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Investigations on the thermal and flexural properties of plain weave carbon/epoxy-nanoclay composites by hand-layup technique

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Abstract The matrix of carbon fiber/SC-15 epoxy composites was modified with Nanomer® I-28E nanoclay, a surface modified montmorillonite mineral, to determine the effects of particle reinforcement on the response of these materials to flexural and thermomechanical loading. Different weight percentages of nanoclay were dispersed in SC-15 epoxy using sonication route. The nanophased epoxy was then used to manufacture plain weave carbon/epoxy nanocomposites using hand-layup process followed by vacuum bagging. Control samples of woven carbon fiber/epoxy were fabricated for comparison purposes. Effect of post curing on these samples was also investigated. 3-point bend flexure and Dynamic Mechanical Analysis (DMA) studies were carried out on 8- and 3-layered samples respectively. Results of flexural tests indicate significant improvements in flexural strength and modulus for nanoclay reinforced composites as compared to the control samples. DMA studies also showed enhancement in thermomechanical properties especially in storage modulus though no appreciable change was noticed in glass transition temperature, T_g. Scanning electron microscopy (SEM) studies were carried out to comprehend the effect of nanoclay on the microstructure and the failure modes.

Introduction

Inorganic nanoparticles have gained acceptance as potential reinforcing materials because of their low cost and ease of fabrication [1, 2]. Many researchers have attempted to enhance properties of different types of polymers by the addition of nanoclay. Over the past decade, a significant amount of work has focused on the improvement of properties of epoxies using nanoclay. Some of the earliest work on inorganic strengthening was performed in 1987 at Toyota. Researchers demonstrated the possibility of functionalizing the surface of layered inorganic silicates to make it compatible with the polymeric matrix [3, 4].

Pinnavaia and co-workers [5–7] showed that by adding organophilic montmorillonite into Diglycidylether of Bisphenol A (DGEBA), it was possible to significantly increase the tensile strength and modulus of the epoxy over the control samples. This increase is more distinct if the strength and modulus values of the starting epoxy are low. Park and Jana [8] reported that the degree of exfoliation of the nanoclay material in the epoxy matrix is thought to be responsible for the increase in the properties of the materials. The hypothesis of exfoliation of nanoclay suggests that the elastic forces developed in the clay galleries during epoxy curing are responsible for exfoliation of the clay structures.

In the last two decade, some studies have shown the potential improvement in properties and performances of fiber reinforced polymer matrix materials in which nano and micro-scale particles were incorporated. The technology of nano and micro-scale particle reinforcement can be categorized into inorganic layered clay technology, single walled and multi-walled carbon

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nanotube, carbon nanofiber technology, and metal particle technology. It was also shown that the mechanical properties of CFRP were improved at a loading of little wt% clay. Schmidt [9], Novak [10], Kojima et al. [4], Mark [11], and Hussain et al. [12] demonstrated the possible technology of dispersing Al_2O_3 particles in the matrix and investigated their effect on the mechanical properties of CFRP. Gilbert et al. [13, 14] and Timmerman [15] have demonstrated that fracture toughness and mechanical properties are increased by incorporation of metal and inorganic particles. In these studies, they have developed the concept of La PolynanoGrESS (Layered Polynanomeric Graphite Epoxy Scaled System) which utilizes the nanoparticle effect in an epoxy matrix and scales to continuous carbon fiber reinforced composites systems. Karaki et al. [16] incorporated layered clay, alumina, and titanium dioxide into an epoxy matrix and fabricated continuous carbon fiber reinforced polynanomeric matrices to study tension-tension fatigue behavior. They found that the number of microcracks in each layer depended on the type of particles and their concentration. Hackman and Hollaway [17] studied the potential applications of clay nanocomposite materials to civil engineering structures. They concluded that their ability to increase service life of materials subjected to aggressive environments could be utilized to increase the durability of glass and carbon fiber composites. Miyagawa et al. [18] studied the influence of biobased epoxy clay nanocomposites as a new matrix for carbon fiber composites. In their study, they found that the flexural strength and modulus did not change with the use of nanoclay. However, they reported increased interlaminar shear strength. Haque et al. [19] studied the effects of nanoclay on the improvement of mechanical and thermal properties of S2-Glass/epoxy composites. They showed that by dispersing 1% by weight nanosilicates, S2-glass/epoxy-clay nanocomposites exhibited an improvement of 44, 24 and 23% in interlaminar shear strength, flexural strength and fracture toughness respectively. Subramaniyan et al. [20] observed that addition of 5% by weight of nanoclay increased elastic modulus of resin under compression by 20% and the compressive strength of glass fiber composites with nanoclay when made by wet layup technique increased by 20–25%. Roy et al. [21] studied the compressive strength of pultruded thermoplastic composites using nanoclay reinforcement and observed consistent improvement in the compressive strength and modulus up to about 122% at 10% clay loading. Mahfuz et al. [22] studied the tensile response of carbon nanoparticle/whiskers reinforced composites and observed 15–17% improvement in the tensile strength and modulus. Chisholm et al. [23] investigated response of carbon/SiC-epoxy nanocomposites and showed 20–30% improvement in mechanical properties. Mohan et al. [24] evaluated the tensile performance of S2-glass epoxy composites dispersed with alumina nanoparticles up to 1.5% weight fraction and found an increase of 12% in tensile modulus and 8% in tensile strength.

In the present study, plain weave carbon/epoxy panels dispersed with 1–3% by weight of Nanomer® I-28E nanoclay were fabricated by hand-layup process. The platelet structured-Nanoclay is unique in its dimension. Its thickness is in the order of 1 nanometer while the other two dimensions vary from 300 to 1000 nm. This gives rise to an extraordinary high aspect ratio. We wanted to investigate the effect of such nanoparticles in our epoxy based composites. Carbon nanotubes (CNT's) also have very high aspect ratio, but nanoclay is much cheaper than CNT's. The price factor also played a role in choosing nanoclay as the nanoparticles. Ultrasonic cavitation route was employed to disperse the nanoclay in SC-15 epoxy resin system. Flexural strength and modulus were determined through a 3-point bend flexural test on 8-layered samples. Thermomechanical characterization was carried out with dynamic flexural tests on 3-layered samples using TA-Instruments' DMA 2980 with which dynamic mechanical properties like storage modulus, loss factor, tan delta were extracted. Glass transition temperature was identified as the temperature corresponding to the peak of tan delta versus temperature curve. The cantilever fixture used for DMA test in the present study can accommodate samples with a maximum span length of about 17.5 mm. In order to have the sample subjected to predominantly flexural mode of stress, it is necessary that a minimum span to thickness ratio of 10 be maintained. This warranted the samples to have a thickness of around 1.75 mm. Hence, laminates with 3 layers of plain weave fabric were fabricated for the DMA tests. On the other hand, the 3-point bend flexural tests were carried out with a 3-point bend fixture. If 3-layered samples were used with ASTM recommended span to thickness ratio of 16, it was required to have a minimum span length of about 24 mm (with average sample thickness of about 1.5 mm). However, the minimum span that could be possibly achieved with fixture used in the current study is about 28 mm without the supports getting loaded by the loading fixture. Hence, it became necessary to fabricate laminates with 8 layers for the flexure tests. Scanning electron microscopic approaches were used to investigate the influence of nanoclay on the microstructure of the composites and their fracture behavior. Fiber volume content of composites was also determined by matrix digestion method.

Experimental

Resin preparation

Ultrasonic mixing

Ultrasonic cavitation technique is one of the most efficient means to disperse nanoparticles into a polymer [25]. *Sonics Vibra Cell* ultrasonic processor (Ti-horn, 20 kHz, 100 W/cm^2) as shown in Fig. 1a was used to obtain a homogeneous mixture of epoxy resin and Nanocor Nanomer® I-28E nanoclay, a surface modified montmorillonite clay. SC-15 epoxy, manufactured by Applied Poleramic, Inc, comes with two parts: part A and part B. At first, the part A of SC-15 epoxy (mixture of: Diglycidylether of Bisphenol A, 60–70%, Aliphatic Diglycidylether, 10–20%) was sonicated with nanoclay at an amplitude of 55% with a total mixing time of 30 min. Nanoclay pick up moisture from air very easily. To get rid of the moisture, nanoclay was heated to 100 °C for 2 h before sonication. A pulser cycle (turning on and off-time ratio of 2:1) was used to control the temperature of the mixture. It was recommended to keep the temperature of the mixture at around 40–50 °C for good mixing, which was monitored using an infrared thermometer. The pulser cycle helps to achieve this goal. In addition, water bath was also used.

Mixing of Part A and Part B

After sonication, the homogeneous mixture was cooled down to room temperature in a refrigerating cooler maintained at 5 °C as shown in Fig. 1b. The sonicated part A with nanoclay was then mixed with part B of SC-15 epoxy (epoxy hardener, cycloaliphatic amine 70–90% and polyoxylalkylamine 10–30%) at a ratio of 10:3 using a high speed mechanical stirrer for about 5 min. Fig. 1c illustrates the mechanical mixing of part A and part B.

Degasification of resin

The mechanical mixing introduces air bubbles into the resin. This warranted a need of removing the trapped air and reaction volatiles from the reaction mixture. This was done using high vacuum for about 30 min. The resin mixture was kept in a sealed desiccating chamber, as can be seen in Fig. 1d, and connected to a vacuum suction pump. The high vacuum pulls out the air from the mixture and the resin is consolidated at the bottom. Two distinct layers are evident in Fig. 1d.

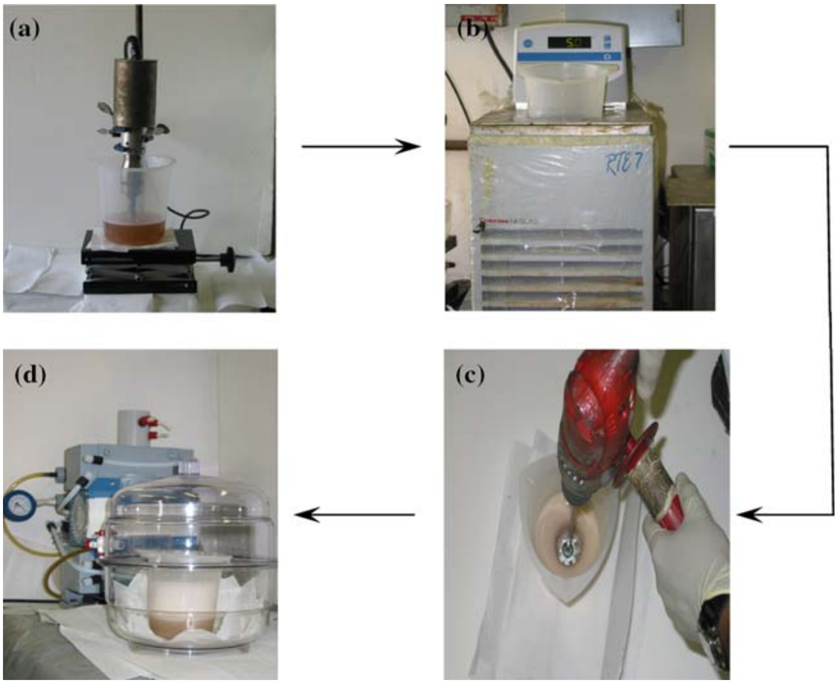


Fig. 1 (**a**) Vibra-cell ultrasonic processor (**b**) Cooling of sonicated Part A (**c**) Mechanical mixing of part A and part B of SC-15 epoxy (**d**) Degasification of resin mixture

Fabrication of nanocomposite by Hand Layup process

Hand-layup technique is sequentially illustrated in Fig. 2. A sheet of non-porous Teflon release film was provided at the back on which the carbon fabric was laid up. On each layer some prepared resin mixture was poured and spread out by a hand roller. The gentle rolling action of hand roller ensured the wetting of the carbon fabric and the excess resin was squeezed out of the panel layup by the roller. Another sheet of non-porous teflon release film was provided on top of the fabric layup. A suction line was provided on one side of the layup as shown in Fig. 2. An Aluminum plate placed on top of non-porous Teflon release film gave a good surface finish. Finally, the entire layup was vacuum-bagged and left to cure for 24 h.

Thermal post curing

The room temperature cured material was taken out from the vacuum bagging and the edges were trimmed. Test samples were machined for thermal and mechanical characterization. They were thermally post cured at 100 °C for 5 h in a mechanical convection oven to study the effect of post curing.

Determination of fiber volume fraction

The fiber volume fraction and void content of VARIM panels were estimated by matrix digestion test according to ASTM D 3171-99(2004) [26]. Samples of dimensions 3 by 3 by 0.2 cm were cut from the panels and the sides were subjected to grinding to have smooth edges. The sample dimensions were measured and weighed accurately. The samples were then placed in a bath of 80% concentrated nitric acid maintained at 75 °C for about 5 h. At that stage, the matrix was digested completely and only the fibers remained. The fibers were then washed with acetone and water. The fibers were then dried on an over maintained at 100 °C for about an hour. The weight of the fibers was measured. Fiber volume percentage in the composite was then calculated as follows:

Fiber, vol.%,

$$V_F = [(W/F)/(w/c)] \times 100 \tag{1}$$

Matrix Volume was calculated as follows:

$$\text{Matrix vol.\%}, V_m = \frac{(w - W)/\rho_m}{V_c} \times 100$$

$$\text{Void, vol.\%}, V_v = 100 - (V_m + V_f)$$

where W = weight of fiber in the composite, w = weight of the initial composite specimen, F = fiber density, ρ_m = density of the matrix, V_c = volume of the composite, and c = composite density.

Test procedure

A 3-point bend flexure test was performed to evaluate the flexural modulus and strength of each of the material systems. This test was carried out according to ASTM D790-02 [27]. Tests were conducted in displacement control mode with crosshead speed of 1.2 mm/min. Load-deflection data for each sample was collected. Flexural stress and strain were then computed. Flexural modulus was calculated from the slope of stress-strain plot. Five samples made of 8-layers of plain weave carbon fabric of each type were tested and the average values of flexural strength and modulus were determined.

Dynamic Mechanical Analysis (DMA) of various specimens was carried out on a DMA 2980 (Manufacturer: TA Instruments, Newark, DE). The samples

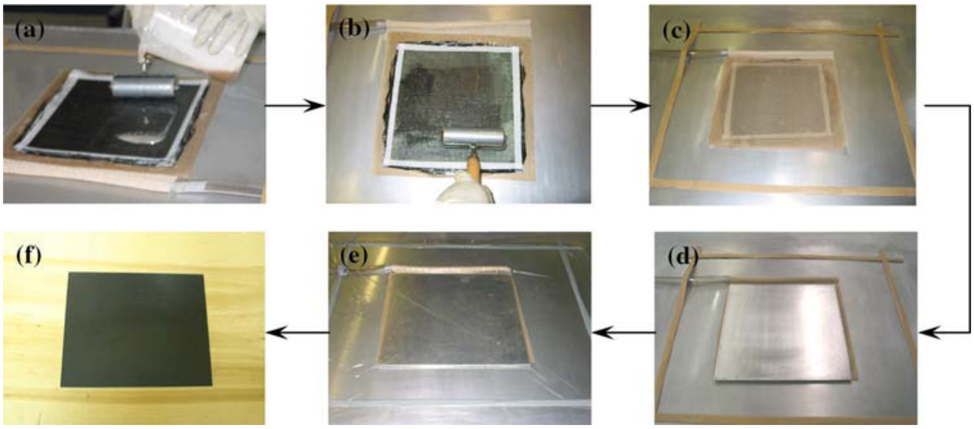


Fig. 2 (**a**) Resin is poured onto the carbon fabric, (**b**) fibers are wetted with the rolling action, (**c**) non-porous Teflon release film at the top after fabric layup, (**d**) Aluminum plate on top of non-porous Teflon release film, (**e**) layup is vacuum bagged and (**f**) edge trimmed hand-layup panel

made of 3-layered plain weave carbon fabric were cut into small pieces using diamond cutter and machined using the mechanical grinder to maintain the specified sample dimensions. The width of the samples was 12 mm and span length to thickness ratio was 10. The test was carried out according to ASTM D4065-01 [28]. The tests were run on a single cantilever beam mode with a frequency of 1 Hz and amplitude of 15 μm. The temperature was ramped from 35 to 150 °C at a rate of 3 °C/min. This heating rate was particularly maintained through out the test runs so that there is a minimum temperature lag between the sample and the furnace environment. At least five samples were tested for each kind. From the test data, storage modulus, which gives the dynamic elastic response of the samples; loss modulus, which gives the dynamic plastic response of samples and tan delta, which is the ratio of loss modulus/storage modulus were determined.

Results and discussion

Composite constituent analysis

Fiber volume fraction and hence the void content in the composites were estimated by matrix digestion method. Table 1 summarizes the results of matrix digestion test. It is seen from Table 1 that fiber volume fraction of 1 wt% and 2 wt% nanoclay samples is 55% while it is about 56% for the control and 3 wt% nanoclay samples. The void content is found to be close to 3% for all the samples. The improved mechanical properties of hand-layup panels can be attributed to the low void content in the samples.

Flexural properties

Flexural tests of hand-layup samples (with or without thermal post curing) were performed to evaluate the bulk stiffness and strength of carbon/epoxy nanocomposites. Typical stress–strain behaviors from the flexural tests are shown in Figs. 3 and 4. Some degree of non-linearity is seen before reaching the maximum stress especially in the case of 1 wt% and 2 wt% nanoclay samples. The positive effect of thermal post curing is also evident if we compare Fig. 3 with Fig. 4. In Figs. 3 and 4, it is evident that composite strength is improved up to 2 wt% of nanoclay loading. The increase in modulus is seen up to 3 wt% nanoclay loading.

Table 1 Matrix digestion test result for hand-layup panels

	Control	1% Nanoclay system	2% Nanoclay system	3% Nanoclay system
Fiber volume fraction, v_f (%)	56.56	55.34	55.14	56.17
Void content, v_v (%)	2.78	2.76	2.89	2.63

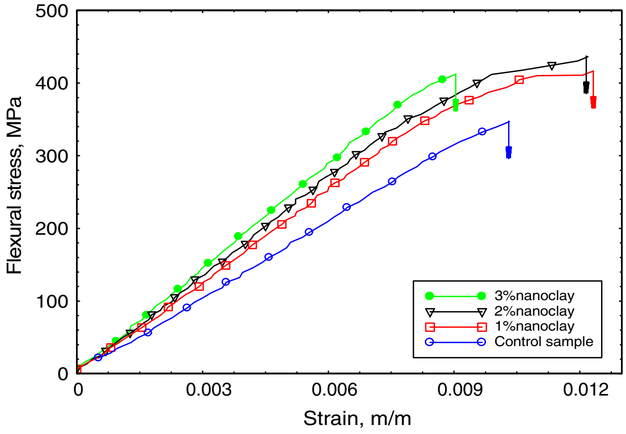


Fig. 3 Flexural stress–strain plot of hand-layup samples without post curing

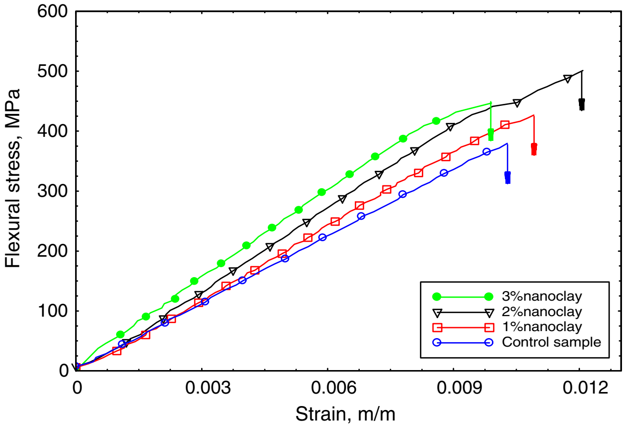


Fig. 4 Flexural stress–strain plot of post cured hand-layup samples

Tables 2 and 3 show the average properties of room temperature cured and thermal post cured samples respectively. From Table 2, it is seen that for 2 wt% nanoclay loading the flexural strength and modulus increased by about 25% and 22% respectively while Table 3 indicates the increase of strength and modulus by about 31% and 21% percent respectively for 2 wt% of nanoclay loading over control samples. It is very clear from these tables that the absolute values of strength for thermally post cured hand-layup samples are better than the room temperature cured samples.

Table 2 Flexural test results of room temperature cured hand-layup samples

	Flexural Strength, MPa	% Gain/ Loss in strength	Flexural Modulus, GPa	% Gain/ Loss in modulus
Control sample	350 ± 12.73	-	37.11 ± 1.16	-
1% Nanoclay	413 ± 6.45	18.0	43.5 ± 1. 03	17.21
2% Nanoclay	439 ± 7.32	25.42	45. 35 ± 1. 68	22.2
3% Nanoclay	411 ± 10.56	17.42	47.1 ± 1. 23	26.9

Table 3 Flexural test results of thermally post cured hand-layup samples

	Flexural Strength, MPa	% Gain/Loss in strength	Flexural Modulus, GPa	% Gain/ Loss in modulus
Control sample	380 ± 3. 3	–	37.57 ± 0.77	–
1% Nanoclay	426 ± 10.81	12.10	43.8 ± 2. 13	16.58
2% Nanoclay	498 ± 12. 81	31.05	45.6 ± 0. 81	21.37
3% Nanoclay	446 ± 8. 95	17.36	46.9 ± 1. 22	24.8

Thermal post curing does not have any appreciable effect on the average modulus values. It can be seen from Tables 2 and 3 that the modulus values remained almost the same in both room temperature cured samples and thermally post cured samples.

Thermal response

The variations of the storage modulus with temperature for room temperature cured hand-layup samples (control, 1, 2, and 3% nanoclay samples) are shown in Fig. 5. The storage modulus improves with the addition of nanoclay content up to 2 wt% and then decreases for 3 wt% nanoclay loading. Table 4 shows a maximum improvement in storage modulus of about 24% for 2 wt% nanoclay samples over control samples at about 35 °C. In Fig. 5, the sharp drop in the storage modulus indicates glass transition temperature, T_g. This sharp drop in storage modulus divides the entire temperature range into two segments—one is below T_g zone and the other is above T_g zone. The below T_g zone is also referred as the operating region while the above T_g region is referred as the rubbery plateau. The operating region of nanoclay samples up to 2 wt% loading remained almost the same as the control sample except for the increase in storage modulus in nanoclay samples in the below T_g zone. The rubbery plateau of the nanoclay samples seemed to remain same as control samples. In general, the crosslink density of nanoclay samples (derived from É in the rubbery plateau) is same as that of control samples. 3 wt% nanoclay samples showed poor thermal properties than other nanoclay

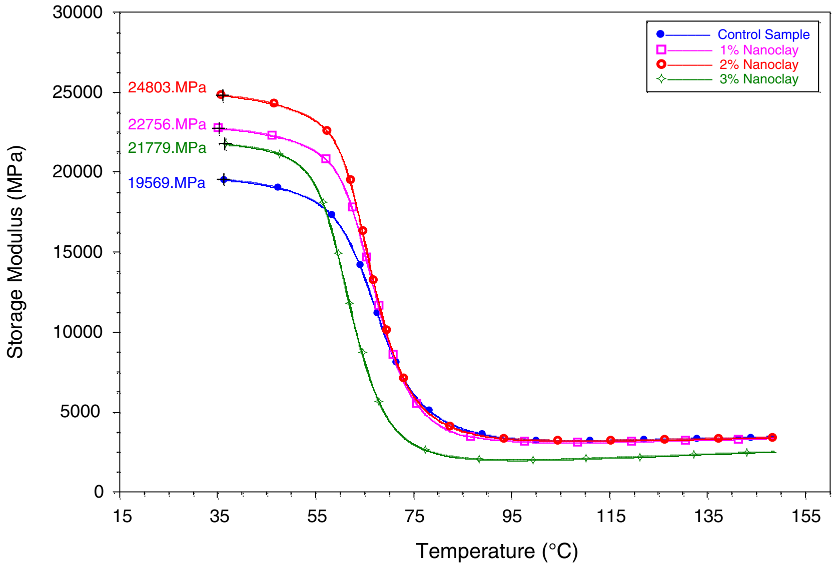


Fig. 5 Temperature dependence of storage modulus for room temperature cured hand-layup samples

Table 4 DMA results of room temperature cured hand-layup samples

	Storage Modulus (MPa)	% change	Loss Modulus (MPa)	% change	T_g (°C)
Control Sample	19683 ± 198	–	2780 ± 66	–	72.21 ± 0.53
1%Nanoclay	22833 ± 139	16.00	3289 ± 42	18.31	73.38 ± 0.40
2%Nanoclay	24457 ± 281	24.25	3640 ± 54	30.94	72.68 ± 0.63
3%Nanoclay	21780 ± 141	10.65	3495 ± 69	25.72	69.19 ± 0.30

samples by having a smaller operating region and lower storage modulus in the above T_g region.

Figure 6 shows the variation of loss modulus with temperature for room temperature cured hand-layup samples. Loss modulus increases up to 2 wt% and Table 4 indicates an increase of about 31% over control samples. There was no apparent shift in peak for nanoclay samples indicating hardly any improvement in T_g. The peak loss modulus of 3 wt% nanoclay samples shifted slightly towards lower temperature than the control samples.

The variations of the tan δ with temperature for samples without thermal post curing are shown in Fig. 7. Table 4 indicates that the control sample shows a peak at an average glass transition temperature of about 72 °C. For nanoclay samples, these peaks are more or less the same. No changes in the laminate glass transition temperature (T_g), as determined by the peak in tan δ were seen. Therefore, any changes in mechanical properties were due to the physical presence of the nanoclay as opposed to changes in the polymeric network structure [9]. These peaks generally increase in intensity and broaden with increase in the amount of nanoclay content.

Variation of the storage modulus with temperature for thermally post-cured hand-layup samples (control, 1, 2, and 3% nanoclay samples) is shown in Fig. 8. The storage modulus improves with the addition of nanoclay content up to 2 wt% and then again decreases for 3 wt%. Table 5 shows a maximum improvement in storage modulus of about 36% for 2 wt% nanoclay samples over control samples at about 35 °C. The effect of post curing is readily seen when Figs. 5 and 8 are compared. There is an increase in absolute values of storage modulus. The operating zone increased considerably in the below T_g zone for thermally post cured samples. The rubbery plateau of nanoclay samples is somewhat same or slightly higher than the control samples.

Figure 9 shows the variation of loss modulus with temperature for thermally post cured hand-layup samples. Loss modulus also increased with the increase in nanoclay content up to 2 wt% and Table 5 indicates an increase of about 46% over control samples. There is hardly any shift in loss modulus peak to higher temperature indicating no change in T_g.

The variations of the tan δ with temperature for thermally post cured hand-layup samples are shown in

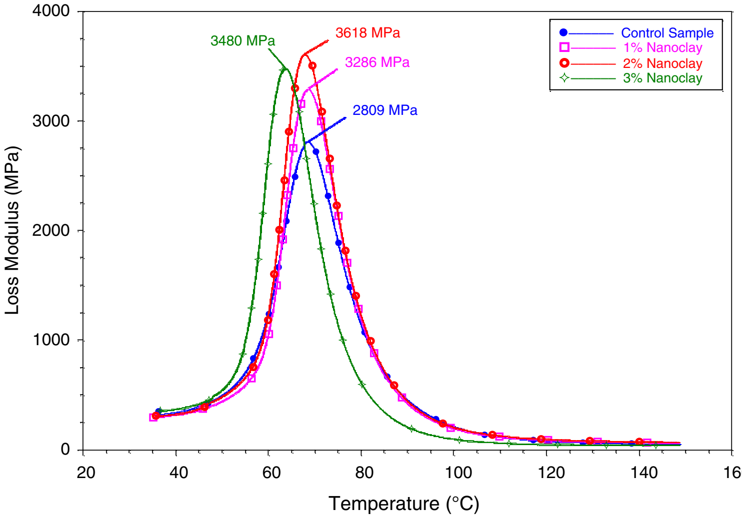


Fig. 6 Temperature dependence of loss modulus for room temperature cured hand-layup samples

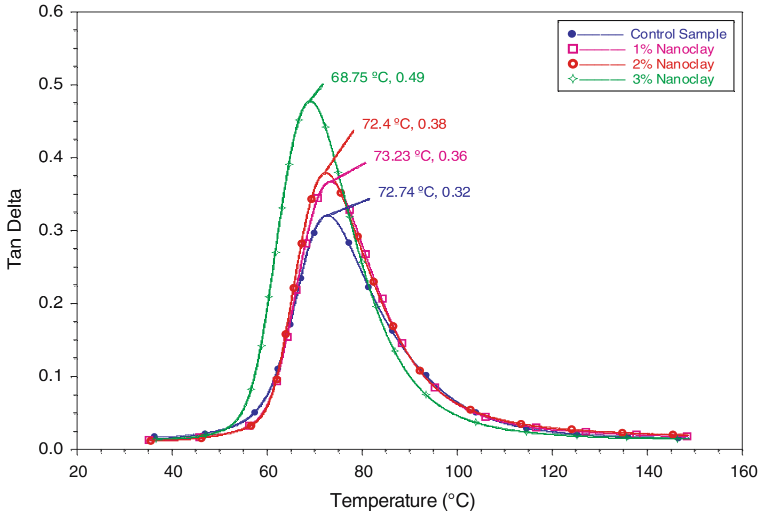


Fig. 7 Variation of loss tangent (tan δ) with temperature for room temperature cured hand-layup samples

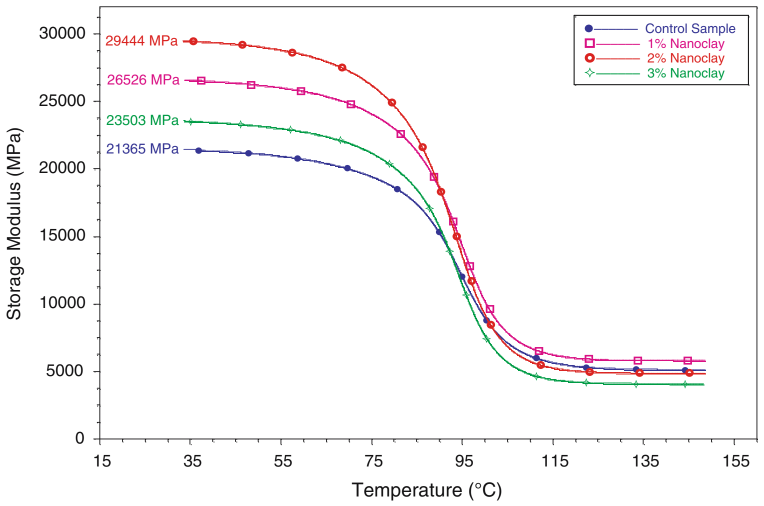


Fig. 8 Effect of thermal post curing on the variation of storage modulus with temperature of hand-layup samples

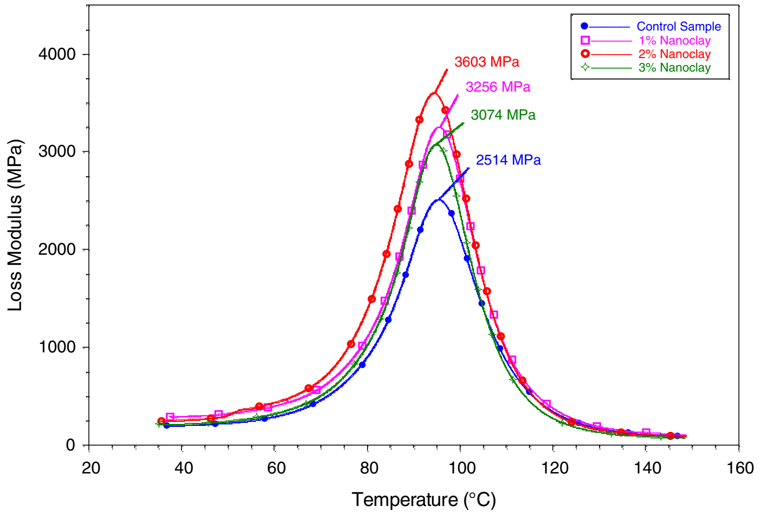


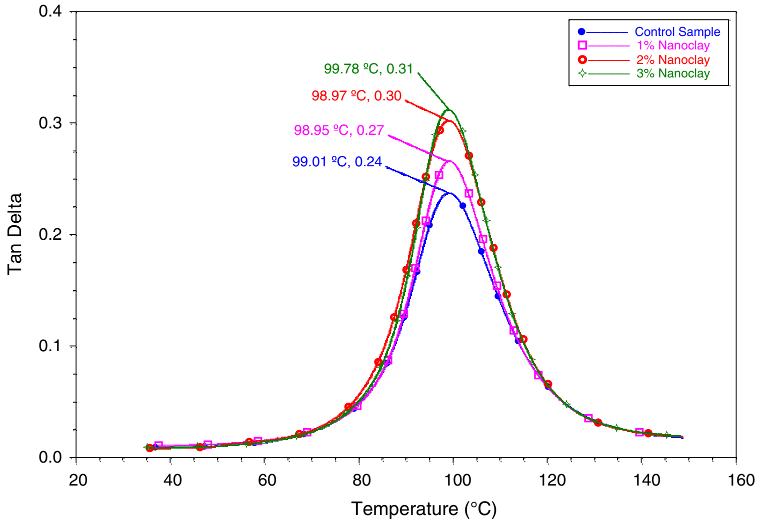
Fig. 9 Effect of thermal post curing on the variation of loss modulus with temperature of hand-layup samples

Fig. 10. The control sample has a T_g of about 99 °C. The peaks of nanoclay samples did not have any shift. The only change associated with the peaks of nanoclay is the broadening of peaks with the increase in nanoclay content. Table 5 shows glass transition temperatures for different wt% of nanoclay samples.

Table 5 DMA results of thermally post cured hand-layup samples

	Storage Modulus (MPa)	% change	Loss Modulus (MPa)	% change	T_g (°C)
Control Sample	21593 ± 149	–	2483 ± 43	–	99.03 ± 0.31
1%Nanoclay	26560 ± 70	23.00	3279 ± 39	32.06	99.26 ± 0.25
2%Nanoclay	29440 ± 71	36.34	3632 ± 37	46.27	99.55 ± 0.21
3%Nanoclay	23400 ± 89	8.37	3080 ± 31	24.04	98.81 ± 0.03

Fig. 10 Effect of thermal post curing on the variation of loss tangent (tan δ) with temperature of hand-layup samples



Failure surface analysis

Scanning Electron Microscopy (SEM) studies were carried out for the failure surfaces. Figure 11 illustrates the fracture surfaces of hand-layup samples for control and different weight percentage of particle loading. It can be seen from this figure that control samples exhibits some fiber pullout failure while 1 wt% and 2 wt% nanoclay showed fiber breakage failure indicating good bonding between fiber and matrix. It is also evident that control sample splits open more than the nanoclay samples. For 3 wt% nanoclay samples fiber pullout is seen in Fig. 11d in the tension side of the flexural specimen while a characteristic failure is seen in the compression side of the flexural specimen. A kink band, as shown in Fig. 11e, is observed in the compression side of 3 wt% nanoclay sample which is a typical failure for brittle fibers like carbon.

Conclusions

Woven carbon/epoxy laminates were fabricated with the epoxy modified using Nanomer® I-28E nanoclay, an organically modified clay at different weight percentages. Thermomechanical and flexural properties of the nanophased composites were determined and compared with those of control samples. Microstructural characterization was carried out through scanning electron microscopy. The following are the summary of the above investigation:

- Flexural test results of thermally post-cured samples indicate a maximum improvement in strength and modulus of about 31% and 21% respectively.
- From DMA results, it is seen that storage modulus increased by significant amount while glass transition temperature remained almost unchanged for different weight percentages of nanoclay samples as determined by the peak in tan δ. Therefore, the changes in mechanical properties were due to the physical presence of the nanoclay as opposed to changes in the polymeric network structure.
- 2 wt% nanoclay seems to be an optimum loading for carbon/SC-15 epoxy composites in terms of mechanical and thermal properties.
- Micro-structural studies revealed that nanoclay promotes good adhesion of fiber and matrix thereby increasing the mechanical properties.

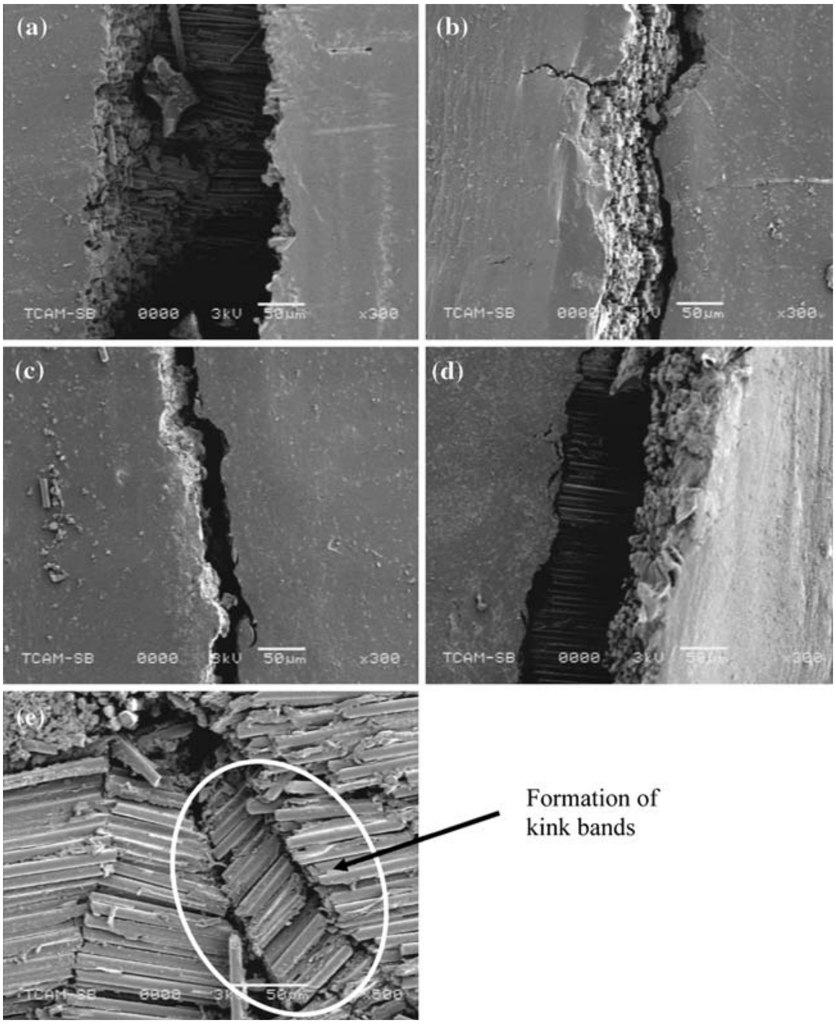


Fig. 11 SEM micrographs of failed hand-Layup samples for (**a**) control sample, (**b**) 1% nanoclay sample, (**c**) 2% nanoclay sample (**d**) 3% nanoclay sample subjected to flexural loading; and (**e**) microbuckling leading to formation of kink zones observed in the compression side of 3 wt% nanoclay sample

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