

Transverse cracking in the bulk and at the free edge of thin-ply composites : experiments and multiscale modelling

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Abstract

Thin-ply composites were shown to exhibit significantly delayed transverse cracking, but the linear onset of damage scaling with ply thickness reported by Amacher et al. [1] did not correspond to the established LEFM based in-situ strength model. This study further investigates this experimental behaviour by simultaneously comparing in-situ free edge crack observation with acoustic emission measurements as well as performing ex-situ X-ray tomography observations of crack propagation. A multi-scale FE model was used to better understand the damage mechanisms at play, and showed a decreasing trend of the apparent toughness with decreasing ply thickness, which explains the deviation from the existing model. Transverse cracking at the free edges was observed to propagate quickly towards the center of the specimens for the thickest plies, while in the thinnest plies it is significantly delayed, up to a point where no cracks can reach the center of the sample before final failure.

Keywords: A. Carbon fibres, B. Mechanical properties, C. Finite element analysis, D. Mechanical testing

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1. Introduction

Ply thickness effects in fibre reinforced polymers were documented very early on with the work of Parvizi et al. [2], but became a topic of renewed interest in the last 10 years due to advances in manufacturing techniques allowing the production of prepreg tapes down to a ply thickness of less than 20 μm . Besides the much increased design space offered by these materials, as well as the significantly improved mechanical properties, the notable increase in transverse tensile strength with decreased ply thickness t was one of the most striking feature of these new materials, and naturally drew a lot of attention to their experimental characterization.

Sihn et al. [3] performed unnotched tensile tests on quasi-isotropic specimens of either 40 μm or 200 μm ply thickness, with the thicker ones obtained by sub-laminate or ply-block stacking of 5 of the thinnest ones. They noticed a strength improvement for the specimens produced with the sub-laminate stacking sequence. They also recorded an improved fatigue life in both unnotched and open hole tensile tests, and reduced delamination as well as increased compression after impact resistance for the specimens produced with the thinner plies. Wisnom et al. [4] performed tensile tests of unidirectional and quasi-isotropic specimens of varying sample and ply thickness. If a small increase in strength was noted when increasing the thickness of quasi-isotropic specimens by sub-laminate stacking, a much larger effect was observed when varying the ply thickness by ply-block stacking, accompanied by a change in failure mechanism from fibre failure after partial delamination with the thinner ply-blocks (250 μm or less) to delamination only with the thicker ones (500 μm or more). In an other study, the transverse crack propagation in 90° layers of different thickness embedded between 0° plies was studied by Saito et al. [5], who reported the existence of a transverse crack suppression effect for the thinner plies, and related it numerically to a decrease in energy release rate at the crack tip with decreasing ply thickness. The fibre-matrix debonding would appear at similar strains, but their coalescence into formed transverse cracks and their propagation through

31 the thickness of the ply were delayed with thinner plies. They also related the
32 higher crack density observed in the thinner plies at high strains to the reduced
33 stress relaxation obtained with smaller transverse cracks. Similar observations
34 regarding the crack density as well as a dependency to the adjacent ply stiffness
35 were reported by Sebaey et al. [6], though on relatively thick plies ($\geq 183 \mu\text{m}$), by
36 using an in-situ optical observation of the tested specimens' polished free edges.
37 Yokozeki et al. [7] also reported a retardation of matrix cracking accumulation
38 in specimens made of thinner plies coupled with a decreased crack density in the
39 bulk of their samples when observing them by means of x-ray radiographs. Ama-
40 cher et al. [1] reported both an increase in ultimate strength when decreasing
41 the ply thickness of quasi-isotropic tensile specimens of constant dimensions, as
42 well as an increased onset of damage determined by acoustic emission.

43
44 The numerical modelling of such phenomenon has already been worked on,
45 with Canal et al. [8] subjecting an RVE of 30 fibres in random homogeneous
46 dispersion with periodic boundary conditions to transverse tension and out-of-
47 plane shear. This work was carried out by studying an embedded cell using the
48 actual microstructure of a notched beam sample, tested in three-point bending,
49 to study the intraply fracture of fibre-reinforced composite, obtaining an accu-
50 rate prediction of the material's macroscopic behaviour [9]. Arteiro et al. [10]
51 used an advanced elasto-plastic model including damage for the matrix in which
52 90° orthotropic fibres were distributed in a statistically equivalent distribution
53 to a real one. The RVE thus defined was embedded between two $\pm\theta^\circ$ layers and
54 the thickness of the micro-modelled zone varied to study the ply thickness effect
55 on the in-situ strength. This model was shown to capture the increase of stress
56 required to grow a crack with decreasing ply thickness. A critical review of this
57 approach compared to experiments and analytical modelling was also published
58 [11].

59
60 Before the beginning of the numerical modelling effort in this field, an analy-
61 tical model was proposed by Camanho et al. [12] based on the previous work of

62 Dvorak and Laws [13], where the concept of the in-situ strength of a (constrained)
63 ply as a function of ply thickness was developed. This model was validated
64 against reported observations of the transverse cracking at the free edge of some
65 samples. The onset of damage scaling reported more recently [1] where an acoustic
66 emission cumulative energy threshold was used to define the onset of damage
67 did however deviate from this model, especially for very thin plies, showing a
68 linear scaling consistent with some numerical results of Arteiro et al. [10]. This
69 raised the questions of whether or not the cracks visible at the edge of the specimens
70 could be used as reliable damage indicator for very thin plies, and of what
71 kind of damage mechanism was reflected by the acoustic emission measurements.

72
73 The goal of this work is therefore to compare simultaneous observation of
74 the damage at the free edge, in the bulk and by recording the acoustic emission
75 activity. To this effect, in-situ observations of the free edge similar to the
76 ones performed by Sebaey et al. [6] are conducted simultaneously with acoustic
77 emission measurements similar to the ones performed by Amacher et al. [1]
78 in order to directly compare the two phenomena. Subsequent cross-sectional
79 micrographies and x-ray tomographic images of selected specimens that were
80 only partially loaded, to study the crack propagation through the width of the
81 sample at different load levels, are also conducted. An embedded cell multi-scale
82 finite element (FE) model is also developed to study the transverse cracking of
83 the 90° plies. Finally, the experimental data are compared to the results of the
84 FE model which aims to provide an understanding of the transition between
85 damage mechanisms involved in the size effect of transverse cracking. Boundary
86 conditions equivalent to both free edge and bulk locations are developed for
87 comparison with the experimental data.

88 2. Materials and methods

89 2.1. Experimental Study

90 The samples used in this study are made of unidirectional prepregs of Toray
91 M40JB carbon fibres and TP80ep epoxy resin supplied by NTPT in 4 different
92 ply thicknesses, namely $t = 30 \mu\text{m}$, $75 \mu\text{m}$, $150 \mu\text{m}$ and $300 \mu\text{m}$. The samples
93 were therefore similar to the ones used by Amacher et al. [1]. All of the prepregs
94 supplied were made with same fibre and resin batches and were manufactured
95 on the same production line in order to minimise any potential relevant scatter.
96 The stacking sequence used to produce the symmetric, quasi-isotropic laminates
97 is $[45/90/-45/0]_{nS}$ where n is varied to ensure a constant laminate thickness
98 of $2.41 \pm 0.1 \text{ mm}$ for all the samples, no matter which ply thickness was used.
99 The composite plates were cured in an autoclave following the supplier's recom-
100 mended curing cycle (peak of 80°C for 8 hours at 3 bar after an initial 2 hour
101 long plateau at 60°C and $1^\circ\text{C}/\text{mn}$ ramp rates) using a machined aluminium
102 mould to ensure a uniform and constant volume fraction and a good flatness.
103 The produced plates were then cut with a diamond disc saw into $240 \times 24 \text{ mm}$
104 specimens following the ASTM D3039 standard [14]. One of the free edges of
105 each sample was finally manually polished along its whole length, down to a pa-
106 per grit size of $3 \mu\text{m}$. Tapered 2 mm thick $\pm 45^\circ$ GFRP tabs were glued at their
107 extremities and an HBM 6/120 LY41 strain gauge was installed in the middle
108 of the gauge length on one of the faces. The proper alignment of the grips was
109 checked on the first sample of each test series accordingly to the standard with
110 a second strain gauge on the opposite side of the sample. The volume fraction of
111 all plates produced was measured between $51,7\%$ and $57,9\%$ with a mean value
112 of $55,5\%$ and a standard deviation of $2,68\%$. The stress measurements reported
113 here were consequently all normalised to 55% volume fraction to account for this
114 small discrepancy according to the formula $\sigma = \frac{55\%}{vf_n} \sigma_n$, where σ is the stress
115 normalised for 55% volume fraction, vf_n the effective volume fraction of each
116 plate and σ_n the stress actually measured during the experiment.

117

118 At least six samples of each ply thickness were tested in tension following the
119 ASTM D3039 standard [14] whilst recording the damage activity by simulta-
120 neously performing acoustic emission measurements, and monitoring the visible
121 crack growth on the polished lateral free edge using a video microscope. The
122 acoustic emission onset of damage was defined as the stress at which the cumu-
123 lative acoustic energy reaches a 10^{-15} J threshold, as acquired by a Mistras-2001
124 measurement equipment, using a 65dB minimum acquisition threshold and an
125 appropriate ΔT -front-end filter to reject acoustic activity taking place out of
126 the gauge length [1]. This procedure ensures that the reported values were com-
127 parable with prior measurements performed on the same material [1, 15, 16].
128 The pictures of the polished free edges were recorded using a Guppy Pro F-146
129 CCD camera mounted on an Olympus SZX12 stereo microscope at 1Hz. This
130 slow acquisition speed coupled with a slower loading speed of 0.125 mm/ min
131 were required to be able to adjust the focus during the test without significant
132 loss of data. The size of the observation window, always located close to the cen-
133 ter of the gauge region, ranged from 1.1x0.8mm for the 30 μ m plies to 3.1x2.3mm
134 for the 150 μ m and 300 μ m ones, corresponding to an optical magnification rang-
135 ing from 2.5x to 6.5x depending on the ply thickness. The magnification was
136 kept as low as possible for the thicker plies in order to capture as much 90° ply
137 length as possible whilst ensuring that the cracks remained visible. At the other
138 end of the spectrum, the magnification could not be increased further for the
139 thinnest plies as the resulting depth of field would not allow a proper focus.
140 After testing, the free edges of selected specimens were observed under a higher
141 magnification optical microscope or in SEM to ensure that the limited resolution
142 of the optical equipment used in-situ did not prevent the observation of some
143 smaller damage elements during the tests.

144

145 Selected samples were used to study the propagation of the free edge cracks
146 into the width of the sample by means of interrupted tests. The stress levels at
147 which the loading was stopped were chosen following the results presented in
148 section 3.1 to either be representative of a point where free edge cracks were

149 present but the onset of damage determined by acoustic emission had not been
150 reached, or of a point where this threshold was reached but the sample had not
151 yet failed. Consequently, one sample made out of 150 μm plies was loaded until
152 0.64% strain, another one until 0.89% strain, and a sample made out of 75 μm
153 plies loaded to 0.89% strain at the same loading rate as used during the pre-
154 vious complete tests. It was not possible to obtain a sample made of 75 μm plies
155 where the loading exceeded the acoustic emission threshold as it ruptured before
156 the loading was halted. After the desired stress levels were reached, the loading
157 was swiftly interrupted and the sample removed from the testing apparatus. A
158 section of the gauge length close to its center was then cut through the middle
159 of its width as shown on Fig. 1 using a diamond wire saw with a nominal wire
160 diameter of 130 μm . The cut line revealing the middle of the sample was then
161 polished, and a new set of tabs glued to it. This "partial sample" was finally re-
162 loaded in a small screw driven jig to reopen possible cracks, at a loading inferior
163 to 0.1% strain. This loaded section was then observed under a Keyence VHX
164 500 microscope at an optical magnification of 15x, using a mix of concentric and
165 ring lighting, over a length of approximately 10mm. The same observation pro-
166 cedure was applied to the original polished free edge of the sample. A reference
167 line drawn across the width of the gauge length before the cut ensured that the
168 observation at the free edge and in the newly revealed bulk could be spatially
169 superimposed for comparison.

170

171 A similar interrupted testing procedure was applied on a second set of
172 samples in order to observe the crack propagation through the bulk of the spe-
173 cimens by means of X-ray tomography. An RX-Solutions Ultratom micro CT
174 scanning device was used to reveal the crack presence, where a resolution of
175 approximately 7 μm per voxel allowed the observation of 10mm gauge length
176 over half of the width of the sample for each scan. The micro damage detec-
177 tion at this resolution was made possible by the use of a ZnI_2 -based contrasting
178 penetrant solution which was shown to produce good contrast [17] and has the
179 additional benefit to free the imaging process from time constraints as the iodide

180 compounds formed in the solution stay in the sample even after evaporation of
181 the solution. To ensure a good penetration of this contrasting agent, samples
182 were immersed for 24 hours whilst being slightly reloaded (typically 0.065%
183 strain) to reopen the cracks previously created during the test.

184 Two samples made of 150 μm plies were again loaded until 0.64% and 0.89%
185 applied strain, and three samples made of 100 μm plies were loaded to 0.64% ,
186 0.83% and 0.89% strain. The choice of the 100 μm ply thickness over the pre-
187 viously used 75 μm ones was solely due to material availability. The goal was
188 to obtain an insight into the bulk damage present at a stress level inferior to
189 the acoustic emission onset threshold, but superior to the onset of free edge
190 cracking. This could then be compared to the damage state in another similar
191 sample loaded at a stress level exceeding the acoustic emission threshold.

192 A final 100 μm sample was then tested to 0.83% applied strain, which at this ply
193 thickness is very close to AE onset but does not exceed it, before the machining
194 of a thin (2 mm) slot in the middle of the sample prior to infiltration. This allo-
195 wed the penetrant solution to enter any potential crack from the center of the
196 specimen as well as from its free edge, revealing any damage originating at the
197 center of the sample. A summary of the selected samples used for interrupted
198 testing can be found in Table 1.

199

200 *2.2. Numerical Study*

201 A FE model was built using a multi-scale “embedded cell” modelling ap-
202 proach in which a finely discretised region corresponding to the 90° ply was
203 embedded in a larger continuum model of the laminate, where the total model
204 length was at least three times the embedded cell length. To account for the
205 differences that were observed in microscopy, microstructures taken from actual
206 samples were used to build the micromechanical zones of the embedded cell.
207 Either manual fibre identification or an in-house Matlab script working through
208 grey level thresholding, erosion and watershed algorithm were used to recons-
209 truct the fibre centre locations. The fibres’ cross sections were then modelled

210 as circles of varying diameter, with the goal to be as close as possible to the
211 nominal fibre diameter whilst allowing a fibre separation gap large enough to
212 fit at least one properly conditioned matrix FE element (minimum 5% of the
213 nominal diameter). This embedded cell approach was shown to work well in the
214 past, as demonstrated in the work of Canal et al. [9]. A transition zone with
215 increasing matrix and interface fracture properties was built-in at the edges of
216 the embedded cell to avoid localization at the interface with the continuum mo-
217 del, and the simulation results were only evaluated outside of these zones. The
218 function used to this purpose is shown on Fig. 2. The embedded cell boundaries
219 are not simple straight edges but rather follow the outer fibres as this is a bet-
220 ter way to represent how two adjacent plies actually "interlock" with each other
221 in the observed laminates, where no clear resin rich interply interface could be
222 observed in the samples used.

223

224 Three different microstructures, one for each of $t = 30 \mu\text{m}$, $68 \mu\text{m}$ and $150 \mu\text{m}$,
225 were embedded in a $[0/45/90/-45/0]$ stack to replicate the stress state in a
226 quasi-isotropic sample. It was validated that this subjected the embedded cell
227 to the same stress state as an internal 90° ply would be experiencing in the core
228 of the laminate. The obtained microstructures were used in a 3D model repre-
229 senting a thin slice ($4 \mu\text{m}$) of material, with the embedded cell 10 to 20 times
230 longer than the ply thickness, and the continuum zone length on each side of
231 it at least as long. Because of the inherent numerical instabilities, the problem
232 was solved using a dynamic explicit solver (Abaqus[©] Explicit) with mass sca-
233 ling recalculated at each increment to represent the quasi-static response of the
234 material. A study was performed on the variable mass scaling's desired stable
235 time increment to ensure that it was converged at the chosen value of 5×10^{-6} .

236

237 In order to simulate damage events in the bulk of the material, boundary
238 conditions must be enforced to reach a plane-strain state of stress, namely a
239 symmetry on the front side of the model and a planarity constraint on all the
240 nodes of the back face to allow plane displacements due to Poisson effects. These

241 boundary conditions are illustrated on Fig. 3 for the 68 μm model case.
242 The free edge behaviour was simulated by a submodelling approach, where the
243 submodel was defined as the micromodelled zone and a part of its homogenised
244 neighbouring plies only, with the same mesh as the bulk model. The displa-
245 cement field imposed on the boundaries of this submodel, as shown on Fig. 4,
246 was driven by the previously calculated displacement field of a larger continuum
247 model discretised with a coarser and highly biased mesh, subjected to the same
248 boundary conditions as the bulk model but without planarity constraint. This
249 allowed the imposition a realistic 3D displacement boundary condition on the
250 submodel, thus allowing a good representation of the stress-state at the free edge
251 of the samples without having to run a full 3D model of the micromodelled zone.
252 The continuum model used to drive the submodel represents the half width of a
253 physical sample (12mm) and a length 10 times longer than the micromodelled
254 zone, which is sufficient to ensure that no undesirable edge effects are imposed
255 upon the submodel.

256 The mesh uses the C3D8R and C3D6 element formulations offered by Abaqus[®],
257 with a typical element size of 20 μm in the homogenised region and 0.5 μm in the
258 embedded cell region. The curvature control parameters used for the meshing
259 algorithm resulted in the definition of about 30 element over the perimeter of
260 the fibres to ensure that the fibre-matrix interface can be discretised finely en-
261 ough. A single element was used in the depth (4 μm) of the model resulting in a
262 mesh varying between 390k nodes for the 30 μm model and 1Mio nodes for the
263 150 μm model. The mesh of the 150 μm model is shown on Fig. 5.

264 The material properties described below, summarised in Table 2, were partially
265 taken from [1], and amplified by additional experimental tests performed for
266 this work. These properties were kept constant throughout this study to ensure
267 that the effects observed were only caused by the changes of microstructure and
268 ply thickness. The thermal residual stresses were neglected in this analysis due
269 to the low curing temperature of the resin used (80 $^{\circ}\text{C}$), which only leads to a
270 maximum of 9.3 MPa as calculated by classical laminate theory. Furthermore,
271 as the neat resin was observed to exhibit significant creep under load at room

272 temperature, it is expected that the highly stressed zones will experience a signi-
273 ficant relaxation during the cooling phase, which further reduces their influence
274 on damage response.

275 *Simulation parameters : Fibres*

276 A transversely isotropic elastic model was used to model the fibres. It re-
277 lies on the properties published by the fibre manufacturer for the longitudi-
278 nal direction. The transverse properties were obtained by inverting the Chamis
279 semi-empirical micromechanical model [18] based on the experimental trans-
280 verse characterization of the material used. The transverse-transverse Poisson
281 ratio, calculated as $\nu_{23} = \frac{E_2}{2G_{23}} - 1$, exceeded physically achievable values and
282 was therefore capped at 0.45. The calculated fibre transverse modulus E_2^f was
283 within the standard deviation of the values obtained experimentally by nano-
284 indentation on the same kind of fibres by Maurin et al. [19] which in turn were
285 in good agreement with the generally accepted values found in Hughes [20].

286 *Simulation parameters : Matrix*

287 The matrix was modelled using a hyperbolic Drucker-Prager model with re-
288 gularised damage ([9, 21]) and non-associated flow based on properties measured
289 on bulk matrix specimens. The general exponent formulation of this model offer-
290 red by Abaqus[©] was used, and its parameters identified by least square fitting
291 of three points, as shown on Fig. 6. The two first ones are the uniaxial tensile
292 (σ_t^y) and uniaxial compressive (σ_c^y) yield strength which were measured experi-
293 mentally following the ISO 527-2/93 [22] standard for the tensile tests and based
294 on the ASTM D695 standard [23] for the compressive test. The last point is the
295 initial hydrostatic tensile strength p_t which was defined by the introduction of
296 σ_t^y and σ_c^y values in the yield model proposed by Melro et al. [24] in a triaxial
297 tensile stress-state. The flow-angle β can then be retrieved by a tangent calcu-
298 lation of the resulting potential function at high confining pressure.
299 The tensile hardening as well as critical damage initiation strain (ultimate plas-
300 tic strain) could not simply be defined as the ones obtained experimentally due

301 to the notable size-effects that such tests present, as shown by Fiedler et al. [25]
302 and Hobbiebrunken et al. [26]. Considering that the matrix properties at the
303 micro-level are used in this simulation, an extrapolation using a second order
304 polynomial fit was performed on the averaged experimental tensile stress-strain
305 curve until a perfectly plastic behaviour is reached (horizontal tangent) to ex-
306 tend the ductility of the material. The resulting curve, shown on Fig. 7 is in
307 good agreement with the experimental results obtained on very small samples
308 [25]. The strain at onset of damage in tension ϵ_t^c was then defined considering
309 an elastic offloading from the last point in the extended stress-strain curve.

310 It can be observed that if the extended behaviour is considered, the hydrostatic
311 strength calculated as proposed is in good agreement with both the void ini-
312 tiation failure criterion $\sigma_H \geq \sigma_{ut}/3$ relating the hydrostatic strength σ_H to the
313 uniaxial strength σ_{ut} , and previous experimental measurements, both discussed
314 by Gross et al. [27].

315 Once the critical strain is reached, a linear softening, energy based regulari-
316 sed damage model reduces the stiffness of the matrix elements. The damage
317 variable SDEG is defined as the scalar stiffness degradation variable (Ductile
318 damage model in Abaqus [28]).

319

320 Finally, nano-indentation tests were performed on cast matrix specimens and
321 the resulting force-displacement curves used for the inverse-identification of the
322 dilatation angle Ψ . A 2D axisymmetric FE model is used to this effect where the
323 indenter tip is modelled with a 70.3° equivalent half-angle to simulate the Berko-
324 vitch tip used experimentally. These simulations were performed using Abaqus
325 Explicit, with mesh and mass-scaling convergence studies performed to ensure
326 that precise results were obtained. The obtained numerical force-displacement
327 curve is in good approximation to the experimental one, as shown on Fig. 8.

328

329 A sensitivity analysis using the bulk embedded cell model was performed
330 on the matrix toughness, using values $G_C^m = 64, 128$ and 256 J/m². It showed
331 an independence of the first crack appearance to this parameter within the

332 tested range. This can be explained by considering the weak interface behaviour
333 exhibited here, where debonding and crack coalescence is the preferential energy
334 dissipation mechanism compared to matrix crack propagation. Consequently,
335 G_C^m was fixed at the lowest tested value of 64 J/m².

336 *Simulation parameters : Interface*

337 The damage process at the fibre-matrix interface was modelled using the
338 linear softening, energy based, cohesive model present in Abaqus[®], with the
339 damage variable CSDMG representing the loss of stiffness of the interface due
340 to damage. In order to set the interface stiffness, a small RVE of 4 µm thick-
341 ness containing a single fibre embedded in a matrix cube and meshed similarly
342 to the embedded cell of the main model was defined. The goal was to set the
343 stiffness as low as possible to improve computation time whilst ensuring both
344 a converged force-displacement curve and a continuous stress field in the RVE
345 when submitting it to a transverse tensile stress state.

346 The normal interface strength was shown to be a parameter to which the em-
347 bedded cell model is only weakly sensitive, as long as it remains weaker than
348 the matrix. Typically, no difference in applied stress required to reach the first
349 transverse crack could be observed between $\tau_1^0 = 20$ MPa and $\tau_1^0 = 40$ MPa. It
350 was thus set by an inverse identification process [29], by comparing the trans-
351 verse tensile strength measured experimentally on unidirectional samples of this
352 material to the simulation of a small RVE of similar fibre volume fraction to
353 the experimental samples, containing 180 randomly placed fibres, and using
354 the same material properties as the main model. Both the transverse tensile
355 and embedded cell laminate simulation were insensitive to the interfacial shear
356 strengths when they were set between the normal interfacial strength and twice
357 that value. They were therefore set to 1.5 times the normal value following [30].

358

359 The interface toughness was clearly the dominating parameter of the quasi-
360 isotropic embedded cell model, and was consequently set such that the first
361 crack predicted by the bulk model for the 150 µm thickness model would be in

362 good agreement with the experimentally observed onset of damage measured by
363 acoustic emission for this ply thickness. The obtained value of 3 J/m^2 , as well
364 as the other interface properties discussed above, are consistent with the values
365 used by Arteiro et al. [10].

366

367 **3. Results and Discussion**

368 *3.1. Experimental Work*

369 A change in the damage mechanisms with decreasing ply thickness is ob-
370 served at the free edge of the samples. Samples with the thickest ($t= 300\ \mu\text{m}$)
371 plies exhibit the first transverse cracks between 0.3 and 0.32 % strain, as shown
372 on Fig. 9a at 0.31% strain, which in a few instances appear simultaneously to
373 some free edge delamination and delamination along the 90° plies. The tips of
374 the transverse cracks then trigger a transverse cracking induced delamination
375 process from 0.5% strain until the failure of the specimens around 0.8 % strain,
376 as shown on Fig. 9b at 0.55% strain. The specimens made with $150\ \mu\text{m}$ plies
377 exhibit a higher onset of transverse cracking at the free edge, found between
378 0.41% and 0.51 % as shown on Fig. 9 c for 0.5% strain. Some less extensive
379 matrix cracking induced delamination appears past 0.8% strain as shown on
380 Fig. 9d at 0.85% strain. The onset of transverse cracking at the free edge is once
381 again delayed in the samples made with $75\ \mu\text{m}$ plies, with the first ones obser-
382 ved between 0.52% and 0.66%, shown for instance at 0.9% strain on Fig. 9e.
383 This transverse cracking does however not lead to any delamination with this
384 ply thickness. Finally, for the samples manufactured using the thinnest ($30\ \mu\text{m}$)
385 plies, no damage can be observed at the free edge before the final failure as
386 shown on Fig. 9f. To ensure that the optical resolution of the equipment used
387 does not preclude the observation of certain damage, the free edge of selected
388 samples was observed in SEM after failure. Whilst transverse cracks did remain
389 clearly visible in thick and intermediate ply thicknesses, no sign of transverse

390 cracking could be found for the thinnest plies as shown on Fig. 10.

391

392 The stress at which the first damage was observed at the free edge, as well
393 as the onset of damage defined by acoustic emission and the ultimate strength
394 of the different ply thicknesses tested are shown on Fig. 11. As no free edge da-
395 mage could be observed for the samples made with 30 μm plies, the UTS value
396 is reported there instead of the free edge crack onset for this ply thickness.

397 One striking feature of the comparison between the free edge observations and
398 the acoustic emission measurements, as shown on Fig. 11, is the drastically dif-
399 ferent scaling that can be observed between the free edge crack onset and the
400 onset of damage obtained by acoustic emission. The observations performed at
401 the free edge are in good agreement with a $1/\sqrt{t}$ scaling, as shown in green in
402 Fig. 11. To the contrary, the onset of damage determined by acoustic emission
403 increases much more linearly with decreasing ply thickness, as shown in red in
404 the same figure. Such a discrepancy between these two indicators can only be
405 explained by the fact that their respective underlying mechanisms are different.

406

407

408 These two different mechanisms can be observed in the partially loaded and
409 cut samples. Indeed, for the one manufactured with 150 μm plies and loaded
410 until 0.64% strain, no damage could be observed in the bulk of the sample,
411 whereas the free edge showed obvious signs of transverse cracking, as shown on
412 Fig. 12b. For the similar sample loaded until 0.89% strain, the free edge reveals
413 much more free edge damage as can be seen on Fig. 12 a, with delamination
414 growing from the transverse cracks and secondary damage propagating into the
415 $\pm 45^\circ$ layers or growing within the 90° plies. At this applied strain, transverse
416 cracks could be observed in the middle of the samples. Finally, the sample ma-
417 nufactured with 75 μm plies and loaded until 0.89% strain, shown on Fig. 12c,
418 reveals a reduced free edge damage compared to the sample manufactured with
419 150 μm plies that was tested up to the same applied strain. Transverse cracking
420 is the only observed damage mechanism, which is consistent with the observa-

421 tions made during the in-situ tests. Furthermore, no damage can be observed
422 in the middle of this 75 μm sample. It can thus be established that the presence
423 of transverse cracking in the bulk of the samples is linked to the crossing of the
424 acoustic emission threshold as defined in this work (section 2.1).

425

426 Further evidence that transverse cracks originate from the free edge of the
427 samples and then propagate into the bulk at the onset of damage defined by
428 acoustic emission is brought by the tomographic observation discussed in sec-
429 tion 2.1. Fig. 13 represents a maximum intensity projection performed with Fiji
430 [31] of all the slices laying in a typical 90° ply, and clearly shows that in both
431 the $t=150\ \mu\text{m}$ and $t=100\ \mu\text{m}$ samples loaded to a stress inferior but close to the
432 acoustic emission onset, no free edge crack propagates markedly into the bulk
433 of the sample (Fig. 13 a,b). To the contrary, in the samples of both $100\ \mu\text{m}$ and
434 $150\ \mu\text{m}$ ply thicknesses loaded to a stress level superior to the acoustic emis-
435 sion threshold, transverse cracks clearly propagate in the bulk of the 90° ply
436 (Fig. 13 c,d). The presence of delamination is evident for the $150\ \mu\text{m}$ laminate
437 loaded until 0.89% strain, but it is interesting to note that it does not extend far
438 into the width of the sample despite a stress level very close to failure. Owing
439 to the observation of the failure mode of these samples, it is most likely that
440 the propagation of this delamination throughout the width of the sample is the
441 last damage occurring and immediately leads to the failure of the specimens.
442 No such delamination can be seen in the $100\ \mu\text{m}$ laminate despite having been
443 subjected to the same applied strain of 0.89%. Furthermore, the developed crack
444 density at a given load is clearly smaller in the samples made of thinner plies
445 (Fig. 13 c,d) which is consistent with [7] but opposite to the trend generally
446 seen at the free edge as for instance in [6].

447

448 The $t=100\ \mu\text{m}$ sample used to assess the absence of damage originating from
449 the bulk is shown on Fig. 14. If the machined cut is clearly visible, no trans-
450 verse cracks are revealed in the bulk, proving that this kind of damage always
451 propagates from the free edges in such samples.

452

453 The angle exhibited by the transverse cracks at the free edge, close to 45° ,
454 are due to a large in plane shear stress component induced by the quasi-isotropic
455 laminate chosen, as shown in the stress analysis of section 3.2. This leads to a
456 principal stress at the free edge of the samples which is normal to the obser-
457 ved cracks. This also explains the observed symmetry in crack angle around
458 the specimens' mid-plane due to the symmetry plane of the chosen stacking
459 sequence. Using the tomographic observations, it is possible to assess that this
460 angle steadily decreases until transverse cracks perpendicular to the ply are ob-
461 served, around 2 mm into the width of the samples.

462

463 In summary, considering the demonstrated different underlying mechanisms
464 between free-edge and bulk behaviour, the increasing strength with decreasing
465 ply thickness observed can be explained by the fact that the behaviour of the
466 samples manufactured using the thickest ($300\ \mu\text{m}$) plies is dominated by damage
467 at their free edges. Any damage appearing there immediately propagates into
468 the bulk of the sample. With decreasing ply thickness, the onset of free edge
469 damage is not only delayed, but the ease with which it can propagate towards
470 the center of the sample is also reduced. The onset of damage as recorded by
471 acoustic emission therefore diverges from what is observed at the free edge, until
472 a different failure mechanism such as the translaminar fracture of the 0° plies
473 becomes predominant, as is the case in the sample manufactured with the thin-
474 nest plies ($t=30\ \mu\text{m}$) where the acoustic emission threshold is reached before
475 free edge damage can be observed.

476

477 Finally, a comparison can be made between the acoustic emission measure-
478 ments reported in [1] and the ones performed at a much reduced loading speed
479 during this experimental study, both shown on Fig. 11. For the two largest ply
480 thicknesses, the onset of damage is delayed at the lower loading speed, as the
481 highly loaded free edge region of the 90° ply region benefits from stress relaxa-
482 tion due to viscoplastic flow. To the contrary, the two smallest ply thicknesses

483 show little dependency on the loading rate, which is further proof that thin-ply
484 composites are less sensitive to damage initiation at the free edge.

485

486 *3.2. Numerical Work*

487 The sequence of damage events in the bulk model is remarkably similar for all
488 three simulation. The first damage to appear is fibre-matrix debonding, which
489 initiates for all ply thicknesses around 0.23% strain. This then leads to local
490 stress concentrators in the matrix, plastic damage accumulation and eventually
491 transverse matrix cracking through coalescence of micro-damage. It is interes-
492 ting to note that this coalescence always takes place in regions where the fibres
493 are closely packed due to increased stress triaxiality in those regions. The main
494 difference noted in the sequence of events between the three simulations is an
495 increased capability to accumulate distributed damage before localisation with
496 decreasing ply thickness. This is illustrated in Fig. 15 where the average cohesive
497 damage indicator (CSDMG) show a very similar onset and increase for all three
498 ply thicknesses modelled. It is only once coalescence happens and thus damage
499 localises that the remaining matrix ligaments experience an increase of plasticity
500 (PEEQ). Typical damage development is represented in Fig. 16. Another point
501 that was noticed during the model development is the much higher sensitivity
502 of the larger ply thickness models to variations in their microstructures compa-
503 red to thinner plies, where early localisation could very easily be triggered in
504 highly packed region. This was not observed for thinner plies, and reinforces the
505 importance to use realistic microstructures in the numerical models. The larger
506 microstructure heterogeneity found in thicker plies is believed to be the cause
507 of this phenomenon [32].

508

509 The onset of transverse cracking in the numerical models is defined as a
510 local unloading (drop in σ_{xx}) over a zone spanning at least a quarter of the ply
511 thickness in the micro-modelled region, as shown on Fig. 17. This also corres-
512 ponds to the first drop in averaged stress in the micro-modelled zone due to

513 the local stress relaxation induced by the crack-opening. It is interesting to note
514 the typical slanted free edge cracks present in the free edge model compared to
515 the purely transverse cracks of the bulk model, which is in agreement with the
516 experimental observations. The macroscopic stress at which this first crack is
517 detected in the bulk FE simulations is then compared to the one at which the
518 acoustic emission threshold is reached at high loading rate, considering that no
519 viscous model is implemented in the simulations. Similarly, the stress at which
520 cracks are detected in the free edge model are related to the onset of free edge
521 cracking. The results reported on Fig. 11 show both a very good agreement
522 between the bulk model values and the experimental onset of damage defined
523 by acoustic emission, as well as a clear difference in behaviour between the bulk
524 and the free-edge models, with the latter tending towards what is observed ex-
525 perimentally at the free edge.

526

527 Regarding the stress-state at the free edge, an analysis of the linear elastic
528 homogenised 3D model used to drive the submodel is performed in order to un-
529 derstand the reason for the angled transverse cracks observed at the free edges.
530 No significant differences are noticed between the different ply thicknesses when
531 computing the mean stress components in the zone corresponding to the em-
532 bedded cell.

533 For the $t=68\ \mu\text{m}$ model, at an applied strain of 0.95%, the mean transverse ten-
534 sile stress $\bar{\sigma}_{22}$ in the 90° layer at the free edge is nearly doubled compared to
535 the mean stress obtained from CLT prediction, with a mean value of 115 MPa.
536 Simultaneously, the 90° ply at the free edge is under a tensile out of plane stress
537 $\bar{\sigma}_{33}$ close to the same mean value. This "through the thickness" stress tends to
538 split the $\pm 45^\circ$ layers away from the 90° one and thus promotes delamination.
539 Furthermore, the mean shear stresses $\bar{\tau}_{12}$ and $\bar{\tau}_{13}$ are negligible within the 90°
540 ply, increasing only very close to the ply boundaries. However, the component
541 $\bar{\tau}_{23}$ cannot be neglected, as it reaches a mean value of 75 MPa, as shown on
542 Fig. 18. At the free edges, the 90° ply within the selected quasi-isotropic lami-
543 nates is thus under combined bi-axial tension and transverse shear. It is this

544 combined stress-state that is responsible for the angle of the transverse cracks
545 observed at the free edge. With a principal stress analysis, the angle of the crack
546 can be easily deduced, assuming that the crack is normal to the principal stress
547 direction. With the above mentioned values, it is possible to obtain the crack
548 angle $\phi = 1/2 \arctan [\bar{\tau}_{23}/(\bar{\sigma}_{22} - \bar{\sigma}_{33}/2)] = 42.5^\circ$ which is very close to what
549 is observed experimentally.

550

551 Regarding the onset of damage scaling with respect to ply thickness, the
552 finite size of the process zone observed during crack development, as shown by
553 the unbroken matrix ligaments on Fig. 16 (damage evolution in 68 μm ply) is of
554 utmost importance. In such a case, the underlying assumptions of LEFM of a
555 fully developed process zone of negligible size is not satisfied. Due to the strong
556 confinement exerted by the neighbouring plies, the crack opening displacement
557 remains very limited in thin plies, which prevents a complete degradation of
558 the ductile matrix ligaments. Thus, only a fraction of the total fracture energy
559 can effectively be dissipated in thin plies. An R-curve effect of the critical ERR
560 associated to transverse cracking is therefore to be expected, as postulated in
561 [15]. To evaluate this hypothesis, the energy dissipation increment during crack
562 nucleation was computed from the embedded cell simulation and an effective
563 critical ERR was computed as the energy dissipated during crack growth divided
564 by the crack area. As shown in Fig 19, the simulation results show a clear
565 reduction of the critical ERR with decreasing ply thickness. The linear scaling in
566 onset of damage is thus attributed to a combination of this reduction of apparent
567 toughness due to an incomplete fracture process zone with the well known ply
568 confinement effect described in the in-situ strength model proposed by Camanho
569 et al. [12]. A modification of this latter model to include the discussed reduction
570 of ERR with decreasing ply thickness was recently proposed by Catalanotti [33].

571

572 4. Summary and Conclusion

573 The appearance of damage in tensile CFRP specimens was monitored in-situ
574 simultaneously at the free edge using a video microscope and in the bulk using
575 acoustic emission. It was shown that their respective onset of damage scalings
576 in terms of the ply thickness differed strongly, indicating two different events. A
577 good correlation between the acoustic onset of damage criterion and the actual
578 observable propagation of transverse cracks into the bulk of the samples was
579 demonstrated by ex-situ micrographies of cut samples and tomographic obser-
580 vations. Furthermore, it was shown that transverse cracks do not originate from
581 the center of the specimens but instead propagate from the free edge into the
582 bulk. Free edge cracking and bulk transverse cracking are thus two different
583 mechanisms with different stress onsets. This propagation of transverse cracks
584 from the free edge into the bulk was shown to be specific to intermediate ply
585 thicknesses, typically in the 50 μm to 200 μm range, as delamination dominates
586 the damage behaviour of the samples when larger plies are used, whereas the
587 translamina fracture strength of the 0° plies becomes dominant with thinner
588 ones.

589
590 The onset of damage predicted by the embedded cell FE model in the bulk
591 of the material showed a very good agreement with the values obtained experi-
592 mentally using acoustic emission. When the free-edge behaviour was simulated,
593 using what is essentially the same model using different boundary conditions,
594 results in good agreement with the experimentally observed transverse cracking
595 onset at the free edge were obtained. Attention should therefore be paid in
596 the future to not compare plane-strain models of transverse cracking to experi-
597 mental free edge observations without first ensuring that these observations are
598 representative of the damage in the bulk of the material. The finite size of the
599 fracture process zone could also be observed in the FE models, which leads to a
600 decrease of apparent toughness for thinner plies. The discrepancy between the
601 experimental onset of damage measured by acoustic emission and LEFM-based

602 analytical models can thus be explained by the incomplete development of the
603 fracture process zone in confined thin plies which limits the ERR and therefore
604 leads to a deviation from the $1/\sqrt{t}$ scaling derived from LEFM under the as-
605 sumption of constant ERR.

606

607 Further experimental characterization of the crack propagation into the bulk
608 of the samples as well as an extension of this work to other materials should
609 help gain an insight into the generic mechanisms at play, paving the way to
610 modelling of the in-situ strength mechanism for very low ply thicknesses at the
611 meso- and macro-scale.

612 **Acknowledgements**

613 This research was funded by the Swiss National Science Foundation, project
614 200021_156207. The authors wish to thank North Thin Ply Technology for the
615 material supply and Gary Perrenoud from PIXE/EPFL for the X-Ray scans.

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TABLE 1: Summary of the selected samples used for the interrupted tests

ply thickness (μm)	applied strain (%)	applied strain (%)
	(observation technique used) <i>before AE onset</i>	(observation technique used) <i>past AE onset</i>
75	0.89 (sample sectioning)	-
100	0.64 (tomography)	
	0.83 (tomography)	0.89 (tomography)
	0.83 (tomography with slit)	
150	0.64 (sample sectioning)	0.89 (sample sectioning)
	0.64 (tomography)	0.89 (tomography)

TABLE 2: Material properties used in the FE model, taken from [1] and manufacturer’s specifications. Newly obtained or assumed data as discussed in this work is denoted by a (*)

Fibre Properties	
Density ((kg m ⁻³))	1750
Young’s modulus E ₁₁ ^f (GPa)	377
* Young’s modulus E ₂₂ ^f (GPa)	11
* Poisson ratio ν ₁₂ ^f (-)	0.29
* Poisson ratio ν ₂₃ ^f (-)	0.45
* Shear Modulus G ₁₂ ^f (GPa)	44
* Shear Modulus G ₂₃ ^f (GPa)	3.5
UD lamina Properties	
Density (kg m ⁻³)	1500
Young’s modulus E ₁₁ (GPa)	220
Young’s modulus E ₂₂ (GPa)	7.01
Poisson ratio ν ₁₂ (-)	0.314
Shear Modulus G ₁₂ (MPa)	4661
Shear Modulus G ₂₃ (MPa)	2417
Matrix Properties	
Density (kg m ⁻³)	1200
Young’s modulus E _m (MPa)	3261
Poisson ratio ν _m (-)	0.35
* Drucker-Prager friction angle β (°)	24
* Drucker-Prager dilatation angle Ψ (°)	35
* Init. hydrostat. tensile strength p _t (MPa)	35
* Onset of damage compressive strain ε _c ^c (%)	25
* Onset of damage shear strain ε _s ^c (%)	1.75
* Onset of damage tensile strain ε _t ^c (%)	1.75
* Critical Energy Release Rate G _m (J/m ²)	64
Interface properties	
* Interface normal stiffness K _{nn} (N/mm ³)	2E7
* Interface shear stiffness K _{ss} & K _{tt} (N/mm ³)	4E7
* Interface normal strength τ ₁ ⁰ (MPa)	20
* Interface shear strength τ ₂ ⁰ & τ ₃ ⁰ (MPa)	30
* Interface critical Energy Release Rate G _i (J/m ²)	3

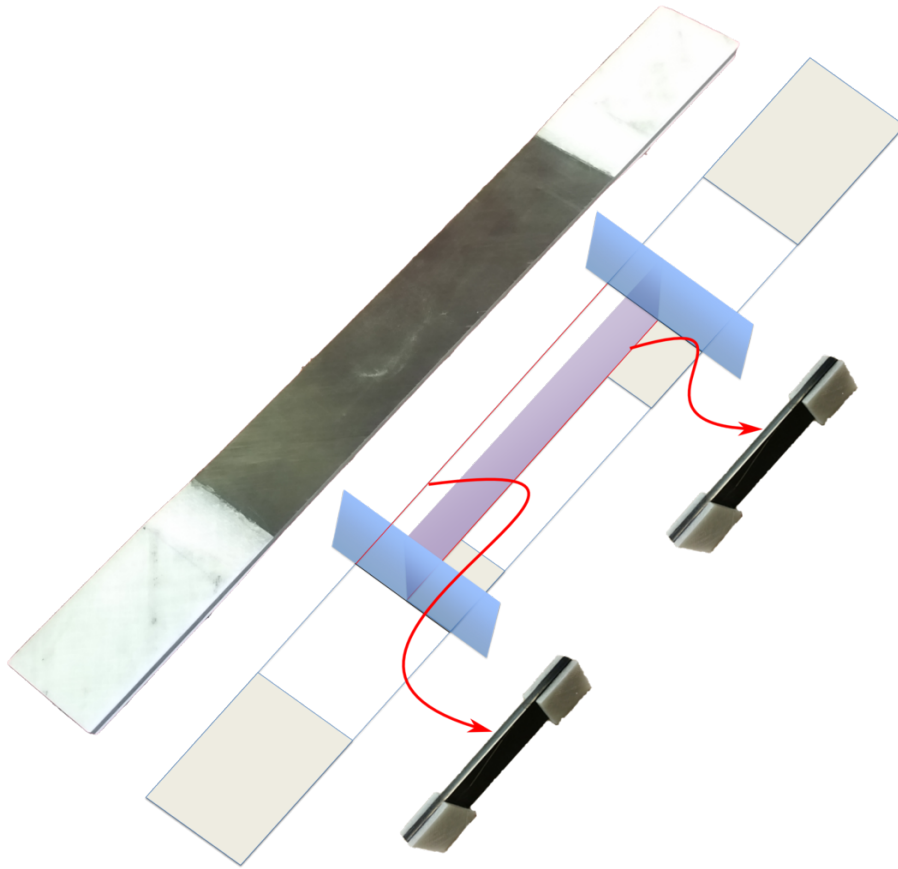


FIGURE 1: Schematic of the cut done to the samples to reveal the center of the specimens

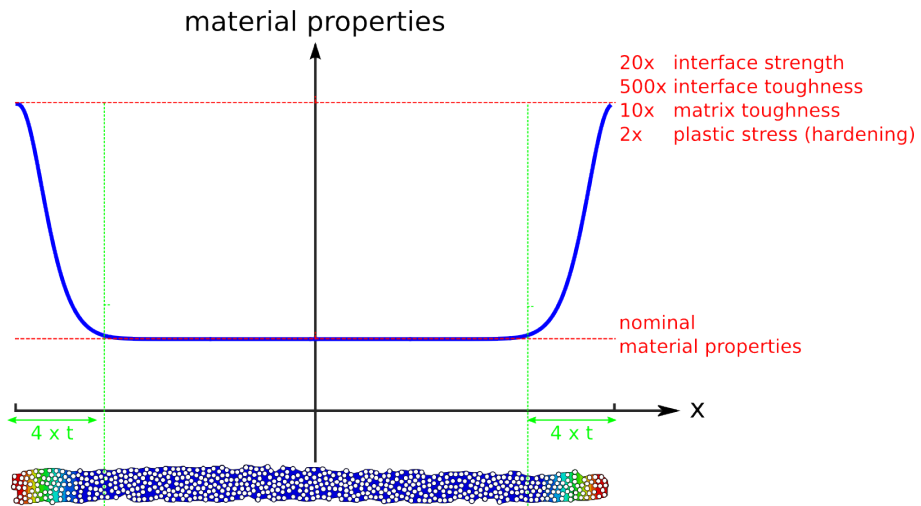


FIGURE 2: Transition zone used at the edge of the embedded cells, showing the function used to increase the fracture properties as a function of the x position. The overlaid embedded cell at the bottom with contour plots varying from the nominal properties to the maximal ones shows an example of its application

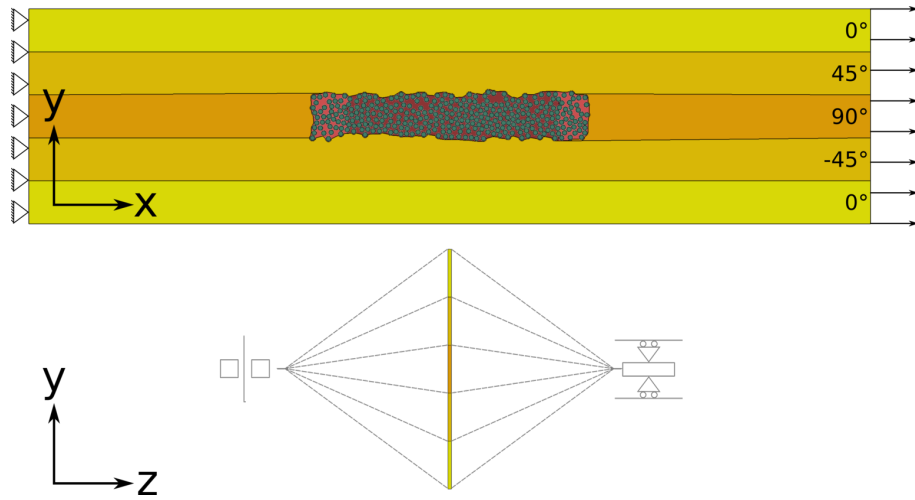


FIGURE 3: Bulk numerical model description. The applied boundary condition impose a plane-strain state of stress. The transition zone is highlighted by a lighter red than the rest of the matrix, with all fibres coloured green.

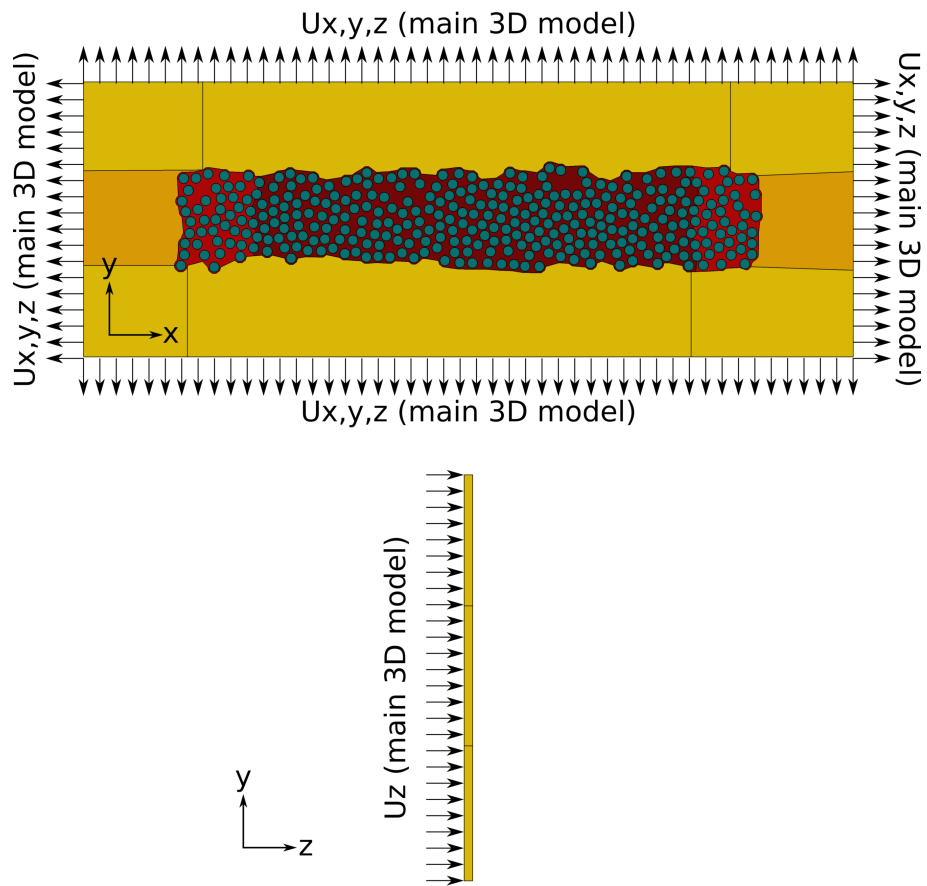


FIGURE 4: Submodel numerical model description, used to represent the free edge behaviour. It is driven by the displacements fields obtained from a linear elastic, ply-level homogenised model in the x and y directions in the microstructure's plane, and in the z direction on its back face.

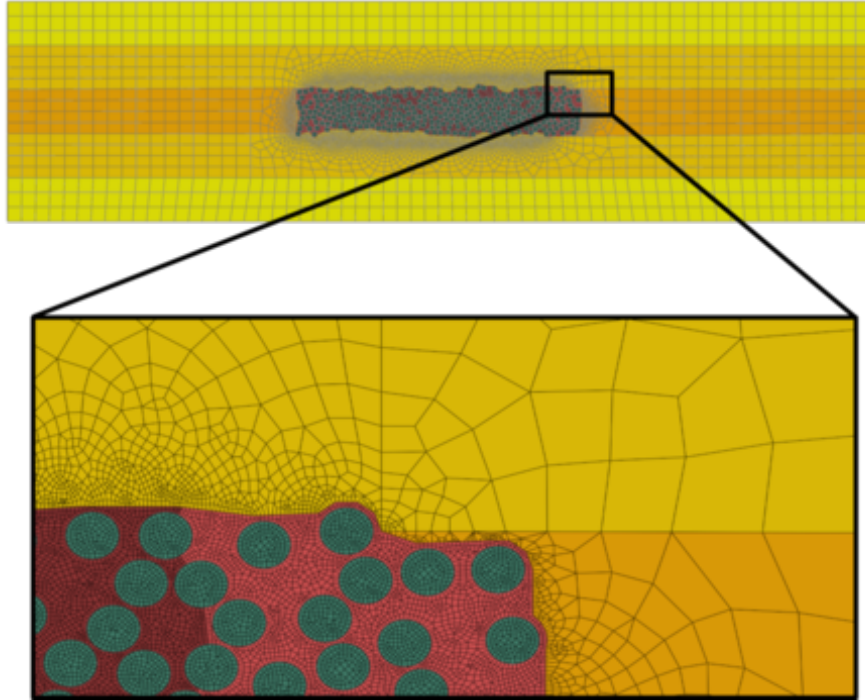


FIGURE 5: illustration of the mesh on the $t=150\ \mu\text{m}$ model

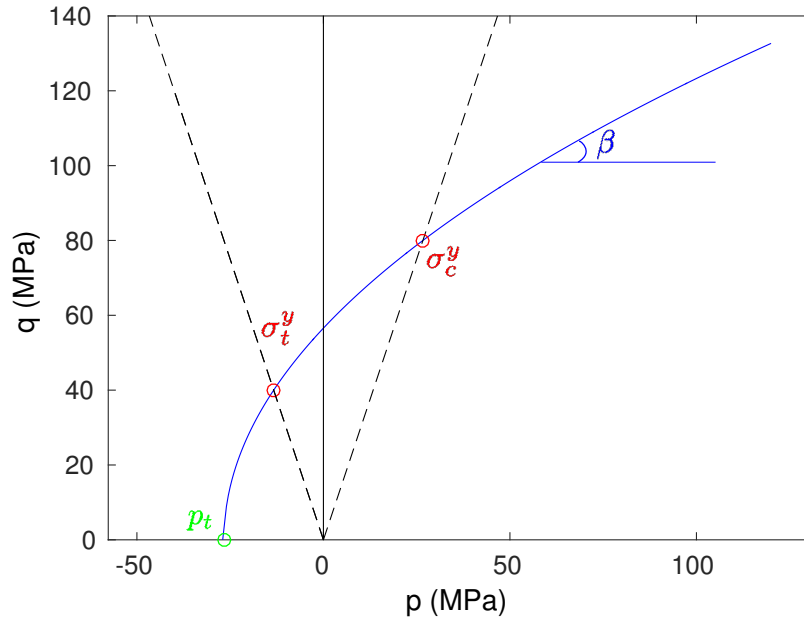


FIGURE 6: Identified Drucker-Prager model, with the experimentally measured uniaxial results shown in red and the deduced hydrostatic strength shown in green. p is the hydrostatic pressure and q the equivalent von Mises stress

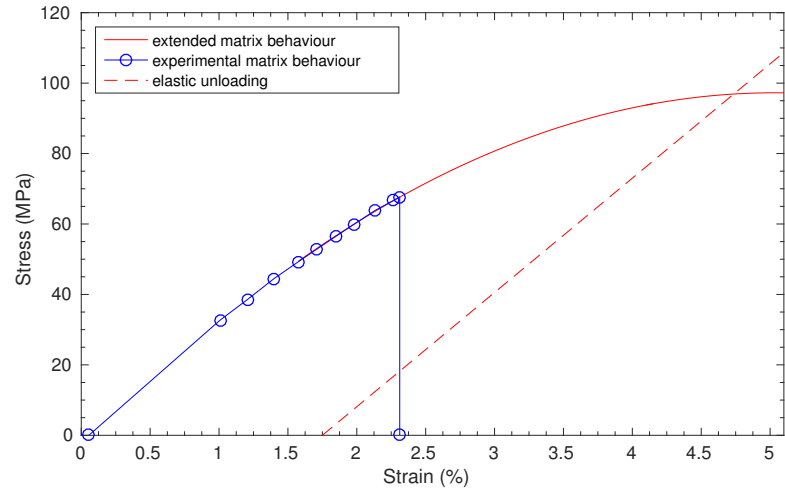


FIGURE 7: TP80 elasto-plastic model, with the experimental values in blue and extended curve in red. The dashed line is the elastic unloading, used to predict the maximal plastic strain

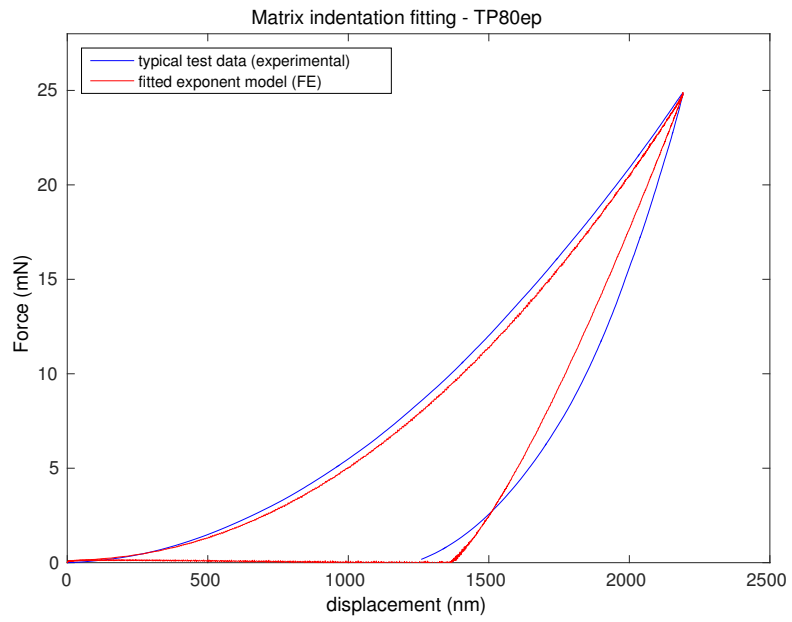


FIGURE 8: Nano-indentation Force-Displacement comparison between experimental and numerical identified model

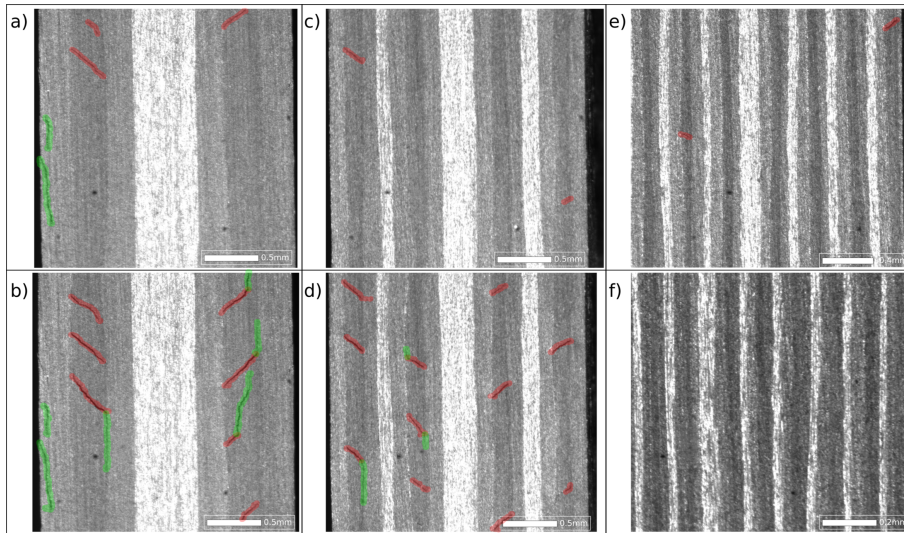


FIGURE 9: Optical in-situ micrographies showing the damage development at the free edges of chosen specimens. Transverse cracking is highlighted in red and matrix cracking induced delamination in green. $t=300\ \mu\text{m}$ at 0.31% strain in (a) and 0.55% in (b), $t=150\ \mu\text{m}$ at 0.5% strain in (c) and 0.85% strain in (d), $t=75\ \mu\text{m}$ at 0.9% strain in (e) and $t=30\ \mu\text{m}$ at 1.05% strain in (f)

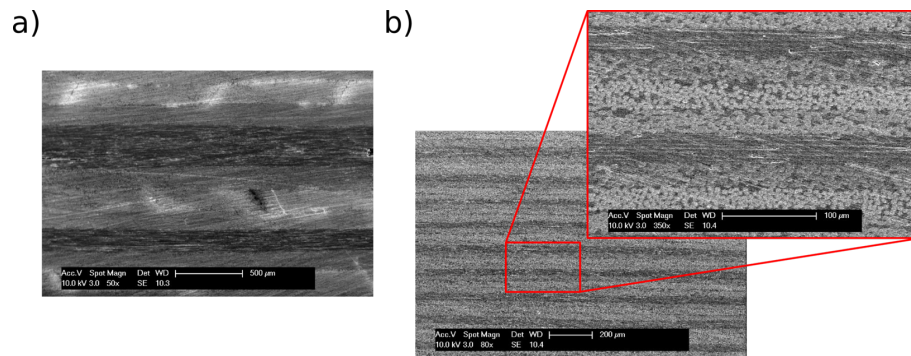


FIGURE 10: SEM visualisation of the polished edge of broken samples. a) for 150 μm plies, with clear signs of transverse cracking, and b) for 30 μm plies, with no visible damage in the 90° plies. If tell-tale signs of transverse cracking are extremely visible in (a), they are totally absent in (b)

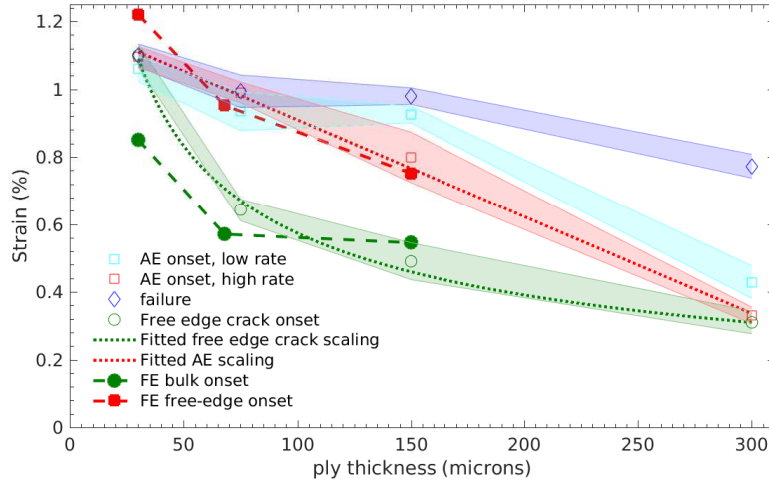


FIGURE 11: Macro-Scale Results, showing the good agreement of the free edge cracks to a $1/\sqrt{t}$ scaling, whereas the onset of damage defined by an acoustic emission threshold is in better agreement with a linear scaling, and lightly loading-rate dependant. The corresponding numerical results are shown in dashed line, showing the good agreement with experimental results

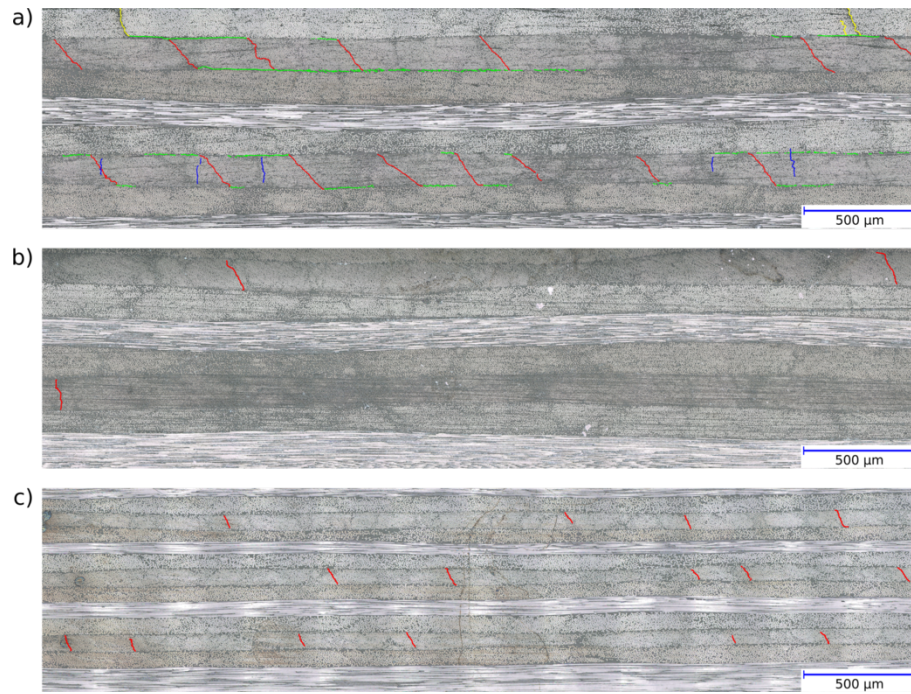


FIGURE 12: Free edge micrographs of the partially loaded specimens, manufactured with 150 μm plies and loaded until 0.89% strain in (a), manufactured with 150 μm plies and loaded until 0.64% strain in (b), and manufactured with 75 μm plies and loaded until 0.89% strain in (c). Transverse cracks at the free edge are highlighted in red, the same damage type reaching the middle of the sample in blue, delamination in green and secondary damage in yellow

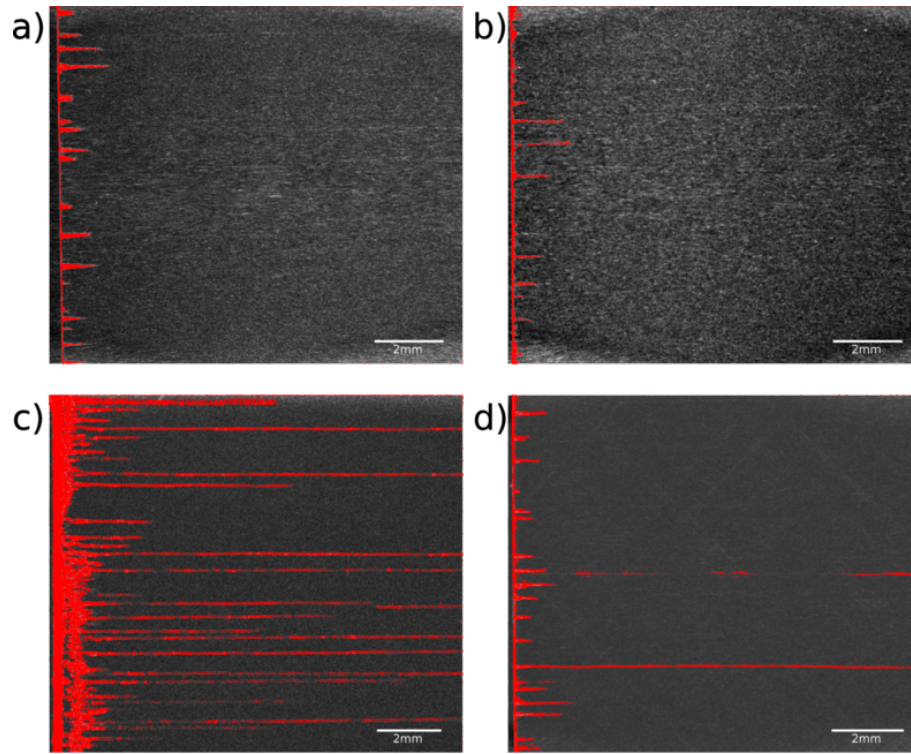


FIGURE 13: Typical 90° plies : a) $t=150\ \mu\text{m}$ loaded until 0.64% strain, b) $t=100\ \mu\text{m}$ loaded until 0.83% strain, c) $t=150\ \mu\text{m}$ loaded until 0.89% strain, d) $t=100\ \mu\text{m}$ loaded until 0.89% strain. Transverse cracking clearly propagates into the bulk of the samples past AE onset.

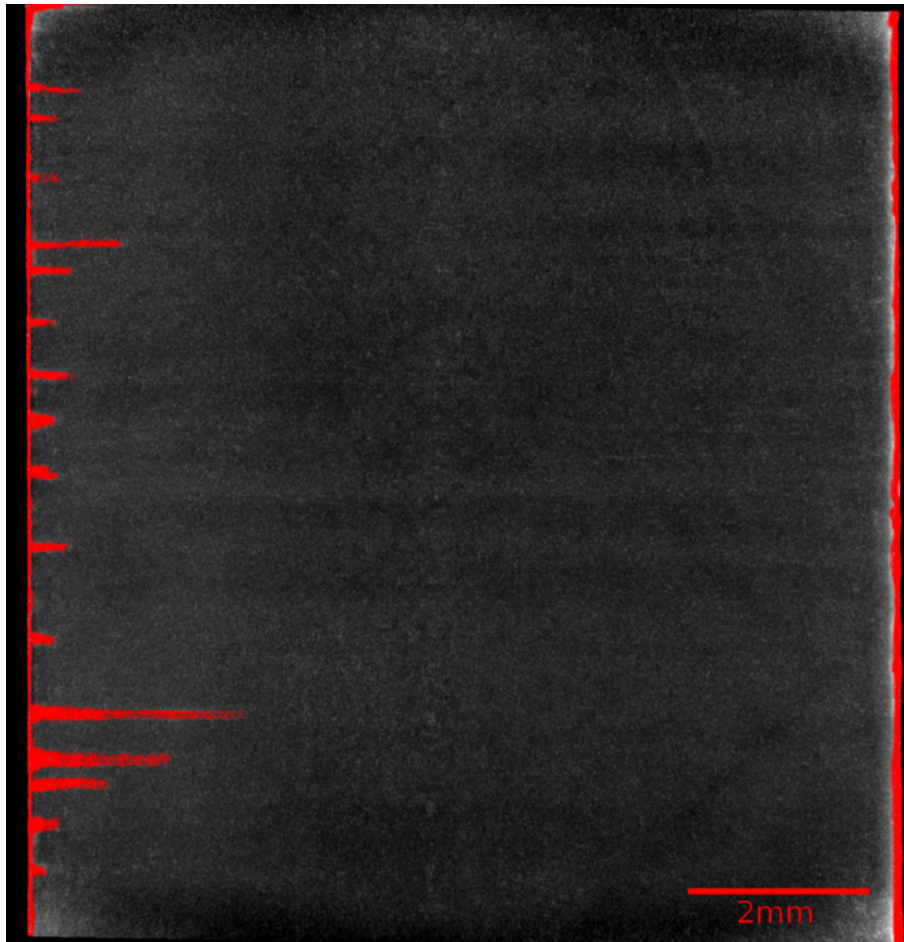


FIGURE 14: Typical 90° ply of the machined sample, revealing no evidence of damage originating from the center of the specimens

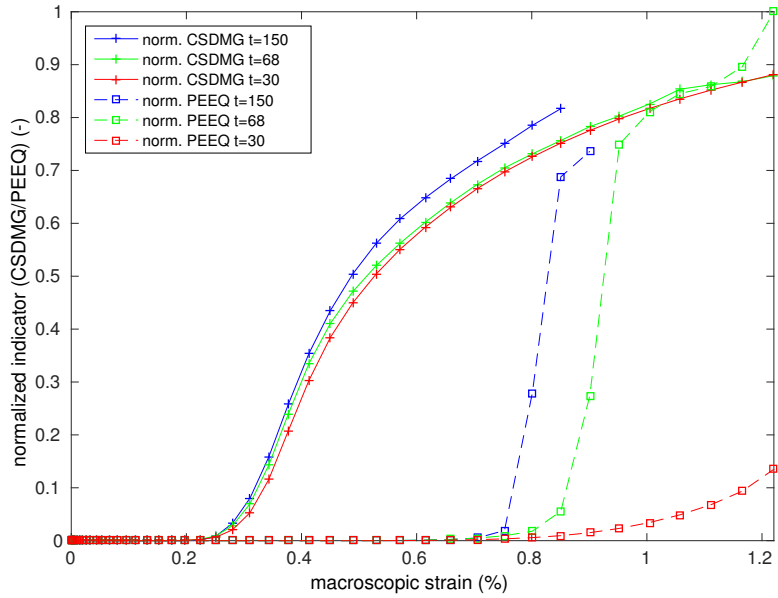


FIGURE 15: Average interfacial (cohesive) damage indicator CSDMG and equivalent plastic strain PEEQ for the three ply thicknesses, normalised by the maximum value reached for all ply thicknesses. Clearly, the decrease of ply thickness delays damage localisation.

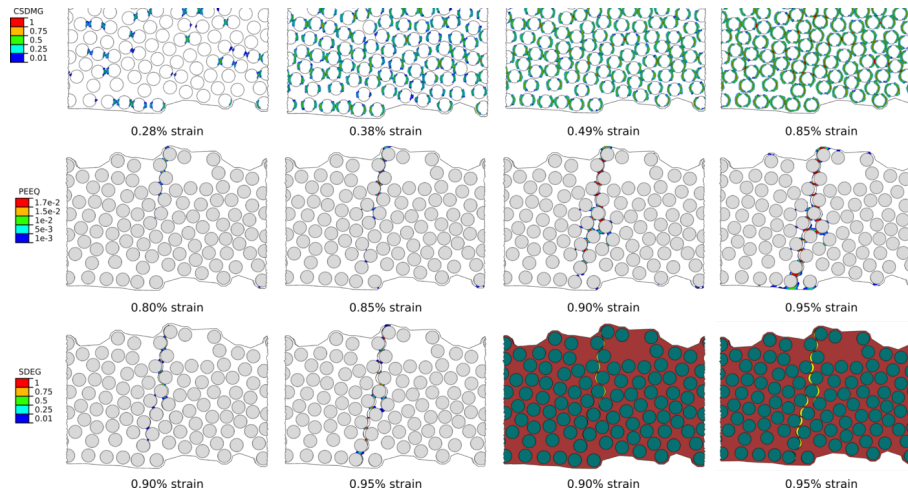


FIGURE 16: Damage evolution of a typical crack in the 68 μm model. The typical damage process starting by progressive debonding (CSDMG damage variable) followed by plastic straining of the matrix ligaments represented by their equivalent plastic strain (PEEQ) leading to their eventual damage (SDEG damage variable) is highlighted. The two figures at the lower right corner highlight the progressive crack opening, with the opening voids shown in yellow.

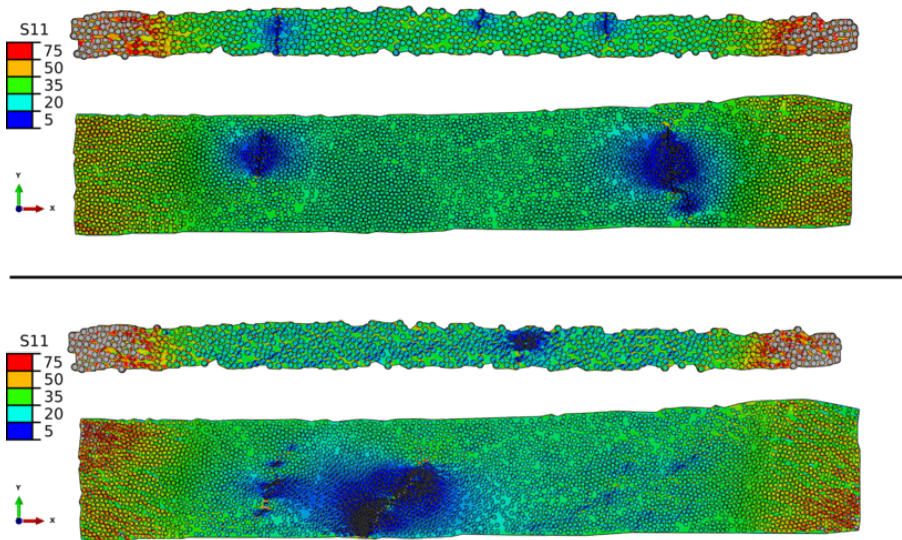


FIGURE 17: σ_{11} at 1.3% applied strain for the 30 μm model and 0.82% for the 150 μm model in the bulk and at 0.85% for the 30 μm respectively 0.53% for the 150 μm at the free edge. The crack propagation lasts for a few time steps of the simulation and the present illustrations represent the state at which the onset of damage was defined.

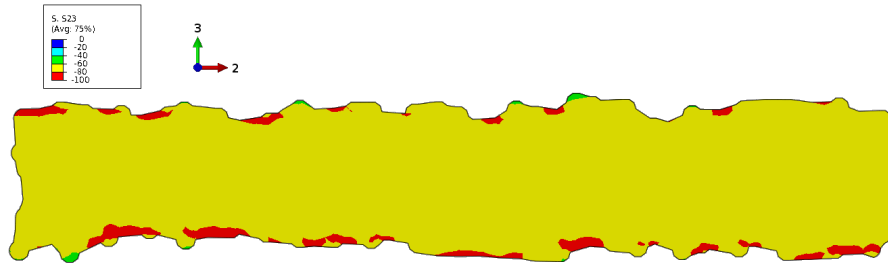


FIGURE 18: τ_{23} stress at the free edge of the $t=68\ \mu\text{m}$ M40JB-TP80ep model, explaining the angle of the transverse cracks observed both experimentally and numerically at the free edge

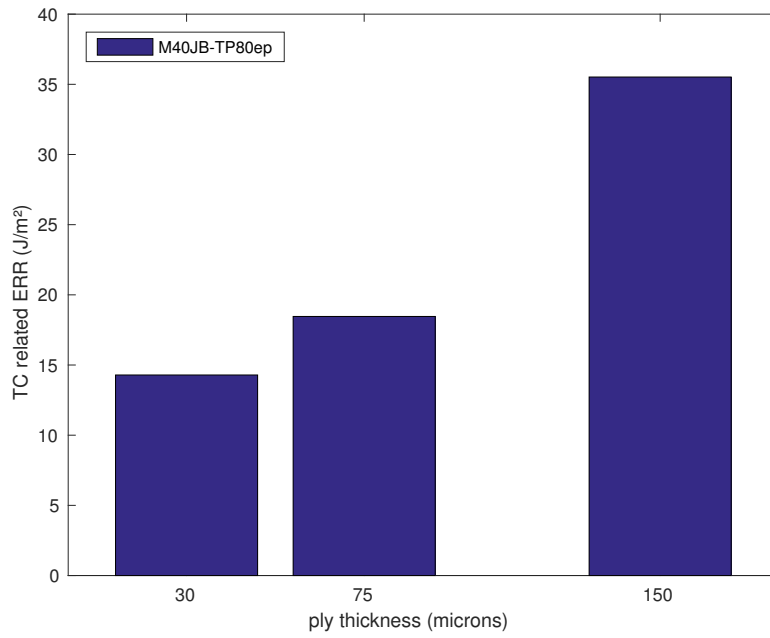


FIGURE 19: Critical transverse cracking related ERR obtained from the bulk numerical model.