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Investigation of Mechanical and Electrical Properties of Carbon Filler Reinforced Epoxy Composites

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Abstract

Polymer composite components are embedded with various filler materials. The selection of fillers are important for enhancing the required properties of composites. Filler materials are used in polymers to increase heat resistance, increase mechanical properties, modify electrical properties and improve dimensional stability. The objective of the study is to judicially select a carbon-based filler material which offers balanced mechanical and electrical output characteristics on polymer composites. In order to get a clear understanding of the relation between mechanical properties and the electrical conductivity of filler reinforced polymer composites, the specimens were subjected to scanning electron microscopic, tensile and four-point probe testing methods. This study revealed noticeable dependencies on the electrical conductivity and mechanical properties on the carbon filler content. Reinforcement of conductive fillers on epoxy matrix improves the electrical conductivity from $8x10^{-13}$ S/m to $7x10^{-2}$ S/m. When the epoxy matrix is reinforced with Milled Carbon Fibre (MCF), only 4.12% reduction in tensile strength has been observed at 8 wt% of filler loading.

Keywords: *Carbon fillers; Conductivity; Dispersion; Elongation; Epoxy*

Introduction

Use of polymer composites are rapidly increasing in engineering, infrastructure, aerospace, automotive, marine and electronic applications. High strength to weight ratio, relatively good durability, ease of preparation and handling, low density and corrosion resistance are the preferable properties of epoxy polymer composites. The unique characteristics of epoxy polymer composite materials make them as an attractive alternative to traditional materials in various applications. Tensile properties of epoxy composites are improved by reinforcing with lesser weight % of carbon-based fillers (Reza Taherian., 2014; Lohar et al., 2018; Ni et al., 2015). Polymer composites are dielectric materials. Insulating material like polymer is converted into an electrically conductive material by developing a linked conductive network inside of them (Fang et al., 2020). Carbon based conductive filler reinforcements in polymers are mostly used to improve electrical conductivity. Derivatives of carbon, such as carbon black, graphite, carbon nanotubes, and chopped carbon fibres are used as the filler materials in polymer composites. High conductivity, low density, high aspect ratio, low resistance and easy production are the promising characters of carbon-based fillers. Polymer/carbon filler composites exhibits high improvement in electrical conductivity (Moriche et al., 2016).

The main drawbacks of polymer composites are that they are sensitive to vulnerable environmental influences of extreme temperatures, fluctuating hydrothermal conditions and high electric current (Hirano et al., 2010). Polymer composites are unable to withstand at these conditions and sufficient protection methods are required to prevent them from destruction due to environmental conditions (Wang et al., 2016). Merging of filler materials with polymers will

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result in a range of materials (Yokoyama., 2013). Performance gaps on polymeric composites are bridged by the proper selection of filler materials. Fillers can be assimilated with any type of polymers. The electrical, mechanical characteristics and the dimensional stability of polymer composites highly depend on the type and amount of filler materials used (Majeed et al., 2014). Proper dispersion of filler materials with matrix medium enables good packing density and thus improved conductive network (Planes et al., 2012). The use of different carbon-based fillers would be a good way to get balanced mechanical and electrical properties in polymer composites (Szeluga et al., 2015; Hallad et al., 2017). Mixing of conductive fillers with polymers leads to increase in conductivity, increase in Young's modulus and increase in tensile strength of the material. Insertion of conductive fillers reduces the elongation at break and toughness of the material (Joshi et al., 2020; Nayak et al., 2020). High filler contents and better ordering structure of conductive fibers inside the thermoplastic results in high electrical conductivity (Zakaria et al., 2015). For critical filler content levels named percolation threshold, the electrical conductivity of polymers increases sharply of several orders of magnitude (Planes et al., 2012).

All the above said experimental works exhibit an inversely proportional relation between the mechanical and electrical properties of manufactured composites. From the literature, it can be noticed that the combined effect of the electrical and mechanical properties of polymer epoxy composites reinforced with different types of particulate carbon fillers was seldom investigated by the researchers. At the same time, it can be found that only a few researchers have conducted experiments to study the influence of the conductive carbon fillers and their effects on output characteristics of epoxy composites. This study aims to investigate the combined effects of mechanical and electrical properties on the polymer composites. Carbon fillers with different morphological characteristics are being used as reinforcements. In this experimental study, mechanical and electrical characteristics of CB/epoxy, NGF/epoxy, MCF/epoxy composites were investigated at varying weight percentage of conductive fillers. The results show the impact of three different carbon filler materials on epoxy composites under a set of processing parameters.

Materials & Method

In this study, Carbon Black (CB), Natural Graphite Flakes (NGF) and Milled Carbon Fibre (MCF) were used as filler materials and the matrix part is epoxy resin. The specifications of materials used in this experiment are given in Table No 1.

Mixing parameters play a major role in increasing the performance of filler reinforced epoxy polymer composites [18]. At first stage, the epoxy resin is heated to 70°C to reduce the viscosity. Reduction in viscosity ensures the easy dispersion of filler particles. The output characteristics of polymers highly depend on the dispersion state of fillers [19]. The conductive carbon fillers were heated for 50 minutes at 110°C and degassed under vacuum.

The epoxy resin and the curing agent were mixed at 10:1 ratio using a high speed mechanical mixer model RM 20- KIKA-WERK for 40 seconds at a rotating speed of 1200 rpm. The graphical process flow chart in Figure 1 shows the intermediate steps in making filler reinforced polymer composites. Uniform mixing of filler and resin is ensured by this melt compounding method [20, 21]. The mixture of CB/epoxy or NGF/epoxy or MCF/epoxy were poured into open glass moulds and allowed to cure at room temperature.

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Table 1. Specifications of materials used

Phase	Material	Particle size	Type
Matrix (10 : 1 by weight)	Epoxy resin	--------	LY556 (1200 kg/m^3)
	Hardener	--------	HY951
Filler	Carbon Black (CB)	50 to 75 microns	VXC72R
	Natural Graphite Flakes (NGF)	50 to 75 microns	--------
	Milled Carbon Fibre (MCF)	50 to 100 microns	FP-MCF-004
Degreaser	Acetone	--------	--------

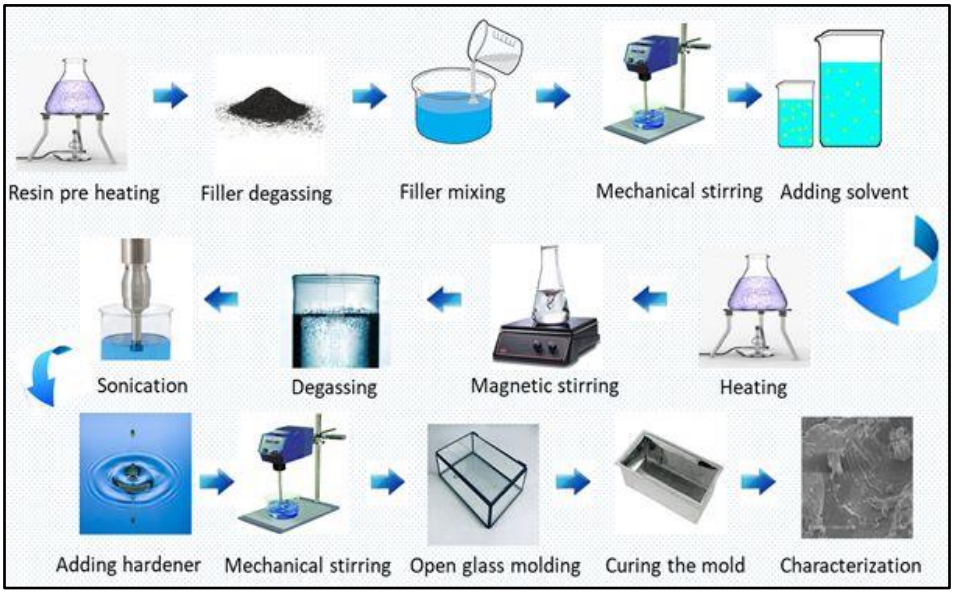

Fig. 1. Graphical process flow chart of making filler reinforced polymer composites

Three different batches of conductive filler epoxy composites were prepared by varying the weight percentage of CB/NGF/MCF fillers. While mixing the conductive fillers with epoxy, whenever the total filler weight percentage exceeds 12, agglomerations started to form. To prevent the formation of clusters and agglomerations, the filler total weight

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percentage is limited up to 12 percentage only. The sample ID and their respecive proportions of epoxy - filler material combinations are listed in Table No 2.

Table 2. Sample ID based on weight percentages of epoxy and conductive fillers

Epoxy (wt%)		100	98	96	94	92	90	88
Filler (wt%)		0	2	4	6	8	10	12
Sample ID	CB/epoxy	Epoxy	CB2	CB4	CB6	CB8	CB10	CB12
	NGF/epoxy	Epoxy	NGF2	NGF4	NGF6	NGF8	NGF10	NGF12
	MCF/epoxy	Epoxy	MCF2	MCF4	MCF6	MCF8	MCF10	MCF12

Testing of Composites

Microscopic Analysis

Specimens were subjected to scanning electron microscope analysis (Zeiss MA15/EVO) to examine the conductive filler distribution and void contents. CB/NGF/MCF fillers were uniformally distributed as shown in Figure 2. Figure 2.(a) represents the distribution of carbon black particle cluster within the matrix, Figure 2.(b) shows the presence of milled carbon fibre in the matrix and Figure 2.(c) shows the presence of graphite flakes in the matrix.

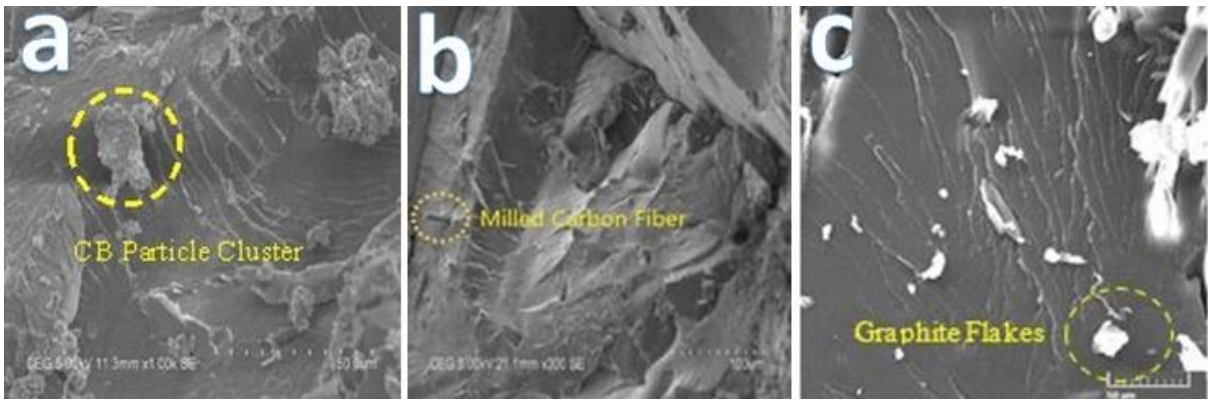

Fig. 2. SEM images of cross section of a) CB/epoxy, b) MCF epoxy, c) NGF epoxy composites

Mechanical testing

The tensile properties and elongation percentage of the CB/NGF/MCF/epoxy composites were examined by tensile testing. Tests were undertaken in ASEW 5 ton universal testing machine supported with digital encoder and a crosshead speed of 1.25 mm/min. The

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samples were cut as per ASTM D 638 [22] standards. Figure 3 shows the tensile testing machine setup and tensile test samples with 3 mm thickness. Three samples tested from each combination of composites for conforming the reproducibility.

DC Electrical conductivity testing

Electrical conductivity of carbon filler reinforced epoxy polymer composite specimens was tested by using four-probe technique under ASTM D 257 standard. Figure 4.(a) depicts the arrangement of electrodes in four point probe testing method. Samples are cut into 30 mm x 30 mm squares with 3 mm thickness as shown in Figure 4.(b). Figure 4.(c) graphically represents the conductivity values for the transformation from insulators to conductors of electric current.

Silver coated copper electrodes used to get sufficient electrical contact with specimens. Four samples from each conductive filler concentration were tested to get the mean value. Conductivity is measured by connecting the measuring instrument with Keithley 6517A electrometer.

Results and Discussion

Mechanical properties

The tensile properties of pure epoxy (zero % conductive filler) and of carbon filler/epoxy composites containing 2, 4, 6, 8, 10 and 12 weight % CB, NGF, MCF are shown in Figure 5.

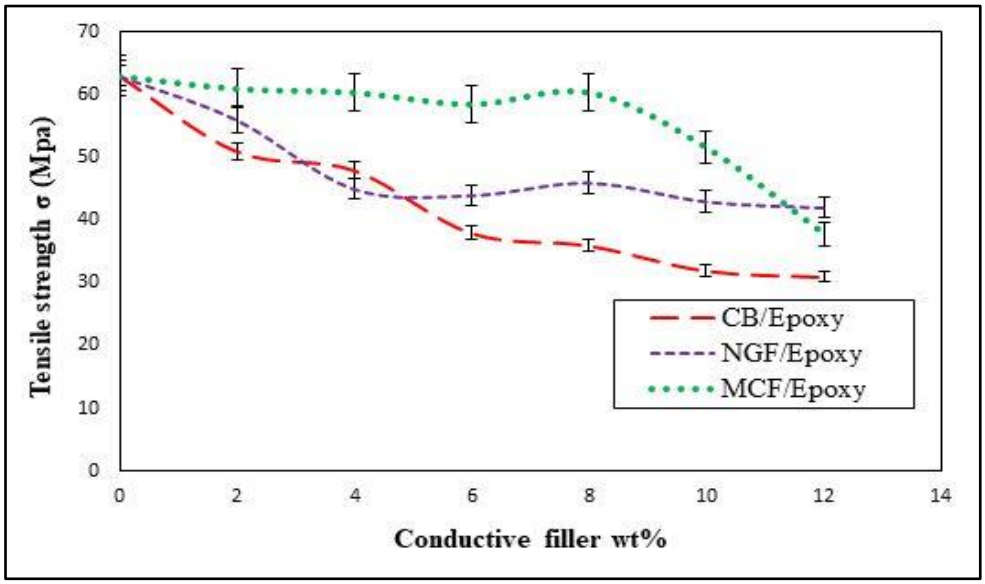

Fig. 5. Tensile strength of filler reinforced epoxy composites

For MCF/epoxy composites between 2 and 8 wt% of MCF reinforcement the tensile strength (σ) value is nearly same around 60 Mpa. Reinforcement of 10 wt% and 12 wt% of MCF results drastic decrease in tensile strength reaching a minimum value of 37.8 Mpa. For NGF/epoxy composites between 4 and 8 wt% of NGF reinforcement the σ value is nearly same around 45 Mpa. The existence of thin graphene layer in NGF and weak Van der Waals bond between the layers [23] and affects the tensile strength of NGF/epoxy composites [24]. When the CB content in epoxy exceeds 6 wt%, the CB particles starts to rejoin and they forms aggregates. It reduces the bonding between CB and epoxy molecules. Incorporation of CB content in epoxy

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matrix reduces the tensile strength of CB/epoxy composites at all wt% of filler concentrations. Tensile strength (σ) of carbon filler reinforced epoxy composites decreases with increasing carbon based filler contents [25]. Figure 5 reveals that tensile strength of all the tested samples decreases with high amount of carbon filler concentrations.

Figure 6 shows the variation in elastic modulus for different filler reinforcements. For NGF/epoxy and MCF/epoxy composites, increment in elastic modulus (E) values is observed proportionally to the increasing wt% of filler content.

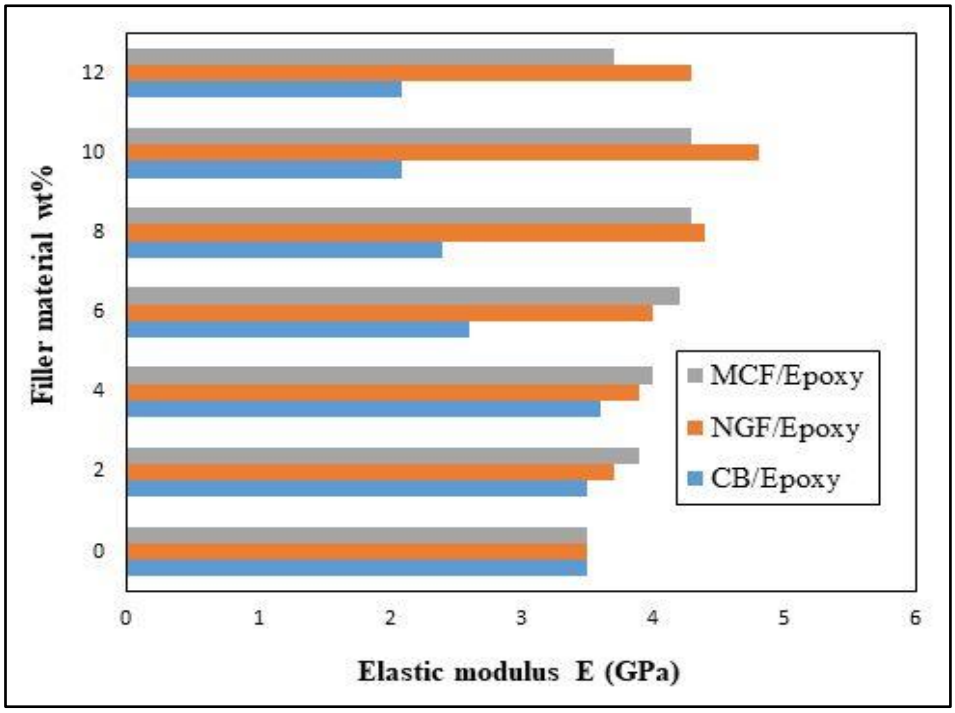

Fig. 6. Elastic modulus of filler /epoxy composites

The magnitude E is high at 10 wt% of filler content for above said two batches of composites. When the filler loading is 12 wt% the E value reduces 10.4% and 14.9% from their maximum values for NGF/epoxy and MCF/epoxy composites respectively. This represents further increase in filler loading will affect the bonding between long chain molecular structures on the epoxy groups. Elastic modulus (E) of NGF/epoxy, MCF/epoxy composites increase with increasing carbon filler contents. From 6 wt% onwards, the CB particles get inserted with the reactive functional group of epoxy and it leads to decrease in E values. The variation in Elastic modulus (E) values of composites with CB concentration deviates from the trend. The chemical filler polymer bond between CB/epoxy composites produces a softening effect and tends to breaking of the composites with low tensile properties [26]. Weaker adhesion of filler particles with epoxy resin, micro and macro pores and agglomerations at higher wt% of fillers on epoxy results the detrimental performance.

The mechanical energy absorption characteristics of epoxy composites were strongly influenced by the amount of filler content on it. The change in elongation properties of epoxy composites with varying filler concentration are shown in Figure 7.

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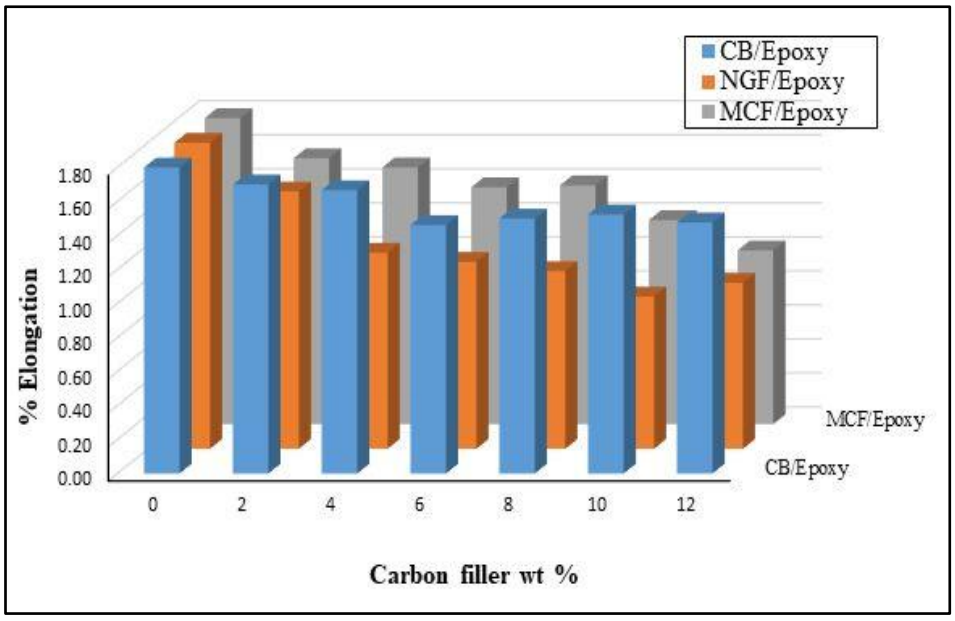

Fig. 7. Percentage elongation of carbon filler/epoxy composites

The percentage elongation is less for all types of filler/epoxy composites with higher wt% of filler content [25]. From figure 7, it can be seen that the percentage elongation of pure epoxy (0 wt% filler content) is 1.8% and percentage elongation of composites reinforced with 12 wt% of CB, NGF, and MCF filler content are 1.48%, 0.98% and 1.02% respectively. This represents restrained neighborhood epoxide groups at higher filler concentrations under the application of tensile loading. All the tested composites with higher filler content possess brittle characteristics [27].

Figure 8 represents the SEM images illustrating tensile fracture surface morphology of the prepared composites. For SEM analysis samples with highest tensile strength values (2 wt% of CB, MCF and NGF fillers) and samples with the lowest tensile strength values (2 wt% of CB, MCF and NGF fillers) were selected. The composites with MCF fillers possessed better tensile strength at composition compared to composites with CB and NGF fillers. Figure 8a) represents the presence of well bonded CB filler particles which is attributed to the better strength. Figure 8b) shows well bonded MCF material to matrix. The MCF material imparts better load bearing capacity in the tensile direction and the better bonding improved the strength further. The failure of the composite was observed to be due to matrix cracking. From Figure 8c) NGF particle cluster was noted and the failure mode was observed to be delamination. For composites with 2 wt% reinforcement fillers the one with MCF filler showed highest strength and CB filler showed lowest strength. Intermediate strength was observed for the composite with NGF filler.

Figure 8d) represents the presence of severe delamination and particle pull out of CB fillers from the matrix. This in turn could be accounted for the degradation of tensile strength. Figure 8e) represents de-bonding of MCF materials from the matrix along with degradation. The increased amount of MCF in the matrix might have resulted in the improper dispersion resulting in the poor filler matrix interface bonding. This might have reduced the tensile strength of the composites with 12 wt % MCF filler. Figure 8f) represents the presence of voids, matrix softening and matrix cracking as a result of increased addition of NGF filler.

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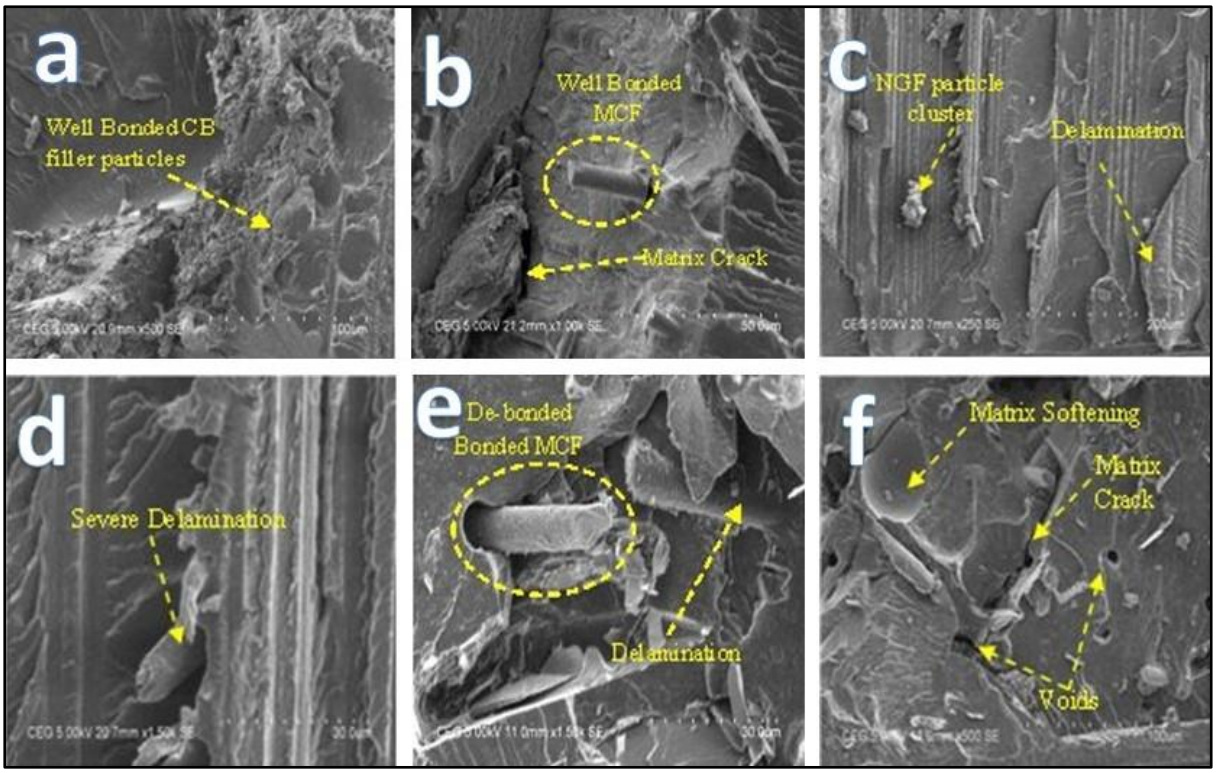

Fig. 8. SEM fracture tensile surface morphology. a) 2wt% CB filler b) 2wt% MCF filler c) 2wt% NGF filler d) 12wt% CB filler e) 12wt% MCF filler f) 12wt% NGF filler

DC Electrical conductivity

The electrical conductive behavior of polymer matrix carbon filler composites are based on the morphological as well as amount of filler material added. Figure 9 shows the volume electrical conductivity of the composites prepared by open glass molding technique at ambient conditions as a function of weight % of different carbon filler content. The homogeneous dispersion of conductive filler with epoxy resin reveals decrease in resistivity [28] and a gradual improvement of conductivity even at low filler concentrations. Composites loaded with Milled Carbon Fiber (MCF) fillers exhibit uniqueness in improved conductivity from 4% of filler content. At increased weight percentage of conductive filler content, remarkable conductive network is formed [29, 30] by reduced inter particle distance or by the physical contact between the conductive filler particles. At critical filler content, percolation threshold formed by tunneling and conductivity increased to several orders.

Natural Graphite Flakes (NGF) reinforced composites reach the percolation threshold at 10 % of filler content. At this region conductivity increases by four orders. Composites reinforced with Carbon Black (CB) reaches the percolation threshold at 8 % of filler content and the conductivity increases by five orders. This value is seven times higher than that of NGF/epoxy composites. The two-dimensional structure of MCF attributes to form a better conductive network even at lower filler concentrations. MCF reinforced composites achieves the percolation value at 6% of MCF content, which is comparatively less wt% than other two. From 4% to 6% of MCF content, the magnitude of volume conductivity increased by five orders. However the conductivity of MCF/epoxy composites are not as good as CB/epoxy composites when the filler content exceeds 8 weight %. Since the dispersion of MCF with epoxy matrix are not uniform at higher filler contents.

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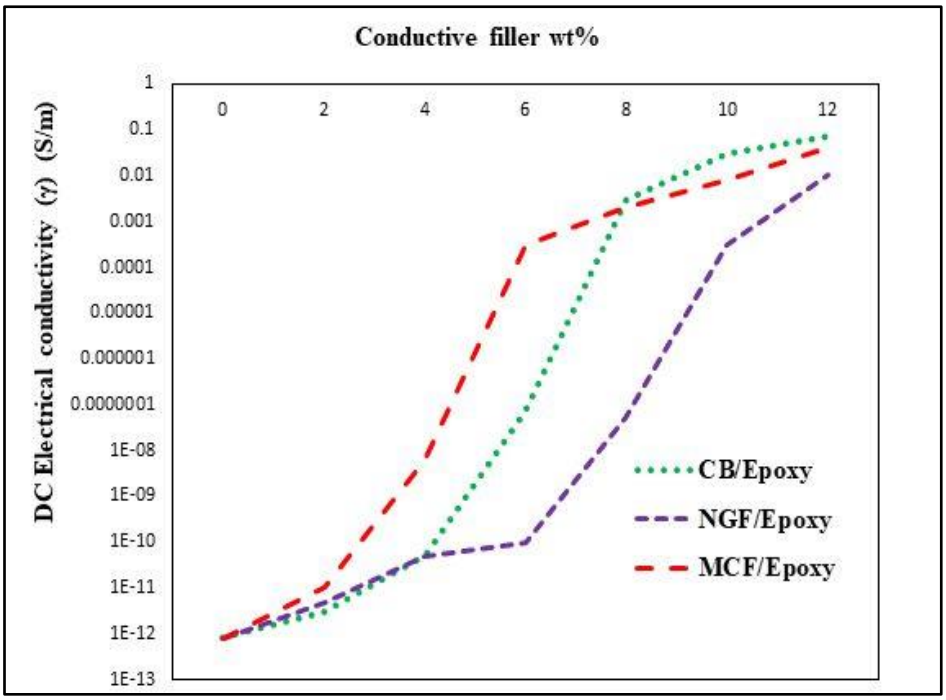

Fig. 9. DC electrical conductivity of filler reinforced epoxy composites

. High aspect ratio and good dispersion properties of CB filler on epoxy matrix tend to improved conductive network [31]. At 12 wt% of conductive filler content, CB/epoxy composites possess six folds higher conductivity ($7x10^{-2}$ S/m) than NGF/epoxy composites ($1x10^{-2}$ S/m) and 0.75 folds higher than MCF/epoxy composites ($4x10^{-2}$ S/m). Weaker covalent bonds between the layers of NGF leads to reduced tunneling and comparatively lesser electrical conductivity. Results obtained from four point probe testing method shows that the conductive filler/epoxy composites transfer from insulating characteristics into semiconductors.

Evaluation of mechanical and electrical properties

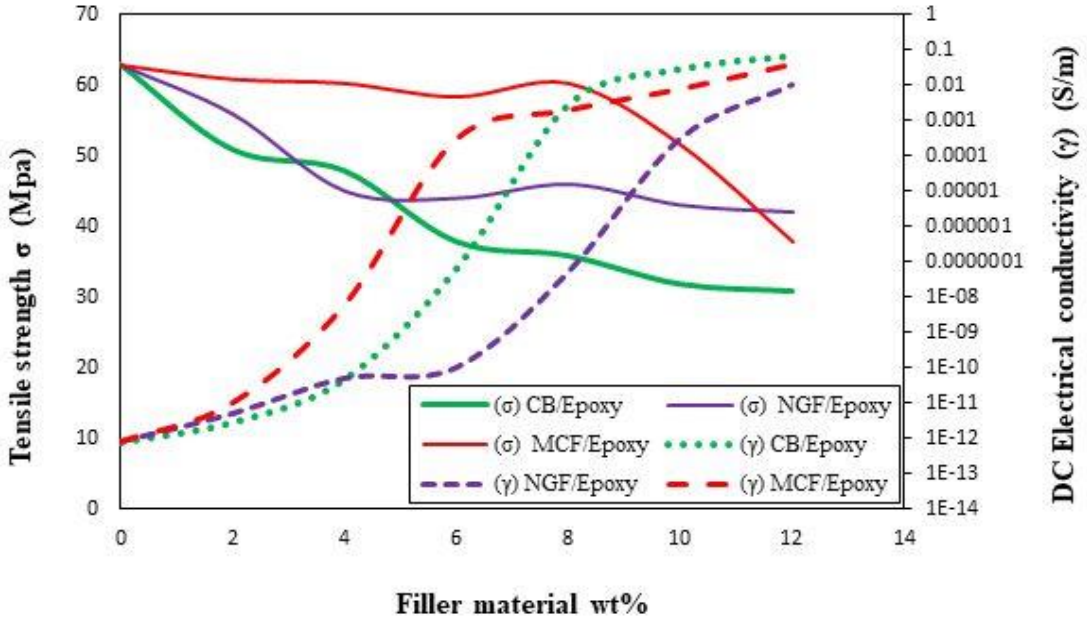

Fig. 10. Evaluation of mechanical and electrical properties of filler/epoxy composites

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Noticeable coincidence on the tensile strength and DC electrical conductivity based on filler content is mentioned in Figure 10. CB filler loading on epoxy leads to the generation of conducting CB paths from lower concentrations (2 wt%); which attributes higher conductivity, but also the reduction in tensile strength. The composite loaded with NGF filler drastically loses its tensile strength even at lower (2 wt%) filler concentration and the conductive network also poor when compared to other two types of filler reinforcements. For composites loaded with MCF filler, a steep drop in tensile strength occurs from the filler concentration after percolation threshold only. At 8% weight of MCF loading on epoxy, the composite holds $2x10^{-3}$ S/m of DC electrical conductivity as that of CB reinforced composites. Carbon fiber fillers are able to form a conductive path way with respect to the amount of fillers reinforced in epoxy composites [32]. However the loss in tensile strength at 8% weight of MCF reinforcement is only 4.12% on the tensile strength of pure epoxy (0% filler). It is obvious that the conductive network formation substantially improves the conductivity of composites and falling off on the mechanical property.

Formation of good conductive network will be a better choice to improve the electrical conductivity of filler reinforced polymer composites. In this study, carbon based fillers with different morphological properties were used. Combination of these fillers can give better packing density on epoxy composites.

Conclusion

Remarkable differences were found in the tensile properties and electrical conductivity of MCF/epoxy composites when compared to the properties of composites prepared by CB/epoxy and NGF/epoxy combinations. For 8% weight of filler reinforcements, CB/epoxy composites possess 50% higher electrical conductivity but 68% reduced tensile strength than MCF/epoxy composites because of high conducting network capability and poor chemical reaction of CB with epoxy which weakens the transfer of stress from matrix to reinforcement. MCF/epoxy, CB/epoxy, NGF/epoxy composites reach the percolation threshold at 6% weight, 8% weight, 10% weight filler reinforcements respectively. Reduced inter-particle distance of MCF reinforcement enhances the conductivity. 8% weight of MCF reinforcement in epoxy exhibits balanced mechanical and electrical properties caused by high ordering structure and conductive path of two dimensional MCF particles. Tensile strength, elastic modulus and elongation percentage values started to diminish for all batches of composites when they are reinforced with more than 10 wt% of carbon fillers due to the formation of agglomerations and void contents.

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