glazing and changing of shape during the test, leading to some local blur effect on the pictures. This type of disturbance has rather been noticed with tests of longer duration (creep tests at 0°C), and the induced error in the measured position of one or two markers. The second source of lighting disturbance is a 'smog' effect on the whole image, appearing during the short injection blows of cold nitrogen into the chamber, with a moderate up to a strong loss of contrast between the marker and its background as consequence. This second type of disturbance causes similar positioning errors of all markers, with in the most severe cases a detection failure of some or all markers by the vision algorithm.

The occurrence of condensation spots is more random with less detection failure and less obvious deviation in measurement curves, and therefore more difficult to track, especially when they occur along the edges of the field(s) of view used for

the optical measurements. The second type of lighting disturbance ('smog' due to coolant blows) can probably less easily be avoided[24], but seems easier to detect on the individual displacement curves of the optical markers due to their regular cyclic nature. Related issues with marker detection and measurement accuracy of the crack opening are further discussed in next paragraph.

Different trails can be proposed for reducing this type of lighting disturbance problems typical to lower test temperature :

1) to refine the design of the test configuration, in terms of position of the lighting source, possibly further improved by modifying the air circulation inside the chamber;

2) to modify the configuration of the optical markers, in order to increase the redundancy – for instance, by increasing the number of markers on each fragment considered as a rigid body – or to reduce edge's effect – for instance, the obtained detection precision has been approximately halved by replacing round shaped stickers (first markers layout, Figure V.6b) by round shaped dots printed on stickers of larger dimensions (second markers layout, Figure V.6c)[25];

3) to change the reference picture from which the marker template is selected for being used by the pattern recognition algorithm.

Optical measurement errors cannot be completely avoided. Whether disturbance of lighting conditions are severe or not, there is always a measurement uncertainty. Attempts at reducing these should be preceded by a reliable and comprehensive quantitative estimation : this is the topic of the next paragraph.

V.2.6.2. Optical measurements of crack opening

The acquired pictures (frames) series were post-processed on the same way for all tests, by means of a routine programmed in LabView Vision Builder. For the reasons identified previously in Chapter II paragraph II.6, Chapter IV paragraph IV.3 and here above, no effort has been made in quantitative measurement of the progression of the delamination fronts.

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[24] The sensitivity to this problem seems peculiar to the configuration of the climatic chamber, the cooling system used and in less extent to the lighting field considered.

[25] The measurement precision has been estimated by calculating standard deviation on measures of fixed distances, between upper and lower markers respectively; as a result of the change of markers layout, a diminution of about 0.015 mm to less than 0.008 mm (in absence of severe variation of the lighting conditions as assessed by a simple visual evaluation of consecutives frames). The obtained precision is then a fraction of a pixel's dimensions (the image resolution was about 0.035 / 0.065 mm/pixel respectively for the two configurations used).

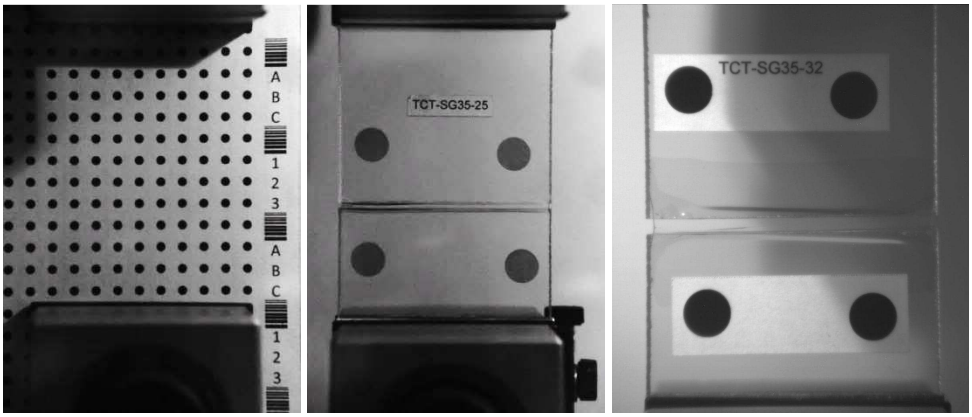

Figure V.6 – Frames used for optical measurements (from left to right) :
a) calibration grid with 5 mm interspaced dots,
b) first markers layout, c) second markers layout

The opening of the initial cracks is measured by means of a pattern recognition algorithm tracking the position of four round markers stuck on the specimen front face. Quantitative optical measurements require the determination of the scale of the image, obtained by placing a calibration grid with the same thickness as the test specimens into the grips prior to the test (Figure V.6a). The initial picture with the grid is used by the analysis routine for calculating a calibration matrix which accounts for non-linearity's caused by optical effects (lens distortion, etc.). The crack opening d_{opt} is then obtained as the average difference in measured positions between the two upper and the two lower markers in comparison with their initial positions (Figure V.6b/c). Besides the quality of the acquired pictures, the obtained measures are affected by the calibration step (parameters for the recognition of the pattern of the calibration grid by the analysis algorithm) and the choice of the marker template.

For the tests for which severe lighting disturbances were noticed on the acquired pictures (principally the tests of series s7 to s9 at colder temperature), some detection failure and measurement's errors required manual corrections during the post-processing step, among others by cutting sections of the position's curve of one or more particular markers (consecutive to detection failure or obvious error) up to cutting of complete sequences of frames of the derived crack opening curve in the most severe cases.

Errors or inaccuracies with optical measures for tests performed at colder temperatures (test series s7-s9 at -20 and 0°C) tend to be amplified by the specific response of the test specimens in these low temperature ranges. The deformation patterns of the SG-laminates were generally more irregular (see paragraph V.3.1), and the critical crack opening leading to final failure (end of the test) were generally smaller altogether with less regular shapes of the loading or creep curves (see paragraph V.3.2).

However, there are also sources of systematic errors in measured values of crack opening by this optical method, which modify the accuracy of the measures without modifying their precision. The bias on the measured value of crack opening, or total systematic deviation, is roughly estimated around 0.04 mm (in absence of severe disturbance of lighting conditions mentioned here above). This one is principally determined by the quality of the calibration step, in particular the positioning of the grid plane with respect to the position of the front face of the specimen with the markers in test conditions.

Table V.3 – Measurement ranges and uncertainties for TCT-test

Parameter or uncertainty type	Value or range [a]
Dimensions of TCT-specimen	
Accuracy on width measurement (unit)	~ ±0.1 mm
Standard deviation on measured width (sample)	0.8 mm
Accuracy on measurement of total thickness (unit)	~ ±0.02 mm
Standard deviation on measured (total) thickness (sample)	< 0.01 mm
Displacement and crack opening during TCT-test	
Short opening range (2.t)	0 .. 1.78 mm
Initial clearance testing configuration Δd_0	0.05 .. 0.2 mm
Image resolution	0.035 .. 0.065 mm/pixel
Random uncertainty of optical measures (round markers)	~ 0.008 .. 0.015 mm
Systematic uncertainty of optical measures (round markers) - estimation	~ 0.04 mm
Detection of position of delamination front (estimation of *achievable* accuracy with the used testing and lighting conditions)	~ 0.2 mm
Other measurement uncertainties	
Accuracy of load cell	< ± 1%

[a] Note : the ranges of values mentioned in this table, in particular those related to measurement uncertainties, are estimations based on the performed TCT-specimens and –tests of the sample SG35 reported in this chapter, for displacement rates ≤ 10 mm/min. See also related comments in main text.

The determination of a criterion on acceptable value of systematic error on results of optical measurements should be balanced with other possible systematic deviations peculiar to the test configuration, in function of the defined global accuracy level. This is an essential aspect in a more global management of uncertainties, and in particular with respect to the assessment of systematic experimental uncertainties discussed in Chapter IV. It seems also clear that the achievable accuracy in measuring the progression of delamination fronts would anyway remain an order of magnitude larger (less accurate) than the measurement accuracy of the crack opening obtained by the method presented in this section; in particular, it is only once the crack opening has reached a value of about 0.5 mm that delamination fronts and deformation patterns can be distinguished on the acquired pictures from the line corresponding to the initial pre-cracked section.

Table V.3 summarizes orders of magnitude of the different measurement ranges and measurement uncertainties obtained with the used test configuration. It highlights in particular that for some parameters the systematic uncertainties are larger than the observed random ones. The reached measurement accuracy of the crack opening by optical method is judged satisfactory with regard to the carried analyses following in this chapter and to the other experimental uncertainties.

V.3. Results of the experimental campaign TCT-tests

An overview of the performed experimental program has been given in Table V.1 with an indication about the success grade by test series. The amount of failed tests mentioned in this table correspond to different types of failure : failure in execution (for instance, due to steering problem of various nature, slip of the specimen…) and failure at analysis which led to the rejection of the test results. The first type of failure has already been commented in the previous section; the second type of failure is further commented in the present section.

The presentation of the results is split into four parts :

- a description of the failure patterns as observed by means of the acquired pictures (paragraph V.3.1);
- a description of the processing method, consisting in the merging of the raw results provided by the two parallel acquisition systems described in paragraphs V.2.5.1 and V.2.6.1, the reconstitution of the loading and creep curves with the optical measurements, and the identification of the particular points of the curves (paragraph V.3.2); as rejection of some test results occurred at this step, this step corresponds to the analysis of individual test results and results by test series;
- the comparative analysis of the test series by loading mode at the different temperatures, based on results of the initial series s1 to s8 (paragraph V.3.3);
- the analysis of the two identified border effects, namely the influence of the width and of the initial ageing state of the test specimens, based on results of test series s1(aX), s1b, s2(aX) and s2b(aX) (paragraph V.3.4).

V.3.1. Deformation and failure patterns

Analysis of acquired images series used for the measurement of the crack opening for each test leads to identify different *deformation* and *failure* patterns[26] for TCT-test configuration (Figure V.7 and Figure V.8) :

1) a **regular delamination** pattern (**RD**) occurs when the overall crack opening is mainly fed by the delamination mechanism, with limited further stretching of the delaminated part of the ligament outside the zones close to the delamination fronts. The latter keep a regular shape, straight and parallel to the initially cracked cross-section, during the delamination process;

[26] The response of a TCT-test can show a succession of different deformation patterns; the failure pattern is the deformation pattern for the identified failure point. In other words, deformation patterns arise from the analysis of the pictures alone, whereas the failure pattern is defined with respect to the loading curve. This distinction is important to make as the determination of the failure point will appear as non-univocal.

2) a **crack propagation** pattern (**CP**) refers to situations where a visible tearing of the interlayer ligament is observed, in the form of one or more holes appearing in the central zone of the ligament through the interlayer thickness (as seen on a front view as in Figure V.7). These holes are subsequently growing in size by further tearing of the interlayer ligament along the width of the specimen : this happens generally together with further irregular delamination, namely the appearance of one or more holes in the ligament is accompanied by an irregular progression of the delamination fronts along the width of the specimen (a change of straight shape into a more irregular one).

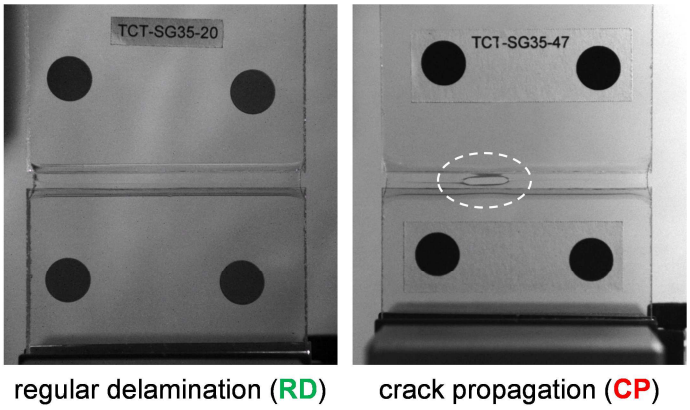

Figure V.7 – TCT-test : two deformation patterns (front view)

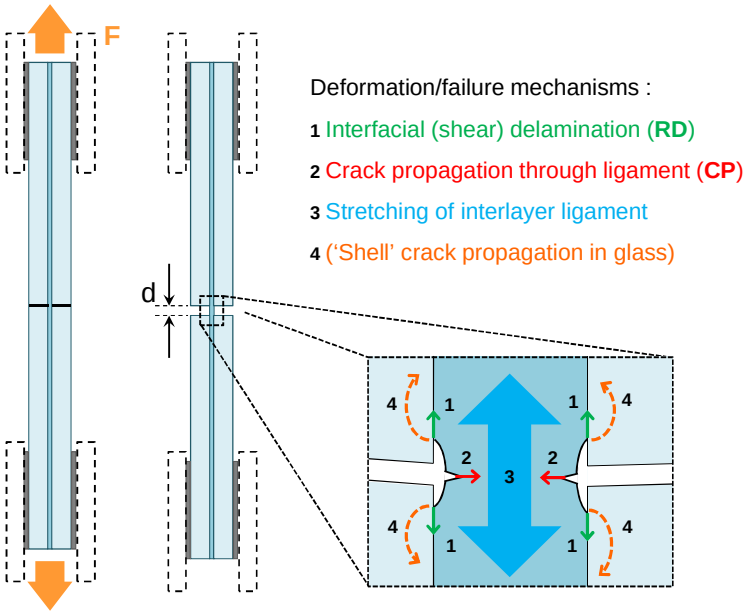

Figure V.8 – TCT-test : test configuration (lateral view), deformation and failure mechanisms

Experimental distinction between the two deformation patterns is possible only for a crack opening above a minimal value, which appears to be already large compared to the identified small crack opening range (see Chapter II section II.6).

Some cdr-tests allow to observe that a CP pattern is consecutive to a RD one, when a noticeable tearing of the interlayer starts only from a relatively large value of the crack opening *d* (Figure V.9). The apparition of a hole through the thickness of the ligament (between points d3 and d4) corresponds clearly to a force drop on the loading curve. However, before this transition point, there is already a sign of the presence of an irregularity on the images announcing the appearance of the hole (pictures corresponding to points d2 and d3). Looking at the shape of the loading curve, this one is characterized by a peak force at small crack opening (point d1) preceding a first load drop; a regular delamination pattern under a steady-state[27] deformation (at constant value of applied force F_{ss}) follows (up to point d3), and the test ends with a crack propagation pattern associated to an irregular decrease of the applied force (after point d4). Between points d4 and d5, the imposed crack opening rate is obtained by a combination of delamination and hole extension along the width of the ligament.

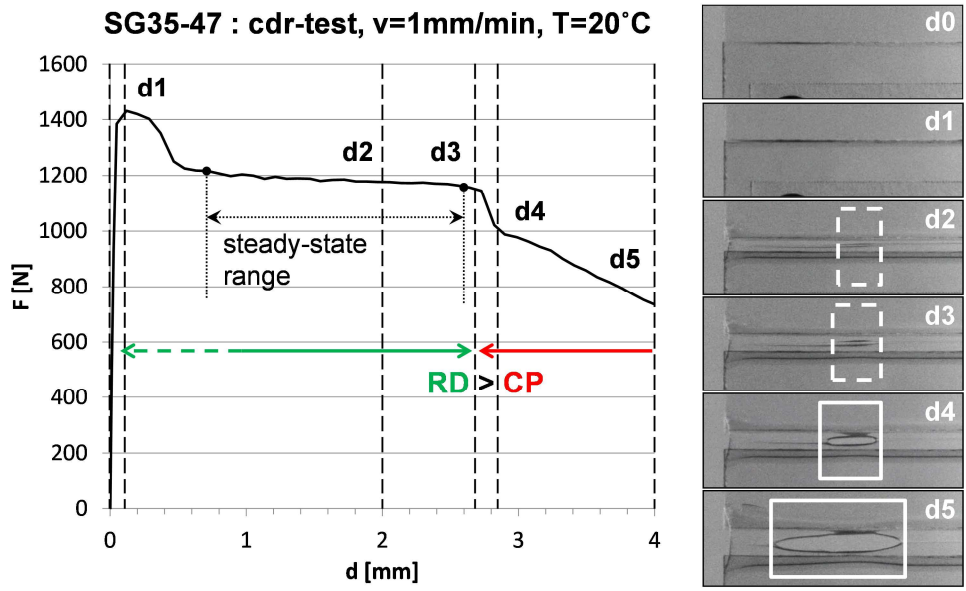

Figure V.9 – Observation of consecutive deformation patterns in a cdr-test (series s1(a3))

However, for many cdr-tests carried out at or below 20°C (series s1 and s7), the possible presence of consecutive RD and CP patterns cannot be distinguished, contrary to the example illustrated here above, because the crack propagation

[27] The steady-state deformation mode refers to a range of deformation where the response force keeps a constant value during a TCT-test performed at constant displacement rate (cdr-test).

pattern begins at small value of the crack opening *d*, with no noticeable steady-state deformation range (examples of such loading curves appear in Figure V.14). Accordingly, image analysis does not allow determining univocally for each test whether the peak force corresponds to initiation of the delamination mechanism at glass-interlayer interfaces or to a yielding mechanism (initiation of plastic flow) in the bulk material[28] of the interlayer ligament announcing the start of the breakage of the ligament cross-section.

For creep tests, a transition from a RD to a CP pattern could generally not be distinguished between the successive digital images and the loading curve (creep curve, see next paragraph), even when this happened at larger value of crack opening : indeed, the apparition of the CP pattern and the full breakage of the ligament are relative close events when considered at the time scale of the whole test duration[29].

A more accurate examination of the CP pattern allows to distinguish two steps in the crack propagation process through the ligament cross-section (Figure V.10). Firstly, strain localisation occurs along the width of the specimen, and cracks initiate from the two outer, delaminated surfaces of the ligament, through the ligament thickness : the start of this mechanism is called *initiation of the CP deformation pattern*, and corresponds with the mechanism 2 in Figure V.8). When the two corresponding crack fronts, situated on each side of the ligament thickness, join each other, namely where there is coalescence of the two crack surfaces, a hole appears in the middle of the ligament[30], and the cracks further propagate then mainly along the width of the specimen from the tips of the hole. The moment of this change of dominant crack propagation direction occurs between the points d3 and d4 (Figure V.9 and Figure V.10). This *'coalescence point' of the CP deformation pattern* is accompanied, in the merging point, by a sudden change of direction of the crack propagation direction. In fact, it is rather this coalescence point of the CP deformation mode which is detected with the kind of image analysis presented in this chapter, and consequently associated with a *failure pattern*. Looking back to the pictures of Figure V.9, initiation of CP already begins between the points d1 and d3; it is however hard to determine if this is already a true crack propagation process or a severe strain localisation.

This remark about the CP pattern highlights that it is getting experimentally obvious only from the moment that an irregularity appears in the shape of the

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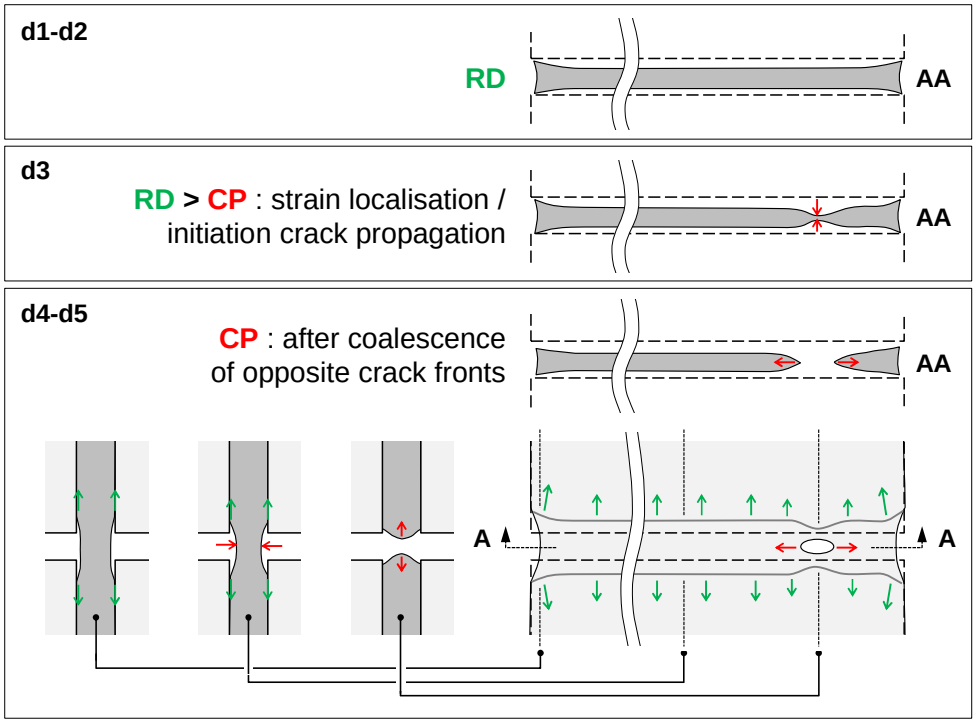
[28] This corresponds to the concept of yield point as defined in Chapter III.

[29] Namely the duration between a visible transition from a RD-mode to a CP-mode and the total breakage of the ligament was an order of magnitude smaller than the acquisition period between two consecutive pictures. However, the acquisition frequency was also lower for the longest creep tests, see paragraph V.2.6.1.

[30] It is impossible to distinguish at this experimental scale if this process is preceded or accompanied by cavitation in the bulk of the interlayer.

crack propagation front along the width of the specimen and through the ligament thickness, and accordingly it is preceded by a localisation (or necking) phenomenon[31]. It seems unlikely that a CP-initiation can occur without preliminary strain localisation, but if it would, the occurrence of a CP-initiation is hardly detectable on the pictures as long as the crack propagation through the thickness remains regular along the width. As a consequence, it is not possible in some cases to determine univocally to which mechanism initiation (RD or CP) the peak load point (d1 in Figure V.9) corresponds; however, it seems clear that these two crack propagation patterns require a local yielding of the material to be initiated[32].



Note : the values of crack openings dX in the left upper corners correspond to the ones used in Figure V.9

Figure V.10 – Transition between crack propagation modes

Accordingly, the *crack propagation pattern (CP)* is getting a *failure mode* caused by crack propagation through the cross-section of the ligament up to its full breakage at a relatively small value of the crack opening; by symmetry, the *regular delamination pattern (RD)* is associated to a *failure mode* when a too

[31] See also Chapter III, paragraph III.2.3.

[32] See also in parallel with Chapter II, section II.4.

large value of the crack opening *d* is attained, disregarding the corresponding values of delamination lengths *a*. However, to this point, the value of the crack opening to consider for determining the transition between the two failure modes is not defined[33] ! Consequently, the peak load point of a cdr-test could principally correspond to the initiation of one of these two failure modes, but in general the RD pattern seems activated firstly (and this corresponds to a safer failure mode of the TCT-configuration[34]).

Among all the TCT-tests performed on specimens of the SG35-sample, CP deformation and failure patterns were only observed for tests carried out at temperatures lower or equal to 20°C, for both loading modes. For tests carried out at 40 and 60°C, only regular delamination patterns were observed in the two loading modes, and consequently not any of these tests ended in a breakage of the interlayer ligament.

In summary, each TCT-test result could be associated on the basis of image analysis to one of the three following categories of failure patterns :

CP : TCT-test for which a CP pattern is observed in the small crack opening range (namely for $d_{opt} < 2.t \approx 1.7\ mm$)[35];

RD > CP : TCT-test for which a CP pattern follows a RD deformation pattern out of the small crack opening range (namely for $d_{opt} > 2.t \approx 1.7\ mm$);

RD : TCT-test for which only a RD pattern is observed out of the small crack opening range (namely for $d_{opt} > 2.t \approx 1.7\ mm$);

However, some tests were stopped before the crack opening could become larger than the small crack opening limit.

In conclusion, we observed that the response of a TCT-test configuration is generally ruled by more than two complementary mechanisms : besides the interfacial delamination mechanism and the ligament's material stretching and yielding, a crack propagation mechanism through the thickness of the interlayer can also be initiated.

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[33] It seems meaningful to relate the determination of a criterion on the value of the crack opening distinguishing the two failure modes to the concept of the small crack opening range (see also Chapter II paragraph II.6). Its value is temporarily (relatively arbitrary) fixed to 2.t, with t the thickness of the interlayer.

[34] This corresponds it fact to a crack penetration-deflection criterion, see Chapter II section II.4.

[35] The small crack opening range defined here is thus different from the short crack limit defined in literature, where it is defined among others in relation to the delamination length *a* (Chapter II section II.6).

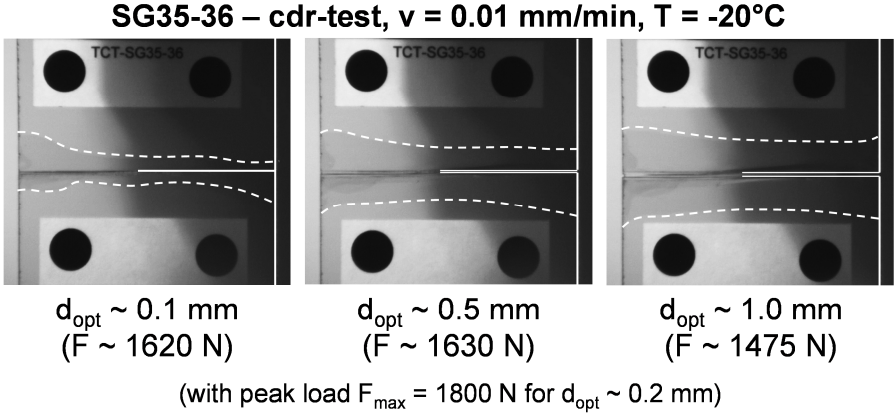

Figure V.11 – Example of iridescent zones (limits highlighted with dotted lines) appearing in the visible spectrum and growing far beyond the delamination fronts for tests carried out at low temperatures (0 and -20°C)

In order to be complete, it is worth mentioning that a supplementary deformation pattern appeared in the visible spectrum on the acquired digital images of the tests at low temperature, in the form of iridescent zones[36] extending far beyond the delamination fronts. In comparison to their appearance in naked eye observation, these iridescent zones are less noticeable on the acquired greyscale images where they appear as slightly "darkened" zones (Figure V.11). This effect was the most visible for the coldest tests (series s9); these zones disappear once the specimen's temperature raises back to room temperature after the end of the test.

This phenomenon seems due to a photoelastic effect, namely caused by a change of stress/strain state in the interlayer. The size extension of these iridescent zones with the increase of crack opening suggests a change of shape of the stress field ensuring the load-transfer between the ligament zone and the glass sheets far beyond the pre-cracked section, perhaps associated with a phenomenon of micro-delamination. It could be understood as an extension of the zone of influence of the TCT-configuration, or necessary length of the interfacial planes on each side of the cracked section for 'fully' transferring the tensile force in the ligament into the glass fragments[37]. However, the observation of this effect does not necessarily imply that it could be used for making some quantifiable measurements by using optical properties of the SG-interlayer.

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[36] Zone appearing with rainbow colours according to the view angles, effect apparently caused by polarization of diffracted light through the specimen.

[37] It is suspected that some correspondence exists between the length of such iridescent zone and the activation length introduced in Chapter II, Section II.4, and that these would have a similar order of magnitude. The extension of the iridescent zone in the early part of the loading curve could be a sign of a change of size of the crack tip field, and of the ratio between crack propagation modes, or of mode-mixity.

V.3.2. Processing of test results

The primary test results for each TCT-test were two sets of data provided by the two acquisition systems. The first consists in measured force and displacement of the transversal beam derived from the control unit of the testing device (see paragraph V.2.5.1), and the second is the value of the crack opening derived from the optical measurement of the marker positions (see paragraph V.2.6.2).

V.3.2.1. Assessment of reliability of optical measurements

The first processing step was the synchronization of the time scales of the two sets of data, which were obtained at different acquisition frequencies. The comparison of the two displacement curves on a common timeline was a first means for assessing the reliability of the optical measurements of the crack opening; the others aspects related to the verification of the reliability of the obtained measures have been commented here above. The following processing steps were slightly different according to the loading mode, and are highlighted in the next two paragraphs accordingly.

V.3.2.2. Processing and analysis of cdr-tests results

The processing and analysis of the cdr-tests results consisted of different aspects or steps (further commented below) :

1) Processing of measurements for each individual test :
   a) Merging and time-synchronization of data from the optical measurement with other data, among others by comparing applied and effective displacement rates (comparison between curves of d_{tb} and d_{opt} in function of time, see Figure V.5);
   b) Analysis of digital images series to determine the failure pattern for each test (qualitatively);
   c) Determination of peak load value and corresponding value of crack opening.

2) Analysis of test results by series :
   a) Drawing of the loading curves ($F - d_{tb}$, $F - d_{opt}$);
   b) Plotting of the peak load values against applied displacement rates on a semi-logarithmic graph, and calculation of a linear regression equation on the data in this form (or one by segment when an inflexion point is detected, see details below);
   c) Grouping of the results of different series on a common semi-log plot (Figure V.13).

The results of the test series at -20°C (series s9, with tests performed at displacement rates of 0.01, 0.1 and 1 mm/min) are not reflected in the final analysis, because they are judged non-representative for different reasons. Their loading curves showed up regular oscillations with a period corresponding to the duration of the cooling cycles. The measured oscillations appeared furthermore to have a real and a virtual origin, but it appeared not possible to separate the two effects in the analysis. In fact, the virtual component is related to the effect of lighting disturbance caused by the coolant blows on the optical measures (see paragraph V.2.6.2), but the oscillations in the measures of crack opening appeared to get an echo in the measured value of applied force (particularly noticeable for the test performed at the smallest displacement rate). There is thus an effective oscillation in the applied displacement rate at the level of the crack opening, assumed to correspond to thermal movements along the loading string caused by significant variations of temperature in the climatic chamber (between -18 and -25°C). The corresponding peak load values (all associated to a CP failure pattern) displayed on the semi-log plot are all situated above the results of the series at 0°C (series s7), but with a very poor alignment in comparison with the results of the other cdr-series. This alignment problem was also a reason for not performing the corresponding creep test series.

It seems not possible to draw conclusions with regard to the effective causes of the failure of this series s9[38] on the basis of the limited amount of performed tests. It seems however obvious that the experimental uncertainties are relatively larger in these test conditions in comparison with the other test series; therefore, these test results cannot be used for drawing conclusions about the effective *quantitative* behaviour of SG-laminates in this temperature range. Nevertheless, it probably gives an indication that similar conditions at the application level probably correspond with a boundary of the application scope "in that direction" (namely by means of an appropriate relation with related Application Field(s), see Chapter I section I.5).

Figure V.14 regroups a large selection of loading curves of the cdr-series (corresponding to test series s1(a0), s3, s5 and s7). The dependence of the peak load value with the applied displacement rate at all test temperatures is obvious (in parallel of Figure V.13). For loading curves showing a clear steady-state behind the peak, also the value of the load drop ($F_{\max} - F_{ss}$) appears to be sensitive to the applied displacement rate (the correspondence between the shape of the loading curve and the observed deformation and failure patterns has already

[38] It remained in fact difficult to allocate the scattering of the test results of the series s9 to one experimental factor univocally : were the thermal oscillations rather modifying the length of the loading string, what modified the effective crack opening rate, or did they more had an influence on the viscously delayed response of the TCT-specimen ? Is the variation in the measured peak load value rather due to this kind of oscillations or rather due to an intrinsic more variable response due to some more instable crack propagation patterns ?

been commented in paragraph V.3.1)[39]. At low displacement rate and higher temperature, the load drop becomes very small or even disappears (for the two lowest rate tests of the series s5 at 40°C). In comparison, the variation of the value of the crack opening corresponding to the peak load is small, and is generally smaller than 0.2 mm.

The dependence of the peak force value F_{max} on displacement rate ($v = \dot{d}$) and test temperature appears more clearly on a semi-log plot as shown in Figure V.13. The failure patterns (paragraph V.3.1) are distinguished by the type of symbol used : a hollow symbol represents a RD (or RD > CP) failure mode, and a filled symbol a CP failure mode. On this basis, the following observations can be done :

- a transition in failure modes is noticed on the test series performed at 0° and 20°C, from a CP-failure mode towards a RD-failure mode with decreasing value of the applied displacement rate. This transition does not seem to affect significantly the rate dependence of the peak load value : the peak load values are relatively well aligned on the semi-logarithmic plot of Figure V.13;
- on the contrary, the cdr-test series at 40 and 60°C show an inflexion point in the rate dependence of the peak load force while the failure pattern remains of the RD type. Besides, the peak load value shows a less sensitive dependence on the applied displacement rate in the lower range for these two series.

The results of cdr-tests as represented in Figure V.13 are further analysed below in parallel of creep test results in paragraph V.3.3 and following.

The influence of other investigated border effects is further discussed for the two loading modes in section V.3.3.

Results of the linear regression (step 2b) were used for determining the first value of creep load and step value for the following tests of the corresponding creep series (as explained in section V.2.3 here above).

[39] A parallel can be seen with the characteristic parameters of the intrinsic loading curve (Chapter III paragraph III.2.2), between the peak load and the yield stress and between the load drop and the yield drop respectively. However, it does not imply that there is a direct correspondence...

V.3.2.3. Processing and analysis of creep tests results

The processing and analysis of the creep test results consisted of different aspects or steps (further commented below) :

1) Processing of measurements for each individual test :

   a) Merging and time-synchronization of data from the optical measurement with other data, among others by comparing applied and effective displacement rate (comparison between curves of d_{tb} and d_{opt} in function of time);

   b) Drawing of creep curves[40] and comparison of applied and effective creep rate (comparison between d_{tb} and d_{opt}), allowing to determine the initial crack opening $d_{ini} = d_{opt}(t_1) - d_{opt}(t_0)$ and the initial clearance[41] of the test configuration $\Delta d_0 = d_{tb}(t_1) - d_{opt}(t_1)$, with t_0: start of loading step, and t_1: start of creep load step;

   c) Analysis of digital images series for determining the failure pattern for each test (qualitatively);

   d) Drawing of the creep curve (d_{cr} - t) and determination of time-to-failure values corresponding to the breakage point (corresponding to the full breakage of the ligament cross-section) and to a defined set of characteristic points on the creep curve (see details below);

2) Analysis of test results by series :

   a) Drawing of the creep curves (d_{cr} - t) by series on semi and double logarithmic plots;

   b) Plotting on a semi-logarithmic graph of the time-to-failure values of the different reference points identified at step 1d) in correspondence of the creep load value for each test of the series (with creep load value along the vertical axis and a logarithmic time-scale as horizontal axis);

   c) Grouping of the results of different series on a common semi-log plot (Figure V.15).

The analysis of creep tests is a little different from the one of cdr-tests. Following the analysis step 1b) here above, the creep curve for a TCT-configuration has been defined by withdrawing the initial, instantaneous crack opening due to the loading step preceding the creep load :

$$d_{cr}(t) = d_{opt}(t) - d_{ini} = d_{opt}(t) - d_{opt}(t_1) \qquad \text{(V.1)}$$

---

[40] The creep curve is defined here as a time-displacement curve with a linear time-scale.

[41] See description of the test configuration in paragraph V.2.5.

In the results of creep tests presented below, the value of the initial crack opening did not exceed a value of 0.2 mm.

Besides the time-to-breakage corresponding to a full breakage of the interlayer ligament (this point is further called “breakage point”, or BP), different types of singular points can be distinguished on the creep curves (a few ones are visible on the tiled frames in Figure V.12).

The examination of the creep curves on a linear time-scale allows distinguishing consecutive segments with different creep rate, separated by more or less pronounced inflexion points. Inflexion points can probably be associated to instant of activation of deformation mechanisms, followed by a time period of stable crack growth and/or stable creep, characterized by a constant value of the deformation rate depending on the loading level. The most obvious activation mechanism is probably the initiation of interfacial crack growth of the RD deformation pattern, which occurs at value of initial crack opening near zero[42]. It seems logical to assume that a stable progression of the delamination fronts is associated to a secondary creep mechanism of the ligament, characterized by a constant creep rate (see Chapter III paragraph III.2.1). The measured crack opening rate is thus resulting of two different processes with a constant rate, the crack propagation rate of the interfacial delamination fronts and the bulk creep of the free ligament delimited by these[43].

However, it did not seem obvious to select singular inflexion points based on univocal criteria, because of the variety of shapes of the measured creep curves and the variation of the crack opening value corresponding to a breakage point; therefore, it has been preferred to determine loading duration times corresponding to some fixed reference values of crack opening, with regard to the formulation of the design problem (see Chapter II section II.3). The defined set of crack opening reference values d_i is rather arbitrary, but purposely limited approximately to a small crack opening range : for this analysis, the considered values of d_i are equal to 0.1, 0.3, 0.6 and 1.0 mm. As the determination of a failure criterion caused by excessive deformations at the element scale is project-dependent, every value of crack opening corresponds with a potential failure criterion; it is

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[42] A slightly increase of the crack opening before the activation of the delamination mechanism is in practice observed, due to the creep of the interlayer ligament on its free length, delimited by the initial delamination lengths a_0 (see Chapter II paragraph II.4), and possibly because of systematic measurement error due to small change in planar alignment of the specimen during the early-loading step (see paragraph V.2.5).

[43] This neglects a possible stable grow of the CP-deformation pattern (corresponding to a stable crack propagation mode) between the initiation and coalescence points (see paragraph V.3.1), which would then contribute to a stable crack opening rate. The distinction between creep and crack propagation phenomena in the polymer ligament is probably a question of scale of observation, and should be considered in parallel of the discussion about the distinction between reversible and irreversible deformations in polymers (see Chapter III).

therefore also relevant to name any loading duration to reach a characteristic crack opening value as a potential 'time-to-failure'.

Figure V.12 shows obtained semi-logarithmic graphs for each temperature series separately, where time-to-failure values for the set characteristic points are plotted against the applied creep load value for each individual test (corresponding to analysis step 2b); the corresponding creep curves represented on small tiles besides are drawn on a linear time-scale and in a fixed range of the crack opening.

Let us add a few comments about experimental uncertainties with this type of tests and analysis. For the most flat (part of) creep curves, namely corresponding to lower values of creep rate[44], a small deviation on the measured value of the crack opening can lead to a relatively large deviation on the corresponding value of time-to-failure. As low creep rate generally occurred for small values of crack opening, the determined values of time-to-failure are relatively less accurate for the characteristic points corresponding to a smaller value of d_i (with respect to a linear time scale); when the corresponding values of time-to-failure are displayed on a semi logarithmic plot (analysis step 2b), the corresponding measurement uncertainties are further amplified by the logarithmic scale in accordance.

A first trend can be identified based on the analysis of the creep test results : the creep curve shows up with a more pronounced curvature downwards (namely the crack opening rate is increasing with the loading duration and value of crack opening) when the applied creep load is larger and when the test temperature is lower, and simultaneously the crack opening at breakage is smaller. Conversely, the creep curves of tests performed at 40°C are almost flat on the small opening range used to draw the creep curves of Figure V.12, what corresponds to a more constant creep rate. Furthermore, the tests carried out at warmer temperature (40 and 60°C) have a creep curve with an inverted curvature oriented upwards (namely the crack opening rate is decreasing with loading time, what is a behaviour similar to a hardening mechanism), but such a response generally only became noticeable in a larger crack opening range. By comparing the orientation of the creep curve with the deformation and failure patterns observed on the acquired digital pictures, it seems that an increase of the crack opening rate is mainly associated with the initiation of a CP deformation pattern.

[44] "Creep rate" is expressed here in terms of a crack opening rate, in relation with straight segments on the creep curve of the TCT-test.

The results of the different creep test series are collected on a semi-logarithmic plot (Figure V.15, analysis step 2c). In comparison with the 'mirror' plot of the cdr-series where each point corresponds to one test (Figure V.13), characteristic points with a same value of crack opening d_i belonging to different creep tests of a same series are joined by means of dotted lines, therefore called **isometric curves**[45].

However, some of the characteristic points drawn in Figure V.15 are laying behind time duration values usually considered as a lower limit of meaningful creep data : Moore and Turner (Moore and Turner 2001) defines this lower limit as ten times the duration of the loading step. For the creep tests presented here, the duration of the loading step ($t_1 - t_0$) was usually about 10 seconds; accordingly, characteristic points with time-to-failure value smaller than 100 s (2 on the log scale) should be disregarded. On the other side, the upper limit is determined by the definition of a "short duration test", in relation with the concept of progressive physical ageing[46] (see Chapter III paragraph III.2.2); in the case of this campaign, this limit was usually about 2-3 days (5.2 .. 5.4 on the logarithmic time scale with time values in seconds).

The analysis of the creep test results can be completed by the determination of the creep rates in every characteristic point or along straight segments of the creep curves. For all characteristic points represented in Figure V.15, the minimum value of creep rate remained larger than 2.10^{-6} mm/s.

[45] by analogy with representation of creep test results on bulk polymer materials

[46] However, it seems that defining a "short duration test" criterion in function of the occurrence of significant progressive physical ageing is only practically relevant for tests performed 'far enough' below the glass transition temperature.

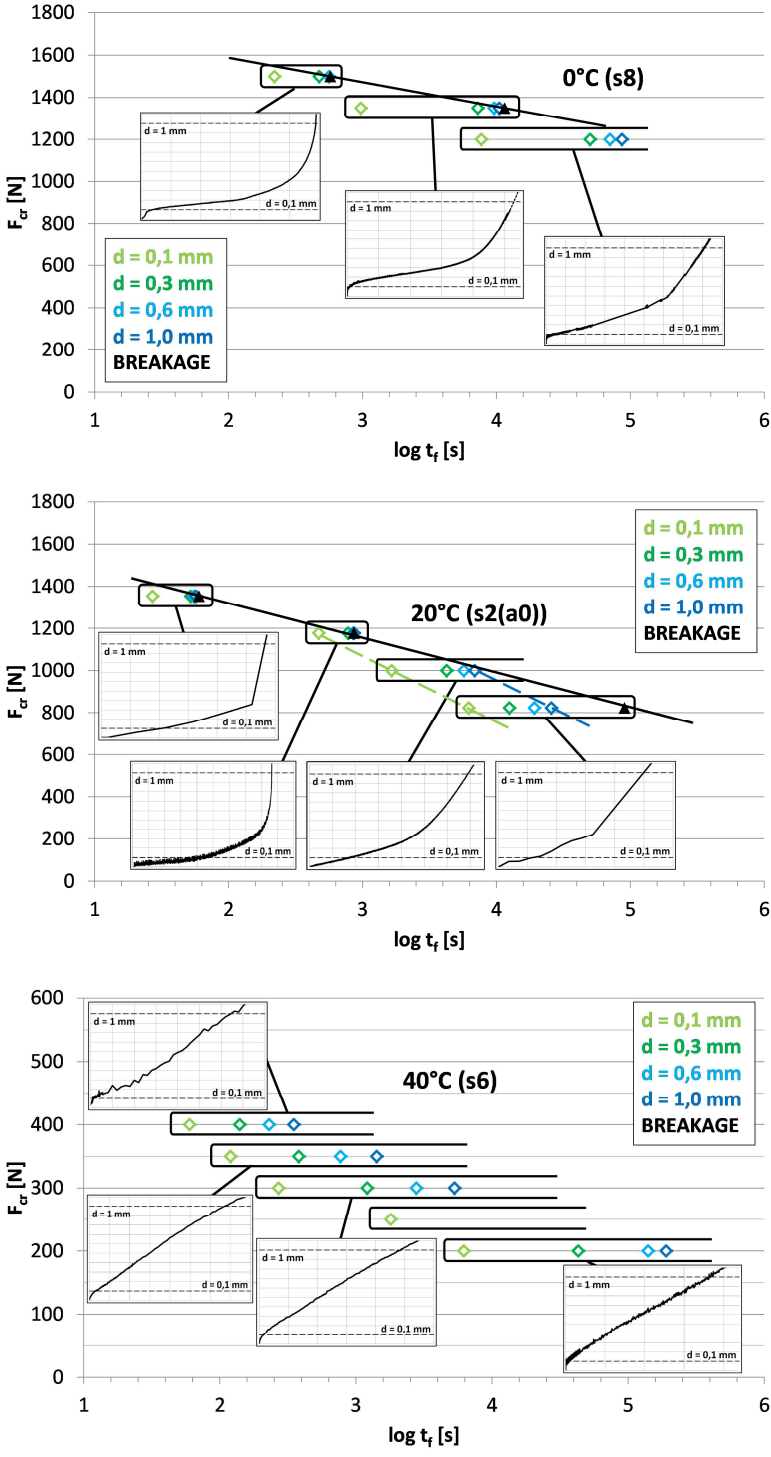


Figure V.12 – Creep test results : characteristic points on semi-logarithmic plot (main frame) and corresponding creep load curves on linear time-scale (tiled frames)

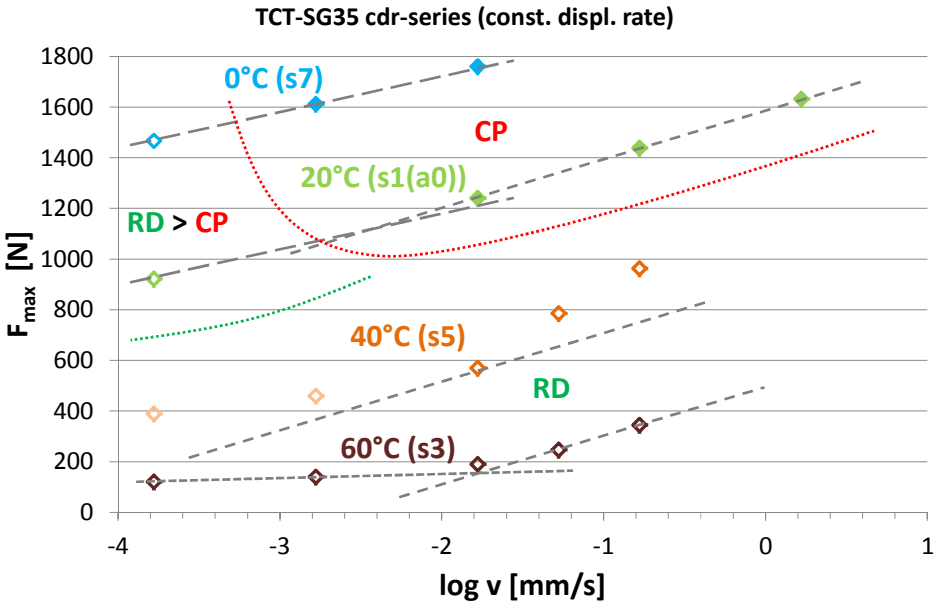


Figure V.13 – Time-temperature dependence of the measured peak load for the initial cdr-test series. The dotted lines are drawn to guide the eye.

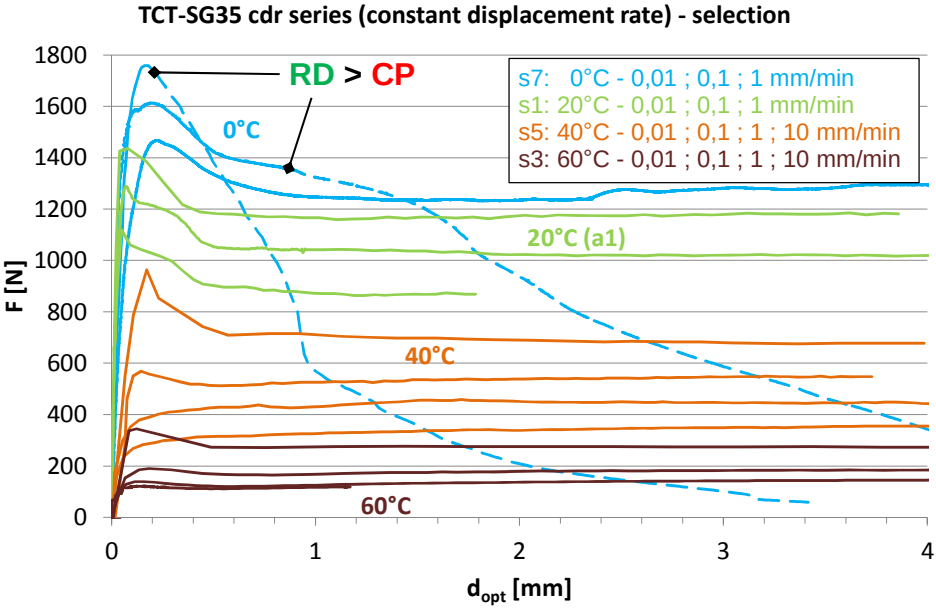


Figure V.14 - Loading curves of cdr-tests grouped by temperature series (selection)

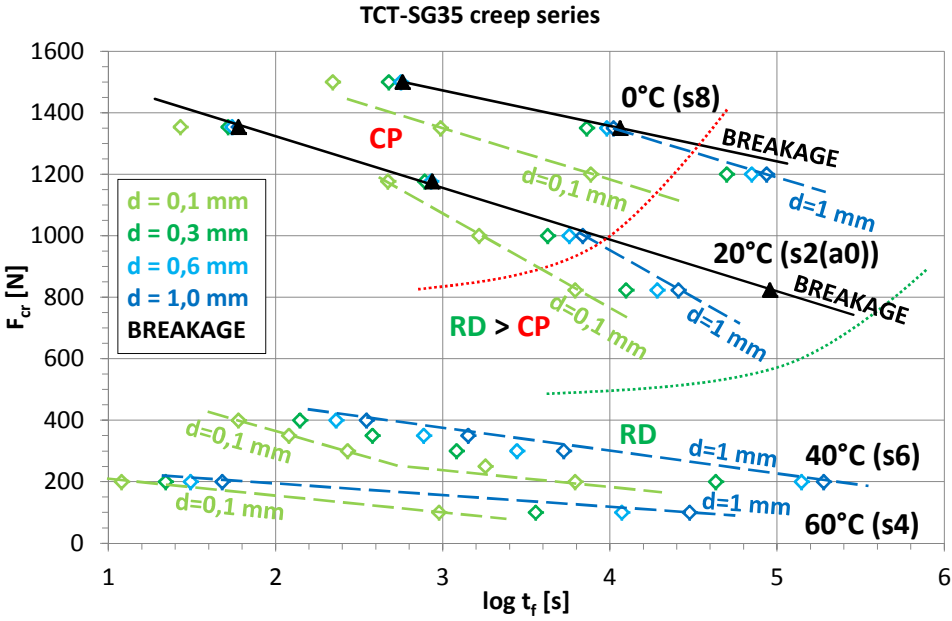


Figure V.15 – Time-temperature dependence of characteristic points of creep curves for the initial creep test series. The stripped lines are drawn to guide the eye in accordance with the definition of isometric curves given in the main text; the continuous lines linking the breakage points of a test series are not a isometric curve

V.3.3. Comparative analysis of cdr and creep tests results

The construction of the two semi-log plots of Figure V.13 and Figure V.15, collecting results of cdr- and creep test series respectively, has been explained in the previous paragraph. The two figures were completed by indicating trends in failure modes on basis of the criteria developed in paragraph V.3.1.

A parallel reading of these two figures allows seeing some symmetry between the response of TCT-specimens of SG-laminates under the two loading modes.

At an intermediate step during the development of this campaign TCT-tests, an attempt has been made to correlate the results of the initial cdr and creep tests series. The analysis consisted in fitting a thermorheological complex model on the cdr-test results by assimilating the TCT-configuration to an intrinsic behaviour, or homogeneous uniaxial loading state (the related concepts are defined in Chapter III paragraph III.2.1), namely by replacing the infinitesimal parameters of stress, strain and strain rate by their macroscopic equivalent, the applied force, the crack opening and the crack opening rate in the equations of the corresponding model[47]. Figure V.16 summarizes the used equations and the parameters determined on basis of the experimental results, and shows that a relative good match can be reached by means of such an approach[48]. However, for this analysis, the critical displacement is defined independently for each temperature series (whereas the corresponding 'intrinsic' parameter, the critical equivalent plastic strain, is considered as a material constant, see Chapter III paragraph III.2.1). By letting this last parameter vary with test temperature, it allows to adapt the horizontal position of each individual creep failure curve independently of each other with regard to the position of the regression curve of the corresponding cdr series (test series performed at the same test temperature). Different reasons for this need for an extra degree of freedom are identified, which can be related to the change of experimental scale and the influence of different border effects. One of these border effects is related to the initial ageing state of the specimen (see further in next paragraph).

[47] Compare the equations (V.2) and (V.3) below with the equations (III.11) and (III.14) of a thermorheological complex model in Chapter III paragraph III.2.2.

[48] The model fitting summarized and illustrated in Figure V.16 has been performed on partially processed experimental data.

$$F_{\max} = F_\alpha + F_\beta = F_{0,\alpha}.\operatorname{arcsinh}\left(\frac{\dot{d}}{\dot{d}^*_{0,\alpha}(T)}\right) + F_{0,\beta}.\operatorname{arcsinh}\left(\frac{\dot{d}}{\dot{d}^*_{0,\beta}(T)}\right)$$

$$\dot{d}^*_{0,x}(T) = \dot{d}_{0,x}.\exp\left(\frac{-\Delta U_x}{R.T}\right) \qquad F_{0,x} = \frac{k.T}{V^*_x} \qquad x = \alpha,\ \beta \tag{V.2}$$

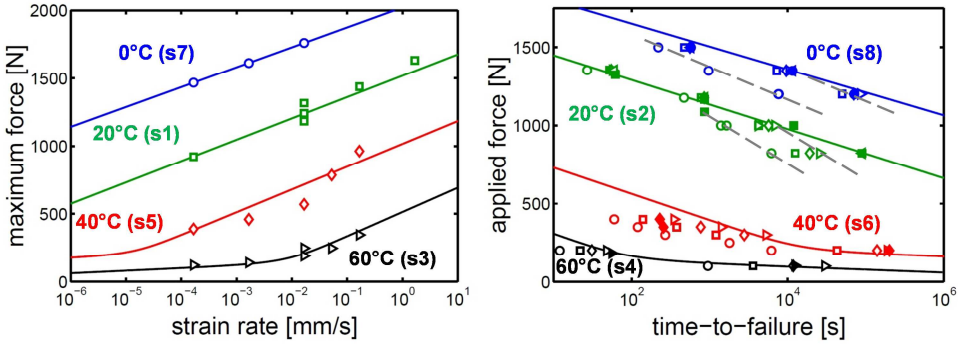
Parameters : $F\,[N],\ d\,[mm],\ \dot{d}\,[mm/s],\ T\,[K],\ V^*_x\,[mm]$

Constants : $k = 1.38.10^{-23}\,[J/K],\ R = 8.314\,[J/(K.mol)]$

$$t_f(F_{cr}) = \frac{d_{cr}}{\dot{d}(F_{cr}, T)} \tag{V.3}$$

Parameters α-transition		
V^*_α	$56.49.10^{-23}\,[m]$	("Activation volume")
ΔU_α	$6.578.10^{5}\,[J/mol]$	(Activation energy)
$\dot{d}_{0,\alpha}$	$1.558.10^{94}\,[mm/s]$	Pre-exponential factor
Parameters β-transition		
V^*_β	$6.62.10^{-23}\,[m]$	("Activation volume")
ΔU_β	$2.768.10^{5}\,[J/mol]$	(Activation energy)
$\dot{d}_{0,\beta}$	$3.960.10^{41}\,[mm/s]$	Pre-exponential factor

T [°C]	d_{cr} [mm]
0	0.3
20	3.6
40	0.2
60	0.7



Experimental results (dots) corresponding to Figure V.13 (cdr series, left) and Figure V.15 (creep series, right) and fitted model (continuous lines) for each temperature series

Figure V.16 – Model fitting on preliminary test results of cdr and creep test series

Despite the approximations, a few interesting trends are arising from the performed analyses.

A straight line through breakage points (BP) of creep tests which ended by a ligament breakage (only in series s2 and s8 corresponding with tests performed at 20 and 0°C respectively) shows a fairly good match with the 'mirror' slope of a similar regression line through the peak load points of the corresponding cdr-series, and this independently of the observed failure patterns in the cdr-tests. However, not such an equivalency between results of corresponding cdr- and creep test series can be found for the steeper slope of the isometric curves (shaped with stripped lines in Figure V.13, Figure V.14 and Figure V.16). Comparison of the data points between corresponding series at 40 and 60°C (corresponding only with RD failure patterns) shows a parallel evolution toward less sensitivity of the response to time-dependent effect, where the decreasing slope in cdr-series finds an echo in the results of the corresponding creep test series.

No direct correspondence appears between transitions in failure modes (failure patterns) and the presence of inflexion points in the rate-dependent response (cdr-mode) and in the isometric curves (creep mode). The apparent thermorheological complex response (associated with the presence of inflexion points and change of slope on the semi-log plots within a test series) seems thus effectively due to different kinetics of molecular processes in the bulk of the interlayer component. The apparent symmetry between Figure V.13 and Figure V.15 is suggesting that the activated deformation mechanisms in cdr and creep loading modes are of similar nature, but it seems difficult to distinguish stretching and crack propagation processes (with regard to RD and CP deformation patterns), especially in the small crack opening range.

Consequently, the ductility of an interlayer ligament in a fractured laminated glass element depends on the ratio between delamination rate and activation of molecular mobility in the bulk of the interlayer component. The ligament ductility appears to increase with lower values of crack opening rate, whatever the loading mode; simultaneously the critical failure mode is evolving from a risk of sudden breakage of the ligament (CP failure mode) to a problem of too large deformation possibly in combination with a too large creep rate (associated to a RD failure mode). This seems a general trend based on the experimental investigation scope of the sample SG35.

From a more practical point of view, cdr-tests seem at the time being mainly useful to determine values of force to use for reaching failure in creep tests for relative short test durations; but they seem relatively useless with regard to the prediction of deformation and accordingly of failure modes due to excessive crack opening in creep load configurations.

V.3.4. Border effects for TCT-tests on SG-laminates

The analysis of TCT-tests results so far showed some trends in the typical response of the ligament for different test conditions of temperature and loading rate. However, in order to assess the feasibility of developing a characterization method based on TCT-tests, it seems necessary to refine the analysis on a more quantitative way. This implies identifying further sources of systematic deviations peculiar to the used TCT-configuration, and estimating these as far as possible.

Two aspects were further investigated with the sample SG35, the influence of the width and of the initial state of the TCT-specimen. The second effect appeared to interfere on results between series initially aimed to investigate the influence of the width; the detection of this interaction of effects arose a little incidentally.

The influence of the width has firstly been investigated by means of a cdr-series similar to the initial series s1, performed at 20°C and for same values of applied displacement rate but on narrower TCT-specimens, with a width of 30 mm in place of 50 mm (series s1b, see also Table V.1 and paragraphs V.2.2 and V.2.3). To allow a comparison between the results of the two series, the measured peak load values were plotted by unit of width against the logarithm of the applied displacement rate (series s1(a0) and s1b in Figure V.18). The rate dependence of the results of both series appeared similar (same slope on the semi-log plot), with similar CP failure patterns, but with a significant distance between both regression lines. This last difference was consequently assumed to be due to edge effects (a particular type of size effect), thus mainly in relation with differences of geometry between the test specimens.

In a later step of the experimental campaign, after the initial test series s1 to s9 had been performed, the defined purpose was to start investigating the reproducibility of some testing conditions and to complete some earlier test series. A cdr-test carried out at intermediate displacement rate for completing the initial cdr-series at 20°C (series s1(a0)) gave a result deviating noticeably from the initial regression curve : not only the measured peak load value for the new TCT-test appeared to be significantly larger, also the failure mode had changed into a RD failure pattern. The trend was confirmed by means of a couple of new tests, which were regrouped in the series named s1(a1).

Figure V.18 compares the respective measured peak load values by unit of width of the TCT-specimen for test series s1b and s1(a1), with the results of the initial series s1(a0). The difference between the test specimens of these three series, in term of width and of initial state (described in term of the storage duration between lamination and testing times), are summarized in Figure V.17. The respective deviations of the regression curves for the narrower and ‘older’ specimens (respectively of the series s1b and s1(a1)) with the preceding results of the initial series s1(a0) are in line with the assumption that the ageing process due

to stationary storage conditions decreases as a logarithmic function of storage duration (see Chapter III paragraph III.2.2). Accordingly, the initial state of the narrow specimens of the series s1b can be supposed much more closer to the one of series s1(a1) than to the one of series s1(a0), based on their respective storage durations. This triangular comparison suggests that edge effects due to geometry of the specimen (variation of width) and of possible geometry dependent ageing phenomena (for instance transport processes through the lateral free edges) are of smaller extent than firstly suggested by a simple comparison of results of test series s1b and s1(a0).

However, this interpretation of the results of Figure V.18 does not give an explanation for the observed difference in failure modes between series s1b (CP) and s1(a1) (RD). Issues related to the interpretation of results of TCT-tests in function of observed failure patterns, in particular when these are performed at constant displacement rate, are further addressed at the end of this Chapter.

Following this interpretation of the test results, it was proposed to investigate whether the initial ageing state of the specimens could effectively be modified by a thermal pre-treatment applied previously to the TCT-tests. It was opted for applying a thermal treatment to two TCT-specimens (series s1(a2)), consisting in exposing these at a temperature of 40°C for a couple of days (2.6 days); this conditioning was supposed to correspond to an “annealing” process accelerating the rate of physical ageing in the interlayer of the TCT-specimens, and was thus expected to further increase their resistance to yield (thus the value of the peak load in a cdr-test). The “annealing” character of the treatment was thus related to a conditioning temperature slightly below the glass-transition temperature. After having applied this pre-treatment to the specimens, they were cooled down at a rate resulting from a natural exposure to the room temperature in the lab, and the tests were performed within the next 24 hours.

This thermal pre-treatment appeared however to have an opposite effect to the initial expectation of an “annealing” effect. The measured peak load values of series s1(a2) are intermediate between the ones of the two previous series, and so are the corresponding failure patterns (see Figure V.18 and corresponding loading curves in Figure V.19).

On the basis of these different results, physical ageing seems to have a rather favourable effect on the mechanical performance of an interlayer ligament to a cdr-loading mode, by increasing its bulk tensile yield strength in larger extent than its interfacial strength, what is rather promoting a RD failure pattern.

Would the observed trend be similar for the response of ligament to creep loading mode ? As no correspondence had been found between cdr and creep test results with regard to the isometric curves (see previous paragraph), it was judged useful to investigate the influence of different initial ageing states on the response of

TCT-specimens to a creep load mode on a direct way. It was also decided to investigate in parallel the influence of the TCT-specimen width on creep test results. The test series s2(a3) and s2b(a3) were carried out in accordance, with values of applied creep load on the narrower specimens reduced proportionally to their width, in order to get a same value of the load by unit of width (or same nominal stress) for the two geometries.

The identification of systematic trends in the results of the three creep test series (s2(a3), s2b(a3), s2(a0)) seems however less straightforward than with the previous cdr-tests (an overview of the 'initial ageing states' by series is provided in Table V.4). The corresponding results are presented on semi-log plots and by series in Figure V.23 (series s2(a3)) and Figure V.24 (series s2b(a3)). No significant deviation of time-to-breakage values appear between the three series[49], but the isometric curves appear to be tighter in the two last test series in comparison with the initial one : their position seem to move towards larger values of time-to-failure and thus closer with the time-to-breakage curve, altogether with a reduction of their slope in accordance. This corresponds to creep curves showing a more important variation of the creep rate in function of the loading duration, and this in the small crack opening range. This change in shape of the creep curves for specimens with a longer storage duration is also more pronounced at the lower loading levels. The creep curves in tiled frames of Figure V.23 and Figure V.24 show, in comparison with the ones of Figure V.22, a more pronounced inflexion point in a crack opening range comprised between 0.1 and 0.6 mm (where $d < t$) : the creep rate seems to be reduced below this inflexion point, and increased above.

Table V.4 – Definition of initial ageing states for the different test series

Test series			Initial ageing state	Nb. of tests
cdr	s1(a0)	SG35-04 .. 06	a0 : 1 week after lamination	4
	s1b	SG35-55 .. 57	aX : a0 + 6 months storage	3
	s1(a1)	SG35-43 .. 45	a1 : a0 + 9 months storage	3
	s2(a2)	SG35-46 .. 47	a2 : a1 + 2.6d@40°C	2
creep	s2(a0)	SG35-07 .. 10	a0 : 3 weeks after lamination	4
	s2(a3)	SG35-49 .. 51	a3 : a0 + 12 months	3
	s2b(a3)	SG35-58 .. 60	a3 : a0 + 12 months	3

[49] A deviation of 0.5 decades is not considered as significant.

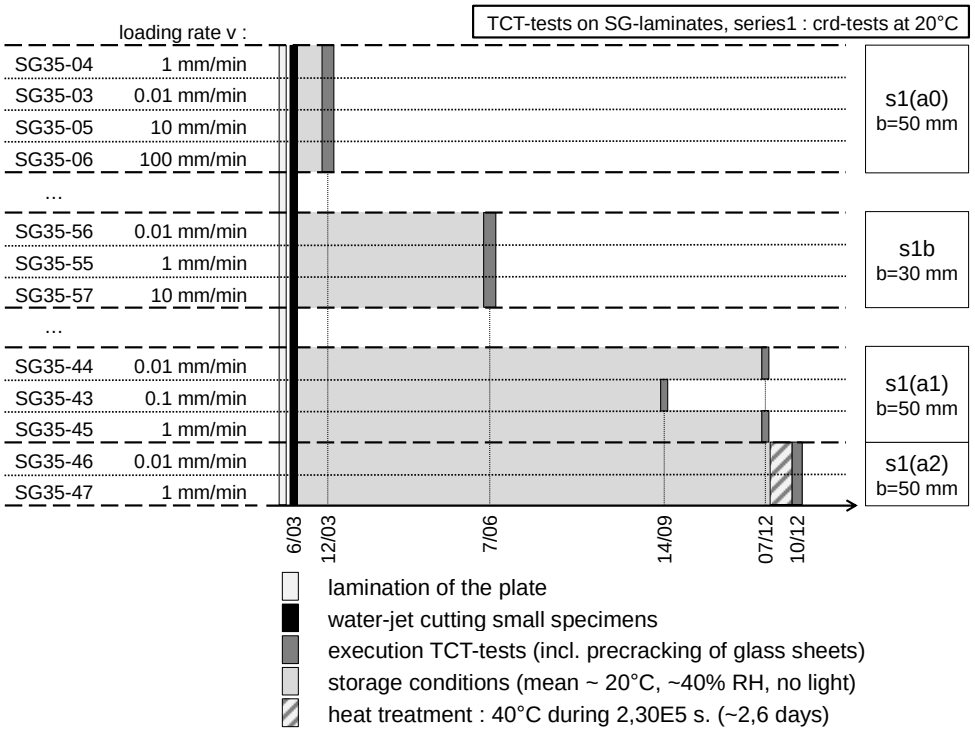


Figure V.17 – Difference of specimen's initial ageing state between cdr-series

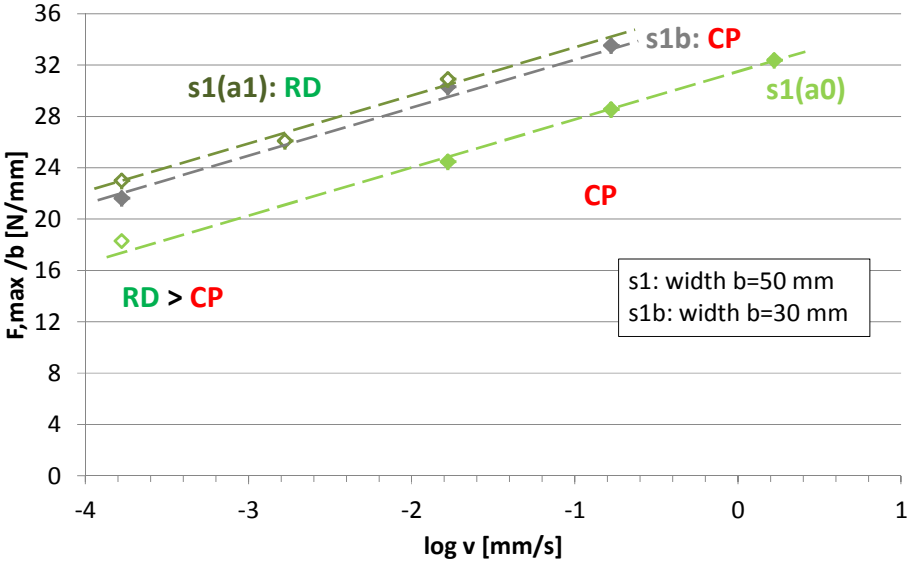


Figure V.18 – Comparison of cdr-series at 20°C : variation of peak load value and failure mode for different initial ageing states (storage duration) and different specimen widths

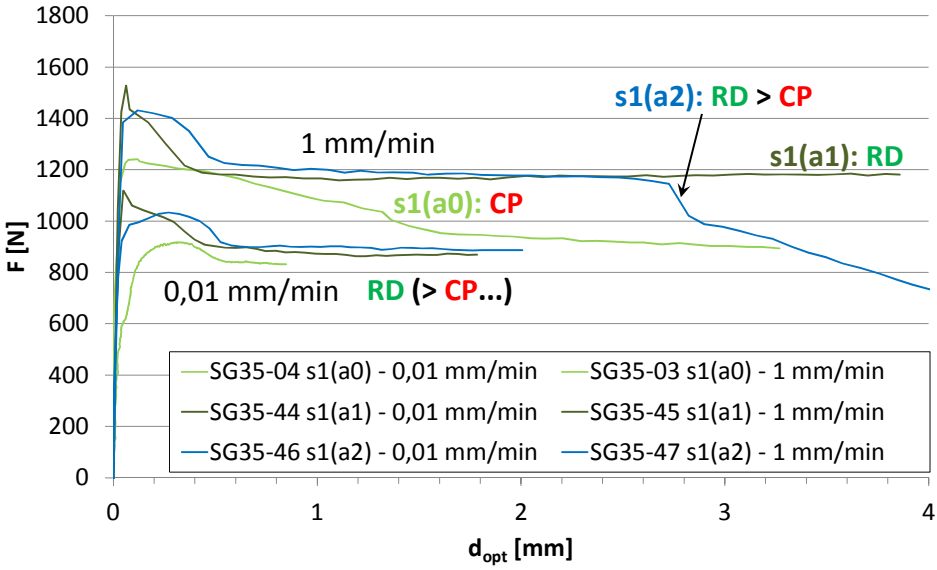


Figure V.19 – Comparison of cdr-series at 20°C : comparison of loading curves for different initial ageing states (storage duration / thermal treatment)

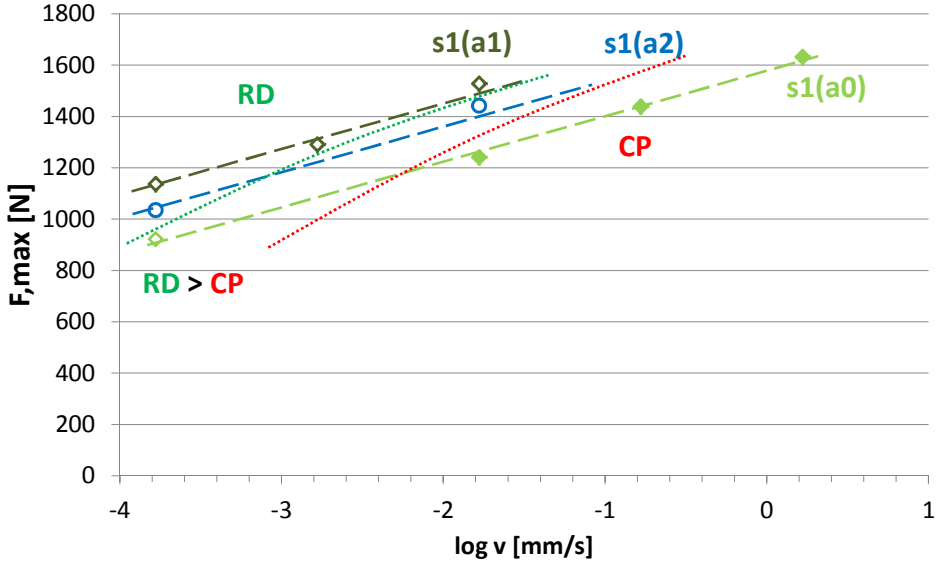


Figure V.20 – Comparison of cdr-series at 20°C : variation of peak load value and failure mode for different initial ageing states (storage duration / thermal treatment)

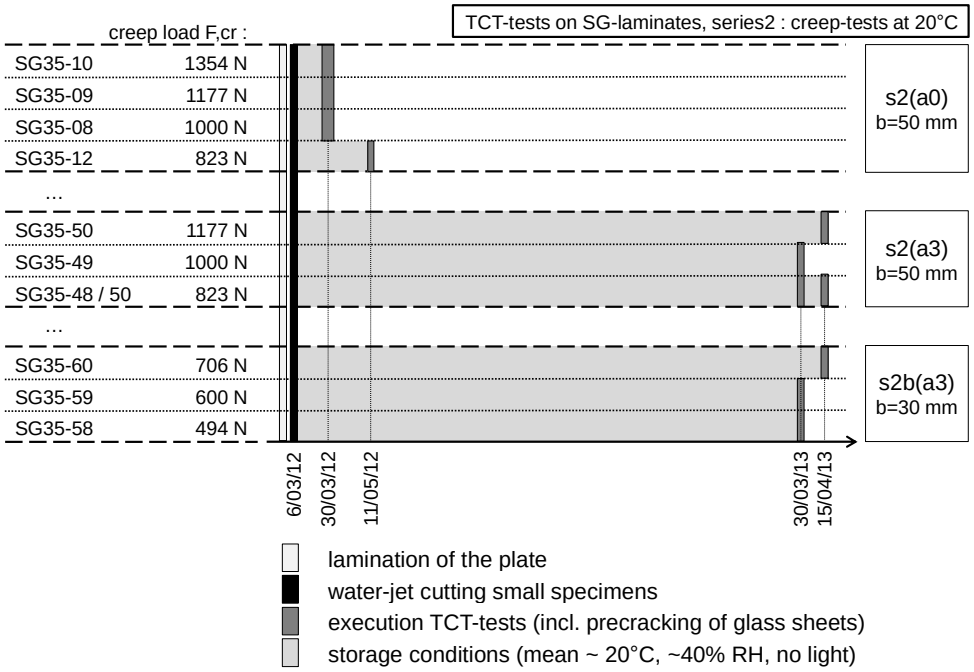


Figure V.21 – Difference of specimen's initial ageing state between creep series

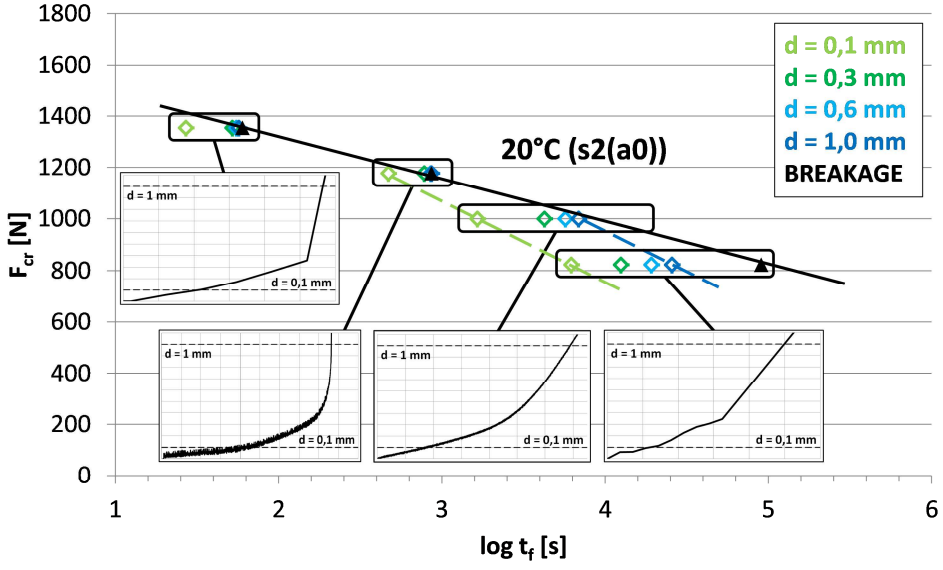


Figure V.22 – Results of creep tests at 20°C on TCT-specimens : initial test series (s2(a0))

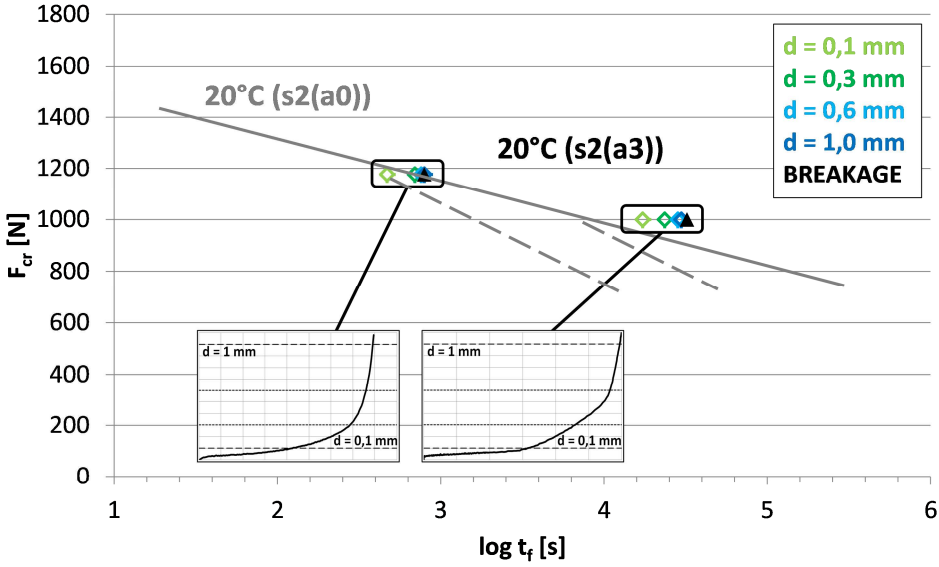


Figure V.23 - Results of creep tests at 20°C on 'older' TCT-specimens (s2(a3))

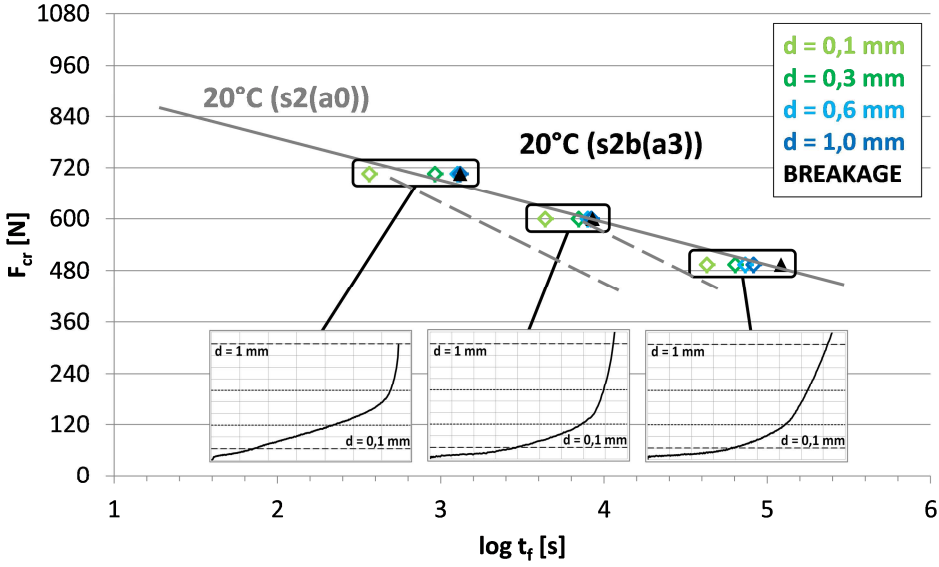


Figure V.24 - Results of creep tests at 20°C on 'older' and narrower TCT-specimens (s2b(a3))

Trends between the respective creep curves are getting more noticeable when considered in a slightly larger range of crack opening, and grouped by loading level (Figure V.25). Segments with a similar creep rate (same slope) appear between the different creep curves corresponding to a same loading level, which seem “activated” at similar value of crack opening but at different values of loading duration. Simultaneously, the value of the critical crack opening corresponding to a breakage of the ligament (point BP) clearly decrease for the ‘older’ specimens of series s2(a3) and s2b(a3). However, this reduction in critical crack opening capacity does not appear when focussing in the small crack opening range (for values of $d \leq 1$ mm $\approx t$), where the creep curves seem shifted in time. This last trend suggests that mainly activation of deformation mechanisms are sensitive to a different initial ageing state, rather than the deformation mechanisms on their own (similar creep rates); besides, this sensitivity seems limited to the time-to-activation (or time-to-failure), and not to the deformation state (value of crack opening for which a new activation is noticed). In other words, ageing modifies the position of first inflexion points on the creep curves along the horizontal axis (loading time), but not (significantly) along the vertical axis (crack opening).

As with cdr-tests, a difference of initial ageing state seems to have a more important influence on the creep response than the width of the TCT-specimen.

These different results show that general conclusions about influence of border effects on the response to creep load mode are not straightforward, and that the trends in response can appear differently according to the range of deformations considered.

A general important conclusion of the present analysis is that the initial ageing state of test specimens in SG-laminates has an important influence on test results performed at usual room temperature, and by consequence, the outcomes of assessment tests can vary on a significant way in function of storage and test conditions. This seems therefore an important aspect to account for in the perspective of assessment or validation tests, and especially when full-scale tests on unique specimens are involved.

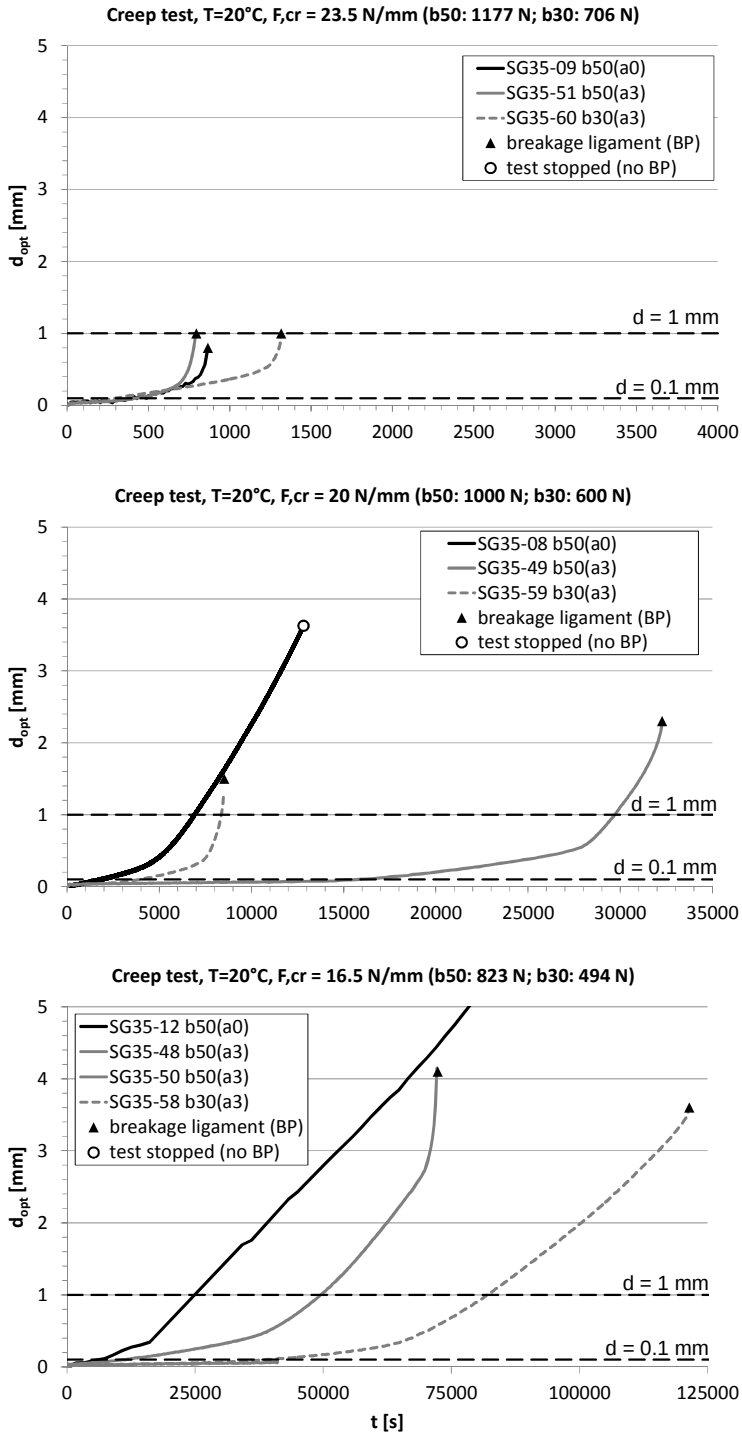


Figure V.25 – Border effects for creep tests at 20°C : comparison of creep curves by loading level, influence of initial ageing state and specimen's width

V.4. Outcomes of the experimental campaign TCT-tests

The experimental campaign TCT-tests performed on a sample of specimens SG-laminates (SG35) reported and analysed in this chapter leads to outcomes of different natures.

The presented test results firstly give a more concrete picture about the time-temperature dependent response of an interlayer ligament in fractured SG-laminates to various test conditions of temperature and loading level which can be observed experimentally. A parallel is observed in the response to two loading modes, when TCT-specimens are loaded at constant displacement rate (cdr-test) and under a constant value of applied force (creep test), and suggests a thermorheological complex behaviour of the interlayer. However, the correspondence of the TCT-response with a thermorheological complex model is solely phenomenological, because of the larger amount of deformation mechanisms involved, and the difficulty to dissociate experimentally their respective contributions on basis of univocal criteria.

The macroscopic ductility results from a combination of different mechanisms, the stretching of the released interlayer ligament and two crack propagation patterns : an interfacial delamination of the interlayer from the glass substrates (named regular delamination pattern – RD pattern) and a crack propagation through the cross-section of the ligament (named CP pattern). When the RD-pattern dominates its response, the TCT-configuration can survive to larger crack opening and appears as more ductile, whereas a dominant CP-pattern leads to critical failure at smaller value of crack opening. RD failure mode and accordingly ductility of the TCT-configuration is promoted at higher test temperature and lower loading level, while CP failure mode is more likely to occur at lower test temperature and higher loading level.

Whereas the presented test results are believed to help getting a comprehensive order of magnitude of the influence of the different test parameters on the behaviour of SG-laminates, a reliable quantitative interpretation of the test results appears to have to account for complementary aspects.

The second analysis axis developed in this chapter aimed to discuss the representativeness and the reliability of the performed TCT-tests and of the obtained results. In order to assess the TCT-test configuration in a more general perspective, specific attention has been dedicated to describe and identify possible and observed sources of systematic deviations on test results. A variety of experimental aspects have been identified and discussed, and orders of magnitude have been estimated for a series of measurement uncertainties. Among experimental border effects, the influence of the initial ageing state of the test specimens due to storage duration and conditions appears as a particularly important effect to take into consideration for experimental assessment of SG-laminates.

The developed experimental approach shows that the application of an incremental experimental strategy based on short successive test series allows to investigate the response of a load-transfer mechanism on a relative large experimental scope with a relative small amount of test specimens. The feasibility of using such an approach in the perspective of characterizing performances of laminated safety glass products is further discussed in the next chapter.

However, the test results presented in this chapter do not provide concrete information about the reproducibility of TCT-tests. Reproducibility is indeed an important feature to assess for developing test standards, and this addresses at first the scattering of results for test series constituted of specimens and for test conditions supposed to be equal. Despite the lack of quantitative results in that regard with the above campaign, it is possible to make some comments and prospective analysis. Firstly, it is expected that with enough care in performing TCT-tests and in controlling sources of systematic deviations, the scattering can be reduced to orders of magnitude similar to other test configurations with similar test conditions, for instance for cdr-tests compared to uniaxial tensile tests on interlayer specimens. However, investigation of scattering in creep test results and of the achievable grade of reproducibility with such tests seems to deserve some priorities with regard to structural applications and the higher level of complexity of creep test results.

Interpretation of result scattering for test series on laminated glass units should be made critically : it cannot be automatically allocated to an effective variation of product properties, and it seems necessary to consider the contribution of experimental uncertainties. Besides, it is expected that for a same test configuration, the scattering of TCT-test results may vary significantly for different test conditions, in function of the characteristics of the tested interlayer material and the specificities of the test configuration. For instance, a larger scattering in results is likely to be expected when test conditions involve transition or ageing mechanisms activated at their highest rate[50], or when crack propagation patterns reach a more unstable mode. Larger dispersion of results is expected accordingly for instance for tests performed at a test temperature in the range of the material glass-rubber transition temperature, and in test ranges where a transition in crack propagation patterns has been observed. One should also remain careful, on a general way, in making comparative interpretation of result scattering based on derived variables involving different order of magnitude of underlying parameters, typically with parameters of the type strain and stretch.

[50] See also Chapter III and Figure III.19.

It should however be kept in mind that a low scattering in TCT-test results (or for any similar test configuration associated to an intermediate experimental scale) is not necessarily a sign of a good accuracy nor of a good representativeness. Again, questions addressed to scattering of test results rise the risk of non-reasonable increase of the amount of required tests, and should therefore be considered cautiously with respect to the identification of systematic sources of deviation, whether the latter are “included” or not in the observed scattering of results.

Chapter VI

Synthesis and perspectives

“Always have a backup plan” (Milena Kunis, American actress, born in 1984 in USSR)
“If you have a backup plan, then you've already admitted defeat”
(Henry Cavill, British actor, born in 1983 in Jersey island)

VI.1. Update of the problem statement and research approach

The assessment of post-fracture performances of laminated safety glass products used in structural applications addresses various issues. A major one concerns the characterization of interlayer properties ruling the time-temperature dependent performances of fractured elements under quasi-static loading conditions. The initial onset of this research and the performed experimental works reported in Chapters IV and V focussed on one particular interlayer material, the SentryGlas® (SG). It appeared that to address the initial question – the characterization of the mechanical properties of a specific interlayer product ruling the post-fracture performances of laminated glass systems – it was necessary to question more fundamentally experimental assessment strategies for a category of construction products.

"Structural glass" as a research area is in fact at the cross-over between different research fields, going from material sciences (with regard to glass and adhesive polymer materials) to development of laminated glass products and to structural engineering. Addressed issues are thus highly multi-disciplinary and inter-disciplinary in nature. Moreover, development of related research activities is confronted to a variety of ongoing 'harmonization processes' on the one hand, and to different interests and priorities on the other.

The development of assessment and design methods for new laminated glass products or new fields of use of existing ones involves closely interlaced processes, with a relatively large variety of stakeholders : designers, manufacturers, contractors, controlling authorities, etc. The amount of (remaining) related questions is proportional to the number of stakeholders and their respective field(s) of interest and intervention in the design process, and to the extent of application scope each is considering (in terms of configurations and of performances, of products and of final applications). These various questions are obviously seldom independent of each other. Nonetheless, they are usually investigated by means of different processes developing on different time scales, which appear not easy to 'synchronize' or make compatible with each other.

Many rationale reasons behind such 'synchronization' difficulties could be identified during this research : most of them appear largely and directly related to questions and issues about experimental investigation methods and their development. They can be related to fundamental difficulties to initiate close collaborative processes at early stages in rather competitive and quickly evolving environments, consequently complicating the development of robust concepts and methods necessary for building a "harmonized" reference framework.

The development of assessment (characterization) methods based on tests at 'intermediate experimental scales' (this thesis) has to be understood in this specific context.

The developed concepts and the findings presented in this thesis are firstly summarized. They are followed by suggestions concerning experimental investigation programs in this field with regard to their support to the development of a "harmonized" reference framework to facilitate the design and the assessment of innovative structural applications with laminated safety glass products. The addressed issues concern more particularly :

- the improvement of the complementarity grade between test results obtained from experimental investigations performed with regard to short-term and long-term objectives, in particular between assessment and research activities and project-related and product-oriented assessment approaches; and
- the related developments of comprehensive and compatible models and design concepts.

VI.2. Summary of the research and main outcomes

The current European standardization context framing the assessment of laminated safety glass products and their use in non-conventional, possibly structural, applications, has been presented and analysed in Chapter I. It appears that the safety performances as assessed by Initial Type Testing in the product standard implicitly consider a relatively narrow application scope, roughly limited to use of laminated glass products as vertical framed glazing elements. The addressed safety performances concern mainly their capacity to resist to different types of impact or other dynamic actions, and the assessment does not deliver design properties. These are obviously not sufficient with regard to performance requirements in many other configurations, and principally for non-conventional ones. The main shortcomings concern the consecutive post-fracture performances in function of the interlayer time-temperature dependent properties. However, it is acknowledged that the assessment of products performances is getting complicated with regard to the simultaneous enlarging of the "family of products", a more vague and evolving "intended field of use" (application scope) and involved smaller production volumes. It led to describe application scopes by means of a combination of *Application Fields (AF)*, to distinguish the nature of 'similar' ones and to identify more precisely the extension fields concerned by non-conventional configurations.

The safety concepts and assessment methods for structural applications in laminated glass were considered closer in Chapter II. The different steps in failure scenarios were detailed. It led to distinguish quasi-static design situations for fractured states by means of a *first important statement* that any crack propagation process in the glass sheets is a dynamic event. This allows to dissociate the assessment of the contribution of the interlayer to post-fracture performances from all the issues related to dynamic behaviour. Load-bearing performances of fractured laminated glass elements are resumed to two load transfer mechanisms, which develop in different proportions according to the

configuration of the fragmentation patterns. The ligament function across a cracked cross-section, or TCT-configuration, is identified as the *critical load transfer mechanism* in ultimate fractured states. The description of physical damage of fractured elements is completed by means of *an initial delamination length* a_0 from the crack tips and along the interface between glass fragments and interlayer. This follows from a *first important assumption* that the ligament cross-section is not damaged during the successive steps of the failure scenario, and relies on an implicit criterion on the adhesion level. In fact, the mechanical performances of the TCT load transfer mechanism is shown to depend on two complementary deformation mechanisms, the stretching of the interlayer ligament and its delamination from the glass substrates. The latter proves to play a dominant role in the ductility of the TCT-configuration, and relies on the balance between bulk and interfacial properties of the laminate. Finally, the TCT load transfer mechanism is shown to require a *minimal activation length* ℓ_{act} on each side of the cracked section to fully develop its load transfer ability, which is suspected to be in most cases an order of magnitude larger than the delamination length. More generally, it is pointed out that the representativeness of a TCT-test configuration is not necessarily straightforward because of possible variable size effects, with respect to the dimensions of the crack-tip stress fields contributing significantly to the delamination processes relative to the other geometric parameters of the problem.

Specificities of polymer materials and their mechanical behaviour are investigated in Chapter III. They appear characterized by a large strain response and different temperature dependent transition mechanisms affecting their processability and their mechanical properties in service conditions. Interlayers can belong to two families of products, thermoplastics and elastomers, distinguished by the nature of their secondary intermolecular bonds, and consequently by the typical temperature ranges in service conditions with regard to their glass-rubber transition temperature. Typical interlayer materials considered (PVB and SG) are thermoplastics. The mechanical response of thermoplastics in service conditions exhibit a viscoplastic behaviour characterized by a time-temperature dependence of the yield stress, which is also a measure of the resistance to creep. The corresponding mechanical behaviour is thermorheological simple or complex according to the amount of relaxation mechanisms present and in function of the test conditions. *Physical ageing* is identified as an important reversible phenomenon to account for at temperatures below the glass-rubber transition, due to a lack of equilibrium of the glassy phase. In particular, progressive physical ageing tends to increase the long-term creep resistance in comparison with extrapolated behaviour from tests of short duration. Physical ageing and progressive physical ageing are described by means of the same ageing state function. When used as adhesive components, viscoplastic properties of the polymer component are supposed to possibly vary differently due to a different effect of physical ageing on bulk and interfacial properties, what is acknowledged

by distinguishing two 'material' state parameters $S_{a,B}(t)$ and $S_{a,I}(t)$ respectively. The various polymer specific aspects induce a series of complementary constraints on experimental investigation methods, among others for evaluating the representativeness of test specimens of small dimensions.

At this state, it appears that the four added parameters – a_0, ℓ_{act}, $S_{a,I}(t)$ and $S_{a,B}(t)$ – to describe fractured states of laminated glass elements cannot be measured or quantified easily. However, they are conceived as important complementary parameters for describing fractured states, together with the description of fragmentation patterns of the glazing components. They are considered as constitutive components for a quantitative description of the physical damage of fractured elements. They also acknowledge for possible non-negligible invisible effects or damages to account for when performing experimental works on specimens laminated glass, whether due to production, storage or test conditions. It could be sufficient in a first step to determine in which conditions these parameters may be assumed to be equal between different conditions or states, and similarly under which their respective value can be assumed to not vary significantly in time.

Finally, the combination of these various material aspects in the context of the assessment of performances of end-products led to prefer experimental investigation methods based on the concept of *critical basic shape*. The transposition of the concept to laminated glass products lead to rather consider the interlayer as a *component* than a material. Accordingly, tests on specimens laminated glass are preferred for characterization purposes, and the TCT-test configuration seems a good experimental configuration for investigating the ligament performance.

Chapter IV reported on a succession of experimental campaigns for investigating the performances of fractured laminates, by means of tests performed at different experimental scales. A first aspect addresses different types of experimental issues related to the development of test methods for investigating the time-temperature response of interlayer ligament. For that purpose, an analysis grid is proposed for identifying and describing different categories of *Experimental Fields of Investigation (EFI).*

With the introduction of this concept in combination with the analysis grid for the Application Fields, *three types of border effects* can be identified, corresponding to different sources of systematic deviations in test results. The first type is mainly related to the representativeness of the test specimens, the second to the test configuration and systematic deviations in measurement, and the third to systematic deviations due to the analysis and modelling methods. The two first types of border effects can be managed by the conception of experimental configurations, in terms of test infrastructures, measurement methods and test protocols, and in terms of test specimens and experimental program. The third

one is rather depending on how test results are further processed and possible propagation of uncertainties, caused or not by border effects of the two first types.

This provides a framework to distinguish intrinsic and extrinsic qualities of test methods, and distinguish systematic deviations arising from the test methods or the modelling approaches. Among others, questions about accuracy and precision of methods should distinguish analysis performed at the level of primary experimental variables, or of secondary, derived parameters. The analysis grid with EFI's is also used for describing *extension potential and limits* for different test configurations. It is shown that developing a test configuration for extending the ranges of *a combination of* experimental investigation fields, in particular of test temperature and loading ranges, is confronted to successive technical limits and experimental issues. The identification of these limits and sources of experimental uncertainties determines the extension potential of a particular test configuration, and consequently its robustness to be used as an assessment method for a range of configurations of laminated glass products.

The developed analysis led to conceive an experimental campaign, reported in Chapter V, based on TCT-tests and performed on one single sample specimens of SG-laminates of about 60 test specimens, for investigating the time-temperature response of the ligament in different conditions of temperature and loading level. The experimental strategy is based on a non-conventional, incremental approach of successive short test series, in order to *investigate with a limited amount of tests an application scope as large as possible*, in terms of *test temperature* and *loading mode and level*. Similarly, the analysis method of the test results has been progressively refined during the campaign. This approach delivered comprehensive orders of magnitude about a series of effects.

The test results for the sample SG-laminates correspond to an (apparent) *thermorheological complex* behaviour, however analysed at a macroscopic and phenomenological level. The relative complex distribution of efforts in TCT-test specimens and the corresponding stress patterns lead to distinguish different deformation mechanisms and failure modes. A correspondence between the time-temperature dependence of the response of TCT-tests conducted at constant displacement rate (cdr-tests) and under constant force (creep mode) is noticeable, when respective values of yield forces and times-to-breakage are compared. However, *it does not seem possible to establish a similar correspondence between the two loading modes for failure criteria due to excessive deformations* (crack opening). The apparent macroscopic ductility under static creep forces appears to be increased significantly, together with values of time-to-failure, by lowering the loading level (which is the only parameter that can be significantly modified by the design in the conditions of a building project).

In comparison, a variation of the specimen width seems to have no significant effect on test results, at least of a lesser extent than the observed effect of physical

ageing due to the different storage durations of the test specimens. When considering the response to cdr loading mode, the effect of physical ageing seems rather favourable, with an increase of the peak resistance together with a more favourable subsequent failure mode. Observed effect of physical ageing on the response to creep loads in tests of relative short duration is however less univocal. A favourable effect of ageing in the form of a diminution of the secondary creep rate, and a consequent increase of times-to-failure in the small deformation range seems accompanied by a non favourable increase of the creep rate at larger levels of deformation. The modified response corresponds to a more sudden failure behaviour at a smaller value of crack opening, due to the start of a non-stable crack propagation through the cross-section of the ligament. Accordingly, effect of physical ageing on the response to creep of the ligament seems to correspond to a diminution of the macroscopic ductility. On an average, physical ageing seems to have a rather favourable effect on mechanical properties, as the design failure modes in practice is likely to correspond to a criterion on the maximal deformation in the range of small values of crack opening.

The experimental basis was however too limited to draw definitive conclusions about the effect of physical ageing, but indicates clearly that it is an important effect to account for in designing experimental programs and test protocols. The parameters showing the largest influence on the failure mode, the yield stress and times-to-failure, are however the test temperature and the displacement rate or the loading level. The graphical format selected for presenting the test results allows to get a first comprehensive order of magnitude of the various effects on the ligament response in the various test conditions. These results show also that the cdr loading mode finally is of limited relevancy with regard to the ranges of behaviour and conditions of practical interest for the considered formulation of the post-fracture quasi-static design conditions. The cdr-loading mode is, in an assessment perspective, essentially useful to determine loading levels of creep tests for achieving failure on a reasonable experimental scale. Trails for further developing an assessment strategy based on TCT-tests are summarized below.

VI.3. Conclusions and perspectives

On the basis of the developed analysis and performed experimental campaigns reported in this work, some elements of answer can be given with regard to the questions formulated at the end of the Chapter I.

1) How could and should existing test methods used for assessment of laminated safety glass products be completed to distinguish the contribution of individual components to the overall safety performances ? What are the *characteristic* properties of each component involved ?

    - Accurate measurement methods of deformations in general are necessary with regard to failure modes by excessive deformations; this is expected to be the critical type of failure mode in a majority of cases when the critical load-transfer mechanisms is the interlayer ligament in fractured stages (Chapter IV and V)

    - The characterization of properties of glass components should account for lower and upper limits of the glass strength, with regard to the risk of damage from the released strain energy at breakage (Chapter II).

    - The determination of the contribution of the interlayer should be assessed by considering it as a component rather than as a material, and test configurations should be designed in accordance. In particular, separate characterization of adhesive properties and bulk material properties of the interlayer seems not really useful with purpose of assessing the end-performances of products (Chatper II and Chapter IV).

2) Which (mechanical) properties of interlayer materials are involved in safety and post-fracture performances of laminated glass units and systems ? According to which methods can these be *characterized* for design purposes, in particular properties potentially significantly sensitive to time-temperature-ageing effects with regard to service conditions ?

    - The contribution of the interlayer can be associated with two main load transfer mechanisms. The load transfer mechanism likely to be the critical one in a majority of design situations of structural element in laminated safety glass is the ligament behaviour in a TCT-configuration. The ductility of the mechanism depends at least as much on the level of adhesion than on the bulk properties of the interlayer (Chapter II).

    - The TCT-test seems an appropriate test configuration, *provided* that the representativeness of the test specimens is assessed with regard to the processing method used for making them, in relation with production methods of end-products. If the TCT-test configuration is judged non-representative or non-achievable with some materials or product configurations, development of alternative test configurations should consider *in parallel* technical limits or issues for developing the test

configuration and for making the test specimens. The fitness for purposes of a test configuration has to be evaluated for each extension of any EFI-field (Chapter IV).

- Each experimental scale is confronted to specific, non-avoidable border effects, which can further vary in extent according to considered *combinations of EFI's*. However, not all present border effects are even or systematically problematic, according to the specific purposes defined for each experimental scale, and the applied analysis method in accordance. Focusing on the elimination or reduction of one or several specific border effects can give rise to other ones potentially more critical for the reliability and representativeness of the test results (Chapter IV).

3) To which extent are safety performances of a laminated glass element as a *construction work* (resistance to impact, etc.) depending on the *product* properties and on other characteristics of the installed *configuration* (in function of element configuration, type and configuration of connections / fixings) ?

   - This question has not been much considered in this work. The analysis grid based on different categories of Application Fields however accounts for the importance of this parameter with a dedicated category for describing design configurations (Chapter I).

4) Which characteristics of the laminated glass product or product family (from preliminary technical documentation) could be accounted for to select or develop a suited experimental investigation program for assessing their safety and/or post-fracture performances ?

   - Estimation of lower and upper strength of glass sheets with regard to the considered ranges of test conditions can be useful for designing test configurations (Chapter II).

   - The results of the reported TCT-tests campaign on a sample SG-laminate give orders of magnitude of the response of a ligament configuration, which can be useful to design other test configurations with this kind of products. However, these results do not give (so far) much more than an order of magnitude… (Chapter V)

   - The family of the interlayer product (thermoplastic or elastomer) and information about the characteristic temperatures are the most useful for situating individual test results with regard to an application scope for the behaviour (Chapter III).

5) How to conciliate application and product-oriented assessment procedures, in particular to keep the amount of requested tests within reasonable proportions in regard to the identified application scopes ? How to integrate vague and evolving application scopes in assessment processes, in particular with regard to particularities of adhesive polymer materials in terms of mechanical

behaviour and influence of production processes on their mechanical properties ? How to express and assess limits of possible fields of use of laminated safety glass products for structural applications ?

- Test configurations corresponding to an intermediate experimental scale have a potential to act as an interface between project-oriented and product-oriented experimental approaches. (Chapter IV and V)

6) Which material and structural models are applicable for characterizing the contribution of interlayer materials to the response of fractured products and systems, and how can the corresponding design parameters be calibrated or validated ?

   - There is no univocal response to this question. There is however a series of aspects to consider in order to obtain quantitatively relevant test results, and which are related to the identification of sources of systematic uncertainties and related border effects (Chapter IV).

   - It could appear useful to develop a third analysis grid, similar in its conception with the analysis grids with the EFI's and the AF's. Such an analysis grid could serve as a supporting tool for designing robust numerical model configurations and for assessing their fitness for purpose, among other by clarifying the (assessed) limits of use to users. It could then lead to define "families of model configurations" identifying the possibilities and limitations of combining different types of model elements into more advanced numerical models. It is expected among others that the sensitivity of (numerical) models and of experiments to uncertainties and to propagation of errors for this kind of problem can be possibly very different, according to the considered modelling approaches. However, modelling issues have not been investigated in this work.

7) In summary, are the safety concepts, assessment approaches and calculation methods developed for laminated glass products used as glazing unit appropriate and transposable for the design and assessment of structural glass works ?

   - If this question has to get only a short answer, then the response is "no"…

VI.3.1. Multiple purposes of tests and assessment strategies

The debate is not so much about which experimental scale or configuration is the most appropriate, but what are the advantages and disadvantages of each, how to evaluate these, in order to determine how the different experimental scales are complementary with each other. Besides, other aspects than border effects have to be considered; some are further discussed here.

There is little doubt that the different experimental scales have complementary functions in experimental assessment strategies of polymer components. There

are possible overlaps between different experimental scales, what means that for some purposes, test methods at different experimental scales[1] probably have the potential to reach a similar level of global accuracy. The complementarity grade between tests at different scales can probably be measured, and even improved, by developing finer methods and analyses for distinguishing the different types of uncertainties and border effects, taking also into account technical limits or issues specific to each test configuration for extending EFI's.

Managing border effects related to time scales, as correspondence between short term and long term behaviour, is certainly trickier, but could be already partly facilitated by a more accurate evaluation of the other border effects. Available theories and experimental observations show that time-temperature and time-stress equivalencies cannot be reduced to simple "shift functions" for thermorheological complex materials or in case of progressive physical ageing during the loading duration. Besides it seems that the different types of ageing effects can affect the bulk and interfacial properties of the interlayer component on differentiated ways.

It seems in general useful to make a clear distinction in analysis between primary (measured) and secondary (derived) experimental variables or results.

VI.3.2. Tests at intermediate scale : necessity and issues

A series of arguments has been given through this work in favour of tests at intermediate scales for assessing the post-fracture performances of laminated glass, in the context of an ITT assessment strategy. Tests on small-size specimens laminated glass are a particular sort of 'intermediate' experimental scales. Associated issues were also identified, which have been expressed by means of three types of border effects.

However, not only scientific arguments have to be considered. Some economic arguments were already mentioned : reducing the costs related to test specimens and the amount of tests, in order to keep a balance between compliance to safety requirements and development and assessment costs, with regard to identified application scope(s) on the one hand and to required experimental assessment program on the other. Besides, selection of intermediate experimental scales should account for a third aspect, namely the role they fulfill as intermediate (or interface) configurations between the different stakeholders, and accordingly between different steps of the design process. These are issues of knowledge transfer and are addressing different conceptual frameworks. This third aspect is essential to understand the problem of the choice of reference test configurations and test conditions, in particular in the context of ITT tests. A selected (initial)

[1] Here mainly the geometric EFI's of specimens and test configurations are considered…

test configuration is not neutral when used for comparing mechanical properties of interlayer materials or performances of laminated glass products : this is an unavoidable issue ! However, the induced bias peculiar to a reference test configuration on a comparison can be more or less harmful, according to the type and extent of (unidentified) border effects in presence, and the comprehension each party has about. It is thus very comprehensible that debates about this type of questions, whether in the context of research activities or technical committees, are difficult and progress slowly.

These considerations can lead to associate the concept of intermediate experimental scale to a role of intermediary between parties participating to the design process and associated decisions; consequently, intermediate experimental scales should probably also be considered as necessary intermediate steps in standardization and harmonization processes providing the framework and support for designers and control bodies. Accordingly, discussions about most suited assessment strategies and the associated imposed or accepted test methods should account for this role of intermediary. As such, tests at intermediate experimental scales developed and conceived for an ITT assessment today, are potentially validation tests for other (future) ITT assessment strategies, which would be based on more fundamental experimental approaches (and thus more general characterization of performances and properties of products).

Similarly, a ‘simple’ ITT determines performances or properties associated to the use of simple design or calculation models, whereas a more advanced ITT procedure involves the use of more complex models. Current technical guidance documents established for technical committees in charge of developing European product standards (CEN) and Guidelines for Technical Agreements (EOTA) promote the use of simple characteristic performances for ITT characterization, but it should not be considered as incompatible with registration of complementary test data usable for other purposes, as the development of more advanced design techniques and calculation models. However, this implies a close(r) collaboration grade between assessment and research activities, which is not always compatible with the expectations or commercial interests of manufacturers (confidentiality of test results,…).

There are however conditions for any test configuration considered as an intermediate experimental scale to get a chance to play such a role of intermediary, which can be taken into account for any experimental investigation step by means of measures at two levels :

1) conception of test configurations and associated measurement methods : as discussed above, it is possible to associate different purposes to similar or identical test methods, which can possibly find an echo into a distinction between primary and secondary experimental and measurement fields. An example has been given in Chapter V for TCT-tests, where applied force and

crack opening were considered as primary measurements, and delamination lengths as potential secondary ones (which appeared finally as non-measurable in most of the considered test conditions…);

2) structuration of test reports and results analysis, by distinguishing test results of primary and secondary level (respectively obtained by direct measurement and by derivation of direct measures[2]). This can be useful for different purposes : distinction between different types of border effects (namely identifying sources of systematic deviations or errors), back-up for allowing alternative analysis levels (in comparisons), modelling approaches or model developments (dissociation of experimental and modelling issues), testing different simplification techniques, etc. It is valuable especially when test results are not a few unique values, but more complex acquisition data sets, and when successive processing steps are considered for their analysis.

With this kind of approaches, non-conventional test methods on structural elements in laminated safety glass can be developed within a double strategy, for experimental assessment of a project-specific design and as a potential validation test with regard to the development of alternative assessment strategy of properties of interlayer components and performances of laminated glass products and systems.

Above considerations on intermediate experimental scales and intermediate reporting and analysis levels can be completed by a few statements by Moore & Turner (Moore and Turner 2001), about tests on (thermo)plastic products in general :

" […] the translation of force into stress and deformation into strain is a source of errors and approximations, so much so, that the transformed results may bear little relation to the fundamental properties"

" […] experimental results and quoted property data should not be divorced from the storage history of the test specimen. […] pretreatment, sample, specimens and test procedure should be regarded as a single entity and no property datum in isolation should be regarded as a unique characterizing quantity."

"The main subsequent difficulty rests on how results obtained in non-standard procedures may be utilized safely in a wider context where they may be at variance with corresponding data from a standard test. Ideally, the issue should be resolved by open debate but there are many practical obstacles to such cooperative and collective activity."

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[2] This type of distinction could also be based on measurement accuracy of the individual parameter, and their effect on the global accuracy.

This last thought can thus be completed : issues arising when comparing results from tests at different experimental scales should be resolved by open debates, which certainly can be supported by appropriate analysis and reporting methods.

VI.3.3. TCT-tests : most suitable intermediate experimental scale ?

TCT-tests on small specimens laminated glass appear as a convenient test configuration for investigating the time-temperature-ageing dependent mechanical performances of interlayer ligaments in fractured SG-laminates. The TCT-test configuration is judged as a potentially suitable experimental configuration for making a characterization of the ligament response[3], provided that the assumption that it has not been significantly damaged[4] by the event which led to the fractured state can be accepted, and that size effects are not significant or can be quantified. Different border effects were identified and a first quantitative estimation has been done for a series of them; Table VI.1 gives an overview of experimental issues for TCT-tests by means of the analysis grid developed in Chapter IV.

A validated expression of the mechanical behaviour at this level should allow to use it in simple structural models for fractured elements, by considering (simplified) critical fractured configurations combining rigid segments (in units working as linear elements) or fragments (planar elements) separated by TCT-sections where the ligament behaviour can be inserted in order to take over tensile efforts.

The suitability of TCT-tests in an assessment strategy can be addressed with respect to two different questions : how to complete the assessment of post-fracture performances of SG-laminates on the one hand, and how to apply and develop similar method to other interlayer materials on the other ?

For assessment of ligament properties in SG-laminates, different complementary development axes seem possible :

- For the considered interlayer configuration (fixed thickness), it is suggested to pursuit the investigation of ageing effects for different initial ageing states. The initial ageing state at the beginning of the TCT-test can be modified by different pre-treatments (annealing effect,…). However, it does not seem straightforward to specify a limited set of thermomechanical pre-treatments to identify and represent limits to possible variation range of state of the polymer component during service conditions. The choice of pre-treatments will be confronted to the duality of approaches on the same way than for artificial

[3] The considered expression is a relation between the applied force by unit of (crack) width and the opening of the pre-cracked section (crack opening), see Chapter II and Chapter V.

[4] Accounting for the three supplementary components identified for describing the level of physical damage of an element (the initial delamination lengths and the two ageing states functions).

ageing tests, between application oriented exposure conditions, and investigation of product/material sensitivity to exposure conditions. For such experimental campaigns, it seems useful to still foresee some test series with TCT-specimens of two different widths, in order to verify that edge effects (border effects due to geometry of TCT-specimens) are not increasing too significantly for different pre-treatment/testing conditions;

- The assessment can be completed by means of tests on different configurations of interlayer component (other thicknesses; other adhesion level/processing conditions). The amount of complementary tests for each variation of EFI can be kept under control by selecting the order of tests within an incremental strategy.
- Ligament models could be developed by means of TCT-test results, and should be validated by comparing the behaviour with results of tests at element scale (for instance creep tests on fractured elements), including tests on more complex laminated glass products, namely on test specimens with dimensions and composition closer to the ones used for structural components (with more than 2 glass sheets). For this process to be conducted successfully, proposed analysis grids can be a useful tool (to further develop…) for the identification of experimental border effects.

The assessment of ligament performances of other interlayer materials can probably be investigated by similar approaches, however the reference geometry and production process of the TCT-specimen should be considered carefully. Some border effects between TCT-specimens and larger test specimens could have another order of magnitude in comparison with SG-laminates, in particular for products fabricated by means of different lamination processes (cast-in-place interlayers,…), and also vary with changes of testing conditions. It is recommended to start with short test series for one component geometry (one thickness), with two widths of TCT-specimens, and to compare results with tests on larger elements (element scale), before launching more extensive experimental programs TCT-tests. In fact, it could appear that alternative test configurations are required in complement to the TCT-test for completing or extending a characterization procedure, or that these constitute better alternatives to the TCT-test configuration for characterizing TCT load transfer mechanism with regard to specifically considered application scopes.

Developing ligament models for different interlayer components is one thing. Assessing the application scope of such models in terms of application scopes (with regard to the different categories of Application Fields) is another one : this addresses the question how representative the test configuration used for investigating the ligament behaviour is in comparison to formed ligaments in other fractured configurations. It is thus necessary to determine further criteria, possibly based on measurement or test methods, for validating this correspondence.

Table VI.1 – Experimental Fields of Investigation for TCT-tests

Experimental Field of Investigation		**Main points of attention with regard to testing ranges, extension possibilities and limits**
	Specimen : Material	- limits for glazing sheets : annealed float glass - limits for interlayer : not damaged by the pre-cracking step of the glass sheets; edges not (too) sensitive to ambient air (storage / test conditions)
	Specimen : Geometry	'simple' laminated glass units, with - thickness glass sheets large enough for load-transfer - limited total thickness and limited width → jaws - length large enough for fixing the specimen on the testing machine without damaging the glass sheets
	Specimen : Processing	variable border effects possible, due to production method of the specimens (lamination, cutting…) and storage conditions between processing and test
	Specimen : Pre-treatment, Conditioning	- initial state of TCT-specimen related to : # pre-cracking step : initial delamination length # thermo-mechanical history : initial ageing state - equipment of specimen for fixing on the testing machine and for optical measurements
	Test configuration : Basic device	- universal testing machine, equipped with appropriate grips (jaws) - climatic chamber → pull-rods for grips required
	Test configuration : Geometry	geometry range limited by grips type and loading capacity range
	Test configuration : Loading configuration	uniaxial tensile loading state depends on : - system for load transfer in grips + distance to pre-cracked section + stiffness interlayer - alignment specimen in testing machine
	Test configuration : Control mode, Loading range	- steering modes limited by tensile machine + grips - loading range limited by tensile machine + load cell
	Test configuration : Measurement methods and measurement configuration	measurement method and calibration (deformations) : - compatibility / precision → testing conditions (with climatic chamber), loading rate and range - acquisition frequency → loading mode / range - calibration → loading / deformation ranges and rates + test conditions (with climatic chamber) - synchronisation of optical measures with main acquisition system (load,…)
	Test conditions : temperature,…	- temperature range : constraints on all other EFI's - lighting conditions for optical measures in combination with a climatic chamber

VI.4. Perspectives for further research

Different trails have been proposed above for conducting experimental campaigns relying on combinations of mechanical tests at different experimental scales, within an incremental assessment strategy.

Not much has been said on development of models and safety concepts, in particular with regard to 'harmonization' purposes mentioned in the first chapter. It is clear that developments in these fields are preferably conducted in close relationship with development of experimental investigation methods. This would help to keep a balance between achievable and required precision levels in tests and in models, and to specify reasonable levels in regard to considered application scopes and design practice. Compatibility of use between test results and models for design practice should also be further addressed. For that purpose, developing a consistent but flexible framework for the expression of uncertainties together with analysis tools for dealing with propagation of uncertainties could be valuable for accompanying further research efforts. Alternative to 'heavy' methods assuming (or requiring) that every uncertainty is determined on a statistical basis could be provided by methods and models based on fuzzy logic, allowing to perform analysis of propagation of uncertainties with no need for statistically relevant data.

So far only destructive experimental investigation methods have been considered and discussed. Non-destructive methods for measuring the adhesion level and the state of the interlayer component would be useful, especially if they could be applied at the level of ligaments in fractured states, for obtaining measurements in relation with parameters a_0, $S_{a,I}(t)$ and $S_{a,B}(t)$). In fact, if the physical/damage state of the ligament represented by these three parameters could be quantified by direct non-destructive measurement, it would allow to dissociate the description of the state from the causes, namely from the thermo-mechanical history. Such a measurement method would be useful among other to prevent to be too intrusive in the description and control of the production processes and interlayer material chemistry. However, it is uncertain whether measurement techniques exist that are potentially applicable in this perspective.

Notwithstanding the various issues possibly supported by further research, and in particular by means of further experimental investigations, it seems useful to take into account the evolutions in standardization developments at a relative early stage, in order to facilitate the valorisation of findings into design practice.

VI.5. About research, harmonization and implementation

It is clear that the development of an "integrated safety approach" (Bos 2009) for design of structural glass constructions requires the support of coordinated efforts in developing compatible experimental methods, calculation models and safety concepts. In that regard, the assessment of post-fracture performances of laminated safety glass products and systems addresses essential issues. However, these can probably not be supported by only more research in this field, but require also more coordination between different research initiatives and better integration of research processes. It is not sufficient to want to promote collaborative dynamics, it is necessary to think about the mechanisms and policies promoting such dynamics and making them attractive for all involved parties.

The development of "harmonized" standardization in this field is confronted to a variety of interests and expectations, among which particular industrial and national interests are not the less important in the ongoing debates. However, acknowledging the existence of different interests should not overlook other aspects, and in particular sources of misunderstanding. Some misunderstandings are caused by the state of development of the European standardization framework, its relative complexity, and the lack of comprehension and visibility of standardization processes and implementation strategies (especially for parties not closely involved in their development). In particular, there seems still to exist relatively little practical understanding about the fundamental philosophy of the "performance-based approach" promoted by European directives and regulations, and in particular about the practical implications it could or should have on the way research is conducted, structured and reported. In other words, the European standardization framework embodied successively by the CPD and the CPR, while appearing rather powerful and robust in its conception, seems weakened essentially because it is not *assimilated* to a sufficient degree. It seems in particular necessary for researchers to develop a sufficiently detailed understanding of these aspects, in order for them to support its consistent development and anticipating problems and questions likely to arise at the various steps of the implementation path.

The range and significance level of some specifications of technical guidelines seems often giving rise to misunderstandings, leading to too strict interpretations of specifications of some reference documents. Possibilities are missed to do one step back, namely choosing guidelines of a more general application level as main reference (as for instance consider EOTA Guidance Documents above ETAG's, etc.), and to reinterpret some principles independently of the already derived application rules when it appears as necessary.

It is probably also useful to keep in mind that adhesive polymers and interlayers are a category of products of which production processes are much more flexible than for other 'structural materials' they are used with, such as glass and steel

products. Float glass products and hot-rolled steel profiles are obtained in large volumes by heavy industrial production processes, which are not easily modified, whereas in comparison many polymers used as adhesive products are produced in smaller quantities, and the production processes are much more easily, and more often adapted. This is probably also an important aspect to account for for developing appropriate assessment strategies.

It is also necessary to acknowledge and to not underestimate the difficulties structural engineers have with polymer mechanics and related models, and with classification of polymer product families, especially with polymer adhesives. In fact, these products fall outside our implicit reference conceptual framework, namely based on small strain theories and solid mechanics. In that regard, the concept of 'intermediate scales' as intermediary between stakeholders, in particular between manufacturers and designers, should be considered. This could be already a subject of reflection to standardization committees, to allow establishing test standards or guidelines for experimental investigation and assessment methods which could be implementable before complete design models are available and validated. In this way, production and share of quality experimental data would be encouraged, which seem a preliminary requirement for the development of robust assessment methods and calculation models, taking into account that the development of calculation models for non-standard applications in buildings is a fragmented and progressive process.

It seems difficult to propose here more concrete trails about how to deal with such issues, which remain quite subjective in nature. Nevertheless, it seems very useful to encourage more initiatives for improving inter-disciplinary approaches in standardization and research activities; hopefully can this work contribute to forthcoming reflections and developments in that direction…

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Appendix A – Details of the experimental campaign SG35

This appendix gives a more detailed overview of the characteristics of each test specimen and complementary TCT-test results belonging to the experimental campaign SG35 reported in Chapter V.

Explanations and details given in the main text about the preparation of the specimens and about the test configurations and conditions are not duplicated in this appendix.

Table A1 details individual data for each TCT-test specimen and follows the numbering order of individual specimens.

Table A2 and Table A3 regroup key data and results by test series, respectively for the cdr-tests and for the creep tests.

Table A1 – Overview of characteristics of test specimens

Wide specimens (b = 50 mm)

Specimen	width	thickness	prepared on	tested on	statute	test series		
	b,ave [mm]	t,ave [mm]				nr.	loading mode	test temp. [°C]
SG35-01	51.0	0.87	9/03/2012	9/03/2012	failed	s1(a0)	cdr	20
SG35-02	50.4	0.87	9/03/2012	9/03/2012	failed			
SG35-03	50.7	0.85	9/03/2012	12/03/2012				
SG35-04	50.4	0.87	12/03/2012	12/03/2012				
SG35-05	50.4	0.87	12/03/2012	12/03/2012				
SG35-06	50.4	0.86	12/03/2012	12/03/2012				
SG35-07	50.6	0.86	30/03/2012	30/03/2012		s2(a0)	creep	20
SG35-08	50.5	0.87	30/03/2012	2/04/2012				
SG35-09	50.5	0.87	5/04/2012	5/04/2012				
SG35-10	49.4	0.86	5/04/2012	5/04/2012				
SG35-11	49.1	0.87	24/04/2012	24/04/2012	rejected	s3	cdr	60
SG35-12	48.6	0.85	11/05/2012	11/05/2012		s2(a0)	creep	20
SG35-13	49.2	0.84	21/05/2012	21/05/2012		s3	cdr	60
SG35-14	49.2	0.86	21/05/2012	21/05/2012				
SG35-15	48.6	0.85	21/05/2012	21/05/2012				
SG35-16	49.4	0.86	21/05/2012	21/05/2012		s4	creep	60
SG35-17	49.4	0.86	21/05/2012	21/05/2012	limit			
SG35-18	49.4	0.87	22/05/2012	13/06/2012		s5	cdr	40
SG35-19	49.2	0.85	13/06/2012	13/06/2012				
SG35-20	49.2	0.86	13/06/2012	13/06/2012				
SG35-21	49.5	0.86	20/06/2012	20/06/2012		s6	creep	40
SG35-22	48.6	0.86	21/06/2012	21/06/2012				
SG35-23	49.9	0.87	31/07/2012	31/07/2012	failed	s5	cdr	40
SG35-24	49.4	0.86	31/07/2012	31/07/2012				
SG35-25	49.4	0.85	31/07/2012	31/07/2012				
SG35-26	48.5	0.85	31/07/2012	31/07/2012		s3	cdr	60
SG35-27	49.4	0.87	31/07/2012	31/07/2012				
SG35-28	50.0	0.85	17/08/2012	17/08/2012		s6	creep	40
SG35-29	50.0	0.87	19/08/2012	19/08/2012	failed			
SG35-30	49.2	0.87	20/08/2012	20/08/2012				

Notes :

- the value of the width is an average value of two direct measurements performed with a (sliding) calliper above and below the initial pre-cracked section; the value of the thickness of the interlayer is a derived value, calculated from the average of four direct measurements of the total thickness of the specimen above and below the initial pre-cracked section and along each lateral edge, performed with a micrometric calliper, and by withdrawing the thickness of the individual glass components taken equal to 3.85 mm (see Chapter V paragraph V.2.2).

Table A1 (continued) – Overview of characteristics of test specimens

Specimen	width	thickness	prepared on	tested on	statute	test series		
	b,ave [mm]	t,ave [mm]				nr.	loading mode	test temp. [°C]
SG35-31	49.5	0.85	3/09/2012	3/09/2012		s7	cdr	0
SG35-32	49.3	0.86	3/09/2012	3/09/2012				
SG35-33	49.2	0.86	3/09/2012	3/09/2012				
SG35-34	49.5	0.85	3/09/2012	4/09/2012	rejected	s9	cdr	-20
SG35-35	49.5	0.85	3/09/2012	4/09/2012	rejected			
SG35-36	50.1	0.85	3/09/2012	4/09/2012	rejected			
SG35-37	50.1	0.85	3/09/2012	4/09/2012	failed	s8	creep	0
SG35-38	48.5	0.87	4/09/2012	4/09/2012				
SG35-39	49.2	0.86	4/09/2012	4/09/2012	failed			
SG35-40	49.5	0.86	4/09/2012	5/09/2012				
SG35-41	49.5	0.86	5/09/2012	5/09/2012				
SG35-42	49.4	0.87	5/09/2012	12/09/2012		s2(a1)	creep	20
SG35-43	49.5	0.86	6/09/2012	14/09/2012		s1(a1)	cdr	20
SG35-44	49.4	0.86	*19/09/2012	7/12/2012				
SG35-45	49.4	0.87	*19/09/2012	7/12/2012				
SG35-46	48.5	0.87	*19/09/2012	10/12/2012		s1(a2)	cdr	20
SG35-47	49.2	0.87	7/12/2012	10/12/2012				
SG35-48	48.5	0.87	29/03/2013	12/04/2014	failed	s2(a3)	creep	20
SG35-49	49.4	0.86	30/03/2013	1/04/2013				
SG35-50	49.4	0.86	12/04/2013	12/04/2013	limit			
SG35-51	49.3	0.85	14/04/2013	14/04/2013				

Narrow specimens (b = 30 mm)

Specimen	width	thickness	prepared on	tested on	statute	test series		
	b,ave [mm]	t,ave [mm]				nr.	loading mode	test temp. [°C]
SG35-55	29.3	0.86	7/06/2012	7/06/2012		s1b	cdr	20
SG35-56	29.4	0.86	7/06/2012	7/06/2012				
SG35-57	30.0	0.85	7/06/2012	7/06/2012				
SG35-58	29.4	0.83	29/03/2013	30/03/2013		s2b(a3)	creep	20
SG35-59	29.4	0.84	29/03/2013	1/04/2013				
SG35-60	30.0	0.86	12/04/2013	12/04/2013				

Notes (continued) :

- the preparation date corresponds to the moment when the initial cracks were made (see Chapter V paragraph V.2.2) (a * indicates specimens which initial cracks have been "refreshed" shortly before the test by bending the specimen again as during the preparation step); the test date indicates when the TCT-test has been started.

- statute : "failed test" is used when an obvious failure is detected during the test, making the test results totally or partially irrelevant or non-usable; "rejected test" is used when no obvious failure is detected but the test results were judged non reliable during the analysis step (see also Chapter V paragraph V.3.2); "limit test" indicate when some potentially significant issue has been detected but the result still has used by lack of alternative.

Table A2 – Overview of test results (key numbers) for cdr-test series

			Parameters test				Test results				
Series	T [°C]	Specimen	v [mm/min]	v [mm/sec]	log v [mm/s]	end test	F,max [N]	t(F,max) [s]	log(t) [s]	Failure mode	F,ss [N]
s7	0	TCT-SG35-31	0.1	0.0016667	-2.778	breakage	1612	262	2.4183	CP	
		TCT-SG35-32	0.01	0.0001667	-3.778	breakage	1467	2454	3.3899	RD > CP	1235
		TCT-SG35-33	1	0.0166667	-1.778	breakage	1760	26.5	1.4232	CP	
s1(a0)	20	TCT-SG35-03	1	0.0166667	-1.778	stopped	1241	31.4	1.4969	CP	
		TCT-SG35-04	0.01	0.0001667	-3.778	stopped	922	3458	3.5388	RD (> CP)	835
		TCT-SG35-05	10	0.1666667	-0.778	stopped	1439	5.1	0.7076	CP	
		TCT-SG35-06	100	1.6666667	0.222	breakage	1632	4.2	0.6232	CP	
s1(a1)	20	TCT-SG35-43	0.1	0.0016667	-2.778	stopped	1291	162.6	2.2111	RD	1045
		TCT-SG35-44	0.01	0.0001667	-3.778	stopped	1137	1249	3.0966	RD	865
		TCT-SG35-45	1	0.0166667	-1.778	stopped	1528	15.9	1.2014	RD	1160
s1(a2)	20	TCT-SG35-46	0.01	0.0001667	-3.778	stopped	1035	2780	3.4440	RD	900
		TCT-SG35-47	1	0.0166667	-1.778	breakage	1442	16.4	1.2148	RD > CP	
s5	40	TCT-SG35-18	1	0.0166667	-1.778	stopped	570	16.5	1.2175	RD	522
		TCT-SG35-19	10	0.1666667	-0.778	stopped	963	1.4	0.1461	RD	714
		TCT-SG35-20	0.01	0.0001667	-3.778	stopped	389	83167	4.9200	RD	389
		TCT-SG35-24	0.1	0.0016667	-2.778	stopped	460	1067	3.0282	RD	460
		TCT-SG35-25	3.162	0.0527	-1.278	stopped	786	3.7	0.5682	RD	620
s3	60	TCT-SG35-13	0.01	0.0001667	-3.778	stopped	121	1127	3.0519	RD	116
		TCT-SG35-14	10	0.1666667	-0.778	stopped	345	1.2	0.0792	RD	272
		TCT-SG35-15	1	0.0166667	-1.778	stopped	190	13.5	1.1303	RD	166
		TCT-SG35-26	3.162	0.0527	-1.278	stopped	247	6.1	0.7853	RD	227
		TCT-SG35-27	0.1	0.0016667	-2.778	stopped	140	106	2.0253	RD	120
s1b	20	TCT-SG35-55	1	0.0166667	-1.778	breakage	889	11.2	1.0492	CP	
		TCT-SG35-56	0.01	0.0001667	-3.778	stopped	636	710	2.8513	CP	
		TCT-SG35-57	10	0.1666667	-0.778	breakage	1005	1.3	0.1139	CP	

Legend cdr-tests (Table A2) :

- v : applied displacement rate (measured at the level of the displacement of the transversal beam of the testing device)
- end test : indicates whether the test is stopped before the ligament breakage or not
- F,max : measured value of the peak force
- F,ss : measured value of the steady-state force (in case of RD failure pattern)

Legend creep tests (Table A3) :

- F,cr : applied value of the creep load
- FPdXX : characteristic point of a creep curve corresponding to an opening of the initial crack equal to X,X mm
- BP : breakage point
- t( ) : time-to-failure of a characteristic point
- dr( ) : crack opening rate (determined with a graphical tangent to the creep curve through the characteristic point considered)
- d(BP) : deformation level at breakage (measured crack opening)

TCT-SG35-39* : test failed by shortage of coolant; only early part of the creep curve is ok
TCT-SG35-29* : test stopped earlier because of steering issues (resonance loading device)
TCT-SG35-17* : test is considered “limit” because of initiation loading step and resulting short time values for a creep test

Table A3 – Overview of test results (key numbers) for creep-test series

		Parameters test			Test results													
					FPd01			FPd03		FPd06		FPd1			BP			
Series	T	Specimen	F_cr	end test	t_d01	log(t_d01)	dr(d01)	t_d03	log(t_d03)	t_d06	log(t_d06)	t_d1	log(t_d1)	dr(d1)	t_BP	log(t_BP)	d(BP)	
	[°C]		[N]		[s]	[s]	[mm/s]	[s]	[s]	[s]	[s]	[s]	[s]	[mm/s]	[s]	[s]	[mm]	
s8	0	TCT-SG35-38	1500	breakage	220	2.3424	3.30E-04	475	2.6767	562	2.7497			4.30E-02	575	2.7597	0.8	
		TCT-SG35-39*	1200	stopped	7600	3.8808												
		TCT-SG35-40	1350	breakage	970	2.9868	1.50E-04	7260	3.8609	9600	3.9823	10500	4.0212	5.00E-04	11500	4.0607	1.5	
		TCT-SG35-41	1200	stopped	7700	3.8865	3.30E-06	50000	4.6990	70500	4.8482	86600	4.9375	3.20E-05				
s2(a0)	20	TCT-SG35-07	1000	stopped	1400	3.1461	5.00E-05					4200	3.6232	8.00E-04				
		TCT-SG35-08	1000	stopped	1650	3.2175	1.00E-04	4250	3.6284	5710	3.7566	6880	3.8376	5.00E-04				
		TCT-SG35-09	1177	breakage	470	2.6721	1.67E-04	780	2.8921	852	2.9304				865	2.9370	0.8	
		TCT-SG35-10	1354	breakage	27	1.4314	5.00E-03	52	1.7160	55	1.7404	57	1.7559	1.33E-01	60	1.7782	1.8	
		TCT-SG35-12	823	breakage	6200	3.7924	1.00E-05	12500	4.0969	19200	4.2833	25700	4.4099	7.50E-05	90400	4.9562	6.3	
s2(a3)	20	TCT-SG35-49	1000	breakage	17251	4.2368		23551	4.3720	28351	4.4526	29770	4.4738		32270	4.5088	2.3	
		TCT-SG35-50	823	breakage	13108	4.1175		30508	4.4844	42808	4.6315	49800	4.6972		72350	4.8594	4.1	
		TCT-SG35-51	1177	breakage	469	2.6712		694	2.8414	759	2.8802	794	2.8998		794	2.8998	1	
s6	40	TCT-SG35-21	400	stopped	60	1.7782	1.71E-03	140	2.1461	230	2.3617	350	2.5441	3.00E-03				
		TCT-SG35-22	300	stopped	270	2.4314	1.23E-04	1210	3.0828	2780	3.4440	5300	3.7243	1.33E-04				
		TCT-SG35-28	200	stopped	6200	3.7924	6.25E-06	43000	4.6335	140000	5.1461	190000	5.2788	2.50E-05				
		TCT-SG35-29*	250	stopped	1800	3.2553	2.50E-05											
		TCT-SG35-30	350	stopped	120	2.0792	6.67E-04	380	2.5798	770	2.8865	1420	3.1523	5.33E-04				
s4	60	TCT-SG35-16	100	stopped	950	2.9777	5.00E-05	3600	3.5563	11800	4.0719	30000	4.4771	1.60E-05				
		TCT-SG35-17*	200	stopped	12	1.0792	1.82E-02	22	1.3424	31	1.4914	48	1.6812	1.82E-02				
s2b(a3)	20	TCT-SG35-58	494	breakage	42709	4.6305		6.37E+04	4.8042	7.33E+04	4.8652	82309	4.9154		121459	5.0844	3.6	
		TCT-SG35-59	600	breakage	4364	3.6399		7.00E+03	3.8453	7.95E+03	3.9004	8380	3.9232		8526	3.9307	1.5	
		TCT-SG35-60	706	breakage	365	2.5623		9.22E+02	2.9647	1.27E+03	3.1028	1317	3.1196		1317	3.1196	1	