Effect of Extrusion Parameters and Nanofillers on Mechanical Properties of PPSU Tufting Yarns

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Abstract. Yarn diameters ranging from 160µm to 720µm of unfilled PPSU and PPSU nanocomposites with 1 wt.% of either carbon nanotubes (CNT) or carbon nanofibers (CNF) were prepared using a twin screw compounder. The tensile properties of these yarns generally improve with the addition of CNT or CNF at higher values of screw speed-to-haul off ratio. This effect is correlated with the yarn draw down ratio and attributed to nanofiller orientation and crystallinity induced in thermoplastic matrix. The fibres have as much as a 23% increase in Ultimate Tensile Strength (UTS) for the same parameter set when loaded with CNT, while the greatest increase observed for larger particle size fillers CNF is just 3.77%. Depending on filler and processing parameters set, yarns varied in UTS from 24.84MPa to 206.23MPa for unfilled PPSU and PPSU-CNT, respectively.

INTRODUCTION

Carbon fibre (CF) reinforced polymer composites offer significant improvement of material strength-to-weight ratio, when compared with materials traditionally used in aerospace and automotive industries.[1] However, delamination of composite structures is a considerable drawback that can be potentially solved by a through-plane stitching of CF fabrics before a resin impregnation step. Such a simple method of improving resistance to delamination requires identification of a suitable tufting yarn material and optimal yarn characteristics. Tufting arrests the displacement caused by crack opening displacement in mode I loading and crack sliding in mode II. The tufting thread functions as a pin, retaining the adjacent laminate regions following the transit of a delamination crack front through said region.[2] Preliminary testing has shown that polyphenylsulfone (PPSU) is a potential candidate for such a tufting yarn due to its excellent mechanical and physical properties which can be further modified by the control of processing conditions and the addition of nanofillers. In particular CNTs have shown great success with increasing the mechanical properties of polymers even at extremely low percentage volumes.[3]

EXPERIMENTAL

Materials

R-5500-NT grade Polyphenylsulfone (PPSU) from Solvay Advanced Polymers was used for all extrusions.

PR-24 carbon nanofiber (CNF) supplied by Pyrograf as heat treated materials with increased inherent conductivity had average fiber diameter 100nm and length ranging between 50-200um (aspect ratio 500-2000). The CNFs were homogeneously mixed in a charge bag with milled PPSU in order to give 1.0wt.% volume of CNF to PPSU.

The NC7000 multi-walled carbon nanotubes (CNT) supplied by Nancy as industrial grade (95% pure) had average fiber diameter 9.5nm and average length 1.5um (aspect ratio c.a. 160). The Multi-walled carbon nanotubes (MWCNTs) were homogeneously mixed in a charge bag with milled PPSU in order to give 1.0wt.% volume of MWCNTs to PPSU.

Polymer Preparation

PPSU pellets of ≈3mm were placed into a vessel with liquid nitrogen for 5 minutes in order to make them more brittle before being fed into a Fritsch Pulverisette 14 rotor mill, rotating at 15,000rpm.

Prior to the mixing with nanomaterials/extrusions the milled PPSU was dried in an over at 150°C for a period of 2 hours to expel any moisture within the material.

Extrusion

A Thermos Scientific HAAKE Rheomex PTW 16 OS twin-screw extruder combined with a Thermoscientific Type L-002-0061 pelletizer, modified to be used asa haul off unit, were used for all of the PPSU extrusions. The modified pelletizer was used as a continuous rate haul-off system. A ChargePoint PharmaSafe Powder Transfer Contanement Valve system was used for the extrusion of the PPSU nanocomposite yarns. The desired concentration of nanomaterials and milled PPSU were mixed in a Charge Bag. This Charge Bag is subsequently attached to the Pharma Safe Charge Point butterfly valve and both then are attached to the extruder feeding mechanism.

The extrusion barrels temperature profile began at 330°, in the zone closest to the hopper, and gradually heating up to 390°C before gradually cooling to 350°C at the die.

Optical Microscopy

A SZ-PT Olympus optical microscope was used to examine the shape and surface topography of the particles before and after milling. Image-J software was then used to take average sizing measurements from each pellet/particle

Scanning Electron Microscopy

In this study a JEOL JSM-IT100 InTouchScope SEM™ was used for the SEM imaging. The fibre samples were cut into sections of 2cm and these placed into an Emitech K500x sputter coater and the vacuum was set at 8*10-2 millibars of pressure for 2 minutes and 30 seconds at 3 milliamps (mA). The fibres were then turned over and the process repeated. The coated samples were mounted on stubs and placed into the SEM. 10kV at a magnification of *45 was employed giving a clear image of the surface topography of the fibres while still allowing enough of a field of view to perform diameter analysis on the fibres. The measure function within the SEM was exercised 5 times on each fibre sample, measuring the diameter at 500µm intervals.

Tensile Testing

An Instron 3344 tensile testing machine, equipped with a 500N load cell and Series 2710-200 Screw Action Grips, was employed throughout the fibre tensile testing. ASTM D3039/D3039M-15 “Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials” and ASTM D3217-07 “Breaking Tenacity of Manufactured Textile Fibres in Loop or Knot Configurations” were followed for the tensile testing. Paper tabs were bonded to either ends of the polymer yarns, with SP106 resin, to avoid the grips causing damaged to the fibres. The

gauge length between the grips in this case is set to 1 inch (25.4mm). The samples were tested at a constant speed of 20mm/min until failure. .

RESULTS AND DISCUSSION

PPSU pellets and milled PPSU can be seen in Figure 1 below:

(a) (b)

FIGURE 1: (a) PPSU Pellet (b) Milled PPSU

The average diameter was reduced by 90.36% (3.47mm) from 3.84mm to 0.37mm. This should aid heat transfer ensuring a homogenous melt within the extrusion process, assisting the production of a more stable extruded yarn.[4] It will also aid the mixing of PPSU and nanomaterials.

This increase in extrusion stability is evident from the minuet error bars in Figure 2 where yarn diameters at the varying processing conditions are shown. The minute error The first number, followed by an "E" denotes the screw speed, in rpm, and the second, which is followed by a "P" represents the haul-off speed in rpm.

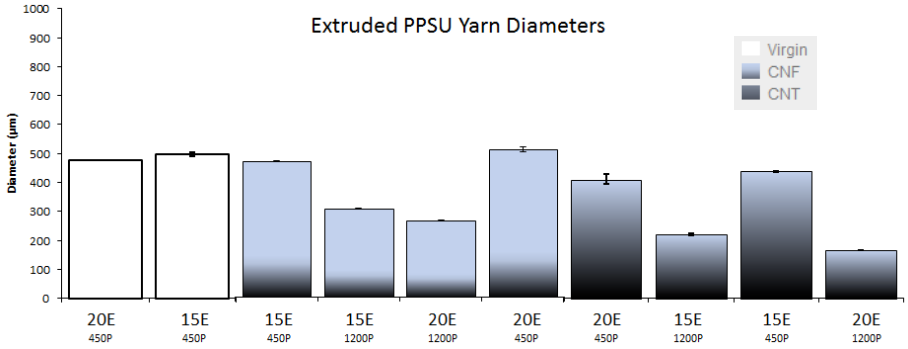

FIGURE 2: Extruded PPSU Yarn Diameters

Previous experimental work by Domenech et al .has shown structuring of extruded nanocomposites can be optimized by working under higher specific mechanical energy (SME) until a critical limit. In this case processing at a higher SME was limited by the maximum torque the machine was capable of achieving.[5]

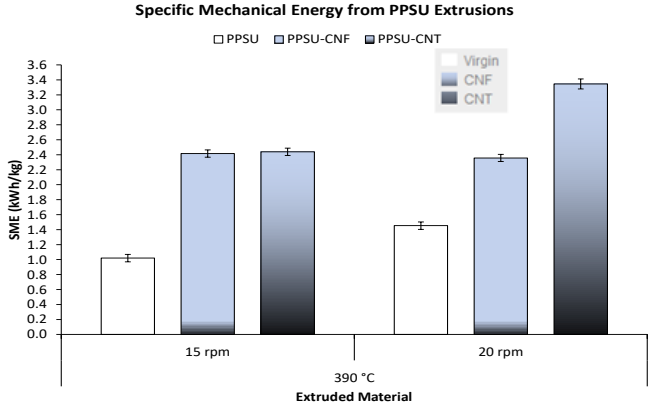

FIGURE 3: Specific Mechanical Energy from PPSU Extrusions

Figure 4 displays the ultimate tensile strength (UTS) for the PPSU and PPSU nanocomposites. It can be seen that little or no advantage in straight UTS was obtained from the edition of CNFs, the higher screw speed of 20rpm actually caused an 10.7% decrease in UTS. At the lower screw speed the CNF reinforced samples' UTS increased by an insignificant 3.77%. However, once carbon nanotubes (CNTs) are introduced as much as a 23.31% increase was observed at the same screw speed compared with the lower speeds.

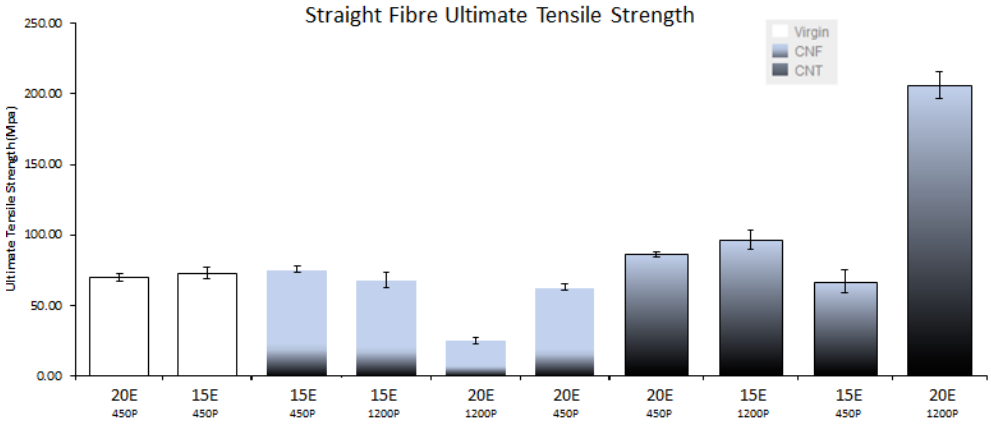

FIGURE 4: Straight Fibre Ultimate Tensile Strength

The UTS of each of the yarns calculated from the looped tensile testing are show in figure 5 below. The smooth surface topography allows the yarns to be pulled apart, when looped, without any risk of initial cutting or initial damage.

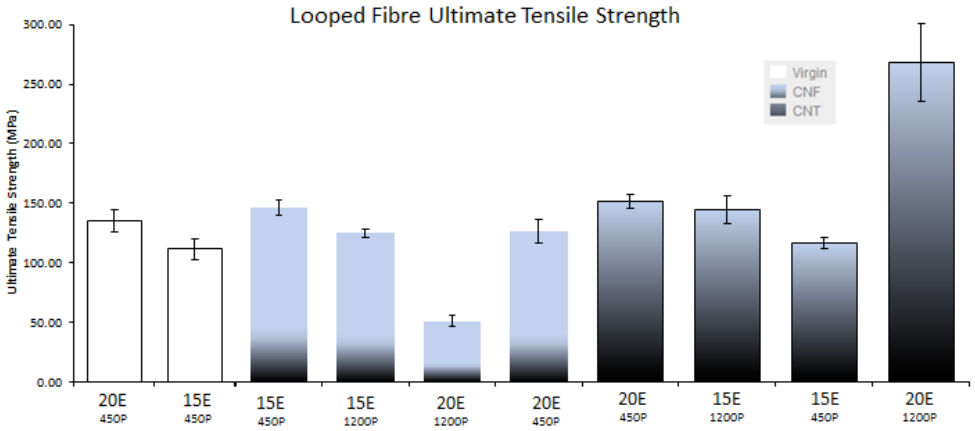

FIGURE 5: Looped Fibre Ultimate Tensile Strength

It is evident from both Figures 4 and 5 that a higher SME aids the increase of UTS of CNT reinforced PPSU, whereas lower SME increases the UTS of CNF reinforced PPSU.

In both the straight fibre and looped fibre tensile tests one particular parameter set exceeded the UTS substantially: screw speed of 20rpm and a pelletizer speed of 1200rpm. Compared to the other CNT reinforced extrusion at 20rpm the increase in haul-off resulted in a 138.5% increase in straight tensile UTS. This could be partially attributed to both the increased alignment of the nanotubes, as a result of the increased haul-off speed and as a result of the increase in SME.[5]

CONCLUSION

CNF reinforcement did not provide any significant improvements in mechanical properties and in certain situations actually resulted in a reduction in mechanical properties. CNTs did provide an increase in UTS under all tensile testing configurations. In both the straight fibre and looped fibre tensile tests one particular extrusion parameter set for the CNT reinforced PPSU exceeded the UTS substantially: screw speed of 20rpm and a pelletizer speed of 1200rpm. This could be partially attributed to both the increased alignment of the nanotubes, as a result of the increased haul-off speed and as a result of the increase in SME.

To date, some of the virgin PPSU yarns from this testing have been successfully tufted with little issue. Diameter consistency, where the diameter of the yarn varied across its length posed some minor slippages or yarn breakages. Testing with pellets and milled PPSU revealed that it was possible to get a more consistent diameter with the lower sized milled PPSU than the PPSU pellets. This could be as a result of both the removal of the air pockets within the pellets and by the smaller particles having a better heat transfer within the extruder, giving a more homogenous melt.

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