Tribological and Mechanical Performance Characteristics of Epoxy-Resin Composites Reinforced with Multi-Walled Carbon Nanotubes for Sustainable Applications

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Abstract

The increased awareness on devastating effect of synthetically developed materials towards the nature has resulted in the development of eco-friendly and sustainable materials. Natural fibers are weak on its own so, they are used as reinforcement in polymers. The objectives of the current work are focused on fabricating and testing epoxy-nanocomposites reinforced with different concentrations of multi-walled carbon nanotubes (MWCNTs). MWCNT-epoxy resin nanocomposites are prepared with the help of ultrasonic cell crusher and compression moulding process. Samples are subjected to different mechanical testing such as tensile strength, tensile modulus, flexural strength, flexural modulus, impact strength and hardness test in accordance with ASTM standards. It is found that the mechanical performance of MWCNT-epoxy nanocomposites are improved significantly with the addition of MWCNTs. Pin-on-disc (POD) was used to analyse the tribological characteristics of nanocomposites at varying loading conditions. Worn surfaces of POD samples are analysed using Scanning electron microscope (SEM) to elucidate the wear mechanisms. In addition, it is
experimentally proven that strong interaction between epoxy and MWCNTs exist and it helps to improve the interfacial strength between the particles.

**Keywords:** Eco-friendly, Sustainable, Polymer, MWCNT, Nanocomposites, Tribology

**1 Introduction**

Recently, importance in the synthesis and properties of nanoparticles has progressively grown because of the great potentials for their applications in different fields of material science and technology [1, 2]. This is due to their exclusive properties such as optical, catalytic, magnetic, anti-friction, anti-wear and others [3]. The evolution of carbon nanotubes (CNTs) has led to their application in development of different composite materials with varying properties [4]. Kaya and Parlar [5] investigated that based on variations in the reinforcement material and its direction, and the type matrix material, composites exhibit different tribological behaviour. Application of CNTs as mechanical and wear reinforcement for epoxy resin composites were studied by Zhu [6, 7]. Allaoui et al. [8] studied the performance of MWCNTs in rubbery epoxy matrix, and concluded that there was considerable increase in Young’s modulus and strength by adding MWCNTs upto 4% wt. Upadhyay and Kumar [9] successfully improved the wear and friction characteristics of the CNT added composites. Campo et al. [10] investigated the tribological performance of epoxy composites for different contents of CNTs and concluded that best performance was shown by 5% CNT composites. Lim et al. [11] studied the improved wear resistance in the polymer matrix by the incorporation of MWCNTs.

Nowadays the main application of CNT concentrates over the area of semiconductor and remote sensor other than automotive and aerospace [12, 27]. Since then after Iijima [13] discovered MWCNT and single-walled carbon nano tubes (SWCNT) [14], the interest for carbon nanotube composites got highlighted. Mixing of CNT and polymer matrix produces inimitable enriched electromagnetic absorption [15] and electrical properties [16, 17, 18,19]. Because of low production cost, better properties and their easy availability MWCNT can be the best choice as reinforcement. Low percentage of MWCNT in epoxy matrix can enhance its mechanical properties because of its greater aspect ratio [20]. As a part of strengthening the composite material, a MWCNT Nano filler with greater surface area is used which ensures better transfer of stress to the fillers [21]. Graphene sheets of two or more layers are used in MWCNT structural forms especially in the form of concentric tubes [22]. Low tensile strength nanocomposite is obtained due to the poor interfacial interaction between nano filler and polymer matrix. Further results in the production of lumps of nano filters and it is found to be held together by the impact of Van der Walls force [23, 24].
However it is noticed that limited literature is available on the work of epoxy-MWCNT nanocomposites [25]. The present work primarily focuses on the fabrication and testing of MWCNT based epoxy nanocomposites using compression moulding technique. The composition of MWCNT is varied from 0 to 2 wt% of epoxy weight percentage [26]. After the fabrication process suitable testing specimens as per the standard ASTM procedure are made for the evaluation of mechanical and tribological properties [27]. Furthermore, an Extensive study on the wear mechanisms is evaluated using scanning electron microscope (SEM) on the worn MWCNT-epoxy composite pin surfaces.

### 2 Materials and Experimental Methods

#### 2.1 Materials

Epoxy material is a combination of resin and hardener. In this work, LY556 is used as the resin and HY951 is used as the hardener. The ratio of resin to hardener is 10:1. MWCNT (supplied by Platonic Nanotech Pvt. Ltd.) is used as the nanoparticle reinforcement. Table 1 shows the properties of MWCNT nanoparticles.

<table>
<thead>
<tr>
<th>Properties</th>
<th>Range/Size</th>
</tr>
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<tbody>
<tr>
<td>Diameter</td>
<td>5 ~20 nm</td>
</tr>
<tr>
<td>Length</td>
<td>2-10 microns</td>
</tr>
<tr>
<td>Surface area</td>
<td>250-270 m²/g</td>
</tr>
</tbody>
</table>

#### 2.2 Fabrication of Composites

300 g of epoxy and 30 g of hardener is used for fabrication of each composite. Steel moulds are used for preparing the composite material, which consists of a base plate and a top plate. The plates are cleaned using an abrasive paper of 1000 grit size and is further washed in acetone. Surfaces are then dried in a hot air oven for 10 min. Compositions of the prepared nanocomposite materials are shown in Table 2. In the initial step, the epoxy resin and MWCNT particles are mixed in definite proportion in an ultrasonicator for 2 h. The mixed resin-nanoparticle dispersion is then treated with hardener in the proportion of 10:1. The MWCNT epoxy-hardener mixture is then poured over the metal mould uniformly and compressed for a curing time of 3 h. The size of the fabricated composite is restricted to 200 ×
200 × 6 mm. Then specimens are cured under room temperature by applying uniform pressure on the mould using the compression moulding apparatus. After the curing processes, these test samples are cut using water jet machining to the required sizes as prescribed in the ASTM standards for different characterizations. The photographs of the prepared laminates and POD specimens are shown in Fig. 1 (a, b)

### 2.3 Mechanical Testing

The details of tensile, flexural, impact, hardness and tribo tests conducted in the present work are explained below. Under each test method three trials were conducted and the average value observed under each trial is explained in Figs. 2, 3, 4, 5 and 6. Error analysis was carried out for the experiments and it was found that the trial values lie between the standard deviation.

<table>
<thead>
<tr>
<th>Laminate code</th>
<th>Material Combination</th>
</tr>
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<tbody>
<tr>
<td>A</td>
<td>Epoxy (300 g) + Hardener (30 g) + MWCNT (0 wt%)</td>
</tr>
<tr>
<td>B</td>
<td>Epoxy (300 g) + Hardener (30 g) + MWCNT (0.5 wt%)</td>
</tr>
<tr>
<td>C</td>
<td>Epoxy (300 g) + Hardener (30 g) + MWCNT (1 wt%)</td>
</tr>
<tr>
<td>D</td>
<td>Epoxy (300 g) + Hardener (30 g) + MWCNT (1.5 wt%)</td>
</tr>
<tr>
<td>E</td>
<td>Epoxy (300 g) + Hardener (30 g) + MWCNT (2 wt%)</td>
</tr>
</tbody>
</table>

**Figure 1 (a)** Prepared nanocomposite laminates
2.4 Tensile and Flexural Test

The mechanical behaviour of the composites prepared with the fabricated samples are tested using universal tensile testing machine with testing load range of 5 ton. The experiments are conducted at normal room temperature. The tensile strength is determined as per ASTM D638. The test specimens are cut as per ASTM standards using water jet machining. Flexural test obtained by three point bending test is done with ASTM D790 standards.

2.5 Impact Test

Impact strength of the composite specimens are carried out using izod impact testing machine according to ASTM D256 standard. A notch depth of 2.5 mm is cut on each specimen for carrying out the operation.

2.6 Hardness Test

The hardness test was carried out using a shore D testing machine. Corresponding hardness value was found by the penetrating indenter foot of the durometer into the sample in accordance with ASTM D2240.

2.7 Wear Test

The pin-on-disc (POD) machine is used for conducting wear tests as per ASTM G99-05 standards. Composite specimens were used as the pin material and the disc used here is made up of steel EN31 with a hardness number of 60 HRC. Square pins of size 5 x 5 mm and length 30 mm are used for the experimentation. 10 N, 15 N and 20 N were the different loads applied at the surface interface.
2.8 Surface Topography Analysis

The surface topography of MWCNT-Epoxy composites are examined using a SEM. SEM studies are conducted by scanning the samples at an accelerating voltage of 25 kV at a vacuum level of $1.5 \times 10^3$ Pa. The specimen surfaces are examined at different magnification levels to assess the surface topography.

3 Results and Discussion

The results of mechanical and tribological testing and the analysis of surface morphology of worn surfaces are detailed in next session.

3.1 Tensile Properties

The results of tensile strength and modulus, respectively, of MWCNT-epoxy composites are depicted in Fig. 2 (a, b). The combination B shows higher tensile strength and modulus of 72.33 MPa and 3550 MPa, respectively. Comparing among all other concentrations in the epoxy matrix, the greater tensile strength was achieved with 0.5 wt% of MWCNT. In this case between the epoxy and MWCNT nanoparticles, a better interfacial interaction is being observed. Moreover 0.5 wt% MWCNT exhibits better nanotube dispersion with little agglomeration. However concentrations beyond 0.5 wt% MWCNT in epoxy matrix exhibits non-monotonous behaviour due to agglomeration of nanotube in epoxy composites.

![Figure 2 (a) Tensile Strength of Composites](image-url)
3.2 Flexural Properties

The results of flexural strength and flexural modulus of MWCNT-epoxy composites are depicted in Fig. 3 (a, b). The combination C (1 wt% MWCNT) exhibits higher flexural strength of 113 MPa and the combination D (1.5 wt% MWCNT) exhibits higher flexural modulus of 3290 MPa. A betterment in flexural strength and flexural modulus of 25% and 40.5% has been noticed for nano-based epoxy composites with material composition C (flexural strength 1 wt% MWCNT) and D (flexural modulus 1.5 wt% MWCNT) respectively when compared with material composition A (Neat-0 wt% MWCNT). The enhancement of flexural strength for 1 wt% nanocomposites may be due to stronger resin and MWCNT interfacial interaction. These strong interactions between epoxy and MWCNT nanotubes help to improve the inter-laminar shear strength between the particles.
3.3 Impact Properties

Fig. 4 shows the results of the impact test. The test reveals that the combination B (0.5 wt% MWCNT) exhibits higher impact strength of 47.10 J/m. An improvement in impact properties of 15.9 % has been noticed for nano-based epoxy composites with material composition B, when compared with material composition A. It is remarkable to note that impact properties are in concurrence with tensile properties with highest values for 0.5 wt% MWCNT-epoxy nanocomposites. The excess of MWCNT increases the viscosity of epoxy resin. But because of the greater specific surface area of MWCNT, some surfaces of the same will not be completely covered by the epoxy matrix. This scenario leads to a difficult situation of uniform distribution of nanotubes and further load transfer.

3. Hardness Properties

Fig. 5 shows the result of hardness test and is evident that the hardness of the combination C (1 wt% MWCNT) and the combination E (2 wt% MWCNT) exhibits higher hardness value of 88 shore D. Following are the reasons behind the enhancement in hardness properties. High crystallinity exhibited by nanocomposites and incorporation of MWCNT in epoxy resin are the primary and secondary reasons for the greater hardness [25]. MWCNT comes under materials with larger strain energy, rigidity and hardness of the nanocomposites can be increased by the homogeneous dispersion by interfacial collaboration in MWCNT [26].

3.5 Wear Properties

POD wear test is carried out for the sample of size 5x5x30 mm. The sample is subjected in a perpendicular direction against a rotating counter
face. Wear test graph’s for Neat composite, 0.5 wt%, 1 wt%, 1.5 wt%, and 2 wt% MWCNT-epoxy for 10 N, 15 N and 20 N are shown in Fig. 6. The test reveals that the combination B (0.5 wt% MWCNT) exhibits better tribological performance for all the load range among the combinations selected for this study. It is also verified as per Archard’s law that wear loss increases as the load range is increased for each material combination. A detailed explanation on the wear mechanism with the help of SEM images is exhibited in the following section.

3.6 SEM Images of MWCNT-Epoxy Composites

Fig. 7 depicts the SEM images of pin surfaces after the tribological test on POD. The images are taken for a magnification scale of 200X, using secondary electron imaging (SE), for an image size of 1000X1000 µm. Fig. 7(A) shows the pin surface after sliding of neat epoxy resin only. It is clear
that the surface is rough with a significant amount of burr formation and wear debris during the sliding process, which is clearly indicated in the image. Figs. 7 (B, C, D, and E) shows the pictures of pin surfaces after sliding with 0.5 wt%, 1 wt%, 1.5 wt%, and 2wt % MWCNT-epoxy composites respectively. From this it is observed that the smoothest surface is obtained for pin surface with 0.5 wt% MWCNT. This is in agreement with the wear studies where the wear rate is minimum for composite with 0.5 wt% MWCNT. It is also noticed that, as we increased the concentration percentage of MWCNT from 0.5 wt% to 2 wt%, wear debris, deep shallow grooves, stepped fracture, plugging, crack and crack propagation are exhibited in the SEM images. As the concentration of MWCNT increases to higher levels, it is also observed that the adhesive wear dominates which results in excessive wear rate. Furthermore it could be noticed that, from the surface of worn pin the quantity of materials peeled off is very much minor for nanocomposites compared with neat epoxy. It is also evident that the presence of exfoliation, plastic deformation and adhesion is very minimal for 0.5 wt% MWCNT added epoxy resin as observed from the SEM image.

Figure 6 Wear test at load 10 N, 15 N, 20N
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4 Conclusions

Epoxy based MWCNT composites are fabricated by compression moulding method. After testing and characterization the following observations are made from this study:

- The comparison of tensile strength and tensile modulus reveals that the combination 0.5 wt% MWCNT-epoxy composite has 72.33 MPa and 3550 MPa exhibit higher tensile strength and higher tensile modulus. Whereas the neat composite exhibits only tensile strength of 49.97 MPa and tensile modulus of 2750 MPa.
- 1 wt% MWCNT-epoxy exhibits better flexural strength of 113 MPa and 1.5 wt%, MWCNT-epoxy exhibits better flexural modulus of 3290 MPa. However, neat composite exhibits only flexural strength of 90.40 MPa and flexural modulus of 2340 MPa.
- Impact tests reveal that 0.5 wt%, MWCNT-epoxy composite possess higher impact strength of 47.10 J/m which is much higher than the neat composite of 40.62 J/m.
- Hardness value of 1 and 2 wt% MWCNT-epoxy composite exhibits better hardness of 88 shore D, whereas neat composite exhibits only 85.
- Tribological studies revealed that the addition of MWCNT decreases wear loss. The best result is achieved with 0.5 wt% MWCNT. The wear mechanisms found for the nanocomposites are adhesive wear, fatigue and delamination.
- SEM images indicate that 0.5 wt% nanocomposites have the lowest abrasive wear, less number of wear debris and high filler bonding. The reason for the same is found to be the improved interaction developed between MWCNT and epoxy matrix.

Figure 7 SEM images of A, B, C, D, E nanocomposite samples after sliding of wear regions at 15 N
Overall, the specimen B (0.5 wt% MWCNT-epoxy nanocomposite) exhibited good tribological and mechanical performances.

References


Biographies

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