Manufacturing and Characterization of Experimental Polycarbonate Composites with Reinforced Vapor Grown Carbon Fiber

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SUMMARY: Polycarbonate composites based on vapor grown carbon fiber (VGCF) were successfully fabricated by solution-evaporation and rolling methods. Microscopic analysis has shown that the fibers were well dispersed in a polycarbonate matrix.

Increasing the fiber content resulted in the electrical and tensile strengths decreasing in both materials. The accumulation of voids increased in the nanocomposites, which influenced a decrease in the mechanical properties of the material.

The VGCFs delayed the thermal decomposition of the PC which is likely a result of absorption, by the carbon surface, of free radicals generated during the polymer decomposition. The twinrolling process enhanced the degree of fiber orientation and improved its mechanical and electrical properties. Thin composites showed a high Young's modulus due to the surface alignment of the fibers where thin composites were more concentrated.

KEYWORD: Vapor grown carbon fiber, Polycarbonate, Nanocomposit sheets, Electrical resistivity, Mechanical properties, Decomposition

INTRODUCTION

Carbon fiber and carbon fiber reinforced composites have attracted worldwide interest as high performance materials. Not only for their light weight and high mechanical properties but also for their potential of having extraordinary electrical and thermal properties, carbon fibers are being used in the aircraft and antistatic industries as well as computer housings and exterior automotive parts where the reduction of static electrical charge is necessary [1-2].

Because of a small diameter the VGCFs cause various problems such as a difficult incorporation into a highly viscous polymer matrix and a tendency to agglomerate. This agglomeration especially occurs as fiber content increases when dispersed in a matrix during its production: carbon nanofibers are more attracted to each other than to the liquid. Some researches results indicated poor mechanical properties because of poor fiber wetting, poor fiber dispersion and the presence of voids [3-6].

Some investors also obtained well dispersed carbon nanotubes (CNTs) in a polymer by high energy sonication of a solution of polymers containing dispersed CNTs. This was followed by a solvent-evaporation method, which successfully achieved homogeneous composites [7-12]. The main objective of this study is to investigate precisely the influence of dispersion processing conditions and the analysis of the mechanical, electrical properties using a scanning electron microscopy (SEM) and a wide angle X-ray diffraction (WAXD) on the nanocomposite sheets. We have also corroborated on the mechanical and electrical properties that were a result of the thickness.

EXPERIMENTATION

The matrix used in this study is the polycarbonate (NOVAREX 7020A grade, M_w of 19000g/mol) in pellet form, which were purchased from Mitsubishi Engineering plastics. Vapor grown carbon nanofibers (VGCFs) are VGCFTM from the Showa Denko K.K Company of Japan with diameters of 100-200nm and length of 10-20 μ m. PC pellets and VGCFs were dried at 120°C in a vacuum for 6hours and 24hours, respectively to remove the water. Because VGCFs are tightly compacted they can only be infiltrated if they are precisely prepared before mixing them with the polymer matrix. We performed a sieving process at 106 μ m followed 53 μ m in order to reduce the diameter of clumps.

To obtain uniform dispersion of VGCFs, PC pellets were completely dissolved in the tetrahydrofuran (THF) for about 2hours and the VGCFs were dispersed in a separate THF beaker by sonification and the stirring process for 15minutes. Then the mixture of PC solution was added to the mixture. Sonification and stirring processes were continued for another 15minutes. Next, the solution-evaporation method and rolling processes were performed, respectively, to fabricate the nanocomposite sheets. The thickness constant was about 0.12mm using a twinroller at 150°C for 20minutes. The nanocomposite sheets's dispersion state was examined using a SEM. X-ray diffraction pattern collected from both the cast and rolling composite sheets at room temperature. The WAXD measurement used $CuK\alpha$ X-ray source radiation at 40kV and 140mA to examine the orientation of VGCFs in the nanocomposite sheets. The degree of the VGCF orientation (f_0) were then obtained using the following equation:

$$f_o = \frac{3 < \cos^2 \Phi > -1}{2}$$
 where $< \cos^2 \Phi > = \frac{\int\limits_0^{\pi/2} I_{(\Phi)} \cos^2 \Phi \sin \Phi d\Phi}{\int\limits_0^{\pi/2} I_{(\Phi)} \sin \Phi d\Phi}$

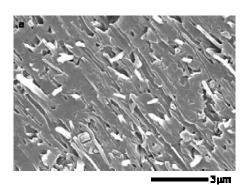
where $I(\Phi)$ is the azimuthal intensity distribution. Curves derived from the azimuth scans were fitted to the profiles of the mathematical model investigated with a Pearson VII functions [13]. The volumes of electrical resistivities were measured at room temperature by a standard four-point probe method on a digital high tester.

The tensile tests were carried out with a minimum of 10 samples. The tensile strength and the Young's modulus were obtained from the tensile test, which was carried out at room temperature, according to ISO 527-3 and performed using a Shimadzu tensile tester (AGS-J) at a cross-head speed of 1mm/min.

In order to investigate the effects of the VGCFs on the thermal properties of the matrix, thermogravimetric analysis (TGA) measurements were performed on composite sheets at all concentrations of VGCF. We used the DTG-50 DTA-TGA instruments simultaneously under a nitrogen atmosphere. 10mg of either pure PC and nanocomposite sheets were used for these measurements. The heating rate was 10°C/min at a temperature range of room temperature to 600°C.

RESULTS AND DISCUSSIONS

In order to determine the quality of the structure, Fig.1 shows the micrographs of the PC/VGCF nanocomposite sheets with VGCF loading of 10wt% for the cast and rolling nanocomposites, respectively. In the samples examined, the VGCFs were found to be evenly well dispersed and the fibers were enough to create the strong network. Voids were observed when VGCF content increases i.e. in the 25wt% composites. We concluded that the Voids diminished on the nanocomposite rolling sheet because of the rolling process. The voids could be under the influence of mechanical properties [5].



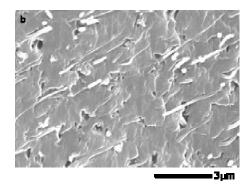


Fig.1 Dispersion of VGCF in a PC matrix; a: 10wt% cast and b:10wt% rolling nanocomposite sheets

The orientation of VGCF was analyzed using the X-ray for the cast and rolling nanocomposite sheets which were reinforced by 10wt% VGCF. The orientation was calculated to be 0.6 and 0.8 for the cast and the rolling nanocomposite sheets, respectively. We estimated from these results that the VGCFs are randomly oriented in the PC matrix and the rolling process enhanced the orientation of the VGCFs.

Fig.2 shows the TGA curves of PC and various filler contents for the nanocomposite rolling sheets. All samples started to decompose above the melting point and the masses remaining at 600°C are due to the remaining VGCFs. Comparing with the TG curves of pure PC and PC/VGCF blending are that the decomposition of materials shifted to a high temperature when increasing VGCF content. This retarding effect is likely to be a result of absorption, by the carbon surface, of free radicals generated during the polymer decomposition [14-15].

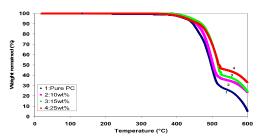


Fig.2 TGA curves of pure PC and various nanocomposite sheets

The influence of VGCF content and thickness on volume electrical resistivity was measured for the casting and the rolling composite sheets. The electrical resistivity decreases with loading. For rolling composite sheets, the high resistivity was measured only at 25wt% not 10 and 15wt%. It is estimated that the VGCF's networks were not well in a wet mixing process because of the high viscosity of the polymer unlike the cast polymer (Fig.3a).

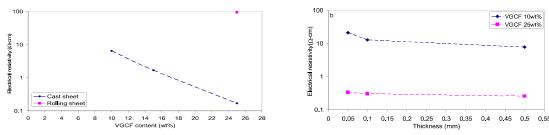


Fig.3 Electrical resistivity of PC/VGCF as a function of the fiber loading and thickness; a: casting and rolling composite sheets, b: cast composite sheets with various thicknesses.

Similar influences of the electrical resistivity are shown at various thicknesses in that the resistivity decreased with an increase of thickness in the cast nanocomposite sheets (Fig.3b). The dependence of the tensile strength and Young's modulus on fiber content for the cast and rolling composite sheets are shown in Figs.4a and b. In Fig.4a, both the maximum tensile strength of the cast and rolling composites resulted in 10wt% of VGCF. For comparison, the tensile strength of the rolling sheets was higher than the casting sheets method. There are two main reasons for this. First, the degree of orientation of VGCF for the rolling sample is higher than that of the cast sheets, determined by using WAXD. In general, the rolling procedure enhances the orientation of VGCFs and polymer chains.

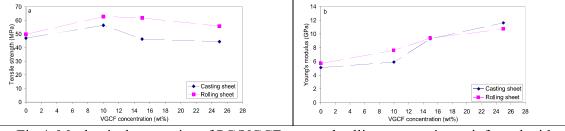
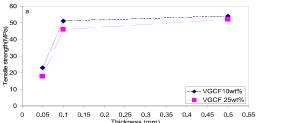


Fig.4. Mechanical properties of PC/VGCF cast and rolling composites reinforced with various amount of VGCF; a: Tensile strength; b: Young's modulus

Second, the rolling process reduces the voids. When the voids decrease, the mechanical properties should be increase. However, there is a major decrease in tensile strength with the incorporation of VGCFs. This suggests that there is a presence of voids in the PC matrix. But the Young's modulus appears largely unaffected by the addition of the

VGCFs, which results in an increases of the tensile modulus (Fig.4b).

We observed on the SEM that there were fractured surfaces on both materials, content of 25wt% of VGCF, and that the majority of the fibers were well coated by the polymer. This indicates that the load transfer from the polymer to VGCF was sufficient to fracture the VGCFs. Fig.5 illustrates the dependence of tensile strength and modulus on thickness for the 10wt% and 25wt% nanocomposite sheets. The tensile strength increases with increasing thickness. We estimated that the reinforcement fibers are probably more uniformly dispersed when the thickness or the number of stacks increases.



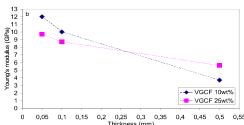


Fig.5 Mechanical properties of PC/VGCF cast composites reinforced with 10 and 25wt% of VGCF; a: Tensile strength; b: Young's modulus.

We noted that the thickness of 0.1mm is the critical point of the tensile strength. Young's modulus decreases with increasing thickness on both the composites. This decreasing is due to the surface effect of the composites. This means that the surface alignment of the fibers is higher where thin composites are more concentrated than the thick composites surfaces. This was confirmed by using a SEM of the surfaces of thin and thick composites at VGCF 25wt%.

CONCLUSION

This research work describes a successful route of the fabrication of the nanocomposite sheets containing VGCF with a homogenous dispersion in a polycarbonate matrix.

The electrical resistivity decreased with increasing VGCF loading because of a good fiber network. However, the voids were increased in the nanocomposites, which influenced a decrease in the mechanical properties of the materials.

The VGCFs delayed the thermal decomposition of PC since it is likely to a result from absorption, by the carbon surface, of free radicals generated during the polymer decomposition. The high orientation of the rolling composites fibers were recorded because the rolling process enhanced the fiber orientation and decreased the voids, suggesting the mechanical properties of the VGCF/PC composite greatly improved, especially in rolling direction. The tensile strength was increased with increasing thickness, while the Young's modulus was decreased with increasing thickness on both the composites due to the surface alignment of the fibers where thin composites were more concentrated.

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