Mechanical Properties of Vapor Grown Carbon Fiber/Epoxy Nanocomposites With Different Dispersion Methods

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Abstract : Effect of dispersion methods for Vapor Grown Carbon Fibers (VGCF) in epoxy caused the change in mechanical properties of VGCF/epoxy nanocomposites, such as tensile modulus and tensile strength. The influence of VGCF types – atmospheric plasma treated (APT) VGCF and raw VGCF – and their contents was discussed in detail. Treating VGCF with atmospheric plasma enhanced the surface energy, therefore improved the bonding strength with epoxy matrix. Two different methods used to disperse VGCF were ultrasonic and mechanical homogenizer methods.

When using dispersion solutions, the VGCF demonstrated good dispersion in ethanol in both homogenizer and ultrasonic method. The uniform dispersion of VGCF was investigated by scanning electron microscopy (SEM) which showed well-dispersion of VGCF in epoxy matrix. The tensile modulus of raw VGCF/epoxy nanocomposites obtained by ultrasonic method was higher than that of one obtained by homogenizer method. APT VGCF/epoxy nanocomposites showed higher tensile strength than that of raw VGCF/epoxy nanocomposites.

Key Words: *VGCF, Nanocomposites, Atmospheric Plasma Treatment, Ultrasonic, Homogenizer.*

1. Introduction

VGCF is a promising candidate as reinforcing nanofillers in polymer nanocomposites. They can be produced at lower price than other conventional nano carbon fillers. They are expected that the thermal, electrical and mechanical properties of these composites can be improved [1-4].

Epoxy is a widely used thermoset, with applications ranging from household glues to high-performance composites. The properties such as performance, tensile modulus, tensile strength and other properties are desirable to be improved. A number of studies have used VGCF as fillers in epoxy matrix and some successes have been achieved in the reinforcement of epoxy composites using small amounts of VGCF [5-8].

It is well established that the dispersion

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state of nanoparticles is a crucial factor in determining the final properties of nanocomposites. With conventional processing, mechanical mixing and normal processing, it is usually difficult to achieve very good particle distribution at nanoscale. Moreover, the mechanical properties of VGCF/epoxy nanocomposites with excellent dispersion increased significantly compared to nanocomposites with somewhat worse dispersion [9-12]. The processing of VGCF/epoxy matrix is still in its infancy. Although, VGCFs have been incorporated into composites commercially, the literatures described the processes are limited. Significant issues remain to be solved about dispersion and nanocomposite processing. Dispersion method has been achieved primarily by sonication of VGCF in a solvent and then both the polymer and VGCF are dispersed in solution [13-15].

The properties of the composite heavily depend on good bonding of the fibers to the matrix. In the as grown state, the surface of VGCF is essentially graphitic with low surface energy, while high surface energies are needed for fillers. Surface energy will be increased with plasma enhanced treatment. In addition to these facts, the bonding at the VGCF/epoxy interface is extremely important for the improvement of the mechanical properties with increasing amount of VGCF.

When the interface between nanofillers and epoxy matrix is increased significantly, then the mechanical properties of nanocomposites can be improved [16-23].

In this paper, epoxy nanocomposites were produced by sonication and homogenizer techniques using different kinds of raw VGCF and APT VGCF. The ethanol was used as a dispersion solution for VGCF. The effect of dispersion of the VGCF was observed by SEM. The changes of the tensile modulus and tensile strength of the VGCF/epoxy were also measured.

2. Experimental

2.1 Materials

2.1.1 Vapor grown carbon fibers

Commercially available VGCF (VGCF®-H, Show Denko KK) is used in this study. VGCF®-H is the VGCF specifically designed to enhance the electrical and thermal properties of high performance materials, as well as to improve the dispersion and homogeneity. VGCF®-H has specific surface area of 13 m^2/g, the diameter and the length of VGCF®-H are 150 nm and 10~20 μm, respectively. The real and bulk densities of VGCF were 2.0 g/cm^3 and 0.04 g/cm^3, respectively.

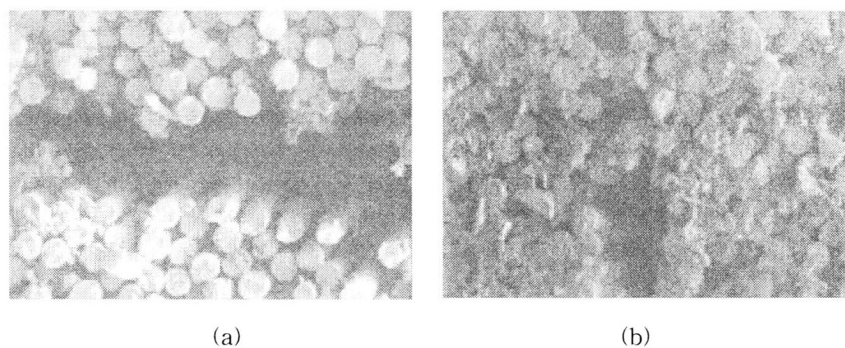(a)   (b)

Fig.1. (a) Raw VGCF and (b) VGCF with atmospheric plasma treatment.

Atmosphere plasma treatment was applied to modify the surface of VGCF in order to increase the surface energy and wettability. As a result, the adhesion at the interface between VGCF and epoxy can be improved [24].

2.1.2 Epoxy matrix

Diglycidyl ether of Bisphenol A, (YD-128, Kukdo Chemical) processed with the anhydride curing agent Methyl tetra hydro phthalic anhydride, (MTHPA, Kukdo®, KBH-1089, Kukdo Chemical). The mixing ratio of epoxy and curing agent was 100/90 (w/w)[25].

2.2 VGCF/epoxy nanocomposites by homogenizer

To the VGCF/anhydride-cured epoxy nanocomposites (up to 3 wt%), the VGCF were homogenized in ethanol at 10,000 rpm of speed for 30 minutes by using Specification® homogenizer (Global Lab). The solution contained more than 100 ml of ethanol per 1g of VGCF. The epoxy was then added and mixed by continuous mechanical mixing for an additional hour. The mixture was divided into two layers 1 hour later (the separated upper layer was ethanol). The ethanol was poured out and the remaining portion was removed by the vacuum extraction at 85°C for 2 hours. Then, curing agent was blended on the mixture

with mechanical stirring for 1 hour. The air bubbles were removed by vacuum at 70°C for 30 minutes. The epoxy matrix was injected into metal mould with vacuum. The specimens were cured at 80°C for 30 minutes, and then for 3 hours at 120°C.

2.3 VGCF/epoxy nanocomposites by ultrasonic

Sonic Mater® Sonicator (The Sonic Tech, Co. Ltd) was used for sonication during for 30 minutes, and next steps were similar to those of homogenizer method.

2.4 Tensile Test

The specimen was fabricated in accordance with ASTM D638. The specimen thickness was 3.0 mm, 12.0 mm of width, total specimen length was 160.0 mm, and the strain gauges length used was 5.0 mm. The Instron 5567 was used for tensile strength test. The machine was rated under displacement control mode with 2.00 mm/min of speed, and all tests were performed at room temperature. Five specimens were cut from each panel for each condition (Fig.2).

2.5 Scanning electron microscopy

The fracture surfaces of VGCF/epoxy nanocomposites were observed by SEM after the tensile test. Au coating of a few nanometers in thickness was applied to fracture surface.
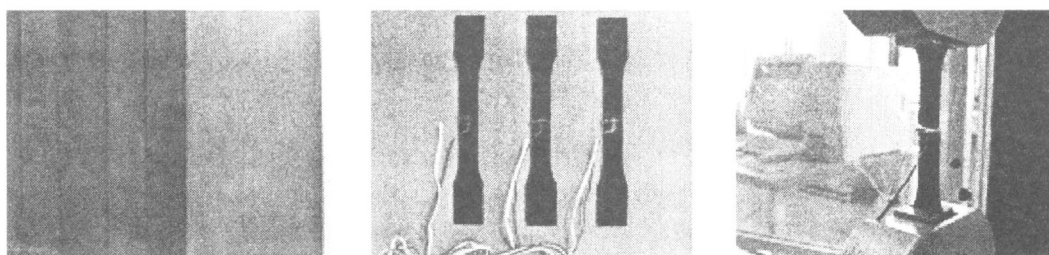

Fig. 2. Panels, specimens and machine for tensile testing.

3. Results and discussion

3.1 Tensile Modulus of VGCF/epoxy nanocomposites

Epoxy-based nanocomposites containing 3 wt% of VGCF got the maximum tensile modulus value. The relationship between tensile modulus and VGCF contents with sonic method and homogenizer method are graphically presented as filled symbols in Fig. 3. The tensile modulus of VGCF/epoxy showed a non-linear increase with increasing VGCF content up to 3.0 wt%. The tensile modulus of VGCF/ epoxy nanocomposites increased 7% and 37.5% with the addition of 1.0 wt% and 3.0 wt% of VGCF, respectively. However, lower tensile modulus increased from 4.7% up to 29.0% by using homogenizer method at the same VGCF contents. As expected, with small amounts of VGCF, the improvement was greater with dispersion by ultrasonic method.

The tensile modulus of APT VGCF and raw VGCF/epoxy had similar value with ultrasonic method.   However, lower tensile modulus of VGCF/epoxy matrix was obtained by homogenizer method (Fig. 4.).
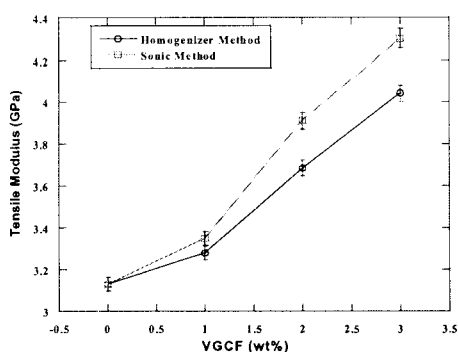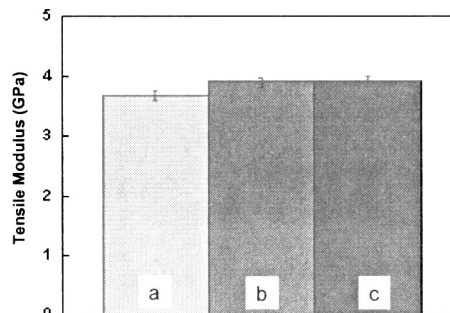
Fig. 4. The tensile modulus with 2.0 wt% VGCF, (a) raw VGCF with homogenizer method, (b) raw VGCF with ultrasonic method, (c) APT VGCF with ultrasonic method.

3.2 Tensile strength of VGCF/epoxy nanocoposites

The tensile strength-strain curves of the nanocomposites containing 0, 1, 2 and 3 wt% raw VGCF with ultrasonic method are shown in Fig. 5. The observed behaviour was continuous increase of tensile strength with strain until rupture point. The tensile strength increased with VGCF content. The strain decreased with increasing VGCF content. However, the tensile strength-strain curves showed considerable nonlinearity before reaching the maximum stress. For only 3.0 wt% of VGCF, the tensile strength of raw VGCF/epoxy nanocomposites at 25000 μm of strain increased about 22.9%, but the strain decreased compared with pure epoxy.

The tensile strength-strain cures of nanocomposites with 2.0 wt% APT VGCF and raw VGCF obtained by ultrasonic method and mechanical homogenizer method are shown in Fig. 6. The maximum tensile strength of raw VGCF/epoxy matrix by ultrasonic method showed higher value than that of homogenizer method.

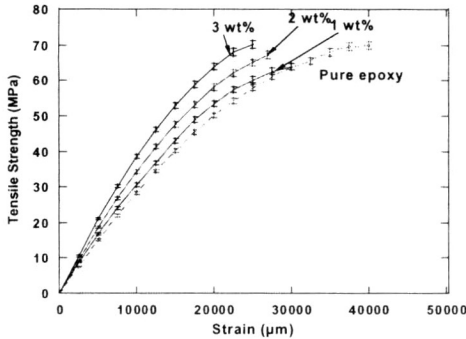Fig. 3. The tensile modulus of raw VGCF/ epoxy matrix with different dispersion method.

Fig. 5. The relation between the tensile strength, raw VGCF with ultrasonic method.
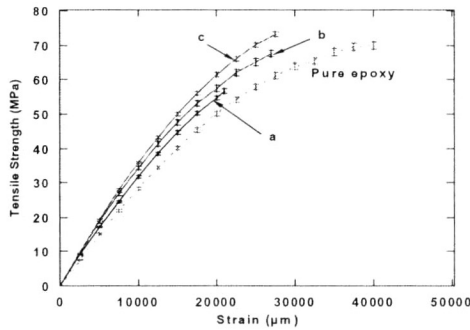

Fig. 6. The tensile strength vs strain with two dispersion methods, 2 wt% VGCF contents: (a) raw VGCF with homogenizer method, (b) raw VGCF with ultrasonic method, (c) APT VGCF with ultrasonic method.

The surface of APT VGCF was improved the bonding at the interface between VGCF and epoxy matrix. The tensile strength for 2.0 wt% APT VGCF at maximum point was increased by 7.1% than that of pure epoxy. At the same VGCF content, tensile strength was decreased by 10.9% for raw VGCF and the strain was decreased compare with pure epoxy. Correspondingly, the APT VGCF had an excellent interfacial bonding with epoxy matrix.

3.3 Scanning electron microscopy micrographs.

The low magnification SEM showed an excellent dispersion of raw VGCF in epoxy matrix with homogenizer method and with ultrasonic method (Fig 7(a) and 7(b)), as well as, APT VGCF obtained by ultrasonic method showed excellent dispersion (Fig. 7(c)).

In Fig. 8(a), the high magnification SEM photographs of the fracture surfaces shows the exposed pulled-out VGCF's all over the fracture surfaces. This implies that the adhesion between the raw VGCF and epoxy matrix was weak. A large portion of VGCF's were easily pulled-out without breakage under tensile test. Actually, several holes after pulled-out of VGCF can also be seen on fracture surfaces.

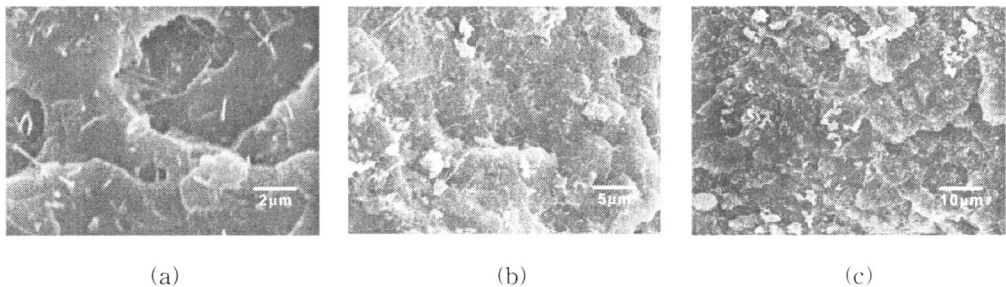(a)   (b)   (c)

Fig. 7. SEM micrographs revealing fracture surfaces: (a) homogenizer method, 2 wt% raw VGCF, (b) ultrasonic method, 2 wt% raw VGCF, (c) sonic method, 2 wt% APT VGCF.
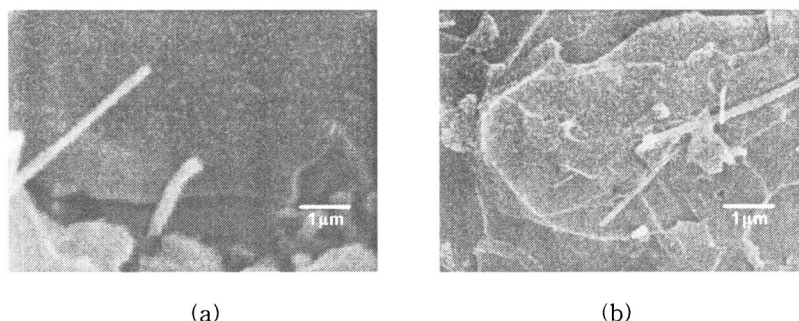
(a)   (b)

Fig. 8. SEM micrographs revealing fracture surfaces with 2.0 wt%
VGCF, ultrasonic method. (a) raw VGCF, (b) APT VGCF.

A large number of VGCFs were cracked on the fracture surfaces, stronger adhesion at the VGCF/epoxy interface was obtained from the APT VGCF, which created higher surface area on the VGCF surfaces due to very high interfacial bonding strength, which resulted in higher tensile strength value of APT VGCF/epoxy nanocomposites than that of raw VGCF/epoxy nanocomposites (Fig. 8b).

result, the tensile strength of APT VGCF/epoxy nanocomposites was higher than that of raw VGCF.

Acknowledgement

4. Conclusions

In this study the mechanical properties of epoxy nanocomposites reinforced by VGCF and the effect of dispersion methods for VGCF in epoxy matrix were investigated. The ultrasonic method and homogenizer method are used to produce well-dispersed raw VGCF and APT VGCF in the anhydride-cured epoxy matrix. However, with mechanical homogenizer method, the tensile modulus and tensile strength were lower than with the case of ultrasonic method.

The atmospheric plasma treatment appears to have a positive effect in improving the interfacial adhesion between VGCF and epoxy matrix. Various adhesion mechanisms can be promoted by atmospheric plasma treatment, the increased surface area and and physic bonding resulted in the improvement of mechanical properties of nanocomposites. As

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