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# Mechanical Properties of Nanoclay Reinforced Epoxy Adhesive Bonded Joints Made with Composite Materials

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**Abstract**

This study was intended to characterize the mechanical properties of nanoclay filled epoxy adhesive single lap joints under both static (tensile) and dynamic (impact) loadings. The nanoclay contents were 1, 3 and 5 wt% of epoxy resin (Araldite LY5052). The nanoclay particles were dispersed in the epoxy resin by a stirring device and then the mixture of resin and clay particles was subjected to sonication using an ultrasonicator. Glass/epoxy composite adherends were fabricated and used to study the behavior of epoxy adhesive bonded joints. The composite adherend surfaces were prepared according to ASTM D2093. According to ASTM D3165, the properties of the reinforced adhesive in shear were obtained by tension loading of single lap joint specimens. Also, the joints were subjected to in-plane and out-of-plane Charpy impact tests. The results showed that the adhesive joints with 1% nanoclay particles had the maximum strength in tensile loading and the highest values of Charpy impact energy were found for the joints with adhesives filled with 3% nanoclay particles.

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**Keywords**

Nanoclay particles, adhesive bond, composite adherends, impact, tensile loading

## 1. Introduction

The number of applications of adhesives in bonding structural components is rising rapidly with particular interest in aerospace applications and microelectronics packaging which require bond integrity over a spectrum of loads and temperature ranges. Features which make adhesive bonding attractive include improved appearance, good sealing, high strength to weight ratio, low stress concentration, low cost,

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corrosion resistance, and fatigue resistance. The rapid development of structural adhesives has led to the widespread use of adhesive joining technique in defense, aerospace, rail and ground transportation applications. In these applications, the joints are designed to carry in-plane loads, although they are also prone to transverse loading from crashes, bullets, fragments, tool drops or flying debris. The usage of bonded joints in structures, especially in aerospace and military applications, makes it important to understand their failure mechanisms under transverse and in-plane loadings.

The thermal evaluation of adhesive structure was carried out by various authors [1, 2]. Graphite materials were bonded by adhesives and heat-treated at temperatures ranging from 200 to 1500°C. Several high-temperature adhesives (HTAs) were prepared: a neat phenol-formaldehyde (PF), a phenol-formaldehyde (PF) resin with boron carbide ($PF + B_4C$) and a PF resin with $B_4C$ and fumed silica ($PF + B_4C + SiO_2$). Results showed that the PF adhesive which was reinforced with $B_4C$ particles and $SiO_2$ nanoparticles was found to have outstanding high temperature properties for graphite bonding. The adhesive structure was dense and uniform even after the graphite joints were heat-treated at 1500°C, with a bond strength of 17.1 MPa. The addition of the secondary additive, i.e., fumed silica, improved the bond performance considerably. A borosilicate phase with better stability was formed during the heat-treatment process, and the volume shrinkage was restrained effectively, which was responsible for the satisfactory high-temperature bond performance of graphite.

Single lap adhesively bonded joints were subjected to in-plane and out-of-plane loads in [3]. The response of the joint to a transverse normal impact load was investigated by LS-DYNA 3D finite element software. It was found that the transverse normal load results in higher peel stress concentration in the adhesive layer as compared to in-plane loading. The increase in peel stress was due to considerable deflection of the joint under transverse normal load. Experiments involving low velocity impact (LVI) tests were carried out on the bonded joints to verify the results from the finite element model. The addition of nanoclay was found to increase the Young's modulus of the adhesive by 20%, while decreasing the ultimate failure strain by 33%. However, no significant difference in the failure energy was observed for the joint fabricated with neat epoxy *versus* that fabricated with nanoclay-reinforced epoxy.

The mechanical properties of nanocomposites and carbon fiber reinforced polymers (CFRPs) containing nanoclay particles in the epoxy matrix were investigated in [4]. Morphological studies using Transmission Electron Microscopy (TEM) revealed that the clay particles within the epoxy resin were intercalated or orderly exfoliated. The nanoclay particles brought a significant improvement in flexural modulus, especially in the first few wt%, and the improvement in flexural modulus was at the expense of a reduction in flexural strength. Flexural properties of CFRPs

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containing nanoclay modified epoxy matrix generally followed a trend similar to the epoxy nanocomposites although the variation was much smaller for the CFRPs.

Adhesive properties of thermoplastic polyimide (PI) filled with multi-wall carbon nano-tubes (MWNTs) and aluminum nitride (AlN) nano-powder were investigated in [5, 6]. The MWNTs and AlN in low wt% (up to 1.5%) were added to the PI and mixed by mechanical agitation and sonication. Adhesive strength of bonded steel samples prepared with PI-MWNT and PI-AlN composite films were determined using single lap joint tests. The results showed that the elastic modulus and strength at break of PI composite films increased with an increase in powder content, whereas strain-at-break decreased. However, elongation-at-break of PI-MWNT composite films and lap shear strength (LSS) of bonded samples were found to initially increase with an increase in powder content, but decreased after a critical value.

The present study was intended to characterize the mechanical properties of single lap adhesively bonded joints with nanoclay reinforced epoxy adhesive films under both static (tensile) and dynamic (impact) loads. Experiments were carried out to compare the effects of various contents of nanoclay particles in epoxy resin.

## 2. Experimental

### 2.1. Adhesive and Sonication Procedure

Nanoclay particles (Southern Clay Products Inc. (USA) Cloisite® 30B Nanoclay) were used as nano-reinforcement for this study. Cloisite® nanoclays are high aspect ratio additives based on montmorillonite clay, designed and manufactured for the plastics industry. Benefits from Cloisite® technology result, in part, from the very high surface area of the montmorillonite clay and its high aspect ratio.

The nanoclay particles are formed from clay platelets which are stacked together. The thickness and aspect ratio of the clay platelet are about 1 nm and 100, respectively [7]. The particle size, bulk density and moisture content of the nanoclay particles were 2 μm, 0.2283 g/cm$^3$ and less than 2%, respectively.

The nanoclay particles used in this work were a natural montmorillonite, modified with a quaternary ammonium salt. Since montmorillonite clay is hydrophilic, it is not inherently compatible with most polymers and must be chemically modified to make its surface more hydrophobic. The most widely used surface treatment is the use of ammonium cations, which can be exchanged for the existing cations on the surface of the clay. The treatment works on the clay to minimize the attractive forces between the agglomerated platelets [8].

Nanomaterials, e.g., metal oxides, nanoclay particles or carbon nanotubes, tend to be agglomerated, when mixed into a liquid. Uniform dispersion of nanoclay particles in polymer matrix without aggregation and entanglement remains the biggest issue in the preparation of nanoclay-polymer composites. Investigations on dispersions of nano-particles with a variable solid content have demonstrated a consider-

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able advantage of ultrasound when compared with other technologies, such as rotor stator mixers, piston homogenizers, or wet milling methods. Hielscher ultrasonic systems can be used at fairly high solids concentrations.

Dispersion and deagglomeration by ultrasonicator is a result of ultrasonic cavitation. When an ultrasonic wave passes through a liquid medium, a large number of micro-bubbles form, grow, and collapse in a very short time of about a few microseconds; this effect is called ultrasonic cavitation. When exposing liquids to ultrasound, the sound waves that propagate into the liquid result in alternating high-pressure and low-pressure cycles. Sonochemical theory calculations and the corresponding experiments suggest that ultrasonic cavitation can generate local temperatures as high as 4500°C and local pressure as high as 50 MPa, with a high heating and cooling rate, thus creating a very rigorous environment [9]. This condition applies mechanical stress to the particles. Ultrasonic cavitation in liquids causes high speed liquid jets of up to 1000 km/h. Such jets press liquid at high pressure between the particles and separate them from each other. Smaller particles are accelerated with the liquid jets and collide at high speeds. This makes ultrasound an effective means not only for dispersing, but also for the milling of micrometer size and sub-micrometer size particles. Therefore, ultrasound is used extensively for dispersion of nano-particles in polymers. By taking advantage of ultrasound, the aggregates and entanglements of nanoclay particles are broken down using this method. It should be noted that the energy input to disperse the nanoclay particles tends to break them into shorter segments decreasing their aspect ratio in the final composite, while simultaneously increasing their dispersibility [10]. The shearing action during mixing of these particles in the epoxy causes exfoliation or separation of the platelets composing each particle, which is responsible for the reduction in particle size to the nanometer scale.

The epoxy resin and hardener selected for this study were Araldite LY 5052 and Aradur 5052 (HUNSTMAN Inc., Basel, Switzerland), respectively. According to the manufacturer, the combination of the resin and the hardener results in a brittle adhesive. The properties of the adhesive are: ultimate tensile strength: 49–71 MPa, tensile modulus: 3.3–3.5 GPa, inter-laminar shear strength (cured with 60% unidirectional E-glass fibres): 57–61 MPa, fracture toughness: 0.77 $\mathrm{MPa}\sqrt{\mathrm{m}}$, and elongation-at-break: 1.5–2.5%.

Nanoclay/epoxy composite samples were fabricated by using a mechanical mixing process with nanoclay particle contents 1, 3 and 5 wt%. The nanoclay content was used in order to achieve maximum mechanical strength of the adhesive bonded joint. Nanoclay particles were dispersed in the epoxy resin. The mixture was hand stirred for 15 min, until the epoxy resin and the nanoclay particles were well mixed at room temperature. Ultrasound sonication was employed to further disperse the nanoclay particles in the epoxy resin. The sonication time was fixed at 25 min for all the samples in order to ensure the maximum mechanical performance [11]. Hardener was added into the sonicated mixtures in a ratio of 1:10 by weight.

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### *2.2. Adherends and Surface Preparation*

Glass/epoxy composite adherends were chosen to study the behavior of adhesively bonded single lap joints under static (tension) and dynamic (impact) loads. The composite adherends were fabricated from eight layers of epoxy resin and woven C-glass fibers with 0°/90° direction. The thickness of each layer was 0.2 mm.

As is well known that when using adhesives, surface preparation is one of the most vital parameters in achieving high bond strength and optimising durability and lifespan of bonded joints. Different adherend materials require different types of adhesives for bonding and different methods for surface preparation. The bonding surfaces were prepared according to ASTM D2093-97 [12]. First, the bonding surface was cleaned with acetone, and then sanded with 240- and 1000-grit silicon carbide papers. It was then immersed in dichromate sulfuric acid solution for 60 min at 20–30°C and cleaned with acetone again and wiped in dry air at 40°C with a lint-free paper to remove any foreign particles.

### *2.3. Joint Specimen Preparation and Test*

The joint fabrication started with wetting the surface area to be joined. Therefore, the adhesive was placed on both adherends along the center line of the bond area and the adherends were brought in contact along the bond area. Application of pressure squeezed the extra adhesive out. The specimens were allowed to cure at room temperature for 20 days. Specimens were prepared with four different types of adhesives: neat epoxy and epoxy reinforced with 1, 3 and 5 wt% nanoclay particles.

Specimens for static loading were prepared according to ASTM D3165-95 [13]. This test method was intended for determining the comparative average shear strength of the adhesive, when tested in a standard single lap joint specimen under tension. The dimensions of the adherends used were 101.6 mm × 25.4 mm × 1.6 mm. The bonded area of the joint was 25.4 mm × 12.7 mm with a thickness of 0.2 mm (Fig. 1).

The adherends for Charpy impact test were cut into dimensions of 40 mm × 6 mm × 1.6 mm and the bonded area of the joint was 6 mm × 10 mm (Fig. 2). Charpy impact tests (Fig. 3(a)) were conducted on the specimens and the Charpy

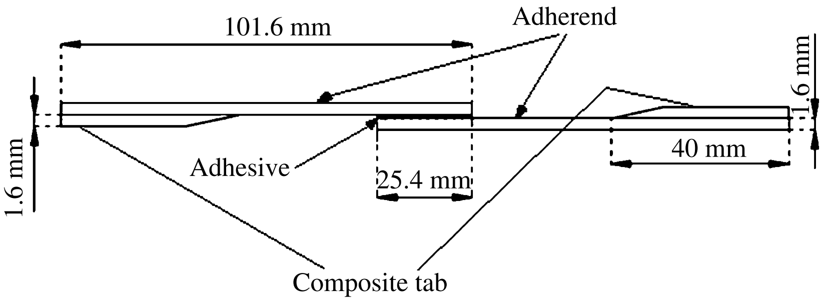

**Figure 1.** Single lap joint specimen geometry for tensile tests.

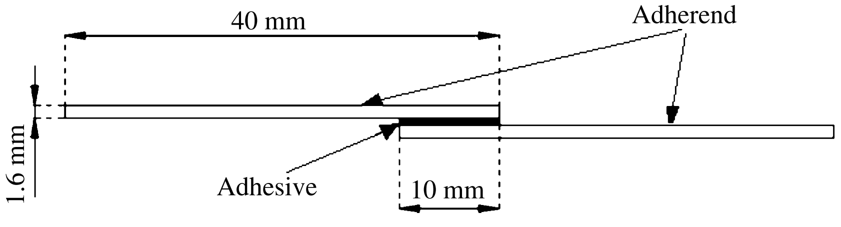

**Figure 2.** Single lap joint specimen geometry for Charpy impact tests.

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impact energy was obtained for both in-plane and out-of-plane impact loads as shown in Fig. 3(b) and 3(c). The middle section of the bondline was subjected to impact loads.

## 3. Results and Discussion

### *3.1. Lap Shear Strength*

Adhesive joints prepared with epoxy/nanoclay composite films were tested for lap shear strength (LSS) in tension. Joints prepared with various contents of nanoclay particles were tested to establish a comparison. The adhesive strength mainly depends on two parameters: (a) mechanical properties of the epoxy resin and nanoclay particles and (b) resin viscoelastic behavior and adhesion properties. In previous studies on adhesive joints with epoxy [5], it was observed that with increasing nanoclay particle content, the mechanical properties increased, but their viscoelastic behavior made a transition from liquid-like to solid-like. Therefore, it was likely that epoxy containing high wt% of nanoclay particles will not have good adhesion properties.

The results of LSS measurements in tension on bonded joints prepared with epoxy containing various contents of nanoclay particles were obtained using the load-displacement diagrams and then converted to shear stress–shear strain (%) diagram as shown in Fig. 4. The shear stress was calculated from load applied on adherend divided by adhesive area, and the joint shear strain in Fig. 4 was obtained from joint displacement divided by overlap length. The standard deviations for the results shown in Fig. 4 vary between 0.06 to 0.16 MPa. The average single lap shear strength is evaluated using the maximum tensile shear stress obtained in Fig. 4. It can be observed that the LSS increased by adding nanoclay particles, but above 1% nanoclay particles, LSS decreased. The LSS values for neat epoxy and epoxy reinforced with 1, 3 and 5 wt% nanoclay particles are 7.26, 7.77, 6.49 and 5.92 MPa, respectively. The LSS increased about 7% by adding 1 wt% nanoclay particles in the epoxy resin. Table 1 shows the results obtained from the above tensile test. From Table 1, it can be concluded that the highest joint stiffness (557 MPa) is obtained in the case of the joint with epoxy containing 1 wt% nanoclay particles. It increased by 28% with respect to neat epoxy. The joint with the neat epoxy had the lowest

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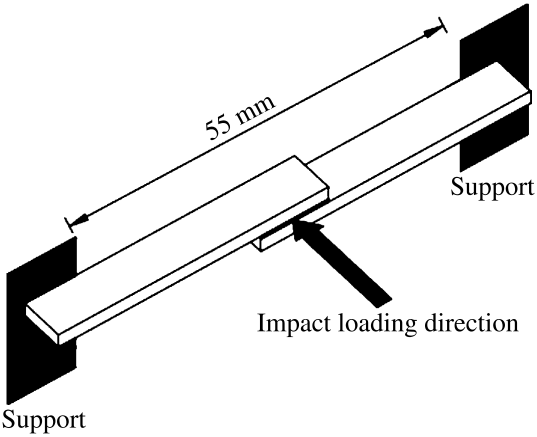
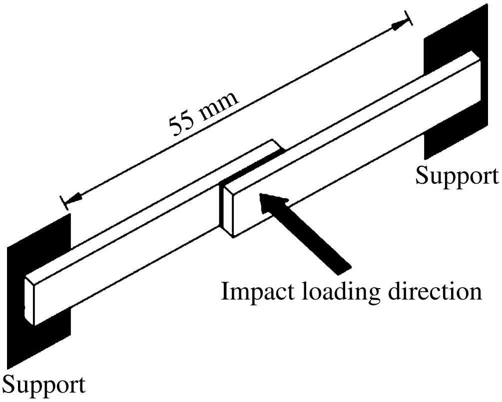
(a)



(b)



(c)

**Figure 3.** (a) Charpy impact test. (b) Charpy impact test, in-plane loading. (c) Charpy impact test, out-of-plane loading.

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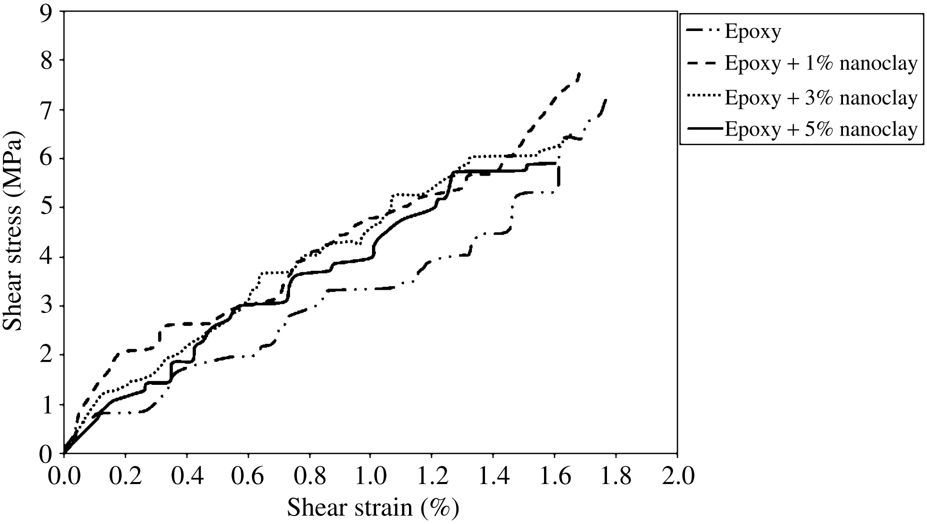

**Figure 4.** Shear stress–shear strain curves for single lap adhesive joints under tensile loading.

**Table 1.**
Results on single lap joints tested in tension

| Property | Sample | | | |
|---|---|---|---|---|
| | Neat epoxy | Epoxy + 1 wt% nanoclay | Epoxy + 3 wt% nanoclay | Epoxy + 5 wt% nanoclay |
| Average lap shear strength (LSS) (MPa) | 7.26 | 7.77 | 6.49 | 5.92 |
| Standard deviation | 0.035 | 0.087 | 0.084 | 0.14 |
| Strain at break (%) | 1.78 | 1.68 | 1.65 | 1.6 |
| Joint stiffness (MPa) | 434 | 557 | 514 | 485 |
| Energy absorption (J) | 0.27 | 0.33 | 0.29 | 0.23 |
| Joint stiffness increase in comparison to neat epoxy (%) | – | 28.3 | 18.4 | 11.8 |

stiffness (434 MPa). Adding nanoclay particles leads to increase of the stiffness of the joint. The energy absorption indicated in the table means the area under the shear stress-strain curve in Fig. 4. The energy absorption for the specimen with 1 wt% nanoclay particles is 22% greater than that of the neat epoxy bonded joint specimen. Adding nanoclay particles to the adhesive results in a better toughness behavior of the joint, but the strain at break of the joint reduced with increasing nanoclay particles in the epoxy resin. In effect, adding the nanoclay particles to the adhesive causes the adhesive bonded joints to be stiffer and hence suitable for applications where the elastic deformation should be less.

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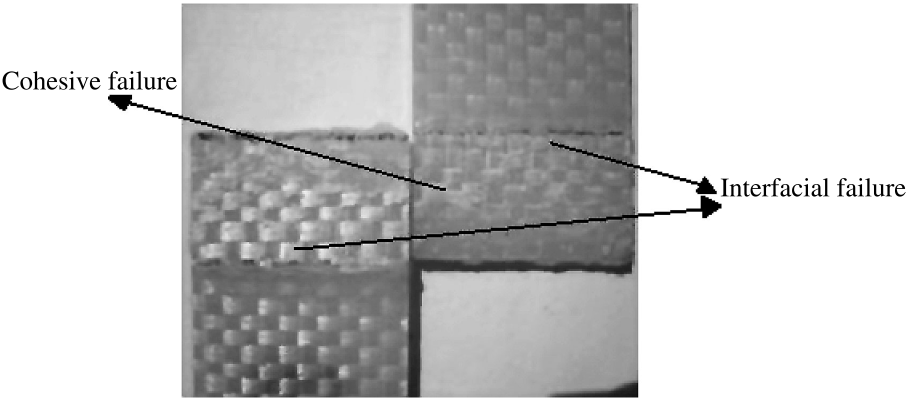

**Figure 5.** Fracture surfaces of adhesive joint specimens after failure in the lap shear test (epoxy + 1% nanoclay).

Despite the fact that the joint stiffness and the average lap shear strength of the adhesive joint were found to increase with increasing nanoclay particle wt%, it also resulted in a poor adhesion between adherends and adhesive. This poor adhesion hinders the proper wetting of the adherend surface, therefore results in a reduced LSS compared to that of the neat adhesive joint. In addition, stress singularities at the interface edges in bonded lap joints are present for all specimens. This is mainly because of joint geometry and sharp discontinuities at the bond edges. The stress concentration at the edges of the joint has two components: the in-plane shear stress, and the transverse normal stress (peel stress). The peel stress is caused by the eccentricity in the load path, which can also cause significant bending of adherends, thus magnifying the peel stress. The peel stress is more detrimental to the joint performance than the shear stress. As shown in Fig. 5, the stresses cause the adhesive to separate from the surface of the adherends at the edges. As a result, fibers in the composite adherends were visible and hence the interfacial failure had occurred. The analysis of fractured surfaces indicates a mixed interfacial and cohesive failures. Near the edges, the failure is interfacial and in the middle of the overlap there is a cohesive failure, as shown in Fig. 5 for 1 wt% nanoclay particle filled epoxy adhesive.

### *3.2. Charpy Impact Energy*

The results from the Charpy impact tests for the specimens with nanoclay-reinforced epoxy and neat epoxy are shown in Table 2. This table shows the work done by the impactor (energy absorbed by the joint) for both in-plane and out-of-plane loads. The Charpy impact energy increased with nanoclay particle content up to 3 wt% and decreased for 5 wt% nanoclay particles. The 3 wt% nanoclay addition to the epoxy led to an increase in Charpy impact energy by almost 1.5 times (percentage increase is 45%) and 3 times (percentage increase is 173%) for in-plane

**Table 2.**
Charpy impact energy test results

| Property | Sample | | | |
|---|---|---|---|---|
| | Neat epoxy | Epoxy + 1 wt% nanoclay | Epoxy + 3 wt% nanoclay | Epoxy + 5 wt% nanoclay |
| Energy for in-plane loading (kJ/m$^2$) | 3.33 | 3.83 | 4.83 | 4.17 |
| Standard deviation | 0.12 | 0.15 | 0.2 | 0.1 |
| Energy for out-of-plane loading (kJ/m$^2$) | 0.67 | 1.42 | 1.83 | 1.00 |
| Standard deviation | 0.1 | 0.11 | 0.06 | 0.1 |

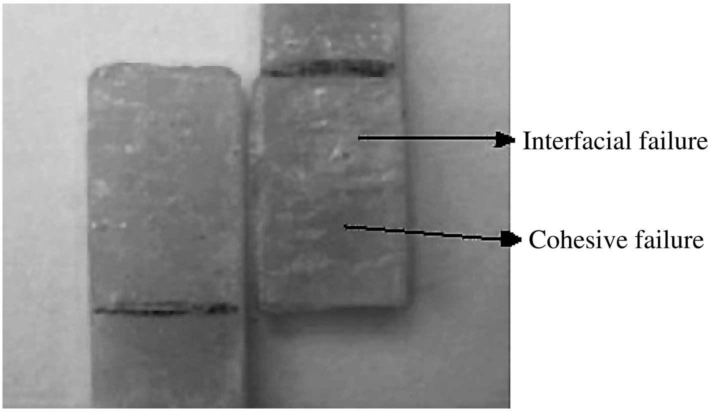

**Figure 6.** Fracture surfaces of adhesive joint specimens after failure in out-of-plane impact test (epoxy + 3% nanoclay).

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and out-of-plane impact loads, respectively, when compared with neat epoxy. It was found that the out-of-plane impact load results in higher peel stress concentration in the adhesive layer as compared to in-plane loading. The increase in peel stress is due to considerable deflection of the joint under transverse normal load. As can be seen in Fig. 6, the failure in the case of out-of-plane loading initiates by interfacial failure and then changes to cohesive failure. In the case of in-plane impact loading, where shear loading dominates, the failure is more cohesive. It seems that the adhesive joint with nanoclay particles has higher tensile strength and stiffness than shear strength and stiffness due to the high tensile properties of adhesive and nano-particles, the aspect ratio of the nanoclay platelets and the interfacial behavior between the clay and the adhesive matrix. Therefore, the effect of nanoclay particles in out-of-plane impact loading is more tangible than in-plane loading.

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The exfoliated nanoclay platelets were re-aggregated into nanoclay particles during the composite preparation process. When the nanoclay weight concentration is higher, thicker particles can be found and the nanoclay particles dispersion is less uniform with a further increase in nanoclay content. The high nanoclay concentration in the matrix as well as the large aspect ratio and high surface to volume ratio of the nanoclay platelets make it difficult to obtain a uniform dispersion in the epoxy [7]. Furthermore, the number of micro-bubbles, which are caused by ultrasonic cavitation, is increased with higher nanoclay particle content. In addition, by adding 5 wt% nanoclay particles, poor adhesion occurred due to poor wetting of the adherend surface. Therefore, the impact energy absorption is reduced for reinforced joint with high wt% nanoclay particles for both in-plane and out-of-plane impact loadings.

## 4. Conclusion

In this paper, an experimental study to investigate the behavior of adhesive joints using adhesive reinforced with nanoclay particles in static tension and impact loadings was carried out. Various concentrations of particles, namely 1, 3 and 5 wt% of nanoclay particles, were added to the adhesive in single lap joints and the mechanical properties were studied. The following experimental results were obtained:

1. The lap shear strength increased by adding nanoclay particles up to 1 wt% and beyond that the LSS decreased.
2. The stiffness of the joint increased by adding nanoclay particles to the epoxy adhesive. The maximum joint stiffness was obtained for the joint with 1 wt% nanoclay epoxy adhesive.
3. Strain at break was reduced by increasing nanoclay particle content in the epoxy adhesive due to brittle nature of the nanoclay particles.
4. The energy absorption for specimens with 1 wt% nanoclay particles was greater than the neat epoxy bonded joint specimen. Adding nanoclay particles to the adhesive resulted in a higher energy absorption and, therefore, higher toughness of the joint.
5. The highest values of Charpy impact energy were found for the joints with adhesives filled with 3% nanoclay particles for both in-plane and out-of-plane impact loadings. The effect of nanoclay particles in out-of-plane impact loading is more tangible than in-plane loading.
6. The analysis of fractured surfaces showed that the failure occurred in a mixed interfacial/cohesive failure modes for all the joint specimens. But, the percentage of interfacial or cohesive failure depends on the type of loading and the content of nanoclay particles.

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