Synthesis & Mechanical Characterization of Carbon/Epoxy Composites Reinforced with SiC Nano Particles

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ABSTRACT

Room temperature cure epoxy resin has been doped with SiC nanoparticles through an ultrasonic cavitation process. The modified resin was then utilized in a Vacuum Assisted Resin Transfer Molding (VARTM) process with satin weave carbon fibers to manufacture nanophased composite panels. The nanoparticles were spherical in shape and about 29 nm in diameter. The loading of nanoparticles into the resin ranged from 1.5 to 3.0 percent by weight. The mixing of nanoparticles with Part-A of SC-15 resin was carried out in a *Sonics Vibra Cell* ultrasonic liquid processor at 55% of the amplitude for about 30 minutes. At this time, the dispersion of nanoparticles seemed uniform through visual observation. In order to avoid rise in the temperature during sonication, cooling was employed by submerging the mixing beaker in a mixture of ice and water. Part-B (hardener) was then added with the mixture at a ratio of 3:10 and the mixing was carried out mechanically for about 10 minutes using a high speed mechanical stirrer. In the next step, the mixture was used in a VARTM set up with satin weave carbon preforms to fabricate nanophased composite panels. Once cured, test coupons were extracted and subjected to both quasi-static and dynamic loading. Under quasi-static loading tensile and flexural response were of particular interests. Dynamic tests were also carried out at low velocity impact and at high rates of strain. Details of manufacturing, analyses of mechanical tests and enhancements in properties due to nanoparticle infusion are discussed in the paper.

Keywords: nanocomposites, carbon/epoxy, SiC nano scale fillers, reinforced composites, nanotechnology

1.0 INTRODUCTION

Mineral fillers, metals, and fibers have been added to polymers for decades to form composite materials [1-5]. Compared to neat resins, these composites have a number of improved properties including strength, heat distortion temperature, and modulus. As a result, for structural applications composite materials have become an essential entity. With the advent of the new millennium, and with our relentless quest for lighter and stronger materials, the demand for materials has taken a new dimension. No longer are the traditional composite materials capable of satisfying our stringent requirements, nor can they be engineered to control properties at the atomic or molecular levels. The essence of such control in properties has derived from the fact that the aggregate properties of materials under external excitations such as force, pressure or temperature, are largely dictated by their molecular level orientation. Eventually, materials which are fashioned at the molecular level will be highly sophisticated to be engineered according to specific requirements. It is surprising to know that the newly developed material system known as "*nanocomposite materials*" offers the ability to build at the molecular level to create large structures with fundamentally new molecular organizations.

The ability to reorganize materials at the molecular level is what makes nanocomposites an attractive tool for fabricating materials to meet our stringent requirements for materials with enhance mechanical and thermal properties. What makes nano scale building blocks attractive in polymers, is the extremely high surface area which is created by the nanoparticles when interaction takes place with the polymer chain. This interaction creates large interfaces in a composite therefore enhances adhesion energy which translates into increase bonding. This increase in chemical bonding improves the polymer cross-linking and enhances mechanical and thermal properties. An interphase 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 [6]. A negligible contribution made by the interphase provides diverse possibilities of performance tailoring and is able to influence the properties of the matrices to a much greater extent under rather low nano-filler loading. Significant improvement in the tensile properties of polypropylene composites has also been reported in terms of stiffening, strengthening and toughening with a low filled content of about 0.5% [7]. Other studies

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have shown that by using silica clay platelet nano-fillers in polymers, thermal properties can be improved significantly when compared to conventional filled polymers [8].

While these studies revealed significant findings, in a variety of areas such as tensile modulus, strength, and thermal stability in polymers, the development of nanocomposites which can meet the growing demand for structural applications have been slow. With the understanding of how nano fillers interact with polymers, it is feasible to develop a structural composite fashioned from a nano-phased polymer. In retrospect, the use of nanocrystalline materials provides an excellent means for the development of polymer fibrous composites with both enhanced mechanical and thermal performance. In the present study, epoxy resin has been doped with SiC nanoparticles through an ultrasonic cavitation process. The modified (nano-phased) resin was then utilized in a Vacuum Assisted Resin Transfer Molding (VARTM) process with satin weave carbon fibers to manufacture composite panels. Test coupons were extracted and subjected to various loading conditions. Details of manufacturing and analyses of mechanical tests due to nanoparticle infusion are discussed in the following sections.

1.1 Manufacturing of Nanocomposites

The fabrication of nanophased carbon/epoxy composites was carried out in three steps. In the first step, spherical SiC nanoparticles of about 29 nm in diameter (manufacturer: MTI Corporation Inc. 2700 Rydin Road, Unit D, Richmond, CA. 94804, USA) were ultrasonically mixed with part-A (mixture of: Diglycidylether of Bisphenl A, 60 to 70%, Aliphatic Diglycidylether, 10 to 20% and epoxy toughner 10 to 20%) of SC-15 epoxy resin (manufacturer: Applied Poleramic, Inc 6166 Egret Court, Benicia, CA. 94510, USA). SC-15 is a two phase toughened epoxy resin system, it cures at room temperature and is extensively used in Vacuum Assisted Resin Transfer Molding (VARTM) processes. The loading of nanoparticles ranged from 1.5 to 3.0 percent by weight of the resin. The mixing was carried out in a *Sonics Vibra Cell* ultrasonic liquid processor (Ti-horn, frequency=20 kHz, intensity=100W/cm^2) as shown in Fig. 1. The mixing was carried out at 55% of the amplitude for about 30 minutes. At this time, the dispersion of nanoparticles seemed uniform through visual observation. In order to avoid rise in temperature during sonication, cooling was employed by submerging the mixing beaker in a mixture of ice and water. In the next step, Part-B (hardener, cycloaliphatic amine 70 to 90% and polyoxylalkylamine 10 to 30%) was added with

Fig 1. Vibra-cell ultrasonic processing

the mixture at a ratio of 3:10 and the mixing was carried out mechanically for about 10 minutes using a high speed mechanical stirrer. In the final step, the reaction mixture was used in a VARTM set up [9-11] with satin weave carbon fiber preforms to fabricate Carbon/Epoxy nanocomposite panels as shown in Fig. 2. Test coupons were extracted from each category of panels to conduct various mechanical tests.

Fig 2. A Typical VARTM Setup

2.0 RESULTS AND DISCUSSION

2.1 Quasi-Static Tests.

Two types of mechanical tests, namely flexure and tensile, were performed to evaluate the bulk stiffness and strength of each of the material systems on an MTS 8010 tensile testing machine.

A typical stress strain behavior from the flexural test is shown in Fig. 3. It is observed that the system with 1.5% SiC infusion has the highest strength and stiffness among the three systems indicated in Fig. 3. Gain in strength and stiffness
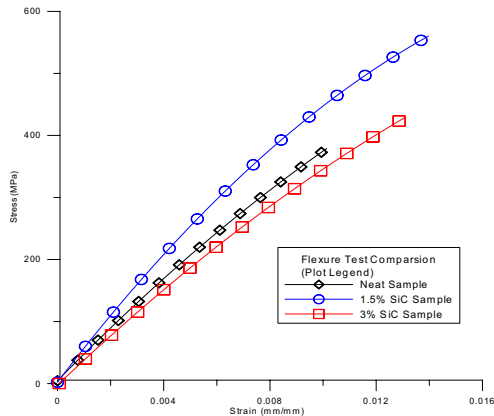

Fig. 3. Flexural testing of Carbon/Epoxy nanocomposites

of the 1.5% system is around 30% and 12% respectively over the neat as shown in Table-1. Enhancement in strength by 30% during flexure was somewhat surprising due the fact that previous studies with nanoclays [12, 13] did not show such improvement. It is also shown in Table-1 that if the particle infusion is increased to 3% SiC there is no proportional improvement in properties. Rather, there is a very nominal increase in strength, and a significant reduction in stiffness with the 3% SiC wt system. Similar reflection in properties with somewhat different ratios is observed during tensile tests as shown in Fig.4 and
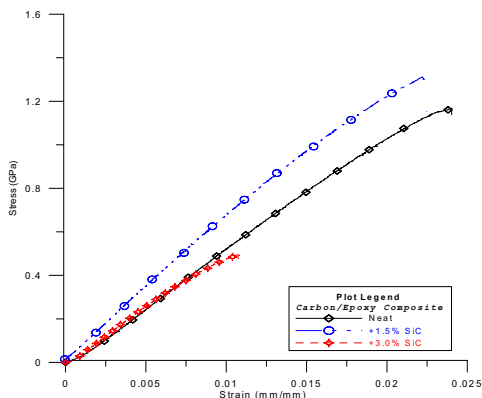

Fig 4. Tensile testing of Carbon/Epoxy nanocomposites

Table-2. It is noted that the enhancement in strength and stiffness is consistent with those shown in Fig. 3. The strain to failure of 1.5% SiC system is seen to be around 2.25% which is almost identical with that of the neat indicated in Fig. 4. Possible reasons for such behavior could be that since the reinforcement is in cloth (satin woven) form it did not contribute much to the elongation, and that the failure was mostly controlled by matrix and delamination related failure modes.

2.2 Dynamic Tests.

Low velocity impact analysis and high strain rate testing were preformed to determine the dynamic mechanical performance of the nano-phased carbon/epoxy systems compared to the neat composite. For low velocity impact testing, the analysis was performed using a Dynatup 8210. Specimens were cut to a 10.2 cm square with a thickness of 3 mm and tested with a pointed head alloy steel impactor weighing 6.33 kg. An electronic sensor was used to record the displacement of the impactor and the data was relayed to the Dynatup/GRC software for analysis. The potential energy (P.E.) was calculated from a height of 37 cm. The maximum calculated P.E. was 23.0 J for all three material systems. The absorption energy versus time graph is shown in Fig. 5. It is observed that the system with 1.5% SiC
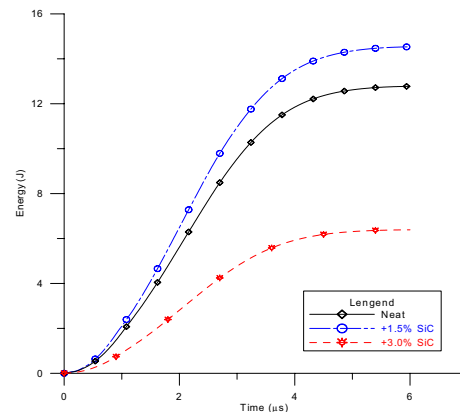

Fig 5. Low Velocity Impact testing at 37 cm height.

infusion has the highest energy absorption among the three material systems indicated in Fig. 5. Gain in impact energy of the 1.5% SiC system is around 14.7% over the neat as shown in the graph. It is also seen in Fig. 5 that if the particle infusion is increased to 3% SiC a degrading of properties are observed. When comparing the neat and 1.5% SiC systems to that of the 3.0%, the behavior of the 3.0% SiC system profile shows a rapid decline in the absorption energy rate. It is observed from then

Fig. 5 that slope of the 3.0% SiC system is much lower by comparison with the neat and 1.5% SiC material systems. This gives a sharp indication to a material system with lower dynamic modulus. The lower energy absorption rate indicates a weaken matrix which does not transfer load from matrix to fiber effectively.

Split Hokinson's pressure bar (SHPB) was used to evaluate dynamic compression and strain rate. For SHPB testing, three specimens were tested for neat, 1.5%, and 3.0% SiC systems with the dimension of 1.6 cm square and a thickness of 1.27 cm. The corresponding inlet pressure for each material system was 124.1 KPa. The specimens were tested in the transverse direction. The incident pulse e_i, reflected pulse e_r (input) and transmitted pulse e_t (output) was recorded using CEA-13-240UZ-120 measurements group strain gages. The data was acquired using gagescope software. Both voltage versus time and strain versus time graphs were plotted. VP3 software was used to merge both plots and stress versus strain response was obtained. A typical dynamic compression versus compressive strain is shown in Fig. 6. It is observed that the system with 1.5% SiC
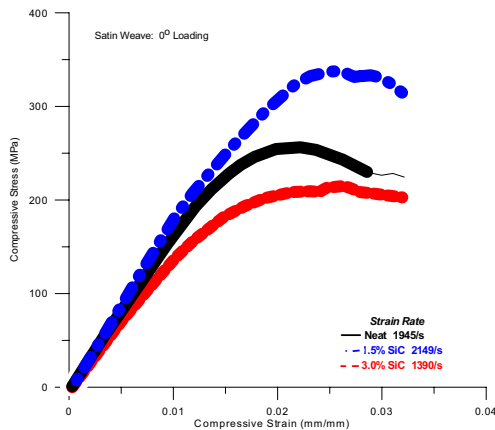

Fig 6. Split Hokinson's Pressure Bar
testing at 121.1 KPa.

infusion has the highest dynamic compression strength as well as modulus among the three systems indicated in Fig. 6. Improvement in compressive strength is shown to be approximately 40% for the 1.5% SiC system. Compressive modulus is around 10.7% over the neat as shown in Table-3. When comparing the 3.0% SiC system with the neat, once again, a drop is noted for the compressive strength as well as compressive modulus. This was expected because of early indications from low velocity impact testing of the three material systems. By inspection of the 3.0%

SiC material system, the damaged specimens showed multiple areas of delimination within the specimen when compared to the 1.5% SiC and neat material systems. The multiple delimination within the specimen strongly indicates a weakened bond between matrix and fibers. Enhancements also could be seen in the strain rate from the 1.5% SiC when compared to the neat.

3.0 SUMMARY AND CONCLUSION

The following is a summary of the above investigation.
- A low cost but reliable manufacturing procedure has been introduced to fabricate large scale laminated structural composites with a nanophased matrix.
- Nanoparticles loading into the base matrix are found to be optimal around 1.5% by weight to derive maximum gain in the mechanical and thermal properties of the structural composites.
- With 1.5% loading, an average of 20-30% increase in mechanical properties has been observed both in tension and flexure.
- Impact properties have been seen to improve by 10-14% with l.5% SiC loading.
- Compressive strength and modulus showed significant improvements with the 1.5% SiC loading.
- Nanoparticles loading at higher loads tend to degrade material properties.

4.0 ACKNOWLEDGEMENT

5.0 Tables

Table-1 Flexural test data for carbon/epoxy

Material	Flexural Strength [MPa]		Gain/Loss Strength in [%]	Flexural Modulus [GPa]		Gain/Loss Modulus in [%]
Neat	334.0		—	45.0		—
	390.0	**381.8**		46.0	**45.60**	
	395.0			39.0		
	390.0			44.0		
	400.0			54.0		
+1.5% SiC	550.5		+39.2	50.3		+12.4
	505.0	**531.3**		51.0	**51.26**	
	525.0			47.0		
	556.0			59.0		
	520.0			49.0		
+3.0% SiC	424.1		+4.7	37.5		-16.4
	390.0	**399.8**		36.0	**38.10**	
	395.0			39.0		
	390.0			41.0		
	400.0			37.0		

Table- 2 Tensile test data for carbon/epoxy

Material	Tensile Strength [GPa]		Gain/Loss Strength in [%]	Tensile Modulus [GPa]		Gain/Loss Modulus in [%]
Neat	1.196		—	50.1		—
	1.192	**1.19**		51.5	**50.1**	
	1.193			39.0		
	1.194			53.9		
	1.194			55.9		
+1.5% SiC	1.330		+11.6	63.2		+23.5
	1.430	**1.33**		59.5	**61.8**	
	1.320			61.9		
	1.290			62.8		
	1.290			61.8		
+3.0% SiC	0.500		-53.9	53.1		+6.3
	0.500	**0.48**		51.3	**54.0**	
	0.450			61.0		
	0.380			50.4		
	0.550			54.0		

Table- 3 SHPB Response @ 121.1 KPa

Mat. Type	Strain Rate (/s)	Peak Stress (MPa)	Strain @ Peak Stress (mm/mm)	Modulus (GPa)
Neat	3762	259	0.023	13.9
	3502	273	0.019	14.8
	3837	232	0.020	12.6
Avg.	**3700**	**255**	**0.020**	**13.8**
+1.5 SiC	2530	330	0.022	20.5
	2634	342	0.024	16.3
	2555	365	0.026	18.4
Avg.	**2573**	**335**	**0.025**	**18.4**
+3.0 SiC	3890	224	0.022	11.9
	3901	237	0.020	12.3
	3906	213	0.023	9.50
Avg.	**3899**	**225**	**0.022**	**11.2**

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