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Effect of wt% of Reinforcement on Tribological Properties of Epoxy/Al_2O_3 Polymer Nanocomposites

Himanshu Tiwari[1*], Sandip Kumar Singh[1], Shashank Dubey[1]

[1]Department of Mechanical Engineering, Veer Bahadur Singh Purvanchal University, Jaunpur, Uttar Pradesh, India 222003

***Corresponding Author :** Himanshu Tiwari
*Email : himanshu.vbspu@gmail.com ORCID : 0000-0002-1045-7798

ABSTRACT

By using an immediate cross-linking procedure, five samples were created. Neat epoxy and epoxy filled with 0.5, 1.0, 1.5 and 2.0 wt% of Al_2O_3 nanorods. At four different sliding distances of 15, 30, 45 and 60 meters friction and wear tests were performed on all samples using a 'linearly reciprocating ball on flat tribometer' against an ASE 304 stainless steel counter surface at room temperature with a constant load of 15 N. It was found that when epoxy is reinforced with 2 wt% of Al_2O_3, the coefficient of friction decreases by 70% and volume loss decreases by 52% compared to that of pure epoxy for a total sliding distance of 60 meters. It was also found that as the wt% of Al_2O_3 nanorods increases in epoxy, average surface roughness decreases, resulting in a lower coefficient of friction. Scanning electron microscopic tests were also performed on all samples. It was found that epoxy containing 2.0 wt% of Al_2O_3 nanorods is smoothest among other images, which is evidence of wear resistance. It is the subject of further research what will happen if the wt% of Al_2O_3 nanorods in epoxy increases beyond 2 wt% but it is clear from this research that the minimum wt% of Al_2O_3 nanorods in epoxy should be at least 2 wt% to achieve efficient wear resistance.

Keywords : Epoxy, Al_2O_3, Nanocomposites, wt%, Friction, Wear, Surface Roughness

INTRODUCTION

Polymers are created via the polymerization of many small molecules (monomers) into a covalently bonded chain. Special features of polymers are resistance to environmental corrosion, ability to sustain impact load, low density, high specific strength, quiet operation due to vibration and noise absorption. However, there are some inherent shortcomings, such as lower load-carrying capacity, higher friction and wear rates than those with liquid-lubricated metals, poor thermal conductivity and limited dimensional stability due to thermal expansion. Hence, neat polymers are generally not adequate for the required end use. To overcome the shortcomings of the above-mentioned neat polymer, the reinforcement of nanosize particles is made in the polymer matrix. In composite materials one of the phases is usually discontinuous, stiffer and stronger which is called reinforcement, whereas the less stiff and weaker phase is continuous and called a matrix. The properties of composite materials depend on the properties of its constituents as geometry, weight percentage etc. Out of which, an important parameter is the volume (or weight) percentage of reinforcement. The degree of reinforcement depends on several factors, including whether the polymer is crystalline or not and the amount of crystallinity that has developed.

Semi-crystalline polymers are more efficiently reinforced than amorphous polymers. Because reinforcement becomes surrounded by a microcrystalline structure, which binds the reinforcement more firmly to the polymer [1]. Reducing the particle size to a nanoscale level is assumed to reach a significant efficiency [2]. Nanosize particles as reinforcing material show their quantum size effect, surface effect and volume effect, which make the nanocomposites have excellent wear resistance and good corrosive resistance, excellent heat resistance, high strength and low creepage [3]. Recently, nanometer sized inorganic compounds such as TiO_2, ZnO, SiO_2, Al_2O_3, Si_3N_4 etc. were tried as the fillers of fabric composites and polymers to improve the tribological properties [4-9]. Epoxy-base matrix composites have tremendous potential to substitute for traditional metallic materials [10]. Epoxy resin (EpR) is one of the most common thermoset polymers. Polymer nanocomposite has the advantages of low cost and simplified manufacturing processes [11].

Wear is the loss of materials on the surfaces of two rubbing bodies. The changes in the surface layer arise from mechanical stresses, temperature and chemical reactions [12]. While a broader definition of wear includes any form of surface damage caused by rubbing processes on one surface against another [13]. The amount of wear in any system will, in general, depend upon a number of factors, such as the applied load, machine characteristics, sliding speed, sliding distance, the environment, and the material properties. The value of any wear test method lies in predicting the relative ranking of material combinations. It does not attempt to duplicate all the conditions that may be experienced in service (for example, lubrication, load, pressure, contact geometry, removal of wear debris, and presence of corrosive environments); there is no assurance that the test will predict the wear rate of a given material under conditions differing from those in the test [14]. In the case of polymethyl methacrylate (PMMA) reinforced with nanofiller DSNP, tested under a normal load at 30 N, the results indicated improved resistance to wear rate and coefficient of friction of pure PMMA by 88.54% and 66.67% respectively by adding 1.2 wt% DSNP. The nanofiller concentration up to 1.5 wt% led to agglomeration, porosity formation, and decreasing mechanical and tribological properties of nanocomposites [15]. In the case of an epoxy polymer matrix reinforced by 0, 1, 3, 5, and 7 wt% of nanoclay, applied with a constant load of 25.5 N, the hardness of the nanocomposite increased by 33.8% up to 5 wt% loading of nanoclay and decreased with further increase in it. The specific wear rate of the nanocomposite having 5% nanoclay was found to be the least (reduced by 20%) as compared to other composites [16].

In the case of a polyester resin matrix reinforced with 4 wt% of glass fiber and 1, 3, 5 wt% of nanosilica particles, the wear rate decreased from (22.5×10^{-4} $cm^3/N.m$) for (pure unsaturated polyester resin) to (0.18×10^{-4} $cm^3/N.m$) for a specimen having 5 wt% nanosilica under an applied load of 7N, a sliding duration of 15 minutes, a sliding speed of 2 m/s and a sliding distance of 7 cm [17].

In the case of epoxy matrix reinforced with 1, 3, 5 wt% of grapheme and 1, 3, 5 wt% of multi-walled carbon nanotubes (MWCNTs) and 10, 30, 50 wt% of chopped carbon fibers separately tested for wear resistance. It was observed that increasing the wt% of any reinforcement reduces the wear rate significantly in its range. Also, graphene enhanced the wear resistance of the nanocomposites by 75% compared to 50% and 38% obtained for MWCNT and carbon fiber composites, respectively [18].

The mechanical and sliding wear properties of epoxy resin (LY556) reinforced with graphite flakes by a pin-on-disc wear test apparatus were tested. It was observed that the specific wear rates of these composites strongly depend on their filler content and applied normal loads. Incorporating 3 wt% graphite flakes into epoxy/glass fabric composites leads to the lowest specific wear rate and the best mechanical performance. A further increase in the graphite content increases the specific wear rate and deteriorates the mechanical behavior [19].

In the case of epoxy/diamond nanocomposites, nanodiamond particles were reinforced up to 5 wt%. It was found that the wear resistance of epoxy/diamond nanocomposites increases up to a 2 wt% concentration of nanodiamonds; further increases in nanodiamond content result in decreased resistance to wear. The friction coefficient of the nanocomposites decreased gradually as the content of nanodiamonds in the composite increased from 0.4 to 5.0 wt%; any further addition of nanodiamonds led to a more efficient decrease in the friction coefficient [20]. The wear behavior of epoxy matrix having different percentages (0.1 and 0.5 wt%) of multi-walled carbon nanotubes (MWCNTs) has been studied using a “pin-on disc” wear testing machine. It was found that wear decreased with the increase in MWCNTs percentage. The nanocomposites with 0.5 wt% MWCNTs had better wear behavior [21]. The tribological properties of epoxy/rubber nanocomposites containing 5, 10, and 16 wt% nanorubber particles were investigated, and it was found that the wear of epoxy and its nanocomposites increased with increasing sliding time and applied load. The content of 5 wt% nanorubber particles was the most effective in reducing the wear loss and friction coefficient. With a further increase in the content of the nanorubber particles, the nanorubber particles agglomerated, and the hardness of the nanocomposites decreased, which resulted in an increase in the specific wear rate [22].

In studying the wear behavior of multi-walled carbon nanotubes (MWCNTs) with different fractions from 0.0 to 2.0 wt%. doped in epoxy resin. Wear behavior was investigated on a linear abrader with visual inspection on an optical microscope and SEM imaging, mass loss measurements and wear volume evaluation on a profilometer. It has been found that the best wear resistance is achieved for reinforcement between 0.25 and 0.5 %wt [23]. In the case of nickel nanoparticles (60–100 nm, filler content 0.5–10 wt%) incorporated in epoxy resin. Dry wear behavior was measured using the pin on disc wear tester. It was found that the friction coefficient and specific wear rate of epoxy resin can be reduced with a lower mass fraction of Ni particles. The lowest specific wear rate of 0.46 × 10-4 mm^3 /N.m (compared to the pure resin value of 2.261024×10^{-4} mm^3) was observed for composites with a filler content of 0.5 wt% [24].

MATERIAL AND EXPERIMENTAL METHODS

Materials

Low molecular weight epoxy with bisphenol A (LY 556) as matrix material and hardener (HY 951) were used in this work. Huntsman Advanced Material, India, provided both of the materials. At room temperature (25 °C), the densities of the epoxy and curing agent hardener utilized are 1.17 g/cc and

0.98 g/cc, respectively. In this study, cylinder-shaped alumina (Al_2O_3 nanorods) were used as reinforcement. These Al_2O_3 nanorods were 5–10 nm in diameter, up to 50 nm in length, 40 m^2/gm of specific surface area, and 4.0 gm/cc in density. These Al_2O_3 nanorods were provided by Sigma Aldrich Pvt. Ltd. and used in the present work to strengthen the epoxy in its natural state without any surface modification.

Synthesis of Epoxy/Al_2O_3 Polymer Nanocomposite

Al_2O_3 nanorod-reinforced epoxy nanocomposites were created using an immediate cross-linking technique. This study employed epoxy, a liquid polymer. Alumina particles of the requisite wt% (0.5, 1.0, 1.5 and 2 wt%) were heated at 150 °C for 4 hours to remove moisture before being used to prepare the samples. After that, dried Al_2O_3 nanorods were manually mixed for 10 minutes with acetone and the necessary amount of epoxy resin. This mixture was then subjected to an hour-long sonication. Following that, acetone was eliminated from the mixture using a hot plate procedure. The mixture was placed in a vacuum for 30 minutes in order to release the trapped gases created by the sonication process. Then hardener was gradually included in the mixture, such that the hardener-to epoxy ratio was 1:10. To release any gases that may have been trapped, this mixture was once again maintained in a vacuum for five minutes. Finally, the mixture was put into a vertical acrylic mold and allowed to cure for 24 hours at room temperature. These samples were then taken out of the molds and post-cured in a hot air oven at 100°C for four hours.

Density Measurement

Weighing equipment ('mettler toledo' ME155DU/A, capacity 152 g - 4 mg, least count 0.1 mg) was used to determine each sample's density. Each sample's density was determined using the Archimedes method.

$$\rho = \frac{M.\rho a}{M - Ma} \qquad (1)$$

Where ρ is density of sample (in g/cc), M is mass of samples in air (in g), Ma is mass of samples dipped in acetone (in g) and ρ_a is density of acetone (in g/cc).

Friction and Wear Measurement

The tribological properties of epoxy and epoxy containing Al_2O_3 nanorods were experimentally evaluated in accordance with ASTM standard G133-05 (2016). Dry sliding wear tests were performed using a ‘linearly reciprocating ball on a flat tribometer’ (specifications: Company Rtec instruments, maximum load up to 5000 N, temperature range 120–1200 °C, stroke 1–30 mm, and frequency up to 70 Hz). A counter surface of ASE 304 stainless steel balls with a 70 HRC room temperature hardness was used. All the samples were shaped like plates with dimensions of 20×10×10 mm. Prior to the wear testing, the samples were polished with sandpaper of varying grades, up to 2000 grade. At room temperature, a normal load of 15 N was used throughout the test, and the reciprocating speed was set at 50 mm/sec. During the test, a total of 60 meters of sliding distance were covered, and observations at 15, 30, 45 and 60 meters were taken. Three different wear tests were performed on each sample, with the average result being utilized.

Frictional force is displayed internally on the 'linearly reciprocating ball on flat tribometer' control panel when the test is being done. The usual applied load was divided by the frictional force to determine the coefficient of friction. The specimen's weight was measured before and after the test with an accuracy of 0.1 mg using an electronic balance. The difference between a specimen's mass before and after wear is referred to as mass loss. The volume loss is then determined by multiplying the mass loss by the density of the corresponding samples.

RESULTS AND DISCUSSION

Friction Test

Figure 1 illustrates the impact of wt% of reinforcement on the coefficient of friction at room temperature (25 °C) for different sliding distances of 15, 30, 45 and 60 meters. From Figure 1, it can be seen that the coefficient of friction for neat epoxy and epoxy filled with 0.5, 1.0 and 1.5 wt% Al_2O_3 nanorods first increases then decreases with sliding distance. However, for epoxy reinforced with 2.0 wt% of Al_2O_3 nanorods, the coefficient of friction increases very rapidly. Also this sample has a minimum coefficient of friction over all other samples at all sliding distances. The variation in coefficient of friction with sliding distance obtained cannot be described by a theoretical formula because it depends on specific surfaces, but its overall qualitative properties can be predicted.

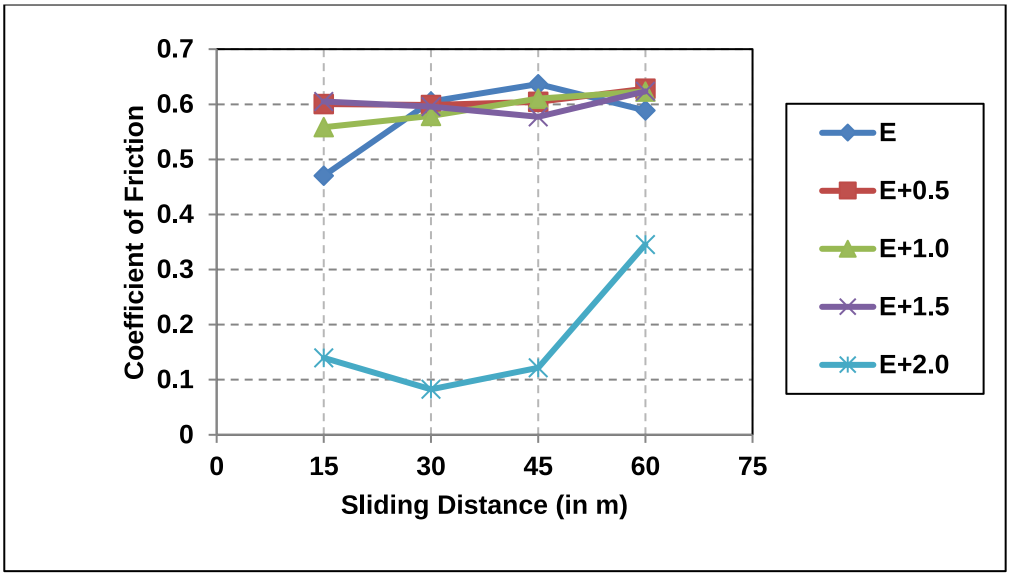


Figure 1. Variation in coefficient of friction with sliding distance for different wt% of Al_2O_3 nanorods in epoxy

Figure 2 illustrates the variation in the average coefficient of friction with the wt% of reinforcement for a total sliding distance of 60 m at room temperature (25 °C). From figure 2, it can be seen that with an increase in wt% of Al_2O_3 nanorods, the coefficient of friction first increases slowly and then decreases very rapidly. The reduction in coefficient of friction for epoxy containing 2.0 wt% of Al_2O_3 nanorods is about 70% greater than that for pure epoxy.

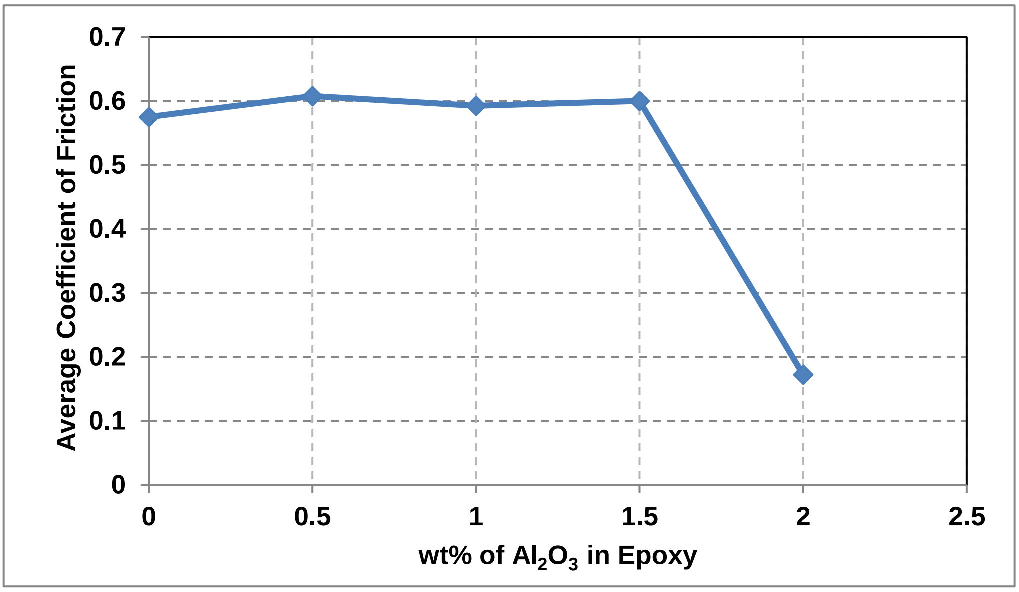


Figure 2. Variation in average coefficient of friction for a total sliding distance of 60 m

Figure 3 illustrates the variation in average surface roughness for neat epoxy and different wt% of Al_2O_3 nanorods in epoxy. From figure 3, it can be observed that with an increase in the wt% of Al_2O_3 nanorods, surface roughness decreases.

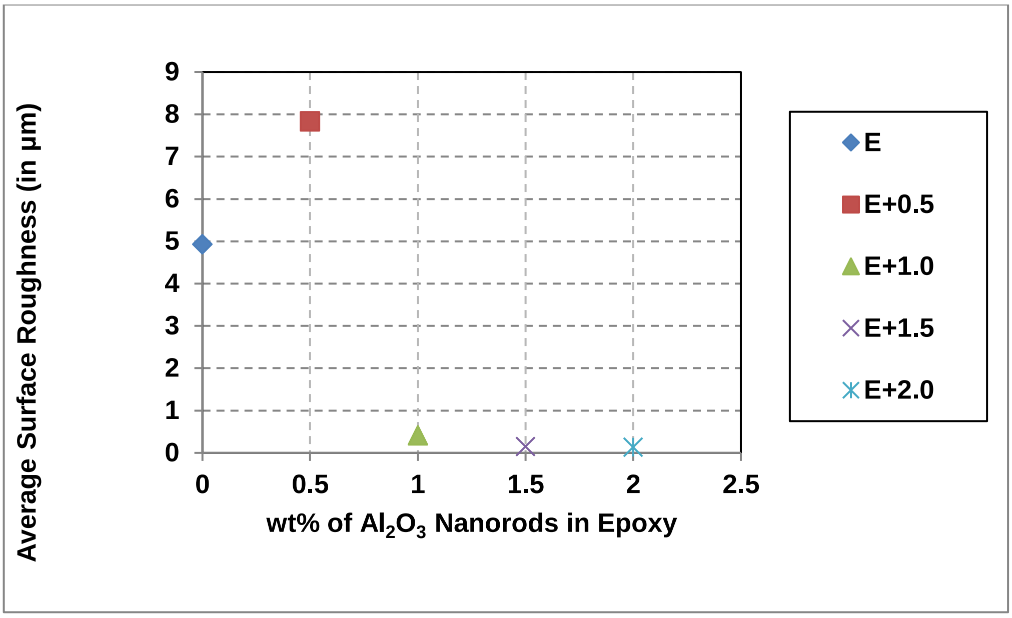


Figure 3. Variation in average surface roughness with different wt% of Al_2O_3 nanorods in epoxy

It is clear from the above graph that as the surface roughness of nanocomposite decreases, the corresponding average coefficient of friction also decreases, and vice versa.

Wear Test

Volume loss can be used to compare results with others only if the wear parameters (normal load, sliding speed, sliding distance) and wear conditions are the same. Figure 4 illustrates the effect of wt% of reinforcement on volume loss of neat epoxy and epoxy containing different wt% of Al_2O_3 nanorods for sliding distances of 15, 30, 45 and 60 m. From figure 4, it can be observed that neat epoxy has very low volume loss over all samples containing 0.5, 1.0 and 1.5 wt% of Al_2O_3 filled in epoxy, but since neat epoxy has limited dimensional stability, thermal stability, and other shortcomings, Hence, it must be reinforced to overcome these limitations. In the case of epoxy filled with 2.0 wt% of Al_2O_3 nanorods, there is minimum volume loss at all sliding distances, which shows the necessary required mimimum wt% of reinforcement to achieve overall capability. For epoxy filled with 2.0 wt% of Al_2O_3 nanorods, volume loss decreased by 52% compared to pure epoxy for a sliding distance of 60 meters. Increased wt% of Al_2O_3 nanorods increases thermal conductivity and strength; hence, loss of material in the form of melting and abrasion decreases.

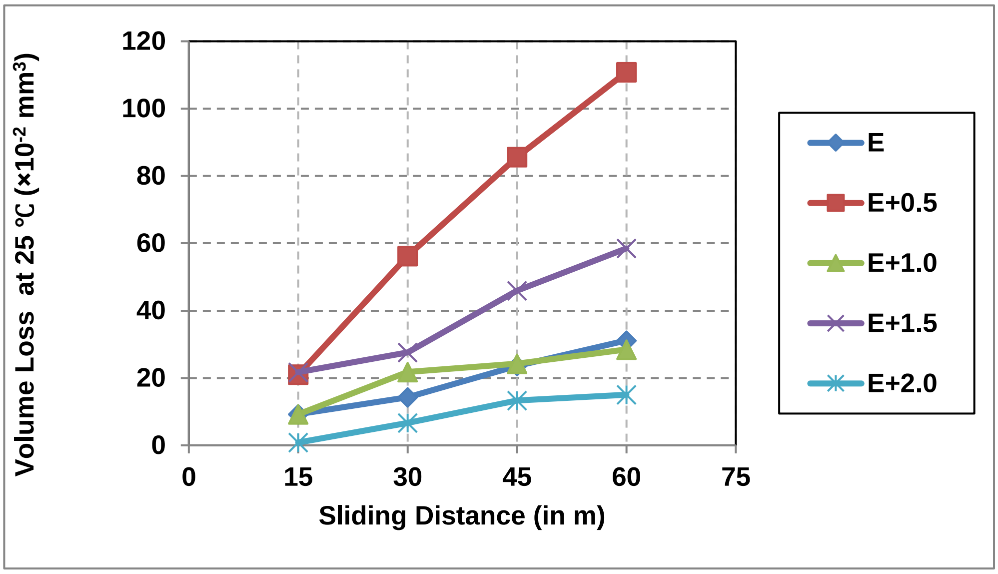
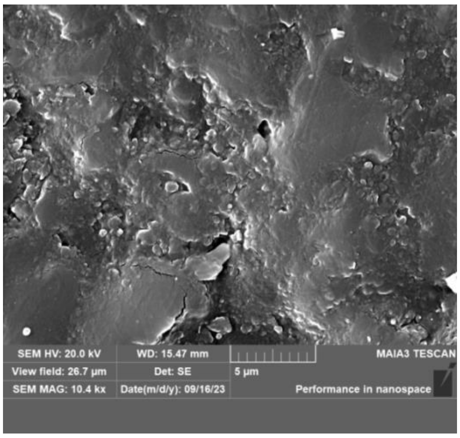


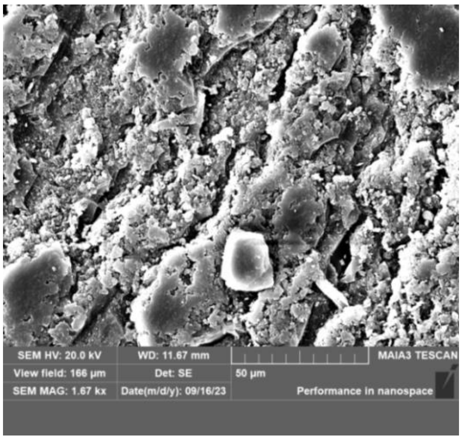
Figure 4. Variation in volume loss with sliding distance for different wt% of Al_2O_3 nanorods in epoxy

Morphology of Wear Surface

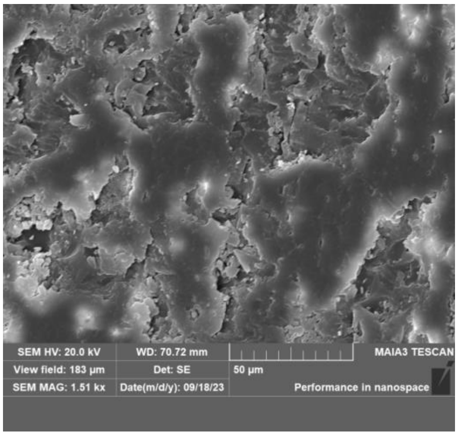
To study the wear behavior of the worn surfaces, scanning electron microscopy (SEM) is done. Figure 5 shows the fracture surface of neat epoxy and epoxy with different weight fractions of alumina nanorods at a room temperature of 25 °C. It is clear from Figure 5 that the surface of neat epoxy is much smoother. Epoxy, reciprocating against the hard counterface, undergoes fatigue wear [25]. Whereas, epoxy containing 2.0 wt% of Al_2O_3 nanorods shows minimum wear of the surface. The reason may be the optimum blending of fillers, leading to a much stronger surface that resists repeated shear loading during wear. Further, it can be concluded that the incorporation of fillers into neat epoxy reduces the severe wear to a milder one, which is evident from the worn surface images.



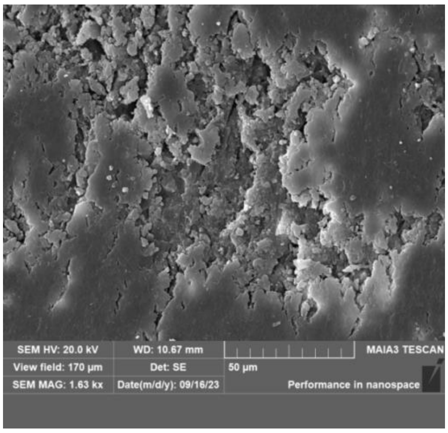
(a) Neat epoxy



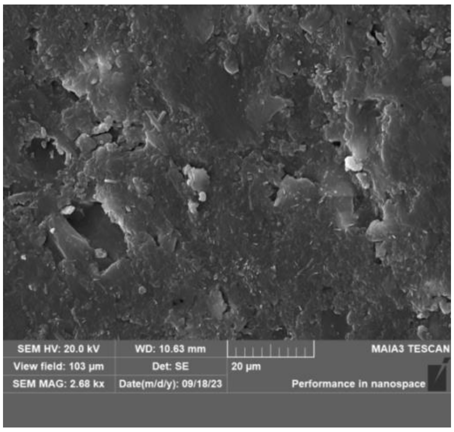
(b) Epoxy with 0.5 wt% of Al_2O_3



(c) Epoxy with 1.0 wt% of Al_2O_3



(d) Epoxy with 1.5 wt% of Al_2O_3



(e) Epoxy with 2.0 wt% of Al_2O_3

Figure 5. Scanning electron microscopic images of weared surfaces at 25℃

In this case, strong interfacial adhesion between Al_2O_3 nanorods and the epoxy reduced damping ability and enhanced resistance to thermal distortion of the nanocomposites, which improved the tribological performance of the epoxy/alumina nanocomposites. It has been known that the interaction between the nanoparticles and epoxy is quite strong. This should make the detachment of the particles from the matrix rather difficult, and thus the amount of material loss is reduced accordingly [26].

CONCLUSIONS

In the present study, epoxy/Al_2O_3 nanocomposites were made by reinforcing 0.5, 1.0, 1.5 and 2.0 wt% of Al_2O_3 nanorods in an epoxy matrix using a cross-linking procedure. At four different sliding distances of 15, 30, 45 and 60 meters, friction and wear tests were performed on all samples using a "linearly reciprocating ball on a flat tribometer" against an ASE 304 stainless steel counter surface at room temperature. The results can be outlined as follows:

- Neat epoxy shows better frictional and wear behavior if the wt% of Al_2O_3 nanorods is less than 2 wt%, but to overcome all other shortcomings of neat epoxy as thermal stability, strength etc reinforcement in epoxy is beneficial.
- Minimum wt% of Al_2O_3 nanorods in epoxy must be 2% to obtain effective tribological properties.
- Over all the samples, epoxy containing 2.0 wt% of Al_2O_3 nanorods has a minimum coefficient of friction (about 70% less than that for pure epoxy).
- As the wt% of Al_2O_3 nanorods increases in epoxy, average surface roughness decreases, resulting in a lower coefficient of friction.
- Over all the samples, epoxy containing 2.0 wt% of Al_2O_3 nanorods has minimum volume loss (about 52% less than that for pure epoxy).
- Scanning electron microscopic tests shown that epoxy containing 2.0 wt% of Al_2O_3 nanorods is smoothest among other images, which is evidence of wear resistance.

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