SHEAR CHARACTERISATION OF UD THERMOPLASTIC COMPOSITES

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ABSTRACT: Intra-ply shear is one of the main mechanisms to accumulate deformations in thermoplastic composites forming processes. In this paper a shear characterisation method for uni-directionally (UD) reinforced thermoplastics in their molten configuration is presented. Bar-like specimens made from UD carbon PEEK material are subjected to oscillating torsional loads. By utilising linear visco-elastic theory, the small strain oscillatory responses are translated to storage and loss shear moduli. The frequency dependent moduli are subsequently translated to the transient domain and show a close-to elastic response for the considered instantaneous strain rates. The application of such characterisation data is demonstrated by modelling the stamp forming process of a complex shaped product with the aid of the finite element method. The process considered deals with an initially flat quasi-isotropic laminate consisting of eight plies, which is formed at high temperatures. Predicted intra-ply shear strains are compared with those measured by photogrammetry in a real formed part. Predicted and measured shear strain distributions and their magnitudes were in good agreement. Moreover, critical regions in which small wrinkles develop were indeed indicated by the forming predictions.

KEYWORDS: *thermoplastic, laminates, uni-directional, shear, characterization, forming, experiments, modeling*

INTRODUCTION

Hot stamp forming of fibre reinforced thermoplastic laminates is ideally suited for the production of thin shelled products with complex curvatures. Nevertheless, process induced defects such as local buckling and subsequent wrinkling appear frequently and disqualify the final product. Better anticipation on these defects results in lead-time reductions and can be achieved by predicting such defects in the early product design stages.

Simulation tools for the stamp forming process of uni-directional (UD) fibre reinforced thermoplastics aim to reduce the number of optimisation cycles during the development phase of a product. Constitutive models are needed to describe the occurring deformation mechanisms, such as intra-ply shear, out-of plane bending, and ply-ply and tool-ply friction. These models subsequently require reliable material property data and corresponding characterization methods.

An alternative method for intra-ply shear characterization by means of torsion is presented. The measured data is subjected to some conversions, such that it can be used in forming simulations. Indeed, the forming process of a complex product is simulated as a next step, with the obtained characterization data involved. The other deformation mechanisms are characterised by other methods and are not considered here. The simulation results are then briefly compared with a real stamp formed product.

SHEAR CHARACTERISATION

The published methods for intra-ply shear characterisation of UD fibre reinforced polymer melts result in distinctly different material parameters, as was summarised clearly by Harrison [1]. For example, a difference of several orders of magnitude was found between plate-plate rheology [2] and picture frame experiments [3]. Therefore, the development of an alternative shear characterisation technique is desired. Reliability, repeatability, and easy accessibility are the desired properties of the envisioned test. The method shown here comprises a straight rectangular bar that is subjected to a torsional load. The set-up makes use of a commercially available rheometer. A set of slightly modified standard fixtures is used, which are generally applied to bar like specimens in their solid to rubbery material state.

Torsion of bars with a rectangular cross section

The method presented here deals with a prismatic bar that is subjected to torsion. The bar has a constant rectangular cross section, as shown in Fig. 1. It is assumed that the bar consists of UD fibres that run parallel to the rotation axis. Torsional loads are introduced via clamps that are attached to both ends of the specimen. At each end the specimen is clamped by two clamping surfaces parallel to the axis of rotation.

Considering an elastic rectangular bar with length L, the torque M can be related to the rotation angle ϕ and a shear modulus G via [4]:

$$M = GJ\frac{\phi}{L} \tag{1}$$

The torsional rigidity J for a rectangular cross section with dimensions t and w reads:

$$J(t,w) = \tfrac{1}{3}t^3 w\left(1 - \frac{192}{\pi^5}\cdot\frac{t}{w}\cdot\sum_{k=1}^{\infty}\frac{1}{(2k-1)^5}\cdot\tanh\left(\frac{(2k-1)\pi w}{2t}\right)\right) \tag{2}$$

It is aimed to subject the rectangular bar to dynamical mechanical tests that involve small amplitude oscillatory deformations through a range of frequencies. Such tests are generally used for the characterisation of visco-elastic materials. Sinusoidally oscillating loads can be represented by replacing the variables in Eqn. (1) with their complex variants. The following relation to determine the complex shear modulus of the tested material then yields:

$$G^* = G' + iG'' = \frac{M^*}{J}\frac{L}{\phi^*} \qquad \text{with} \quad \phi^* = \phi_0 e^{i\omega t} \quad \text{and} \quad M^* = M_0 e^{i(\omega t+\delta)} \tag{3}$$

and can straightforwardly be applied to the measured responses of the device at hand.

Specimen preparation and experiments

Torsion bar specimens were produced with polyether ether ketone (PEEK) UD carbon fibre prepreg, commercially available as CETEX Thermo-Lite® from Ten Cate. The specimen production comprises the hot pressurised consolidation of a stack of 80 equally orientated plies, resulting in an 11 mm thick laminate with a 59±3 % fibre volume fraction. The laminate was subsequently cut with a diamond wheel saw to yield specimens with a width and length of 13.0±0.1 mm and 60.6±0.1 mm, respectively. Thick bar-like specimens are preferred, because thin strip-like specimens are more sensitive to unwanted fibre tensions that might occur [5].

Once the specimen is accurately positioned within the fixtures as in Fig. 1 (centre), it is enclosed by a temperature controlled chamber or cavity. The specimen is heated to 390^{o}C and gentle flow of nitrogen gas is applied within the cavity in order to avoid polymer degradation effects that are driven by the presence of oxygen. Strain sweeps are performed to explore the linear region of the material response. The maximum shear strain that appears in the rectangular cross section is used as a reference throughout all the analyses and appears at the centre of the longest side of the rectangular cross section. Frequency sweeps are carried out to investigate the material response for a range of loading rates.

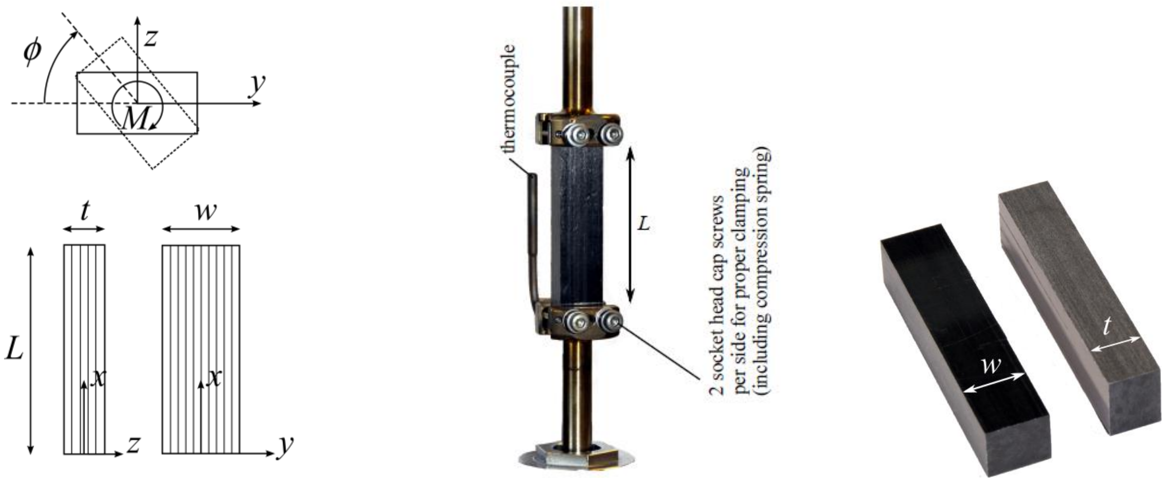

Fig. 1: Left: schematic representation of the UD reinforced straight bar. Centre: fixtures with a mounted specimen. Length *L* indicates the length between the clamps, not the specimen's length itself. Right: typical geometry of the specimens.

Results and discussion

Fig. 2 shows the averaged results of both the amplitude and frequency sweeps for five specimens. The strain range covered during the amplitude sweep is approximately one decade only (Fig. 2, left). This was chosen in order to minimise effects such as fibre migration, which might occur for larger deformations. Such interesting non-linear phenomena could probably lead to deviating responses but are not considered here. The measured shear moduli are constant for the investigated shear strain range. Together with the waveform data that looks visually good, this implies linear visco-elastic material behaviour and thus Eqn. (3) can be applied straightforwardly.

Frequency sweeps were carried out subsequently with shear strain amplitudes of $\gamma_0 = 0.1\%$. The measured moduli are weakly dependent on frequency. A constantly increasing slope can be deduced for the considered range of frequencies. The storage terms are consistently larger than the loss terms within the range of investigated parameters. This behaviour is substantially different from the polymer characteristics without the presence of fibres. The neat polymer characteristics show increasing storage and loss moduli for increasing angular frequencies, as well as a dominating loss modulus. The elastic dominance of the tested composite system here is most likely caused by the presence of the fibres. Multiple fibre-fibre interactions will be present and it is supposed that these invoke elastic effects during material loadings [6].

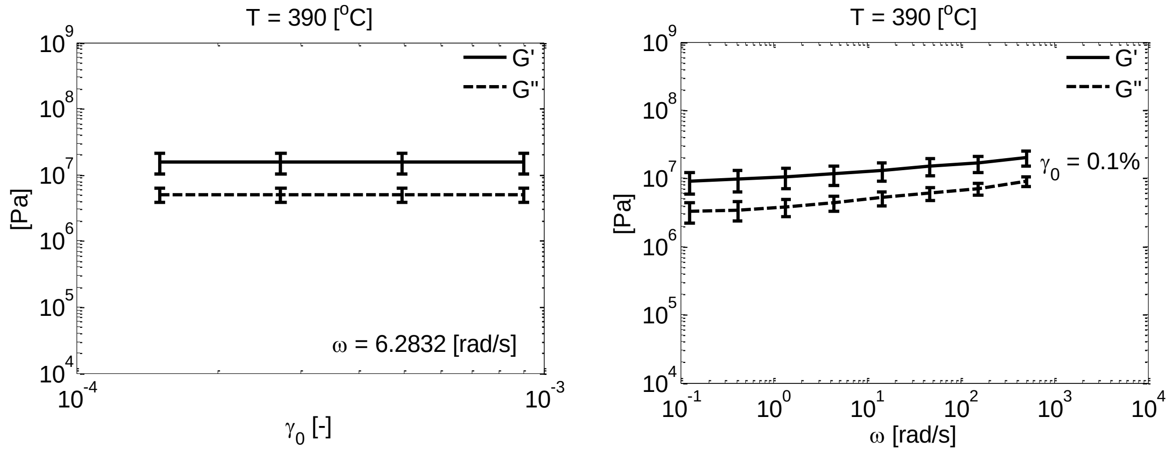

Fig. 2: Storage G' and loss G'' moduli, as function of strain amplitude γ_0 (left), and as function of angular frequency ω (right).

Translation to the transient domain

The shear behaviour for small strains is now known in the frequency domain. Since the forming process takes place in the transient time domain, the measured characteristics in Fig. 2 have to be translated. The well-known empirical Cox-Merz relationship is often used for neat polymer melts with a dominating loss modulus. It relates the complex viscosity as a function of the angular frequency, to the steady shear viscosity as a function of shear rate. Generally, this relationship cannot be applied for dispersed media. This was for example shown by Kitano [7] for short glass fibre filled polyethylene (PE). Generally, transient deformations of linear visco-elastic materials are described via the following constitutive relationship from linear visco-elasticity theory (LVE) [8]:

$$\tau(t) = \int_{s=-\infty}^{s=t} G(t-s)\dot{\gamma}(s)ds \tag{4}$$

where τ is the shear stress, $\dot{\gamma}$ the shear rate, and $G(t)$ is the shear relaxation modulus. The integration is carried out over all past times s up to the current time t. The shear relaxation modulus needs to be determined to describe the transient interaction between shear stress and shear strain and its rate.

Another relation from LVE theory relates this relaxation modulus to the frequency dependent loss modulus [8] via:

$$G(t) = \frac{2}{\pi}\int_0^\infty \frac{G''}{\omega}\cos(\omega t)d\omega \tag{5}$$

where we recognise the inverse Fourier cosine transform. Since the loss modulus shows a straight line in the frequency plot, it can be approximated by a power law function as shown in Fig. 3 (left). Following a similar procedure as was done by Campanella [9], substitution of this fairly simple relation into Eqn. (5) allows for its analytical evaluation. Substitution of the resulting analytical expression into the constitutive relationship from the LVE theory in Eqn. (4), finally results in the following shear stress response for an instantaneously applied constant shear rate $\dot{\gamma}_n$:

$$\tau(t) = \frac{2}{\pi}\dot{\gamma}_n a\Gamma(1-p)\sin\left(\frac{p\pi}{2}\right)\frac{t^p}{p} \tag{6}$$

in which Γ is the error function. In this analysis, the equilibrium modulus has been omitted for now. It will be accounted for in future analyses, together with a more representative description of both the moduli. Fig. 3 (right) shows these stress responses for three different shear strain rates, which are typically the magnitudes as they appear in stamp forming processes. The material shows a close to elastic response, however, slightly dependent on shear rate. Nevertheless, the material will be regarded as behaving elastically within the range of shear rates that appear during the stamp forming processes considered here. Such behaviour has been assumed earlier by Scobbo [10], who states that within the considered range of shear rates the material indeed acts as an elastic solid. Or as cited from [11]: "Although hot rubbery polymers exhibit both solid rubbery and rubbery liquid characteristics, in the limit, thermoforming is a solid phase deformation process".

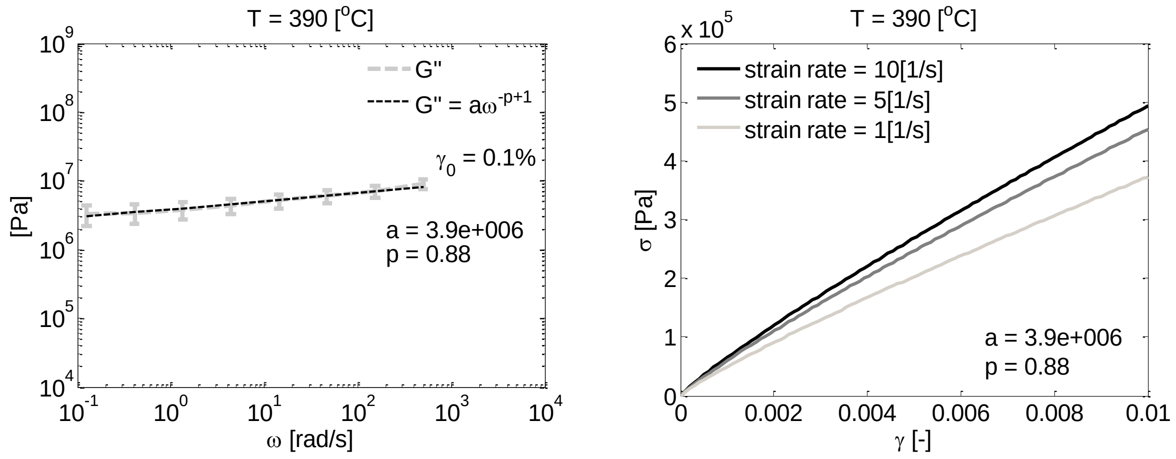

Fig. 3: Left: straight line approximation for the loss modulus. Right: calculated stress-strain response from Eqn. (6) as a result of a instantaneously applied constant shear strain rate.

APPLICATION

The measured shear characteristics find their application in forming simulations. This section shows such an application for which a fairly complex product was selected. It comprises a stiffening rib with features such as flanges and double curvatures as displayed in the centre of Fig. 4. It is one of the many parts of the wing fixed leading edge assembly, designed and built by Fokker Aerostructures. Although the product design is tailored for the stamp forming process of flat sheets of another conventional composite material, we consider an eight layered quasi-isotropically stacked laminate (Fig.4, left) with the UD carbon PEEK material for this research. Firstly, the forming process and the resulting product shape will be shown. Secondly, the modelling and the resulting predictions of the intra-ply shear strains will be discussed.

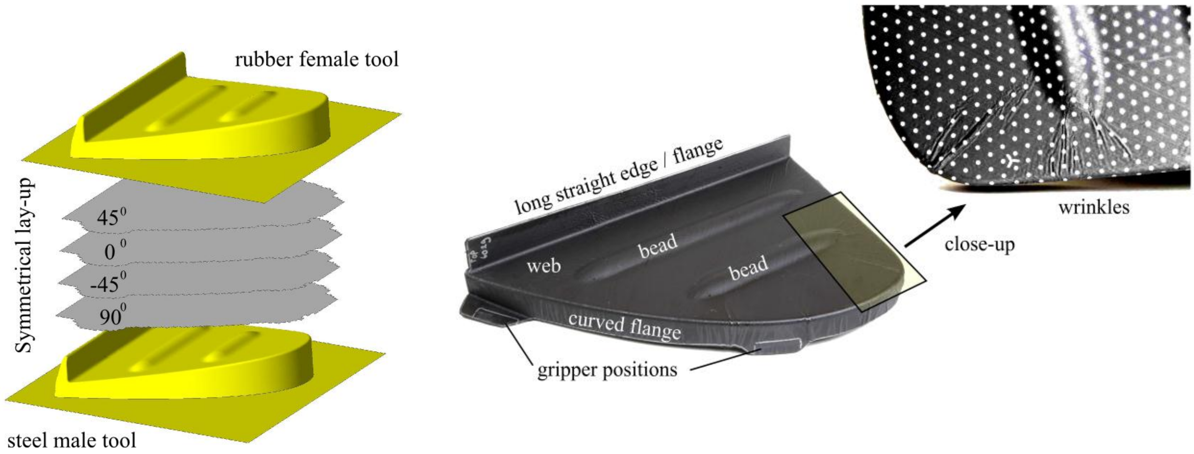

Fig. 4: Left: stamp forming set-up as used for forming experiments and process modelling, the laminate consists of eight plies in total. Centre: the resulting J-nose stiffener with the definition of some features. Right: partial close-up showing process induced defects.

Forming experiment

A flat blank with a shape as shown in Fig. 4 (left) was cut out of a flat pre-consolidated laminate. The blank is subsequently heated in an infra-red oven and transported between a pre-heated steel male and a cold rubber female tool. Subsequently, the hot blank is formed by the downwards movement of the female tool with a typical speed of 20 mm/s. A detailed process analysis was performed in [12]. The resulting product shape is shown in the centre of Fig. 4. The close-up on the right of Fig. 4 shows small wrinkles mainly occurring in the web and originating from the doubly curved bead ends.

As can be seen in the close-up in Fig. 4, blanks were prepared with a white dot pattern. This was applied with paint brush tooling in order to quantify the intra-ply strains. The position of the dots before and after forming was measured with the aid of photogrammetry software. Intra-ply strain components were subsequently determined by calculating the deformation gradients for each triangle, as determined by the dots. The resulting shear strain components of the top ply are shown in Fig. 5 (left). Dominating shear strains are visible near the bead ends, however, these mainly have an apparent character due to the presence of wrinkles in those areas (Fig. 4, right). The strain measures correctly represent the intra-ply shear strains for the regions without wrinkles and vary between -0.02 and 0.02, corresponding to fibre shear angles between $\approx -1^{o}$ and 1^{o}.

Forming predictions and discussion

The AniForm finite element software was employed to model the iso-thermal forming process according to the virtual set-up in Fig. 4 (left). A similar modelling methodology was described in a previous publication [13]. The male and female tools are modelled as rigid surfaces, each containing 37.000 elements. Eight individual plies were modelled separately with 17.000 elements each. These plies consist of 3-node triangular shell elements (discrete Kirchhoff triangles, DKT). An elastic fibre model in combination with the characterised intra-ply shear behaviour was modelled for the in-plane material responses. The characteristics in Fig. 3 (right) were assumed to be elastic and are represented with a Mooney-Rivlin model. An orthotropic elastic model is used for the DKTs to model the bending behaviour of the plies. Tool-ply friction was measured with an in-house developed friction tester [14] and the characteristics were processed in the models that describe contact logic at the modelled interfaces.

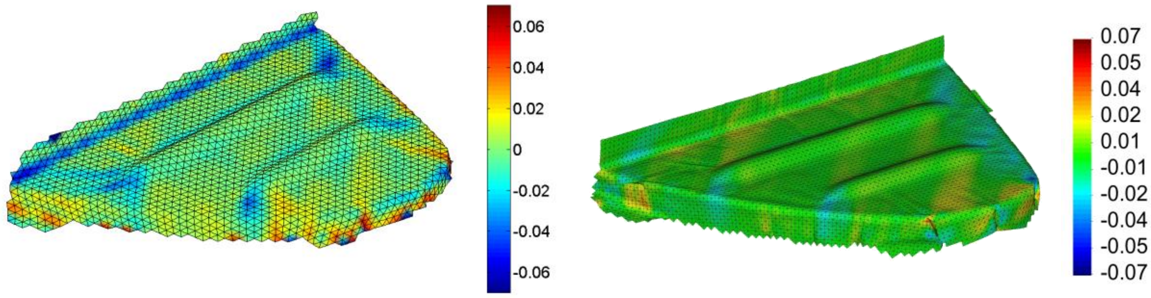

Fig.5: Intra-ply shear distributions of the top ply, expressed in the shear component of the Green-Lagrange strain tensor $\varepsilon_{xy} = \gamma/2$. Left: experimental result, determined with photogrammetry. Right: forming simulation result.

Fig. 5 (right) shows the predicted product shape and intra-ply shear strains of the top ply. A small contribution of intra-ply shear mechanisms is indicated, which was also concluded from the photogrammetry results in Fig.5 (left). Some wrinkles or waves are predicted as well near the bead ends, however, the real process induced defects are too small to be represented with a typical finite element mesh size. Nevertheless, the predicted wrinkles or waves indicate potential problematic areas.

During the forming process of the considered geometry in combination with the quasi-isotropic layup, out-of-plane bending is the most pronounced of all mechanisms that appear. The experiment suggests that probably more energy is required for intra-ply shearing, compared to the local out-of-plane bending. The latter will eventually develop and consequently results in wrinkles. The model prediction indicates this as well, which suggests that the magnitude of the measured intra-ply shear characteristics, as well as the elastic behaviour approximation, is likely to be a good approach.

CONCLUSIONS

An alternative shear characterisation method has been shown. Rectangular bar shaped specimens comprising a UD carbon PEEK material were subjected to oscillatory torsional loads. The measured responses show a storage modulus that is consistently larger than the loss modulus. Moreover, a weak frequency dependency was found which suggests that the material behaves like a solid or weak gel [15]. The frequency dependent moduli were subsequently translated to the transient domain in order to be used in a stamp forming model.

An iso-thermal stamp forming process was modelled by means of the finite element method. A complex product was selected and forming of a quasi-isotropic UD carbon PEEK laminate was simulated. Predicted intra-ply shear strains were compared with these strains in a real-life product. Stamp forming experiments were conducted and strains were extracted with the aid of photogrammetry. Both the simulation and experiment show small intra-ply shear strains, their distribution and order of magnitude in agreement. Local out-of-plane bending was the pronounced mechanism and results in small wrinkles in practise. Simulations were able to indicate these potential problematic regions, which is among others the result of proper modelling of the intra-ply shear behaviour.

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