CHARACTERISING THE FORMING MECHANICS OF PRE-CONSOLIDATED NYLON-CARBON COMPOSITE

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Abstract

Preliminary bending and shear tests are performed on a nylon-carbon fibre pre-consolidated cross-ply 0/90/90/0 laminate. The aim is to produce data for use in macro-scale forming simulations. A novel variation on the usual uniaxial bias extension tests is devised. The modifications are designed to create a well-defined boundary condition at the extremities of the test specimen while avoiding transverse squeeze flows due to clamping pressure and undesirable temperature gradients across the test specimen. A variant of the cantilever test is also developed to characterise the temperature-dependent flexural rigidity of the composite as a function of temperature. The cantilever set-up is designed to maximise the rate of testing. Reasonable results are found using both preliminary set-ups, though various causes of error are highlighted and suggestions for future improvements are made.

1. Introduction

Accurate characterisation of the forming mechanics of engineering fabrics and preimpregnated composites is a necesary step to achieving accurate forming simulations. In this investigation we aim to characterize the high-temperature forming mechanics of advanced thermoplastic cross-ply laminates. Ideally, the characterisation process should also be fast, robust, easy to perform and inexpensive. In [1], a technique for measuring four independent mechanical stiffnesses governing the forming mechanics of engineering fabrics (out-of-plane bending, shear, in-plane bending and torsional stiffness) was demonstrated using two commonly applied test methods; the cantilever bending test and the uniaxial bias extension test. The latter was modified to mitigate the influence of intraply slip and facilitate measurement of the in-plane bending stiffness. In this investigation, the same experiments are adapted to characterize the thermoforming behavior of preconsolidated biaxial cross-ply thermoplastics composites. The requirement for high temperature testing introduces challenges that are highlighted in this investigation. Novel solutions are attempted together with suggestions for future improvements.

2. Materials

A carbon fibre – nylon composite laminate consisting of four plies of TenCate Cetex® TC910 Nylon 6 UD tape in a 0°/90°/90°/0° layup was used. The thickness of the tape is 0.16 mm. The fibre volume fraction is 49% and the polymer content is 40% by weight. The average thickness of the laminate is 0.68 mm with a standard deviation of 0.10 mm. The recommended processing temperature is 249°C to

271°C. The matrix polymer is Ultramid B3W (PA6). Differential scanning calorimetry (TA Instruments model Q20 with a RCS90 cooling system and nitrogen purging gas, flow rate=50 ml/min) measured a melt temperature of 223.4°C. Limited rate and temperature dependent viscosity data for the matrix polymer are provided in the supplier's datasheet [2].

3. Test Methods

3.1 Cantilever Bending Test

The cantilever bending test is an established technique (BS EN ISO 9073-7:1998) [3] used to measure the out-of-plane bending and torsional stiffness of fabrics. The main equation of the standard is based on a truncated power series solution to the system's governing second order differential equation, provided by Peirce [4],

$$G_{P\varepsilon} = \frac{1}{\frac{\tan\phi}{\cos(0.5\phi)}} \times \frac{pL_s^3}{8} \quad (1)$$

where $G_{P\varepsilon}$ is the flexural rigidity (per unit width), Φ is the angular deflection of the cantilever end (see Figure 4), p is the mass per unit area multiplied by the acceleration due to gravity, taken here to be 9.81ms^{-2} and L_s is the cantilever strip length. Use of the simplification,

$$\frac{1}{\frac{\tan\phi}{\cos(0.5\phi)}} \approx 1 \quad (2)$$

when $\Phi = 41.5^{o}$ as recommended in [3], gives

$$G_{P\varepsilon} = \frac{pL_s^3}{8} \quad (3)$$

despite the simplifactions in the derivation of Eq (1) and also the assumption used in Eq (2), the equation was shown previously to be accurate to within ~0.3% of numerical predictions [5]. Various researchers have adapted this test to investigate the out-of-plane bending stiffness of thermosetting [6] and thermoplastic prepreg composites [7].

In this investigation we suggest a modification to the the standard test procedure to facilitate high temperature conditions. A ramp, manufactured from 1mm-thick aluminium sheet (to reduce mass and the associated thermal inertia) was designed to fit inside a Zwick environmental chamber (see Figure 1). Four specimens of different length were fastened to the top of the ramp (see Figure 1a). The temperature in the chamber was slowly increased and monitored using both the chamber's in-built thermocouple together with a temperature probe (two thermocouples sandwiched between two composite specimens, the first positioned 20mm from the ramp, T1, the second 70mm from the ramp, T2 – see Figure 1b). As the termperature increases the bending stiffness of the specimens decreases and the angular deflection of the specimens increases until eventually reaching the slope at 41.5°. By recording the temperature at which each sample touches the ramp, and by using Eq (3), the flexural rigidity can be plotted as a function of temperature. Use of a wide ramp, means that multiple specimens can be tested simultaneously, increasing the rate of data collection.

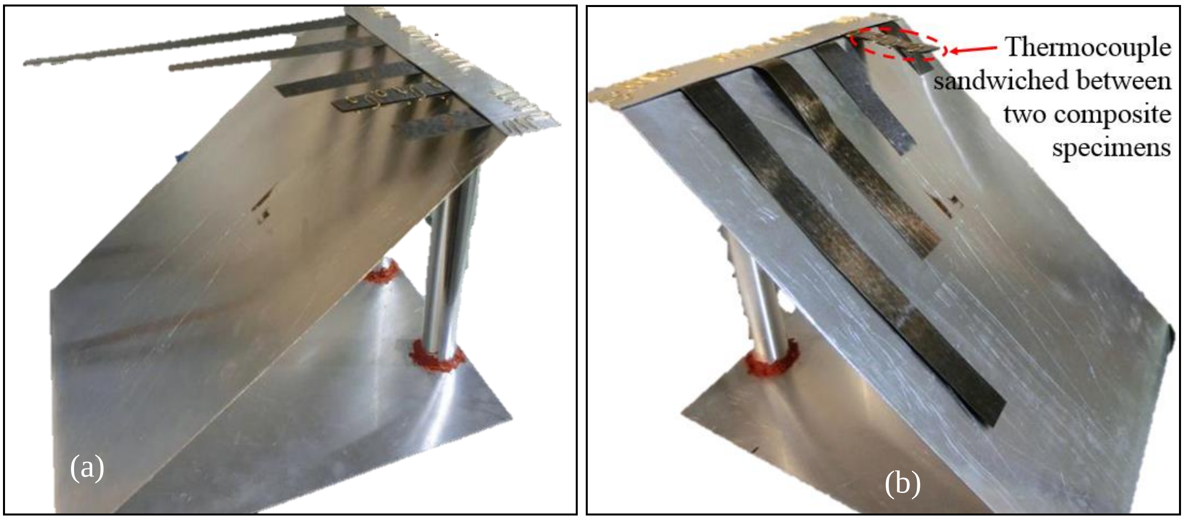

Figure 1. (a) Aluminium ramp manufactured with a slope of 41.5°. Four specimens of different lengths are fixed at the top of the ramp using an aluminium strip. A temperature probe consisting of two short strips of carbon-nylon composite sandwiching two thermocouples (20 and 70mm from the ramp), is used to monitor temperature. (b) The four test specimens after testing.

3.2 Modified Uniaxial Bias Extension Test

The Uniaxial Bias Extension (UBE) test is a common experimental technique used to characterize the shear compliance of engineering fabrics and prepregs [1] (see Figure 1a-c). This is usually done by measuring the axial force while pulling the UBE specimen in the bias direction and measuring the shear angle at the centre of the specimen. The shear force versus shear angle data can then be obtained using appropriate normalisation theory. In this investigation the UBE test is chosen over the picture frame test due to the latter's sensitivity to sample misalignment when testing cross-ply laminates [8].

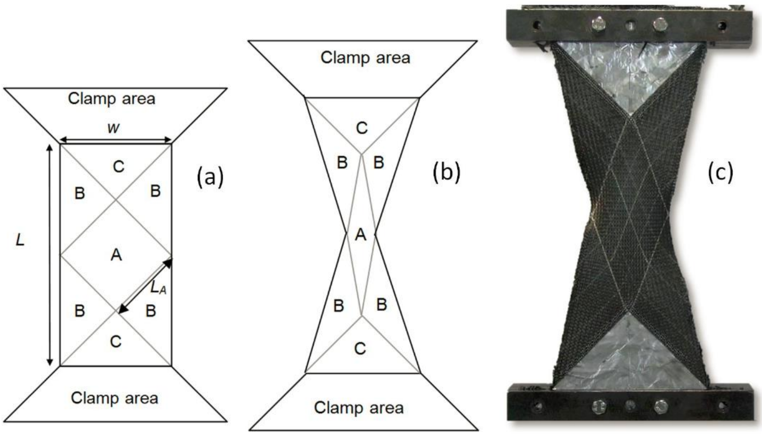

Figure 2: (from [1]). Schematic of the Uniaxial Bias Extension Test showing 3 district regions (A-C) (a) shows te sample before deformation and (b) after deformation. (c) shows an actual test specimen modified to prevent deformation in Region C.

In addition to the shear compliance both the torsional stiffness and the in-plane bending stiffness (equivalent to the 2nd order gradient energy, modelled using a generalized continuum mechanics approach to modelling [9]) can be estimated from the UBE test by measuring the specimen kinematics; the wrinkle onset angle helps identify the torsional stiffness of the sheared specimen, while the shear angle kinematics help identify the in-plane bending stiffness, both via inverse modelling using a suitably comprehensive finite element model [1]. In order to identify the in-plane bending stiffness from the shear kinematics it is important to mitigate intraply slip [10] in Region C of the UBE test specimen (see Figure 2a and 2b) and to carefully control the boundary condition along the edge of Region C. Having a well-defined boundary condition means that the test can later be accurately modelled using finite element simulations in order to estimate mechanical properties (the in-plane bending and torsional stiffnesses) using an inverse modelling approach [1]. For engineering fabrics this can be achieved simply by bonding aluminium foil over Region C (see Figure 2). However, at high temperatures, foil bonded to the molten thermoplastic composite would lose adhesion as the polymeric phase melts and the composite behaves as a fibre reinforced fluid, making prevention of intraply slip in Region C more challenging.

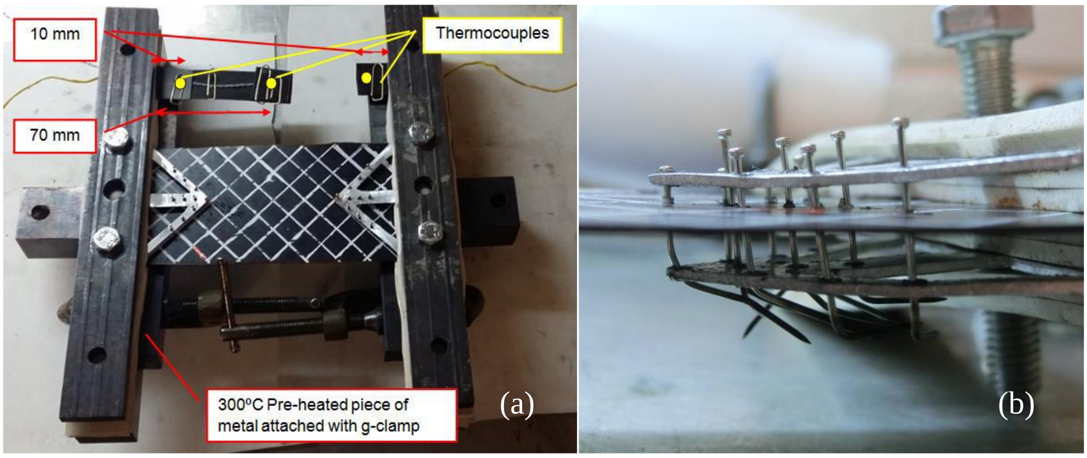

Figure 3. (a) Test set-up before going into the environmental chamber. (b) side view of the arrangement of pinning plates, needles and silicon rubber spacer layers (prior to loading the arrangment in the clamps)

The setup in this investigation involves the use of aluminum 'pinning plates' (see Figure 3), that allow pins or needles to pierce the specimen along the boundary between Regions B and C (see Figure 2). Instead of an encastre boundary condition (see Figure 2c) a well-defined pinned condition is thus imposed along the boundary. The needles help to mitigate intra-ply slip in Region C. Ten needles were pierced through the specimen (with the help of a soldering iron) and passed through holes in the pinning plates (two at either end of the specimen, see Figure 3). The process of piercing the specimen with the needles takes around 30 minutes. Silcone rubber spacer layers were used to: (i) create an air gap between the specimen and the pinning plates to allow convection heating of the test specimen under the plates and (ii) to reduce the temperature-coupling between the specimen and the bulky metal the clamps – the latter were preheated to ensure the specimen under the clamps were close to, but lower than the melt temperature of the specimen during testing. In future electrical heating of the clamps would be desirable. If the specimen melts under the clamps during testing then the pressure of the clamps creates a squeeze flow [11], eventually creating significant and unwanted deformation in Region C of the specimen as well as slippage of the specimen from the clamps during the test. The oven was also pre-heated for several hours prior to testing at the set temperature. By doing so, undesirable temperature gradients between the top and bottom of the environmental chamber were minimized (see Section 4.2.1 for further information).

The dimensions chosen for the unclamped part of the test specimen were 140x70mm, i.e. $\lambda=L_o/w_o=2$. A grid was drawn along the direction of the fibres, and later used to measure the deformation kinematics during the test. Three thermocouples were used. Two were placed 10 mm away from the upper and the lower clamps, and the third was place at the same height as the mid-secion of the specimen. The thermocouples were not placed on the sample itself but sandwiched between two vertical strips of carbon fibre-nylon material (see Figure 3a). The air temperature inside the oven was shown by the display of the environmental chamber. All samples were tested using a Zwick Z2 tensile test machine fitted with a 2kN load cell using a displacement rate of 200 mm/min. A tripod and a Casio EX-ZR 700 camera were used to record the experiments for subsequent shear angle analysis.

4. Results

4.1 Cantilever Bending Test Results

Initial tests were conducted to verify the accuracy of the temperature control inside the oven. The Zwick environmental chamber was pre-heated to 210ºC, and maintained at this temperature until the samples reached a stable temperature. The temperature was then increased by 1ºC per minute up to 220ºC, then at 0.5ºC per minute above this temperature. The time versus temperature profile of the two thermocouples in the temperature probe together with that of the oven thermocouple temperature are shown in Figure 4 and show reasonable agreement.

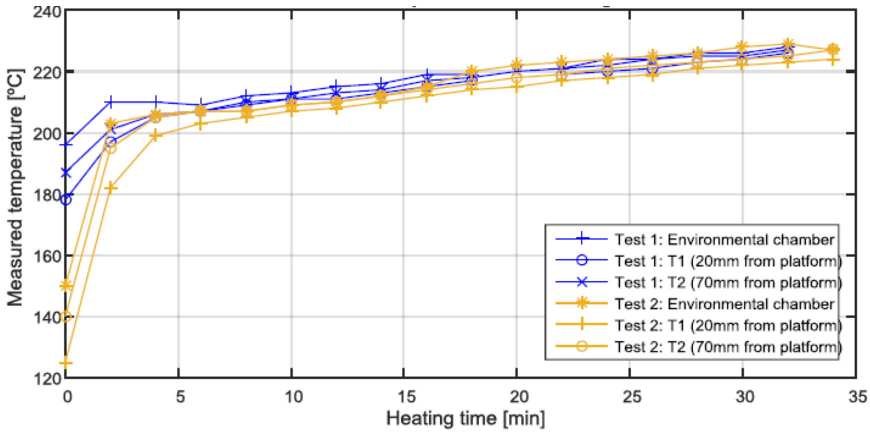

Figure 4. Temperature versus time in the environmental chamber measured using the two temperature probes and the environmental chamber.

Preliminary results from this test are shown in Figure 5. As anticipated, the results suggest a rapid drop in out-of-plane bending stiffness with increasing temperature, the drop is particularly abrupt at about 224ºC; close to the melt temperature of the nylon matrix (223.4ºC according to DSC testing – see Section 2). Just above the melt temperature the specimen has a flexural modulus of ~ 0.002Nm which is about 10x higher than that of a twill weave carbon fabric (areal density ~200gm^{-2}) (see [1]). The value rapidly drops towards that of the carbon fabric as the temperature continues to increase above the melt temperature. In future this test method could be significantly improved by recording the height of the specimens versus temperature throughout the whole test rather than at just the point when the specimens touch the ramp. This can be achieved using Eq (19) from [5] which provides accurate estimates of the flexural modulus of the specimen at any height. Such a modification would permit hundreds of data points to be collected per test rather than just four. Note that this test takes no account of the rate-dependence of the bending modulus, indeed, it is more accurate to discuss the viscosity rather than the modulus of the specimen. However, measurement of an apparent modulus is considered to be good enough (and convenient) for first order accuracy in forming simulations.

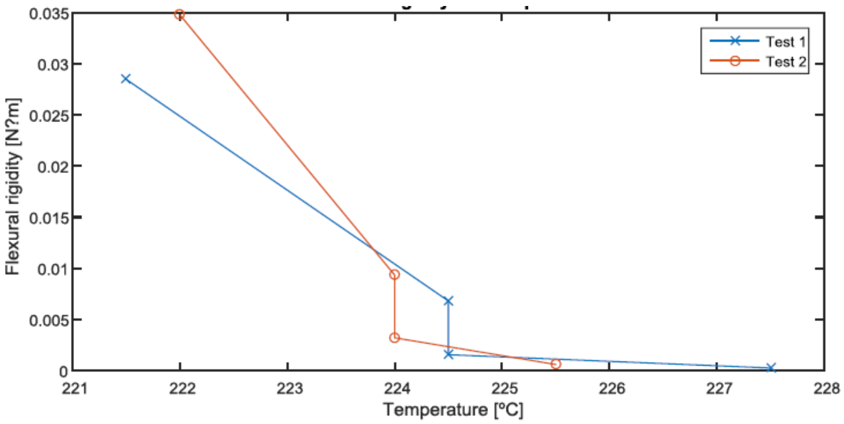

Figure 5. Flexural rigidity per unit width versus temperature.

4.2 Uniaxial Bias Extension Test Results

Two types of result were measured during the UBE tests; the axial force versus shear angle and the shear angle kinematics.

4.2.1 Temperature Measurements

Prior to conducting mechanical characterization using the UBE test, experiments were conducted to examine the temperature distribution in the oven. The temperature of the lower thermocouple was found to be significantly different from the upper thermocouple, even after waiting 25 minutes after closing the pre-heated oven door. At a set temperature of 230°C the upper, middle and environmental chamber temperatures measured ~230 +/1°C, in contrast the lower thermocouple measured a temperature of ~221 +/1°C, approximately 9°C lower than the upper end of the specimen. This temperature gradient is a fundamental problem when conducting UBE tests in a vertical convection oven, especially near the melt temperature of the matrix polymer. Potentially the upper and lower portions of the specimen could exist above and below the polymer's melt temperature. The sensitivity of the polymer rheology around this temperature (see Figure 5), introduces a good deal of uncertainty when conducting UBE tests in this way. More accurate experiments would benefit from implementation of a horizontal oven. Another issue noted during high temperature testing (~240°C) was distortion of the specimen prior to the start of the test; presuambly this effect is due to relaxation of residual stresses in the polymeric phase of the composite. It is unclear whether such deformation adversely affects the shear test results. With these issues in mind, results from the current set-up are presented below.

4.2.2 Normalised Axial Force Versus Shear Angle and Shear Angle Kinematics

The axial force was normalized using the normalization method derived for rate dependent materials (using a Newtonian fluid assumption – an approximation for this non-Newtonian material) [12] and plotted against the measured shear angle at the centre of the specimen (see Figure 6a). Tests 2-8 used a set temperature of 230°C, and tests 9 & 10 used a set temperature of 240°C. However, the temperature recorded by the lower thermocouple was typically around 10 to 20°C lower than the set temperature (see Section 4.2.1) resulting in considerable uncertaintly in the actual temperature of the samples. The results suggest that the specimens were just below the melt temperature for tests 2-8 and just above the melt temperature for test 9 & 10, resulting in about a ten-fold decrease in the shear stiffness of the specimen as the nylon matrix passed through its melt temperature of 223.4°C. A similar reduction in bending stiffness was seen in Figure 5.

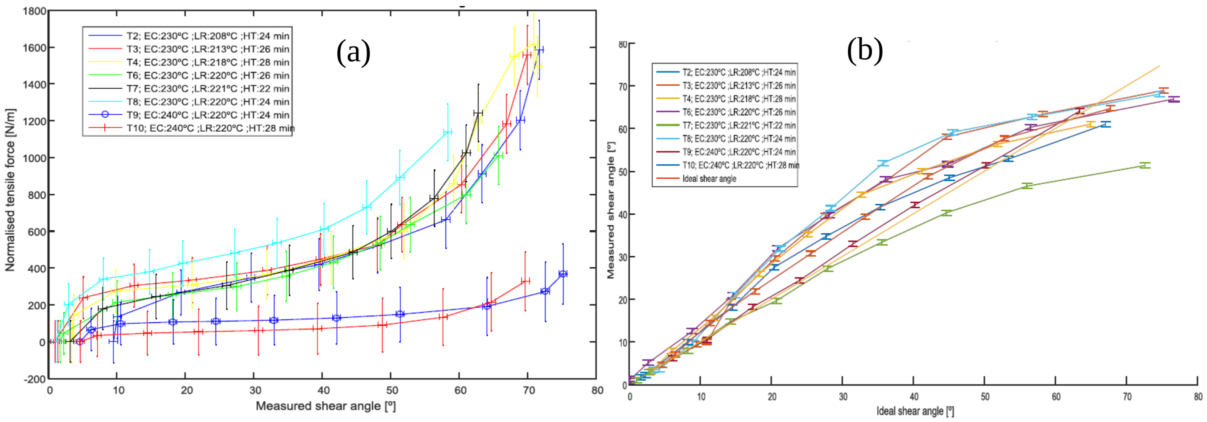

Figure 6. (a) Normalised axial force versus shear angle. EC is temperature of environmental chamber, LR is temperature of lower region of specimen and HT is the heating time before starting the test.(b) Measured versus ideal shear angle kinematics.

Regarding the sample's shear kinematics, as expected, the measured shear angle tends to lie above the ideal shear angle prediction (see Figure 6b). In [1], this effect was attributed to the in-plane bending stiffness of the specimen and importantly, allowed estimates of the fabric's in-plane bending stiffness to be made via inverse modelling. Similar measurements were anticipated in the current experiments. However, in these high temperature experiments the cooling effect of the clamps was found to create a ~5mm wide 'cool' zone. This altered the shear kinematics across the entire specimen; effectively reducing the side length of the central region of the specimen (see Figure 7a). The adverse effect of the frozen region will be eliminated in future by redesigning the pinning plates with an extended vertical section (see Figure 7b).

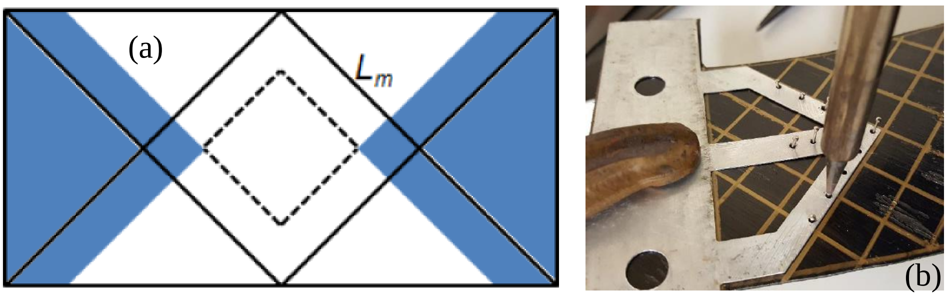

Figure 7. (a) The 'cold' zone nect to the clamps has a significant influence on the kinematics across the entire specimen. (b) An improved design for pinning plate. The extended vertical section eliminates the adverse effect of the cool zone next to the clamps.

5. Conclusions

Forming tests on carbon-nylon preconsolidated cross-ply laminates have been conducted. Variations on existing test methods have been introduced to accommodate the unique nature of thermoplastic composites. While preliminary results are encouraging, the investigation has revealed numerous issues related to temperature distributions occurring in both the environmental chamber and across the samples themselves that adversely affect the recorded results. Several suggestions to improve the test methods have been proposed and will form the basis of continued work in this direction.

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