Evaluation of mechanical and frictional properties of CuO added MgO/ZTA ceramics

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Abstract
The focus of the present work is to investigate the tribological effect of adding CuO as solid lubricant in MgO/ZTA (Magnesia-Zirconia Toughened Alumina) ceramics. Different wt% of CuO (0–5 wt%) have been added in MgO/ZTA ceramics using powder metallurgy process route to study the change in mechanical properties i.e., hardness, fracture toughness and flexural strength, as well as the tribological properties i.e., coefficient of friction (COF) and wear depth, against corundum alumina balls. The addition of CuO shows significant improvement in tribological properties but a diminishment of the mechanical properties. A significant reduction of 53.45% in COF with minimum wear depth is achieved with the addition of 1.5 wt% CuO inside the MgO/ZTA matrix. The improvement in COF and specific wear rate of the composite is observed with increasing sliding velocity due to the presence of uniform patchy layer along with a glossy surface on the wear track. The formation of patchy layer is enhanced with an increase in sliding velocity due to high squeezing and smearing of wear debris. A significant increment in specific wear rate is observed with increasing load due to the delamination process prevailing at the contact surfaces.

1. Introduction

Lubrication plays a vital role in the improvement of component life in industrial applications. Petroleum-based lubricants were introduced early in the 1900s as external lubricants to safeguard the life of components, but their hazardous effects on the environment and human health, notably skin ailments and allergies, restricted their widespread application. The focus of research then shifted towards the incorporation of self-lubricating materials in the base matrix of composites to enhance the service life of components made thereof. Additives such as Graphite [1], CuO [2], MoS₂ [3], CaF₂ [4] and WS₂ [5] were used to enhance desirable properties of ceramic composites, viz. hot hardness, corrosion resistance, chemical stability and abrasion resistance. Nowadays, self-lubricating composites constitute one of the most promising materials for researchers. Stachowiak et al [6] investigated the self-lubricating behavior of zirconia toughened alumina in the late 20th-century, and initially observed high coefficient of friction (COF) (~0.4–0.7) for ceramic-ceramic and metal-ceramic sliding pairs. A moderate value of COF with best wear performance was found for cast iron-ceramic sliding pairs because of two predominant phenomena: plastic deformation and delamination. The delamination process was found to be significant in hard sliding i.e. for ceramic-ceramic contact, whereas the metallic film transfer on to the ceramic surface was observed for metal-ceramic sliding process. Hannink et al [7] carried out experiments to analyze the friction and wear properties of magnesia added partially stabilized zirconia (magnesia-PSZ) composites. The experiments suggested that the developed composites had lower friction along with low wear at elevated temperatures. A detailed investigation to gauge the effect of oxidation on tribological...
properties for solid solution Cu–Al alloys was carried out by Poggie et al [8]. Their analysis suggested that the wear and COF decreased significantly with an increasing percentage of aluminium, due to the formation of uniform Cu2O layer at the interface. Kerkwijk et al[9] investigated the tribological properties of nanoscale alumina-zirconia composites, and their findings suggested that the tribological behaviour significantly improved with incorporation of second phase zirconia or alumina. Alexeyev et al[10] carried out experiments to illustrate the friction phenomenon for self-lubrication composites. These authors demonstrated that the second phase deformation and motion of additives present in the developed composites were jointly responsible for the improvement in tribological properties. Authors also concluded that the physical and mechanical properties of films developed on the surfaces have profound effect on the tribological behaviour of ceramic composites: materials which formed soft films on the surface during sliding showed excellent tribological characteristics. Experiments also revealed that the particle size, limiting shear stress and soft phases were responsible for improving the COF, and these parameters were optimized according to friction conditions. Wang et al[11] investigated the tribological properties of partially stabilized zirconia with CuO. The value of COF diminished from 0.4 to 0.175 with incorporation of CuO inside the PSZ matrix. Pasaribu et al[12] investigated the tribological properties of CuO added zirconia and alumina ceramics in different environmental conditions. The lowest value of COF (∼0.25–0.35) was observed for zirconia mixed with CuO at an ambient temperature. The analysis of Pasaribu et al[12] also showed severe wear in the case of zirconia doped CuO at elevated temperature compared to alumina doped with CuO. The effect of temperature on tribological properties for zirconia doped CuO was further carried out by Valefi et al[13]. Their analyses showed low values of COF at higher temperature, attributed to the plastic deformation of copper-rich wear debris which resulted in the formation of a smooth layer of Cu2O phase at the interface. The aforementioned smooth layer was subjected to continuous removal from and deformation at the interface, and was regenerated and healed by sliding action. This phenomenon was observed up to the certain load, beyond which a complete removal of the soft layer was reported. Xu et al[14] studied the tribological properties of yttria-stabilized zirconia used in the fabrication of ceramic tool and die material. The observation showed a significant reduction in COF of 24% for ZrO2/Ti(C3N3) nanocomposite compared to single-phase ZrO2 ceramic. The selected composites also demonstrated an improvement in wear resistance due to the addition of Ti(C3N3) nanophase. Westergard et al[15] studied the tribological behaviour of plasma sprayed alumina with Cu on the coating of mild carbon steel, and illustrated that wear characteristics significantly improved due to sealing treatment.

Though a large volume of research has been carried out in the field of self-lubrication ceramics, little effort has been made to apply these lubricating composites to structural/engineering applications. Assessment of ceramics for said applications demands comprehensive knowledge of their mechanical and tribological behaviour. In the present investigation, attempts have been made to estimate the optimum percentage of CuO (varied between 0–5 wt%) in MgO/ZTA by studying the variation in COF and wear depth, as well as in the hardness, fracture toughness and flexural strength of the developed composites. Further studies have been carried out to understand the underlying wear phenomena through FESEM imaging. The authors have also attempted to visualize the tribological behavior of the composites by varying sliding velocity and applied load.

2. Experimental details

In this work, powder metallurgy processing route has been adopted to develop (0–5 wt%) CuO/MgO/ZTA composites. Zirconia toughened alumina having 3 mol% yttria (average particle size 60 μm, supplied by Artha Materials, Pune, India) is used as the base matrix, in which different quantities of CuO (average particle size 40 nm, supplied by Merck) have been incorporated. A particle size analyzer (Make: Malvern Model, Nano-ZS90) has been used to measure grain size, while the determination of bulk density of all sintered components has been accomplished through Archimedes’ principle. The details of calculation of the bulk density of components have been reported in an earlier work of the author [16]. The requisite amount of CuO in ethanol was vigorously shaken by a probe-sonicator. The dispersed CuO was mixed with requisite amount of ZTA; 0.6 wt% of MgO was added to act as an inhibitor of grain growth. The mixed powders were placed in a planetary ball mill, with alumina balls (Diameter: 6–10 mm) as milling media, for 45–50 h. After proper blending, the composite powders were placed in a drying oven for 12 h. The blended mixture was gently crushed by hand in a mortar and pestle set. The composites were finally calcined at 800 °C, followed by sintering at 1600 °C.

The mechanical and tribological characterization of all composites was carried out on square samples having dimensions as follows: length = 14 mm, breadth = 14 mm and thickness = 5 mm. The same is shown in figure 1(A). During the preparation of samples, at first, the well-blended calcined powders were pressed in a hydraulic press (Make: Carver, USA) with the help of die-punch arrangement at a pressure of 5 ton cm$^{-2}$. Secondly, the green compacts were placed in a high-temperature furnace at a temperature of 1600 °C for 1 h.
Finally, the sintered specimens were polished till a surface roughness of 1 micron was achieved. This was accomplished in two steps: first, the samples were slowly polished by honing/lapping process using silicon carbide powder of 400 mesh size. The samples were then polished with diamond paste (0.5–1.0 μm) on a Bainpolisher machine to get the required surface finish.

Rectangular specimens having length 50 mm, breadth 10 mm and thickness 5 mm were used to carry the three-point bend test or flexural test on a universal testing machine (Tenius Olsen Serial No H50KS-0537) as shown in figure 1 (B). To prepare the flexural samples, the requisite amount of well blended calcined powder was mixed with 5 wt% polyvinyl alcohol solution in an automatic stirrer machine for 1 h. The well-mixed powder was kept in an oven for 6 h at 100 °C to remove moisture content. The dried mass was then pressed at a pressure of 2.5 ton cm$^{-2}$ in a hydraulic press (Make: Carver, USA) using die-punch arrangement. The samples were slowly removed from the die and kept at 600 °C for 180 min to eliminate the binders from green compacts. After cooling, the samples were again placed in a high-temperature furnace at a temperature of 1600 °C with 1 h dwell time.

Field emission scanning electron microscope (FESEM) (CARL-ZEISS-SMT-LTD, Germany, Model: SUPRA 40) attached with EDAX has been used for microstructural characterization. Phase identification has been carried out using x-ray diffraction (XRD) in the range angles (2θ) of 20° and 70° (figure 2). The hardness measurement was carried out on a Vickers hardness testing machine (Innova test Machine Falcone 500) using micro indentation at a load of 500 gm [17].

$$HV = 1.854(F/D^2)$$

(1)

Where, F (measured in Kgf) is the applied load and $D^2$ (measured in mm$^2$) is the area of the indentation.
The fracture toughness has been calculated using Evans and Charles equation [18] by measurement of crack length as mentioned in equation (2).

\[
K_{IC} = 0.16(c/a)^{−1.5}(H_a^{0.5})
\]  

Where,

- \(K_{IC}\) = Fracture toughness (MPa-m\(^1/2\))  
- \(H\) = Vickers hardness (MPa)  
- \(c\) = Average length of the cracks obtained in the tips of the Vickers marks (microns)  
- \(a\) = Half average length of the diagonal (microns)

The microtribological investigations were carried out on universal Mechanical Tester (UMT-2, Bruker, USA) in atmospheric conditions using 6 mm dia. corundum alumina balls after annealing at 900 °C for 1 h. In these experiments, sliding velocity has been considered to be constant within a single stroke followed by a rapid reversal in the sliding direction for both ends of stroke. A load of 10 N and sliding velocity of 4 mm/s under dry sliding conditions were used to investigate the microtribology for each composite. Stroke length of 6 mm with 45 min of sliding time was fixed for each run. The composition which showed the minimum values of tribological properties i.e. COF and wear depth was selected to visualize the effect of varying sliding velocity (2 mm s\(^{-1}\), 4 mm s\(^{-1}\) and 6 m s\(^{-1}\)) and applied normal load (5 N, 10 N and 15 N). Friction and normal forces were measured using a force sensor in both directions. After completion of tests, astral inspection for worn tribo tracks of each sample was carried out using FESEM images to interpret the microstructural characteristics along with spectroscopic analysis of the worn tracks using electron dispersive spectroscopy (EDS). The specific wear rate after each experiment was evaluated by calculating the scar diameter developed after tribological experiment on the counter surface using an optical microscope. Average of five scar diameter readings was taken to conclude the scar diameter for evaluation of specific wear rate. Three equations viz. Equations (3)–(5) mentioned below were used to evaluate the specific wear rate for different sets of conditions. These equations are well described by Berman et al [19].

\[
\text{Specific wear rate} = \frac{\text{Total wear volume}}{\text{Load} \times \text{sliding distance}} \left( \frac{\text{mm}^3}{\text{N} \times \text{m}} \right)  
\]  

\[
\text{Wear volume} = \left( \frac{\pi h}{6} \right) \left( \frac{3d^2}{4} + h^2 \right)  
\]  

\[
h = r - \sqrt{r^2 - \frac{d^2}{4}}  
\]  

Where \(r\) is the radius of alumina ball used during experiments and \(d\) is the scar diameter developed on the alumina ball after tribological investigation.

### 3. Results and discussion

#### 3.1. Morphological and mechanical characterization

The morphological details and mechanical properties (hardness, indentation fracture toughness and flexural strength) of CuO-reinforced zirconia toughened alumina are presented in table 1. The results of particle size analyzer show that grain size of developed composites increases with an increase in the percentage of CuO. It is concluded that CuO promotes grain growth. From the table, it is seen that the bulk density of developed composites decreases with an increase in the percentage of CuO. This may be attributed to the incorporation of CuO particles inside the matrix, which increases the grain size and porosity of the developed composite. A similar result in the case of alumina doped CuO was earlier noticed by Ramesh et al [20]. It is observed from the results that addition of CuO decreases the mechanical properties of developed CuO/MgO/ZTA composites as compared to parent material (MgO/ZTA). However, it may be concluded that minor addition (up to 1.5%) of CuO does not severely deteriorate mechanical properties; beyond that a rapid deterioration occurs. The mechanism behind the same was well described by Ran et al [2]. According to these researchers, the retention of tetragonal phases that facilitates the toughening phenomenon is hardly benefitted in the case of CuO. Beyond certain percentage (above 1.5 wt%), the Cu-rich phase creates an impurity inside the matrix that favours crack growth, consequently reducing the toughness and strength. The FESEM images of 1.5 wt% CuO doped MgO/ZTA matrix before and after sintering is shown in figures 2(a) and (b). XRD analysis of the composites shown in figure 3 confirms the presence of CuO in the matrix.
Table 1. Density, Grain Size, Hardness and Fracture Toughness of different wt% CuO in MgO-ZTA.

| Composition          | Bulk density | Average grain size (nanometer) | Hardness (GPa) | Fracture Toughness (MPa.m$^{1/2}$) | Flexural strength (MPa) |
|----------------------|--------------|-------------------------------|----------------|-----------------------------------|-------------------------|
| MgO-ZTA + 0.0 wt% CuO | 4.28 ± 0.2   | 28 ± 1.4                      | 17.71 ± 0.88   | 6.28 ± 0.31                       | 468 ± 12.5              |
| MgO-ZTA + 0.5 wt% CuO | 4.18 ± 0.2   | 30 ± 1.5                      | 16.69 ± 0.82   | 6.04 ± 0.30                       | 353 ± 8.8               |
| MgO-ZTA + 1.0 wt% CuO | 4.08 ± 0.2   | 35 ± 1.7                      | 15.93 ± 0.79   | 5.93 ± 0.29                       | 338 ± 8.4               |
| MgO-ZTA + 1.5 wt% CuO | 4.03 ± 0.19  | 47 ± 2.2                      | 15.63 ± 0.73   | 5.24 ± 0.26                       | 302 ± 7.6               |
| MgO-ZTA + 2.0 wt% CuO | 3.96 ± 0.18  | 51 ± 2.5                      | 15.45 ± 0.71   | 5.05 ± 0.25                       | 269 ± 6.7               |
| MgO-ZTA + 5.0 wt% CuO | 3.92 ± 0.18  | 65 ± 3.1                      | 14.50 ± 0.68   | 4.93 ± 0.23                       | 235 ± 6.2               |
3.2. Tribological study on coefficient of friction

The results of sliding tests of all samples in terms of COF for MgO/ZTA added with CuO against alumina ball are shown in figure 4 with constant load of 10 N and sliding time of 45 min. From the figure, it is observed that the COF diminishes from 0.58 to 0.27 with the addition of CuO. The lowest value of COF i.e. 0.27 is observed for 1.5 wt% CuO added MgO/ZTA. Beyond that, the value of COF increases up to 0.488 for 5 wt% CuO added MgO/ZTA. The COF curve of 1.5 wt% CuO added MgO/ZTA shows minimal fluctuations compared to other selected compositions due to the formation of long elastic soft film at the interface. In other cases, the tendency to retain the soft film during sliding is very less. An identical behaviour was well illustrated by Prasebu et al [21] in case of alumina and zirconia added with copper oxide. According to these authors, the soft patchy layer could be developed during sliding by deposition of wear debris at the contact area. This deposition is affected by squeezing and smearing action prevailing during the sliding motion. A similar kind of mechanism for self-lubricating composites was also cited by Alexeyev et al and Kerkwijk et al [22, 23]. They also confirmed the same lubricating mechanism, owed to the presence of second phase material (CuO) dispersed inside the matrix of hard materials.

**Figure 3.** XRD pattern of different percentage doped CuO/MgO/ZTA.

**Figure 4.** Variation in friction coefficient with time for all developed composites under ambient condition. Data generated from the ball loading micro-tribometric studies at normal load of 10 N with sliding speed of 4 mm/sec.
3.3. Tribological study on wear depth

Wear resistance property has been evaluated in terms of wear depth for each composite under similar tribological conditions and illustrated in figure 5. An increase in wear depth (at the initial sliding) has been noticed for MgO/ZTA, followed by gradual increment with sliding time. Interestingly, negative wear depth has been observed in all cases of CuO added MgO/ZTA. However, at lower CuO content (<1.5%), the initial trend of positive wear depth could be seen followed by negative wear depth. A completely reversed trend of wear profile has been observed in case of 1.5% CuO doped MgO/ZTA. This negative trend was earlier observed by Samanta et al [24] and Sudagar et al [25]. Researchers explained that the debris generated during sliding tests adheres with the specimen, resulting in the negative trend. High CuO content (5%) initially shows a negative wear depth followed by sudden reversal of wear depth profile. The reason for the fluctuations could be attributed to the combined effect of surface delamination and grain pull-out phenomenon.

The wear mechanism postulated by earlier researchers involved different phenomena for composite specimens containing varying percentages of CuO as second phase material. Kerkwijk et al [22] used several solid oxide lubricants as the second phase in $\alpha$-$\text{Al}_2\text{O}_3$. They held the formation of a soft layer, due to the presence of second phase materials, responsible for the improvement in tribological properties. The superplastic deformation due to the presence of CuO in second phase complex structures was proposed to be responsible for the improvement in wear properties. Similar results for CuO reinforced ZTA were reported by Dey and Biswas [26, 27], in which the wear rate improved with increase in sliding distance. According to these researchers, the improvement in wear rate was due to the formation of a highly polished surface after a certain distance of sliding which resulted in lower contact stresses. The wear phenomena of the composites have also been correlated with FESEM images shown in figure 6. In case of 1.5 wt% CuO added MgO/ZTA, a thick soft layer (figure 6(B)) has been observed which is long elastic in nature and creates a highly polished wear track with sliding time. This could be the possible reason for a minimum value of wear depth (figure 5(d)). But in case of MgO/ZTA, the patchy layers are found to be absent, and as a result, a higher wear depth is observed as shown in figure 6(A). With higher percentage of CuO, particles aggregate into the counterface and provide some kind of abrasive effect. This may be due to the reduction in hardness of the composite as demonstrated in table 1. For a higher amount of CuO, the formation of the patchy layer cracks down after some slab of sliding due to the abrasion effect as shown in figure 6(C). As a result, a high value of COF and wear depth has been found with higher percentage of CuO in MgO/ZTA matrix.

A systematic mechanism for the formation of self-lubricating film or patchy layer on the wear track during the sliding motion for CuO reinforced MgO/ZTA matrix is shown in figure 7. Figure 7(A) represents the sliding action with the application of load on a reciprocating specimen. During sliding, the particles of second phase material i.e., soft particles of CuO are released from the alumina matrix and start accumulating at the interface as shown in figures 7(B) and (C). These particles are squeezed and smeared due to sliding action, resulting in the formation of the soft layer as illustrated in figure 7(D). These layers act as a stable, anti-frictional transfer film between the rubbing tribo-pair and provide lower COF and correspondingly low wear depth.
3.4. Effect of applied load on tribological properties

As discussed in the above section, an improvement in COF of around 53.45% was observed for 1.5 wt% CuO/MgO/ZTA. Hence, this composition has been selected to study the effect of velocity and load on the COF as well as on the specific wear rate. The variation in the COF, wear depth, scar diameter and specific wear rate with different loading conditions is shown in figures 8(A)–(D). A slight variation in COF is observed with different values of load viz. 0.25 for 5 N, and 0.29 for 15 N. A small change in the COF is attributed either to the increased pressure at the interface of two sliding bodies or to a change in environmental conditions. The systematic graph for wear depth with varying load is shown in figure 8(B), which clearly illustrates a transition mechanism, occurring as the load transcends from low to high values. It is observed that at lower load, the pull-out or
ploughing action of soft particles is the predominant mechanism of wear, which eventually results in the accumulation of soft particles on wear track. This phenomenon results in the formation of stable patchy layer. At higher load, the combined effect of superplastic deformation and a pull-out phenomenon is observed. This combined action results in delamination along with higher stress at the interface, resulting in a higher specific wear rate. Similar results were demonstrated earlier by Wei et al [28] and Stachowiak et al [6].

3.5. Effect of sliding velocity on tribological properties
A significant change in the COF values was noticed in the case of velocity variation viz., 0.31 for low sliding velocity (2 mm sec⁻¹) and 0.24 for high sliding velocity (6 mm sec⁻¹), as shown in figure 9(A). As discussed above, the formation of patchy layers is responsible for the minimization of COF. With an increase in sliding velocity, the accumulation of soft particles increases due to higher squeezing and smearing action taking place at the interface of mating surfaces. These accumulations are responsible for the formation of the long elastic and uniform patchy layer. The generation of high heat at the interface due to increased velocity also favours this formation, together resulting in a low value of COF at higher sliding velocity. Similar results were found by Mondal et al [29] and Lim et al [30] with higher sliding velocities in the case of magnesium alloy. Another mechanism responsible for the improvement in COF at higher velocity was well illustrated by Wei et al [28]. According to Wei et al, the plastic deformation within a glossy surface of alumina particles at high sliding velocity favourably affects the minimization of COF.

Figure 9 (B), (C) and (D) respectively show the variation in wear depth, wear scar diameter and specific wear rate with change in sliding velocity. From figure 9(B) i.e., wear depth with time for different sliding velocities, it can be concluded that the wear depth decreases significantly with increasing sliding velocity. This happens due to the formation of a soft patchy layer, which ultimately lowers wear depth. It is seen in figure 9(C) that with increasing velocity the value of WSD decreases. A similar behaviour is observed for specific wear rate as shown in figure 9(D). The following argument can be invoked to understand the wear phenomenon: at lower sliding velocity, the formation of the smooth surface due to sliding action takes place quickly; but the pullout phenomenon continuously increases, which, in turn, is responsible for higher specific wear rate.

The obtained trends of tribological parameters (COF, wear depth, WSD and specific wear rate) are found to be in good agreement with the FESEM images of wear track shown in figures 10(A) and (B) for sliding velocities of 2 mm sec⁻¹ and 6 mm sec⁻¹ respectively. Figure 9(A) reveals a broken patchy layer with some pull out debris present on the worn track, at a sliding velocity of 2 mm s⁻¹ and load of 10 N. In case of higher sliding velocities
(6 mm \text{s}^{-1} \text{ and load being same}), a uniform patchy layer with a glossy surface is observed on the worn track (figure 10(B)).

4. Conclusion

Different compositions of CuO doped MgO/ZTA have been successfully prepared using powder metallurgy route. It is observed that the mechanical properties of developed composites are diminished marginally with the addition of CuO, whereas the tribological properties are significantly improved for 1.5 wt% CuO doped MgO/ZTA. An improvement of 53.45% in the coefficient of friction values has been observed for the composition of 1.5 wt% CuO doped MgO/ZTA. Studies with variation in sliding velocity clearly depict an improvement in the coefficient of friction with the increase in sliding velocity, notably a COF value of 0.24 for 6 mm sec\(^{-1}\), and of 0.31 for 2 mm sec\(^{-1}\). A negative wear depth has been noticed in all cases of CuO doped MgO/ZTA due to the
adherence of wear debris, generated during sliding tests, with the specimen. The specific wear rate decreases with an increase in the sliding velocity; however, it increases with increasing load. The astral analyses of worn track carried out through FESEM images suggest that the improvement in tribological properties may be due to the formation of soft patchy layer (presence of second phase material as CuO inside MgO/ZTA matrix). These patchy layers are formed due to squeezing and smearing action of wear debris.

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