Effect of Nanosilica on the Mechanical and Thermal Properties of Carbon Fiber/Polycarbonate Laminates

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Abstract: An investigation of nanosilica (SiO₂), influencing the mechanical and thermal attributes of carbon fiber (CF)/ polycarbonate (PC) laminates, is described in this study. Polycarbonates with four different weight percentages of SiO₂ (PC- SiO_2 , 0.1, 0.3, 0.6 and 1.0 wt%) were prepared using a melt-blending technique. The PC-SiO₂ nanocomposites were then used to fabricate planar CF/PC laminates through a hot hydraulic press machine. The prepared laminates were characterized by a number of different techniques; namely, tensile tests, dynamic mechanical thermal analysis (DMTA), differential scanning calorimetry (DSC), thermal gravimetric analysis (TGA) and scanning electron microscopy (SEM). The tensile test findings revealed that when 0.6 wt% of $SiO₂$ was added to the laminate layers, the maximum tensile modulus and yield stress were achieved. The mechanical properties obtained by DMTA supported the tensile test results. It should be noted that the 0.6 wt% of SiO₂ had the highest mechanical properties. The DMTA and DSC analyses were used to measure the glass transition temperatures (T_g) of laminates. We found that with the addition 0.6 wt% of SiO₂ the T_g increased to approximately 1° C compared to 0° C for the neat CF/PC laminate, meaning that by adding up 0.1 to 0.6 nanosilica to the polymer, the value T_g first increased and then decreased. To characterize the mass loss, the thermal degradation of polycarbonate influenced by nitrogen was investigated through TGA. According to our TGA results, the highest thermal stability was achieved by adding 0.6 wt% of SiO₂ to the PC.

Keywords: Polycarbonate, SiO₂, Carbon fiber laminate, Mechanical properties, Thermal properties

Introduction

It is generally assumed that there has been a growing tendency to use thermoplastics instead of thermosets. This is suggested to be due to their noticeable merits, such as a high degree of toughness, shorter manufacturing process, lack of need for refrigerated storage and recyclability [1,2]. Moreover, due to recent ecological challenges, there has been a growing demand in industry to reduce the fraction of metallic parts through such new materials as carbon fiber reinforced polymers (CFRPs). It is worth noting that the idea was first proposed by Margossian et al. [3] and Yokozeki et al. [4].

As far as mechanical properties are concerned, many research studies have already substantiated the advantages of CFRPs over metallic materials [5-8]. It seems reasonable to assume that for the design and manufacture of CFRP composites we need to improve not only their mechanical but also their thermal properties [9]. This could be made possible through developing polymer composite materials, which can ultimately lead to the production of better CFRP [10,11]. It has also been proved that carbon and glass fibers can to a great extent; reinforce such thermoplastics matrices as polypropylene (PP), polyetheretherketone (PEEK), and polyetherimide (PEI) in the form of laminates and composites [12-15]. It is worth mentioning that, due to their outstanding properties, these sorts of polymers are now widely utilized in the aerospace industry [16]. It appears that among all the existing amorphous thermoplastic polymers, polycarbonate (PC) is known to have the most notable mechanical and thermal properties. It is also cost effective and due to its good thermal stability and impact resistance properties [17], it has a wide variety of applications in military, electrical, automotive and sports (e.g., safety helmets) industries; thus, it was used as the matrix for our composites.

It is widely believed that various kinds of nano-fillers, such as clay, nanosilica $(SiO₂)$, and carbon nanotubes (CNT), can be applied to reinforce any matrices of polymer composites. Thus, we attempted to consolidate the mechanical and thermal attributes of PC by the manufacture of CFRP from pure PC, which was accomplished through methods we had previously used for thermoplastic olefin (TPO), high impact polystyrene (HIPS) and epoxies [18-20]. Our research findings revealed that the mechanical and thermal attributes of PC could be improved by adding minerals and fibers. In the same line, Gabr et al. strove to examine the impact of organoclay on the mechanical and thermal attributes of carbon fiber/PP laminates [21]. Their results indicated that the mechanical attributes of the PP laminates could be remarkably consolidated, by approximately 67 %, with 3 % filler content of organoclay. Nanosilica is known as *Corresponding author: mfataliyan@gmail.com an inexpensive mineral and, as it enjoys a high thermal

stability, it has been widely used in various research studies and is also used commercially as a nanofiller for the reinforcement of nanocomposites [22,23]. Nanosilica is composed of tiny SiO₂ particles bound to each other through chemical bonds in aggregates resulting in larger sized particles. It is worth noting that the high surface area of these nano materials can trigger a high reactivity [24-26]. Thus, we chose it as our reinforcing filler.

In this study, nanosilica, in four different weight percentages $(0.1, 0.3, 0.6, \text{ and } 1 \text{ wt\%})$, were mixed with polycarbonate using a melt-blending method so as to create the desired nanocomposites. Then the composite laminates were made by putting four layers of carbon fiber fabric alternately between five $PC/SiO₂$ films (each less than 0.5 mm thick), which was accomplished by hot compression. The fabricated CF/PC laminates were tested through a number of techniques and analyses. A quick look at the existing literature reveals that the impact of nanosilica on the physical attributes of CF/PC laminates has not been previously examined. Our techniques included dynamic mechanical thermal analysis (DMTA), differential scanning calorimetry (DSC), thermal gravimetric analysis (TGA), tensile testing, and scanning electron microscopy (SEM).

Experimental

Materials

PC pellets were obtained from Bayer Material Science (Covestro Makrolon 2207 polycarbonate, Germany) with a melt flow index of 37 $g/10$ min (300 °C, 1.2 kg) and density of 1.19 g/cm³. SiO₂ nanoparticle powder was supplied by the TECMAN Co in Spain. The grasific gurface area of the TECNAN Co. in Spain. The specific surface area of the nanosized SiO_2 was 180-270 m²/g, the average primary particle size was 10-15 nm and the purity was greater than 99 %. The carbon fibers (T300) were obtained from the Toray Industries Co, Japan, with a typical density of 1.76 g/ cm³, an average diameter of 7 μ m and a length of 18 cm.

Preparation of PC-nanoSiO₂ Layers and CFRP Laminates

Polycarbonates with different weight percentages of nanoSiO₂ (0.1, 0.3, 0.6 and $1 wt\%$) were physically premixed, and then melt-blended using a Brabender GmbH & Co. KG. Plasticorder internal mixer (Germany) model TSE 20, possessing a Banbury type rotor design, at $250 \degree C$ at a speed of 30 rpm for 5 minutes. The blends were then placed in a 150 mm×150 mm×0.5 mm mold and heated using a hydraulic press machine (Toyo Seiki Kogyo Co., Japan) at 250 °C and under 20 MPa for 10 min, to form PC-nano-SiO₂
composites, which were 0.5 mm, think. Then, the press composites, which were 0.5 mm thick. Then the press heaters were turned off to let the sample cool off in about 2 h, while maintaining 20 MPa (cold compression). The composite laminates were made by putting 4 layers of carbon fiber fabric alternately between five $PC-SiO₂$ films inside a special mold using hot compression for the same temperature,

Table 1. Components of the manufactured laminates

Sample	$PC(wt\%)$	SiO ₂ (wt ₂)	$CF (wt\%)$	
CF/PC	79.6	0.0	20.4	
$CF/PC-SiO2$ (0.1 %)	79.5	0.1	20.4	
$CF/PC-SiO2 (0.3%)$	79.3	0.3	20.4	
$CF/PC-SiO2 (0.6%)$	79.0	0.6	20.4	
$CF/PC-SiO2 (1.0%)$	78.6	1.0	20.4	

pressure and time for all samples. The compositions of the manufactured laminates are detailed in Table 1. The obtained laminates were nominally 3±0.1 mm thick, possessed a carbon fiber content of 20.4 wt%, and had the density of 1.62 ± 0.03 g/cm³ on average. For various characterizations of the experiments, a diamond cutting wheel was used to cut the laminates into small pieces.

Characterization

The tensile tests for the CFRP laminates were carried out aligned with ASTM D-3039 using a Universal Testing Machine (Model AG-X plus series, Shimadzu, Japan). The experiment was conducted under room temperature with a crosshead velocity of 1 mm/min and a load cell of 1 kN. For the tensile test experiment, the obtained laminates were cut into specimens with the dimensions of 18×120×3 mm. Three specimens from every composition were examined and their mean values are reported.

The dynamic mechanical attributes of the CFRP laminates were determined through a DMA 8000, Perkin Elmer Co. USA. Dynamic mechanical thermal analysis (DMTA) was utilized to gauge the storage modulus, loss modulus and tan δ of the laminates. Samples with dimensions of $14 \times 7 \times$ 3 mm were heated from -100 to 200 $^{\circ}$ C with a ramping rate of 10° C/min in an atmosphere filled with nitrogen. The sample, positioned in a single cantilever mode, was fixedly pinned at one end and the other end was shaken by the bending stress at a frequency of 1 Hz with a displacement of 0.03 mm. The glass transition temperatures, T_g , of the nanocomposites, were determined by the maxima of the tan δ curves.

Differential scanning calorimetry (DSC) analysis was carried out under nitrogen flow using a DSC-1, Mettler-Toledo AG, Analytical, Switzerland. Samples weighing approximately 5 mg were sealed in aluminum pans, heated to 300 $\rm{^{\circ}C}$ for 5 min, and then cooled at room temperature for 5 min and after that reheated to 300 $^{\circ}$ C, with all phases at the same rate of 10 \degree C/min. The glass transition temperatures were specified from the second heating curves as the midpoint of the baseline change in the calorimetric curves.

The thermal stability of the CFRP laminates and their residual weights were thoroughly tested using a TGA Pyris 6, Perkin Elmer Co., USA. Around 5 mg of the specimens were heated from room temperature to 800 $^{\circ}$ C at a heating

rate of 10 °C/min under a nitrogen gas atmosphere.

The dispersion of $SiO₂$ in the polymer matrix and the morphology of the fracture surfaces obtained from the tensile tests for the CFRP laminates were investigated through a Tescan Co. (Vega-II LSH, USA) scanning electron microscope (SEM). SEM micrographs were taken at 20 kV acceleration voltages at diverse magnifications. Before SEM observation, the fracture surfaces of the specimens when the tensile tests were sputter-coated with a slim layer of gold to enhance the sample conduction and to avoid electrical charging throughout the analysis.

Results and Discussion

Mechanical Properties

Tensile testing was initially carried out to gauge the result of nanoSiO₂ on the mechanical properties of the CF/PC laminates; the mechanical properties of pure PC, pure CF/ PC laminates and $CF/PC-SiO₂$ laminates with the four different nano $SiO₂$ contents are listed in Table 2. Examination of the data in the table shows that the pure CF/PC laminate had stronger mechanical properties than the pure PC. Analysis of the trends in the mechanical properties revealed information about the effect of the different contents of nanoSiO₂ in the laminates. The incorporation of nanoSiO₂ in the PC layers led to a rise in the tensile modulus of the laminates from 2.4 to 4.7 GPa, and the yield strength improved from 82.4 to 127.4 MPa, corresponding to 92 % and 55 $\%$ enhancements, respectively, for the CF/PC-SiO₂ (0.6 wt\%) sample. This enhancement of the tensile modulus and yield stress can be ascribed to such various factors as the intrinsic stiffness of $SiO₂$, good dispersion of $SiO₂$ in the PC matrix, especially at low loading, and also some strong interfacial adhesion between $SiO₂$ and also the matrix. Beyond 0.6 wt%, the tensile modulus and yield stress decreased. Furthermore, the nano $SiO₂$ reduced the malleability of the matrix by lowering the strain at break from approximately 7 % for pure CF/PC laminate to 5.5 % for the CF/PC laminates containing 0.6 wt% of SiO₂. We suggest this is related to defects created due to the addition of the $SiO₂₇₁$.

Table 2. Mechanical properties of the PC/CF laminates obtained by tensile testing

Sample	Tensile modulus (GPa) strength (MPa) at break $(\%)$	Failure	Elongation
Neat PC	1.02 ± 0.02	37.95	~ 8.1
CF/PC	2.40 ± 0.08	108.71	\sim 7.1
$CF/PC-SiO2 (0.1%)$	3.48 ± 0.04	132.04	\sim 5.4
$CF/PC-SiO2 (0.3 %)$	4.18 ± 0.03	154.56	\sim 5.4
$CF/PC-SiO_2 (0.6\%)$	4.65 ± 0.02	171.97	\sim 5.4
$CF/PC-SiO2 (1.0%)$	3.67 ± 0.02	118.09	~ 4.5

Viscoelastic Properties

Dynamic mechanical thermal analysis is generally employed to characterize the viscoelasticity of composites, shedding light on their mechanical behavior. The storage modulus (E') refers to the amount of the energy saved elastically, whereas the loss modulus (E'') is attributed the amount of energy lost as heat in a cyclic deformation. The ratio of the loss and storage moduli is called tangent delta $(E''/E'=tan \delta)$; it quantifies the ratio of energy lost to energy stored. The elastic modulus (E') defines the relative sturdiness or the rigidity of a material. The maxima of the tan δ can be employed for the determination of the glass transition temperature (T_g) . The glass transition temperature is one of the most vital attributes of all amorphous polymers, being the center of the temperature zone where the polymer transitions from a hard, glassy material to a soft, rubbery material. Figure 1 shows the plots of E' and tan δ with increasing temperature, measured for the pristine polymer

Figure 1. Storage moduli and tan δ as functions of temperature; (a) storage modulus as function of temperature and (b) Tan δ as functions of temperature.

Table 3. Values of T_g obtained from tan δ and DSC data and the storage modulus (E') at room temperature for the various manufactured laminates

Sample	T_{g} obtained from tan δ $(^{\circ}C)$	T_{g} obtained from DSC $(^{\circ}C)$	Storage modulus (GPa)
CF/PC	152.8	134.0	1.03
$CF/PC-SiO2$ (0.1 %)	153.9	144.3	1.25
$CF/PC-SiO2 (0.3 %)$	154.1	144.8	1.25
$CF/PC-SiO2 (0.6%)$	153.8	144.0	1.38
$CF/PC-SiO2 (1.0%)$	153.6	139.8	1.23

and for the corresponding laminates containing the various amounts of nanofillers. For the CF/PC composite, two tan δ behaviors were visible. The lower tan δ peak relates to the T_g of the PC, and the higher tan δ peak relates to the PC chains whose chain mobility was hindered by the CF [28,29]. Additionally, the values of T_g , as specified by the peak temperature of the tan δ , lower temperature and the value of E' at room temperature are depicted in Table 3, for the five different samples.

As shown in Figure 1(a), the $CF/PC-SiO₂$ laminates exhibited slightly higher E' values compared with the pristine CF/PC. The E' values first increased slightly up to 0.6 wt% of $SiO₂$ and then decreased for temperatures below 150 °C. These results are consistent with the tensile stress findings. For instance, the dynamic modulus of pristine PC at 24° C at the frequency of 1 Hz was equal to 1.03 GPa and, by adding 0.6 wt% of nanoSiO₂ in the PC, the dynamic modulus of the laminate rose to 1.38 GPa. It should be noted that although the tensile modulus of the PC was 1.02 GPa, adding 0.6 wt% nanoSiO₂, could increase it to 4.65 GPa. Furthermore, by increasing the nanosilica content in the PC to 0.6 wt% (and all other contents), the T_g of the PC-SiO₂ laminates were slightly increased by approximately $1^{\circ}C$, compared to the neat CF/PC laminate. It is known that the polymer chain segments in a pure polymer are free from restraints. The results showed that by adding a bit of $SiO₂$ (0.1 wt%) to the polymer phase, T_g increased by 1 °C due to restrictions on the motion of the polymeric chains; however, according to Table 3, by adding 1 wt% SiO₂, T_g decreased, attributed to the agglomeration of $SiO₂$. The combination of fillers decreased the free volume and restricted the mobility of the PC chains, as it was shown by higher T_g values (the additive more silica may add free volume because of disruption in packing of the chain segments, decreasing T_g). The laminates containing different contents of nanoSiO₂ were also tested by DSC to compare the glass transition temperatures, T_{g} , with the values obtained by DMTA. As indicated by the data presented in Table 3, the trend of the T_{g} changes obtained from the DSC analyses of the samples were consistent with the findings achieved through DMTA analysis (this means that results are not the same, but they have the same trend). What makes this finding different is that the pure CF/PC has a T_g around 20 °C lower than that of the observed DMTA value, whereas the T_g for the various $CF/PC-SiO₂$ laminates was 10 °C lower. Generally, as shown in both the DMTA test, the changes in T_g values in the range of 0.1 to 0.6 wt% were negligible.

Thermal Stability

The thermal stabilities of the pure CF/PC and the CF/PC- $SiO₂$ laminates were investigated using TGA. The TGA curves of all investigated samples (pure CF/PC and CF/PC- $SiO₂$ laminates) are shown in Figure 2 with their derivative thermogravimetric analysis (DTGA) curves in Figure 3. For a better overview of the data obtained from the thermal studies, the following characteristics of the investigated laminates are listed in Table 4: degradation temperature at 5% weight loss, T_{onset} and temperature at which the

Figure 2. TGA curves of all investigated laminates.

Figure 3. DTA curves of all investigated laminates.

Table 4. Characteristic temperatures of CF/PC and CF/PC-SiO₂ laminates obtained from TGA and DTGA

Sample	T_{onset} (°C)	T_n ^(°C)
CF/PC	486.1	497.7
$CF/PC-SiO2(0.1%)$	489.3	521.1
$CF/PC-SiO2(0.3%)$	497.6	523.0
$CF/PC-SiO2(0.6%)$	508.1	524.4
$CF/PC-SiO2(1.0%)$	486.7	521.4

Figure 4. SEM micrographs of the PC-SiO₂ nanocomposites with (a) 0.1 wt%, (b) 0.6 wt%, and (c) 1.0 wt% of SiO_2 .

maximal degradation rate was registered in the DTGA curve, T_{peak} . Inspection of Figure 2 shows that all specimens displayed a single decomposition phase in the nitrogen atmosphere, akin to the findings for the pure CF/PC laminates. According to our TGA results, the highest thermal stability was achieved when 0.6 wt% of $SiO₂$ was added to the pure polymer layers. It was observed that the incorporation of $SiO₂$ induced thermal stabilization of the PC laminates, with a rise in the T_{onset} of up to 22.0 °C at 0.6 wt% loading
compared to the performed leminate (nume CE/DC leminate compared to the reference laminate (pure CF/PC laminate. The same pattern was observed for the T_{peak} , where the increments were larger, approximately 23.4, 25.3, 26.7 and 23.7 °C for contents of 0.1, 0.3, 0.6 and 1 wt% of SiO_2 , respectively (the thermal degradation temperatures, such as the initial temperature of degradation (T_{onset}) , increase continuously as a function of nano silica content, but

Figure 5. (a) Optical and (b) SEM micrographs for the PC/CF laminate fracture surfaces after the tensile tests, and (c) fiber orientation after tensile test on PC/CF laminate

decrease slightly at 1 wt%).

Morphological Observations

SEM analysis was conducted on the tensile fracture surfaces of the samples to examine the dispersion of the nano $SiO₂$ in the PC polymer. Figure 4 illustrates the SEM images of the PC composites filled with 0.1, 0.6, and 1 wt% nano $SiO₂$. Many spheres can be observed in these fractographs, with their density dependent on the $SiO₂$ volume fraction. This figure also shows that as the $SiO₂$ content rose to 0.6 wt% the distribution of the nanofiller in the matrix was homogeneous, but as the nanofiller content rose to 1 wt%, agglomerations occurred that led to a decrease of the mechanical strength of the nanocomposite. Moreover, the optical and SEM micrographs for the CF/PC laminate fracture surfaces after the tensile tests are presented in Figure 5.

All carbon fibers were unidirectional, and the tensile directional was the same direction with the fibers. In Figure 5(b), fibers direction (unidirectional fibers) can be seen after the tensile test and the composite failure mode under longitudinal loading (tensile fracture). In Figure 5(c), the areas marked in figure b are zoomed out; therefore, fiber orientations are quite clear.

Conclusion

In this study we focused on the influence of nanosilica on the mechanical and thermal attributes of CF/PC laminates. The focus of the work was on the experimental determination of the tensile and dynamic mechanical properties and the thermal stability of the $CF/PC-SiO₂$ laminates. The findings demonstrated that a $SiO₂$ content of 0.6 wt% resulted in laminates with the best tensile modulus, yield stress and thermal stability. The enhanced mechanical properties of the $CF/PC-SiO₂$ laminates are attributed to the consolidating effects of the nanosilica. SEM observations illustrated that at more than 0.6 wt% of $SiO₂$, the nanofiller agglomerated in the PC matrix which resulted in a drop in the mechanical strength and thermal stability of the $CF/PC-SiO₂$ laminates. Indeed, the additive $SiO₂$ nanoparticles impede the motion of matrix chain segments, which serves to the increase of T_g . The values of T_g all composites increase slightly at low nano silica content (under 0.6 wt\%) and then decrease with the increase of silica content.

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