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Tribological studies of epoxy and its composite coatings on steel in dry and lubricated sliding

V. Kumar¹, S. K. Sinha^{*2} and A. K. Agarwal¹

Industrial lubricants are invariably used with additives (with high sulfur and phosphorous contents) for tribological performance enhancement. However, these additives are environmentally very harmful. Hence, there is an urgent need to find alternate solutions for enhancing the tribological performance of lubricants and components without the use of harmful additives. The objective of this work is to investigate the feasibility of using polymer composite coatings in enhancing the tribological properties of steel surfaces in dry and base oil lubricated conditions. Pure epoxy and its composite (with 10 wt-% of graphene or graphite powder) films were coated onto steel substrates and tested under dry and base oil lubricated conditions. Friction and wear experiments were conducted on a ball on cylinder tribometer between polymer/composite coated cylindrical steel surface (shaft) and an uncoated steel ball as the counterface. Tests were conducted at various normal loads and speeds. In dry condition at 3 N load and 0.63 m s^{-1} sliding speed, the wear life of epoxy was increased by five times and coefficient of friction was nearly the same (0.18) on inclusion of graphene nanoparticle. In lubricated case, epoxy/graphene composite coating performed eight times and more than five times better than pure epoxy and epoxy/graphite respectively.

Keywords: Epoxy, Graphene, Graphite, Lubrication

Introduction

Because of environmental concerns and government regulations in various countries, there are strong efforts made towards reducing the use of harmful lubricating additives, which otherwise form efficient boundary lubricant on steel surfaces.¹ This important societal problem could be solved either by replacing the current additives with environmentally benign formulations or by providing a coating on the interacting surfaces, which can reduce friction and prevent wear of the substrate (mostly steels). Epoxies are thermoset polymers having one or more active epoxide group. Epoxy polymers have excellent chemical resistance, adhesion, durability at high and low temperatures, good electrical resistance, mechanical properties (high strength and toughness), and low shrinkage.^{2–6} Therefore, epoxies are used in various applications such as protective coatings for appliances, automotive primers, pipes, encapsulation of electrical and electronic instrument, adhesives, bonding materials for dental usages, replacement of welding and riveting in aircraft and automobile, matrix material for composites in aerospace industry, printed circuitry, pressure

vessel and pipes, and construction usages such as flooring, paving and airport runway repair.^{2,7,8} Epoxy coating can provide a good protective layer against wear if the epoxy is suitably modified for low friction and wear resistance.

In a previous study, epoxy composites with 0–60 wt-% graphite particles were coated on tin surface.⁸ The report shows that thermal conductivity increases as graphite percentage increases and reaches a constant value beyond 60 wt-% of graphite in epoxy. The wear rate and friction coefficient is minimum at 50 wt-% of graphite due to the formation of a transfer film on the steel ball, which acts as a solid lubricant. By using thermotropic liquid crystalline epoxy and graphene oxide (GO) (1–1.5 wt-%) composite, the impact strength increased by 96%, tensile strength increased by 46%, and flexural modulus increased by 48% for low percentage of GO (1 or 1.5 wt-%).⁹ The decomposition temperature (T_d) was higher by 35.8°C for 1.5 wt-% GO modified epoxy.

The multiwall carbon nanotubes at 1.5 wt-% in epoxy resin were responsible for excellent mechanical properties.¹⁰ It also exhibited low wear rate and lower coefficient of friction (COF). Increment in mechanical and tribological properties is due to the strengthening and self-lubricating properties of multiwall carbon nanotube. The epoxy composites modified with microcapsules of wax lubricant and multiwall carbon nanotubes show reduced hardness and Young's modulus, and there is a reduction in the COF, which leads to lower wear rate.¹¹ This is due to self-lubricating properties of wax and

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multiwall carbon nanotube. Some authors have also used GO, which was octadecylamine functionalised, in epoxy and found a reduction in the COF from 0.5 for neat epoxy to <0.1 for 1 wt-% addition of GO.¹² This also had $\sim 37\%$ reduction in the wear rate.

From the literature data,^{10–15} it is obvious that epoxy shows highly improved mechanical and tribological properties when mixed with appropriate filler material and made in the form of a composite. However, these studies have not tested the efficacy of epoxy and its coatings when used with lubricating oil. Hence, in the current work, we have tested graphite and graphene filled epoxy as tribological coatings on steel for dry sliding as well as in the presence of base oil lubricant without the additives. Such coatings have the potential to be used as boundary layers for the protection of steel and other metallic substrates¹⁶.

Experimental

Materials

Steel (grade EN31) cylindrical shaft (diameter = 40 mm) was used to coat epoxy and its composites of thickness 25–40 μm . The nanoparticle graphene (supplied by Reinste Nano Ventures Pvt Ltd, Noida, India) and graphite powder (supplied by Sigma Aldrich, India) of sizes $<100\text{ nm}$ and $<20\text{ }\mu\text{m}$ respectively were used to make the epoxy composite coatings. The counterface for all tribological tests was stainless steel (440C with hardness of 58 HRC) ball of 4 mm diameter. For base oil lubricated tests, SN 150 (provided by Indian Oil Corporation Limited) of Group I having viscosity index 95 was used under boundary lubrication condition.

Sample preparation

Specimen (cylindrical shaft) of EN31 of diameter $\sim 40\text{ mm}$, width $\sim 12\text{ mm}$ and roughness $\sim 0.40\text{ }\mu\text{m}$ were prepared by turning and then grinding the surface. The specimens were thoroughly cleaned with acetone and dried at room temperature. The cleaned shaft specimens were treated with oxygen plasma for 2 min using plasma cleaner (PDC 32G, Harrick plasma, NY, USA). The oxygen plasma treatment was used to remove the organic contaminants and to generate hydroxyl groups on the surface, which enhance adhesion between the substrate and coating. The epoxy (Araldite AY 103, supplied by Huntsman Advanced Material Pvt. Ltd) and hardener (HY 951 supplied by Huntsman Advanced Material Pvt. Ltd) were mixed thoroughly, and the air bubbles were removed using a vacuum pump. The cleaned steel shafts were coated with epoxy by a lab fabricated dip coating machine and then by rotating the coated shaft at 50 rev min^{-1} in order to distribute the epoxy (or composite) uniformly over the shaft. The coatings were cured initially at room temperature for 20 h followed by thermal curing at 80°C for 4 h. The coating thickness varied between ~ 25 and $35\text{ }\mu\text{m}$. The samples were then stored in a desiccator before any further use.

For the preparation of the epoxy composites, 10 wt-% of graphene or graphite particles were thoroughly mixed in liquid epoxy using stirring rod with hand for 15 min before applying coating. The air bubbles were removed using a vacuum pump.

Tensile strength of epoxy and its composites

The ASTM standard mould was prepared, and tensile specimens of pure epoxy, epoxy/graphene and epoxy/

graphite (Fig. 1) were cast. The dumbbell shaped specimen had dimensions as follows: full length = 60 mm, width at the clamp = 12 mm, thickness = 3.9 mm, gauge length = 10 mm and width near the gauge length = 3 mm. The specimens are shown in Fig. 1a; photos were taken after the specimens were tested. The tensile test (100 KN Universal Testing Machine ITW-BISS) was conducted on epoxy and epoxy composites, and the results are shown in Fig. 1b–d. The tensile strengths of epoxy, epoxy/graphene and epoxy/graphite were recorded as 40 (standard deviation, SD = 3) MPa, 34 (SD = 3) MPa and 27 (SD = 2.3) MPa respectively, and the Young's modulus of elasticity values were 815 (SD = 23) MPa for epoxy, 853 (SD = 31) MPa for epoxy/graphene and 847 (SD = 33.5) MPa for epoxy/graphite composites. These data are based on three repeated experimental values. It is observed that there is some reduction in the tensile strength after adding graphene or graphite; however, the Young's modulus is increased. It is possible that the material tends to become slightly less tough and there is poor bonding between epoxy and the graphite or graphene particles.

Hardness of epoxy and its composites

Hardness of epoxy and its composites coated on steel substrates were measured by Vickers fully automatic hardness tester (Barreiss, Germany) at 0.2 N normal load, and typical indents made on the material are shown in Fig. 2. The hardness values of epoxy, epoxy/graphene and epoxy/graphite were measured as 2.16 GPa (SD = 0.27), 2.35 MPa (SD = 0.18) and 2.33 MPa (SD = 0.14) respectively. The values reported are averages of at least three measured data. We observed a slight increase in the hardness, and hence compressive strength, of epoxy on account of adding graphene or graphite particles. High compressive strength can also give rise to high load bearing capacity during tribological interactions.

Fourier transform infrared spectroscopy characterisation

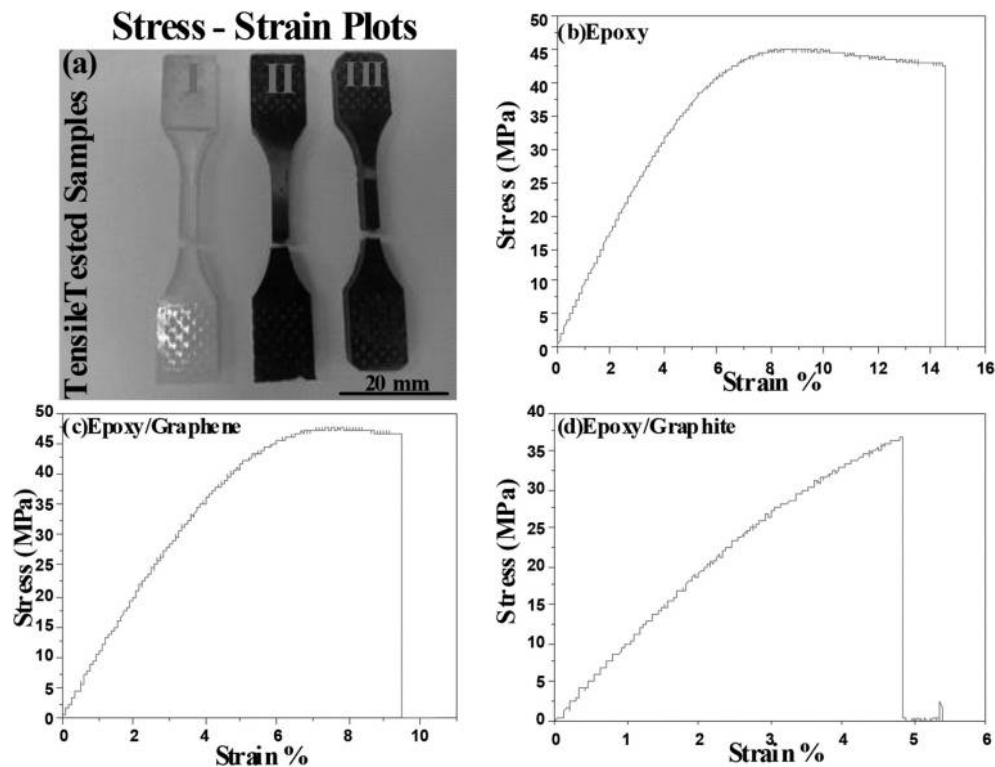
Fourier transform infrared analysis (Thermo Scientific Nicolet 8700 Research using KBr for making the pellet of nanopowder) of graphene and graphite powder was carried out for studying the chemical bonds and the functional group. Figure 3 shows the presence of OH group, which is due to the presence of moisture on the surface. There is carbon–carbon double bond in both graphene and graphite, which indicates the purity of carbon material used.

Thermogravimetric analysis

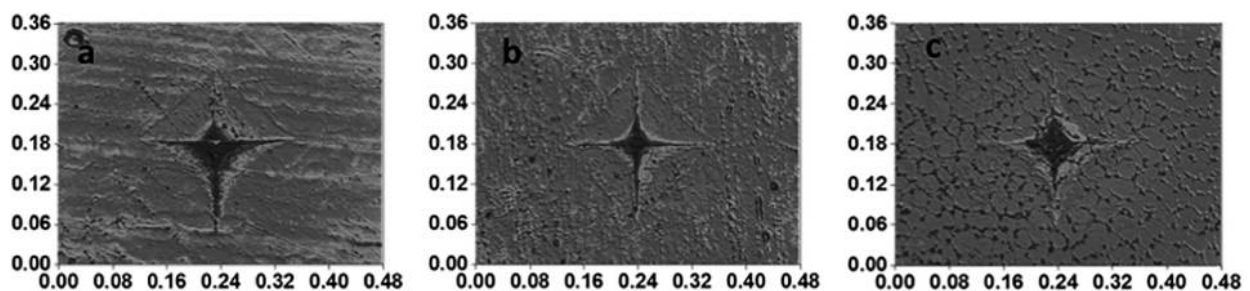
Thermogravimetric analysis (PerkinElmer Thermal Analysis) of epoxy and epoxy composites was carried out to estimate temperatures at which decomposition of the materials started. The tests were conducted by heating the specimen from 30 to 800°C at a rate of $10^\circ\text{C min}^{-1}$ and holding for 5.0 min at 800°C and then cooling from 800 to 30°C at $30^\circ\text{C min}^{-1}$. Figure 4 shows that the decomposition temperatures for epoxy and its composites are nearly 328°C .

Tribological characterisation

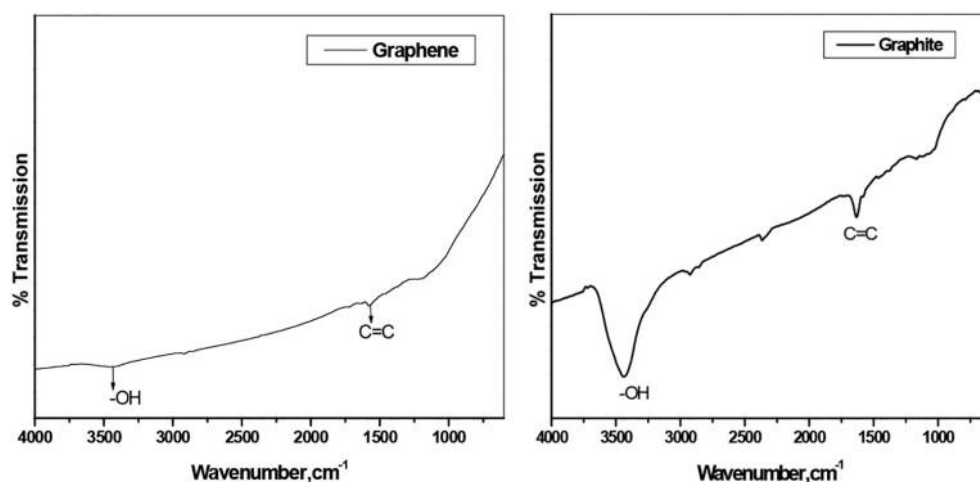
Dry and lubricated (in base oil SN 150) friction and wear tests were carried out using lab fabricated ball on



1 a tensile specimens after test (I, pure epoxy; II, epoxy/graphene; III, epoxy/graphite); stress strain curves of b pure epoxy, c epoxy/graphene and d epoxy/graphite bulk specimens as prepared according to ASTM standard



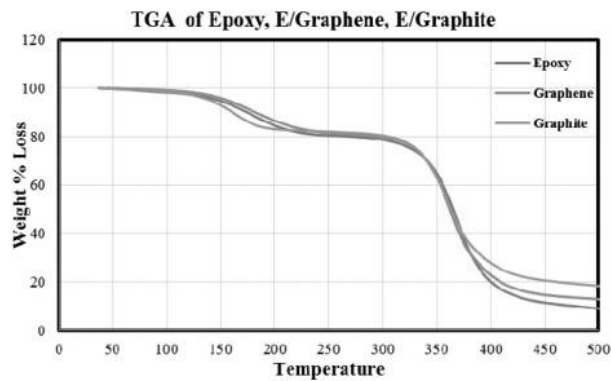
2 Indent mark on a epoxy, b epoxy/graphene and c epoxy/graphite made by microhardness tester



3 Fourier transform infrared spectroscopy analyses of graphene and graphite

cylinder tribometer set-up (Fig. 5). Full bridge strain gauges were attached to the cantilever. The cantilever was pivoted at one end and had ball holder attached at

the other end. It was able to rotate in the vertical plane but constrained in the horizontal plane. Hence, when a horizontal friction force was applied to the ball due to



4 Thermogravimetric analysis (TGA) of epoxy and its composites

shaft rotation, it deflected the cantilever, causing elastic strain in the strain gauges attached to it. The COF was measured with the help of strain measurement system (model SCAD500, Pyrodynamics Ltd, India) connected to the strain gauges, and the data were transferred to Excel spreadsheet using a data acquisition system. Steel (grade: 440C) balls of 4 mm diameter with surface roughness of 50 nm were used as counterface. There was an oil tray that could be brought below the rotating shaft such that the shaft touched the oil. The viscous nature of the oil helped it to be pulled onto the shaft as it rotated and hence lubricate the contact between the shaft and the ball. The dry tests were conducted at different normal loads (1.5, 2.5 and 3 N) and at various speeds (100, 200 and 300 rev min⁻¹ equivalent to linear speeds of 0.21, 0.42 and 0.63 m s⁻¹ respectively) for maximum number of cycles till the COF reached 0.3. Lubricated tests were carried out at normal loads of 1.5, 2.5, 3, 4 and 5 N, and at the same speeds as those for the dry tests at room temperature (25°C) and relative humidity of 45%. For the dry sliding case, the wear life for the tested conditions was taken as the number of sliding cycles for the COF to reach 0.3 or visible wear track was observed on the substrate with abnormally fluctuating friction values, whichever occurred earlier. Specific wear rate was calculated by measuring the wear track profiles using an optical surface profiler. The cross-sectional area of the wear track was measured at several points on the wear track, and the average cross-sectional area was multiplied by the track length to obtain the volume of the material removed due to wear. For the lubricated case, wear track width was measured and wear volume was calculated from wear track topographical profiles as followed for the case of dry sliding. For each set of conditions, at least three tests were conducted and average data are reported in this paper.

Three-dimensional optical profiler analysis

Wear track dimension was measured using a three-dimensional (3D) optical profiler (Bruker, USA model: Counter GTK). The laser light with interferometer principle was used in vertical scanning interferometer mode to find out track width and depth profile of the wear track. The width and depth profile dimensions were used to calculate the cross-sectional area, which was then multiplied by the total track length to obtain the wear volume. The wear rate is calculated as the specific wear rate (W_{sp}), which is given as follows¹⁷:

$$W_{sp} = \frac{V}{LD} \text{ mm}^3(\text{N m})^{-1}$$

where V is the wear volume, L is the normal load and D is the total sliding distance.

Field emission scanning electron microscopy characterisation

The surface morphologies of wear track were studied using field emission scanning electron microscopy (FE-SEM) (SIGMA, Zeiss, USA). Before FE-SEM imaging, the samples were gold coated with thickness 10 nm at 10 mA for 30 s using sputter coater (SC7620-CF, Quorum Laughton Lewes).

Results and discussion

Coatings of epoxy and its composites with film thickness $\sim 30 \mu\text{m}$ were formed on steel (EN 31) shaft. Dry and lubricated wear tests were carried out using ball on cylinder type tribometer at various conditions such as by varying the speed and normal load in normal environmental conditions. The following results were obtained.

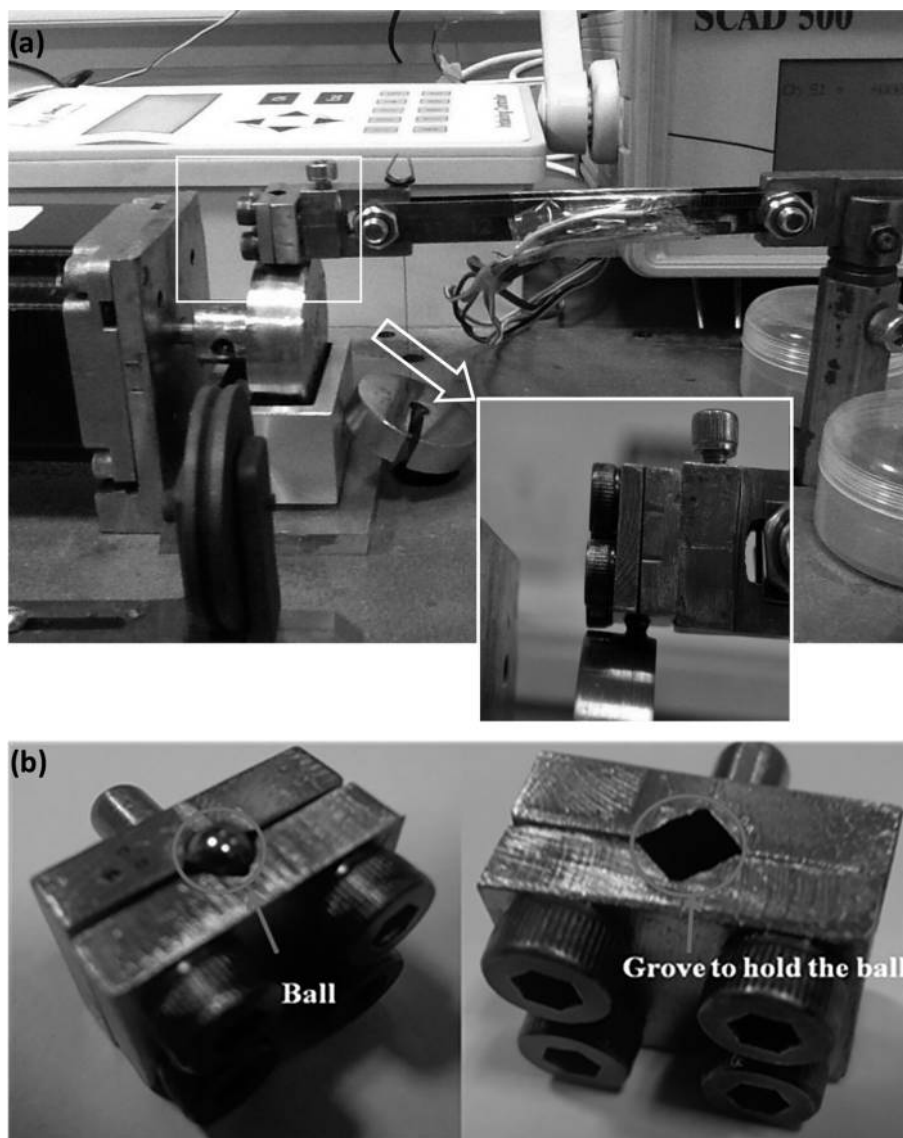
Effects of load and speed on COF

Figure 6 presents the steady state COF data at different applied normal loads for two rotational speeds of the shaft. At 0.21 m s⁻¹ (100 rev min⁻¹) speed, as load increases, the COF decreases, for epoxy from 0.16 to 0.13, for epoxy/graphene composite from 0.15 to 0.14 and for epoxy/graphite composite from 0.22 to 0.18 in dry condition as shown in Fig. 6a. In dry state, the coefficients of friction for pure epoxy and epoxy/graphene are much lower than that of epoxy/graphite coating. There is a drop in the friction with increase in the normal load. The behaviour is obvious for the low speed case. At higher loads, there is high energy dissipation at the interface, leading to heat accumulation and interfacial softening. This is the main reason for lower friction at higher normal loads. In dry condition, the samples could not be tested at a load higher than 3 N as the wear life became extremely short at higher load because of coating failure due to high friction and wear.

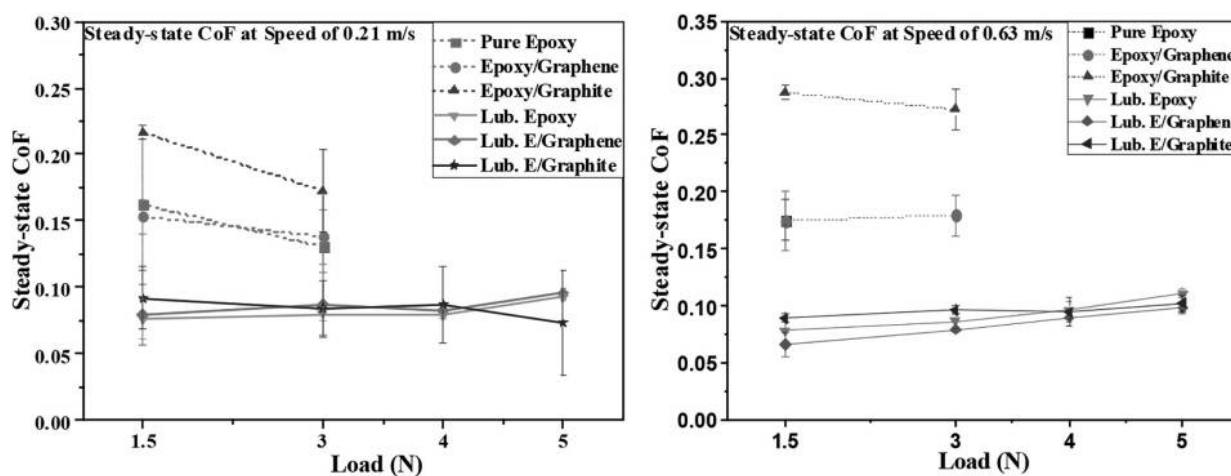
For the base oil lubricated case, the COF remains in the range of 0.07–0.1. There is a trend of increasing COF for higher normal loads for pure epoxy and epoxy/graphene composite.

Under lubricated condition, the tests were carried out up to 5 N normal load at 0.63 m s⁻¹ (300 rev min⁻¹) sliding speed, and at this condition, epoxy has shown maximum COF of 0.11 and epoxy/graphene shows the minimum value at 0.09. Epoxy/graphite had the minimum COF between 0.07 and 0.09 for all loads at 0.63 m s⁻¹ (300 rev min⁻¹) (Fig. 6b). It is important to note that under similar condition of load without the coating, the two steel surfaces provide a COF of 0.62 in dry and 0.14 in the base oil lubricated conditions.

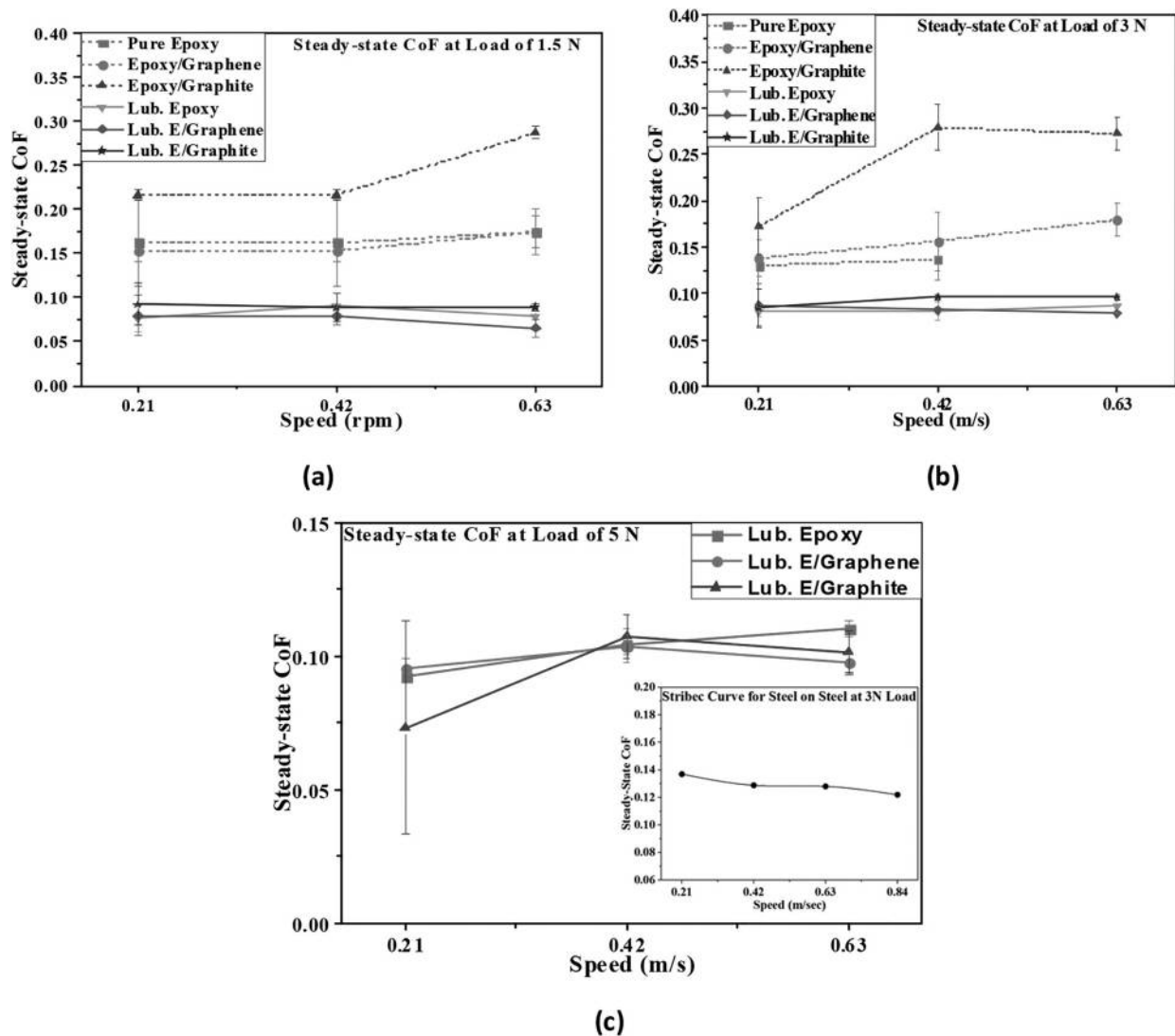
Figure 7 presents the effects of sliding speed on the steady state COF for epoxy and composites in dry and lubricated conditions at different normal loads. It is generally observed that COF is higher at higher sliding speeds. This behaviour is much more obvious for the dry case. In the presence of lubricant, the epoxy/graphene sample does not show any change in COF for the lower loads of 1.5 and 3 N. However, as shown in Fig. 7c,



5 a photograph of ball on shaft type tribometer; inset picture shows larger image of ball on shaft contact zone; b photos of ball holder with and without steel ball; ball was firmly clamped in groove with one more ball on other end of groove for uniform stress application when screws were tightened; rolling of ball was not possible in this ball holder during tribological tests



6 Variation of COF with normal load for epoxy and its composites in dry and base oil lubricated conditions at different normal loads and two sliding speeds: a 0.21 m s^{-1} (100 rev min^{-1}) and b 0.63 m s^{-1} (300 rev min^{-1})



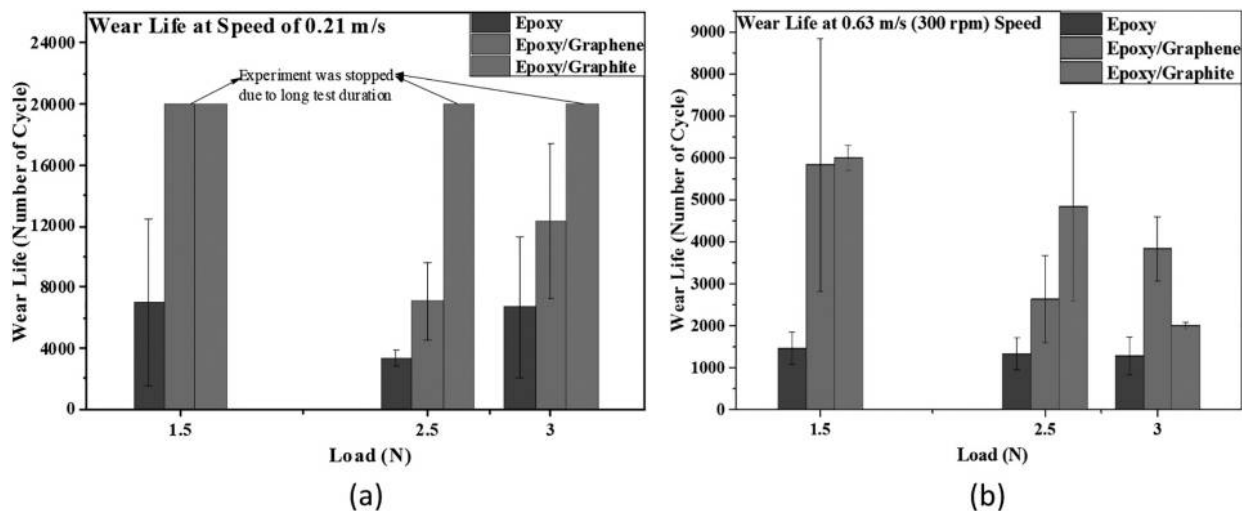
7 Steady state COF as function of sliding speed for epoxy and its composites at different load; *a* normal load = 1.5 N, *b* normal load = 3 N, *c* normal load = 5 N (data presented only for lubricated tests); plot in inset of Fig. 7c is Stribeck curve plotted for uncoated shaft against steel ball

at 5 N normal load, the COF at higher speed of 0.63 m s^{-1} is higher for all samples with epoxy/graphene, giving the lowest value of 0.082 among all. We conducted Stribeck curve plotting for bare steels (ball on shaft) sliding in the base oil lubrication, and the curve is present in the inset in Fig. 7c. The COF was measured under 3 N normal load, and the speed was varied between 0.21 and 0.84 m s^{-1} . The COF remained high in the range of 0.14–0.12, which clearly indicated that the lubrication regime for uncoated steels was also in the boundary lubrication regime.

Wear analysis

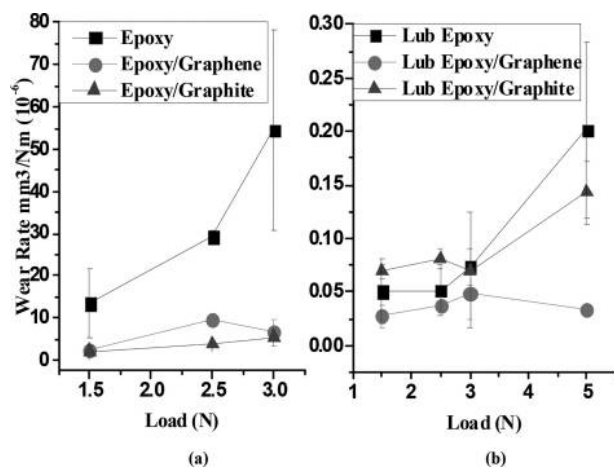
Wear lives (in terms of number of cycles) of epoxy and its composites were estimated from the COF versus number of cycle graphs using the definition of wear life as mentioned in the 'Experimental' section. Figure 8 presents two bar charts for the wear lives of pure epoxy, epoxy/graphite composite and epoxy/graphene composite at normal loads of 1.5, 2.5 and 3 N and for two shaft speeds of 0.21 m s^{-1} (100 rev min^{-1}) and 0.63 m s^{-1} (300 rev min^{-1}). It is clear from these data that pure epoxy coating is much poorer in wear life compared with

the composites at all loads and sliding speeds tested. Further, we find that epoxy/graphite coating gives maximum wear life calculated based on the definition of failure when $\mu \geq 0.3$. The COF remained consistent, though on the higher side compared with that for pure epoxy or epoxy/graphene coating. For the 0.21 m s^{-1} (100 rev min^{-1}) test (Fig. 8a) case, the epoxy/graphite coating had not failed at 20 000 cycles in all repeated tests and the experiments were stopped due to long test duration. Similarly, epoxy/graphene coating had not failed for the lowest load and speed. In these cases, the experiments were stopped at 20 000 cycles due to long test duration. At 0.63 m s^{-1} (300 rev min^{-1}) sliding speed, all samples failed but epoxy/graphite coating had the maximum life at lower load. At the highest load of 3 N and 0.63 m s^{-1} speed, the epoxy/graphite coating furnishes COF higher than 0.3 and was considered to have failed at much lower number of cycles (according to COF as the failure criterion), even though the coating was still intact on the steel shaft. Therefore, in order to further understand the wear behaviour of the coatings, we conducted wear volume measurements, and the analysis is presented below.



8 Wear life as function of normal load for two sliding speeds, *a* 0.21 m s^{-1} (100 rev min^{-1}) and *b* 0.63 m s^{-1} (300 rev min^{-1}), for pure epoxy and composites in dry condition

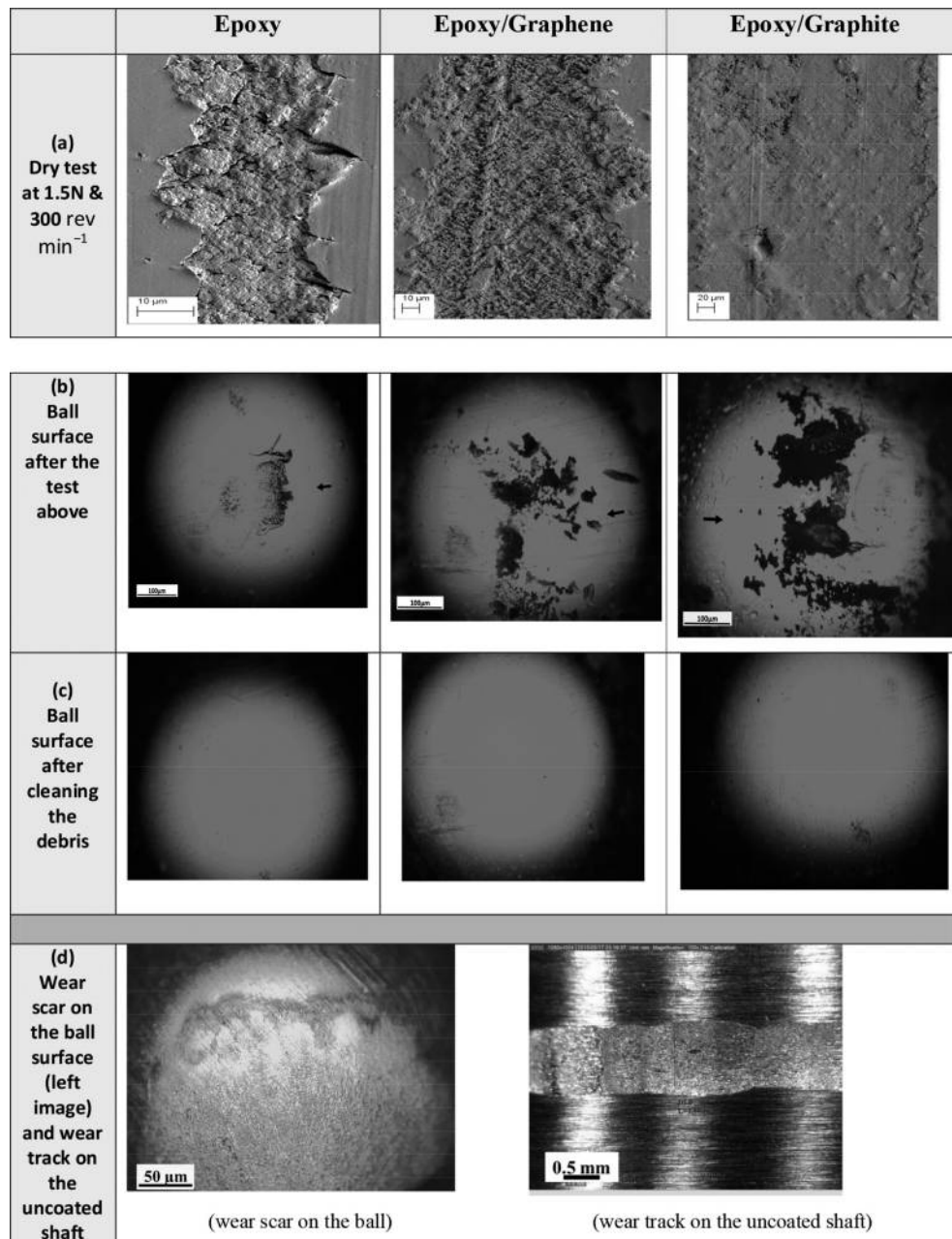
The wear volume of the material removed was computed using the topographical profiles of the wear tracks measured using the 3D surface profiler. The data are presented in Fig. 9 for dry and lubricated (with base oil SN150) sliding. It is to be noted that in the presence of lubricant, the COF remained much below 0.3, and hence, the COF based failure criterion was not applicable. Specific wear rate [$\text{mm}^3 (\text{N m})^{-1}$] is plotted as a function of normal load for 0.63 m s^{-1} (300 rev min^{-1}) sliding speed. The wear rate of epoxy coating in dry condition is several times larger than those for epoxy/graphene and epoxy/graphite composites. Wear rates for both the composites remain nearly independent of the load with epoxy/graphene, giving slightly higher value than epoxy/graphite. Though the epoxy/graphene composite shows a slight maxima in the wear rate at 2.5 N load, it might be considered within the experimental error due to sample variations. In the lubricated condition, epoxy/graphene gives the lowest wear rate, which is much more evident at the higher load of 5 N. At this load, pure epoxy gives eight times higher and epoxy/graphite gives five times higher wear rate than that of epoxy/graphene. Hence, it may be concluded that the epoxy/graphene composite is a very suitable coating for



9 Specific wear rate as function of normal loads at 0.63 m s^{-1} sliding speed for *a* dry and *b* lubricated interfaces for pure epoxy and composites

protecting the steel substrate in base oil lubricated condition. The wear marks on the coatings and steel ball surface after sliding for 15 000 cycles in dry condition and after 50 000 cycles in lubricated condition are shown in Figs. 10 and 11 respectively. The wear tracks for dry conditions are wider with considerable material loss (Fig. 10*a*). It is noted that epoxy gives less width of the track, but the depth is much larger than the other two surfaces, giving very high wear volume. Hence, the actual wear rate for pure epoxy is higher in comparison with those of the composites. The wear marks on the composite surfaces are shallower and wider, and do not change much with load, keeping the wear rate constant with load. Figure 10*b* and *c* presents images of the counterface steel ball immediately after the test and after cleaning the debris from the ball surface respectively. Wear debris is seen on the surface of the ball for each surface after 1500 cycles. After cleaning the ball of the debris material, we do not see any wear mark or scratch on the ball surface, which confirms the protective nature of the coating for the substrate as well as the counterface. Compared to these coated specimens, the uncoated shaft (Fig. 10*d*) suffers severe wear, which is also the case with ball counterface after sliding for 15 000 cycles at a normal load of 1.5 N and speed of 0.63 m s^{-1} . The wear track width on the uncoated steel shaft is $\sim 1 \text{ mm}$, and the wear scar on the ball is $\sim 250\text{--}300 \mu\text{m}$ in diameter. The steady state COF for uncoated steel shaft against steel ball in dry condition was measured as 0.6 ($\text{SD}=0.035$).

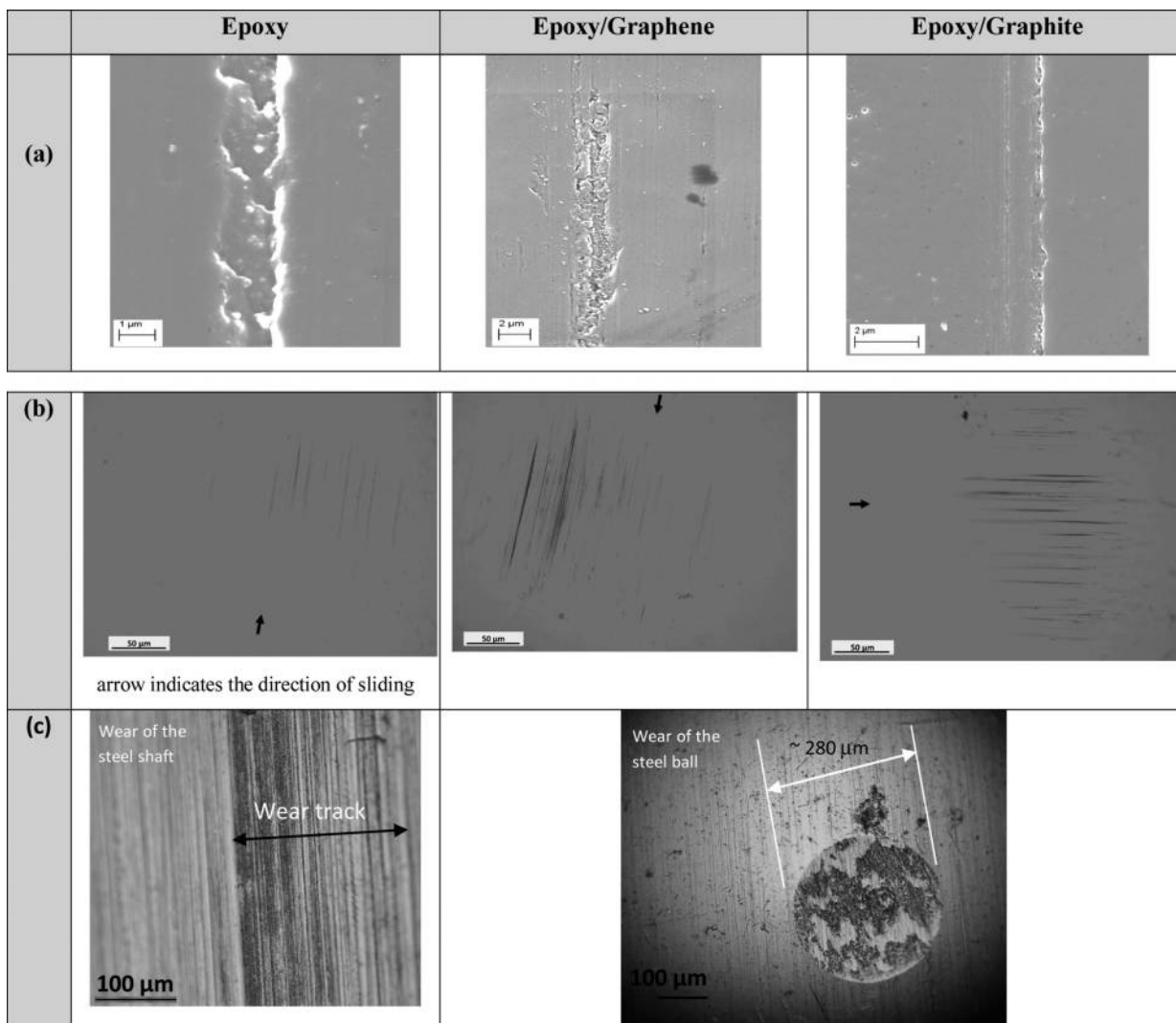
Figure 11 shows images of the wear tracks and the respective ball counterfaces for the coated and bare steel shafts after sliding tests in the presence of base oil lubricant. The images were taken after 50 000 sliding cycles as the amount of wear was very slow for the lubricated case. Epoxy suffers greatest wear among the three coatings. The wear depth is much larger for pure epoxy, whereas epoxy/graphene and epoxy/graphite surfaces show very faint plastically deformed lines. As was presented in Fig. 9*b*, epoxy/graphene gives lowest wear rate in the presence of lubricating oil. All coatings tested with base oil lubricant did not fail until 10^5 sliding cycles and the tests had to be terminated due to long test duration. After 50 000 sliding cycles, the counterface ball



10 *a* SEM images of wear tracks formed on epoxy, epoxy/graphene and epoxy/graphite specimens slid at normal load of 1.5 N and sliding speed of 0.21 m s⁻¹ (300 rev min⁻¹) against steel ball counterface after 15 000 cycles in dry condition; *b* optical images of respective ball surfaces after tests showing debris; *c* same ball surface after cleaning debris; *d* wear scar on ball surface (left image) and wear track formed on uncoated steel shaft at 1.5 N normal load and sliding speed of 0.63 m s⁻¹ (300 rev min⁻¹) tested in dry condition

was also imaged for each test (Fig. 11*b*). Some minor scratches were observed on the ball surfaces, which seemed to be more in number for the ball slid against epoxy/graphene surface. This is because of the slightly higher hardness value for epoxy/graphene in comparison to that for pure epoxy and epoxy/graphite surfaces. Scratches may also be created because of the reflow of the debris or contaminants in the oil to the interface. Figure 11*c* shows images of the wear track on uncoated shaft and the corresponding wear scar on the ball after 50 000 cycles of sliding in lubricated condition. There is ~200 µm wide wear track mark on the steel shaft surface and ~280 µm diameter wear scar on the ball surface. This shows that bare steels wear heavily even in

the presence of liquid lubricant in the boundary lubrication regime because of high friction (~0.14–0.12) (COF data shown in Fig. 7*c*, inset). All sliding components suffer from the problem of wear failure in the boundary lubrication regime. Hence, the present results show that the epoxy based composites can provide excellent wear protection with much lower COF when compared with bare steel interactions in the presence of base lubricant. It is to be noted that the steady state COF for uncoated steel shaft against ball was 0.14–0.12 (SD=0.06) in the presence of base lubricant for all loads and speeds. For all the coated specimens, the COF in the lubricated condition was mostly <0.1, with epoxy/graphene coating giving the lowest COF value of 0.07.



11 **a** SEM images of wear tracks on coated samples after sliding for 50 000 cycles in the presence of base oil lubrication at normal load of 5 N and sliding speed of 0.63 m s^{-1} (300 rev min^{-1}); **b** images of counterface steel ball for respective wear tracks on coated surfaces shown above in **a**; **c** left image: wear track on uncoated steel shaft after sliding against steel counterface ball for 50 000 cycles at 5 N normal load and 0.21 m s^{-1} (300 rev min^{-1}) sliding speed in base oil lubricated condition; right image: corresponding wear scar on the ball surface

Conclusion

Epoxy and epoxy composites (with 10 wt-% graphene and graphite particles) were coated on steel shaft surfaces and tested for mechanical and tribological properties in dry and base oil lubricated conditions. The counterface used for the tribological tests was 4 mm diameter steel (grade 440C) steel. The normal load and sliding speed were varied during the tribological tests. The following conclusions are drawn based on the present study:

1. Filler particles of graphene and graphite in epoxy coating improve the mechanical properties such that Young's modulus increased by 14% and hardness increased by 13% for epoxy/graphene and 22% and 12% for epoxy/graphite respectively.
2. The coefficients of friction in dry sliding for epoxy and epoxy/graphene are nearly the same at 0.18, but for epoxy/graphite, it is 0.2 when tested at 3 N normal load and 0.63 m s^{-1} (300 rev min^{-1}) sliding speed.
3. Wear life (based on the definition of failure when $\mu \geq 0.3$ or μ was fluctuating) for pure epoxy was increased by 5 times on addition of graphene and 10 times on addition of graphite at the severe condition of 3 N normal load and 0.63 m s^{-1} speed under dry sliding condition test.
4. Based on the volumetric wear rate, epoxy/graphene shows specific wear rate of $6.6 \times 10^{-6} \text{ mm}^3 (\text{N m})^{-1}$ and epoxy/graphite $5.5 \times 10^{-6} \text{ mm}^3 (\text{N m})^{-1}$, whereas for pure epoxy, it is $54.6 \times 10^{-6} \text{ mm}^3 (\text{N m})^{-1}$ at 3 N load and 0.63 m s^{-1} speed under dry condition.
5. Under base oil (SN150) lubricated condition, the steady state COF for epoxy/graphene varied between 0.07 and 0.09 and showed the least amount of wear. All coatings gave friction values lower than that for bare steel shaft against steel ball, which was 0.14–0.12 even in the presence of lubricant.
6. Under lubricated condition, the wear specific wear for epoxy/graphene was lowest at $0.025 \times 10^{-6} \text{ mm}^3 (\text{N m})^{-1}$, giving only minor scratches to the counterface ball. Severe wear to both the uncoated steel shaft and ball was observed when tested in all conditions.

Finally, we conclude that epoxy/graphene is a potential coating for steel surfaces, which can give low friction and long wear life under base oil lubricated condition.

Acknowledgements

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References

1. E Ng, N Satyanarayana, SK Sinha, C Lim and Z Zheng: 'Tribological performances of ZDDP and ashless triphenyl phosphorothionate (TPPT) as lubricant additives on Ti-N and Ti-Al-N coated steel surfaces', *Tribology*, 2014, **8**, (4), 209–221.
2. JI DiStasio: 'Epoxy resin technology: development since', 1979, Hoboken, NJ, Noyes Data Corp.
3. B Bilyeu, W Brostow and KP Menard: 'Epoxy thermosets and their applications I: chemical structures and applications', *J. Mater. Ed.*, 1999, **21**, 281–286.
4. CA May: 'Epoxy resins: chemistry and technology', 1988, New York, Marcel Dekker.
5. B Bilyeu, W Brostow and KP Menard: 'Determination of volume changes during cure via void elimination and shrinkage of an epoxy prepreg using a quartz dilatometry cell', *Polimery*, 2001, **46**, 799–802.
6. B Bilyeu, W Brostow and KP Menard: 'Epoxy thermosets and their applications II. Thermal analysis', *J. Mater. Ed.*, 2000, **22**, 107–130.
7. XM Chen and B Ellis: 'Coatings and other applications of epoxy resins', in 'Chemistry and technology of epoxy resins', (ed. B. Ellis), 303–325; 1993, New York, Chapman & Hall.
8. G Pan, Q Guo, J Ding, W Zhang and X Wang: 'Tribological behaviors of graphite/epoxy two-phase composite coatings', *Tribol. Int.*, 2010, **43**, 1318–1325.
9. B Qi, S R Lu, X E Xiao, LL Pan, F Z Tan and JH Yu: 'Enhanced thermal and mechanical properties of epoxy composites by mixing thermotropic liquid crystalline epoxy grafted graphene oxide', *Express Polym. Lett.*, 2014, **8**, 467–479.
10. B. Dong, Z. Yang, Y. Huang and H.-L. Li: 'Study on tribological properties of multi-walled carbon nanotubes/epoxy resin nano composites', *Tribol. Lett.*, 2005, **20**, 251–254.
11. N W Khun, H Zhang, J Yang and E Liu: 'Mechanical and tribological properties of epoxy matrix composites modified with microencapsulated mixture of wax lubricant and multi-walled carbon nanotubes', *Friction*, 2013, **4**, 341–349.
12. R. Shah, T. Datashvili, T. Cai, J. Wahrmond, B. Menard, K. P. Menard, W Brostow and J Perez: 'Effects of functionalised reduced graphene oxide on frictional and wear properties of epoxy resin', *Mater. Res. Innovation*, 2015, **19**, (2), 97–106.
13. H R Le, A Howson, M Ramanauskas and J A Williams: 'Tribological Characterisation of Air-Sprayed Epoxy-CNT Nanocomposite Coatings', *Tribol. Lett.*, 2012, **45**, 301–308.
14. W Brostow, M Dutta, P Rusek and Modified epoxy coatings on mild steel: 'Tribology and surface energy', *Eur. Polym. J.*, 2010, **46**, 2181–2189.
15. N L McCooka, D L Burrisa, G R Bournea, J Steffensa, J R Hanrahanb and W G Sawyer: 'Wear resistant solid lubricant coating made from PTFE and epoxy', *Tribol. Lett.*, 2005, **18**, 119–124.
16. M Abdul Samad and S K Sinha: 'Nanocomposite UHMWPE–CNT polymer coatings for boundary lubrication on aluminium substrates', *Tribol. Lett.*, 2010, **38**, 301–311.
17. I. M Hutchings: 'Tribology—friction and wear of engineering materials', 1992, Oxford, UK, Butterworth-Heinemann.