Compressive failure analysis of alumina nano particles dispersed short glass/carbon fiber reinforced epoxy hybrid composites

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Abstract – This paper investigates the compressive strength and modulus of the alumina nano particle embedded glass and carbon fiber reinforced epoxy based hybrid composite materials through the compression test. In this analysis, the volume fraction of the alumina nano particles, glass and carbon fibers within the epoxy matrix was varied as 1%, 2%, 3%, 4%, 5%, 10% and 15%. The hybrid composite was made by adding a constant 2wt.% of alumina nano particles within the epoxy matrix along with the fibers. From the experiment, it was found that the strength of the compressive strength and modulus both were increased up to 2wt.% fiber addition. Further improvement in the properties was observed in case of hybrid composites. From this analysis, it was found that the improvement of the properties in the composite was restricted to the 2wt.% filler addition and the properties decreases there after.

Index Terms — Thermoset polymer composites, Alumina particles, Glass/Carbon fiber, Compressive strength, Scanning electron microscopy, Young's modulus

1. INTRODUCTION

Thermoset polymer composites are important class of engineering materials used in modern day engineering applications. Thermosetting resin like epoxy is widely used in various engineering applications starting from automobile industries to the aerospace application. Particles and fiber loaded epoxy composites shows excellent properties like low shrinkage, best adhesion, good chemical resistance and mechanical properties.

Many researchers tried to improve the properties of the virgin polymer by introducing different types of reinforcing elements like nano particles, fibers and their combinations. The mechanical properties of the fiber reinforced composite materials were improved due to the addition of nano filler in the polymeric matrix.

Researchers also reported that the improvements to the properties were observed at the lower volume fraction of filler contents. Improvement of one mechanical property associated with the decrease of the other related properties of the composites. The incorporation of the rubber particles within the epoxy matrix improves the fracture toughness [1] whereas, the substantial decrease was observed for the modulus and strength of the composite materials. On the other hand, the incorporation of fibers within the polymer matrix improves the modulus and strength of the composites. The properties like inter laminar shear strength improved due to the SiC whiskers in the polymer matrix [2]. Due to the low-cost and better chemical resistance and ability to make bond to the epoxy matrix, the alumina particles are drawing significant attention for nano-scale polymer reinforcement. The miss alignment of the fibers within the matrix causes the shear instability in compressive mode of failure, and the compressive strength drastically reduced [3], [4]. Stephen et al. has reported that the compression loading for fiber reinforced composite leads to three-dimensional states of stress, and the compression strength improved under pressure [5]. Influence of fibre volume fraction on the mechanical properties of composites under compressive load was studied by many researchers [6], [7], [8]. The experimental and numerical findings suggest that compressive failure of high modulus carbon fibre/epoxy composites are mainly controlled by the compressive response to the carbon fibres themselves [9]. Sharma et al. has reported that the modification of the interphase properties of carbon fiber by growing the carbon nano tube over the fiber surface had enhanced the compressive strengths of the longitudinal and transverse compressive strengths respectively as compared to composites made of carbon fibers without carbon nanotubes growth [10]. The particle impregnated fiber-reinforced composites shows the improvement in flexural and compressive strength at higher temperatures. This may be due to the additional supports provided by the nano particles to the fiber against kinking [11]. Incorporation of nano tubes at the vicinity of the

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fiber's bridges the inter fiber debonding and micro cracks thus the global failure due to the compressive loads gets delayed [12]. The microscopic observation by Srivastava et al. [13] has conformed that the shear mode of failure of fibers is prominent in compressive loading, which then lead to matrix crack and complete failure of the composite. Uddin et al. [14] has reported that the silica nano particle loading improves the compressive strength and modulus of the epoxy composite. The compressive strength of CFRP increases as well as the deflection decreases due to addition of CNT [15]. The composites exhibited higher elastic moduli and yield strengths than the neat epoxy samples. The nano Al- reinforced composite exhibited a higher elastic modulus and static and dynamic compressive strengths than epoxy and micro Al-containing composite due to increased cross-link density and nano Al-containing composite exhibited significant strain hardening effect [16]. The dispersion of CNF into the epoxy improves mechanical properties of the composites, and the CFRP laminates with CNF dispersed resin exhibit higher compressive strength than CFRP laminates [17]. Similar to nano particles, the fullerenes also improves the mechanical properties of the composite. The compressive strengths of (0)8, and 16 ply laminates are improved by incorporating a small amount of fullerenes [18]. The modulus increased with nanoparticle concentration. The increase in modulus is expected because the modulus of Al_2O_3, E = 360GPa [19] is much greater than that of the epoxy matrix (3.2GPa) [20]. Subramaniyan et al. has reported that the elastic modulus and compressive strength and modulus of the resin increased through the increase with the addition of nano clay and seizes after certain volume fraction of loading [21]. The similar kind of results was also reported by some other researchers [22]. The carbon nano tubes also play an important role to improve the mechanical properties to the polymer composite. In the contrary, some researchers also reported that the compressive strength and modulus of the composite materials decrease in the incorporation of fibers in the epoxy resin but these results were confined to the unidirectional fibers only [23].

The main objective of this paper is to study the influence of the effect of alumina nano dispersion in the fiber/epoxy composite on the compressive strength to the bulk material. Furthermore, the compressive strength of hybrid composite prepared by adding a constant 2wt.% of alumina particles to the fiber-reinforced composites was examined for the cylindrical test coupons. Thus in this paper, the compressive test was performed to determine the strength and modulus of alumina particles and randomly distributed short glass/carbon fiber filled epoxy composite for the optimal loading of the fillers in the matrix.

The compressive strength of the test samples was calculated by using the equation (1):

$$\sigma = \frac{P}{A} \tag{1}$$

Where, σ is the failure stress, P is the ultimate braking strength and A is the area of cross section normal to the direction of the applied compressive load.

The modulus of the samples was calculated by taking the ratio of compressive stress to compressive strain in the linear region of the stress-strain curve between strain values of 0.05% and 0.25%. Compressive modulus is calculated using the equation (2):

$$Y_c = \frac{\sigma_2 - \sigma_1}{\varepsilon_2 - \varepsilon_1} \tag{2}$$

Where, ε_1 is a strain of 0.0005, ε_2 is a strain of 0.0025 and σ_1 is the stress at ε_1, and σ_2 is the stress at ε_2.

2. EXPERIMENTAL

2.1. Materials used

The polymeric matrix used during the fabrication of the composite was the blend of unmodified solvent free epoxy resin, specific gravity of 1.14, under the trade name of Bondtite PL-411 and the amine based hardener of specific gravity 0.98 of grade PH-861 supplied by Resinova Chemie Ltd. India. The composition of the base matrix formulation was the mixture of 10 – 12 parts by weight of the hardener with the epoxy which provides the pot life of 30 minutes at 40°C room temperature. Three types of reinforcing elements were used for the preparation of the composite materials (Table 1).

TABLE 1
MECHANICAL PROPERTIES AND DENSITY OF CONSTITUENTS OF THE COMPOSITE MATERIALS.

Constituents	Young's Modulus	Poisson's Ratio	Density (gm/cc)
Epoxy	3416 MPa	0.33	1.2
Alumina	347 GPa	0.22	3.95
Glass Fiber	60 GPa	0.25	2.5
Carbon Fiber	242 GPa	0.33	1.81

The first category of the reinforcing element used was the Alumina particle procured from the SASOL Germany. The second category was the PAN based carbon fibers supplied by the Zoltek-USA, and the last variety is the commercial-grade E-glass fiber supplied by M/s Harsh Deep Industries, India. The hybrid composite was prepared by adding 2%wt. of alumina particles to the glass fiber and carbon-fiber composite in which the % weight fraction varies as 1%, 2%, 3%, 4%, 5%, 10% and 15% (Figure 1).

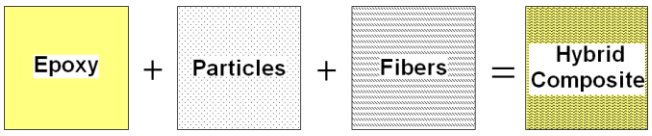

Fig. 1. Schematic illustration of the particles and fibre reinforced hybrid composite

2.2. Specimen preparation and post curing operation

The ASTM D 695-10 standard dimension was followed during the preparation of the test specimen. The stoichiometric amount of the hardener was added to the epoxy resin under the continuous mechanical stirring operation for 5 minutes to ensure thorough mixing of the hardener in the network of polymer. As soon as the exothermic reactions' stats to occur, the viscosity of the mixture reduce, this creates an ideal environment to add the reinforcing elements. Now the as received alumina particles of size less than 90 micron, short glass and carbon fibers of length 1mm to 7mm was added in phase to the mixture. Furthermore, 2 wt.% alumina particles were added individually into the mixture of glass/carbon-fiber epoxy based solution thus forming a new type of hybrid composites. The mixture was poured into the firmly clamped dual piece symmetrical metallic mould containing a cylindrical pocket of required dimension, lubricated with silicon oil and the care was taken to nullify the chance of entrapped air particles during the pouring process (Figure 2).

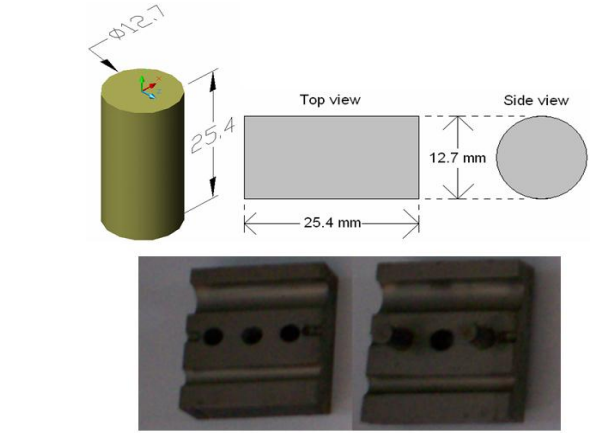

Fig. 2. Geometric representation of Compression test specimen and metallic mould used for the fabrication purpose

The mould was kept at room temperature for 24 hours. The post curing operation was carried out for the composite materials by keeping the test coupons inside the vacuum furnace. Figure 3 shows the post curing operation of all types of the composites followed by the slow cooling in a controlled atmosphere.

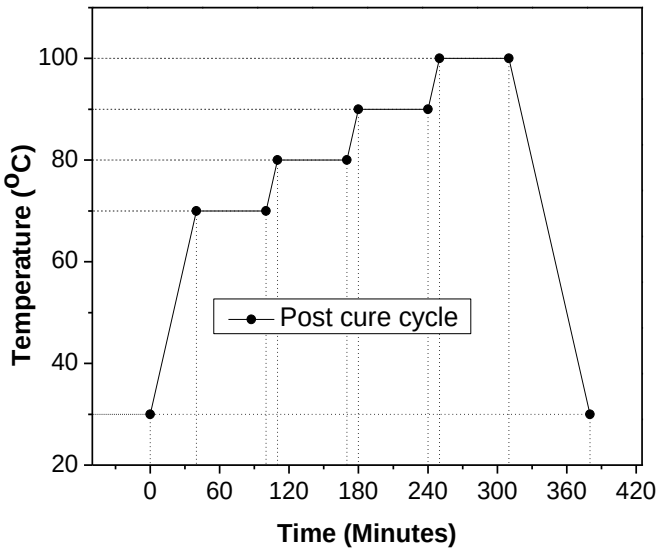

Fig. 3. Post-cure cycle

2.3. Compression Test

The fully cured test specimens were tested at room temperature and atmospheric condition in the ZWICK/Roell Z010 universal testing machine. The cross head speed of 1 mm/min. was maintained throughout the test. The average value of similar kind of five test results was taken in the analysis. Figure 4 shows the brittle failure of the composites.

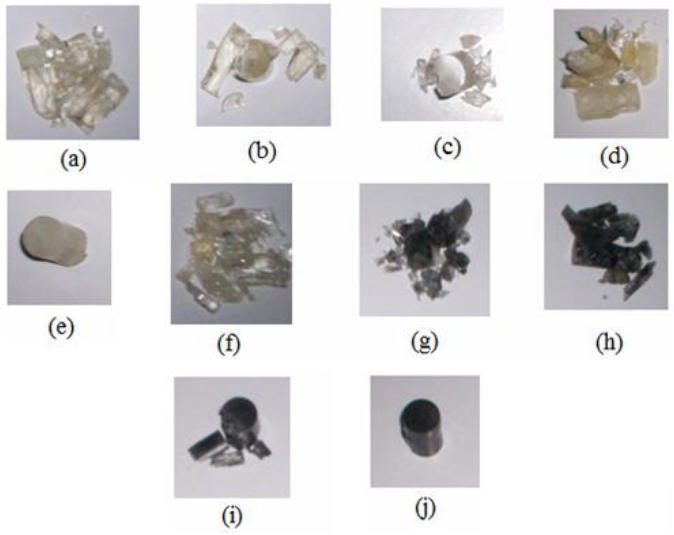

Fig. 4. Fractured test specimens made out of (a) neat epoxy and epoxy composite containing (b) 1 wt.% alumina nano particles (c) 2 wt.% alumina nano particles (d) 1 wt.% alumina nano particles and 2 wt.% short glass fibre (e) 2 wt.% alumina nano particles and 2 wt.% short glass fibre (f) 10 wt.% alumina nano particles (g) 1 wt.% short carbon fibre (h) 2 wt.% short carbon fibre (i) 1 wt.% alumina nano particles and 2 wt.% short carbon fibre (j) 2 wt.% alumina nano particles and 2 wt.% short carbon fibre.

2.4. Scanning electron microscopy

The surface morphology was studied by scanning electron microscopy (Philips XL-20). The structural deformation of the composite samples was avoided by submerging the samples in the liquid air and the samples were (Al_2O_3-reinforced composites) sputter-coated with gold for better electrical conductivity. The voltage of 10kV was maintained during the SEM operation. Figure 5 shows the cluster of alumina nano particles correspond to the 15wt.% filler addition.

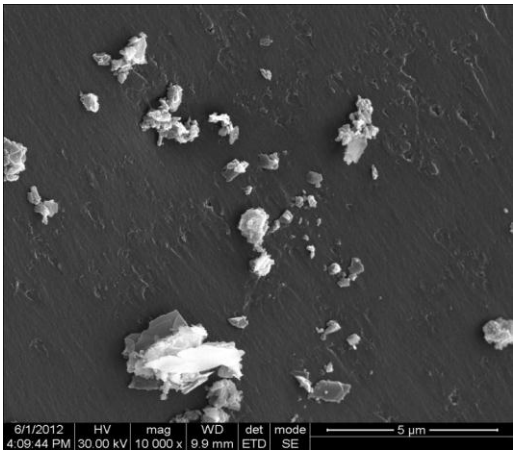

Fig. 5. SEM image of clustered alumina nano particles in epoxy resin

2.5 FTIR analysis

The FTIR spectra was used to study the molecular structural changes of the epoxy resin due to the addition of 1, 3 and 5wt.% of alumina nano particles. From the Figure 6, it was found that the C–H band stretching of SP_2 around 3055cm^{-1}, and the C–H band stretching of SP_3 around 2950cm^{-1}, 2902cm^{-1} and 2873cm^{-1}. Furthermore, aromatic C=C band stretching can be seen at 1608–1456cm^{-1}, C–O band stretching around 1290cm^{-1}, and the existence of epoxide groups around 900cm^{-1}.

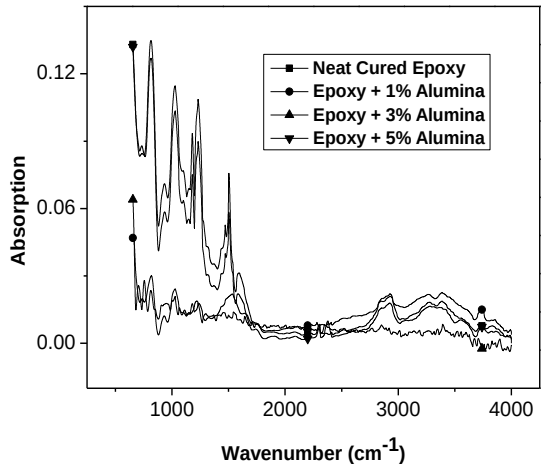

Fig. 6. FTIR spectrum of neat epoxy and alumina particles reinforced composites

After the addition of alumina nano particles to the epoxy, the epoxide group can be seen around 850cm^{-1} and aromatic C=C band stretching at 1456cm^{-1}, which were observed in epoxy, as well as the band stretching of –CN bonding around 2237cm^{-1} and that of C=O bonding around 1623cm^{-1}, which were also observed in composite [24]. The methyl groups (–CH3) of bisphenol A are characteristic of bending vibration at 1382.8cm^{-1} and the absorption band at 1450cm^{-1} indicate the presence of methylene (–CH2–) moieties of epichlorohydrine. The peak at 820cm^{-1} corresponds to the stretching vibration Al-O in tetrahedral coordination. The peaks at 660cm^{-1} correspond to the stretching vibration of Al-O in octahedral coordination [25]. The appearance of stretching vibration band at 3400cm^{-1} is indicative of the presence of hydroxyl groups, which is correlated to the interaction between the amine as curing agent and hydroxyl group of epichlorohydrine. The band at 1249.8 and 1033.7cm^{-1} are ascribed to alkyl aryl ether symmetric stretching and dialkyl ether stretching vibration, respectively. First one shows an interaction between bisphenol-A and epichlorohydrine and the other is attributed to the cross-linking reaction. The band at 1182.2cm^{-1} (C–N stretching) proves the reaction of secondary amine (piperidine) with the epoxy group [26]. It is important to observe that the picks correspond to the wave numbers are prominent for 3% and 5% over 1% alumina in epoxy.

3. Results and Discussion

Figure 7 shows the compressive strength of the particles, and fiber reinforced composite materials. The failure strength of the composites' increases with the incorporation of the reinforcing elements within the matrix of epoxy correspond to a 2 to 3wt.% of filler addition. It was found that, the compressive strength of the alumina particles reinforced composite was increased by 90% in comparison with the neat epoxy with the addition of 2wt.% of alumina particles and there after the strength gradually decreases.

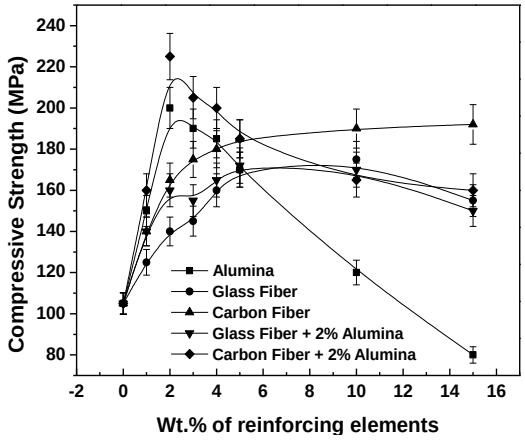

Fig. 7. Effect of filler addition upon the compressive strength of composite

At 15wt.% of alumina particle addition, the strength of the composites' drops by 23%. This drop in strength is due to the agglomeration of the nano particles and formation of air

pockets (Figure 5) which may be due to their higher surface energy to adhere among themselves.
Influence of the 2wt.% reinforcing elements on the modulus of the composite is shown in the figure 8.

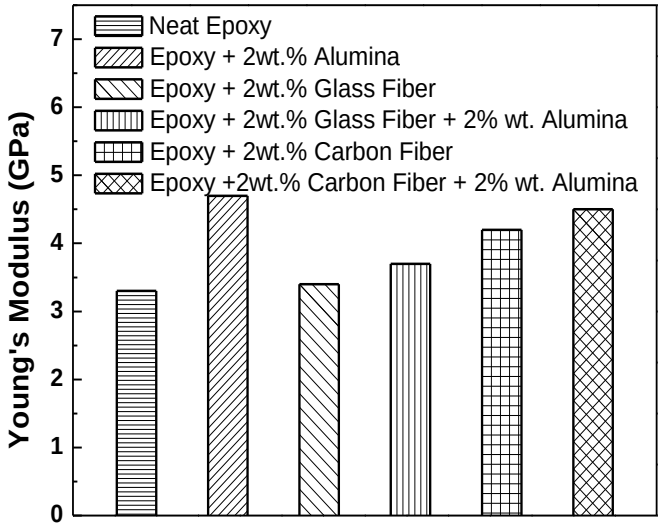

Fig. 8. Variation of Young's modulus for the particles and fibre reinforced composites

The modulus of the composite was increased by 42% due to the incorporation of nano sized alumina particles within the epoxy matrix. There was a marginal improvement 3% of the modulus for the addition of 2wt.% of glass fiber into the epoxy matrix was recorded. Further addition of 2wt.% alumina particles to the glass-fiber composite, the result was quite interesting and the compressive strength was enhanced by 12% as compared to the neat epoxy. The modulus of the composite was improved over the epoxy by 27% due to the addition of short carbon fiber.
The modulus of the 2% volume fraction of carbon fiber reinforced composite was further improved up to 36% due to the incorporation of 2% volume alumina particles along with 2% carbon fiber within the matrix of epoxy. This result thus proves that the compressive strength to the glass/carbon fiber reinforced composite was improved due to the addition of alumina particles within the epoxy matrix. The SEM picture (Figure 9) shows the presence of fiber and alumina particles in the matrix.
The rule of mixture equation (3) was applied to the randomly distributed particle/fiber loaded composite materials;

$$E_{Composite} = K \times \left(E_m \times V_m + E_f \times V_f \right) \quad (3)$$

Where, $E_{Composite}$ = Modulus of the composite, E_f, E_m are the modulus of the fiber and matrix materials; V_f, V_m are the volume fraction of fiber and matrix.

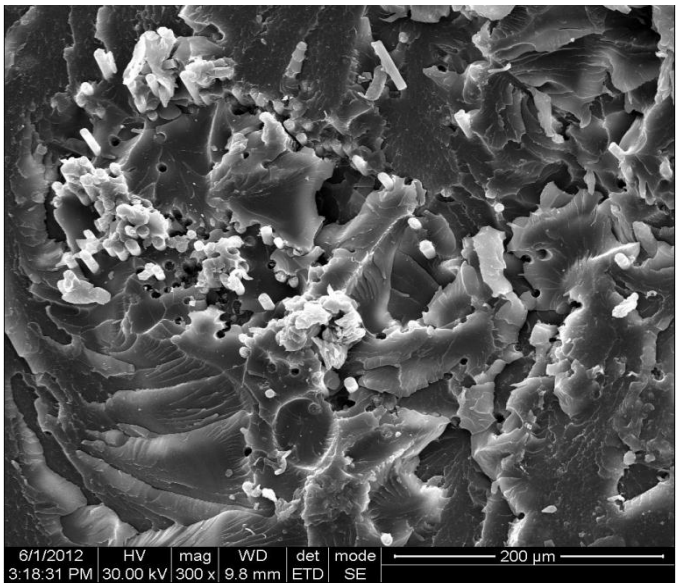

Fig. 9. SEM image of fiber/particle reinforced composites

K is the constant called as fibre efficiency parameter. Figure 10 shows that the values of "*K*" have the close proximity for the alumina particle reinforced composites. This value is in higher side for the glass fiber and intermediate value for the carbon fiber reinforced composite materials.

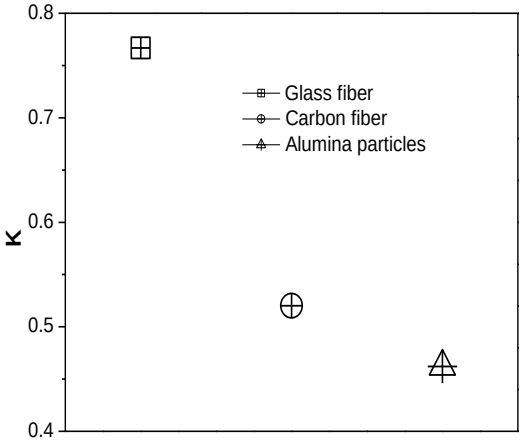

Fig. 10. Variation of fibre efficiency parameter for the fibre reinforced composites

3.1 Post analysis of the fractured test specimens

Each type of the composite materials reacts differently to the externally applied load, and the failure pattern is unique to the same class of composites. Post analysis of the tested compression test specimens undisclosed the secrecy of the stress absorbing mechanism of different grade of composite materials. The common fracture mode of the brittle materials is along the plane which makes an angle other than parallel and orthogonal directions to the direction of the applied load. But the failure of the neat epoxy generally doesn't show such types of fracture's ratter

the failure is the complete collapse of the test coupons forming many segmental parts of the single unit original member (Figure 4). The number of the fragmented pieces gradually decreases with the addition of the filler contents. At an optimum alumina filler content of 2wt.% the fragmented pieces colleted after the test is least in numbers because the failure occurs at the critical plane. From the results, it may be concluded that the composite materials offer maximum resistance to the brittle fracture at 2wt.% addition of alumina particles. The fractured test specimen collected from the compression test shows fewer numbers of fragmentations of the test coupons as it was compared with the failure of the neat epoxy. This proves that, the energy absorption is more for the carbon fiber reinforced composite. Similar pattern also observed for the glass fiber reinforced composite materials. It is interesting to report here that, with the incorporation of 2wt.% nano sized alumina particles along with 2wt.% glass/carbon-fiber fractures without forming segmental pieces as the end products (Figure 9). This proves that, the intermolecular bond strength increases and the crack arresting mechanism comes in order to play by just adding with the addition of 2wt.% alumina particles into the fiber-reinforced polymer matrix.

4. Conclusions

The compressive strength and modulus of the fiber/particle reinforced epoxy composites were measured and the influence of the %wt. fraction and the combined effect of the glass/carbon fibers and alumina particles on the compressive strength of the composites were studied. The compressive strength was enhanced due to the short glass/carbon fiber, and nano sized alumina particle reinforcement. The carbon fiber reinforced to epoxy composite shows better results than the glass fiber reinforced epoxy composite. The compressive strength of the glass fiber reinforced composite material was further improved due to the incorporation of 2% alumina particles within the polymer matrix but the compressive strength of 2wt.% of the glass/alumina hybrid composite was found to be less than the 2wt.% carbon fiber reinforced composite material. It was important to note that the addition of 2wt.% nano sized alumina particles to the glass/carbon fiber reinforced composites results in a remarkable strengthening effect up to 5% of the bulk fiber contents. After this, the strength of the hybrid composites decreases gradually and the value falls below to their fiber reinforced composite.

In general, the nano sized alumina particles reinforced composite materials surpass the performance of the commercially available randomly distributed short glass/carbon fiber reinforced composite materials at lower cost. The alumina particles reinforced glass/carbon fiber composite may be well suited for the under roof structural applications. However an extensive study on the performance of this type of hybrid composite under adverse condition may be an interesting topic of the future research.

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