Effect of nano-crystalline $TiO_2$ addition on reciprocating frictional behaviour of alumina ceramics

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Abstract. We report the effect of normal load and sliding frequency on the tribological performance of alumina ceramics in relation with nano-crystalline $TiO_2$ addition. Tribological studies are conducted by reciprocating a silicon nitride ball on the prepared samples in dry condition in a linear reciprocating tribotester. Reciprocating friction tests are performed with sliding frequency of 15, 30, 45 and 60 Hz and at normal loads of 0.3, 0.5, 0.7 and 1.0 kg. The friction coefficient increases with increasing sliding frequency, normal load but decreases with nano titania addition. The coefficient of friction sharply increases at the level of sliding frequency from 30 Hz to 45 Hz. As the coefficient of friction gradually decreases with increase in $TiO_2$ addition, it can be inferred that alumina ceramics with $TiO_2$ addition up to 4 weight percent can be used as ceramic engine components, cutting tool inserts.

1. Introduction

Alumina ceramics is used for tribological applications due to its high hardness, high melting point, chemical inertness and high wear resistance. It is used in building tools like gas turbine engines, cutting tools, roller and bearings etc. [1-3]. One of the most important factors which impact the mechanical properties and wear resistance in alumina is grain refinement [4-6]. Fine grained ceramics improve wear resistance as compare to coarse-grained ceramics. Nano sized powders enable us to easily fabricate fine grained ceramics. Coefficient of friction (COF) of nano-crystalline $TiO_2$ based alumina ceramics is measured by varying sliding frequency, normal load and weight percentage of nano titania addition when reciprocating against a $Si_3N_4$ counter face. The main claims of this paper are as follows:

- COF is directly proportional to sliding frequency for $TiO_2/Al_2O_3$ system
- COF is directly proportional to normal load for $TiO_2/Al_2O_3$ system
- COF is inversely proportional to weight percent of nano $TiO_2$ addition

2. Experimental work

2.1. Materials

Reactive alumina ($Al_2O_3$) is purchased from M/S Alcoa. Nano-$TiO_2$ is synthesized with the help of chemical grade titanium (IV) isopropoxide ($Ti[OCH(CH_3)]_4$), citric acid ($C_6H_8O_7$) and
ethylene glycol \((C_2H_6O_2)\) (all of these precursors are 99.9% pure, purchased from M/S Merck), which are used as starting material without any further purification.

2.2. Synthesis of nano-crystalline \(TiO_2\) powder

For the synthesis of nano \(TiO_2\) powder, citric acid and titanium (IV) isopropoxide are mixed in stoichiometric ratio in aqueous solution under constant stirring and heating at about 60°C until complete dissolution. Ethylene glycol is then added with the same volume proportion under constant stirring and heating at about 80°C until gelation occurs. Then the gel is oven dried at 120°C for 24 hours. The dried material is ground in an agate mortar and heat treated in a muffle furnace at 800°C for 3 hours for proper crystallisation.

2.3. Sample preparation

Five set of samples are prepared with addition of 0, 0.5, 1, 2 and 4 weight percent of nano-TiO\(_2\) to Al\(_2\)O\(_3\) matrix and the samples are marked as \(ATiO_2−0\), \(ATiO_2−0.5\), \(ATiO_2−1\), \(ATiO_2−2\) and \(ATiO_2−4\) to identify the samples containing alumina and respective weight percentage of TiO\(_2\) in it. To prepare the batches the raw powders are weighed and mixed with required different weight percentages by making slurry with distilled water. The mixed suspension is dried in a drier for 24 hours followed by grinding of the dried powder in agate mortar pestle. 6% PVA (poly vinyl alcohol) is used as the binding agent. The samples are produced by powder metallurgy route. The dried mixtures are cold pressed in the form of cylindrical pellets (diameter \(≈ 12.9\) mm and thickness \(≈ 3.8\) mm) in a hydraulic press at a uniaxial pressure of 1000 \(kgf/cm^2\). After 2 minutes of dwelling time pressure is released by operating pressure releasing valve. All the green compacts are dried in air at 110°C in a drier for 24 hours to remove the binder from the samples. The green samples are then sintered at 1600°C with a heating rate of 8°C/min up to 1000°C then 10 minutes soaking and 2°C/min upto 1600°C is maintained with a soaking time of 2 hours at the maximum peak temperature. After the definite isothermal holding, the samples are slowly cooled down to room temperature in the furnace.

2.4. Test procedure

Friction tester, TR-282, DUCUM is used to conduct the reciprocating friction test of the specimens under observation. Samples of \(φ10mm×3mm\) are produced from the fabricated material. \(Si_3N_4\) ball is used as the counter face. The normal load to the sample is applied through a pivoted lever. The test is carried out by applying different normal loads, sliding frequencies and sliding duration of 10 minutes as indicated in Table 1. The reciprocating stroke is fixed as 1 (one) mm. The COF at the interface of the ball and specimen is recorded once per second.

| Table 1. Process parameters and their levels |
|---------------------------------------------|
| Control parameters | Units | Levels | I | II | III | IV |
| Sliding frequency  | Hz    | 15     | 30 | 45 | 60  |
| Normal load       | kg    | 0.3    | 0.5 | 0.7 | 1   |
| Time              | Min   | 10     | 10 | 10 | 10  |
For all the samples two set of experimental scheme is adopted. In the first set, the normal load of 500 gm, sliding time of 10 minutes are kept constant and the sliding frequency is varied as per levels. In the second set, the sliding frequency of 30 Hz, sliding time of 10 minutes are kept constant and the normal load is varied as per levels.

3. Results and discussions

The nano-crystalline $TiO_2$ based alumina ceramics densify more than 96% with apparent porosity less than 1.5%. Various factors affect the COF [7] but in our work we have concentrated on the impact of normal load and sliding frequency on the COF for $TiO_2/Al_2O_3$ system.

3.1. Effect of reciprocating frequency on coefficient of friction

The variation of COF with sliding frequency at 500 gm normal loads for all the samples has been shown in Fig. 1(a-e). The friction coefficient as obtained in all the graphs captured from the tribotester can be divided into two stages: wear in stage and stable stage [8]. In the wear in stage, contact occurs between the surface asperities of the sample with the ball. At this initial break-in period, the asperities deform, break or get ruptured. As a result the COF is unsteady at this stage. With the progress of sliding, steady level of COF is achieved due to the worn track becoming flat as well as due to polishing process during the wear test [9]. It is seen from the Fig. 1(a-e) that the COF values fluctuate around an average value which is mainly caused by non-uniform wear track or stick-slip phenomenon [10]. The friction coefficients in the stable stage are listed in Table 2 as well as in plotted in a graph as shown in Fig.2.

Figure 1. Variation of COF with reciprocating frequency at 500 gm normal loads for (a) $ATiO_2 - 0$ (b) $ATiO_2 - 0.5$ (c) $ATiO_2 - 1$ (d) $ATiO_2 - 2$ (e) $ATiO_2 - 4$
Table 2. COF of materials at different frequency at constant normal load of 500 gm

| Sliding frequency (Hz) | ATiO$_2$ − 0 | ATiO$_2$ − 0.5 | ATiO$_2$ − 1 | ATiO$_2$ − 2 | ATiO$_2$ − 4 |
|-----------------------|---------------|----------------|---------------|---------------|---------------|
| 15                    | 0.048075      | 0.048062       | 0.046175      | 0.04573       | 0.045408      |
| 30                    | 0.077682      | 0.074356       | 0.068590      | 0.06247       | 0.057290      |
| 45                    | 0.253962      | 0.22315        | 0.198313      | 0.183151      | 0.171339      |
| 60                    | 0.344184      | 0.328330       | 0.303150      | 0.272650      | 0.23523       |

Figure 2. COF as function of reciprocating frequency

In general, the COF increases with the increase in sliding frequency for any of the samples. It is clear from the Fig.2 that the rate of increase in the value of COF as the sliding frequency increases from 30 Hz to 45 Hz is more than any other consecutive intervals. With increase in reciprocating frequency, the temperature at the contact zone rises, so cooling time per stroke of the Si$_3$N$_4$ ball is reduced. Heat generated at the contact surface results more or increased adhesion with the ball [11] leading to increase of friction coefficient. It is also observed that as the weight percentage of TiO$_2$ in alumina increases, the COF decreases.

3.2. Effect of normal load on friction coefficient

In this section, the variation of COF with different normal loads (0.3 kg, 0.5 kg, 0.7 kg and 1.0 kg) at 30 Hz sliding frequency has been studied and has been shown in Fig. 3(a-e) respectively. The stable COF data of Table 3 depicts that with increase in normal load, COF increases. Similar observation is reported for alumina based ceramics by Wang et al. [8]. This is due
to formation of plastic deformation zone on the material surface due to larger value of normal load. Moreover, with increase in TiO₂ in alumina, the COF decreases for all the cases. COF of different samples under observation are plotted as a function of normal load in Fig. 4.

Table 3. COF of materials at different normal load at constant sliding frequency of 30 Hz

| Normal load (gm) | ATiO₂ − 0 | ATiO₂ − 0.5 | ATiO₂ − 1 | ATiO₂ − 2 | ATiO₂ − 4 |
|------------------|-----------|-------------|-----------|-----------|-----------|
| 300              | 0.061038  | 0.059317    | 0.05305   | 0.04874   | 0.0427    |
| 500              | 0.077682  | 0.07436     | 0.06859   | 0.06263   | 0.05529   |
| 700              | 0.141231  | 0.132787    | 0.11551   | 0.092543  | 0.070873  |
| 1000             | 0.205162  | 0.198183    | 0.18132   | 0.166795  | 0.150881  |

Figure 3. Variation of COF with normal load at 30 Hz sliding frequency for (a) ATiO₂ − 0 (b) ATiO₂ − 0.5 (c) ATiO₂ − 1 (d) ATiO₂ − 2 (e) ATiO₂ − 4

4. Conclusions
The normal load and sliding frequency indeed have a considerable effect on the friction behavior of nano-crystalline TiO₂ based alumina ceramics. For the samples under observation, with constant normal load of 500 gm, with increase in sliding frequency the value of COF increases, this is due to more adhesion of Si₃N₄ ball to the ceramic specimen. The rate of increment in COF from 30 Hz frequency to 45 Hz is more as compared to other consecutive frequency intervals. As the TiO₂ weight percent in alumina ceramics increases, the value of COF decreases for any
constant sliding frequency level at 500 gm normal load. On the other hand, for the samples under observation, with constant sliding frequency of 30 Hz, with increase in normal load the value of COF increases. This is due to formation of plastic deformation zone on the material surface due to larger value of normal load. As the TiO$_2$ weight percent in alumina ceramics increases, the value of COF also decreases for any constant normal load at 30 Hz sliding frequency. This study opens a new area of research on the ceramic engine components, cutting tool and other relevant field of work.

References

[1] Ajayi O O and Ludema K C 1988 Wear 124 237
[2] Kato K and Adachi K 2002 Wear 253 1097
[3] Lee S W, Morillo C, Lira-Olives J, Kim S H, Sekino T, Niihara K and Hockey B J 2003 Wear 255 1040
[4] Cho S J, Hockey B J, Lawn B R and Bennison S J 1989 J. Am. Ceram. Soc. 72 1249
[5] Zum Gahr K H, Bundschuh W and Zimmerlin B 1993 Wear 162 269
[6] Kerkwijk B, Winnubst A J, Verweij H, Mulder E J, Metselaar H S C and Schipper D J 1999 Wear 225 1293
[7] Sarkar P, Modak N and Sahoo P 2016 Int. J. Surf. Eng. Interdiscip. Mater. Sci. 4 25
[8] Wang J, Cheng Y, Zhang Y, Yin Z, Hu X and Yuan Q 2017 Ceram. Int. 43 14827
[9] Rodriguez-Suarez T, Bartolomé J F, Smirnov A, Lopez-Esteban S, Torrecillas R and Moya J S 2011 J. Eur. Ceram. Soc. 31 1389
[10] Parchovianský M, Balko J, Švancárek P, Sedláček J, Dusza J, Lofaj F and Galusek D 2017 J. Eur. Ceram. Soc. 37 4297
[11] Yin Z, Yuan J, Huang C, Wang Z, Huang L and Cheng Y 2016 Ceram. Int. 42 1982