

Effect of nanoclay on the toughness of epoxy and mechanical, impact properties of E-glass-epoxy composites

K. Krushnamurthy¹, I. Srikanth², B. Rangababu², S. K. Majee², R. Bauri³, Ch. Subrahmanyam^{1*}

¹Department of Chemistry, Indian Institute of Technology (IIT) Hyderabad, Hyderabad 502205, India

²Defense Research and Development Organization (DRDO), ASL, Hyderabad 500058, India

³Department of Metallurgical and Materials Engineering, Indian Institute of Technology (IIT) Madras, 600036, Chennai, India

*Corresponding author. Tel: (+91) 40-23016050; Fax: (+91) 40-23016032; E-mail: csubbu@iith.ac.in

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ABSTRACT

Organically modified montmorillonite nanoclay was added to the epoxy and E-glass-epoxy composites. The influence of nanoclay content (varied between 0 to 5wt %) on the relative crosslink density and the fracture toughness of the epoxy matrix was studied. Differential scanning calorimetry (DSC) indicated that the amino functional groups present on the nanoclay react with the epoxy matrix to increase the crosslink density of about 13 and 18% at 3 and 5wt% addition, respectively. The toughness of the epoxy composites increased by 25% at 3wt% addition of nanoclay, whereas, it decreases at 5wt%. Flexural strength and tensile strength of the E-glass-epoxy composites were found to increase by 12% and 11% respectively at 3wt% addition of nanoclay, while at 5wt% addition these properties decreased due to the matrix embrittlement. Interestingly matrix embrittlement is found to be beneficial in increasing the impact resistance due to spallation of embrittled matrix that ensures the dissipation of the impact energy. 5wt% nanoclay addition increases the impact strength by 29% and reduces the back face bulge of composite by 31%. These results may lead to the design and realization of glass-epoxy composites with better impact strength. Copyright © 2015 VBRI Press.

Keywords: Nanoclay; glass fibers; fracture toughness; mechanical properties; differential scanning calorimetry.

Introduction

There is a growing demand to use a high-performance and low-weight composites to replace the traditionally used metals in various applications [1, 2]. Among the various composites, glass fiber reinforced epoxy composites (E-glass-epoxy) are widely used in civilian applications like automobile bodies, strengthening of walls, slabs to new building frames and bridges and ship hulls. In defense applications glass-epoxy composites are used as armor tiles for protecting various vehicles [3-5]. There is a need to enhance mechanical and impact properties of glass epoxy composites as it will enable realizing light weight structures for both civilian and defense applications [6]. One of the possibilities is to increase the interface compatibility of the epoxy and glass fiber [7, 8]. For this purpose many researchers have explored the possibility of employing various additives like CNTs, CNFs, glass micro balloons, nanoclay etc. to enhance the mechanical properties [9-12] and impact resistance [13-16] of the fiber reinforced epoxy composites. Among various additives, organically modified montmorillonite nanoclay is beneficial due to lower cost,

ease of availability and its compatibility with the conventional FRPs like glass-epoxy [11, 17]. Recent studies indicated that the amino functional groups present on the surface of the nanomaterials reacts with the epoxy resin that improves the crosslink density, which has a positive effect on the toughness of the matrix [18, 19]. So far the effect of toughness on mechanical and impact properties of composites was studied by many research groups [20]. It is generally believed that increasing the toughness of the matrix increases the mechanical and impact properties for the composites [21]. However, there is a need to optimize the nanoclay addition in order to obtain the best results.

In the present work, a systematic study has been carried to understand the influence of nanoclay addition on the toughness of the epoxy matrix and the influence of toughness variation of epoxy on the impact resistance, tensile and flexural strength of the E-glass-epoxy composites. Novelty of the present work is its emphasis on finding the effect of matrix embrittlement along with matrix toughness on the mechanical and impact performance of the E-glass-epoxy composites.

Experimental

Materials

Epoxy resin (commercial name LY556, Huntsman Advanced Materials (India) Pvt. Limited, Mumbai) made from Bisphenol A and epichlorohydrin along with diamine based curing agent (DETDA, commercial name HY 5200, Huntsman Advanced Materials (India) Pvt. Limited, Mumbai) is used as the matrix phase. E-glass fabric with plain-woven (360 gsm, UTS of 40 GPa) was used as the main reinforcement. Nanoclay used in this study is octadecylamine modified montmorillonite (Nanomer I.30E) procured from M/s Nanocor Inc (USA).

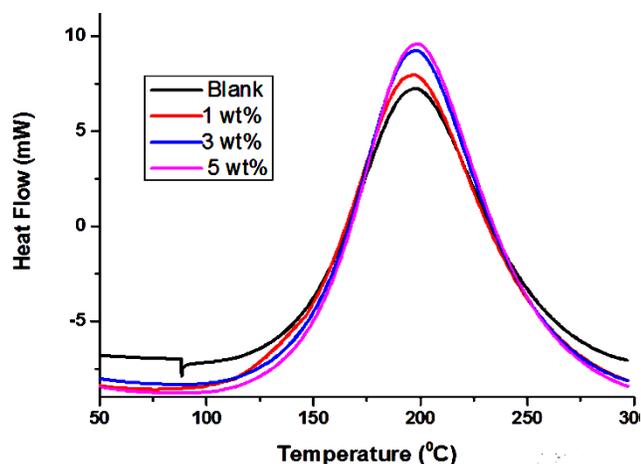


Fig. 1. Heat evolution during curing.

Preparation of nanoclay-epoxy mixture

Initially desired weight of nanoclay (0wt%, 1wt%, 3wt% and 5wt %) was directly added into the epoxy resin. Dispersion of the nanoclay was ensured by mechanical stirring at 2000 rpm for 2 h using a high speed mechanical stirrer followed by sonication at 50MHz frequency in a bath type sonicator for 90 minutes. Hardener in the ratio of 1: 4 to the resin weight was added to this mixture. Hereafter, this mixture is referred as a nanoclay-epoxy system.

Fabrication of nanoclay-epoxy and nanoclay-E-glass-epoxy composites

To realize the nanoclay-epoxy composites, nanoclay-epoxy system was poured into a die and cured for 1h at 110°C followed by curing at 160°C for 2h. Post curing was carried out for 3h at 180°C, which is nearer to the peak cure temperature of the composition under study (Fig. 1). Thus nanoclay-epoxy composites of four different compositions (0wt% or blank, 1wt%, 3wt% and 5wt %) were prepared. For the preparation of the nanoclay-E-glass-epoxy composites, nanoclay-epoxy resin was applied to the E-glass fabric. These fabric layers were stacked up and compacted by sandwiching between two flat steel plates to get required fiber-matrix ratio. Curing was carried out by following the same cure cycle as that of nanoclay-epoxy composites. Volume fraction (V_f) of the E-glass-epoxy composites was controlled at 55% ($\pm 1\%$) by using the same number of layers and by controlling the thickness of the

sample. V_f of the prepared E-glass-epoxy composites was measured by following ASTM D3171. Thus, four different nanoclay-E-glass-epoxy composites corresponding to 0wt%, 1wt%, 3wt% and 5wt% addition of nanoclay were prepared.

Testing of nanoclay-epoxy composites

Nanoclay-epoxy composites were tested to determine the fracture toughness (K_{IC}). For carrying out the toughness test, six samples having 60 mm x 10 mm x 5 mm dimensions were collected. A notch was made at the centre of the specimen using isomet (Isomet 5000, BUEHLER) and tested as per ASTM D 5045-07 on a universal testing machine (United 50KN) at a crosshead speed of 10 mm/min. Scanning electron microscope (FEI-quanta 200, USA) was used to study the fractured surfaces, whereas transmission electron microscope (TEM, model Tecnai G2, FEI) was used to study the dispersion of nanoclay in epoxy composites.

Tensile and flexural strength of nanoclay-E-glass-epoxy composites

Tensile and flexural strength of the samples were measured as per ASTM D 3039 and ASTM D790 standards, using a universal testing machine (United 50kN). Typical sample dimensions for tensile and flexural strength are 200 mm x 10 mm x 2.8mm and 60 mm x 12.7 mm x 2.8 mm respectively.

Impact strength

Drop weight impact test was performed to measure the impact strength. Impact test was carried out as per ISO 6603-01 specifications. Samples having approximately 140 mm x 140 mm x 4 mm were clamped on a 100 mm diameter support ring (Fractovis plus, Ceast). Impactor having 3.56 Kg weight with a hemispherical nose of 20.0 mm diameter was impacted at 5.0 m/s velocity at the centre of the specimen. The bending deformation of sample, energy absorbed by sample and load-time data was recorded directly from the data acquisition system. To study the area of the damage due to impact, infrared thermography images were acquired using a FLIR SC3000 camera with 45° lenses, equipped with an Echotherm flash lamp heating system (9.6 kJ). Heat pulse was impinged on the sample for 5milli seconds and then the thermal images were captured at a frame rate of 25 Hz. C-Scan imaging is used to study the magnitude of bending deformation of the impacted samples across the cross section.

Results and discussion

Crosslinking changes to epoxy

Corsslink density increases on the addition of the nanoclay to epoxy matrix. This was attributed to the amino functional groups present on the nanoclay that can react with the epoxy matrix. Differential scanning calorimetry confirmed the increasing degree of heat evolution with increasing loading of nanoclay (Fig. 1). Thus the addition of nanoclay imparts a higher degree of cross linking with epoxy matrix.

Fracture toughness

Fracture toughness changes of the epoxy due to the addition of nanoclay are shown in **Table 1**. It can be seen that the addition of nanoclay up to 3wt% has improved the fracture toughness.

Table 1. Heat evolution during cross linking, % relative improvement in crosslink density, fracture toughness of neat epoxy composites and mechanical properties of E-glass-epoxy composites at various wt% of nanoclay.

wt% of nanoclay in epoxy	ΔH (J/g)	% improved cross linking density	Fracture Toughness ($\text{MPa}\cdot\text{m}^{1/2}$)	Tensile Strength (MPa)	Flexural Strength (MPa)
0% nanoclay (blank)	179	-	0.982	364	476
1% nanoclay	192	7.2	1.073	376	508
3% nanoclay	203	13.4	1.226	407	532
5% nanoclay	212	18.4	0.873	369	489

The fracture surface of samples with and without nanoclay is shown in **Fig. 2**. The pristine sample (**Fig. 2a**) shows a characteristic smooth surface representing a brittle failure, whereas 3wt% nanoclay-epoxy sample has shown a ductile failure with many planes of cleavage (**Fig. 2b**). This could be because of additional cross linking due to amino groups present on the surface of nanoclay as well as the ability of nanoclay to deflect the crack [**18, 19**]. This infers that, nanoclay zones can resist the crack propagation thereby forcing the advancing cracks to follow a torturous path (**Fig. 2c**). This results in the generation of higher fracture surface area and thus higher toughness for nanoclay reinforced epoxy matrix. However, matrix embrittlement was observed at 5wt% addition of nanoclay to epoxy matrix, even though there was a good dispersion of nanoclay (**Fig. 2f**). Degree of cross linking with epoxy at 5wt% addition of nanoclay resulted in a very high crosslink density. However, crosslink density beyond some optimum value initiates the embrittlement of the epoxy matrix [**18**]. It means that at the higher wt% addition of nanoclay, matrix surrounding the nanoclay zones gets embrittled. This results in an easy spallation of nanoclay rich zones as shown in **Fig. 2d**.

As the nanoclay-E-glass-epoxy composites are having the same matrix system, which showed embrittlement at 5wt% addition of nanoclay (nanoclay-epoxy), the properties of nanoclay-E-glass epoxy composites also should get affected. The subsequent sections discuss this aspect.

Tensile strength of E-glass-epoxy composites

The tensile properties of the E-glass-epoxy composites with varying amounts of the nanoclay (0 to 5wt %) are shown in **Table 1**. It was observed that the tensile strength increased marginally with increasing nanoclay until 3wt%, beyond which the tensile strength decreased. Nanoclay is not known to act as a load bearing reinforcement in the fiber reinforced composites. Yet, nanoclay addition increased the tensile strength. This indicates that the nanoclay present in the composites ensures more load bearing by the E-glass fibers as compared to the composite without nanoclay. This could be attributed to the ability of the nanoclay to ensure

more elongation to the composite before its ultimate tensile failure (**Fig. 4a**). More elongation in turn can be attributed to the enhanced interface toughness in the composite, which ensures more resistance to interface crack propagation [**22**]. This leads to better utilization of the E-glass fiber mechanical properties in the composite. Enhanced matrix toughness due to the nanoclay is evidenced from the strong inter-filament bonding and rough fracture surfaces of the nanoclay-glass-epoxy composites failed under tensile loads (**Fig. 3a & b**).

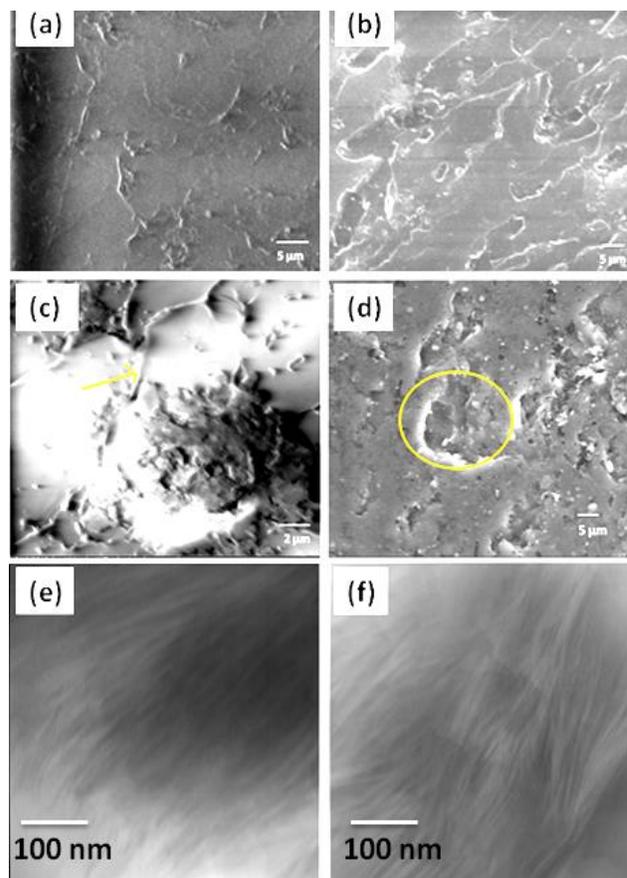


Fig. 2. Typical SEM images (a-d) of the nanoclay-epoxy nanocomposites failed under; fracture toughness test. (a) 0wt% nanoclay-epoxy showing very smooth surface, (b) 3.0wt% nanoclay-epoxy showing ductile like failure with many planes of cleavage giving rough surface, (c) magnified image of 3.0wt% nanoclay-epoxy showing crack deflection around nanoclay (indicated by arrow), (d) 5.0wt% nanoclay-epoxy showing agglomeration and spallation of nanoclay from surface (encircled zone), TEM images of both, (e) 3wt% nanoclay-epoxy and (f) 5wt% nanoclay-epoxy shows good dispersion of nanoclay in an epoxy matrix.

Direct relation between the matrix toughness of the nanoclay-epoxy and the tensile strength of nanoclay-E-glass-epoxy composites can be evidenced (**Table 1**). Both have shown their peak values at 3wt% addition of nanoclay and both have shown reduced values at 5wt% of nanoclay. Fracture surface of 5wt% addition of nanoclay-E-glass-epoxy was observed to have more interfilament debonding (**Fig. 3c**) as compared to 3wt% nanoclay-E-glass-epoxy. This indicates that 3wt% addition of nanoclay is optimum to get better interface toughness. Matrix embrittlement observed at 5wt% addition of nanoclay decreases the tensile strength of 5wt% nanoclay E-glass-epoxy composite.

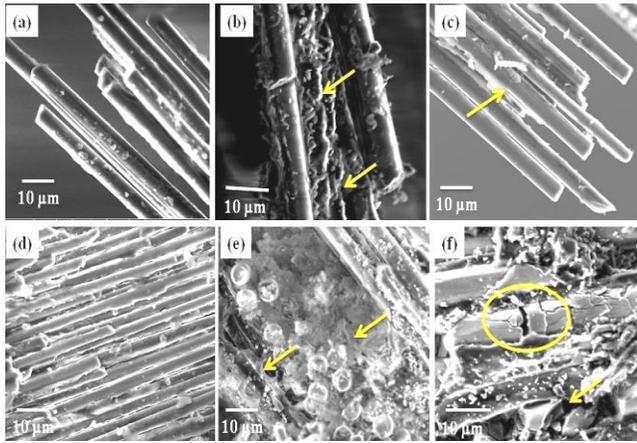


Fig. 3. SEM images of E-glass epoxy composites after failure under tensile (a, b, and c) and flexural load (d, e, and f). (a) Blank showing more fiber matrix debonding with smooth surfaces, (b) 3.0wt% nanoclay E-glass-epoxy showing better resin toughening and strong interfacial bonding between E-glass fiber and matrix, (c) 5.0 wt% nanoclay E-glass-epoxy showing poor interfilament bonding between the matrix and fiber, (d) Blank showing the interfilament debonding with smooth fiber surfaces, (e) 3.0wt% nanoclay E-glass epoxy showing good interfilament bonding and (f) 5.0wt% nanoclay showing micro cracks at the interface (indicated by arrow) and more fiber damage (encircled zone).

Flexural strength

Similar to the trend observed for the toughness of the nanoclay-epoxy composites, flexural strength of the nanoclay-E-glass epoxy composites have shown the highest flexural strength at 3wt% addition of nanoclay, beyond which it decreased (Table 1 & Fig. 4b). SEM images of the samples failed under flexural load indicated the maximum interfilament debonding in the blank (Fig. 3d) as compared to 3wt% nanoclay E-glass-epoxy, which has shown a good, interfilament bonding (Fig. 3e). This feature improves bending resistance. Besides this, improved matrix rigidity due to nanoclay addition could have resisted bending loads effectively, which contributed to the improved flexural strength [23]. However, the addition of higher amounts of clay (up to 5wt %) decreased the flexural strength due to embrittlement of the matrix, leading to early failure of fibers and premature failure of the composite (Fig. 3f).

Table 2. Impact and Damage parameters of nanoclay E-Glass epoxy composites at various wt% of nanoclay.

Wt% of nanoclay	Peak load (N)	Deflection at peak load (mm)	Force at puncture (N)
Blank	10983	4.846	5490.28
1%	11328	4.482	5857.77
3%	14057	3.807	6906.34
5%	14280	3.684	7129.28

Impact response

Typical load-time curves recorded in the impact test for E-glass-epoxy composites are shown in Fig. 5. Peak load in the elastic part of the curves indicates energy to puncture. Total area under the curves indicates the overall energy absorbed by the composite before failure. Peak load, deflection at peak load and force at puncture values are shown in Table 2. It can be seen that as the wt% of

nanoclay increased, the load required to puncture the laminate increased drastically with a simultaneous increase of the absorbed energy. The toughness of epoxy matrix was reduced beyond 3wt% loading of nanoclay impact resistance of the nanoclay-E-glass-epoxy continued to increase up to 5wt% addition of nanoclay. This indicates that, matrix embrittlement that is observed at 5wt% nanoclay addition is helping in imparting increased impact strength to the composites. This can be interpreted as follows.

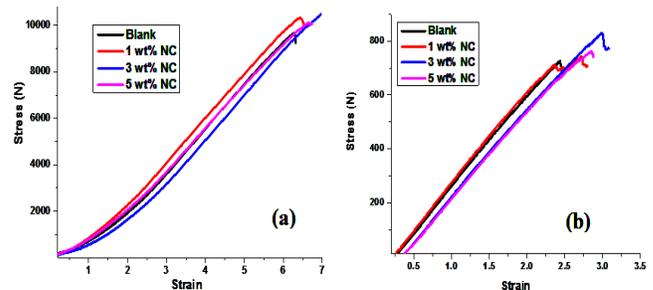


Fig. 4. Stress-Strain curves of various nanoclay-E-glass-epoxy composites failed under (a) Tensile load and (b) Flexural load.

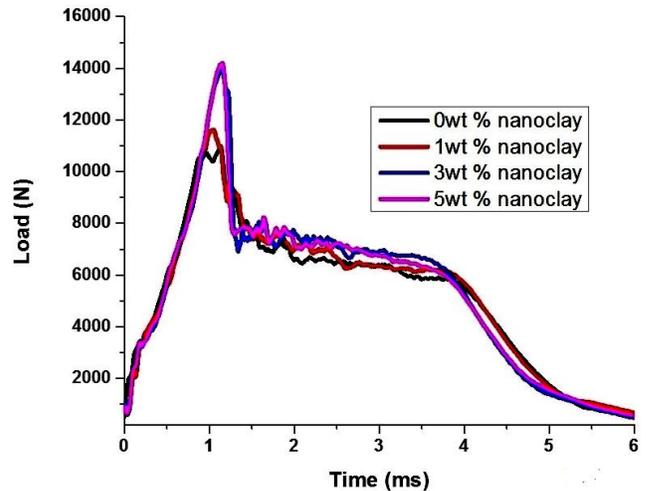


Fig. 5. The load vs time response of the E-Glass-epoxy composites containing 0%, 1.0%, 3.0% and 5wt% nanoclays.

Failure due to impact damage can be divided into two modes as shown below:

- Local region damage due to contact stress: In this mode of failure, damage is due to indentation which is more localized, with fiber fracture and matrix cracking within a distance of one contact radius of the loaded surface with negligible global deflection [24]. In this mode, the impact energy will permeate through the thickness towards the rear side of the composite (a vertical spread of impact energy).
- Transverse shear failure: Out of plane stresses generated due to impact results in flexure, transverse shear forces across the thickness of the composite. This result in mode II shears i.e. delamination growth between various layers. The region affected by this kind of failures is conically expanding towards the non-impacted face of the laminate. This represents the horizontal spread of the impact energy.

In general, during impact, up to a certain thickness of the composite, only indentation damage will be observed. More is the penetration of indenter; more will be the back face damage due to more vertical permeation of impact energy increases. This is because; the reinforcement layers (E-glass fabric layers in the present case) which get indented fail locally. This limits the contribution of these reinforcement's layers to consume impact energy by mode II shear. In other words, it can be said that, if the horizontal spread of damage initiates closer to the front face, impact energy reaching to the back face will be minimized.

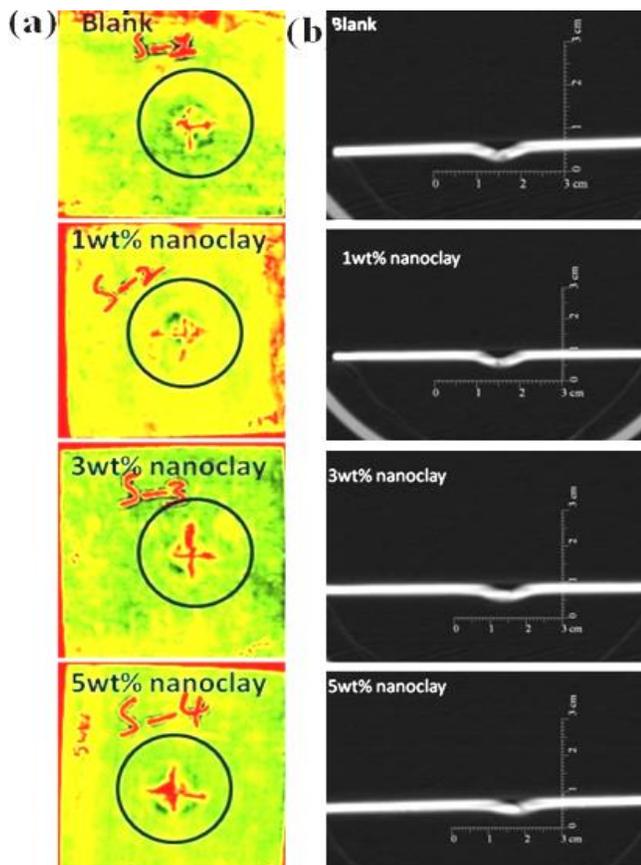


Fig. 6. Images of damaged E-glass-epoxy composite samples after impact test (a) Thermograph images of the front face, 5wt% nanoclay added E-glass-epoxy showing maximum damage area on front face (b) CT-Scan image showing magnitude of bending deformation of 5wt% nanoclay added E-glass-epoxy composite which has shown lower magnitude of damage to the back face.

Damage to front face

It can be seen from the thermography image (**Fig. 6a**) that, a damage area on the front face of the glass-epoxy is minimum for the blank. This indicates that, front layers in the laminate are being indented vertically. This indicates a localized damage to the front layers. As the weight percentage of the nanoclay is increased up to 3wt%, there is only a marginal increase in the damage of the front face, but the damage area increased drastically when 5wt% nanoclay was added E-glass-epoxy composite. Thus, at 5wt% loading of nanoclay, the more horizontal spread of impact energy from the topmost layers of the laminates is observed (**Fig. 6b**). Hence, vertical spread of damage towards the back face is minimized.

Damage to the back face

It was observed that the addition of the nanoclay to glass-epoxy up to 3wt% has not shown a significant reduction in the damage on the back face. This indicates that toughness improvements of the matrix due to nanoclay addition as well as tensile and flexural strength improvements of composites could not control the damage on the back face. However it was seen that, 5wt% nanoclay addition to composite has shown lower damage to the back face than any other composition (**Fig. 6b**). This trend can be reasoned as below:

Mechanism of damage resistance for 5wt% nanoclay-E-glass-epoxy

Additional cross linking that takes place due to the amino functional groups increase the toughness of epoxy up to 3wt%, beyond which it embrittled the matrix. This indicates that, at 5wt% addition of nanoclay, the clay rich regions of the matrix behave like localized embrittled zones. Thus, when the 5wt% nanoclay composite undergoes impacting on the front surface, micro cracks originate and propagate around the localized pockets of embrittled zones. As the micro crack circumvents the embrittled zone, the embrittled zones undergo spallation from the matrix (**Fig. 2d**). Thus, in 5wt% nanoclay-E-glass-epoxy composite, facile shattering and spallation of nanoclay rich zones could spread transverse shear failure of the composite laminates (horizontal damage) even from the topmost layers (**Fig. 6a**) thereby involving more composite area in absorbing the impact energy. Thus, as more energy is consumed in creating horizontal damage in 5wt% nanoclay glass-epoxy composite, vertical penetration of the damage has become minimum thereby causing minimum bending of the back face (**Fig. 6b**).

Conclusion

Nanoclay-epoxy and nanoclay-E-glass-epoxy composites with varying amounts of nanoclay were fabricated. Fracture toughness for the nanoclay-epoxy composites and tensile, flexural and impact properties of the nanoclay-E-glass-epoxy composites were measured. The important findings are summarized as below:

- (1) Amino groups present on the nanoclay react with the epoxy matrix and enhances the crosslink density. Toughness of nanoclay- epoxy composites increases up to a critical crosslink density improvement.
- (2) Even though the toughness of epoxy and mechanical properties of the E-glass-epoxy composites decreases at 5wt% addition of nanoclay, the best impact resistance with minimum damage to the back face was observed. This may be due to highest energy consumption of 5wt% nanoclay composite during shattering of the brittle nanoclay rich zones.
- (3) Observed trends indicated that the creation of localized cells of embrittled zones in E-glass-epoxy with nanoclay addition can effectively resist the impact damage, even though there is a reduction in other mechanical properties. These results may lead to the

design and realization of glass-epoxy composites with better impact strength.

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