THERMAL AND MECHANICAL PROPERTIES OF MICROWAVE CURED SiC/ EPOXY NANOCOMPOSITES

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Abstract
In the present research work, curing of epoxy resin reinforced with SiC nanoparticles were studied by using both traditional thermal curing and microwave irradiation technique. Comparisons of thermal and mechanical properties of these nanocomposites were carried out using Differential Scanning Calorimetry (DSC), Thermogravimetric Analysis (TGA), Dynamic Mechanical Analysis (DMA), Thermal Mechanical Analysis (TMA) and flexural tests. The fracture surface and failure pattern were studied using Scanning Electron Microscopy (SEM). 1% loading of SiC has shown increased mechanical properties in terms of flexural modulus, strength, and maximum strain to failure as compared to the thermally cured nanocomposites. The curing time was drastically reduced for microwave cured for ~30 minutes instead of 12 hours room temperature curing with additional 6 hours post curing at 75°C. However, the maximum strain to failure was increased by 25%-40% for microwave-cured nanocomposites over the room temperature cured samples for corresponding loading of nanofillers. Ductile behavior was more pronounced for microwave cured samples while thermally cured samples showed brittle behavior. The glass transition temperature ($T_g$) was also increased up to ~14°C for microwave cured samples.

1 Introduction

In manufacturing of thermoset polymer nanocomposites, polymer curing is one of the concluding phases to obtain the final product. Therefore, degree of curing play important role on the properties of thermoset polymers. Most of the times a post curing is needed to complete cure and obtain the optimum benefit from the polymer matrix in terms of mechanical, thermal, or electrical properties. If the curing is not completed, it may cause the loss of adhesion in the final product because of presence of solvent, moisture, or un-reacted monomer. External energy and/or catalysts are needed to introduce the polymer chain to react chemically active sites linking into rigid, three-dimensional structures. The curing reaction propagates exothermically to form this 3D network and finally transformed into solid. The field of nanotechnology is still unrefined in certain aspects, curing is one of them. The conventional method of curing is time-consuming. Alternate curing methods have been tried and tested for quite some time. The most prominent alternatives suggested are Electron Beam (EB) and Microwave heating [1]. Since the EB curing is expensive, the microwave curing is viable alternate. The microwave interact with materials through either polarization or conduction process due to dielectric properties of materials. Silicon carbide (SiC) is one of such ceramic nanofillers. SiC nanoparticles are also extensively used because some of its inherent characteristics such as high heat impact, radiation, and oxidation resistance, high chemical stability, catalyst support, strengthening and increasing super plasticity of materials, wear resistance and so on. Silicon carbide (SiC) based polymer nanocomposites have shown remarkable improvement in response to thermal and mechanical properties [2-4] because of its inherent distinctive features.

In microwave curing molecules with permanent dipole momentum, try to align in the direction of electromagnetic field, their rotation, friction, collision cause tremendous heat generation within a very short period of time. Thus, microwave offer a positive effect on polymers curing because of its selective heating with faster curing. Microwave
Irradiation technique is believed to heat the whole system simultaneously. Chaowasakoo and Sombatsompop [4] studied on mechanical and morphological properties on fly ash/epoxy and observed increased mechanical (tensile, flexure, impact) properties with the increasing the percentage of loading of treated fly ash with epoxy resin and also compared between conventional and microwave cured specimen. They observed that microwave curing resulted shorter curing time with higher strain to failure of the composites, although the strength and modulus were relatively lower than those from conventional curing specimen. Previously it was hypothesized that the presence of microwave absorbent materials weakens the localized superheating when microwave in incident because of absorbing the microwave energy during the curing process and the curing rate is required to be decreased with higher loading of nanoparticles into matrix. It is well known that CNT, SiC etc. are excellent microwave absorbents. In recent years, several studies have been conducted to investigate the interaction between carbon nanotubes (CNT) and microwave irradiation [5-7]. Wang et al. [8] successfully attached multi-walled carbon nanotube (MWCNT) using microwave on polyethylene terephthalate (PET), polycarbonate (PC) and polyimide (PI) substrates. This ‘microwave-welding’ was accomplished in 1-5 second of microwave irradiation. They obtained the anisotropic alignment of MWCNT on the substrates by unidirectional stressing. The bulk substrate was found to be unchanged, although high temperature was generated which may be because of short time of heat conduction during the attachment without damaging the chemical bonding of substrates. Rangari et al. [9] studied on the curing behavior of nanocomposite by infusing CNT into epon 862 epoxy resin system and monitored that with microwave irradiation, curing time can be reduced drastically to 10 minutes instead of 8 hours of conventional thermal curing without compromising mechanical and thermal properties. Moreover, they observed increased glass transition temperature (Tg) and strain to failure for the microwave cured nanocomposites.

2. Experimental:

2.1 Materials:
Silicon carbide (SiC) nanoparticles (less than 30 nm) were β-SiC (99.7% pure) purchased from MTI Corporation, USA. OctaIsobutyl (OI) POSS were supplied by the Hybrid Plastics Company, Fountain Valley, California. Epoxy SC-870 was obtained from Applied Poleramic Inc. The matrix system in this study is a commercially available, low viscosity, two part system SC-780 epoxy resin purchased from Applied Poleramic Inc. (API), USA. This SC-780 toughened epoxy resin system was specifically developed for vacuum assisted resin transfer molding (VARTM) process. The recommended temperature for infusion is at 75-80°F. The curing can be performed at 77°F overnight or at 100°F for two hours subsequently post cured at 160°F-170°F for six hours to achieve maximum mechanical and thermal properties.

2.2 Synthesis of Nanocomposite:
The pre-calculated amount of nanoparticles (SiC) and part A of epoxy SC-780 was weighed carefully and mixed together in a 400 ml plastic beaker. The beaker was then placed in a coolant circulating at a constant temperature of 0°C to control the heat generation by sonication. Then mixing was carried out using a high intensity ultrasonic irradiation of sonic vibra cell with Ti horn for 60 minutes in a pulse mode of 20 second on and 10 second off at amplitude of 35%. This time, pulse and amplitude were adjusted after several trials of mixing under ultrasonic irradiation. It is noticed that sonochemical mixing produced a huge number of bubbles inside the nanofiller modified part A of epoxy resin. Therefore the after sonication, the mixture was kept in vacuum desiccators for another hour to remove the entrapped voids. Just after the degasification, the part B which is amine base hardener were added with the nano-modified part A of SC-780 epoxy resin in a container specified for Thinky deforming mixer. THINKY hybrid deforming mixer ARE-250 was used for noncontact homogenous mixing of modified part A and part B. In this technique the material container is set at 45 degrees angle inside the mixer and revolves and rotates at high acceleration with the speed of 2000 rpm for 15 minutes. These dual centrifugal forces were given to
the container that keep pressing materials to outward and down along with the slope of inner wall of the container and accomplish powerful mixing and de-aerating simultaneously.

After that the matrix system containing the nanoparticles were again kept in desiccators for 15 minutes to remove the volatiles from the mixture. Immediate after the matrix were placed in steel flexure mould (according to ASTM standards). The samples in the mould were allowed in a thermal oven at 25°C for 12 hours. After that the samples were post cured at 75°C for 6 hours to complete the curing cycle. For curing under microwave heating, the nanofiller infused epoxy matrix were transferred into Teflon pan and cured under microwave irradiation in two phases of curing cycle. SiC are good microwaves absorbent which produce extremely high temperature while absorbing microwave resulting over burning of epoxy based matrix nanocomposites. Therefore after many trials the procedure was set by splitting the steps of curing into two phases for microwave synthesis with the simple technique that is just placing Teflon pan with matrix into it. In first step mixture was heated using microwave irradiation at 20°C for 90 seconds and then kept it rest for 15 minutes. In second step at same temperature microwave irradiation was carried out for another 60 seconds and rest 20 minutes and relatively superior quality microwave cured samples are obtained. These multistep heating cycles enable to reduce the generation of volatiles during the reaction. It is noticed that with 1.5% loading of SiC into epoxy resin, resulting complete burnt samples due to high microwave absorbance by SiC. After de-molding and trimming according to standard specifications, the samples are ready for mechanical and thermal characterization.

3. Results and Discussion

Figure 1 represent the DSC curves of microwave cured (MC) SiC infused SC-780 epoxy resin system at different loading. Similar trends of the curves were observed with the adding of SiC nanoparticles in SC-780 epoxy and thermally cured at room temperature. The Tg was increased with respect to neat system. The measured Tg was found to be 89°C to 91°C for neat and nanophased epoxy system which are higher than those obtained from the traditional thermal cured nanocomposites. It is well known that Tg increases with the increasing the cross-linking density and decreasing molecular mobility [3]. The addition of SiC nanoparticles up to an optimum level, impart such contribution-causing enhancement in Tg. With higher loading (1.5 %) of SiC, Tg is almost equal to that of neat SC-780 cured under microwave irradiation. This decrease is may be because of: a) agglomeration, b) effect of localized heating. As SiC absorb high microwave radiation, the localized heating caused overheating, resulting the burning of samples and damaging bonding properties of matrix.

![Stress-Strain curve from flexural test for microwave cured nanocomposites](image-url)

The flexural test graphs for microwave-cured nanocomposites are shown in figure 2 for different weight fraction of SiC. This figure also shows the similar trend as stress strain curve for conventional cured epoxy nanocomposites. For SiC loading the flexure strength and modulus increased up to 1% weight fraction. However, for 1.5% loading of SiC, both flexure strength and modulus were decreased because of over burning, soldering or over welding effect [8,9].
4. Conclusions

Microwave processing is successfully used in producing epoxy-based polymer nanocomposites infused with SiC nanoparticles. In this technique, the nanocomposite curing time is drastically reduced from 18 hours to 30 minutes without compromising the thermal and mechanical properties. The flexural strength and modulus of microwave-cured samples were increased by 12% and 9.5% respectively as compared to the room temperature cured nanocomposites with 1% loading of SiC. For microwave cured specimen, the maximum strain to failure were increased by 25-40% for 0.5% and 1% loading of SiC with respect to neat epoxy cured at room temperature without compromising the flexural strength and modulus. The glass transition temperature for room temperature cured nanocomposites was found to be increased by maximum 7°C for 1% SiC loading.

References