

Effect of particle size on the mechanical properties of polymer matrix nanocomposite

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ABSTRACT

In the present investigation, we have studied effect of inclusions size on mechanical properties of nanocomposite materials containing SC-15 epoxy resin and TiO₂ particles. The size of particles varied from macro (0.02mm) to nano (5nm) scale. The particles were infused into the part A of SC-15 through sonic cavitations and then mixed with part B of SC-15 using a high speed mechanical agitator. The trapped air and reaction volatiles were removed from the reaction mixture using high vacuum. Three-point bending tests were performed on unfilled, 0.5wt.%, 1.0wt.% and 1.5wt.% TiO₂ particles filled SC-15 epoxy to identify the loading effect on mechanical properties of the composites. Experimental results show that, micro sized particles have little effect on modulus and strength of epoxy at such low loading, and modulus and strength increased as the size of particles decreased to nano scale. Results also show that 1.0wt% nanoparticles reinforced system exhibit the highest mechanical performance. Higher than 1.0%, strength of composite decreased because of poor dispersion.

Keywords: Nanoparticles, Size, Strength

1 INTRODUCTION

The incorporation of inorganic particulate fillers has proved to be an effective way of improving the mechanical properties, such as modulus and strength, of resin. However, the typical filler content needed for significant enhancement of these properties can be as high as 10-20% by volume. At such high particle volume fractions, the processing of the material often becomes difficult, and since the inorganic filler has a higher density than the resin, the density of the filled resin is also increased. Nanoparticle filled resins are attracting considerable attention since they can produce property enhancement that are sometimes even higher than the conventional filled polymers at volume fractions in the range of 1 to 5%. It has been established that with the addition of small amounts of nano-particles (<5 wt. %) to a matrix system can increase thermal and mechanical properties without compromising the weight or process-ability of the composite [1-5]. These polymer-based nanocomposites derive their high properties at low filler volume fractions owing to the high aspect ratio

and high surface area to volume ratio of the nano-sized particles. According to Reynaud et al. [16], an interface of 1 nm thick represents roughly 0.3% of the total volume of polymer in case of micro particle filled composites, whereas it can reach 30% of the total volume in case of nanocomposites. However, high specific surface area causes strong tendency to agglomerate, which reduces the strength of nanocomposite by stress concentration effect.

The effect of particle size on mechanical performance of composites has been addressed by many authors. Leidner and Woodhams [14] demonstrated that the tensile strength of glass beads/polyester composites increases as the size of glass beads decreases. Ng et al. [19] measured tensile properties and scratch resistance of TiO₂ (32 nm)/epoxy nanocomposites and compared them to those of composites with micron size TiO₂ (0.24 μ m). In comparison, higher failure strain and scratch resistance were observed in the nanocomposite [6]. Flexural modulus and strength measurements were performed with alumina (Al₂O₃) particles of 40 nm, 1 μ m and 3 μ m in diameter with a vinyl ester resin [7]. The flexural modulus was not affected by the particle size, but the strength was found to be lowered as the particle size decreased. The decrease in strength relative to the particle size was attributed to poor dispersions of nanoparticles in the composite.

In this study, TiO₂ particles with diameters of 5nm, 10nm and 30nm will be filled in SC-15 epoxy with different weight fraction. Flexural test will be used to evaluate the mechanical properties of neat and nanophased epoxy. Fracture surface and failure mechanism will be studied by using SEM.

2 EXPERIMENTAL

The resin used in this study is a commercially available SC-15 epoxy obtained from *Applied Poleramic, Inc.* It is a low viscosity two phased toughened epoxy resin system consisting of part-A (resin mixture of Diglycidylether of Bisphenol-A, Aliphatic Diglycidylether epoxy toughner) and part-B (hardener mixture of, cycloaliphatic amine and polyoxylalkylamine). The particles diameters are 5nm, 10nm, 30nm and 20 μ m. The weight fraction of particles is 1.0wt.%.

Pre-calculated amount of particles and part-A were carefully weighed, and mixed together in a suitable beaker.

The mixing was carried out through a high intensity ultrasonic irradiation (Ti-horn, 20 kHz *Sonics Vibra Cell, Sonics Mandmaterials, Inc, USA*) for half an hour with pulse mode (50sec. on/ 25sec. off) since part-A is insensitive to ultrasound irradiation. To avoid a temperature rise during the sonication process, external cooling was employed by submerging the beaker containing the mixture in an ice-bath. Once the irradiation was completed, part-B was added to the modified part-A then mixed using a high speed mechanical stirrer for about 10 minutes. The mix-ratio of part A and part B of SC-15 is 10:3. The rigorous mixing of part-A and part-B produced highly reactive volatile vapor bubbles at initial stages of the reaction, which could detrimentally affect the properties of the final product by creating voids. A high vacuum was accordingly applied using *Brand Tech Vacuum* system for about 30 minutes. After the bubbles were completely removed, the mixture was transferred into a plastic and Teflon coated metal rectangular molds and kept for 24 hours at room temperature. The cured material was then de-molded and trimmed. Finally, test samples were machined for thermal and mechanical characterization. All as-prepared panels were post-cured at 100°C for five hours, in a Lindberg/Blue Mechanical Convection Oven.

Flexural tests under three point bend configuration were performed according to ASTM D790-86. The tests were conducted in a 10 KN servo hydraulic testing machine (MTS) equipped with Test Ware data acquisition system. The machine was run under displacement control mode at a cross head speed of 2.0 mm/min, and all the tests were performed at room temperature. Test samples were cut from the panels using a Felker saw fitted with a diamond coated steel blade. Five replicate specimens from four different materials were prepared for static flexure tests.

3 RESULTS

The stress strain curves of 5nm nanoparticle reinforced epoxy with different weight fraction are shown in Figure 1. The stress-strain curves show considerable non-linearity before reaching the maximum stress. Each curve shows a maximum stress, which is assumed to be the flexural strength of the material. For neat and 0.5% system, specimens failed when the stress reach the tensile strength. But for 1.0% and 1.5% system, after flexural strength point, stress decreased steadily with strain until fracture occurred.

Figure 2 shows the variation of strength with particles content. Optimal loading of particles was found at 1wt. %. Figure 2 also showed the influence of particle on strength of nanocomposite. The manner in which flexural strength changed with particles size can be seen more clearly by plotting σ_b as a function of diameter of particles (Figure 3). At the same weight fraction (1%), flexural strength showed maximum value in 10nm nanoparticle reinforced epoxy.

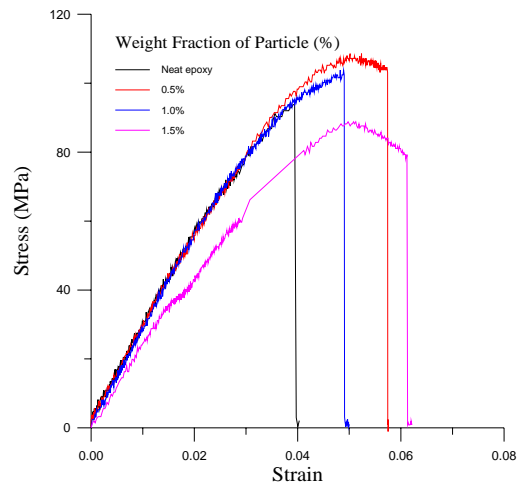


Figure 1 Stress strain curves of 5nm particle reinforced epoxy

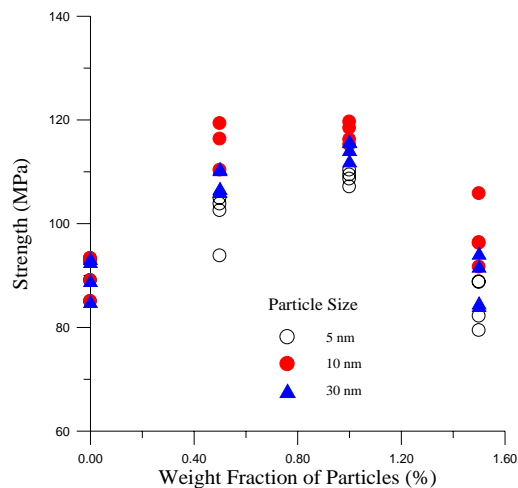


Figure 2 Relationship between flexural strength and weight fraction

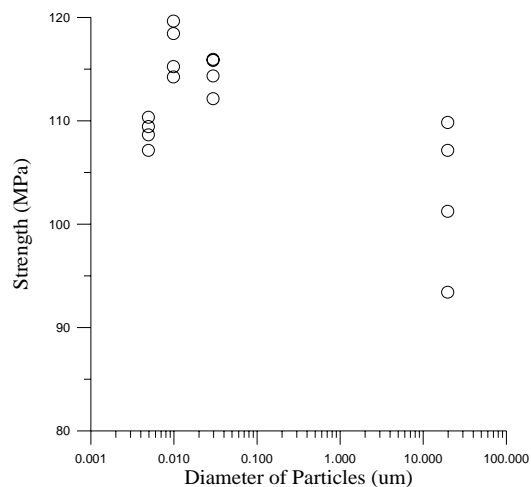
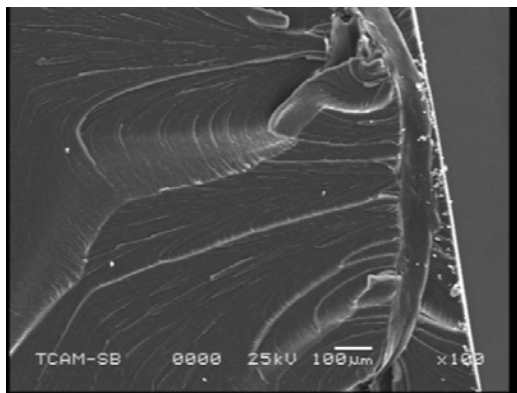
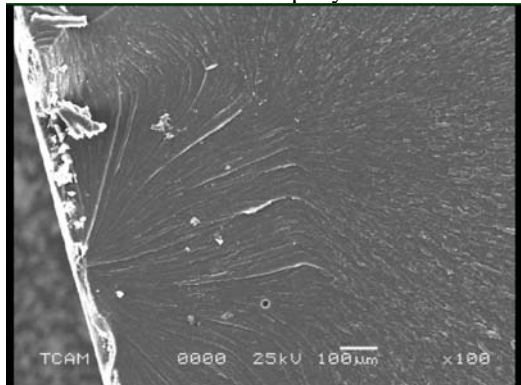


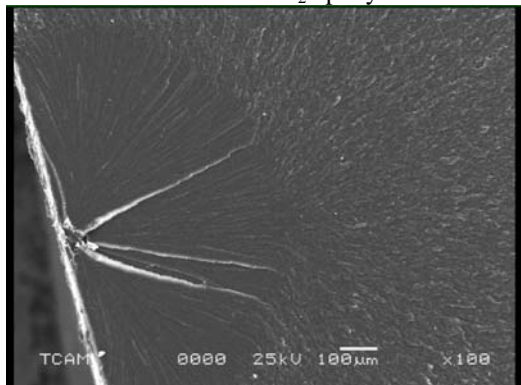
Figure 3 Effect of particle size on flexural strength



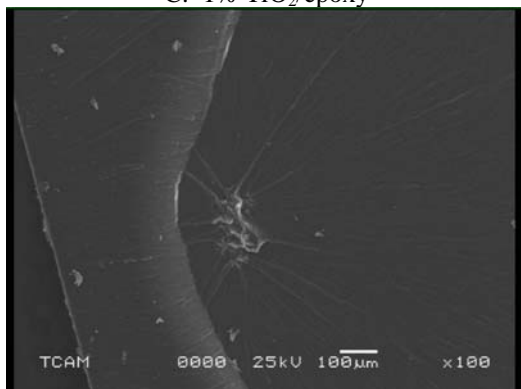
A: Neat epoxy



B: 0.5% TiO₂/epoxy

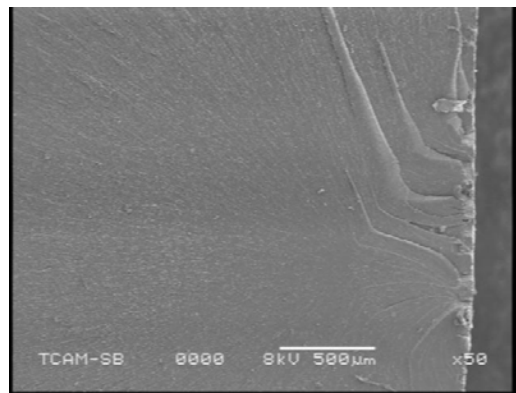


C: 1% TiO₂/epoxy

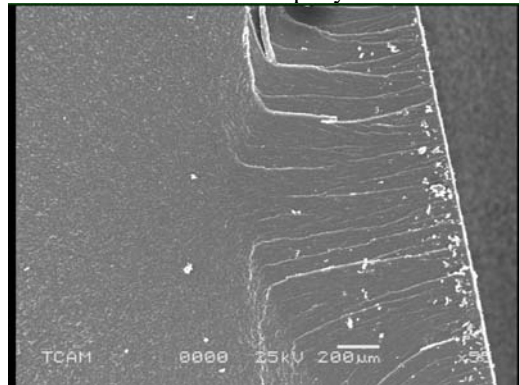


D: 1.5% TiO₂/epoxy

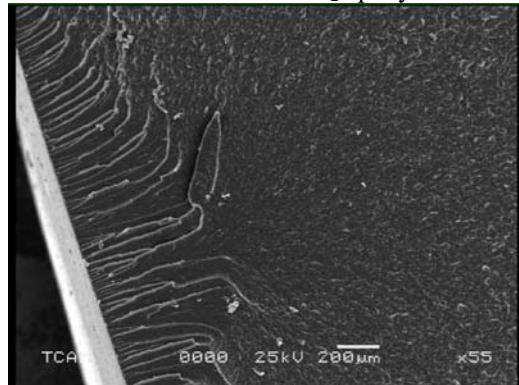
Figure 4 Fracture surface of neat and nanophased epoxy



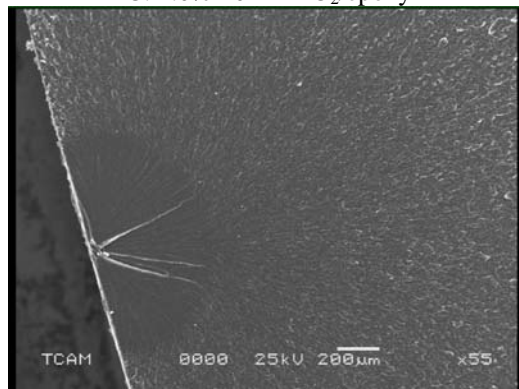
A: Neat epoxy



B: 1.0% 30nm TiO₂/epoxy



C: 1.0% 10nm TiO₂/epoxy



D: 0.5% 5nm TiO₂/epoxy

Figure 5 Fracture surface of neat and nanophased epoxy

The fracture surfaces of neat epoxy and the nanocomposites were comparatively examined using SEM. It can be seen in Figure 4a that neat epoxy resin exhibits a relatively smooth fracture surface and initial crack occurred at the tension edge of specimen. Figure 7a indicates a typical fractography feature of brittle fracture behavior, thus accounting for the low fracture toughness of the unfilled epoxy.

Compared to the case of neat epoxy, the fracture surfaces of the nanocomposites show considerably different fractographic features. As a representative example, the failure surface of the nanocomposite containing 0.5 wt % and 1.0 wt % nanoparticle are shown in Figure 4b and 4c. Generally, a much rougher fracture surface is seen upon adding nanoparticle into the epoxy matrix. The increased surface roughness implies that the path of the crack tip is distorted because of the nanoparticles, making crack propagation more difficult.

When the particle content increased to 1.5 wt %, failure mode of nano composite changed. The initial crack did not occur at the tensile edge, but in the middle of specimen (Figure 4d). The failure was initiated at a large particle that appears to be an agglomeration of several nanoparticles. The crack initiation was caused by the stress concentration caused by the agglomerated particle.

Figure 5a-d show the effect of particles on micrograph of epoxy. When diameter of nanoparticle decreased to 5nm, failure of specimen initiated at one point. The decrease in strength in 5nm system was attributed to poor dispersions of nanoparticles in the composite.

4 CONCLUSION

Ultrasonic cavitation has been used to infuse nanoparticles into the SC-15 epoxy resin. Flexural tests and SEM results show that strength of materials increase with decreasing of particles size until 10nm. The decrease in strength in 5nm system was attributed to poor dispersions of nanoparticles in the composite. Results also show that optimal loading of nanoparticle is 1.0%, high than this value, strength decreased due to stress concentration effect of agglomerated particles.

Acknowledgements

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