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Effect of nano-α-Al_2O_3 Particles on Mechanical Properties of Glass-Fibre Reinforced Epoxy Hybrid Composites

***Anil Kumar Veerapaneni*[1]**
***Chandrasekar Kuppan*[*,2]**
***Murthy Chavali*[*,3]**

[1] *Aarshanano Composite Technologies Pvt. Ltd., Guntur District, Andhra Pradesh, India*
[2] *Division of Chemistry, Vignan's Foundation for Science, Technology and Research University (Vignan's University), Vadlamudi 522 213 Guntur District, Andhra Pradesh, India*
[3] *NTRC-MCETRC, Tenali 522201 Guntur District, Andhra Pradesh, India*
[*]*e-mail:* ChavaliM@gmail.com and ramachan16@gmail.com

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Abstract. The present work deals with the mechanical properties of hybrid nanocomposites made of epoxy/glass fibre dispersed with different weight percentages of nano-α-Al_2O_3 powder . The nanoparticles were synthesized by a high energy ball milling technique (60 and 200 nm).. The effect of nano-α-Al_2O_3 size and content (wt%) on mechanical properties, such as tensile, flexural, interlaminar shear stress (ILSS) and hardness was investigated. The addition of nano-α-Al_2O_3 enhanced all measured mechanical parameters because of their higher surface area and interfacial polymer-metal interaction. The nanoparticle-embedded laminates showed an improvement in flexural strength and hardness compared to laminate without nano-α-Al_2O_3. Among all the wt% of varied sizes of nano-α-Al_2O_3, the highest tensile strength was shown by the addition of 0.5 wt% 200nm nano-α-Al_2O_3 (167.80 N/m^2). The highest flexural strength (378.39 N/m^2) Vickers hardness (86.72) were observed for laminates containing 1.5 wt% of 60nm nano-α-Al_2O_3, while the highest ILSS (31.21 Ksi) was observed for 0.5 wt% of 60nm nano-α-Al_2O_3. This study showed that there was a higher interaction between the nanoparticle and polymer resin, which led to increasing the mechanical properties of the laminate. This finding show that diversifiying the application of these hybrid materials was possible by adding nano-alumina.

Keywords: Alumina, epoxy resin, hardener, mechanical properties, SEM and XRD

INTRODUCTION

The development of novel reinforced composite materials or alteration of existing composite material to face reality is very challenging for most materials scientists (Rongli et al., 2019). Compared to other old-fashioned thermoset or thermoplastic resins, epoxy resins have diverse advantages, like, increased mechanical and fatigue strength, impact-resistant, excellent moisture resistance and chemical resistance, good electrical properties and low shrinkage during cure. The addition of fibres to the polymer matrix is proven to increase the mechanical properties of the composite material as compared to the neat polymer, especially glass fibres (Abdul-Hussein et al., 2016).

Previous studies have indicated that there is a significant improvement in

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mechanical properties with epoxy matrix modification (Bahereh et al., 2016). One approach to modify the polymer matrix with nil or less covalent interaction or supramolecular interaction is to incorporate metals in their nanoscale either as free metal or metal oxide (Chavali et al., 2019). Some of the nanoparticles which are reported to enhance the properties in the presence of fibre-reinforced plastic (FRP) are nano clay, Al_2O_3, SiO_2 and TiO_2 (Merad et al., 2011; Ikram et al., 2013). Iron nanoparticles (NPs) embedded in glass fibre/epoxy composite enhanced the mechanical and magnetic properties (Fathy et al., 2015). The addition of nano clay particles also resulted in improvement of the tensile properties, which is due to higher interaction of the polymer matrix with the metal oxides in the clay structure via hydrogen bonding (Battacharya et al., 2016). Kardar et al. (2008) studied the effect of nano alumina particles on UV cured epoxy acrylate's physical and mechanical properties via nano-indentation (Kardar et al., 2008) and observed that the scratch resistance and self-healing of the film improved in the presence of nano alumina particles. Nano SiO_2 fillers showed higher elastic moduli for the nano SiO_2/epoxy composite to that of neat epoxy resins (Zhao et al., 2008) and effectively increased both the toughness and strength of epoxy resin even at low loadings (Yun et al., 2009). Previously stated well in all the works, the final properties are affected by the change in particle size, shape, and size distribution of the loaded filler, i.e., nanoparticle and resin crosslink density. Other promising fillers such as SiC, AIN and BN were also found to improve the mechanical properties (Sim et al., 2005; Ohashi et al., 2005). Among these, ceramic fillers are considered an ideal candidate for improving thermal properties. Alumina is an engineering ceramic material (Nachum et al., 2010) with many interesting properties such as wear resistance, good dielectric application (Periasamy et al., 2020), resistance to acid and alkali at elevated temperatures, excellent thermal conductivity, and can be obtained in high purity up to 99.5% (Wagih et al., 2018; Kang et al., 2005). Nano-alumina resists plastic deformation when used as a reinforcing material when dispersed with a copper matrix (Liu et al., 2016; Melaibari et al., 2019). Previously found that alumina improved wear resistance (Nassar et al., 2012), but its mechanical strength decreased with an increase in the weight proportion of the reinforcement.

In this work, we have investigated the mechanical properties of S-fibre glass-reinforced epoxy resin composite by embedding nano-α-Al_2O_3 (60 and 200 nm) with three different weight percentages (0.5, 1.0, 1.5 wt%). Subsequently, these materials were characterized by SEM and XRD for their surface morphology. Mechanical properties of nano-α-Al_2O_3 embedded glass fibre/epoxy hybrid composites were evaluated by testing their tensile, flexural, ILSS and Vickers hardness.

Despite the advantages of fibres and resins in improving the properties of laminates, several outputs cannot be controlled. Extensive research on this area led to an improvement in these properties by the addition of inorganic nanofillers. The main property which gives an improvement in the property is their surface area and their interfacial interaction. Though there are many works done on nanofillers, work on alpha-alumina is very limited. So we have attempted to study the property of the nanocomposite laminates synthesized using various sizes of α-alumina. The work is designed to study the effect of nanofiller size (here it is α-Alumina

with two different sizes 60nm and 200nm) on mechanical properties like tensile strength, flexural strength, and hardness.

MATERIALS AND METHODS

To attain the various nanocomposites, two different sizes of nano-α-Al_2O_3 (60nm and 200nm) were embedded with fibre-glass/epoxy bolstered laminates as composites. The nanocomposite samples were taken in triplicate, with 0.5%, 1% and 1.5% by weights of nano-α-Al_2O_3 along with blank without nano alumina, for concordance and a better understanding of their mechanical properties after the addition of nano-α-Al_2O_3 to the epoxy composites. The materials were made strictly following the ASTM standards (ASTM 1986; ASTM 1988) and were set for the testing of composites.

Materials

In this study, commercially available Al_2O_3 (bulk) particles were purchased from Qualitech Systems; Ludhiana. Al_2O_3 nanopowder fillers were prepared using a high energy ball milling technique (IKON INSTRUMENTS), having 14 stainless steel balls, a greener approach. Two different sizes of nanomaterials were predominantly obtained (60±5 nm and 200±5 nm) by the milling process. The main characteristics of the synthesized Al_2O_3 nanopowder are listed in Table 1.

Table 1. Main parameters of synthesized Al_2O_3 nanopowder

Crystal phase	Purity (%)	Average size (nm)	Specific surface Area, BET (m^2/g)	Density (g/cm^3)
α	≥99.90	60±5	42±5	3.11
α	≥99.85	200±5	7.6±2	3.27

Commercially available S-fibre glass woven cloth (Style-6533, 200 GSM, 6oz Plain Weave 30-inch Aerialite, see Fig. 1), which is compatible with polyester, vinyl ester and epoxy resin systems were used for the analysis. The glass fibre sheets chosen for the study were weaved in two directions. The glass fibre chosen for the study closely resembles E-fibre glass. It shows superior properties, such as 30-40% higher tensile strength, and 15-20% higher modulus, with a greater abrasion resistance, a higher temperature resistance, an increase in ind fatigue resistance, and a higher impact resistance. Therefore, with all of these features, it was expected that the mechanical properties of the designed composites will be improved.

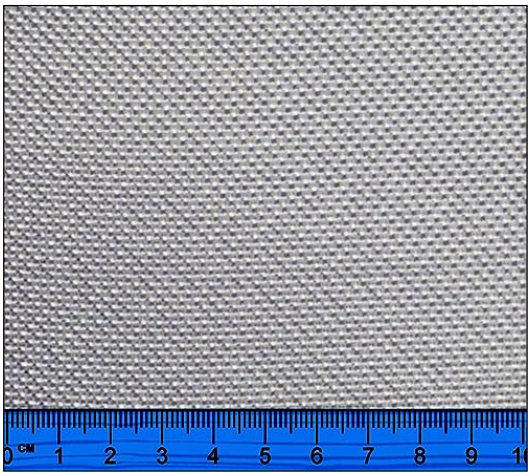

Fig. 1: White standard S-fibre glass cloth

Fabrication of hybrid nanocomposite

Hybrid nanocomposite materials with different weight percentages of nano-α-Al_2O_3 fillers were fabricated by a well-known hand lay-up method. The weight percentage of epoxy, fibre, filler and hardener were fixed, and their respective compositions were given in Table 2. The weight ratio of glass fibre to binder was 1:1.5, and preliminary experiments were conducted to assess the

dispersibility of alumina nanoparticles with varying size in epoxy glue using a mechanical stirrer. Commercial grade Epoxy resin (LY556) and hardener (HY951), was taken in the weight ratio of 10:1, respectively to make the composite material. Before the addition of the hardener, the nanomaterial in weight % was added to the matrix and dispersed evenly using a mechanical agitator for a period of 30 min. The mould was cleaned well, and the base was set with a Teflon sheet to facilitate easy removal of the composite.

The first layer of the composite was made by spreading the epoxy resin over a sheet of glass fibre inside the mould by carefully applying using a brush. Seven layers of composite with S-glass fibre were prepared to make a thickness of about 4mm. After each layer, a mild steel roller was rolled over the composite to remove the entrapped air and to maintain a uniform thickness all over the composite material. The prepared composites were allowed to cure in the mould by placing them in the oven at a temperature of 35°C for 72 hours. The cured composites were smoothened and cut into ASTM standard dimensions using a diamond cutter to measure tensile strength, flexural strength and Vickers hardness.

Characterization

The surface morphological features of the materials synthesized were studied using high-resolution scanning electron microscopy (SEM). The samples were characterized for their crystallinity and the crystal parameters by using an X-ray diffractometer (Shimadzu XRD-6000; X-ray tube: Cu Kα radiation (1.54060Å), voltage: 40.0 keV and current: 30.0 mA). The XRD-6000 boasts an integrated design featuring high speed and a high precision vertical goniometer suitable for diverse applications and data processing software supporting the Windows XP user interface. The 2θ scanning range was between 10 and 90◦, at a 5º/min scan rate.

Table 2. Description and composition of composites

Sample	Size of nano Al_2O_3	Composition of the composites
A-X-0	-	Epoxy resin (LY556) and hardener (HY951) in 10:1 ratio + S-glass fibre **(Control)**
A-X-1	60 nm	Epoxy resin (LY556) and hardener (HY951) in 10:1 ratio + S-glass fibre + 0.5 wt.% nano Al_2O_3
A-X-2	60 nm	Epoxy resin (LY556) and hardener (HY951) in 10:1 ratio + S-glass fibre + 1.0 wt.% nano Al_2O_3
A-X-3	60 nm	Epoxy resin (LY556) and hardener (HY951) in 10:1 ratio + S-glass fibre + 1.5 wt.% nano Al_2O_3
A-Y-0	-	Epoxy resin (LY556) and hardener (HY951) in 10:1 ratio + S-glass fibre **(Control)**
A-Y-1	200 nm	Epoxy resin (LY556) and hardener (HY951) in 10:1 ratio + S-glass fibre + 0.5 wt.% nano Al_2O_3
A-Y-2	200 nm	Epoxy resin (LY556) and hardener (HY951) in 10:1 ratio + S-glass fibre + 1.0 wt.% nano Al_2O_3
A-Y-3	200 nm	Epoxy resin (LY556) and hardener (HY951) in 10:1 ratio + S-glass fibre + 1.5 wt.% nano Al_2O_3

RESULTS AND DISCUSSION

The influence of nanoparticle on the enhancement of material properties of epoxy-based fibreglass reinforced composites was analyzed by using nano alpha-alumina, synthesized by a ball milling approach. The measured size of the as-synthesized nanoparticle by SEM was around 60 and 200nm, (Fig. 2a). The purity of the nanopowder was confirmed using XRD, where the peaks corresponding to the crystalline phase of α-alumina (Fig. 2b) matched with the XRD patterns reported in the literature (Pu et al., 2015).

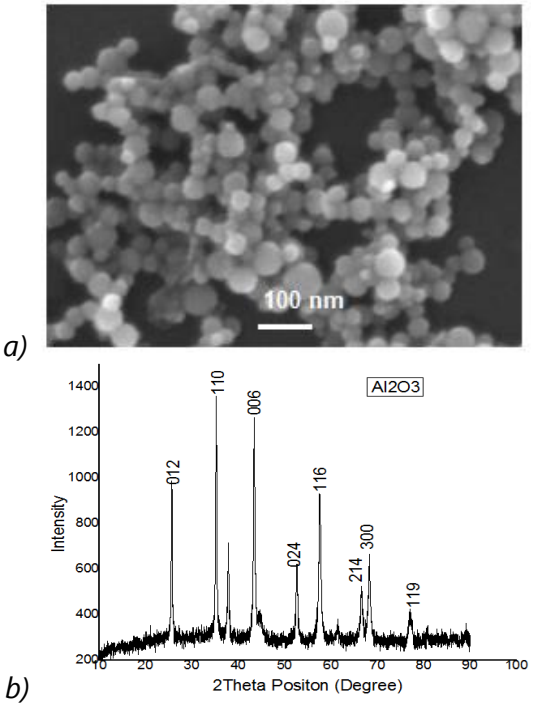

Fig. 2a: SEM image of the nano-α-Al_2O_3 powder, **2b**: X-ray diffraction pattern of nano-α-Al_2O_3

Tensile strength

The quality of engineering materials was assessed by their ability to withstand applied stress. The maximum tolerable force or stress while being stretched or pulled before breaking was defined as Tensile Strength. For this study, the composites were prepared as per ASTM D-638 standards with and without nanofiller. The tensile tests were measured using Universal Testing Machine. Using the stress-strain data from UTM, the tensile strength was estimated for alumina nanoparticle embedded epoxy-GF composites of two different sizes (60 nm and 200 nm) as a function of its wt% (Fig. 3). It is observed from the results that the tensile strengths for 60 nm laminate showed a decreasing trend with the increase of % addition of nano alumina. Whereas for 200 nm nano alumina samples, the highest tensile strength of 167.8 N/m^2 was recorded for 0.5 wt% nano alumina. Further increase in alumina content showed a decrease in tensile strength. These results indicate that the composition of the materials and the particle size are the major factors controlling the mechanical properties of the composites.

Compared to the blank nano composite (136 N/m^2), 60 nm hybrid materials showed a gradual decrease in the tensile strength from 128 to 96 N/m^2, which can be attributed to either non-uniform dispersion of the nanomaterial in the composite or higher % of nanomaterial (i.e. the 60nm composite might exhibit a maximum tensile strength below 0.5 wt% of alumina). The decreasing trend of tensile strength with an increasing content of alumina in the composite might be due to lesser interaction of the epoxy with the nanofiller.

Despite the smaller size and high surface area (45 m^2/g), the strength of the laminate was not increased compared to the blank due to agglomeration of the nanoparticle before it was dispersed into the matrix or a decreased % of surface hydroxy over alumina. Since increasing alumina percentage in the composite did not improve

the tensile strength, we concluded that 60 nm alumina fillers were not the optimum nano size for alumina-based epoxy-GF laminates.

In the case of 200nm particle composites with 0.5wt% alumina, the tensile strength value increased 20%, from 136 to 168N/m^2, which may be caused by the higher interaction of the metal nanoparticle surface with the epoxy-glass filler matrix. With increasing content of alumina in the composite, the tensile strength decreased drastically up to 78N/m^2 for 1.5% alumina, where the chance of nanoparticle agglomeration increases with increasing wt%, which reduces the number of surface anchoring hydroxy groups (the larger the particle size, the lesser the surface area) and ultimately the strength of the laminate.

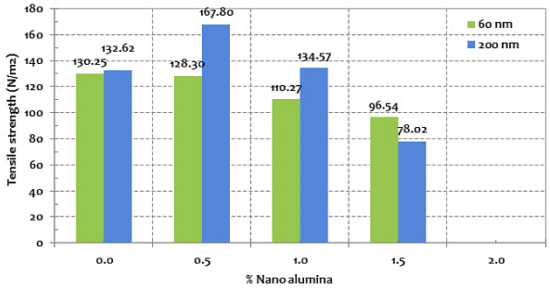

Fig. 3: Tensile strength for nano-α-Al_2O_3 based epoxy-Glass fibre hybrid laminates at 60 nm and 200 nm as a function of Wt%

Flexural strength

Flexural strength (FS) was calculated by performing short beam shear (SBS) test; conducted as per the ASTM- D2344/D2344M-00 standards at room temperature. Samples with dimensions of 25 x 11 x 5.5 mm and a span length of 22mm were used for the analysis. The flexural strength was calculated using Eq. 1. This equation is suitable for a rectangular sample under a load in a three-point bending setup.

$$FS = \frac{3\,PL}{2bt^2} \quad \text{..... (1)}$$

Where FS is flexural strength (MPa), P equates force at fracture (N), and L, b, and t represents the length (mm), the thickness (mm), and the width (mm) of the samples (mm). Flexural strength shows the amount of force or stress a material can withstand before it breaks. The relation between tensile strength and flexural strength depends on the homogeneity of the material. An increase in inhomogeneity of the composite will increase the flexural strength and lower the tensile strength, and vice versa. For the hybrid composites prepared in this study, the surface inhomogeneity was lesser than the bulk inhomogeneity. Because of this reason, for all samples containing alumina, the flexural strength was much higher than the tensile strength.

The flexural strength of the prepared hybrid laminates was calculated using Eq. 2, for varying wt% of nano-α-Al_2O_3 for both 60 nm and 200 nm particle sizes. It was observed that the flexural strengths for 60 nm laminate showed an increasing trend with the increase % addition of nano-α-Al_2O_3 (from 252.69 to 378.39 N/m^2). Though the tensile strength values are not convincing for 60nm, the flexural strength values showed that the particles were homogeneously spread and a lesser value in tensile strength was only due to more agglomeration of nanoparticle at 0.5 wt%. The increasing trend was due to a higher amount of homogeneous materials in the system, making, making the composite adhere very strongly and resulting in higher flexural strength. The addition of nanofiller with a high surface area increased the polymer/nanofiller interface area and the tensile strength. This increased interfacial strength reduced the stress concentration at the interface and enhanced the load transfer efficiency effectively, which was observed in the case of 60nm laminates with increasing

wt% of nano alumina.

In the case of 200 nm nano-α-Al_2O_3 hybrid laminates, flexural strength showed a decreasing trend from 252.69 to 193.31 N/m^2, as shown in Fig. 4. With the increasing particle size of the nanomaterial, the possibility of more aggregation may induce imperfection at the surface and the bulk material, resulting in the flexural strength decrease with increasing wt% of nano alumina. The flexural strength at 1wt% of 200 nm alumina was the lowest as both larger particle size and larger nano alumina amount increased the imperfection and decreased the strength drastically.

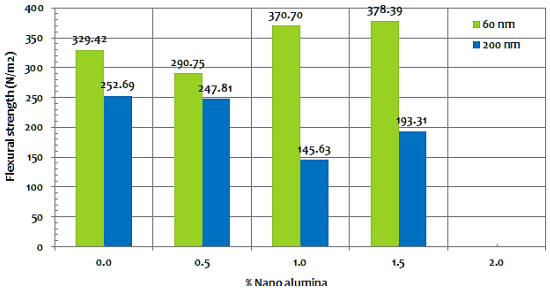

Fig. 4: Flexural strength for nano-α-Al_2O_3 based epoxy-glass fibre hybrid laminates at 60 nm and 200 nm as a function of Wt%

Inter-Laminar Shear Stress

One of the major problems in laminate composites wasdelamination. Delamination is the damage happening at the interface of the composites. Delamination was caused by the high interlaminar stress at the interface. Inter-Laminar Shear Stress (ILSS) measures the in-situ shear strength between the polymer matrix and the nanofiller/fibre. The emergence of delamination induced a significant reduction in the mechanical and thermal properties of the composites. The ILSS was calculated by performing short beam shear (SBS) test (ASTM- D2344/D2344M-00). The dimensions of the sample was 25 x 11 x 5.5 mm with a span length of 22mm.

$$\text{ILSS} = \frac{3P}{4bt} \quad \text{..... (2)}$$

Where, ILSS is interlaminar shear strength (MPa), P is force at fracture (N), b is thickness (mm), and t is width (mm).

The ILSS for the laminate with 0, 0.5, 1.0, and 1.5 wt% addition of nano-α-Al_2O_3 for 60 nm showed a gradual increase from 22.84Ksi for blank to 31.21 Ksi for 0.5 wt% of nano-α-Al_2O_3 addition. The delaminating property decreased with 0.5wt% addition, as shown in Fig. 5.

For 200 nm nano-α-Al_2O_3, the ILSS values for 0.5% slightly increased to 23.94 Ksi from 22.84 Ksi (blank) and zstabilized the laminate against delamination with stress. The strength decreased below the blank when alumina wt% higher than 0.5% was used. The minimum value was obtained for 1wt%, which was 16.03Ksi. From Fig. 6, it was understood that for interlaminar shear strength compared to 200 nm particle size, 60 nm particle laminates withstood the stress against delamination which was supported by the ILSS data.

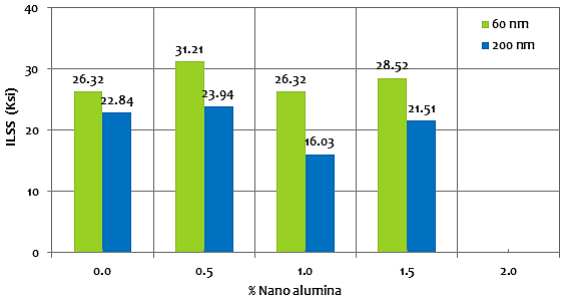

Fig. 5: Interlaminar shear stress for nano-α-Al_2O_3 based epoxy-glass fibre hybrid laminates at 60 nm and 200 nm as a function of Wt%

Vickers hardness

Vickers hardness test was performed by using hardness (Shore D) and according to (ASTM DI-2240) standard at room temperature. Samples were cut into a diameter of 40mm and a thickness of 5mm. The results showed that there was no significant change in hardness for the laminate with the addition of 0.5, 1.0, and 1.5 wt% nano-α-Al_2O_3 for 60 nm (from 86.50 to 86.72) laminates. However, compared to the blank, the hardness of samples prepared by nano-alumina addition were significantly improved from 27.24 to around 86. For 200 nm nano-α-Al_2O_3, the hardness did not improve much compared to the blank from 27.24 to 35.244 for 1.5%. For both particle sizes, the highest value was observed for 1.5 wt% addition of nano-α-Al_2O_3 in 60 nm and 200 nm composites, with hardness values of 86.72 and 35.22, respectively (see Fig. 6). The hardness value over the varying wt% of nanoparticle in 200 nm composite was lower than 60 nm composite. This result may be caused by the poor adhesion of the nanoparticle with the epoxy matrix and the reduced surface area of the nanoparticle.

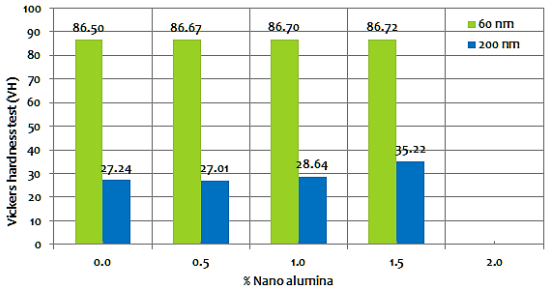

Fig. 6 Hardness for nano-α-Al_2O_3 for sizes 60 nm and 200 nm

CONCLUSIONS

In this work, the fibreglass nanocomposites were prepared and investigated for their mechanical properties as a function of different wt% (0, 0.5, 1, 1.5 wt%) and sizes (60 and 200 nm) of nano-α-Al_2O_3. The mechanical parameters measured were tensile strength, flexural strength, interlaminar shear strength and Vickers hardness.

The effect of the surface area over the binding efficiency of the epoxy showed that the flexural strength, ILSS and hardness was much higher for 60 nm filler than the 200 nm sample as the surface area of 60 nm alumina are higher. The wt% of nano-alumina in the nanofiller also contributes to the mechanical properties. A higher content of alumina led to agglomeration of the nanoparticle, which decreased the tensile strength by lowering the surface area and the adhesion properties.

The highest tensile strength was observed for 0.5 wt% of 200nm nano-α-Al_2O_3, which was 167.80 N/m^2. The highest flexural strength was observed for composite that contains 1.5 wt% of 60nm nano-α-Al_2O_3, which is 378.39 N/m^2. The highest interlaminar shear strength was observed for 0.5 wt% of 60nm nano-α-Al_2O_3, which was 31.21 Ksi and the highest Vickers hardness (86.72) was given by adding 1.5 wt% of 60nm nano-α-Al_2O_3..

For aero applications, addition of 1 wt% nano-α-Al_2O_3 to the composite improved the tensile strength compared to the standard composite. The percentage of increase was 37.82% for 1% nano-α-Al_2O_3 with 200 nm. The flexural strength showed a 42% increase for 1% nano-α-Al_2O_3 compared with standard material.. In the case of ILSS property, the samples that possess nano-α-Al_2O_3 have shown better properties compared to the standard material. All samples showed an increase in tensile strength, but the highest strength was seen in 0.5% nano-α-Al_2O_3 followed by 1% nano-α-Al_2O_3.

In-depth investigation of other reinforcements, such as kevlar, other glass fibres, MNOPs, etc., must be analyzed with the same parameters to investigate the feasibility for aerospace applications.

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NOMENCLATURE

T	:	temperature [k]
t	:	time [s]
ρ	:	density [kg m^{-3}]
μ	:	dynamics viscosity [kg $m^{-1}s^{-1}$]
θ	:	angle [degree]
ILSS	:	interlaminar shear strength [MPa]
P	:	force at fracture [N]
b	:	thickness [mm]
t	:	width [mm]
FS	:	flexural strength [MPa]
L	:	length of the sample [mm]
α	:	crystal phase
%	:	purity [%]
S	:	sp. surface Area, BET [m^2/g]
D	:	density [g/cm^3]

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