Intraply Shear Characterization of a Fiber Reinforced Thermoplastic Composite

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ABSTRACT: Acquiring complex curvature shapes during thermoforming of thermoplastic composites involves flow phenomena such as interply slip, intraply shear and squeeze flow. To date the intraply shear mechanism is the least well understood. Approaches have been made to determine both longitudinal and transverse steady intraply shear viscosities of molten unidirectional continuous fiber-reinforced composites in the past but with conflicting results. The present study concentrated on experimentally establishing the intraply shear behavior of carbon fiber reinforced polyetheretherketone (APC-2). To induce true intraply shear in a composite laminate APC-2 panels, were cut and ground into long, square-faced blocks, which were then rotated through 90°. A series of experiments was carried out in which a central plate was drawn out at constant velocity using a custom-built pull-out shear apparatus. Values of longitudinal and transverse shear viscosities were determined for shear rates between 0.025 and 0.02 s^{-1}. The viscosity was highly shear-rate dependent, and was best modeled using a 'power-law'. At a rate of $0.01 s^{-1}$ the longitudinal viscosity was in the 1 MPa.s range, and approximately 2.5 times the transverse value. These values are several orders of magnitude greater than those determined using more traditional means, such as torsional rheometry, but agree with results obtained using a picture frame test method.

KEYWORDS: Thermoplastic, unidirectional composite, intraply shear, viscosity.

INTRODUCTION

Many modern composites consist of a highly viscous resin or matrix reinforced by long, continuous strands of virtually inextensible fibers. These composites are often supplied in the form of tape-like sheets referred to as preimpregnated tapes or 'prepregs' in which the fibers have been highly collimated and aligned in the matrix in order to maximise their reinforcing potential. This alignment of the fibers together with their inextensibility makes the properties of the bulk composite highly directional or strongly anisotropic, with preferred directions along and transverse to the fibers. Forming complex shapes from sheets of preimpregnated thermoplastic composite, in their melt state, may be considered as a difficult process when compared to the forming of unreinforced thermoplastic sheet, or indeed, sheet metal [1]. For a thermoplastic composite such as APC-2, comprising approximately 60% by volume carbon fibers embedded in a PEEK matrix, however, circumstances are complicated by the high degree of anisotropy associated with this composite, and successful part manufacture is not as straightforward [2]. Deformation of the composite is constrained by the fiber reinforcement but facilitated by the matrix, which acts as a form of lubricant in between individual fibers and between plies of different orientation, decreasing the internal friction during the motion of fiber bundles in a laminate, thus lowering the forming forces [3-6].

To model the flow and deformation of strongly anisotropic materials in a commercial forming process, mathematicians (Spencer [7], Rogers [8]) have developed macroscopic theories in which constitutive equations are used to relate the state of stress at any point and time in the material to the strain or rate-of-strain. The simplest form of these equations, which assumes the case of a highly anisotropic viscous liquid reinforced by a single family of inextensible fibers, consists of two important rheological parameters, these being the longitudinal viscosity η_L, which is associated with shearing along the direction of the fibers, and the transverse viscosity η_T, which is associated with shearing perpendicular to the fiber direction. Clearly, if models of this type are to be successfully employed in the simulation of composite forming, then an accurate experimental database for η_L and η_T is vital.

In this paper a custom-built experimental apparatus designed to provide experimental values for the longitudinal and transverse viscosities of a thermoplastic composite system undergoing steady-shear deformation is introduced. The operation of the instrument is based on the principle of drawing a thin, flat plate from a composite specimen using a constant pull-out velocity and measuring the pull-out force as a function of the plate's displacement from its initial position. By aligning the fibers parallel or normal to the pull-out direction the anisotropic behavior of the test specimen can be characterized, and through appropriate mathematical analysis, the readings of pull-out force can be converted into corresponding values of longitudinal and transverse shear viscosity.

DEFROMATION BEHAVIOR

For unidirectional thermoplastic composites, four principal mechanisms have been identified and observed to occur when manufacturing parts from preimpregnated tape [3]. These are (i) resin percolation through the fibers (ii) transverse flow of the fibers (iii) interply slip of the individual plies across one another and (iv) intraply shearing of the individual fibers within each ply. To realise acceptable wrinkle free complex or double curvature parts some form of shear mechanism must be present to allow for the fiber re-orientation. During the shaping stage, fibers must be displaced relative to each other within individual plies, due to the shear stresses that are induced. As each fiber within a ply is separated from its neighbours by an amount of viscous polymer this is the material that is sheared during deformation and the mechanism of accommodating inter-fiber shear is called intraply shear. Two types of intraply shear can occur within a ply, namely axial and transverse as illustrated in Figure 2.1. Axial intraply shear must be present whenever a double curvature shape is to be formed successfully and wherever the relative orientation between neighbouring plies changes.

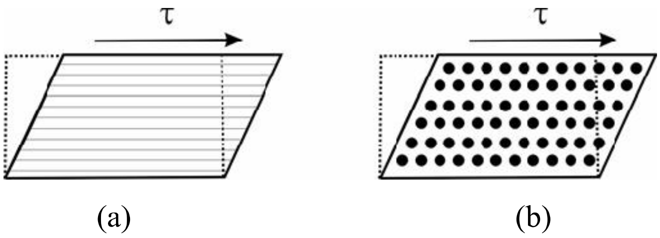


Figure 2.1: Two modes of intraply shear in composite materials: (a) axial or longitudinal and (b) transverse.

To date it appears that relatively few experimental studies into the characterization of the intraply shear mode of deformation in commercial composite systems have been made. More effort has concentrated on trying to characterize fully the interply slip of these advanced composites. A number of successful research studies have been carried out on the 'model' composite systems consisting of long, rigid and virtually inextensible fibers embedded in a liquid, Newtonian matrix, at room temperature whereby the intraply shear behavior was much more readily achievable throughout their composite structure than the models' commercial counterparts [9-12].

APPARATUS

An illustration of the complete experimental apparatus, with a sample to be tested *in situ*, is given in Figure 3.1, highlighting the three principal influences on the composite material during an experiment, namely the normal pressure over the surface area, the application of heat and the lateral straining action.

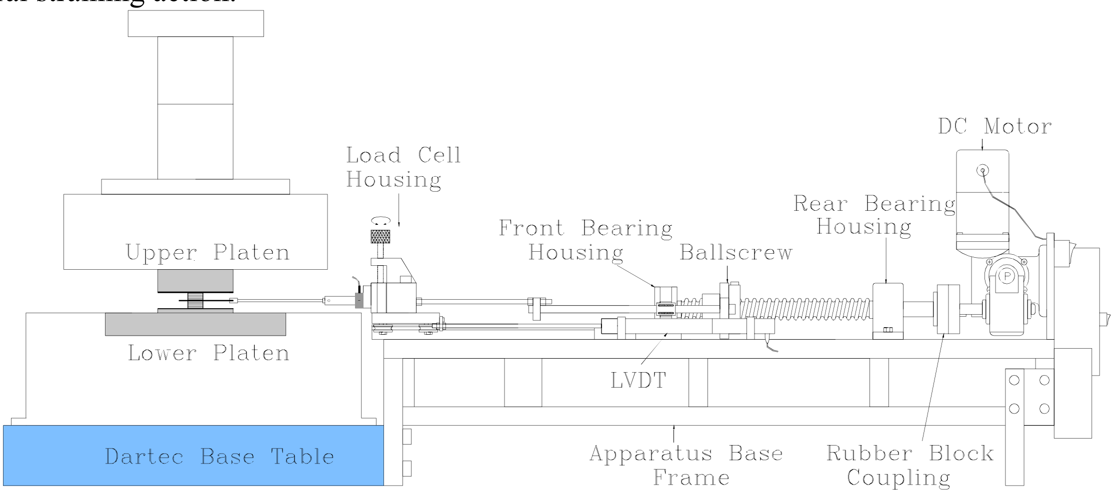


Figure 3.1: Complete shear apparatus, indicating main parts of the rig.

The vertical gap height and normal pressure was controlled using a commercially available hydraulic straining unit, Dartec 100 kN RK, the heating was effected using cartridge heaters embedded in steel platens. The shearing action was achieved and monitored using the custom built experimental rig.

The drive mechanism of the shear rig used to generate the desired (constant) pull-out velocity consisted of a motor driven ball-screw. This was attached through a connecting shaft to a main housing, which sat on a sliding trolley. The trolley was fitted with four contoured wheels that ran on the mating rail or track. The main housing supported the load cell, a 10 N maximum capacity Model 31 from Cooper Instruments (U.S.A.) Ltd., with a linearity rating of ±0.15% F.S. The load cell had two male threads along its main axis. One of these threads screwed into a T-slide on the main housing, thereby facilitating a change in the load cell height (for centralisation) by rotating a threaded shaft fitted to the slide. The other end of the load cell, called the active stud, was screwed into a rod of machineable glass ceramic, commercially known as MACOR, to minimise the conduction of heat from the composite to the load cell. Next, was a stainless steel rod to enable correct angular alignment of the pull-out plate, i.e. horizontal. A titanium tube, of outside diameter 3 mm and wall thickness 0.5 mm, was screwed directly to this steel rod. At the other end, a steel clip joined the titanium extension tube to the titanium pull-out plate. A ±25 mm linear variable differential transformer (LVDT) supplied by RDP Electronics (U.K.) Ltd., model number DCT1000C (±0.5% linearity), was employed to measure the pull-out plate displacement. The LVDT armature was connected to the rear of the housing trolley.

SPECIMEN PREPARATION

Early shear tests were performed on composite blocks consolidated under higher than recommended pressures in order to achieve a thick 'single' ply of unidirectional material, i.e. to overcome the formation of resin rich interlayers. However, subsequent shear tests resulted in interply slip of the plies. In order to encourage intraply shear in the composite and preclude the effect of the resin layers on the deformation process a novel approach would have to be adopted in order to induce the deformation sought. The only practical solution to this problem was to align blocks of composite such that the individual planes or layers of the plies were perpendicular to the line of action of the pull-out force as depicted in Figure 4.1.

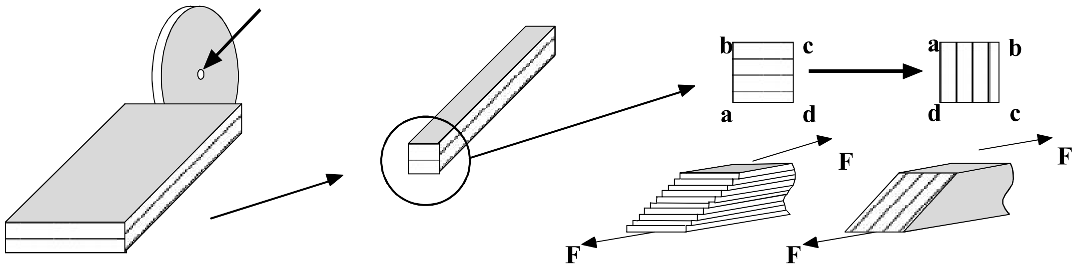


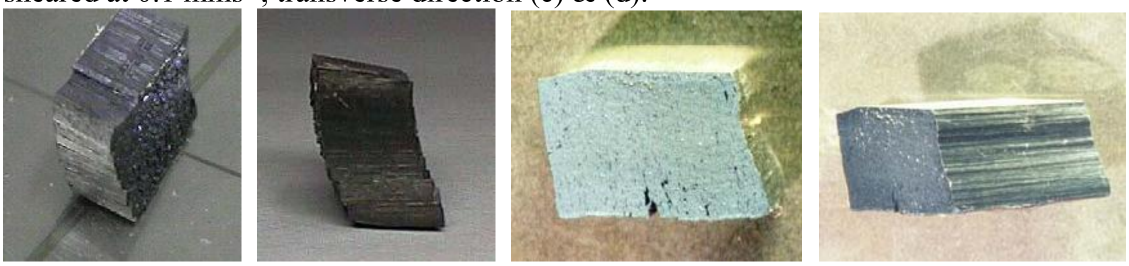
Figure 4.1: Schematic of the composite machining process and desired deformation.

Panels comprising 80 plies of unidirectional APC-2 were manufactured in an autoclave. The consolidated panels were then machined using a diamond tipped 'slitting' saw to have a constant height of 10 mm of varying lengths.

EXPERIMENTAL PROCEDURE

One half of the assembly of blocks to be tested was placed on the lower platen according to prescribed datum lines. A thermocouple probe tip was placed in close proximity to the side of the composite in order to monitor the temperature as close as possible to the assembled blocks, but without disturbing them. Prior commissioning tests were carried out to check the temperature distribution throughout the upper and lower composite block assemblies by inserting thermocouple probes into predrilled holes in the specimens and monitoring time the time taken to achieve near isothermal conditions within the material with a set point temperature of 380°C. The central titanium pull-out plate was, already attached to the extension arm, was simultaneously laid on top of the composite and into the ceramic attachment affixed to the load cell. The upper half of the composite assembly was then placed on the titanium plate directly above the block, or blocks, below. Finally, the upper platen was lowered into position, such that it made slight contact pressure with the whole assembly. When the composite attained the required temperature the motor of the shear rig was activated and the load - deflection readings were logged. The predominant pull-out velocity was set at 0.1 mms^{-1}. The effect of normal pressure on the intraply shear behavior was also limited due to the size of the assembled blocks used in the test programme, i.e. their relatively small surface area, and correspondingly, the minimum recordable normal force on the composite. A complete series of experiments was carried out on blocks of composite, with increasing block, hence fiber, length. All of these composite blocks sheared through the thickness in a truly intraply shear mode of deformation up to and including blocks of 10 mm length. With the sample length greater than 10 mm, the increased fiber length prevented the composite from shearing. A comprehensive test series was also performed in which the pull-out velocity was varied to investigate a shear rate effect on the shear behavior.

A similar programme of experimentation was carried out with the fibers aligned in a direction transverse to the pull-out force. It became evident that during the transverse shear tests that the composite tended to deform more readily than when aligned in the longitudinal direction, up to a certain extent of shear. Thereafter, the pull-out plate began to slip relative to the composite blocks, and the tests degenerated to simple friction tests. Again, a comprehensive test series was also performed in which the pull-out velocity was varied to investigate a shear rate effect on the shear behavior. Two images are given in Figure 5.1, (a) and (b), taken of a 6 mm sample sheared at 0.2 mms^{-1}, longitudinal direction, and two photographs of a 20 mm in length composite block sheared at 0.1 mms^{-1}, transverse direction (c) & (d).



(a) (b) (c) (d)

Figure 5.1: Images of samples of APC-2 post shear; (a) & (b) longitudinal direction, (c) & (d) transverse direction.

RESULTS

The full range of viscosity values extracted from the results of the test programme in the longitudinal direction is given in Figure 6.1 (a). Three distinct values of shear rate were applied during the testing phase. These corresponded to pull-out velocities of 0.025, 0.1 and 0.2 mms^{-1}. Pull-out velocities above 0.2 mms^{-1} were tried but did not result in a successful test, i.e. the pull-out plate slipped thereby instigating zero degree of intraply shear within the composite blocks. The titles in the legend of this graph refer to the block or fiber length. The viscosity values were calculated by using the average force value over the range of displacement from 1.25 to 2.25 mm of shear, when the fibers were uniformly distributed and the structure of the material was least disturbed. There exists a shear-thinning effect in the shape of the viscosity curve. There also existed a fiber length effect on the viscosity values obtained, i.e. for increasing fiber lengths the viscosity increased for all values of shear rate.

The power-law model can be used when the shear-rates being studied are not negligibly small and can use a small number of experimentally determined values to provide an empirical method for determining viscosity data. Its basis lies in the logarithmic plot of shear stress versus shear rate. The apparent viscosity, or shear–dependent viscosity, is not absolute but a function of the shear rate,

$$\eta = \frac{\tau}{\dot{\gamma}} \qquad (6.1)$$

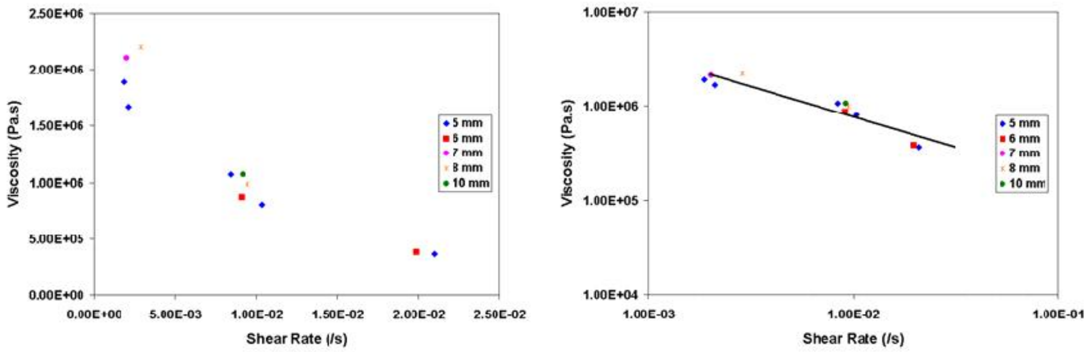
A linear region of the logarithmic plot suggests the following relationship,

$$\tau = K(\dot{\gamma})^{n} \qquad (6.2)$$

then, an expression for the viscosity, η, reduces to:

$$\eta = K(\dot{\gamma})^{n-1} \qquad (6.3)$$

n is called the power-law index and K is called the ‘consistency’. The plot of shear viscosity versus shear rate, Figure 6.1 (b), indicates that a power-law model could be applied to the composite deformation behavior, the form of which is given in equation 6.4.

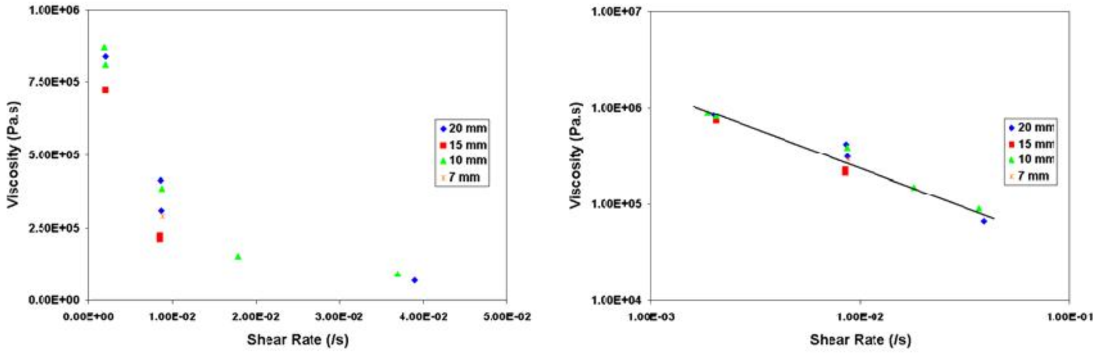


(a) (b)

Figure 6.1: (a) Viscosity values for APC-2, for increasing values of shear rate – longitudinal direction; (b) Power-law region indicated from the tests.

$\eta_L = 39637(\dot{\gamma})^{-0.646}$ (6.4)

A similar analysis was applied to the force-displacement data collated from the tests performed in the transverse direction. The viscosity – shear rate dependence plot is given in Figure 6.2 (a).



(a) (b)

Figure 6.2: (a) Viscosity values for APC-2, for increasing values of shear rate – transverse direction; (b) Power-law region indicated from the tests.

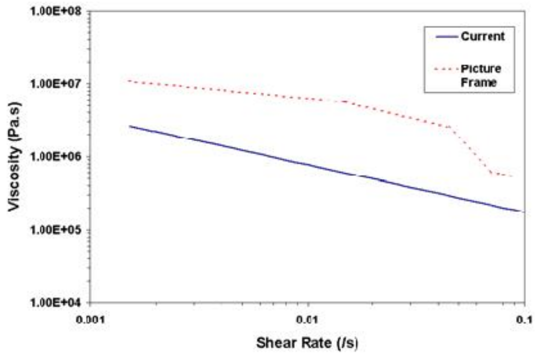
The magnitudes of the viscosity values for the composite, with the fibers aligned in the transverse direction, turned out to be 40 % (approx.) of those when the blocks were aligned in the longitudinal direction. Again, shear thinning, the viscosity fall-off with increasing shear rate, was evident for the material during this phase of experimentation. No discernible fiber length effect was evident in the viscosity values collated. A power-law model was also applied to the results with the following equation determined from the graph in Figure 6.2 (b).

$\eta_T = 6720.1(\dot{\gamma})^{-0.7798}$ (6.5)

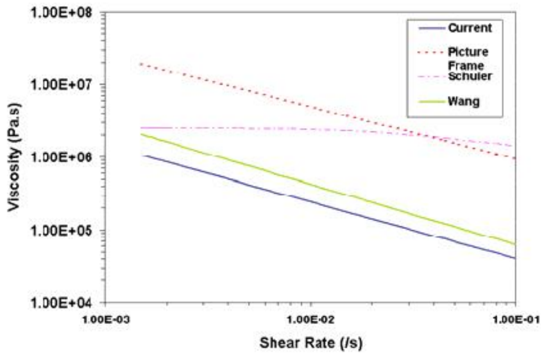
DISCUSSION

The values collected for the intraply shearing viscosities for the thermoplastic composite presented in this study far exceed those arrived at by oscillatory and torsional shear testing [13-15]. Values for steady flow and dynamic viscosities for both longitudinal and transverse shear varied between 3,200 Pa.s and 7,400 Pa.s. The longitudinal viscosities were on average higher by a factor of 1.3 than the transverse values. The findings from this research are in the MPas. range, three orders of magnitude in difference. A significant factor that may have influenced the torsional rheometry and oscillatory shear results would be the formation of resin-rich layers between the surfaces of the composite and the platens of the test rig. When the oscillatory deformation was applied to the samples, a large proportion of the resulting shear may occur purely within the resin-rich layers, and only a small amount being transmitted through to the bulk of the composite.

However, the resultant values arrived at through the picture-frame technique [16-18] and the transverse squeeze flow experiments [19,20] are comparable. Figure 7.1 (a) compares the power-law model proposed by the current findings and that presented by McGuinness et al [18] from the experiments done using the picture-frame method, longitudinal direction.



(a)



(b)

Figure 7.1: (a) Comparison of the power-law model from the current study and that proposed by McGuinness [18] for APC-2 in the longitudinal direction; (b) Comparison of the power-law model from the current study and the models proposed by McGuinness [18], Schuler [19], and Wang [20] for APC-2 in the transverse direction.

The power-law model proposed by McGuinness is not constant across the range of shear rates of interest at a temperature of 380°C. Thus the behavior could be described by using two power-law models that give viscosity values over particular shear-rate ranges, resulting in the distorted shape of the plot in Figure 7.1(a). In the transverse direction, the transverse flow experiments, in addition to the picture-frame work can now be compared with the current research. The testing temperature used throughout the range of experiments was between 370°C and 390°C. The models proposed by each researcher to describe the transverse shearing flow behavior are plotted in Figure 7.1(b). Again, McGuinness used a power-law to model the viscosity of the composite in the transverse direction. In this instance, one model described the behavior over the full range of shear rates used. Schuler and Advani [19] employed a Carreau model for the transverse shear viscosity of APC-2 at the testing temperature of 370°C. Wang and Gutowski [20] used a power-law model with parameters n = 0.17 and K = 9300 $Pa.s^n$. As with the longitudinal case, the transverse shear values from the picture-frame experiments far exceed those from the current investigation. The larger sample size (200 mm^2) could have cased the higher viscosities due to some degree of fiber entanglement, the probability of which would be higher with an increase in the sample dimensions. A limitation of the results or viscosity values arrived at using the picture-frame technique is that cross-ply laminates (32 ply $[0_4/90_4]_{2S}$) were used as the test samples and the force response from these tests were approximately 25% higher than the unidirectional tests, although it was not possible to deduce the viscosity values from these tests due to the complicating factor of the surrounding compliant diaphragms. Therefore, the unidirectional values of shear viscosity could be far less than 75% of the cross-ply values. Moreover, by the very nature of the rhombus deformation induced in the sample, there was a through-thickness effect also. Schuler has applied a Carreau model to describe the transverse flow behavior of APC-2 at 370°C. A 10°C increase in temperature would reduce the viscosity by approximately 17% [21]. This would account for some of the difference in the magnitude of values from this transverse flow experiment and the current study. However, over the range of shear-rates used during the current study, the viscosity values are in the Newtonian plateau in Schuler's proposed Carreau model. Only, towards the higher rates of shear, does his viscosity dependence change over to the power-law behavior. One difficulty in interpreting squeeze flow experiments is the non-uniform nature of the flow field of the samples, which can induce large variations in shear rates throughout the material, and thus affect the overall shear rate dependence. In a similar type of experimental technique, Wang [20] applied a power-law model to describe the transverse flow of APC-2. The results compare rather favourably with current results, with the viscosity values of the current study approximately half the transverse flow values.

CONCLUSIONS

A custom built shear apparatus has been developed and applied to investigate the steady-shear deformation of a commercial high fiber volume fraction carbon fiber reinforced thermoplastic composite material. The rheological behavior of the composite has been investigated for different pull-out shear rates both along and transverse to the principal fiber directions. A novel specimen block design was introduced. Assemblies of 5 to 20 mm long blocks, 10 mm square, were tested in the rig. When values of viscosity for the composite were plotted against the shear rate, shear-thinning response or behavior was evident. The viscosity measured from the tests was approximately 2 MPa.s at the lowest shear rate of 2.5 x 10^{-3} s^{-1} in the axial direction. This value reduced by a factor of ten approximately, with a tenfold increase in shear rate. The transverse viscosities were consistently less than the longitudinal viscosities for all shear rates. The highest viscosity measured was approximately 0.9 MPa.s at 2 x 10^{-3} s^{-1}. The longitudinal values of shear viscosity were approximately 2.5 times the corresponding transverse values. A power-law model was also used to describe the shear behavior in the both directions. The values of viscosity determined during this current study were far greater than those determined using oscillatory shearing techniques. A picture-frame experiment, however delivered results of a similar magnitude to values found in this study. The results were also compared to two transverse shear/squeeze flow investigations. The results for one of these studies were modeled using a Carreau expression, but were significantly different from the current results, modeled using a power-law, although still in the MPa.s range. Another investigation, this time with the results modeled using a power-law, were quite comparable with the current results.

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