

Friction and wear properties of nano-Si₃N₄/nano-SiC composite under nanolubricated conditions

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Abstract: Friction and wear properties of nano-Si₃N₄/nano-SiC composite were studied under nanolubricated conditions. Mineral oil mixed with nanoparticles of diamond was used as lubricant. A friction coefficient of 0.043 and a wear coefficient of 4.2×10^{-7} were obtained for nano-Si₃N₄/nano-SiC composite under normal load of 600 N with mineral oil + 0.5 wt% nanodiamond, whereas a friction coefficient of 0.077 and a wear coefficient of 10.3×10^{-7} were obtained for nano-Si₃N₄/nano-SiC composite under normal load of 600 N with mineral oil. 3D surface profilometer was used to study the surface morphology of wear scars. Scanning electron microscopy (SEM) and X-ray diffraction (XRD) studies were conducted to illustrate reduction in friction and wear.

Keywords: nano-ceramics; nanolubrication; nano-Si₃N₄/nano-SiC; friction; wear

1 Introduction

Engineering ceramics are used for extreme conditions of load, velocity, and temperature in various applications, *viz.*, hybrid bearings, cutting tools, internal combustion engines, cam assembly, etc. [1,2]. It is well known that ceramics experience severe wear damage and high friction during unlubricated sliding contact, especially under conditions of high load, speed, and temperature [2]. Compared with other ceramic composites, nano-Si₃N₄ composites possess higher hardness and high wear resistance [1–3]. However, their poor frictional properties restrict the use of such class of ceramics in engineering applications [2]. Recently tribological studies conducted on nanodiamond particles mixed with conventional lubricants have proved effective in reduction of friction

and wear in ceramics, under boundary lubricated conditions [4,5]. Composite of nano-Si₃N₄/nano-SiC is considered as a potential candidate material for various engineering applications for near future [6]. Therefore, there is a need for improving the friction and wear properties of nano-Si₃N₄/nano-SiC composite through proper lubrication, and as such, it has become inevitable to develop appropriate lubricants as a step towards wider engineering applications of Si₃N₄ and its composites.

Extensive research has been reported in open literature for incorporation of liquid and solid lubrication in ceramics, particularly for Si₃N₄ and its composites with a goal to reduce friction and wear [7–11]. However, liquid lubricants on ceramic surfaces are less efficient, due to their poor reactivity with ceramics. Solid lubrication of ceramics involves the use of thin lubricious surface coating that is often limited by its short lifetime. Therefore, there is scope for unconventional lubrication scheme to overcome the

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difficulties of pure liquid/pure solid lubrication. Tribological studies on nanoparticles have proved that lubricants with different nanoparticles as additives are effective in reducing friction and wear for metallic tribo pairs, whereas limited work has been reported in ceramics. A number of researchers have reported that the addition of nanoparticles, such as copper oxide, molybdenum disulphide, etc., to the conventional lubricants are effective in reducing friction and wear in both metallic and ceramic tribo pairs [8–24]. Nanodiamond additives to mineral oil have already proved effective in friction and wear reduction in various engineering applications, under higher load and low velocity conditions. Therefore, its use for friction and wear reduction in the case of nano-Si₃N₄/nano-SiC composite offers opportunity to explore the nanodiamond based mineral oil through tribological tests.

The objective of the present study is to evaluate the tribological properties of a ceramic tribo pair consisting of nano-Si₃N₄/nano-SiC composite disc and Si₃N₄ ball under high load, with nanodiamond as additive to the mineral oil based lubricant at ambient temperature.

2 Experimental procedures

2.1 Sample preparation

In the present experimental studies, Si₃N₄ ball of 10 mm in diameter was rubbed against disc of nano-Si₃N₄/nano-SiC composite of 50 mm in diameter and 6 mm in thickness. The method of preparation included the formation of silicon carbide nanograins using the carbothermal reduction of SiO₂ by carbon in the Y₂O₃-SiO₂ system at sintering temperature [6,25,26]. The hardness and density of the nano-Si₃N₄/nano-SiC composite were 17 GPa and 3.06 g/cm³, respectively. The disc was polished at a uniform pressure of 2.5 MPa by 5 μm diamond paste, resulting in the mirror polished finish of the disc before the actual tribological tests were performed. Nanodiamond particles of 40–50 nm size were procured commercially from reliable source namely Intelligent Materials Pvt. Ltd. Nanoshel LLC (in collaboration). The particle size and other details of the nanodiamond, such as spherical morphology, were authenticated by X-ray diffraction (XRD) analysis provided by the supplier. Nanodiamond particles (0.5 wt%) were dispersed in the lubricant

mineral oil for about 2 h using WENSAR ultrasonicator to obtain a homogeneous mixture. Samples of ball and disc were washed in acetone in an ultrasonic cleaner for about 10 min and dried at room temperature for 15 min, before loading on the tribological test rig.

The properties of the mineral oil are shown in Table 1. There was no significant change observed in the physical and chemical properties of the mineral oil with the addition of 0.5 wt% diamond nanoparticles. The quantitative energy-dispersive X-ray spectroscopy (EDX) results of the nano-Si₃N₄/nano-SiC composite disc are given in Table 2.

Table 1 Properties of lubricant mineral oil

| | | |
|---------------------|--------|------------------------|
| Kinematic viscosity | 40 °C | 112 mm ² /s |
| | 100 °C | 13 mm ² /s |
| Viscosity index | | 128 |
| Dynamic viscosity | -10 °C | 2800 mPa·s |
| Density | 15 °C | 878 kg/m ³ |
| Flash point | | 234 °C |
| Pour point | | -29 °C |

Table 2 Quantitative EDX result of nano-Si₃N₄/nano-SiC composite

| Analyte | Result (wt%) | Standard deviation | Calculating procedure | Line | Intensity (a.u.) |
|---------|--------------|--------------------|-----------------------|-------|------------------|
| Si | 90.043 | [0.621] | Quan-FP | Si Kα | 6.2085 |
| Y | 7.230 | [0.022] | Quan-FP | Y Kα | 40.6907 |
| P | 2.413 | [0.114] | Quan-FP | P Kα | 0.0921 |
| Fe | 0.108 | [0.005] | Quan-FP | Fe Kα | 0.1453 |
| Sm | 0.004 | [0.024] | Quan-FP | Sm Kα | 0.0221 |
| Mn | 0.040 | [0.007] | Quan-FP | Mn Kα | 0.0387 |
| Cu | 0.034 | [0.004] | Quan-FP | Cu Kα | 0.0855 |
| Cr | 0.028 | [0.007] | Quan-FP | Cr Kα | 0.0199 |
| Ni | 0.015 | [0.003] | Quan-FP | Ni Kα | 0.0311 |

2.2 Test apparatus

The high speed tribological test rig, ball on disc (Fig. 1) developed by Rtec USA, is a high precision machine.

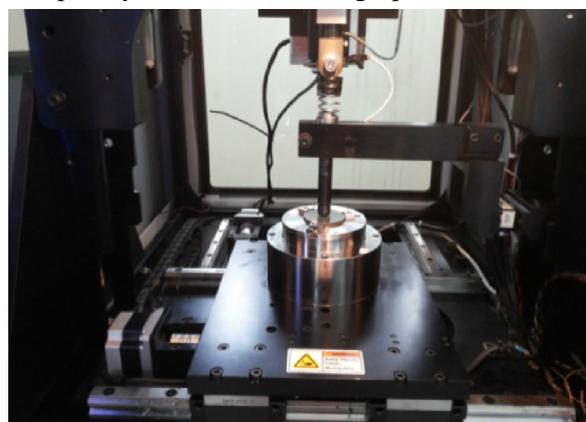


Fig. 1 High precision tribological test rig.

The specific features of the tribo test rig include high sliding speed of 50 ms⁻¹ and a maximum load of 5 kN. The test rig has a high resolution piezo load arm. The test specimen disc of 50 mm diameter was mounted on a sliding attachment on the main table which was moved in X or Y direction using a jogbox. The ball was mounted in a cylindrical assembly exactly above the specimen loaded in Z direction which was also controlled by the jogbox.

3D profilometer working on the principle of white light interferometry (WLI) was used to study the wear in detail by scanning the wear scars. Rotation of the objective turret was used to select the magnification and the sample was positioned under WLI objective using “X” and “Y” controls of the profiler jogbox. Once the sample was appropriately positioned, then “Z” stage was moved to the appropriate magnification to begin the scanning process. Schematic diagram demonstrating the scanning of the wear scars is shown in Fig. 2. Three Nikon objectives, viz., 4×, 10×, and 50× were used for the analysis of the worn surfaces. After scanning, 3D model image was generated. Scanning electron microscopy (SEM) and XRD analyses of the unworn and worn out surfaces were conducted on Hitachi 3600 at 2000× magnification and Philips XPERT-Pro using Cu Kα (λ = 1.54060 nm) with a 2θ range of 35.65°–133.35°, respectively.

2.3 Test conditions

In these experiments, tribological tests were performed

at constant normal loads of 500, 550, and 600 N and a sliding speed of 0.02 m/s with a stroke of 1 mm for 15 min at ambient temperature. Low sliding speed of 0.02 m/s was used to ensure the boundary lubrication conditions. The lubricant was supplied at the point of contact before the commencement of the test by a syringe. The samples of nano-Si₃N₄/nano-SiC composite disc were mirror polished with average surface roughness of all samples equal to 0.06 and 0.01 μm for nano-Si₃N₄/nano-SiC composite disc and Si₃N₄ ball, respectively.

3 Results and discussion

3.1 Film parameter

An important parameter that indicates the effectiveness of lubrication is the film parameter given by [27]:

$$\Lambda = \frac{h_{\min}}{\sqrt{R_{qa}^2 + R_{qb}^2}} \tag{1}$$

where h_{\min} is the minimum film thickness; R_{qa} and R_{qb} are the surface roughness of the two surfaces. The film parameter is used to define the four important lubrication regimes which include: (i) boundary lubrication, $\Lambda < 1$; (ii) partial lubrication, $1 \leq \Lambda < 3$; (iii) hydrodynamic lubrication, $3 \leq \Lambda$; (iv) elastohydrodynamic lubrication, $3 \leq \Lambda < 10$. These values are only approximate, but give useful insight

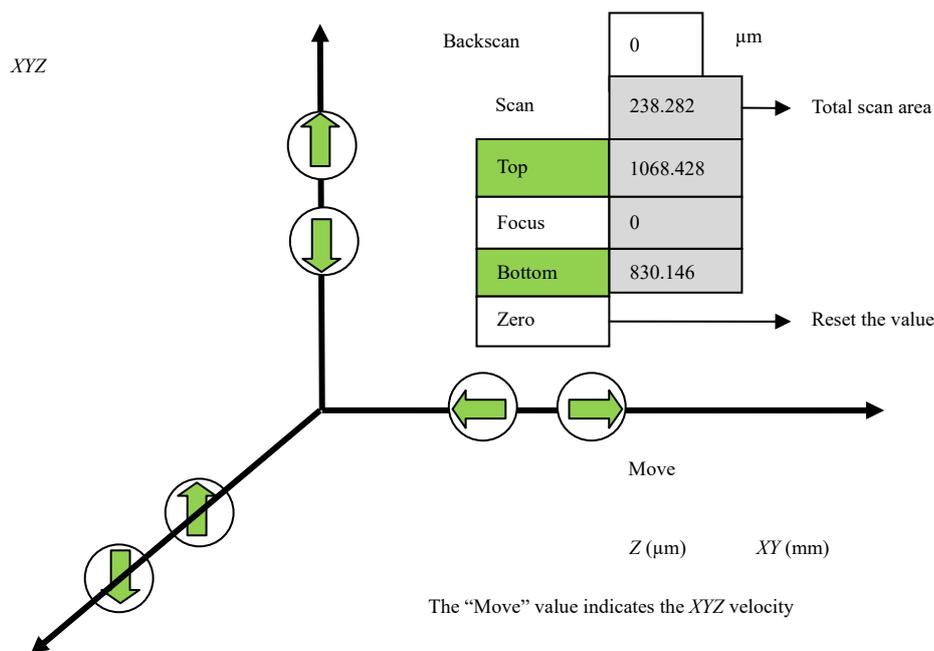


Fig. 2 Schematic diagram of scanning wear scars on 3D profilometer.

into the mechanism of lubrication. The minimum film thickness was calculated by Eq. (2) given below:

$$h_{\min} = 3.63RU_{\Sigma}^{0.68}G_{\Sigma}^{0.49}W_{\Sigma}^{-0.073}(1 - e^{-0.68k_c}) \quad (2)$$

where $U_{\Sigma} = \eta_0 u_s E^* R$; $G_{\Sigma} = \alpha_p E^*$; $W_{\Sigma} = P/(E^* R^2)$; η_0 is the dynamic viscosity (26.8×10^{-3} Pa·s); α_p is the viscosity–pressure coefficient (3.4×10^{-8} Pa $^{-1}$); R is the reduced radius of curvature of the ball (2.5 mm); E^* is the reduced Young's modulus (328.94 GPa); u_s is the entrainment speed which was constant for sliding tests (0.02 m/s); P is the normal load (500, 550, and 600 N); and k_c is the ellipticity parameter. The calculations for all the experiments using Eq. (1) and Eq. (2), gave the λ ratio well under unity which means that the lubrication occurred in the boundary lubrication regime only.

This research study was intended for applications such as cam and tappet in high speed engines, ball and race in hybrid bearings, etc., which give rise to a very high Hertzian pressure, as such, the maximum Hertzian pressure (P_{\max}) calculated in this study varied from 7.4 to 7.9 GPa at the applied normal loads of 500–600 N. Equation (3) was used to calculate the maximum Hertzian pressure, while Hertz diameter “ a ” was found to be 357.26, 368.78, and 380 μ m at normal loads of 500, 550, and 600 N, respectively.

$$P_{\max} = \frac{3P}{2\pi a^2} \quad (3)$$

where $a = \left(\frac{3RP}{2E^*}\right)^{1/3}$.

3.2 Coefficient of friction

Friction and wear behaviours observed between Si_3N_4 ball and nano- Si_3N_4 /nano-SiC composite disc in all reciprocating sliding experimental studies are shown in Figs. 3–11. All experiments were repeated three times to ensure repeatability of the results.

Coefficient of friction (COF) obtained from tribological studies for nano- Si_3N_4 /nano-SiC composite disc against Si_3N_4 ball with mineral oil and mineral oil + 0.5 wt% nanodiamond as lubricant under various normal loads are shown in Figs. 3–5. It is evident from these figures that a low COF of 0.0426 is observed under constant normal load of 600 N using mineral oil + 0.5 wt% nanodiamond. However, higher COF of 0.0773 is observed for Si_3N_4 ball and nano- Si_3N_4 /nano-SiC composite disc when lubricated with mineral oil.

The overall percentage decrease in COF is more than 40% with 0.5 wt% nanodiamond. The decrease in COF is attributed to the rolling motion of nanoparticles at the interface of tribo pair under high normal load, as these nanodiamond particles dispersed in the lubricant get entrapped between the asperities of the two surfaces in motion [4]. It is possible that at higher loads, more nanoparticles get entrapped between the asperities of the two surfaces in contact resulting in reduced COF. Surface morphological studies and surface analysis of unworn and worn out surfaces of the nano- Si_3N_4 /nano-SiC composite disc are shown in Figs. 6 and 7.

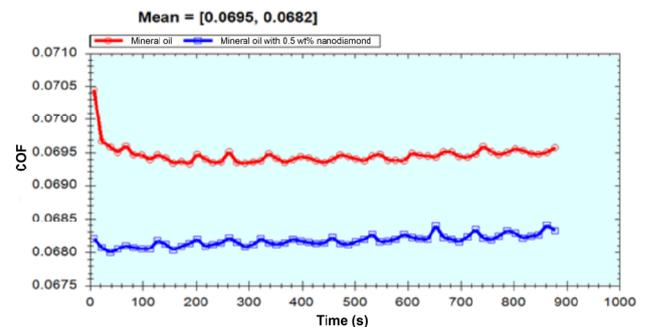


Fig. 3 Friction coefficient of mineral oil and mineral oil + 0.5 wt% nanodiamond at normal load of 500 N.

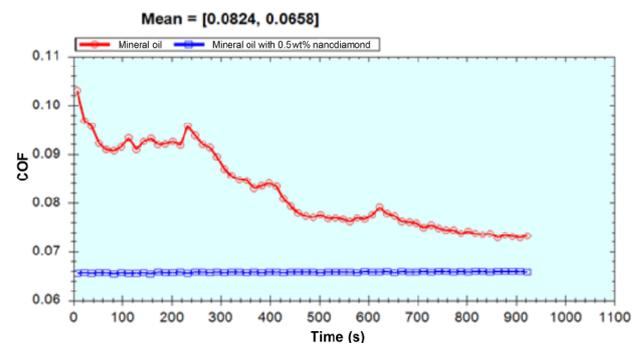


Fig. 4 Friction coefficient of mineral oil and mineral oil + 0.5 wt% nanodiamond at normal load of 550 N.

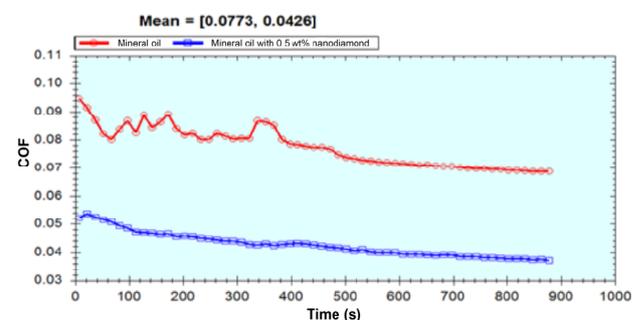


Fig. 5 Friction coefficient of mineral oil and mineral oil + 0.5 wt% nanodiamond at normal load of 600 N.

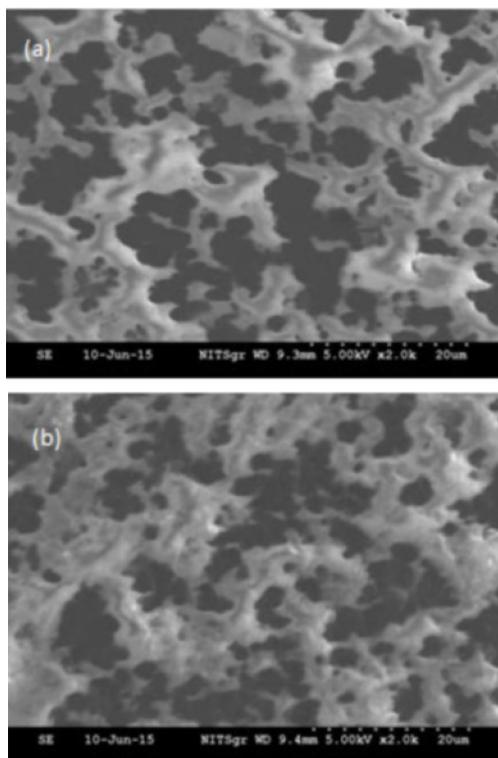


Fig. 6 SEM micrographs of nano-Si₃N₄/nano-SiC composite disc at 2000× on (a) unworn surface and (b) wear scars at 600 N load with mineral oil + 0.5 wt% nanodiamond.

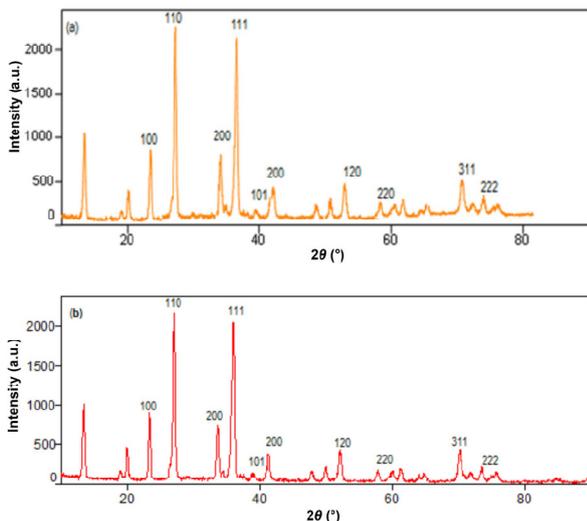


Fig. 7 XRD patterns of nano-Si₃N₄/nano-SiC composite disc on (a) unworn surface and (b) wear scars at 600 N load with mineral oil + 0.5 wt% nanodiamond.

SEM micrographs of unworn surface and worn out surface on the nano-Si₃N₄/nano-SiC composite disc at 600 N load with mineral oil + 0.5 wt% nanodiamond are shown in Figs. 6(a) and 6(b), respectively. It is evident from these micrographs that no film formation

or transfer film is present on the worn out surface. This is also supported by the XRD patterns obtained on unworn and worn out surfaces shown in Figs. 7(a) and 7(b), respectively. The XRD pattern on unworn surface is shown in Fig. 7(a), whereas Fig. 7(b) shows the XRD pattern on worn out surface of nano-Si₃N₄/nano-SiC composite disc at 600 N normal load when lubricated with mineral oil + 0.5 wt% nanodiamond. The Si₃N₄ was characterized by XRD as per JCPDS Card No. 33-1160. The peaks appeared corresponding to (100), (110), (200), (101), and (120) locating at 23°, 27°, 34.08°, 38.62°, and 52.5°. The SiC was characterized by XRD as per JCPDS Card No. 29-1129. The peaks appeared corresponding to (111), (200), (220), (311), and (222) locating at 35.65°, 41.4°, 58.18°, 71.76°, and 75.49°, respectively.

The COF observed in this research study is lower than the COF (0.6–0.7) observed between the same tribo pair for normal load of 5–15 N under dry sliding conditions as reported by Šajgalík *et al.* [6]. The counter intuitive effect of reduction of COF is attributed to rolling effect of the nanoparticles.

3.3 Wear

Wear behaviour of Si₃N₄ ball and nano-Si₃N₄/nano-SiC composite disc under lubricated conditions is shown in Fig. 8. Wear coefficient was calculated using well established Archards equation:

$$K_w = \frac{W_v H}{S_d P} \tag{4}$$

where K_w is the wear coefficient; W_v is the wear volume (mm³); H is the hardness (N/mm²); P is the normal load (N); and S_d is the sliding distance (m).

Wear coefficients of 4.2×10^{-7} and 8.1×10^{-7} for Si₃N₄ ball and nano-Si₃N₄/nano-SiC composite disc were obtained with lubricant mineral oil + 0.5 wt%

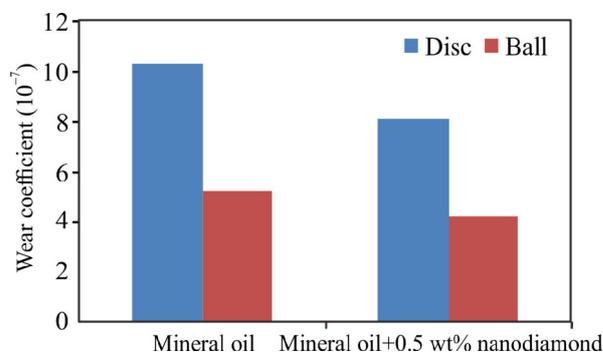


Fig. 8 Wear coefficients of Si₃N₄ ball and nano-Si₃N₄/nano-SiC composite disc at normal load of 600 N.

nanodiamond respectively, whereas wear coefficients of 5.2×10^{-7} and 10.3×10^{-7} were obtained for Si_3N_4 ball and nano- Si_3N_4 /nano-SiC composite disc respectively, using mineral oil as lubricant at normal load of 600 N. It is evident from these studies that Si_3N_4 ball shows higher wear resistance, as compared to nano- Si_3N_4 /nano-SiC disc under lubricated conditions without and with nanodiamond. Surface morphological studies of wear scar diameters of Si_3N_4 ball after sliding distance test for 15 min are shown in Figs. 9(a) and 9(b) under 600 N normal load without and with nanodiamond, respectively. It is evident from Figs. 9(a) and 9(b) that higher wear scar diameter of the order of 900 μm is observed on the Si_3N_4 ball without additive, as compared to 760 μm wear scar diameter obtained in the case of lubricant with nanodiamond. Surface profile of wear scars shows that abrasive wear is the predominant mode of wear under these conditions of load and speed. It is evident from the surface profile (Fig. 10) that dark rough patches on wear tracks are found on the Si_3N_4 ball surface when mineral oil is used. However, very smooth track is found on the ball with mineral oil + 0.5 wt% nanodiamond. This may be attributed to three body wear in the case of the Si_3N_4 ball on nano- Si_3N_4 /nano-SiC composite disc with mineral oil, whereas two body wear takes place

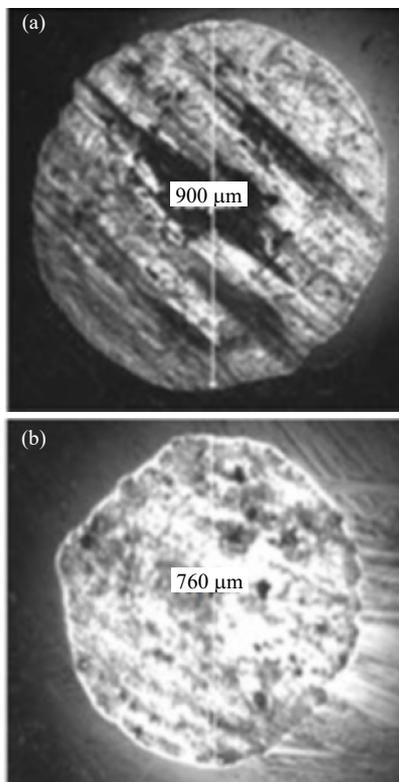


Fig. 9 Wear scar morphology of Si_3N_4 ball under (a) mineral oil and (b) mineral oil + 0.5 wt% nanodiamond.

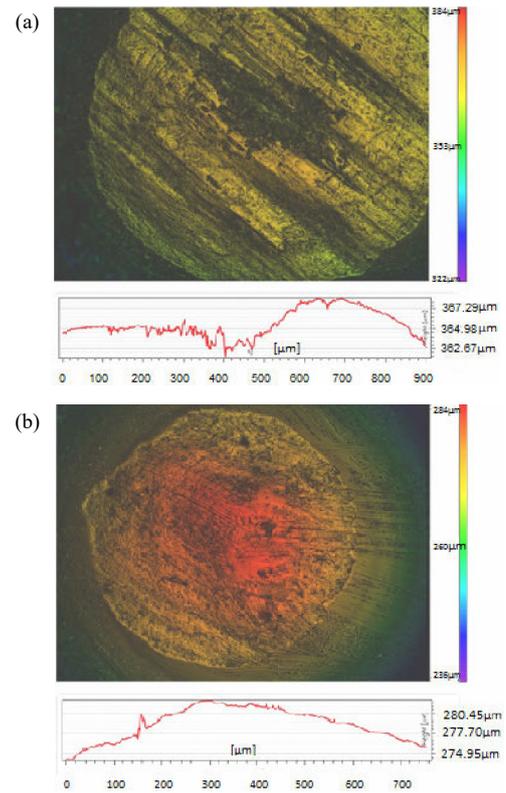


Fig. 10 Surface profile of wear scars of Si_3N_4 ball in (a) mineral oil and (b) mineral oil + 0.5 wt% nanodiamond.

between the ball and disc when mineral oil + 0.5 wt% nanodiamond is used. Figures 11(a) and 11(b) shows wear scar diameters in the case of nano- Si_3N_4 /nano-SiC composite disc without and with lubricant additive, respectively. Lower wear scars (900 μm) for the disc were obtained, as compared to wear scars (1040 μm) obtained in the case of the disc when lubricated without additive. Thus it is established through presented results that nanodiamond helps in enhancing the wear resistance under high load conditions.

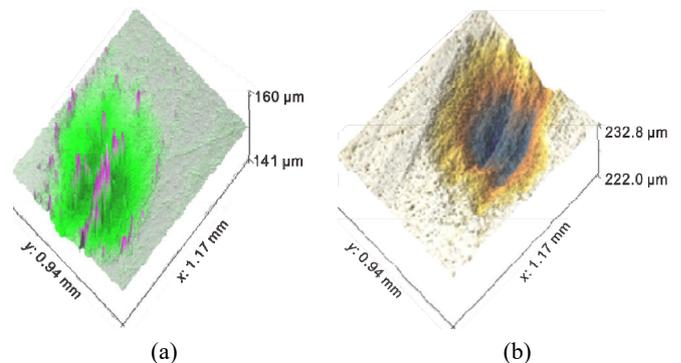


Fig. 11 3D view of wear scars on nano- Si_3N_4 /nano-SiC composite disc with (a) mineral oil and (b) mineral oil + 0.5 wt% nanodiamond.

4 Conclusions

Tribological studies were conducted on tribo test rig using ceramic tribo pair of Si₃N₄ and nano-Si₃N₄/nano-SiC composite with nanodiamond as additive to the mineral oil under reciprocating sliding test conditions. It is inferred from this research that nanodiamond has strong influence on friction and wear properties of Si₃N₄ and nano-Si₃N₄/nano-SiC composite under high normal load. Coefficient of friction decreases by more than 40% at normal load of 600 N with nanodiamond as additive to the lubricant which is also manifested by decrease in the wear coefficient. A very low wear coefficient of 4.2×10^{-7} is obtained for Si₃N₄ ball at normal load of 600 N with nanodiamond as additive to the lubricant. The wear scar diameter of nano-Si₃N₄/nano-SiC composite disc is reduced by 140 μm with nanodiamond as additive to the mineral oil. The research studies in this article reveal that nanodiamond has strong potential as lubricant additive for nano-Si₃N₄/nano-SiC composite. Further research studies need to be carried out under high load and high temperature conditions to determine the suitability of nanodiamond as an additive for high temperature applications.

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