Tribological investigation of alumina/graphene nanoplatelets reinforced epoxy nanocomposites

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Abstract
Epoxy nanocomposites are widely used in sliding conditions of different applications such as structural components, automotive and marine vessels, etc. Thus, the study of such type materials currently drawing wide attention to the research communities. In this work, the micro hardness, the average coefficient of friction and the specific wear rate of the neat epoxy and alumina (Al₂O₃)/graphene nanoplatelets (GnP) reinforced epoxy nanocomposites were investigated. The various mix ratios of Al₂O₃/GnP particles were incorporated into the epoxy resin using the probe sonication method followed by the ball milling process in order to achieve the homogeneous dispersion in the epoxy matrix. The results showed that the micro hardness value for the combined loading of 2 wt% of Al₂O₃ and 0.5 wt% of GnP into the epoxy matrix was increased to 47.64% whereas the average coefficient of friction and specific wear rate value for the combined loading of 2 wt% of Al₂O₃ and 0.5 wt% of GnP reinforced epoxy nanocomposites was decreased to 18.3% and 76.92% when compared with the neat epoxy test coupons. The scanning electron microscopy (SEM) and atomic force microscope (AFM) images of worn out surfaces of composite specimens were used for the understanding of dispersion of the fillers and wear mechanisms.

1. Introduction
Polymers based composites are emerging nowadays as an alternative material for the substitute matrix materials in many anti-wear industrial applications. Because of the demand in the anti-wear applications like polymer-based bearings [1], automobile brake pads, artificial joints [2] and gears [3] are increasing, the enhancement of the tribological properties of polymer-based components is significantly required. Epoxy-based composites are generally used material for the structural, aerospace and coating industries, due to its better tensile strength, high corrosion resistance and chemical resistance [4–7]. The epoxy resin is a thermosetting material which generally has a high brittleness and weak wear resistance which limits its usage in the tribological applications and hence, it is significant to improve the tribological properties of epoxy with suitable reinforcement particles. Such effort has been carried out by many researchers with different filler materials, and existing results are reported [8–18].

In traditional routes, the combination of short carbon, graphene, multi walled carbon nanotubes and internal lubricants like graphite or polytetrafluoroethylene in an epoxy resin matrix leads to a significant enhancement in the dry sliding condition on opposing the metallic counterparts [19]. Nano fillers such as alumina, silica [20], and nanodiamonds are usually applied to enhance the tribological properties of the epoxy resin matrix [21–26]. The importance of alumina and graphene in epoxy composites are well established in many articles and hence, alumina and graphene are used as the filler materials in this study. The fabrication of Al₂O₃ and GnP reinforced epoxy nanocomposites is an important aspect to take into consideration for homogeneous dispersion of Al₂O₃ and GnP in the epoxy matrix. The best method to achieve good dispersion is sonication [27], planetary ball milling [28, 29], magnetic stirring and planetary ball milling [30] and both sonication and planetary ball milling process [31].
In this study, the composite samples were fabricated by incorporating Al₂O₃ and GnP at different loading conditions into an epoxy matrix to improve the tribological properties and the corresponding optimum wt% of the filler content was determined. Moreover, this paper also examined the micro hardness property of the neat epoxy and epoxy based nanocomposites and the correlation between the tribological and micro hardness properties of the epoxy composites was established.

2. Experimental methods
2.1. Materials
For the preparation of nanocomposites, the graphene nanoplatelets (GnP) and Alumina (Al₂O₃) powder were used as a filler materials. GnP is purchased from Sigma Aldrich, Bangalore, India and Al₂O₃ was purchased from SASOL, Germany. Epoxy resin of grade DGEBA (Lapox L-12) and the hardener (Lapox K-6) was purchased from Atul Limited, Gujarat, India. Acetone AR grade was procured from S D Fine-Chem Limited, Mumbai, India was used for the cleaning purpose in respect to the fabrication and during the experimentation process. All the materials were used as received condition. The properties of GnP, Al₂O₃, epoxy and hardener used in this study is shown in tables 1 and 2.

2.2. Preparation of alumina Al₂O₃ and GnP epoxy nanocomposites
The desired quantity of Al₂O₃ and GnP fillers shown in table 3 was added into epoxy resin. The combined solution was then sonicated (probe sonication apparatus Make: Johnson Plastosonic (India); Specifications: Probe diameter is 12 mm and Probe Material is titanium) at the frequency of 20 kHz for 30 min. During the sonication process, ice cubes were added in the ice bath to maintain the temperature of 25 °C. Epoxy/Al₂O₃/GnP mixtures were then ball milled by using a planetary ball mill machine (Make: VB Ceramics Inc. Chennai; Specifications: tungsten carbide jar and tungsten carbide balls) at 200 revolutions per minute for 120 min. Following that, a 10:1 mass ratio of epoxy to hardener was added and degassed at 80 °C for 10 min in the vacuum oven to remove the trapped air bubbles. Finally, the mixture was poured into an open mould and kept for 24 h for curing at ambient temperature. The post curing operation was performed in the oven at 80 °C for 2 h. A similar procedure was followed for the fabrication of all the variants of the composite specimens.

| Table 1. Physical properties of GnP and Al₂O₃. |
|-----------------------------------------------|
| Materials     | Properties          | Value          |
| GnP           | Carbon content (%)  | 99.5           |
|               | Surface area (m² g⁻¹) | 120–150        |
|               | Density (g cm⁻³)    | 0.03–0.1       |
|               | Particle size (μm)  | 25             |
|               | Thickness (nm)      | 6–8            |
| Al₂O₃         | Carbon content (%)  | —              |
|               | Surface area (m² g⁻¹)| 95             |
|               | Density (g cm⁻³)    | 0.35–0.65      |
|               | Particle size (μm)  | 35             |

| Table 2. Specifications of epoxy resin and hardener. |
|----------------------------------------------------|
| Specification of chemicals                         |
| Epoxy resin (Lapox L-12)                          |
| Epoxide equivalent (g eq⁻¹)                       | 182–192         |
| Viscosity at 25 °C (Pa.s)                         | 9–12            |
| Density at 25 °C (g cm⁻³)                         | 1.2             |
| Parts by weight                                   | 100             |
| Hardener (K-6)                                    |
| Density at 25 °C (g/cm³)                          | 0.9             |
| Water content (% of Max.)                         | 1               |
| Refractive index at 25 °C                         | 1.494–1.5       |
| Parts by weight (Mixing ratio)                    | 10              |
2.3. Characterization

2.3.1. Characterization of GnP and Al₂O₃ fillers

The morphology GnP filler was characterized using high resolution transmission electron microscope (HRTEM) setup (Tecnai, G2 20 twin).

The morphology of the Al₂O₃ filler was obtained using the scanning electron microscope (SEM) (Carl Zeiss India (Bangalore) Private Limited (Model: EVO18)).

2.3.2. Characterization of epoxy nanocomposites

Hardness of neat epoxy and epoxy based nanocomposites samples were measured by using micro hardness tester (Make: Matsuzawa Co., Limited (Japan); Model: MMT-X7; Specifications: Test load from 1 gf to 2000 gf). Diamond indenter (squared based pyramid) was used for applying the load of 100 gf on the surface neat epoxy and epoxy nanocomposites at the dwell time of 15 s [8]. The actual setup of hardness tester used for the measurement is shown in figure 1. The dimensions of the specimens used for hardness test is 8 mm diameter and 10 mm thickness (figure 2).

The hardness test was performed according to ASTM E 384. The average of five indentations was taken at five different arbitrary locations and corresponding hardness values were reported. After hardness test indentation was mapped using optical microscope and SEM. The hardness indentation optical microscope images were taken at a magnification of 40 × and SEM images were taken at a magnification of 2000 ×.

The tribological properties of neat epoxy and epoxy nanocomposites specimens were examined by using a pin on disc tester (Make: Ducom, Bangalore, India; Model: TR–20–PHM 600) under dry sliding (ambient temperature) contact condition. The actual setup used for wear test is shown in figure 3. Specification of the

| Samples codes | Descriptions |
|---------------|--------------|
| EP            | Neat epoxy   |
| EPA1G0.1      | EP reinforced with 1 wt% of A and 0.1 wt% of G |
| EPA1G0.25     | EP reinforced with 1 wt% of A and 0.25 wt% of G |
| EPA1G0.5      | EP reinforced with 1 wt% of A and 0.5 wt% of G |
| EPA1G1        | EP reinforced with 1 wt% of A and 1 wt% of G |
| EPA2G0.1      | EP reinforced with 2 wt% of A and 0.1 wt% of G |
| EPA2G0.25     | EP reinforced with 2 wt% of A and 0.25 wt% of G |
| EPA2G0.5      | EP reinforced with 2 wt% of A and 0.5 wt% of G |
| EPA2G1        | EP reinforced with 2 wt% of A and 1 wt% of G |
| EPA3G0.1      | EP reinforced with 3 wt% of A and 0.1 wt% of G |
| EPA3G0.25     | EP reinforced with 3 wt% of A and 0.25 wt% of G |
| EPA3G0.5      | EP reinforced with 3 wt% of A and 0.5 wt% of G |
| EPA3G1        | EP reinforced with 3 wt% of A and 1 wt% of G |

Note: A = Al₂O₃; G = GnP.
machine load range from 0 to 60 N (in this case 15 N load was applied) and rotational speed from 0 to 500 rpm. During the wear test, the pin is pressed against the counter surface of the rotary disk with a specified load being applied to arm attachment provided for the setup. The dimensions of the rotary disk with a diameter of 165 mm and a depth of 8 mm, EN-31 hardened steel with a hardness of 50–62 HRC and surface roughness value (Ra) 0.55–0.65 μm was used. Flat-faced composite pin with the dimensions of 8 mm diameter and 50 mm length. Figure 4 shows the fabricated pin specimens of neat epoxy, Al₂O₃, and GnP reinforced epoxy nanocomposites for the wear test.

Before the wear test, the contact surface of the pins was polished with various grades of silicon carbide sandpapers ranging from 220 to 600 grit size. Then, the surfaces of both the composite pin and the rotary disc were cleaned with acetone using a cotton cloth to remove the impurities presented on the specimen surface. The weights of the composite pins were measured to an accuracy of ±0.1 mg in a digital weighing balance (Mettler-Toledo India Private Limited), before and after the wear test.

The pin on disc test was performed under the operating parameters of sliding speed of 1 m s⁻¹, applied loads of 15 N, wear track diameter of 80 mm and sliding distance of 2000 m. The coefficient of friction (μ) was determined by taking into an account of the applied load and the frictional force at the contact surface. The specific wear rate (k) was measured by using equation (1). To determine the specific wear rate and coefficient of friction, ASTM G 99 standard was adopted. The wear test was repeated for 5 specimens of neat epoxy and Al₂O₃/GnP reinforced epoxy nanocomposites samples.
\[ k = \frac{\delta M}{\rho P x} \]  

Where \( k \) is the specific wear rate \((\text{mm}^3 \text{Nm}^{-1})\), \( \delta M \) is the specimen weight loss \((\text{g})\), \( \rho \) is the density of composite pin specimens \((\text{g} \text{mm}^{-3})\), \( P \) is the applied load \((\text{N})\) and \( x \) is the sliding distance \((\text{m})\).

After the wear test, the SEM images of the worn surfaces of composite specimens were obtained to discuss the wear mechanism. All the composite specimens were sputtered with gold coated before the SEM analysis. The roughness (Ra) of the worn surface of the tested neat epoxy and epoxy nanocomposites was also measured by using Nanosurf easy Scan 2 Atomic Force Microscope (AFM).

3. Results and discussions

3.1. Morphological analysis of GnP and Al\(_2\)O\(_3\) fillers

Figure 5 shows the TEM image of the GnP particles used in the fabrication of epoxy composites. These particles were of sheets like crumpled structure and consist of many layers.

The morphology of the Al\(_2\)O\(_3\) was characterized using SEM. Figure 6 shows the SEM image of the as received Al\(_2\)O\(_3\) particles exhibited a spherical shaped structure. The size transformation mechanism of as received micro size of the Al\(_2\)O\(_3\) to the nano size during the curing process of epoxy composites was reported by Mohanty et al \([32]\).

The size transformations mechanisms of micron sized into nano size Al\(_2\)O\(_3\) occurs in three stages (figure 7). In the first stage, as received micron size particles were added to the epoxy matrix. In the second stage, the
mixture of alumina and epoxy mixture was subjected to sonication and ball milling process. Here, the excessive mechanical energy brakes the clustered micro size particles in the epoxy matrix. In this second stage, the epoxy molecules get the path to enter in to and around the particles. In the third stage, the curing of epoxy starts. This creates a high compressive environment and the plastic strain of the composite starts to occur and ends with the permanent fragmentation of the Al₂O₃ particles. In this stage, the micron size Al₂O₃ particles were permanently converted into the nano size. The presence of such nano size of Al₂O₃ particles in the matrix of the epoxy is presented in Figure 8. The mean size of Al₂O₃ particles was found to be in the range of 45 to 81 nm.

3.2. Vickers micro hardness (Hᵥ) testing
The surface hardness of the neat epoxy and epoxy based nanocomposite specimens were measured through the Vickers micro hardness (Hᵥ) test setup to understand the effect of Al₂O₃ and GnP fillers on the epoxy matrix. Figure 9 shows the effect of various filler contents on the hardness value of epoxy based nanocomposites. It is evidenced, the hardness value of specimens was enhanced with the increasing of filler content. The higher micro hardness is exhibited by EPA1G0.5 and EPA2G0.5 composite specimens when compared to the neat epoxy. From this result, it is also evident that the highest micro hardness value was achieved by the EPA2G0.5 specimen with an increment up to 28.2 Hᵥ, i.e. 47.64% higher than that of the EP specimen (19.1 Hᵥ). The increase in the percentage of filler loading above 2 wt% of alumina and graphene nanoplatelets (varying from 0.1, 0.25, 0.5 and 1 wt%) has decreased the micro hardness value compared to the EPA2G0.1, EPA2G0.25, EPA2G0.5 and EPA2G1 specimens.

In most cases, the increased amount of the filler content could increase the micro hardness value of the epoxy composite up to a threshold value [33]. However, in this case, the higher filler contents has decreased the micro hardness value. This is due to the effect of weakness in the bond between the epoxy resin matrix and Al₂O₃/GnP fillers, which is created by the agglomeration of nano particles. Campo et al [25] found that as the filler content is increased, the micro hardness values is slightly decreased. This due to the defects formed during the preparation of the composites such as voids, non-uniform dispersion of nanoparticles in the epoxy resin matrix [27, 33].
Figure 10 shows the depth profile images of the indentation caused during the hardness test of the neat epoxy and epoxy nanocomposites samples. It is observed that the penetration depth of EPA2G0.5 is lower than the EP. This is due to the incorporation of Al₂O₃ and GnP particles in the epoxy which restrict the plastic deformation during the indentation. This is because of the interaction of Al with the –OH groups of the epoxy has leads to the efficient dispersion of the nano particles [8]. This has increased the high bonding strength between the particles and the epoxy matrix. Due to this, Al₂O₃/GnP reinforced epoxy nanocomposites resisted the penetration and the scale of the indentation sign on the surface that stressed higher hardness in EPA2G0.5 sample. The indentation mark of EPA3G0.5 is slightly higher when compared with the EPA2G0.5 sample. This is due to the limited agglomeration at higher filler concentration and due to such effect, the results were decreased [34].

Figure 11 shows the SEM images of after the Vickers micro hardness indentation of the epoxy and epoxy nanocomposites samples. During the Vickers micro hardness indentation process, the depth penetration of EPA2G0.5 is lower than of the EP sample. This is due to the incorporation of Al₂O₃ and GnP particles in the epoxy matrix. The figure 11(d) has more voids in EPA3G0.5 sample compared with EP sample, this is due to the poor bonding between the fillers (Al₂O₃ and GnP) and matrix at a higher filler concentration loaded in this composite. Due to this voids, the micro hardness values were decreased for the higher weight percentage of fillers.

3.3. Wear properties of epoxy nanocomposites

The average CoF value of neat epoxy and epoxy nanocomposites as a function of sliding distance (2000 m) at the applied load (15 N) is shown in figure 12. The average coefficient of friction value of the epoxy resin matrix was decreased due to the addition of filler particles of alumina and graphene nanoplatelets. Furthermore, it was clear that by increasing the filler particles in the epoxy resin matrix, the values of the average coefficient of friction were decreased.

It can be seen that the EPA1G0.5 and EPA2G0.5 composite specimens showed a low friction coefficient when compared with EP specimen. From the results, it is evident that the lowest coefficient of friction value was achieved for the EPA2G0.5 specimen (0.501), which is 18.3% lower than that of the EP specimen (0.613). The increase in the percentage of filler loading above 2 wt% of alumina with graphene nanoplatelets (varying from 0.1, 0.25, 0.5 and 1 wt%) has increased the average coefficient of friction when compared to the EPA2G0.1, EPA2G0.25, EPA2G0.5 and EPA2G1 samples.

Figure 13 shows the specific wear rate of neat epoxy and epoxy based nanocomposite specimens at an applied load of 15 N and sliding distance of 2000 m. There is a decreasing trend in the specific wear rate (k) values of composite specimens were noticed. It is specifying that the filler reinforcements (Al₂O₃ and GnP) have a positive impact on the wear properties of composite specimens. Among the various epoxy composites, EPA1G0.5 and EPA2G0.5 composite specimens showed the lowest specific wear rate (k) compared to the EP specimen. From the figure 13, it is also evident that the lowest specific wear rate (k) value was achieved by the EPA2G0.5 specimen ($4.64 \times 10^{-6} \text{ mm}^3 \text{ Nm}^{-1}$), which is about 76.92% lower than that of the EP specimen ($193.45 \times 10^{-6} \text{ mm}^3 \text{ Nm}^{-1}$). The increase in the percentage of filler loading above 2 wt% of alumina with graphene nanoplatelets (varying from 0.1, 0.25, 0.5 and 1 wt%) has increased the specific wear rate when compared to the
Figure 10. Indentations images of Vickers micro hardness indenter of the neat epoxy and epoxy nanocomposites samples (applied load of 100 gf) with magnification of 40×. (a) EP, (b) EPA1G0.5, (c) EPA2G0.5 and (d) EPA3G0.5.
Figure 11. SEM images of after Vickers micro hardness indenter of the neat epoxy and epoxy nanocomposites samples: (a) EP, (b) EPA1G0.5, (c) EPA2G0.5 and (d) EPA3G0.5.
EPA2G0.1, EPA2G0.25, EPA2G0.5 and EPA2G1 samples. This is due to the higher filler content added in the EPA3G0.1, EPA3G0.25, EPA3G0.5 and EPA3G1 specimens.

The reason for the low CoF and low wear rate values are predominantly due to the self-lubricating effect of the GnP. The presence of GnP has induced the contact surface during the sliding. This lubricant film was the main reason for the improvement in the CoF and wear rate. Whereas, the higher CoF and wear rate values due to the higher filler concentration. The wear debris were formed during the wear test. This is due to the poor bonding strength between the fillers and matrix, the material has easily removed debris from the epoxy matrix, this debris acts like a third body and sticks to the contact surface. This debris has enhanced the higher coefficient of friction and wear rate for the higher filler weight percentage reinforced epoxy nanocomposites.

Figure 14 shows the photographic image of the wear tracks for EP, EPA2G0.5 and EPA3G1 specimens. It can be seen that the surface texture of the disc (counterpart material) was changed due to the composite pin specimen wrapped with the disc for the specific applied load. From figure 14(a) it was observed that higher wear was wrapped in the counterpart disc. It is due to the formation of tiny brittle fractured particles of the epoxy matrix at the contact surface of the pin material. From figure 14(b), it was observed that the lower wear was wrapped in the counterpart disc when compared to figure 14(a). This is due to the lubricating effect of the GnP in the epoxy matrix. Again from the figure 14(c) it is observed that the higher wear is wrapped in the counterpart disc when compared to the figure 14(b), it is due to the third body mechanism in which the chances of sticking of the debris to the counter metallic plate is more prominent.
For an understanding of the improvement of the wear properties in the neat epoxy and particle reinforced epoxy nanocomposites, the worn faces in the immediate vicinity of the sliding surfaces of the polymer samples were examined by SEM for the specimens. Figure 15 shows the worn surface of the epoxy and the epoxy based nanocomposite specimens containing 0.5 wt% GnP along with 1, 2 and 3 wt% Al$_2$O$_3$ particles. It can be seen that the worn surfaces of EP specimen are rough along with the localized shining patches. This is specifying the brittle failure of the worn surface (figure 15(a)). It can be assumed that the worn surface toughness of this specimen is poor [8]. The worn surfaces of the EPA1G0.5 specimen (figure 15(b)) are having more peaks and valleys than that of EP specimen (figure 15(a)). This shows that the toughness value was moderately improved. Figure 15(c) is showing the wrinkled structure along with distinguished peaks and valleys in the worn surfaces of EPA2G0.5 samples. This shows that the toughness value was optimised. Figure 15(d) shows the rough surfaces along with the presence of the debris. This shows that the worn properties are controlled not only by the toughness of the material but also by the third body, called debris, plays a vital role for the determination of wear property of such type of materials.

Figure 16 shows the roughness value (Ra) of the worn surfaces of the neat epoxy (figure 16(a)) and epoxy nanocomposites which were obtained by the AFM instrument. The worn surface roughness improvement of the nanocomposites is the resistance to surface shearing due to the incorporation of Al$_2$O$_3$ and GnP particles. When
the Al$_2$O$_3$ and GnP incorporated with the epoxy, the Ra of the epoxy nanocomposites decreased. The Ra value for the EPA1G0.5 (figure 16(b)) is 32.61% lower than the neat epoxy samples whereas the maximum reduction of the surface roughness of 40.53% was observed for the EPA2G0.5 samples (figure 16(c)) in comparison to the

Figure 15: SEM images of the worn-out surfaces of (a) EP, (b) EPA1G0.5, (c) EPA2G0.5 and (d) EPA3G0.5.
neat epoxy samples. After this threshold value, the surface roughness started to increase for the EPA3G0.5 (figure 16(d)) samples by 30.49%. Thus, it can be concluded that the AFM results are in agreement with the SEM images.

Figure 16. AFM (3D) images of the worn-out surface topography of (a) EP, (b) EPA1G0.5, (c) EPA2G0.5 and (d) EPA3G0.5.
4. Conclusions

The tribological properties of neat epoxy and epoxy nanocomposites reinforced with Al$_2$O$_3$ and GnP particles were studied in this work.

- The Al$_2$O$_3$ and GnP particles at different mixing ratios were successfully dispersed in the epoxy resin matrix by sonication and planetary ball milling.
- The micro hardness value of composite specimens was increased by the inclusion of filler particles in the epoxy resin matrix. It was observed that the highest micro hardness value was obtained for the EPA2G0.5 specimen (28.2 H$_{v}$), which is about ~47.64% higher than that of the neat epoxy specimen (19.1 H$_{v}$).
- The average friction coefficient and specific wear rate (k) of epoxy composite specimens was decreased by the addition of filler particles in the epoxy resin matrix.
- It was observed that, the lowest friction coefficient and specific wear rate of EPA2G0.5 specimen (0.501 and $44.64 \times 10^{-6}$ mm$^3$ Nm$^{-1}$) is lower than that of the neat epoxy specimen (0.613 and $193.45 \times 10^{-6}$ mm$^3$ Nm$^{-1}$).
- The worn surfaces of neat epoxy and epoxy nanocomposites specimens was successfully examined by SEM. The worn surfaces of composite specimens shown the more the effect of Al$_2$O$_3$ and GnP particles are reducing the brittleness of the epoxy resin matrix.

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