Friction and Wear Properties of Si$_3$N$_4$/TiC Ceramic Composite under Nano Lubrication

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Abstract. The present research evaluated the tribological behavior of Silicon Nitride based composite reinforced with 1 wt. % of Titanium Carbide under dry and lubrication conditions. The lubricant base oil used in this study is 85W140; with nanoparticles additives poly tetra flouro ethylene and Copper (PTFE & Cu) Nanoparticles were added in the base oil to review the performance of the nanoparticles as additives. Ball-on-disc wear tests were conducted to explore the effects of Nano additive in lubricant for ceramic-ceramic tribo-pair. Results showed that friction and wear decreased using nanoparticle in the lubricant oil, as compared to, dry as well as base lubricant oil conditions. It was reported that 0.1 wt. % of PTFE Nano particles and 0.3 wt. % of Cu Nano particles shows minimum value for the coefficient of friction (COF). Rheological studies were also done on these lubricants samples. The findings from the present work encourage the modification of nanoparticle based lubricant to improve the friction and wear properties and to improve the life of the component.

1. Introduction

Friction, wear and lubrication are the main aspects of the tribological properties/characterization. Ceramics are the special class of materials having a wide range of applications from pottery to furnace brick or hard refractory inorganic compounds, which are formed by heating the base material in powder form to a high temperature and controlled atmosphere for solid state reaction. Ceramic materials exhibit properties that make them suitable candidates for a number of industrial and engineering applications. Advanced structural ceramics differ from conventional ceramic consumer goods in that they are made from extremely pure, microscopic powders that are consolidated at high temperatures to yield a dense, durable structure. Compared with the metals advanced structural ceramics are the material which possesses high strength and hardness, good corrosion resistance, good chemical resistance and good wear resistance than the most metals. Also with the merits of relative high strength compared with its low density, low coefficient of friction, excellent anti-corrosion capability, engineering ceramics are widely used as wear resistant material in extremely harsh environment condition where poor or no lubrications available, such as bearing in aerospace machines, ocean engineering machines, food processing machinery and mechanical equipment used in high temperature and corrosive environment. For enhancement in the mechanical properties, tribological properties, chemical stability etc., combinations of materials are most commonly used in present and have a very bright future. These combinations of materials are – laminates, composites and matrices of different types to provide strength, corrosion resistance, dimensional stability, high temperature sustainability and other properties that are not present in conventional form of materials. M. Belmonte observed the wear behavior of textured silicon nitride (Si$_3$N$_4$) ceramics with aligned microstructures under abrasive wear conditions [1]. He performed dry reciprocating self-mated ball-on-flat disc wear tests to study the influence of different micro structural plane/orientation combination on the Si3N4 tribological behavior. He found that the textured materials showed superior wear resistance than non-textured reference Si$_3$N$_4$ for the whole range of loads and contact pressures, 5 – 50 N and 1.7 – 3.6 GPa, respectively, with an increase of about 70% for the maximum applied load. J.M. Carrapichano observed the tribological behavior of Si$_3$N$_4$ – BN composites performing unlubricated sliding tests by pin-on-disc were carried out with three grades of composite materials with 10, 18 and 25 vol% of BN [2]. He found that the addition of BN to
the silicon nitride (Si₃N₄) matrix resulted in a slight reduction of the friction coefficient, which decreased from 0.82 for monolithic Si₃N₄ to 0.67 for Si₃N₄–10% BN materials. Wear coefficients (K) were above 10⁻⁵ mm³ N⁻¹ m⁻¹ for all materials tested and increased sharply with increases in BN volume fraction greater than 10%, e.g. K ≈ 10⁻³ mm³ N⁻¹ m⁻¹ for Si₃N₄–25% BN. A.H. Jone observed the mechanical and tribological properties of Si₃N₄–TiB₂ ceramic composites produced by hot pressing and hot isostatic pressing [3]. A novel dual atmosphere sintering profile combined with low temperature hot pressing has been used to successfully produce fully dense materials. Wear coefficients and friction coefficients for Si₃N₄–TiB₂ composites have been measured and compared to monolithic Si₃N₄ materials. Composites containing TiB₂ show significant improvements in hardness, fracture toughness and wear. P. Tatarko observed the wear resistant properties of the hot–pressed Si₃N₄/SiC micro/nanocomposites as well as monolithic silicon nitrides sintered with the rare–earth oxide sintering additives (La₂O₃, Nd₂O₃, Y₂O₃, Yb₂O₃, and Lu₂O₃) under dry sliding conditions [4]. The friction coefficient decreased with a decreasing ionic radius of rare–earth elements either in monolithic or composite materials. Ouyang et al., [5] studied the high temperature tribology and solid lubrication of advanced ceramics. The high temperature friction and wear characteristics of different ceramics incorporated with various solid lubricants have investigated from room temperature to 1000 °C. General design considerations relevant to solid lubrication were proposed on the basis of friction and wear data of self-lubricating CMCs. The self-lubricating composites incorporated with SrSO₄ and CaSiO₃ exhibits low and stable friction coefficients of 0.2 to 0.3 and small wear rates in the order of 10⁻⁶ mm3/Nm from room temperature to 800 °C.

2. Experimental Procedure

2.1. Sample Preparation
The samples were prepared in the form of disc 30 mm diameter and 8 mm thickness. The surface of samples was prepared by grinding and then polishing by using emery papers with different grit sizes. After polishing on silicon carbide emery paper, the surface was further polished with the help of diamond paste with particle size of 0.5 μm and 0.25 μm.

2.2. Lubricant Sample Preparation
The nanoparticles were added to the lubricating oil at different concentrations on weight basis. The required quantity of nanoparticles was accurately weighed using a precision electronic weighing balance and then mixed with the lubricating oil. An ultrasonic shaker and probe sonicator was used for mixing the nanoparticle additives in the lubricating oil. The time of agitation was fixed at 30 minutes based on the past experience in producing a stable suspension without sufficient time for sedimentation to begin.

2.3. Microstructure Study
Microstructure study consists of Scanning Electron Microscopy (SEM), Energy Dispersive X–Ray Spectroscopy (EDX) and optical etc. Figure 1 shows the SEM image of composite material. With the help of these studies we can easily understand the microstructure, atomic structure, composition. These studies give proof to the composition of the material i.e. can identify the different material used in the composites and also proof to the bond structure of the composites. SEM microstructure of polished and chemically etched specimens was carried out by Phenom TM G2 Pro, The Netherlands Portable SEM instrument revealed formation of 2 to 5 μm needle shaped grains in silicon nitride composite containing 1 wt % TiC.
2.4. **Tribo Testing**

Dry as well as wet test for friction and wear measurement, were conducted on reciprocating ball-on-disc universal tribometer (R-tec, USA). The tests were performed at following conditions: different concentration of the nanoparticles in the lubricant (80W140) by keeping the normal load and sliding distance constant for each test and measure the values of friction coefficient and wear. Load Tests – which are performed by variation in normal load from 50 N to 110 N, keeping the sliding distance and sliding velocity constant for each test and measure the values of friction coefficient and wear. The samples were cleaned before and after the test by immersion in acetone with agitation in an ultrasonic bath for 10 min, drying in an oven at 50 °C for 10 min, and then being left to cool. The silicon carbide ball with a diameter of 10 mm, was fixed in the holder and positioned vertically, silicon nitride – titanium carbide composite disc that was in turn fixed to the base of tribometer.

2.5. **Boundary Lubrication Condition**

Equation (1) was used to determine whether the lubrication was under boundary lubrication or not [6]:

\[ h_{\text{min}} = 7.43R \left( 1 - 0.85e^{-0.31k} \right) \left( \frac{\eta u}{E^*R} \right)^{0.65} \left( \frac{L}{R^2E^*} \right)^{-0.21} \]  

(1)

Where k is the ellipticity parameter, R the composite radius (m), \( \eta \) the absolute viscosity (Pa s), u the sliding velocity (m/s), \( E^* \) the composite elasticity modulus (Pa) and L the load (N).

\[ \frac{1}{E^*} = \frac{1-v_a^2}{E_a} + \frac{1-v_b^2}{E_b} \]  

(2)

\[ \frac{1}{R} = \frac{1}{R_a} + \frac{1}{R_b} \]  

(3)

\[ \lambda = \frac{h_{\text{min}}}{\sigma^*} \]  

(4)

\[ \sigma^* = \sqrt{(\sigma_a^2 + \sigma_b^2)} \]  

(5)

Where \( R_a \) is the radius of ball, \( R_b \) is the radius of Flat disc (Infinite), k is the ellipticity parameter (1); \( \eta \) is the viscosity of oil (Pa s), u is the mean velocity (m/s), is the Poisson’s ratio of steel ball is the Poisson’s ratio of disk, \( E_a \) is the elasticity modulus of ball, \( E_b \) is the elasticity modulus of disk, L is the normal load. For boundary lubrication the value of should be less than 1.

2.6. **Rheological Studies**

To study the rheology we measure the viscosity and shear stress at shear rate varies from 1 to 1000. The rheological tests were performed on the base oil and on the optimum percentage of the Nano additives.
in the base oil. The temperature were also varied during the test. The test were performed at 25°C and 40°C.

3. Results and Discussion

3.1. Analysis of wear under dry conditions
The dry test were conducted at 50 N load, sliding distance 90m, stroke 2mm, frequency 20 Hz at room temperature. Under dry condition maximum wear and highest value for the COF was reported under these conditions. Wear scar can be easily visible by naked eye and the SEM and EDS of wear track is shown in Figure 2. Optical microscopy, SEM & EDS was used to examine the wear of the material. It was reported that the main reason for the wear is abrasion mechanism as clearly visible from Figure 2.

![Figure 2](image)

**Figure 2.** SEM and EDS analysis of wear track under dry conditions.

3.2. Effect of PTFE nanoparticle concentration on COF
The tribological test were performed to study the effect of the varying concentration of nanoparticles in the lubricating oil under constant load and sliding distance room temperature conditions. It is observed that the COF was maximum under dry sliding conditions but when we use the base lubricant oil the value of the COF was reduced from 0.11 to 0.10. The COF Vs conc. of PTFE is shown in Figure 3. The value of the COF was reduced by adding the PTFE nanoparticle to the base oil with concentration varies from (0.05 wt. % to 0.2 wt. %). It is reported that the minimum value for the COF is obtained for 0.1 wt. % of PTFE nano particles as shown in Figure 3 and was supposed to be the optimum nanoparticle concentration in case of PTFE nanoparticles.
3.3. Effect of PTFE optimum concentration on load
The load tests were performed on the optimum concentration of PTFE Nanoparticles (0.1 wt. % PTFE nanoparticles). The concentration, sliding distance, stroke and frequency remain constant and there is a variation in the load from 50 N to 110 N. COF Vs load for optimum concentration with varying load is shown in Figure 4. It is clear from Figure 4 that the value for maximum COF was obtained at 70 N load and the minimum value was obtained at 50 N load. It was reported that in the initial load test from 50N to 70N the value of COF increases after that the COF value decreases with an increase in the load.

3.4. Analysis of wear with PTFE as nanoparticle additive
With PTFE as an additive the lubricating oil report negligible wear. It might be due to the antiwear property of the PTFE nanoparticles and formation of the protective tribofilm which prevent direct surface contact and reduce the wear scar. Scar was difficult to examine by naked eye. A very minute scratch was reported by using optical microscope as shown in Figure 5.
3.5. Effect of Cu nanoparticle concentration on COF
The tribological test were performed to study the effect of the varying concentration of Nano particles in the lubricating oil under constant load and sliding distance at room temperature conditions. It was observed that the COF was maximum under dry sliding conditions but when we use the base lubricant oil, the value of the COF was reduced from 0.11 to 0.10. The COF Vs conc. of Cu is shown in Figure 6. The value of the COF was also reduced by adding the Cu Nano particle to the base oil with concentration varies from (0.05 wt. % to 0.4 wt. %). It is clear from Figure 6 that the minimum value of COF was reported for 0.3 wt. % of Cu Nano particles and was supposed to be the optimum nanoparticle concentration in case of Cu nanoparticles.

![Figure 6. COF with varying Cu concentration.](image)

3.6. Effect of Cu optimum concentration on load
The load tests were performed on the optimum concentration of Cu nanoparticles (0.3 wt. % Cu nanoparticles). The concentration, sliding distance, stroke and frequency remain constant and there is a variation in the load from 50 N to 110 N. The COF Vs load is shown in Figure 7. It is clear from Figure 6 that the maximum value of COF was obtained at 70 N load and the minimum value was obtained at 110 N load. It is also evident from Figure 7 that in the initial load test from 50N to 70N the value of COF increases after that the COF value decreases with an increase in the load.

![Figure 7. COF with optimum Cu concentration with varying Load.](image)
3.7. Analysis of wear with Cu as nanoparticle additive
With Cu as an additive the lubricating oil report negligible wear. It might be due to the rolling nanoparticle lubrication mechanism. The spherical nanoparticles roll between the mating surfaces which prevent direct surface contact and reduce the wear scar. Scar was difficult to examine by naked eye. A very minute scratch was reported by using optical microscope as shown in Figure 8.

![Figure 8](image.png)

**Figure 8.** Optical micrograph of wear surface with Cu nanoparticles as additive.

3.8. Comparison of COF between PTFE & Cu nano particles
While comparing the performance of two different nanoparticles additives on the basis of their concentration it was reported that by the addition of the both of this nano additive, there is an enhancement in the lubricant properties. Figure 9 compares the COF of PTFE and Cu with varying percentage. It is clear from Figure 9 that the COF reduce and the minimum value of the COF was obtained for 0.1 wt. % of PTFE particles and the maximum value of the COF was obtained for 0.05 wt. % of PTFE particles. For Cu nanoparticles the minimum value for COF was obtained at 0.3 wt. % of nanoparticles. Further the load test were compared with varying load from 50N to 110N as shown in Figure 10 and all parameter along with optimum concentration of both the nanoparticles remain constant. It is clear from Figure 10 that the minimum COF was reported at 50 N load for the optimum percentage of PTFE.

![Figure 9](image.png)

**Figure 9.** Comparision of COF between PTFE and Cu nanoparticles with varying concentration.
Figure 10. Comparison of COF between PTFE and Cu nanoparticles with optimum concentration under varying load.

3.9. Rheological Analysis

Rheological studies as shown in Figures 11 and 12 are used to measure the viscosity and shear stress at a shear rate varies from 1 to 1000 for 0.1 wt.% PTFE and 0.3 wt.% Cu in base oil. The rheological tests were performed on the base oil and on the optimum percentage of the Nano additives (PTFE and Cu) in the base oil. The temperature was also varied during the test. The tests were performed at 25°C and 40°C. Viscosity Vs share stress for 0.1 wt.% PTFE and 0.3 wt.% Cu is shown in Figures 13 and 14. It is clear from Figures 13 and 14 that the viscosity of the base oil remain constant with respect to the share rate at 25°C, as the temperature increases to 40°C the value for the viscosity decreases as compared to the lower temperature but remain constant with respect to the share rate. The shear stress curve is linear at both the temperature but the value for the shear stress is less at 40°C. As the nanoparticle concentration increases the viscosity and the shear stress also increases. By this it is reported that the lubricant is Newtonian fluid. The rheological test of the nanoparticle additive lubricant was done at the optimum concentration for both nanoparticle additive.

Figure 11. Shear stress to shear rate of base oil and optimum % of PTFE nanoparticles in base oil at 25°C and 40°C.

Figure 12. Shear stress to shear rate of base oil and optimum % of Cu nanoparticles in base oil at 25°C and 40°C.
Figure 13. Viscosity to shear rate of base oil and optimum % of PTFE nanoparticles in base oil at 25ºC and 40ºC.

Figure 14. Viscosity to shear rate of base oil and optimum % of Cu nanoparticles in base oil at 25ºC and 40ºC.

4. Conclusion
1. It was observed that the COF was maximum under dry sliding conditions with COF 0.11 which was reduced from to 0.10 under base lubricant.
2. The value of the COF was further reduced by adding the PTFE nano particle to the base oil with concentration varies from (0.05 wt. % to 0.2 wt. %).
3. It was reported that the minimum value for the COF was reported for 0.1 wt. % of PTFE Nanoparticle and 0.3 wt. % of Cu nano particles. It was supposed to be the optimum Nanoparticle concentration in case of PTFE nanoparticle and Cu nanoparticles.
4. It was concluded that the inclusions of the nanoparticles in the lubricating oil enhance the friction and wear properties of the oil which furthermore help to enhance the life of the material.

References
[1] Belmontea M, Miranzoa P, Osendia M I, Gomesb J R 2009 Wear 266 6–12.
[2] Carrapichano J.M, Gomes J R, Silva R F 2002 Wear 253(9-10) 1070-1076.
[3] Jones A H 1997 PhD thesis University of Warwick, Coventry UK.
[4] Tatarkoa P, Kasiarovaa M, Duszaa J, Morgielb J, Sajgalik P, Hvizdos P 2010 Wear 08 020
[5] Ouyang J H, Murakami T, Sasaki S, Li Y F, Wang Y M, Umeda K and Zhou Y 2008 In Key Engineering Materials 368 1088-1091.
[6] Raina A and Anand A 2017 Applied nanoscience 7 371–388.