

**A Pilot Study on
Flexural Properties of Epoxy Nanoclay Reinforced Composites**

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Abstract: Kinetix epoxy resin was filled with nanoclay to increase tensile properties of the composite for civil and structural applications by the Centre of Excellence in Engineered Fiber Composites (CEEFC), University of Southern Queensland (USQ). This project manufactured samples with different percentages by weight of nanoclay in the composites in steps of 1 wt %, which were then post-cured in an oven. The samples were then subjected to flexural tests. The results showed that composite with 3 wt % of nanoclay produces the maximum flexural strength, strain and modulus. It is hoped that the discussion and results in this work would not only contribute towards the development of nanoclay reinforced epoxy composites with better material properties, but also useful for the investigations of fracture toughness, tensile properties and flexural properties in other composites.

Keywords: Flexural strength, maximum flexural strain, flexural modulus, epoxy resin, and nanoclay.

1. Introduction

The composite materials exhibit characteristics of both inorganic materials and organic polymers. It has been established in recent years that polymer reinforced with a small percentages of strong fillers can significantly improve the mechanical and thermal properties [1].

The most widely used and least expensive polymer resins are the polyesters and vinyl esters; these matrix materials are used primarily for glass fiber-reinforced composites. The epoxies are more expensive and, in addition to commercial applications, are also

utilized extensively in polymer matrix composites for aerospace applications; they have better mechanical properties and resistance to moisture than the polyesters and vinyl resins [2]. This research project investigates the flexural strength, maximum flexural strain and flexural modulus of kinetic epoxy composites reinforced with varying percentage by weight of nanoclay with a view to finding out the optimum percentage by weight of the nanoclay that can be added to the composites.

Nanocomposites are a type of composite in which the scale of the dispersed phase is less than 100 nm, in at least one dimension. Because of the nanoscale dispersion and the high aspect ratios of the inorganic clays, polymer-layered silicate nanocomposites (PLSNs) exhibit light weight, dimensional stability, heat resistance, high stiffness, barrier properties, and improved toughness and strength with far less reinforcement loading than conventional composite counterparts. The synthesis and characterization of PLSNs has become one of the frontiers in materials science [3].

In general, PLSNs can be divided into two categories: intercalated and exfoliated composites. In an intercalated nanocomposite, a few polymer chains diffuse into the silicate galleries with fixed interlayer spacing. In contrast, an exfoliated nanocomposite is formed when the silicate nanolayers are delaminated and well dispersed in the continuous polymer matrix. The exfoliated state may maximize interfacial contact between the organic and inorganic phases, and as a result, nanocomposites with optimum performance properties can be achieved [3]. Nanoparticles have enormous surface area and the interfacial areas between the two intermixed phases in a nanocomposite will therefore much larger area than micro scale composites. This brings about increased bonding between the matrix and the

matrix. The mechanical, thermal and electrical properties of nanocomposites are therefore observed to be better than their traditional counterparts. In many cases, the nanoparticle reinforced resins have been used to fabricate conventional microcomposites [4].

2. Literature review

Some studies showed that the addition of a small amount of nanoparticles caused the significant improvement in the mechanical properties of pure polymers. The high aspect ratio and high modulus of these particles may be the reasons for this. When the nanoparticles were dispersed in the matrix materials, they produced a very large amount of surface area and confined the chain mobility of the polymers under stresses [5-6]. The overall molecular mobility in the nanocomposites had been due to two grounds: (1) the loosened molecular packing of polymer chains; this was because of the presence of nanoparticles and their interactions with the polymer chains. This led to increased free volume and enhanced molecular mobility, and (2) the formation of a layer of bound polymer around the nanoparticles and this led to decreased molecular mobility [5, 7].

Khan et al. made nanoclay/epoxy composites and discovered that the flexural strength of the composites initially increased with increasing particulate loading but dropped back after 3 wt % of nanoclay. They also found that the flexural modulus of the composite increased with particulate loading. They also made nanoclay-CFRP/epoxy hybrid composites and found that the flexural strength and modulus behaved in the

same way as the flexural strength of nanoclay/epoxy composites; the flexural strength and modulus peaked at 3 wt % of nanoclay [8].

Zhou et al. mixed vinyl ester resin (tensile strength = 41.27 MPa) with nanoclay and found that the tensile strength of the nanoclay/vinyl ester composite would initially increase and peak at 2 wt % of nanoclay (44.68 MPa) and then drop significantly at 4 wt % of nanoclay (23.33 MPa). However, the tensile modulus of the composites behaved a little bit different as the maximum elastic modulus was at 1 wt % of nanoclay. After this, the elastic modulus dropped with increasing particulate loading [5].

Yasmin et al. found that by adding clay particles the elastic modulus of pure epoxy was significantly improved, and the higher the clay loading, the higher the modulus of the nanocomposite. The elastic modulus of the nanocomposites was, however, found to decrease by degrees with temperature [5].

Mahfuz et al. made composite samples from low density polyethylene and carbon nanoparticles. They were then tensile tested. They found that the failure stress of the extruded polyethylene sample doped with carbon nanoparticles/whiskers is about 17% higher than the neat extruded sample. They also found that the improvement in tensile modulus was around 16% and that the gain in modulus was at the cost of a reduction in failure strain of 12% [9].

3. Epoxy resin

The epoxy resin used in this study is Kinetix R246TX Thixotropic Laminating Resin, an opaque liquid, and the hardener used is kinetic H160 medium hardener which has a pot life of 120 minutes. Other hardeners like H126, H128, H161 and H162 can also be used [10]. The nanoclay was first mixed with epoxy resin, after this the hardener, kinetic H160 medium was added. The by weight ratio of resin to hardener used was 4:1 [10]. The composite was then cast to moulds of flexural test pieces and left to cure under ambient conditions for 24 hours. The flexural test specimens were taken out of the moulds and then post-cured in oven at 40 °C for 16 hours, and then at 50 °C for 16 hours and finally at 60 °C for 8 hours. This is to ensure the heat distortion temperature (HDT) is above 63 °C. To bring the ultimate HDT to 68 °C, another 15 hours of post-curing will be required [10]. The specimens were then subjected to tensile tests.

In many cases, the resin components (filler and resin) are mixed together and simply gravity poured into a low-cost mould of metal or some other material. One precaution must be kept in mind with epoxy casting and laminating resin; the polymerization reaction produces heat; it is exothermic, and if a large batch of resin is mixed with a curing agent and allowed to stand for too long, it may catch fire. People familiar with dealing with epoxies are well aware of this characteristic and deal with it using the resin immediately after adding the curing agent or by mixing small quantities [10].

Although some epoxies are formulated to crosslink at room temperature to initiate the crosslinking and a well-controlled temperature history throughout the process to

crosslink as intended. Many epoxies also require subsequent post-curing to completely crosslink to achieve optimum properties. The crosslinking times required by epoxies are normally long as compared with other resin, e.g. unsaturated polyesters [11].

4. Nanoclay

On top of reducing costs, the addition of fillers into polymers is to make it electrically conducting. These fillers must be electrically conducting and is usually discontinuous for the sake of convenience in composite fabrication, e.g. slurry casting. These composites are widely used in electronic packaging [12]. Moreover, the addition of some filler to polymers will increase the heat dissipation capability of the composites. This is very important for the proper operation of electronic devices because the accumulated heat generated from the performance of those devices should be removed quickly [13].

The reinforcement used in this study is nanoclay. The product name of the nanoclay used is CLOISITE® 30Bm manufactured by SCP Rockwood Additives; its synonym(s) is organoclay. Cloisite® 30B is a natural montmorillonite modified with a quaternary ammonium salt. Cloisite® 30B is an additive for plastics to improve various plastic physical properties, such as reinforcement, HDT, CLTE and barrier. The moisture content is less than 2%. The typical dry particle sizes are 10 % less than 2µm, 50 % less than 6µm and 10 % less than 13µm. The colour is off white. Its density is 1.98 g/cc [14].

5. Flexural properties

The three point bending flexural test provides values for the modulus of elasticity in bending E_B , flexural stress σ_f , flexural strain ε_f and the flexural stress-strain response of the material. The main advantage of a three point flexural test is the ease of the specimen preparation and testing. However, this method has also some disadvantages: the results of the testing method are sensitive to specimen and loading geometry and strain rate [15].

The standard used is ISO 14125:1998(E) because the results can then be compared with the work of others [16]. The centre uses a universal machine MTS Alliance RT/10 at 10kN couple with the software TESTWORK 4. The dimensions of the specimens of resins were 250mm x 10mm x 4mm and tested at a crosshead speed of 4 mm/min.

The equation used for calculating the flexural stress,

$$\sigma_f = \frac{3PL}{2bh^2} \quad (1)$$

The equation used for calculating the flexural strain,

$$\varepsilon_f = \frac{6Dh}{L^2} \quad (2)$$

The equation used for calculating the Young's modulus,

$$E_B = \frac{L^3 m}{4bh^3} = \frac{\text{Flexural strength}}{\text{Flexural strain}} \quad (3)$$

where: σ_f : stress in outer fibre at midpoint, MPa;

ε_f : strain in the outer surface, %;

E_B : modulus of elasticity in bending, MPa;

P: load at a given point on the load deflection curve, N;

L: support span, mm;

b: width of test beam, mm;

h: depth of test beam, mm;

D: maximum deflection of the centre of the beam, mm;

m: slope of the tangent to the initial straight line portion of the load deflection curve, N/mm.

6. The Composite Samples

The reinforcement was nanoclay (Cloisite® 30B) and they were made 0 to 7 wt % in the cured epoxy composites EP/nanoclay (X wt %), where x is the percentage by weight of the filler. As the raw materials of the composites are liquid and powder, the mixture of the resin, hardener and the nanoclay was cast into a baking tray.

The resin is an opaque liquid and is first mixed with the nanoclay. After that the hardener is added to the mixture, they are then mixed to give the uncured composite. Nanoclay was first added to the resin rather than the hardener because it would give one more time to mix and spread the nanoclay across the resin without the effect of gelling. This would give a better mixture of the resin and the nanoparticles. Table 1 shows the mass in grams of resin, hardener and nanoclay required respectively to make 1000 grams of uncured composite of 2 wt % of nanoclay.

Finally, before pouring the uncured composite into the mould (tray), baking paper was laid on the surface of the tray to enable easy release of the composite slab after curing. The uncured composite was then cast into the tray and cured under ambient

conditions. After initial 72-hour curing when the test pieces were removed from the mould, they were post-cured. This was achieved by baking the pieces in an oven. The oven temperatures and times were:

- 16 hours at 40°C
- 16 hours at 50°C
- 8 hours at 60°C

The test pieces were then tensile-tested.

7. Results and Discussions

Figure 1 illustrates flexural strengths of varying nanoclay wt % reinforced Kinetix epoxy matrix composite. The flexural strength of the neat resin post-cured in an oven was 58.83 MPa. This rises slowly to a peak value of 72.42 MPa at 3 wt % of filler but then remains steady for the rest wt % of filler. Table 2 shows the values of flexural strengths mentioned above with their standard deviation in brackets. The increase in flexural strength at 3 wt % of nanoclay was 23 % above that of neat resin.

Siddiqui et al. [17] used organoclay I30.P to fill Epon828 epoxy resin. These were clay/epoxy nanocomposites. They found that there was a gradual decrease of flexural strength with increasing particulate loading as depicted in Figure 2. This was due to the agglomeration and or non-uniform distribution of the clays in the matrix, particularly at high clay contents. The agglomerates could also act as crack initiation sites, leading to a loss of flexural strength. They also argued that the different curing speeds between the bulk epoxy and the silicate interfaces might induce internal stresses, leading to premature failure [17]. The results of the flexural strength of this

study were better than those of Siddiqui et al. [17]'s study because the flexural strength in this study increased with nanoclay content and peaked at 3 wt % of reinforcement. Of course, the resin and nanoclay used were different. The results are not surprising as Cease studied the mechanical properties of different types of epoxy resins and discovered that the tensile strength of Epotek 301-2 epoxy resin was 2.4 times than that of Hysol 9361 epoxy resin [18].

Siddiqui et al. [17] also fabricated laminate composites from carbon fiber plain woven fabric and organoclay-filled epoxy resin (Epon828). These were CFRP (carbon fibre reinforced plastic) nanoclay epoxy composites. The flexural strength values were normalized with carbon fibre volume fraction of 20% and shown in Figure 1, which shows that the flexural strength marginally decreased with increasing particulate loading. The variations of flexural strength were very small and the changes had to be related to the matrix properties which were affected by the clay content, dispersion and morphology [17]. The trend of the flexural strengths of the clay/epoxy nanocomposites and the CFRP composites was different from that of this study and the results of this study are more promising than those of Siddiqui et al. [17]. Moreover, it can be argued that the dispersion of the clay and morphology of this study are better than its counterparts.

Chowdhury et al. [19] used Nanocor Manomer I-28E nanoclay and SC-15 two-part epoxy resin to manufacture nanoclay reinforced epoxy composites. The flexural properties results were shown in Table 3. It can be found that the flexural strength of composites with any wt % of nanoclay was higher than the neat resin. The flexural strength peaked at 415 MPa at 2 wt % of nanoclay [19]. The trend of the flexural

strength of Chowdhury et al. [19]'s study was similar to that of this study except that the flexural strength peaked at 3 wt % of nanoclay in this study. Both returned promising results but the maximum improvement in flexural strength in this study (23%) is better than its rival (13.7%). However, this is not surprising as Wang and Pinnavaia, Lan Wang and Pinnavaia, and Wang et al. had pointed out that the increase in strength in composites would be more distinct if the strength values of the starting epoxy were low [20-22].

Khan et al. used epoxy (Epon828) resin and organoclay, Nanomer I30P to make clay-epoxy nanocomposites. They found that the flexural strength of the composites increase with particulate loading and peaked at 121.5 MPa at 3 wt % of nanoclay as depicted in Figure 2. The flexural strength dropped back to 110.8 MPa at 5 wt % of filler but it was still higher than the neat resin composites (108 MPa). From Figure 2, it can be found that the results of the flexural strengths of clay-epoxy composites of Siddiqui et al. were much inferior to those of Khan et al. even both used the same resin and nanoclay. This can be attributed to the better dispersion of the nanoclay in the matrix because Khan et al. shear-mixed the resin and the nanoclay at 3000 rpm, while Siddiqui et al. subjected the mixed resin and nanoclay to sonication [8, 17].

The trend of the flexural strengths of Khan et al.'s study was similar to that of this study and both peaked at 3 wt % of nanoclay. The maximum improvement in flexural strength in Khan et al.'s study was 12.5 % while that of this study was 23 %. This is because the increase in strength in composites would be more distinct if the strength values of the starting epoxy were low [20-22].

Khan et al. also fabricated laminate (hybrid) composites from unidirectional carbon fiber and organoclay-filled epoxy resin (Epon828). These were CFRP (carbon fibre reinforced plastic) nanoclay epoxy composites. They found that the flexural strength of the composites increase with particulate loading and peaked at 718.4 MPa at 3 wt % of nanoclay as depicted in Figure 1. The flexural strength dropped back to 678.9 MPa at 5 wt % of filler but it was still higher than that of the neat resin plus carbon fibre composites (663 MPa). The flexural strength of neat resin plus carbon fibre laminate composite of Khan et al. was 663 MPa (Figure 1) but that of Siddiqui et al. was only 368.4 MPa (Figure 1) which was 80 % lower than its counterpart. This was due to the different carbon fibre format used. Furthermore, the flexural strengths of CFRP nanoclay composites of Khan et al. were even better than those of Siddiqui et al. (Figure 1). This proves that the manufacturing processes employed by Khan et al. were more superior than those of Siddiqui et al. [8, 17].

The increase in tensile strengths of the composites from those of neat resin could be due to two reasons; one was from nano-particle point of view and the other from cross-linking. If a matrix were more cross-linked, its strength and stiffness would be higher. On the other hand, if nano-particles were infused into a polymer, they would form a relatively weak particle-polymer interface which would act as a crack dissemination mechanism at the very early stage of crack growth and eventually delay the formation of the dominant crack [22]. Thus, the improved tensile strength of nano-composites was believed to have been caused by enhanced cross-linking in the matrix due to nano-particle infusion [22].

Figure 3 illustrates the maximum flexural strain of nanoclay filled epoxy composites. The maximum flexural strain of the neat resin was 0.029 mm/mm. This rises to a peak of 0.032 at 3 wt % of nanoclay. It then drops back to 0.021 mm/mm at 5 wt % of filler and remains more or less the same with up to 7 wt % of nanoclay. The peak value of the maximum flexural strain occurred at 3 wt % of nanoclay and this was in line with that of flexural strength. The trend of the maximum flexural strains was also in line with that of the flexural strength. It can be argued that the trends of values of both were correct and reliable. Table 2 shows the values of flexural strengths mentioned above with their standard deviation in brackets. As the values of the standard deviations were small, it can be argued that the values of maximum flexural strains were correct and reliable.

Figure 4 shows the flexural moduli of varying by wt % of nanoclay reinforced epoxy matrix composites. The Young's modulus of the neat resin post-cured in an oven was 2.30 GPa. This rises very slowly to 2.43 GPa at 3 wt % of nanoclay. The change was only 5.6%. In fact, the flexural modulus values did not change much with varying wt % of filler. The lowest value was 2.09 GPa at 7 wt % of nanoclay. Table 2 shows the values of Young's modulus mentioned above with their standard deviation in brackets. Since most of the deviations were small, it can be argued that the results are correct and reliable.

Siddiqui et al. used organoclay I30.P to fill Epon828 epoxy resin. These were clay/epoxy nanocomposites. They found that there was a significant increase of flexural modulus with increasing particulate loading as depicted in Figure 4. They discovered that the increase was particularly significant in the first few wt % of

nanoclay [12]. The increase from neat resin (2.87 GPa) to a composite with 7 wt % of nanoclay (3.89 GPa) was 35.5%. The composite with 7 wt % of nanoclay gave the highest flexural modulus. This is in line with the work of Khan et al. as depicted in Figure 4. The increase from neat resin (2.96 GPa) to a composite with 7 wt % of nanoclay (3.70 GPa) was 25%. Again, the composite with 7 wt % of nanoclay gave the highest flexural modulus. However, their results were different to those of this study because the flexural modulus values of in this study barely changed much with varying wt % of nanoclay. Of course, the resin and nanoclay used were different and the Young's modulus values of different epoxy resins were different as pointed out by Cease et al. [18]. However, it can be argued that the trend of having Young's modulus to increase with nanoparticle loading, like in the cases of Siddiqui et al. and Khan et al., would not continue with increasing wt % of nanoclay; the reverse would happen as proved by Chan et al. [23].

Figure 5 shows the flexural modulus of CFRP composites with varying clay content obtained by Siddiqui et al. [17]. It can be found that the values of the flexural modulus hardly changed with particulate loading like the ones obtained in this study. On the other hand, Figure 5 illustrates the flexural modulus of CFRP composites with varying clay content obtained by Khan et al. [8]. It can be observed that the flexural modulus increased slightly with nanoclay content. The flexural moduli of neat resin and hybrid composite with 3 wt % of nanoclay were 78.9 GPa and 83.9 GPa respectively. The increase was only 5 %. The composite with 3 wt % of nanoclay gave the highest flexural modulus. However, the increase in flexural modulus of composite with 3 wt % of filler from that neat resin was not far from that (5.6%) of

this study and composite with 3 wt % of nanoclay in this study also gave the highest flexural modulus.

Figure 6 shows the SEM neat epoxy resin sample post-cured in an oven for 4, 4 and 2 hours at 50 °C, 80 °C and 100 °C respectively at a magnification of 150 times. The striations and flow were the characteristics of neat epoxy resin. Epoxy/nanoclay (3 wt %) specimen post-cured in an oven for 4, 4 and 2 hours at 50 °C, 80 °C and 100 °C respectively at a magnification of 300 times was depicted in Figure 7. It can be found that the nanoclay powder clusters were dispersed quite evenly and no porosity *was* found. Figure 8 shows epoxy/nanoclay (7 wt %) composite specimen post-cured in an oven for 4, 4 and 2 hours at 50 °C, 80 °C and 100 °C respectively at a magnification of 300 times. Here the porosities were more and that is why the specimen with 7 wt % of nanoclay was weaker than its 3 wt % counterpart.

8. Conclusions

This study has evaluated the flexural strength, maximum flexural strain and flexural modulus of varying percentage by weight of nanoclay reinforced epoxy resin; in all cases, the fluidity of the slurry composite was high and could be cast easily into moulds. The values of flexural strengths and flexural moduli had also been compared with those found by other studies and the values obtained in this study were sometimes higher but at the other times lower than their rivals; this is due to the different epoxy resins and types of nanoclay used. However, the trends of the flexural properties were the same. The values of flexural strength and maximum

flexural modulus as well as flexural modulus of this study peaked at 3 wt % of nanoclay and in another study (Khan et al.), the values of flexural strength peaked at 3 wt % of nanoclay but the flexural modulus peaked at 5 wt % of nanoclay. This is due to the different epoxy resins and types of nanoclay used. Finally, it is worth pointing out that in addition to the basic reinforcing mechanisms of nanoclay composites: intercalation and exfoliation, interlocking and bridging effects do exist in nanocomposites and pointed by Chan et al. [24].

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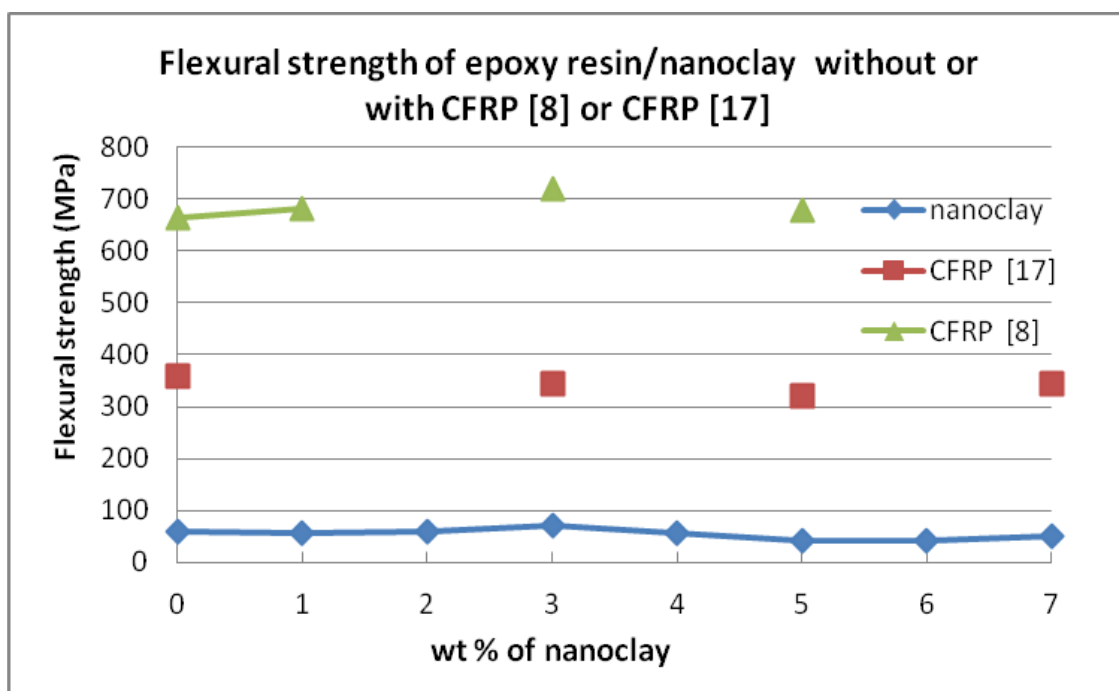


Figure 1: Differences in flexural strength of samples in this study and CFRP filled samples of others

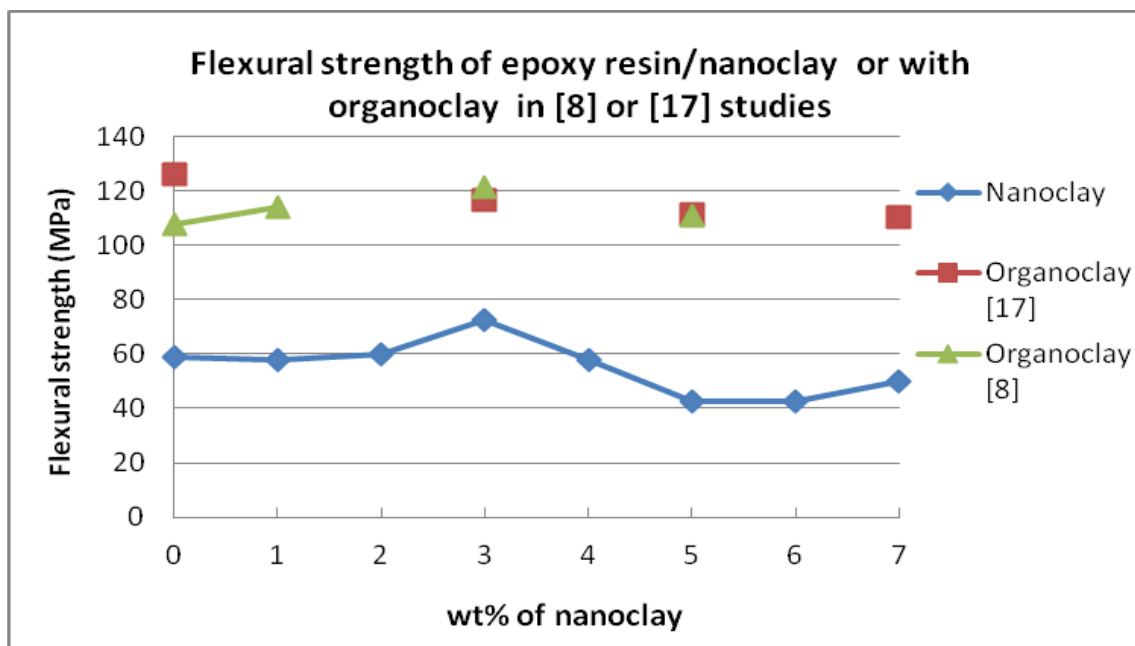


Figure 2: Differences in flexural strength of samples in this study and organoclay filled samples of others

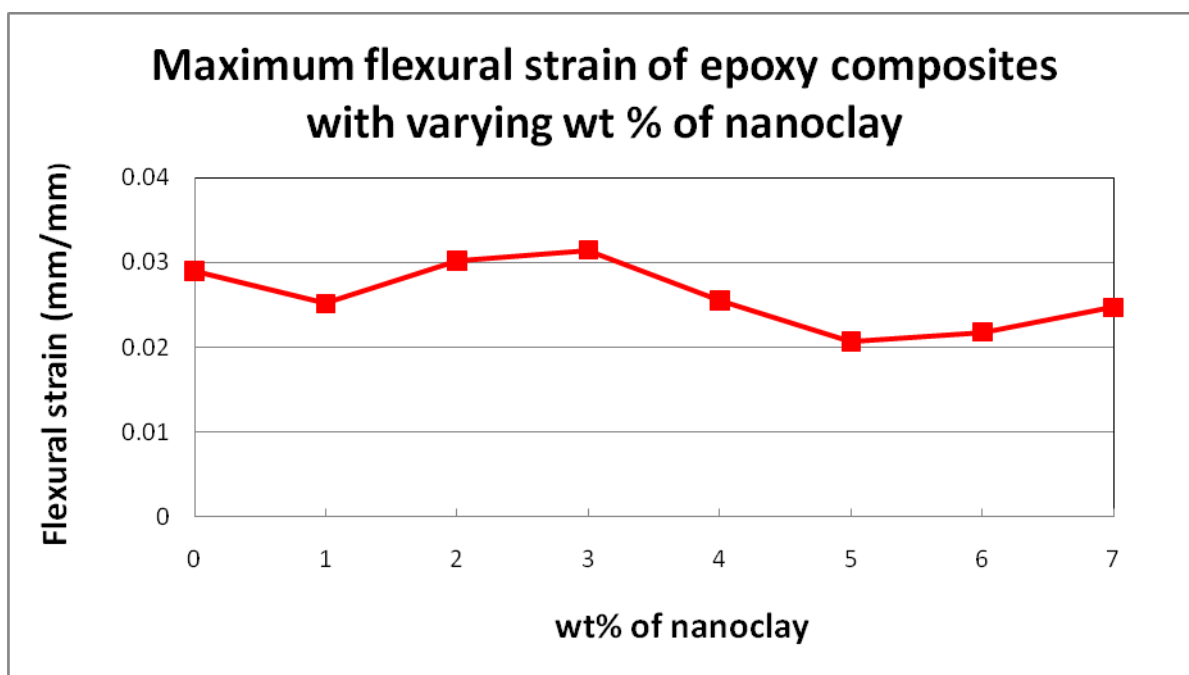


Figure 3: Maximum flexural strain of epoxy composites with varying wt % of nanoclay

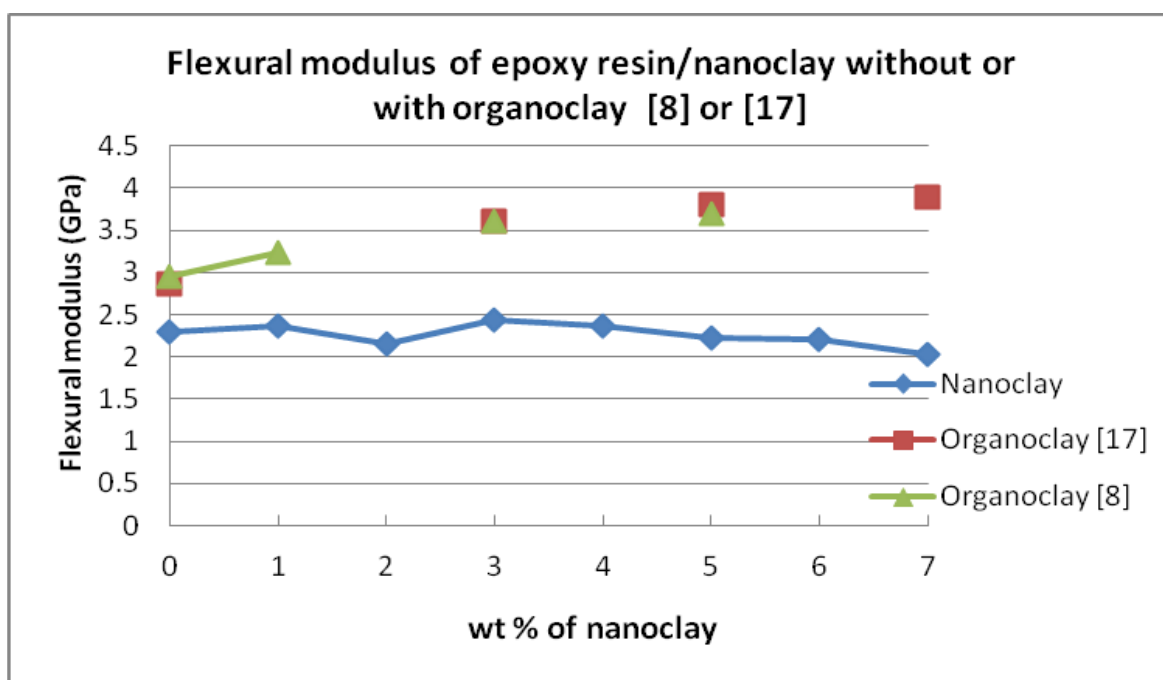


Figure 4: Differences in flexural modulus of samples in this study and CFRP filled samples of others

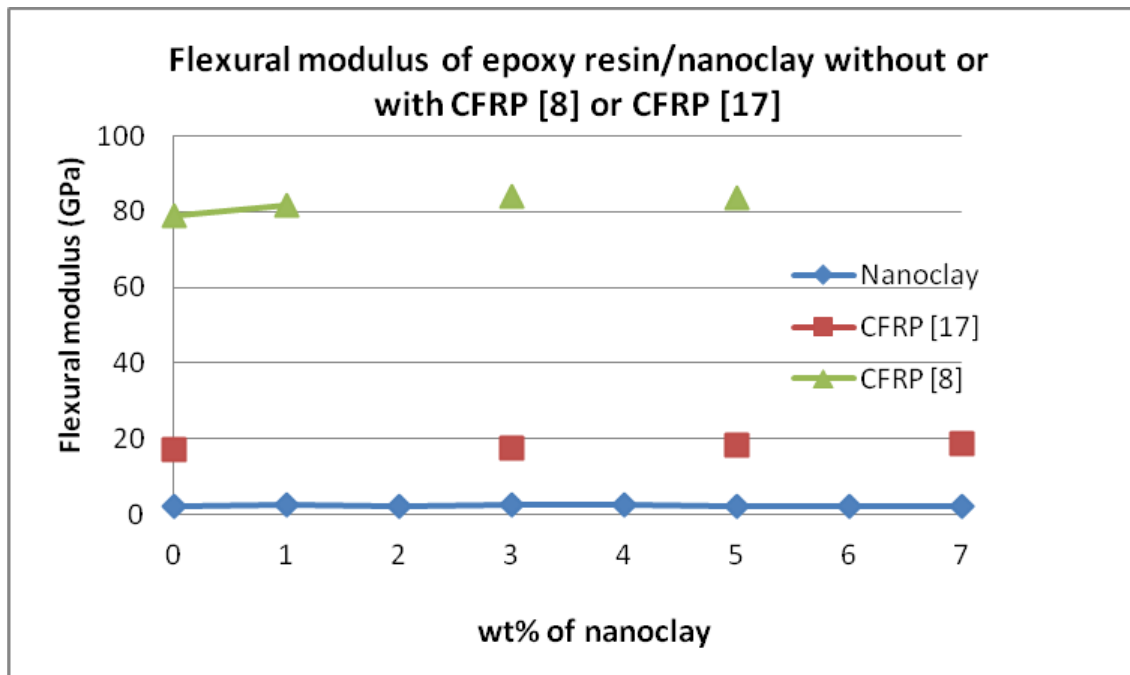


Figure 5: Differences in flexural modulus of samples in this study and organoclay filled samples of others

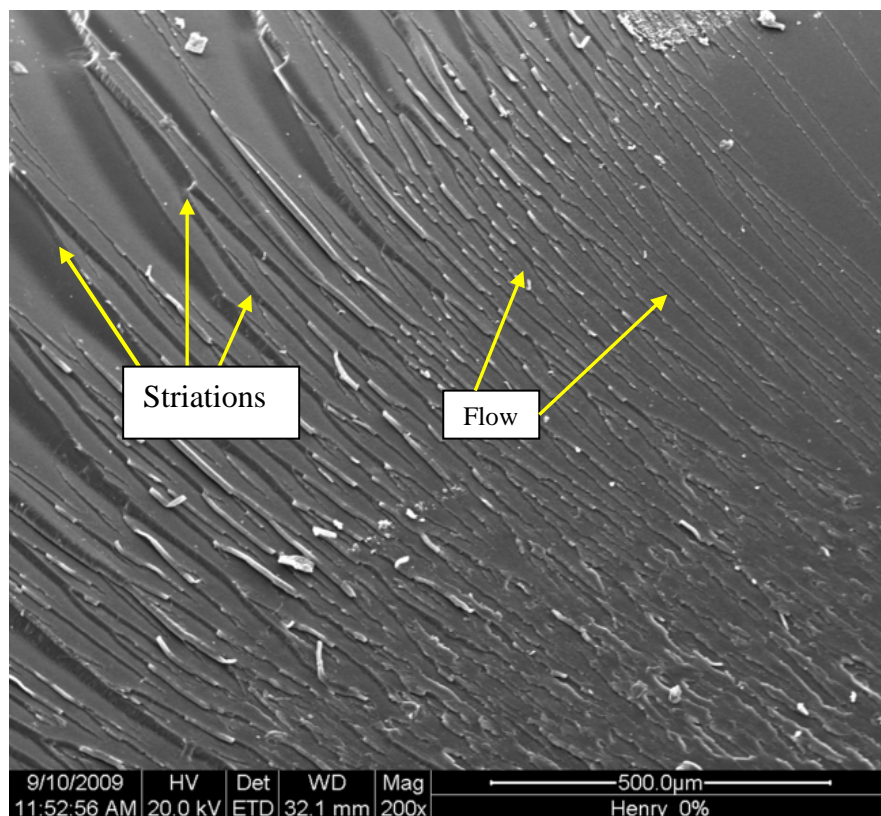


Figure 6: SEM image of fractured neat epoxy resin, 150 X

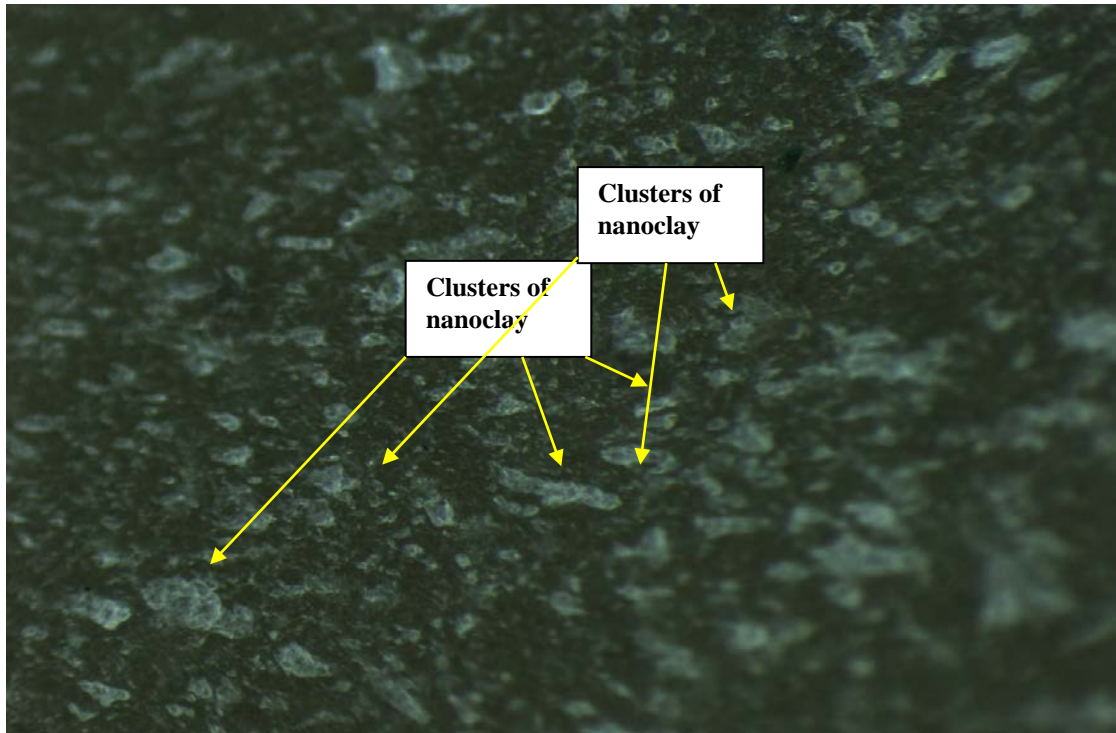


Figure 7: Optical microscope image of epoxy/nanoclay (3 wt %) composite, X 300

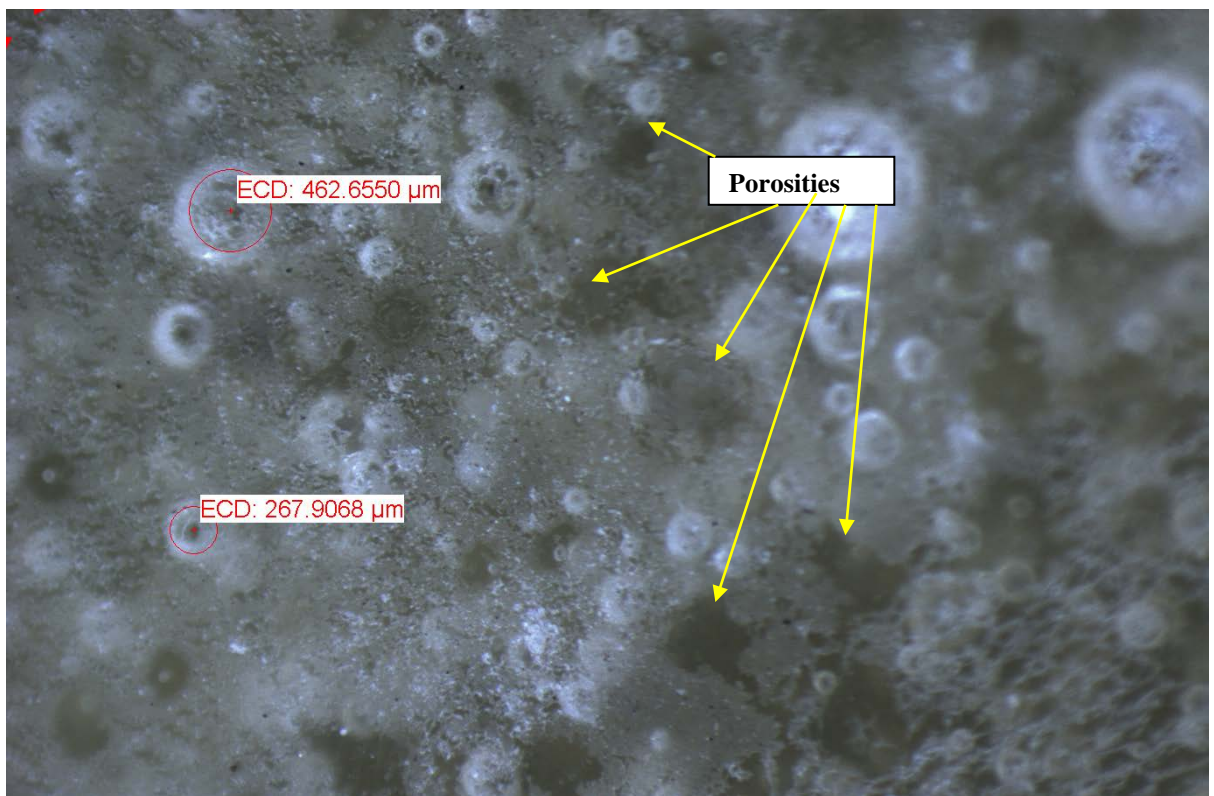


Figure 8: Optical microscope image of epoxy/nanoclay (5 wt %) composite, X 300

Table 1: Weight of materials required to make 1000 g of EP/nanoclay (2%)

Parameters	Materials	Resin (R)	Hardener (H)	R + H	Nanoclay	Composite
Percentage by weight		4	1	---	---	---
Percentage by weight		---	---	98%	2%	---
Weight of materials in 1000 g of Epoxy/nanoclay		784 (g)	196 (g)	980 (g)	20 (g)	1000 (g)

Table 2: Yield strength, tensile strength and Young's modulus of epoxy composite reinforced with nanoclay

Mechanical properties	% w/t of nanoclay	0	1	2	3	4	5	6	7
Standard deviations of flexural strength (MPa)		3.09 (58.8) [#]	3.28 (57.8)	7.99 (64.6)	6.21 (72.42)	5.47 (61.4)	6.93 (48.9)	4.85 (47.9)	9.70 (50.17)
Standard deviations of maximum flexural strain (mm/mm)		0.01 (0.029)	0.002 (0.025)	0.002 (0.030)	0.003 (0.032)	0.003 (0.026)	0.004 (0.021)	0.002 (0.022)	0.002 (0.022)
Standard deviations of flexural modulus (GPa)		0.02 (2.30) [#]	0.31 (2.36)	0.07 (2.16)	0.25 (2.43)	0.02 (2.37)	0.33 (2.22)	0.16 (2.27)	0.04 (2.09)

[#]flexural strength, maximum flexural strain and flexural modulus

Table 3: Flexural test results of thermally post-cured epoxy composites. Adapted from [13]

	Flexural strength (MPa)	% Gain/loss in strength	Flexural modulus (GPa)	% Gain/loss in modulus
Control sample	365 ± 7.57	-	35.3 ± 0.15	-
1 wt % nanoclay	399 ± 18.88	9.31	36.7 ± 0.43	3.97
2 wt % nanoclay	415 ± 17.21	13.70	38.6 ± 0.65	9.35
3 wt % nanoclay	373 ± 9.29	2.19	37.7 ± 0.45	6.80

