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Effect of High Fiber Content on Properties and Performance of CFRTP Composites

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Abstract: Continuously reinforced thermoplastic composites are widely used in structural applications due to their toughness, light weight, and shorter process cycle. Moreover, they provide flexibility in design and material selection. Unlike thermoset composites, continuous fiber content to maximize mechanical properties in thermoplastic composites has not been well investigated. In this paper, three thermoplastic systems are investigated to study the optimum content of continuous fiber reinforcement. These systems include carbon fiber/polyphenylene sulfide (PPS), glass fiber/PPS, and glass fiber/high-density polyethylene (HDPE). Tapes were made at several fiber contents, and samples were compression molded and tested using thermo-gravimetric analysis (TGA), differential scanning calorimetry (DSC), dynamic mechanical analysis (DMA), tensile, 3-point flexure, and short-beam shear tests. Results revealed that higher fiber content led to an increase in the glass transition and melt transition temperatures of the polymer. Some mechanical properties increased with fiber content and then began to decrease upon further addition of fibers, while other properties, such as ductility and interfacial bond strength, decreased with more reinforcement. Furthermore, the optimum fiber contents to maximize mechanical properties are different for different properties and different materials.



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Keywords: thermoplastic composites; fiber-reinforced polymers; continuous reinforcement; optimum fiber content; mechanical properties; glass transition temperature; polymer matrix composite

1. Introduction

Composite materials have seen growth in traditional metal markets where weight, corrosion, and required inspection/maintenance result in expensive parts throughout the life cycle of the part/structure. Both aerospace and marine industries have seen wide acceptance of thermosetting composite materials. The reduction in weight in such vehicles represents either added performance or increased cargo capacity [1–3]. Thermosetting composites require little capital and equipment to produce quality composites, but they have inherent challenges, including being brittle, difficult to recycle, and requiring a longer time to reach full cure at ideal temperatures [4]. While carbon and aramid fiber loading of thermosets composites may reach up to 65% by volume, the maximum loading of fiberglass rarely exceeds 55%, beyond which properties begin to decrease [5].

Thermoplastics reinforced with continuous fibers, known as continuous fiber reinforced thermoplastic (CFRTP) composites, tapes, or organo-sheets, represent an emerging material in the composites marketplace that offers high strength, high stiffness, and recyclable products that are tough and require a setting time as short as a few minutes [6–9]. The variety of available thermoplastics provides additional design flexibility for composite engineers to tailor appropriate strength, stiffness, ductility, service temperatures, and resistance to degradation for relevant design requirements [10,11]. Other benefits of thermoplastic

tapes include custom-design of fiber content, low energy storage as the pre-impregnated tape is stored at room temperature, and flexibility in shape as spools can be made to desired widths, thicknesses, and lengths for various continuous processing applications [12]. In addition, automated lay-up, preheating, and forming have enabled efficient manufacturing of high-temperature thermoplastic composite parts [13]. Simulation studies of 3D-printed CFRTTP composites showed that parts with nearly 30% carbon fiber content by volume can be produced and that the printing parameters have a direct effect on the mechanical properties of the parts [14].

Further analysis, simulation, and testing methods have been developed to account for the viscoelastic behavior of CFRTTP composites. Zschoyge et al. [15] developed a test procedure to determine the elastic, inelastic, and viscoelastic behavior of CFRTTP composites in a single test. The test involved a stepwise loading/unloading test with stress-relaxation and strain-retardation periods. They tested unidirectional and bidirectional samples made of comingled hybrid roving of glass and polypropylene fibers with fiber volume fractions of 54% and 47%, respectively. The researchers went further to develop a viscoelastic/plastic damage model to predict the structural behavior of components made of CFRTTP composites [15].

A major challenge in producing high-fidelity continuous fiber thermoplastic tape is that the high viscosity of the thermoplastic matrix at melt temperature impedes thorough impregnation of the reinforcing fiber tows. This, in turn, limits fiber content to lower volume fractions than in thermoset composites. Various efforts are underway to mitigate this problem. In a recent study, Dong-Jun Kwon et al. [16] introduced comingled fiberglass yarns with different contents of continuous polypropylene filaments to improve the impregnation of glass fibers with the thermoplastic matrix. The researchers reported better impregnation of fiberglass tows at high content of polypropylene filaments up to 50% by weight. Other efforts included using self-reinforcement of a thermoplastic matrix with high modulus and strength thermoplastic fiber of the same material, such as ultra-high molecular weight polyethylene fiber and matrix [17], and using low-viscosity liquid thermoplastics, such as the novel liquid methyl methacrylate thermoplastic matrix known as Elium®. Carbon fiber reinforced Elium® has shown good potential in impact applications. [18].

While vast research has been conducted to determine the ideal fiber content of thermosetting composites in various processes [19–22], the literature is rather sparse in describing the ideal fiber content in unidirectional thermoplastic tapes [23]. Fiber volume fraction is routinely used in the analysis and design of composites, while fiber weight fraction is mostly used in the manufacturing of composite parts. In general, high fiber content improves mechanical properties, but an increase beyond a certain limit is known to inhibit fiber protection, fiber/matrix bond, and load transfer capacity from matrix to fiber. This, in turn, leads to reduced durability and mechanical properties of thermoplastic composites.

The study of the effect of fiber content on the properties of thermoplastic tape and the determination of optimum fiber loadings for different material systems is, therefore, essential for the design and manufacture of high-performance composite parts and structures. Results of an experimental investigation conducted on a range of high fiber content unidirectional thermoplastic tapes are presented and discussed in this paper. Unidirectional tape made from CF/PPS, GF/PPS, and GF/HDPE materials with various fiber content produced by Celanese Inc. was used. Test panels were compression molded using standard molding procedures.

2. Materials and Methods

2.1. Materials

The materials used in this study were unidirectional continuous fiber reinforced thermoplastic tapes provided by Celanese AEM. The tapes were manufactured via a pultrusion process using melt impregnation. The process utilizes continuous fiber reinforcement and thermoplastic compounds, including neat resin, color master batch, and stabilizing additives as the input materials. Those materials are fed independently and combined

within a crosshead die, where the impregnation takes place. The tape is then cooled and wound onto spools at pre-established weights, thicknesses, and widths. A schematic of the process is shown in Figure 1. Three sets of materials with various fiber contents were produced: carbon fiber reinforced polyphenylene sulfide (CF/PPS), glass fiber reinforced polyphenylene sulfide (GF/PPS), and glass fiber reinforced high-density polyethylene (GF/HDPE). Tape thickness was roughly 0.25 mm for glass-reinforced tapes and 0.15 mm for carbon-reinforced tapes.

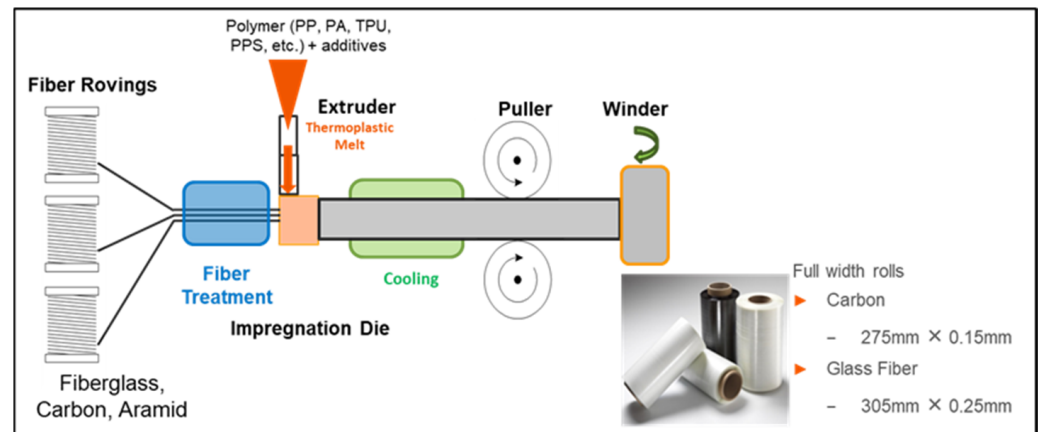


Figure 1. Processing of thermoplastic tape (courtesy of Celanese CE).

High-density polyethylene is a semi-crystalline commodity thermoplastic polymer, and polyphenylene sulfide is a semi-crystalline high-performance polymer. The molecular structure of HDPE and PPS are shown in Figure 2. The mechanical properties of the fiber and matrix materials are given in Table 1.

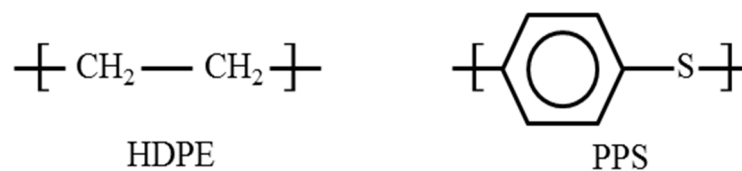


Figure 2. Schematics of the chemical structure of HDPE and PPS, repeat unit.

Table 1. Mechanical Properties of Constituent Materials.

Mechanical Property	HDPE	PPS	E-Glass Fiber	Carbon Fiber
Tensile Modulus (GPa)	0.8	8.1	87	230
Tensile Strength (MPa)	19	74	2900	4900
Strain at Failure (%)	4.1	3.0	4.8	2.1
Compressive Strength (MPa)	22	148	-	-

2.2. Fabrication of Panels and Sample Preparation

Panels were made using a Wabash (Model G30H-15-CX) hot platen press and a two-part steel tool to produce samples in accordance with ASTM standards D 3039 [24], D 790 [25], and D 2344 [26]. The platen temperature was set to the desired mold temperature, and unidirectional tape materials were placed between the two halves of a 152 mm × 305 mm steel clamshell tool. At least one inch of material overhang was used to allow material to freeze at the die edge and prevent fiber-marcelling associated with the use of picture frame tools. The tool was then inserted into the Wabash press with an initial clamping force of one ton until the platen temperature returned to the set temperature. The applied pressure was then increased for a short duration of time, after which the press was

cooled. The pressure, temperature, and duration used were unique for each polymer type, as shown in Table 2.

Table 2. Processing parameters of the CF/PPS, GF/PPS, and GF/HDPE.

Material	Set Temp (°C)	Pressure (kPa)	Dwell Time (Minutes)	Removal Temp (°C)
CF/PPS	340	580	5	135
GF/PPS	340	580	5	135
GF/HDPE	199	193	2	52

All panels produced for this study were unidirectional; however, the same procedure can be used to mold various lay-ups, including quasi-isotropic panels, provided all fibers remain in tension during consolidation. The process yielded uniform fiber distribution at various fiber contents in the panels, as shown in the images obtained by a Nikon Eclipse optical microscope at 200X magnification in Figure 3.

Tensile panels were made of three layers of tape, and flexure panels were manufactured using the appropriate number of layers of tape to achieve a minimum panel thickness of 3.18 mm. Glass fiber panels required 10 to 11 layers of tape, while the thinner carbon fiber panels required 22 to 23 layers of tape. The panels were visually inspected for defects, tabs were added for the tensile panels, and specimens were machined using a diamond wheel on a wet saw. Samples for fiber content analysis, density measurement, thermal analysis, and microscopy were taken from respective tapes and panels prepared for flexure tests.

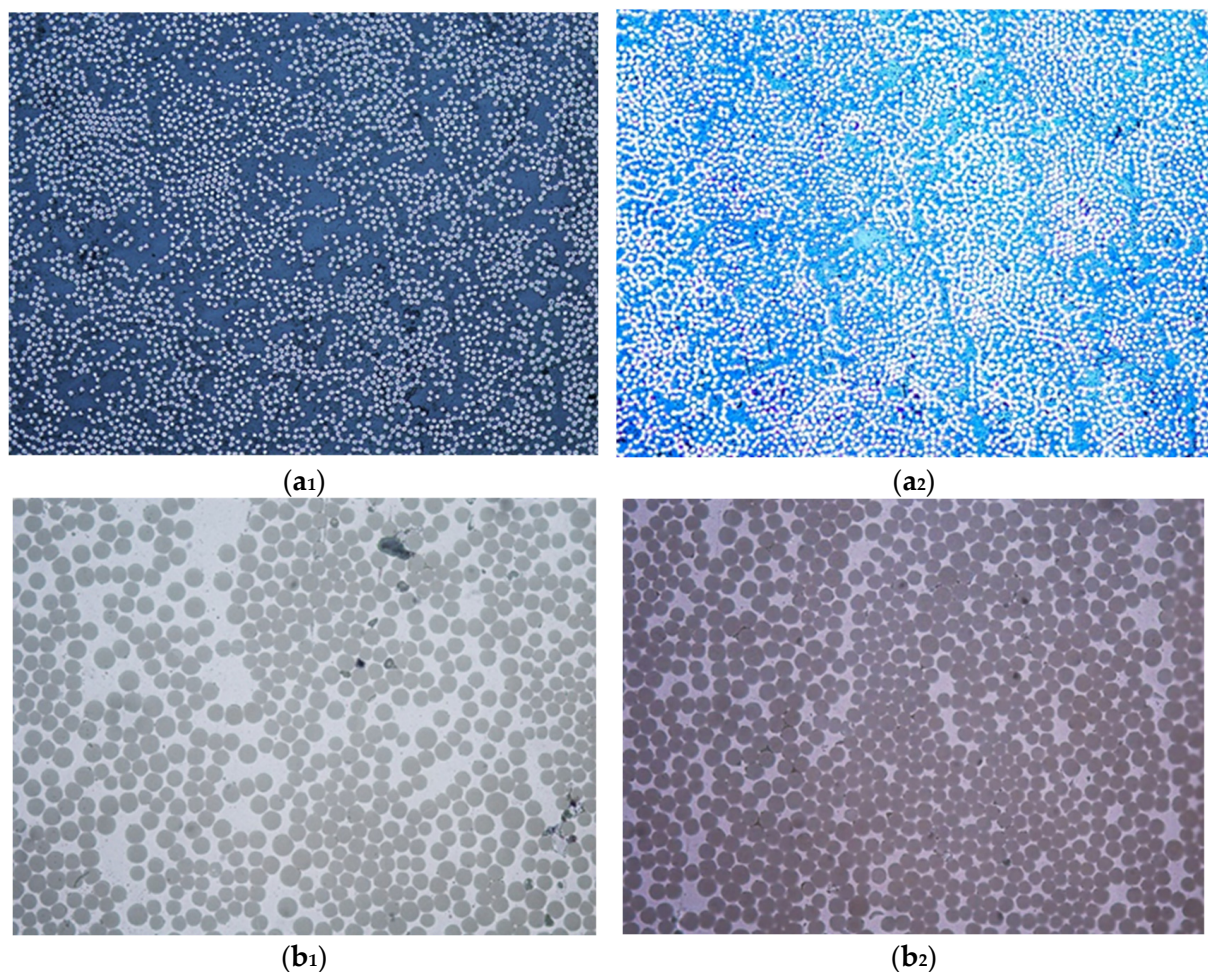


Figure 3. Cont.

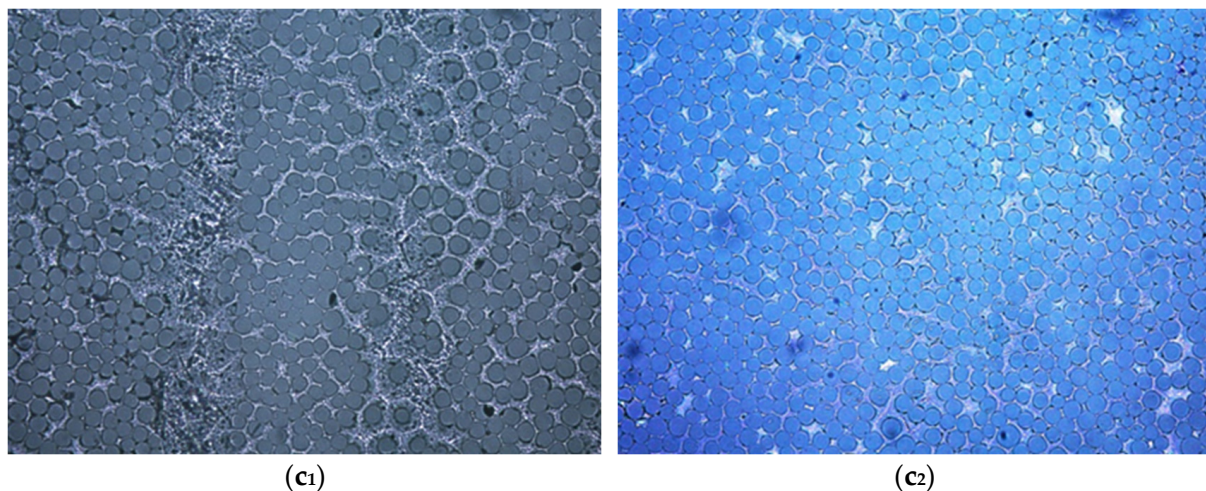


Figure 3. Fiber distribution in panels with low and high fiber volume fractions at 200× magnification: CF/PPS, (a₁) 49.6% and (a₂) 68.1%; GF/PPS (b₁) 54% and (b₂) 70%; GF/HDPE (c₁) 44% and (c₂) 66%.

3. Results and Discussion

3.1. Constituents' Contents

To determine the exact amounts of constituent materials, three types of measurements were conducted. These included nominal fiber weight fraction, defined as the net raw material input in tape manufacturing, matrix burn-off and acid digestion to find weight fractions of fiber and matrix, and density measurements to obtain volume fractions and to correct for void content in the materials. The nominal weight fractions were calculated assuming no voids and no resin loss. Efforts were made to minimize the inherent resin loss in the manufacturing process due to drooling or melt flow irregularities. The GF/PPS and GF/HDPE tapes and panels were tested for fiber content using fiber burn-off according to ASTM D 3171-A7 [27], and the CF/PPS tapes and panels were analyzed using a nitric acid digestion technique, ASTM D3171-A1 [27]. Figure 4a through Figure 4c show graphical comparisons of the average fiber weight fractions obtained by nominal calculations and by experimental measurement of tape and panel samples of each of the three materials systems. It can be observed in these figures that the fiber weight fractions obtained by each measurement follow the same increasing trend as designed for the samples. It was noticed that the fiber contents in the tapes were more variable and slightly lower than that of the panels. The high variability in measurements of the weight fractions of the tape is possibly due to selection bias and limited sample area. The panels were made by compression molding of multi-layered unidirectional tape, a process that affects localized high/low fiber weight fraction due to redistribution of resin during consolidation. The measurement of fiber weight fractions of the panels usually averages out any variability in the tapes due to the presence of multiple layers of thermoplastic tape. It is shown in these figures that the panels provided the most uniform data of fiber weight fractions; therefore, fiber weight fractions in panels were selected for further calculation of fiber volume fractions.

The experimental density of panels was obtained at room temperatures, 22 °C–23 °C, using the ASTM D3800—Archimedes method [28]. The density of PPS, HDPE, glass fiber, and carbon fiber are reported to be 1.35 g/cm³ [29], 0.948 g/cm³ [30], 2.54 g/cm³ [5], and 1.80 g/cm³ [31], respectively. The values of fiber weight fraction obtained in the panels were used to determine the theoretical density of the panels, assuming no void content. Knowing the theoretical and experimental density values, the void content in the panels was calculated using Equation (1):

$$\text{Void Fraction} = \frac{V_{\text{experimental}} - V_{\text{theoretical}}}{V_{\text{experimental}}} \quad (1)$$

where V represents specific volume, with unit of cm^3/g . The fiber volume fractions were then calculated with and without the void contents. Figure 5 shows the fiber volume percent in each of the panel samples. The open and filled markers in Figure 5 represent fiber volume percent without and with voids, respectively.

The void content in all the samples increased with the increase in fiber content and ranged from 0% to 2%. The largest void content appears for CF/PPS panels with the highest carbon fiber content. A micrograph showing void contents and fiber-rich areas of CF/PPS at a fiber volume fraction of 61.3% is shown in Figure 6 below. The highlighted deep pockets in the matrix indicate voids in the specimen. The remainder of the panels had a void content of less than 1%. All properties presented in this paper are given in terms of fiber volume fraction considering the voids in the samples. These fiber volume fractions are shown in Table 3.

The presence of voids could be attributed to factors such as poor impregnation, surface contamination of precursor tape material, poor panel consolidation, or poor machining quality. It should be noted that the processing parameters for the panels produced for this study were not fully optimized for the high fiber content of the tapes. Panels with various fiber contents were consolidated with the same parameters as standard panels, which may not have been ideal. Machining of unidirectional composite materials typically induces internal damage along the cut edge due to the brittle reinforcement and low transverse strength. Similar to thermoset composites, surface contaminants such as dust and oils may lower the bonding strength between the fiber and matrix. Considering these factors, the panels produced in this study appeared to be of good quality based on test results.

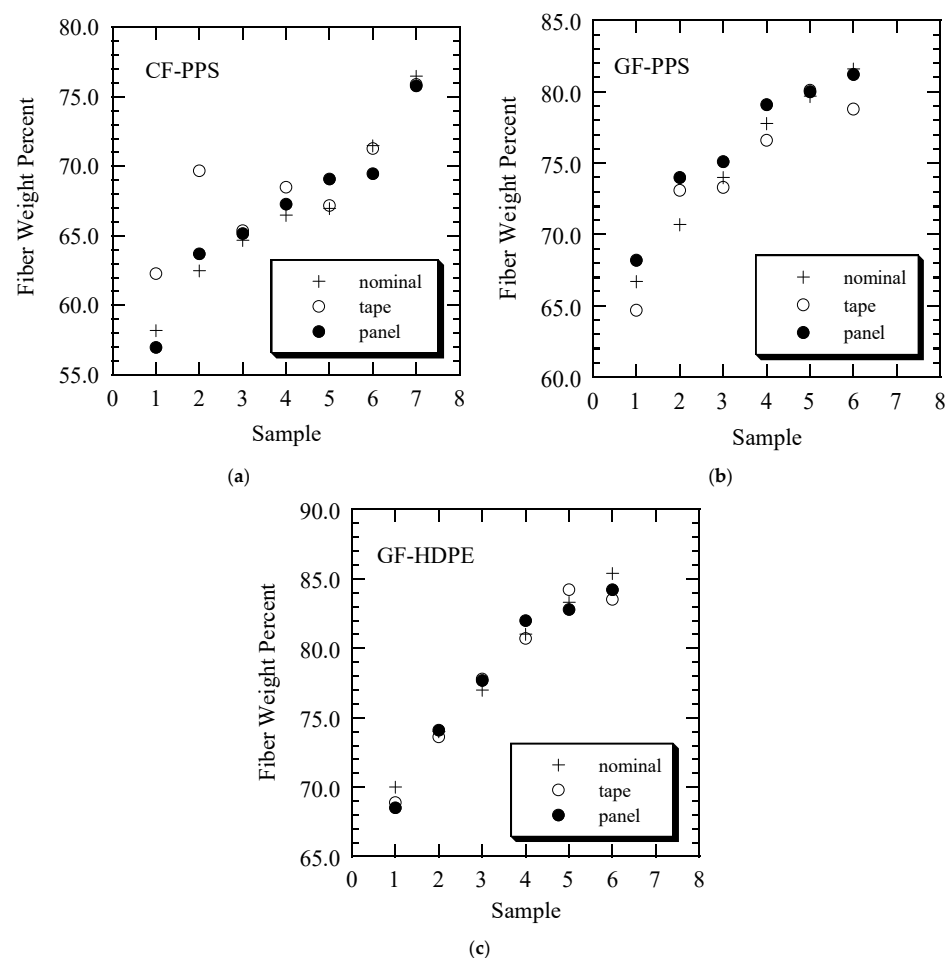


Figure 4. Fiber weight content of tape and fabricated panels: (a) CF-PPS; (b) GF-PPS; (c) GF-HDPE. Nominal refers to desired fiber content.

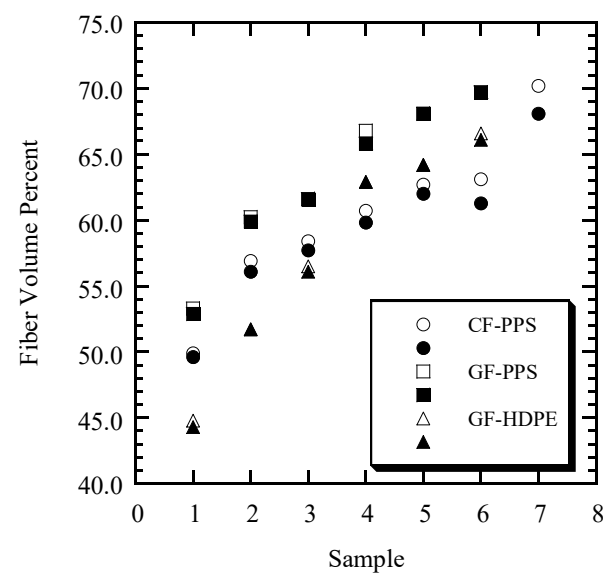


Figure 5. Fiber volume fraction of the composite panels with void contents (filled markers) and without void contents (open markers).

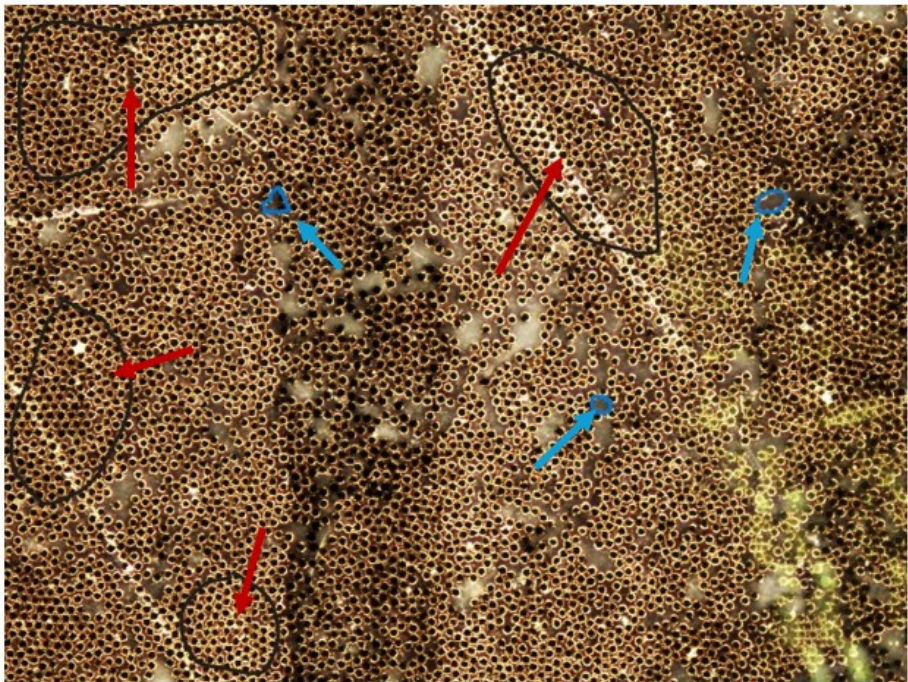


Figure 6. Void and fiber-rich areas (blue and red arrows) in CF/PPS at 61.3% Vf.

Table 3. Nominal and actual fiber weight and actual fiber volume in panels of the three composites.

CF/PPS			GF/PPS			GF/HDPE		
Nominal Fiber Weight %	Actual Fiber Weight %	Actual Fiber Volume %	Nominal Fiber Weight %	Actual Fiber Weight %	Actual Fiber Volume %	Nominal Fiber Weight %	Actual Fiber Weight %	Actual Fiber Volume %
58.2	57.0 ± 0.3	49.6 ± 0.3	66.7	69.1 ± 0.9	53.6 ± 0.7	70.0	68.5 ± 0.0	44.3 ± 0.0
62.5	63.7 ± 0.8	56.1 ± 0.8	70.7	74.1 ± 0.1	60.1 ± 0.1	74.0	74.3 ± 0.2	51.9 ± 0.2
64.7	65.2 ± 0.2	57.7 ± 0.2	74.0	75.3 ± 0.2	61.9 ± 0.3	77.0	77.7 ± 0.1	56.1 ± 0.1
66.5	67.3 ± 0.8	59.8 ± 0.9	77.8	79.2 ± 0.1	65.9 ± 0.1	81.0	81.5 ± 0.4	62.3 ± 0.7
67.0	69.1 ± 0.4	62.0 ± 0.4	79.7	80.2 ± 0.2	68.3 ± 0.3	83.3	83.0 ± 0.3	64.6 ± 0.4
71.5	69.5 ± 0.5	61.3 ± 0.5	81.6	81.7 ± 0.4	70.2 ± 0.5	85.4	84.2 ± 0.0	66.1 ± 0.0
76.5	75.8 ± 1.4	68.1 ± 1.6						

3.2. Effect on Thermal Properties

To ensure that the processing temperatures do not adversely affect the polymer, the thermal stability of neat pellets of PPS and HDPE were tested using a 2390 TA Instruments thermo-gravimetric analyzer, TGA. Nitrogen flow of 10 mL/min and a heating ramp of 5 °C/min were used. The results from TGA showed that mass loss in HDPE and PPS began at about 350 °C and 400 °C, respectively. These temperatures are well above the processing temperatures of their respective polymers, shown in Table 2. Therefore, processing these materials through the heating cycles in Table 2 did not deteriorate the polymers in any way, and no signs of degradation were noticed in the carbon and glass fibers.

A Q100 differential scanning calorimeter, DSC, TA Instruments, New Castle, Delaware, USA, was used to investigate the change in the degree of crystallinity of the polymer matrix with fiber content. The heat of melting and crystallization of neat HDPE and PPS were obtained first. Figure 7 shows the DSC thermograms of neat polymer pellets. Each sample was heated twice at 10 °C/min and cooled at 2 °C/min.

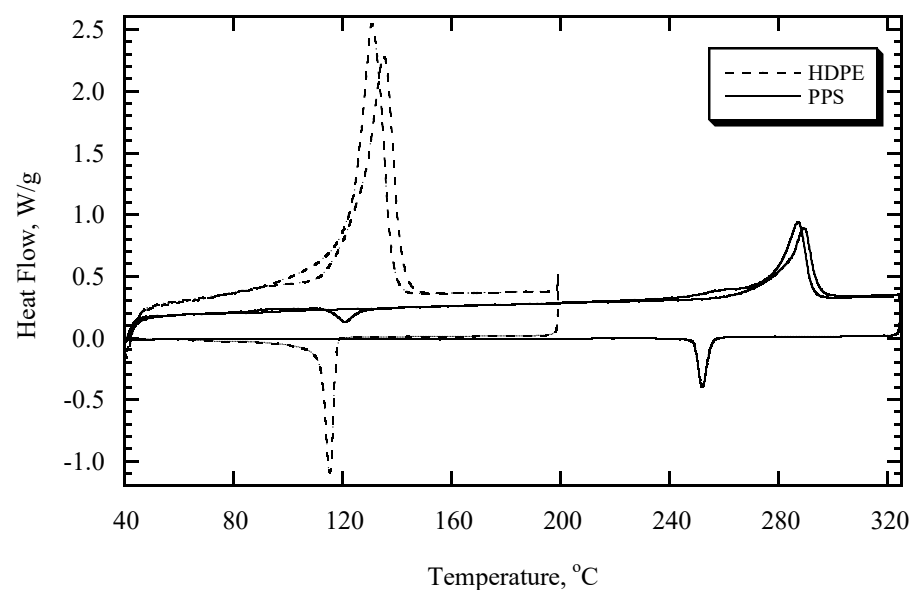


Figure 7. Thermograms obtained from DSC runs on neat HDPE and PPS pellets. A cooling rate of 2 °C/min was used to obtain the maximum heat of crystallization/melting of crystals. Endo Up.

Figure 7 shows the melting temperature of HDPE to be lower than that of PPS but its enthalpy of melting to be larger than that of PPS. There is a small enthalpy of cold crystallization of PPS that appears at about 120 °C in the first heating cycle of the polymer. To interpret the data further, Table 4 was generated.

Table 4. Thermogram results of neat HDPE and PPS; 1st and 2nd refer to heating cycles of samples.

Material	Cooling Rate, (°C/min)	$\Delta H_{\text{melting, 1st}}$ (J/g)	$\Delta H_{\text{melting, 2nd}}$ (J/g)	T_m , 1st (°C)	T_m , 2nd (°C)
HDPE	2	186	189	135	131
	5	220	216	135	132
PPS	2	46	51	289	287
	5	60	59	288	283

In Table 4, the data for DSC experiments include cooling rates at 2 °C/min and 5 °C/min. The magnitude of enthalpy of cold crystallization in both sets of experiments was 6 J/g for PPS and 0.0 J/g for HDPE. The difference in the first heat of melting for HDPE (186 J/g versus 220 J/g) and PPS (46 J/g versus 60 J/g) shows that small DSC samples may

not accurately represent the bulk polymer and to obtain an accurate heat of melting for these polymers, numerous samples should be tested.

To determine the effect of fiber content on the heat of melting of the composites, 15–20 mg DSC samples were carefully cut from tensile panels in such a way that no crushing of matrix or fiber occurred. Tensile bars are thinner and easier to cut with less damage to the samples. This was necessary to keep the fiber content intact and to further analyze the data based on the polymer content only. In all DSC experiments, specimens were heated at 10 °C/min from 40 °C to above the melting peak of their respective polymer with a nitrogen flow of 50 mL/min. The specimens were then cooled at 5 °C/min to 40 °C and reheated to above melting. Heats of melting of PPS and HDPE crystals in panels with various fiber volume fractions were measured. The average heat of melting from samples of the panels, normalized with respect to the weight fraction of their polymers, is shown in Table 5.

Table 5. Average values of heat of melting of PPS and HDPE crystals in tensile panels with various fiber volume content.

Material	Heat of Melting, (J/g)
CF/PPS	43 ± 1
GF/PPS	43 ± 2
GF/HDPE	186 ± 25

There was practically no difference in the heat of melting of crystals in any of the panels made with PPS, and no cold crystallization was observed in reinforced PPS panels. The average heat of melting of GF/HDPE composites had a high standard deviation and a coefficient of variation of 13.4%. This large COV indicates that the addition of glass fiber may have an insulating effect, leading to a slow cooling rate and resulting in a higher degree of crystallinity in HDPE panels. Moreover, different degrees of crystallinity seem to have been developed in different regions due to different cooling rates in these regions of the panels, leading to high variability in the heat of melting. However, no conclusive results were obtained from the DSC test data on the effect of fiber content for HDPE, and no correlation was found between fiber content and heat of melting in HDPE panels.

Dynamic mechanical analysis was performed on all samples using a Q800 DMA, TA Instruments, New Castle, Delaware, USA, to obtain glass transition temperatures and flexural storage moduli [32]. Dual cantilever specimens of average dimensions 35 mm (L), 12.7 mm (W), and 3 mm (H) were tested at 1 Hz and 15-micron amplitude. All HDPE and PPS composite samples were tested from room temperature to 150 °C and 320 °C, respectively, at 5 °C/min. Figure 8 shows a typical plot from DMA experiments of GF/PPS at Vf of 61.1% and GF/HDPE at Vf of 44.3%. The left and right y-axes represent flexural storage and loss moduli, E' and E'' , respectively. The flexural storage modulus at room temperature, glass transition temperature, T_g , and the melting temperature, T_m , are obtained from the data. Glass transition temperatures are read from the peak of loss modulus curves or inflection point of the storage modulus curves. Tests were not conducted at lower temperatures to capture the T_g of HDPE, and the melt temperature, T_m , of the PPS specimens was not clearly defined, as shown in Figure 8.

The variation in glass transition temperature with fiber volume fraction of CF/PPS and GF/PPS composites is shown in Figure 9a and the variation in the melt transition temperature of GF/HDPE in Figure 9b. In these figures, T_g and T_m are obtained using the peak of E'' graphs from the DMA tests. Data are fitted to lines to show an increasing trend in T_g with fiber content.

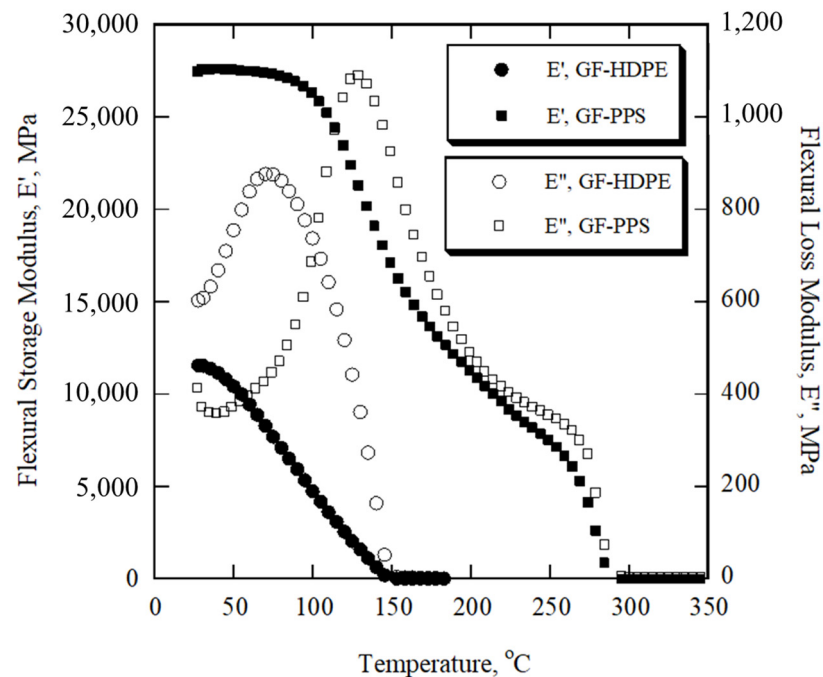


Figure 8. Storage and loss moduli of GF/PPS at 61.1% and GF/HDPE at 44.3% glass fiber by volume.

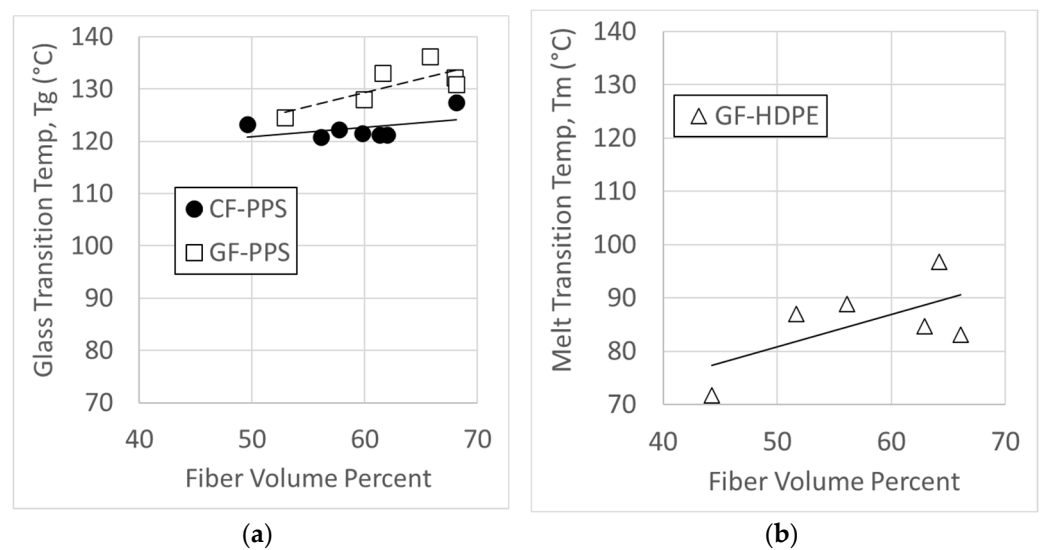


Figure 9. Variation in glass and melt transition temperatures, using peak of E'' curves, with fiber content, (a) CF/PPS and GF/PPS samples, (b) GF/HDPE samples.

There are two main mechanisms that may have influenced these results: change in heat transferability and change in crystallinity [33]. It has been demonstrated that thick specimens have a perceived greater T_g because it takes a slightly longer time to heat through their thickness than thinner specimens. Higher crystallinity also increases T_g since additional thermal energy is required to unravel the tightly packed crystals [33]. When extrapolating these concepts to a fiber-filled polymer composite, an obvious issue would be the use of insulating fiber like glass or conductive fiber like carbon. Glass fiber has high thermal heat capacity, and as its content increases, the heat transfer through specimens will be reduced significantly. On the other hand, carbon fiber will evenly distribute heat due to its higher thermal conductivity. The other aspect that may be causing T_g to increase as fiber content increases is that fiber surfaces can act as nucleation sites for polymer chains to

lay down and add internal energy that would need to be additionally unraveled to reach the T_g . This effect could resemble an increase in crystallinity.

Figure 10 shows the variation in flexural storage modulus, E' , at 30 °C with the fiber content for the three materials. The trend shows that the flexural storage modulus increases with fiber content for all three composites. For the CF/PPS material, the increasing trend is consistent despite the scattered data compared with the other two materials in Figure 10.

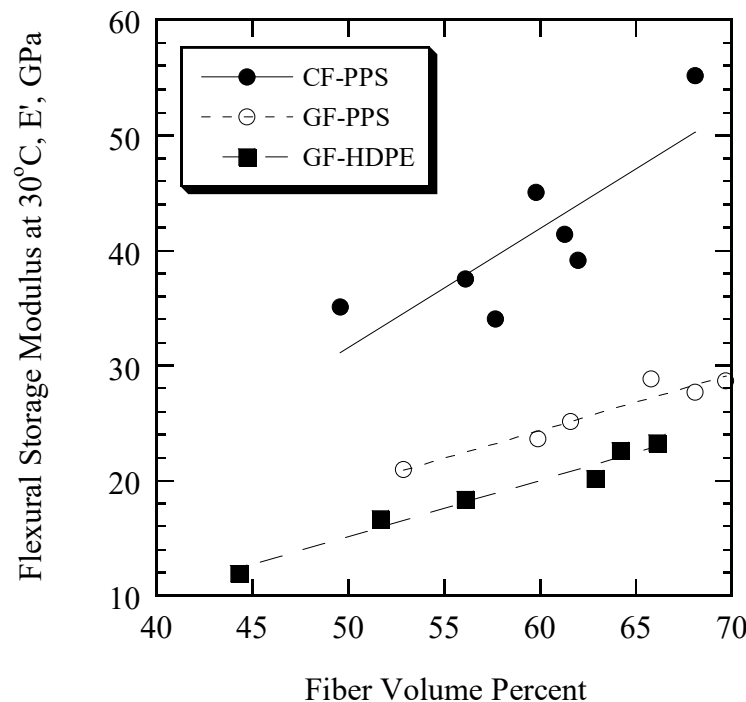


Figure 10. Flexural storage modulus at 30 °C versus fiber content.

3.3. Effect on Mechanical Properties

3.3.1. Tensile Properties

Tensile, flexural, and short beam shear tests were conducted on an Instron Model 5585H load frame. An Instron 2630-100 strain extensometer, Instron, Norwood, MA, USA, was utilized for all tensile testing. Tensile specimens of dimensions 150 mm (L), 12.7 mm (W), and ~1 mm thickness were tested according to ASTM D3039 [24]. The variation in tensile strength, modulus, and strain at maximum stress with fiber content is shown below in Figure 11a–c.

A general trend of increase in longitudinal tensile strength with fiber content up to specific volume fractions is shown in Figure 11a. The maximum tensile strength of the GF/HDPE and GF/PPS composites was achieved at fiber volume fractions of 56% and 64%, respectively. The corresponding strength values were 800 MPa and 1100 MPa, respectively. The tensile strength of the CF/PPS composite seemed to reach a plateau at a fiber volume fraction of 68–70%, with a corresponding strength value of 2480 MPa. The tensile moduli of the three materials follow a similar trend of increasing with fiber content to reach a maximum value and then decreasing with the addition of fiber reinforcement. Similar trends were obtained in discontinuous GF/PP, GF/PET, and CF/PET composites by Lu [34]. The optimum fiber content of GF/HDPE was 64% by volume; for GF/PPS, it was 63% by volume; and for CF/PPS, it was 68% by volume. The corresponding maximum modulus values for these materials were 50 GPa, 58 GPa, and 140 GPa, respectively, as shown in Figure 11b. These results indicate that the optimum fiber content depends on both the thermoplastic matrix and the type of fiber. The optimum fiber content for tensile strength and modulus for the CF/PPS material is in the range of 68–69%, and for the GF/PPS composite, it is in the range of 63–64%, as shown in Table 6 below.

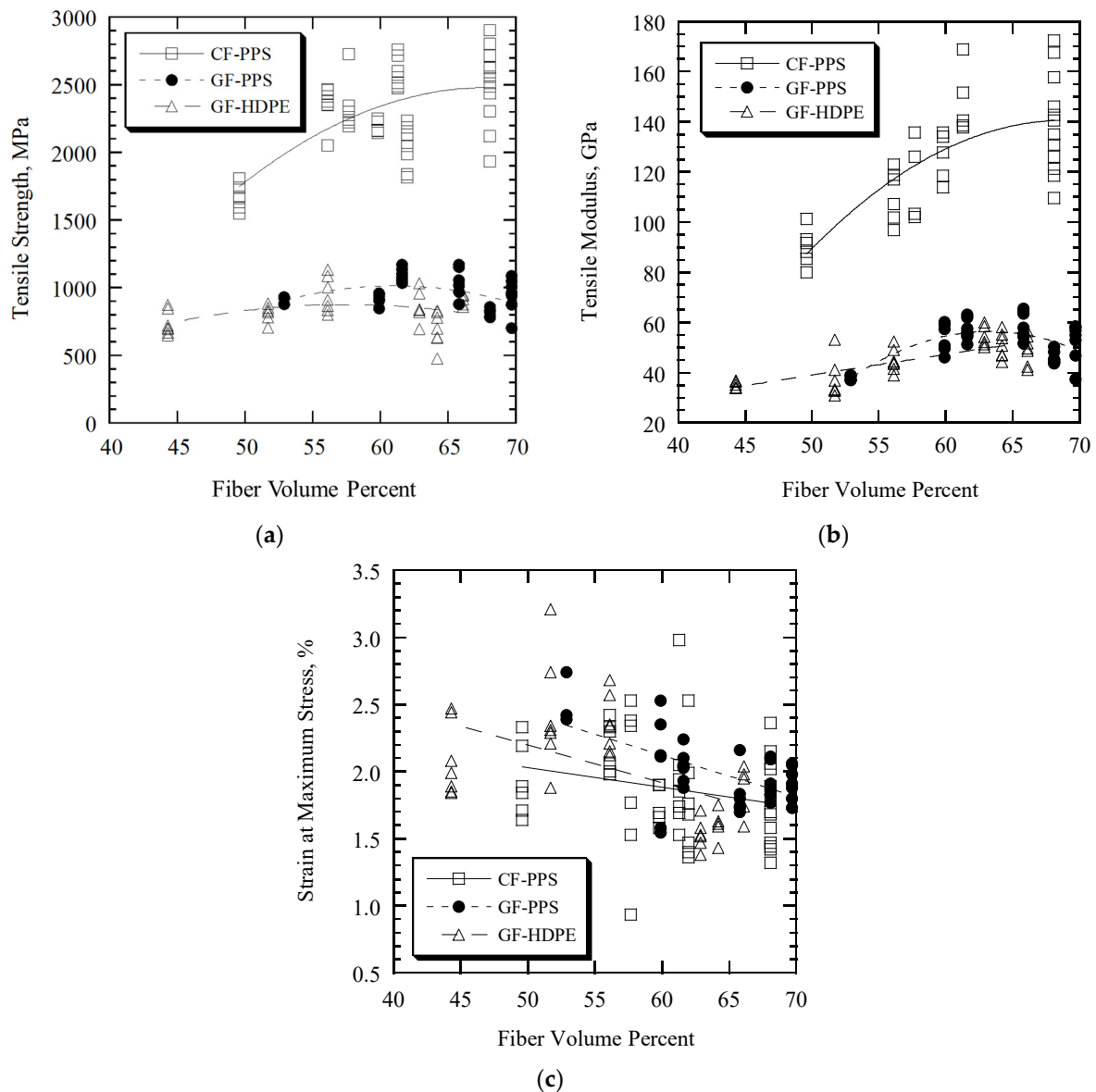


Figure 11. Variation in tensile properties: (a) strength; (b) modulus; (c) strain at maximum stress. All graph properties plotted with actual fiber volume fraction in the x-axis.

Table 6. Mechanical properties at optimum fiber volume fraction (V_f opt.) in percent.

Material Properties	CF/PPS		GF/PPS		GF/HDPE	
	Value	V_f Opt	Value	V_f Opt	Value	V_f Opt
Max Tensile Strength (MPa)	2480	69%	1100	64%	800	56%
Max Tensile Modulus (GPa)	140	68%	58	63%	50	64%
Max Flexural Strength (MPa)	1280	61%	1250	70%	550	65%
Max Flexural Modulus (GPa)	140	68%	55	70%	50	66%
Strain at Max Tensile Stress (%)	1.8	69%	2.2	64%	2.1	56%
Strain at Max Flexure Stress (%)	1.1	61%	3.2	70%	1.1	65%

For the GF/HDPE composite used in this study, the optimum fiber content for a tensile strength of 56% is much lower than the optimum fiber content to achieve the maximum tensile modulus, which is 64% by volume. This could be attributed to the interfacial bond between the HDPE and glass fibers. The non-uniform degree of crystallinity in the HDPE matrix noted above and possible incompatible glass fiber sizing seem to have caused a

weak interfacial bond between the fiber and matrix. The longitudinal tensile strength in a composite depends on both the fiber and fiber/matrix interface. At higher fiber content, cracks initiate and propagate in regions of weak interface. This, in turn, leads to further debonding and a decrease in tensile strength beyond the fiber content of 56% for the GF/HDPE material. The micrographs of tensile failure surfaces of GF/PPS and GF/HDPE in Figure 12 show better interfacial bonds between glass fiber and PPS matrix than between glass fiber and HDPE matrix. A considerable amount of matrix is adhered to the glass fiber after failure of the GF/PPS composite, as shown in Figure 12a, while the fibers in the GF/HDPE in Figure 12b are rather clean with no residue of matrix after failure, indicating poor fiber/matrix adhesion and lowering the tensile strength at high fiber content. On the other hand, the tensile modulus is fiber-driven and less affected by debonding in the fiber/matrix interface.

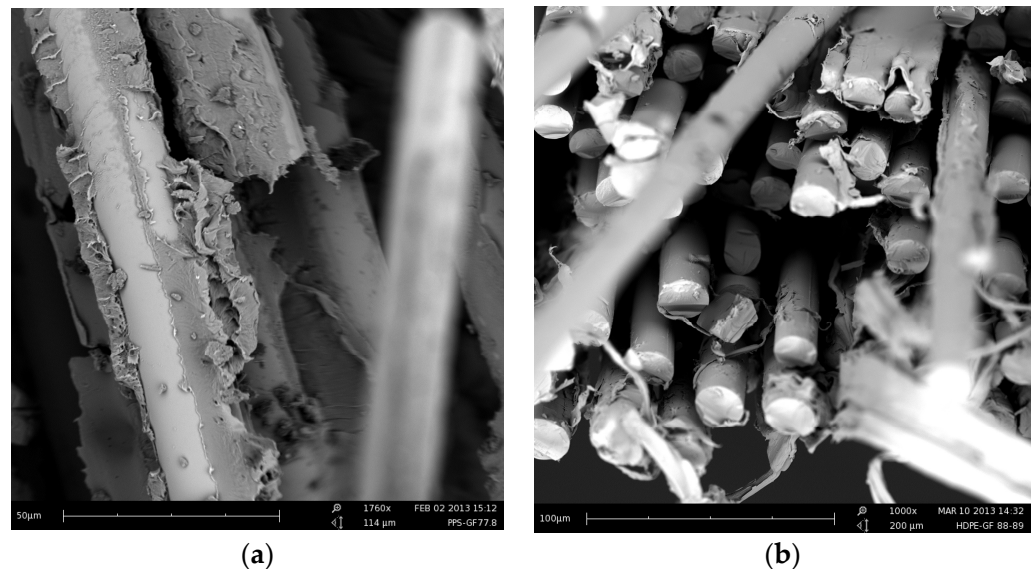


Figure 12. Tensile failure surface of (a) GF/PPS and (b) GF/HDPE.

Figure 11c shows a linear decrease in longitudinal tensile strain-at-maximum-stress with an increase in fiber content as the unidirectional fibers continue to dominate the behavior and stiffen the composite. The average longitudinal strains-at-maximum-stress of the GF/HDPE composite decreased from 2.3% at glass volume fraction, V_{gf} , of 44% to 1.7% at V_{gf} of 66%. The GF/PPS composites strain decreased from 2.5% at 53% fiber content to 1.9% at 70% V_{gf} , while the longitudinal strain-at maximum-stress of the CF/PPS decreased from 2.1% to 1.8% at carbon fiber contents, V_{cf} , of 50% and 68%, respectively. It is shown in Figure 11c that minimum strains occur at maximum fiber contents.

It is important to note that in this study, the same panel processing parameters were used for all fiber loadings in each material system. The properties at optimum fiber content determined above may change if the processing parameters were adjusted based on fiber loading [35]. For example, higher pressure and longer dwell time may be required for adequate consolidation of panels manufactured with higher fiber content to allow uniform distribution of the polymer between the closely packed fibers. Conversely, lower pressure and shorter dwell time at high fiber content would cause lower strength due to inadequate interlaminar bonding [36,37]. Therefore, adjustments in processing parameters could improve the tensile properties of the material systems at higher fiber content.

3.3.2. Flexure Properties

Results of the three-point flexure tests are shown below in Figure 13a–c. A minimum of three replicate specimens with an average width of 12.7 mm, minimum thickness of 3.2 mm, and a span-to-depth ratio of 40:1 were tested according to ASTM D790 [20] for

each fiber content. The variation in flexural strength with fiber volume fraction in each of the three composites is shown in Figure 13a. The flexure strength increases steadily with the addition of fiber in each composite. It reaches the highest value of 1280 MPa at Vcf of 61% in the CF/PPS composite. The maximum flexural strength in the GF/PPS composite is 1250 MPa at Vgf of 70%, and in the GF/HDPE material, the maximum strength is 550 MPa at Vgf of 65%, as shown in Figure 13a below and Table 6 above. Due to compression above the neutral axis in the three-point flexure test, the matrix plays a significant role in the flexure strength. This is indicated by the large difference between the flexure strengths of the GF/PPS and GF/HDPE materials. As seen in the molecular structure in Figure 2 and the constituent properties in Table 1, the PPS matrix has a more robust structure, higher mechanical properties, and better fiber/matrix interfacial bond than the HDPE matrix, as shown in Figure 12 above.

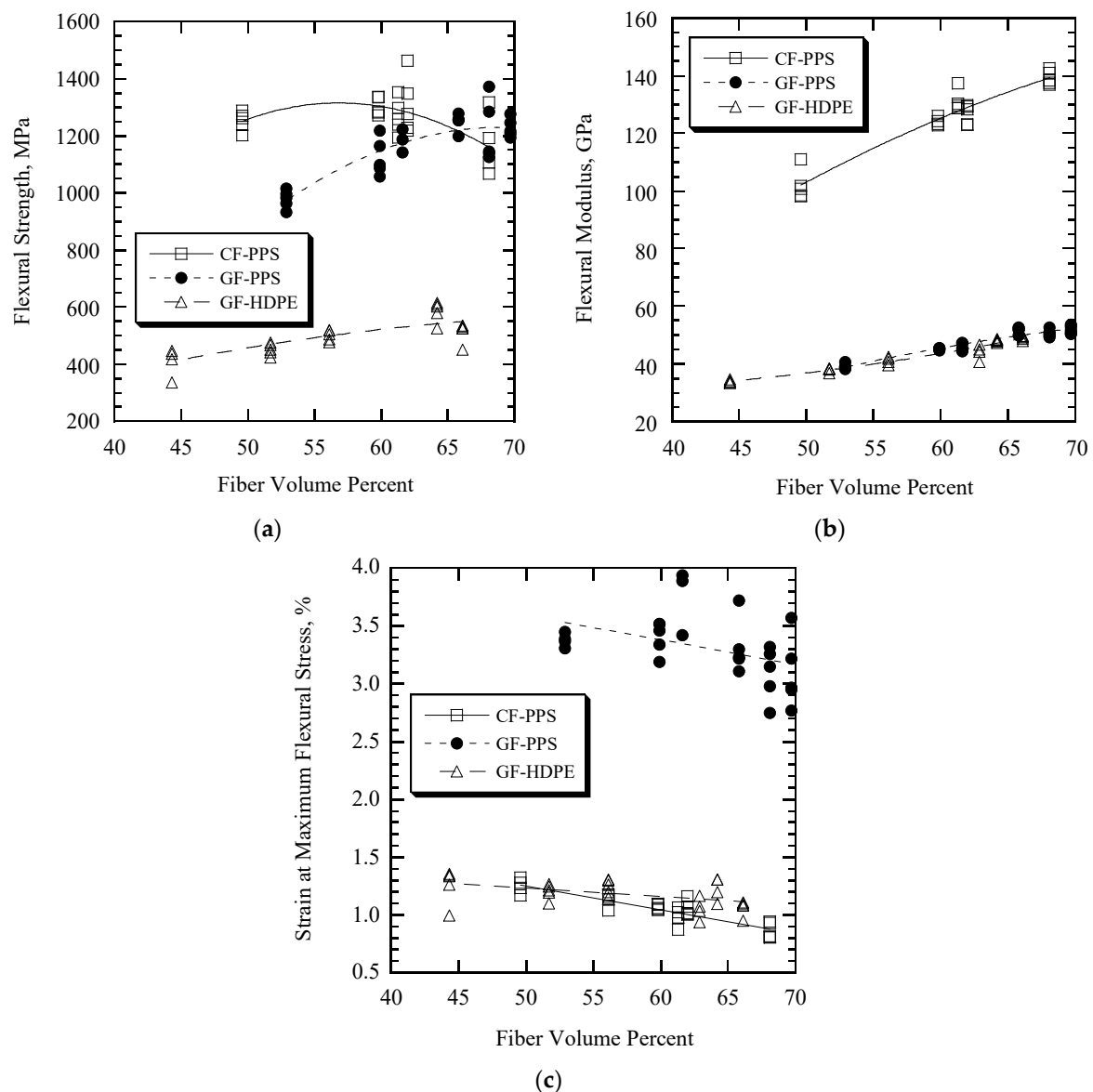


Figure 13. Variation in flexural properties: (a) strength; (b) modulus; (c) strain at ultimate stress. All graph properties plotted with actual fiber volume fraction in the x-axis.

The flexure strength of unidirectional composites is typically lower than the tensile strength, as demonstrated by the data of the CF/PPS and GF/HDPE composites in Table 6. However, in this study, the flexure strength of the GF/PPS composite, 1250 MPa at optimum

V_{gf} is slightly higher than its tensile strength, 1100 MPa, albeit at a higher optimum fiber content, as shown in Table 6. This is apparently due to the high compressive strength of the PPS matrix and the high strain of the glass fiber. The high-strength PPS matrix is capable of resisting higher flexural stress at the top of the specimen, and the high strain of glass fiber prevents sudden crushing failure in compression. This synergistic action, combined with the high optimum volume fraction of 70%, leads to a higher flexure strength of GF/PPS than its tensile strength. Examples of flexure failure of each composite system at optimum fiber content are shown in Figure 14a,b. The figure shows that most of the flexure failure occurred on the compression side and that the compression properties of fiber and matrix play a dominant role in the flexural behavior of the CF RTP composites used in this study. No noticeable failure was observed on the tension side of the CF/PPS and GF/HDPE, as shown in Figure 14b. The GF/PPS demonstrated combined tension and compression resistance to flexure loading, as shown by the compression and tension failure modes in Figure 14a,b. This combined resistance explains the high flexure strength of 1250 MPa of the material, as shown in Table 6. Typical flexure stress–strain curves of the three composites are shown in Figure 15, indicating the high strength and ductility of the GF/PPS compared with CF/PPS and GF/HDPE.

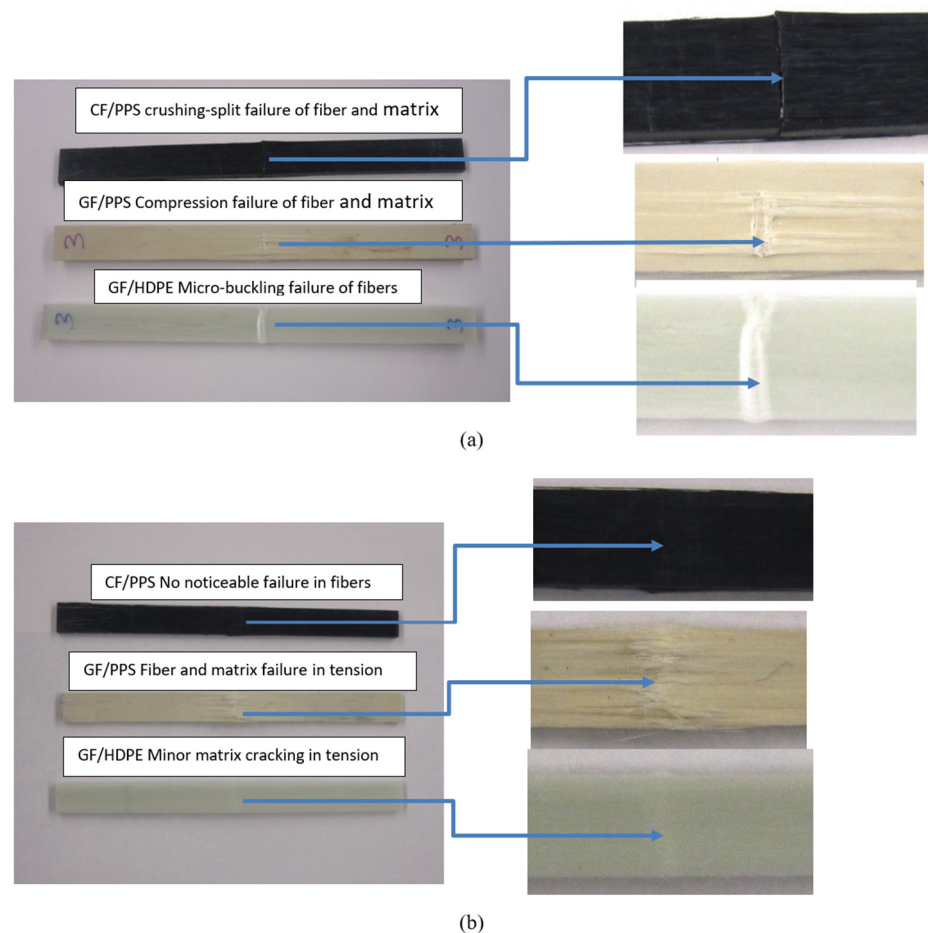


Figure 14. Failure of specimens tested in flexure on (a) Compression Side; (b) Tension Side.

The variation in flexural strain with fiber content, shown in Figure 13c, indicates a linear decrease in strain with the increase in fiber content in the three composite systems. The ultimate strain in the CF/PPS material decreases from 1.2% at V_{cf} of 50% to 0.9% at V_{cf} of 68%. The ultimate strain of the GF/PPS system decreases from 3.5% at V_{gf} of 52% to 3.2% at V_{gf} of 70%. Similarly, the ultimate strain of the GF/HDPE decreases from 1.3% at V_{gf} of 45% to 1.1% at V_{gf} of 66%. While the trend is similar to the tensile strain variation with fiber content, the mechanism is different, as indicated by the range of values

in Figures 11c and 13c. The ultimate tensile strain is dominated by the ultimate strain of the fiber and the bond between fiber and matrix; however, the ultimate flexure strain is influenced by the tensile and compressive strains of fiber and matrix in addition to the bond at the interface.

For example, the flexure strain of the CF/PPS seems to be dominated by the low compressive strain and the crushing–split compression failure of the carbon fiber at high fiber content, as shown in Figure 14a. On the other hand, the flexure strain of the GF/PPS is dominated by the higher compressive strain of the glass fiber and the PPS matrix, as indicated in Figures 14a,b and 15. The GF/HDPE composite exhibited lower flexural strength and strain, as shown in Table 6 and Figures 13c, 14a and 15, due to the weak interfacial bond, which leads to micro-buckling of glass fibers and compressive failure of the composite.

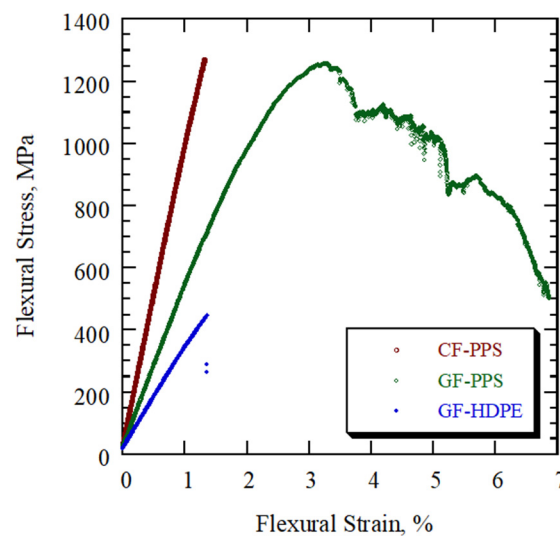


Figure 15. Typical flexure stress–strain curves of the three composite systems at high fiber content.

3.3.3. Interlaminar Shear Properties

Short beam shear tests were conducted according to ASTM D 2344 [26]. Specimens with an average width of 12.7 mm, minimum thickness of 3.2 mm, and a span-to-depth ratio of 4:1 were used. A minimum of three replicate specimens were tested at each level of fiber content. The tests were performed to study the effect of fiber content on the interlaminar shear strength between the layers of the reinforced thermoplastic panels and, to some extent, assess the fiber/matrix interface bond strength with an increase in fiber content. The results of the short beam shear strength are shown in Figure 16. Most of the GF/HDPE specimens did not exhibit proper shear failure due to excessive bending deformation and high strain of the HDPE and, therefore, were not included in the analysis or in Figure 16.

The variations in short beam shear strength with fiber volume fraction for the CF/PPS and GF/PPS materials are shown in Figure 16. Data of all specimens tested are included in this figure. The data show that the short beam shear strength of the CF/PPS material decreases consistently with an increase in fiber content. The maximum average shear strength was obtained at a volume fraction of carbon fiber, V_{cf} , of 50%, and the minimum average short beam shear strength was obtained at V_{cf} of 68%. Samples with V_{cf} less than 50% were not tested to observe the trend at lower fiber content; however, the results show that the interlaminar shear strength and/or the bond between the carbon fiber and PPS matrix decrease with the addition of reinforcement beyond V_{cf} of 50%. This decrease is expected at high V_{cf} due to a lack of encapsulation of all the reinforcing fiber, an increase in void content, and the development of fiber-rich and matrix-starved areas, as shown in Figure 6 above. The smooth fracture surface of fiber and matrix in a short beam shear specimen of CF/PPS at 68% V_{cf} in Figure 17 below indicates poor adhesion

at the fiber/matrix interface. A similar model and observations were made by Lee and Palley for carbon/epoxy composites [38]. The short beam shear strength can be improved with more compatible sizing and functionalization of carbon fibers, but it is interesting to note that the decrease in shear strength does not affect the tensile strength and modulus and flexure modulus of CF/PPS which continue to increase with fiber content up to a volume fraction of 68%, as shown in Figures 11a,b and 13b. It is not known at this point whether improvement in short beam shear strength will further enhance the tensile and flexure properties, as well as other properties such as fatigue and impact properties of this material.

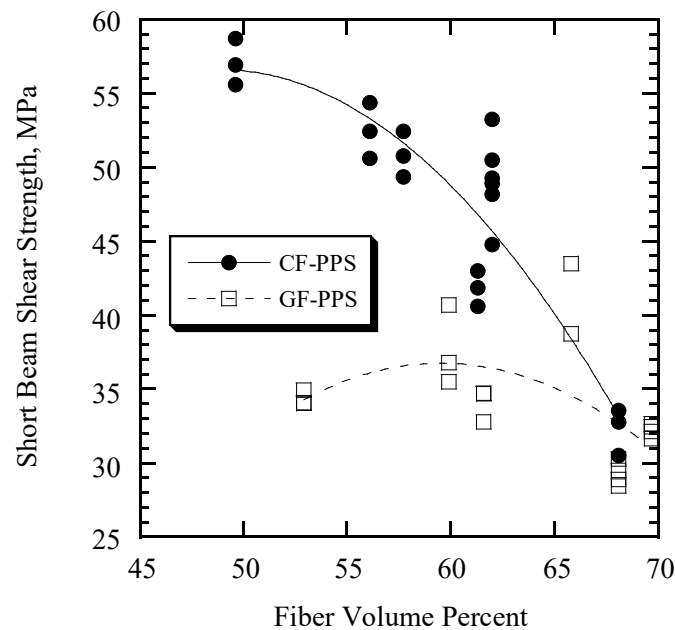


Figure 16. Variation in short beam shear strength of CF/PPS and GF/PPS with fiber content.

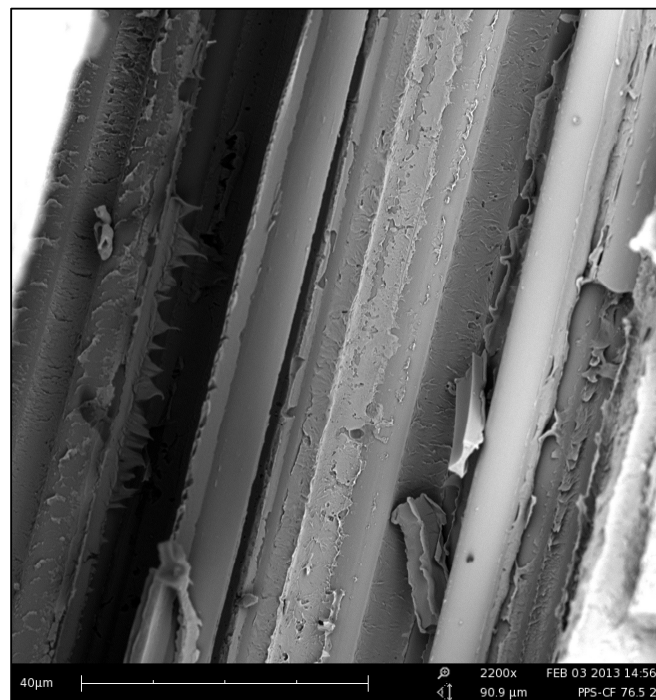


Figure 17. Short beam shear failure surface and fiber debonding of CF/PPS at 68% Vf.

The short beam shear strength of the GF/PPS material increases with glass fiber content up to a maximum of 36 MPa at a volume fraction of glass fiber, V_{gf} , of 60%, then decreases with a higher volume fraction of fiber, as shown in Figure 16. Some data points in the figure show that the short beam shear strength may continue to increase beyond V_{gf} of 60%; however, a definite reduction in shear strength from 36 MPa to 31 MPa is observed at V_{gf} of 68% and 70%. This, again, can be attributed to matrix-starved regions and an increase in void content, leading to a decrease in fiber/matrix interfacial bond strength. A parallel decrease in tensile strength and modulus at higher fiber volume fraction of this material is observed in Figure 11a,b. The optimum values of these properties also seem to occur in the same range of fiber volume fractions of 60% to 64% for the short beam shear strength.

4. Conclusions

Three continuous fiber reinforced thermoplastic composites, CF/PPS, GF/PPS, and GF/HDPE, were investigated to study the effects of high fiber content on the properties and performance of the materials. Microscopy images of samples' cross-sections revealed that uniform fiber distribution can be achieved with the pultrusion melt-impregnation process of tape and compression molding of tape layers using polymer-specific processing parameters. Minor adjustments in the processes can be made for various types of fiber and matrix materials to allow for complete impregnation of fiber roving, reduced resin-starved areas, and to prevent the introduction of voids with higher fiber content.

Results show that an increase in fiber content had no effect on the heat of melting and, consequently, no effect on the degree of crystallinity of PPS composites. No conclusion was reached about the effect on the heat of melting of the GF/HDPE composite. The glass transition temperatures, T_g , of PPS composites and the melt transition temperature, T_m , of the GF/HDPE composite increased steadily with the increase in fiber content. Glass fiber demonstrated a more significant increase in T_g than carbon fiber at higher fiber contents. Similarly, the addition of fibers increased the storage modulus, E' , with carbon fibers providing a higher increase than the glass-reinforced polymers due to the inherent high modulus of carbon fiber. The increases of T_g , T_m , and E' were linear up to the maximum fiber contents used in the study.

Maximum mechanical properties of the same CFRTP material were attained at different fiber contents. For example, the maximum tensile strength of CF/PPS occurs at V_f of 69%, while the maximum flexure strength occurs at V_f of 61%. The opposite is observed for glass-reinforced PPS and HDPE, where the optimum fiber content for maximum flexure strength is higher than that for maximum tensile strength. This indicates that the effect of fiber content is different for different constituent materials, mainly due to the different loading, failure mechanisms, and the role of fiber and matrix in resisting the induced stresses. For example, the flexure behavior of unidirectional composites is influenced by the compression properties of the fiber and matrix; in the case of CF/PPS, an increase in fiber content leads to crushing of the brittle carbon fiber and premature failure in compression, and in the case of GF/PPS and GF/HDPE, the relatively ductile glass fiber can sustain higher compressive strain, but more fiber content is needed to boost the compression strength of the composites.

The optimum V_f to achieve maximum tensile strength and tensile modulus for CF/PPS is 68–69%, and for GF/PPS it is 63–64%. This indicates better melt impregnation of carbon fibers by the PPS polymer at both the tape processing and panel processing stages. In addition, the small diameter of carbon fiber allows higher fiber packing and a high surface-to-volume ratio, leading to better fiber/matrix interaction at higher fiber content. On the other hand, the larger diameter of the glass fiber inhibits efficient fiber packing and wet-out, thus decreasing the fiber bond and lowering the tensile strength and modulus of the GF/PPS at higher fiber content. For the GF/HDPE, the optimum fiber content is 56% for maximum tensile strength and 64% for maximum tensile modulus because strength depends on the fiber/matrix bond, which proved to be weak for this material. The modulus

depends on the contribution of the high-stiffness fiber; that is, higher fiber content is needed for a higher modulus.

The ultimate strain of the PPS and HDPE thermoplastic polymers decreased steadily with the addition of fibers, indicating loss of ductility with high fiber content as the behavior becomes dominated by the high stiffness and brittle fiber. Similarly, high fiber content lowers the short beam shear strength of CF/PPS and GF/PPS due to a decrease in interlaminar and fiber/matrix bond strength. The decrease in strain and short beam shear properties may play a role in the decrease in other mechanical properties beyond the optimum fiber content.

It is concluded that an increase in fiber content beyond the common industry practice, currently at ~60% Wf, leads to higher mechanical properties and enhanced performance of continuous fiber reinforced thermoplastic composites. Efficient contribution of high fiber content can be achieved with minor improvements in and proper adjustments of tape and part processing parameters.

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Data Availability Statement: The data presented in this study are available on request from the corresponding author. The data are not publicly available due to privacy concerns from the manufacturer.

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Conflicts of Interest: Author David Eastep is employed by the company Celanese. Author Saeed Ziaee is currently employed by the company Baxter. The remaining authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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