Tribological Characterization of Iron Based Ceramic Reinforced Self- lubricating Material

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Abstract. In this paper, the effect of ZrO2 reinforcement and MoS2 as solid lubricant on tribological properties of iron-copper-tin composite for plain bearing application have been investigated. This paper includes two studies, one in which wt% of MoS2 is varied keeping wt% of ZrO2 constant and in another wt% of ZrO2 is varied keeping wt% of MoS2 constant to see the effect of ZrO2 as reinforcement and MoS2 as a solid lubricant for improving tribological properties of sintered Fe-Cu-Sn material. The material was prepared by sintering at temperature of 1150°C. The tribological properties of developed materials were analysed by ball on disk test. Least value of COF 0.0421 is shown by sample with 2 wt.% of MoS2 and 2 wt.% of ZrO2. Least wear rate of 0.4581x10^-4 mm/Nm for sample with 2 wt.% of MoS2 and 2 wt.% of ZrO2. Characterization of worn surfaces revealed abrasive and adhesive wear including third body abrasive wear, delamination and micro-ploughing in reinforced composites. The addition of MoS2 has improved tribological properties, whereas ZrO2 addition not only improved tribological properties, but also improved strength and hardness of the composite. Maximum hardness value 208HV (701.4MPa) is shown by the composite with 2 wt.% of MoS2 and 2 wt. % of ZrO2. The findings show that the developed material could be used for antifriction and antiwear plain bearing applications.

Keywords: Iron based composites, ceramic reinforcement, ZrO2, MoS2, self lubricating material, Friction, Wear, Plain bearing material.

1. Introduction
Plain bearings are mechanical elements used to reduce friction between rotating shaft and stationary support members. Plain bearings are used primarily in machinery that has a rotating or sliding shaft component. Plain bearings are used in very critical applications where failure of bearings might have severe consequence, example in turbomachines, such as power plant steam turbines, compressors operating in critical pipeline applications, etc. Plain bearings are subjected to extreme conditions of load, temperature and velocities at operational stage. Various materials have been developed to withstand these extreme conditions. Plain bearing materials are developed by various techniques, Powder metallurgy iron based materials, due to their low cost and excellent mechanical properties hold a significant place in industrial sector. Using powder metallurgy, iron (Fe) based composites are tailored to achieve desirable properties [1]. The research and production of iron-based self-lubricating materials is considerably increasing, due to their higher strength, ease of availability and low cost iron powders in the last decades [2,3]. Adding copper and tin to Fe matrix, stabilizes its pearlite structure, that improves strength and corrosion resistance, and also has a rapid surface diffusion over solid iron [4]. Moreover, to reduce friction, solid lubricants are preferred for eg., graphite, MoS2 etc. Such
solid lubricants have a lamellar structure, with hexagonal layers bonded by weak Vander Waals forces and strong covalent bonds between the atoms in each layer. These layers shear on the application of parallel force, resulting into reduction in friction [5]. On the other hand, it also prevents asperity penetration due to presence of the strong covalent bond between Mo and S. Addition of these solids lubricants in composites develops self lubricating material reduces the need of external lubrication.

Fe-Cu-Sn material were studied by various researchers, where Cu and Sn was added in different wt% to find the optimum wt% for developing material with better properties and various solid lubricants were added to develop self lubricating materials. Varying percentages of Pb were added to Fe- Cu- Sn-MoS₂ antifriction material developed by powder metallurgy technique & observed better frictional and mechanical properties on adding Pb up to 7.5 wt.%, whereas further addition deteriorated these properties [6]. A study conducted for studying the effect of solid lubricant (graphite and talc) content on the formation of microstructure, mechanical and tribological properties of Fe–Cu–Sn alloys concluding that its microstructure with solid lubricants addition is multiphase and heterogeneous. Increasing the sintering temperature to 1000 and 1150°C leads to better tribological properties at moderate and high test loads and leads to formation of hard phase [7]. Also, the effect of addition of tin, nano boron nitride and molybdenum disulphide in Fe-Cu based powder metallurgy composites for plain bearing applications was studied and concluded that composition containing 2.5 wt. % Sn and 7.5 wt. % of nano boron nitride showed the higher value of Vickers hardness and low value of coefficient of friction [8]. On addition of graphite to Fe-Cu-Sn alloy at 423 K, researchers revealed that best mechanical properties has been obtained for 3 wt.% graphite content [9].

It has been observed, when ceramics are added to the iron-based composites, there is an increase in the strength and reduction in wear [10]. ZrO₂ being important structural ceramic, it has excellent mechanical properties, such as high strength, fracture toughness, and hardness. When added to iron matrix they increase the hardness the composites [11]. According to a study, iron metal powder when mixed with varying percentage of zirconium dioxide, results into formation of harder Zr₆Fe₃O phase, due to reactive sintering [12]. There are no such studies in the literature where ZrO₂ as ceramic reinforcement was added to Fe-Cu-Sn based composite for plain bearing applications.

The present study aims to improve the hardness, friction and wear resistance of iron based composites for plain bearing applications. This is achieved by adding ceramic to Fe-Cu-Sn based composite, as ceramics improves hardness by forming harder phases with Fe matrix during sintering, that can reduce the wear rate of the bearing material and addition of molybdenum disulphide as solid lubricant reduces friction coefficient. The friction, wear rate and hardness of sintered composites with varying wt% of MoS₂ and ZrO₂ will be investigated.

2. Experimental Details

2.1 Sample preparation

Material compositions of samples and powder specifications is given in table 1. Powders of iron(Fe), copper(Cu), tin(Sn), molybdenum disulphide(MoS₂) and zirconium dioxide(ZrO₂) were used to prepare four compositions B1, B2, B3 and B4 as shown in table 1. Powders of four compositions were mixed using planetary ball mill (PULVERISTTE 5 Classic line) at a speed of 200rpm for 2 hours. Then compaction was carried out at 500MPa. A cylindrical die was used for compaction and green compact of 30mm diameter and 8mm thickness were obtained. Green compacts were sintered at 1150°C in argon atmosphere for 50 min. The sintered samples were then polished using emery papers and mirror finish was obtained by using diamond paste.

Table 1. Chemical composition of the materials.

| Sample | Composition | Fe wt% | Cu wt% | Sn wt% | ZrO₂ wt% | MoS₂ wt% |
|--------|-------------|--------|--------|--------|---------|---------|
| B1     | Fe-5Cu-3Sn  | Bal    | 5      | 3      | 0       | 0       |
| B2     | Fe-5Cu-3Sn-1ZrO₂-1MoS₂ | Bal    | 5      | 3      | 1       | 1       |
3. Results and discussions

3.1 Density & microhardness

The green densities of compacted sample and sintered densities of sintered samples were measured using Archimedes principle. The theoretical density of composite material was computed by rule of mixtures. Figure 1(a) shows that densities of compositions with varying percentages of MoS$_2$. Theoretical, green and sintered densities follow same trend, this shows suitability of this technique for composite preparation, however in powder metallurgy density of composites are less than theoretical densities due to porosity. Also, it is clear from Figure 1(a), sintered densities are more than green densities due to liquid phase sintering of compositions, copper and tin in liquid phase due to capillary action fill the pores in the green compact and thus increase densities of respective samples after sintering. Figure 1(a) showing the same trend for theoretical, green and sintered densities, and sintered densities being greater than green densities.

**Figure 1.** Comparison of densities of samples (a) B1, B2 and B3 (b) B1, B3 and B4.

The average hardness values (HV) verses load measured for varying wt% of MoS$_2$ and ZrO$_2$ is shown in Figure 2(a,b). As load increases microhardness decreases. However, higher hardness values were obtained in sample containing 2 wt.% MoS$_2$ (B3) as compared to samples with 0 wt.% (B1) and 1 wt.% (B2)MoS$_2$. This behavior is attributed to the hardness imparted by MoS$_2$, due to its strong covalent bond between Mo & S, it reduces asperity penetration and it offers obstacles to the dislocation motion due to good bonding with matrix material. Also in Figure 2(b), higher hardness values were obtained in sample with 2 wt. % ZrO$_2$ (B4) as compared to samples with 0 wt.% (B1) and 1 wt.% (B3) ZrO$_2$. This behavior is attributed to the hardness imparted by ZrO$_2$ due to its reactive sintering at high temperature, which resulted into formation of hard phases of ZrO$_2$ with the matrix Fe, as concentration of ZrO$_2$ increases, hard phases increase which in turn improves hardness of material too.
3.2. Coefficient of friction

3.2.1 Effect of sliding distance on COF

The tribological tests were carried out on samples B1, B2 and B3 against EN8 ball. The variation of COF with different wt.% of MoS2 at varying sliding distances, constant load (15N), sliding speed (0.2m/s) at room temperature (27°C) is shown in Figure 3(a). It shows, as sliding distance increases COF decreases. COF for virgin sample B1 is higher due to initial contact then it decreases due to various tin phases that reduces friction [13] and remains somewhat constant with increase in sliding distance. But for rest of the samples B2 and B3, COF decreases as the sliding distance increases, this is due to formation of low friction film of MoS2 over the surface that reduces coefficient of friction, furthermore with increase in wt% of MoS2, thick layer of MoS2 forms at the interface and thus reduces friction. This is also evident from SEM images Figure 4(a,b,c), smoother surfaces due to smearing of MoS2 at the contact interface. The optimum values of COF are achieved at 550m (sliding distance) for all compositions due to smearing of fine powder of MoS2 on the surface and resulting into formation of solid lubricant layer over the surface. The least value of coefficient of friction of sample B3 obtained is 0.0435. This is also evident from SEM images as shown in Figure 4(a,b,c) that smoother surfaces has been formed due to smearing of MoS2 at the contact interface.

The variation of COF with sliding distance for different wt.% of ZrO2 in samples B1, B3&B4 is shown in Figure 3(b). It is noted that COF decreases with increase in sliding distance, as contact increases, ZrO2 nano particles penetration into the rubbing surfaces that, in turn, changes the material properties at the contact points between mating surfaces (the “mending effect”) [14]. Thus, better results are obtained at 550m sliding distance. However composition B4 has least COF at this sliding distance, this is due to ball bearing effect of ZrO2 powder that results into polishing effect on surface as shown by Figure 4(a,c,d) and thus reduces COF. The least value of coefficient of friction of sample B3 obtained is 0.0421. It is because this sample not only has highest wt.% of MoS2 but also highest wt.% of ZrO2 among others.
3.2.2 Effect of load on COF

The values of COF versus load for samples B1, B2 and B3 with increasing wt.% of MoS$_2$ is shown in Figure 5(a). It is observed that as the load increased the value of COF decreased. As fine powdered MoS$_2$ smears on the surface at high loads, leading to smooth surface formation as shown by Figure 6(b,c). It was found that the beneficial effect of IF nanoparticles increased with the load. Exfoliation of external sheets of IF (inorganic fullerene-like supramolecules of metal dichalcogenide MX$_2$; M = Mo, W, etc.; X = S, Se) nanoparticles was found to occur at high contact loads. The transfer of delaminated IF nanoparticles appears to be the dominant friction mechanism at severe contact conditions, which is evident from Figure 6(a,b,c). However, the least COF (0.3565) is obtained at 25N load for composition B3, because MoS$_2$ forms a thick film over the contact surface and reduces COF. The values of COF versus load for samples B1, B3 and B4 with increasing wt.% of ZrO$_2$ is shown in the Figure 5(b). It is observed that, as load increases the value of COF decreases. This is due to formation of fine powdered ZrO$_2$ that easily penetrate into rubbing surface at high loads, that produces ball...
bearing or surface polishing effect of nanoparticles at the interface, thus removes roughness shown by Figure 6(c,d), which in turn reduces COF. The least value of coefficient of friction of sample B4 at load 25N obtained is 0.2041.

![Figure 5. COF Vs Load for (a) B1,B2 and B3 (b) B1,B3 and B4.](image)

![Figure 6. SEM images of worn surfaces of samples (a) B1 (b) B2 (c) B3 and (d) B4 at 25N, 150m and 0.2 m/s.](image)

### 3.3 Wear behavior

#### 3.3.1 Effect of sliding distance on wear

The values of wear rate versus sliding distance for samples B1, B2 and B3 with increasing wt.% of MoS$_2$ is shown by Figure 7(a). It shows initially that the wear rate of virgin sample (B1) is less as compared to B2 and B3, due to presence of MoS$_2$ in the two samples. As sliding distance increases MoS$_2$ shears out and smears over the surface of B2 and B3 thus wear rate of these composites reduces as compared to virgin samples and least values of wear rate is obtained for sample with highest wt.% of MoS$_2$ (B3) at 550 m sliding distance, as MoS$_2$ shears out and smears over the surface forming a film that reduces further wear forming barrier, which is also evident from Figure 8(a,b,c). The least value of wear rate of sample B3 at sliding distance 550 m obtained is $0.8051 \times 10^{-4} \text{mm}^3/\text{Nm}$ with 1 wt.% ZrO$_2$ and 2 wt.% MoS$_2$. 


Also the variation of wear rate with sliding distance for samples B1, B3 and B4 with increasing wt.% of ZrO2 is shown in Figure 7(b). This graph shows initially the wear rate of virgin sample (B1) is less, as compared to, B3 and B4, due to presence of MoS2 among others in the two samples as compared to virgin one. Furthermore, wear rate of sample B3 is more due to harder surfaces provided by sample with 2 wt.% ZrO2 (B4) composition, then afterwards due to formation of fine ZrO2 powder that smear over the surface and also due to ball bearing effect the wear rate further reduces and the samples B3 and B4 shows least wear rates as sliding distance increases. The reduced wear rates are obtained at 550m sliding distance for all compositions with varying wt. % of ZrO2, which is also evident from Figure 8(c,d). The least value of wear rate of sample B4 at sliding distance 550 m obtained is 0.4011x10^{-4} mm^3/Nm with 2% wt. ZrO2 and 2% wt. MoS2.

3.3.2. Effect of loads on wear

The variations of wear rate with loads for samples B1, B2 and B3 with increasing wt.% of MoS2 at constant sliding distance 150m, 0.2m/s sliding speed is shown in the Figure 9(a). The wear rate for virgin samples is more than B2 and B3 due presence of MoS2 in these samples that shear out and forms a film over their surface that reduces further wear at higher loads. Also wear rates for sample B3 are more than sample B4 due to formation of thick protective MoS2 layer over sample B4. The least value of wear rate of sample B3 at load...
25N obtained is $1.1243 \times 10^{-4}$ mm$^3$/Nm with 1% wt. ZrO$_2$ and 2% wt. MoS$_2$, due to low friction film formed by fine powder of MoS$_2$ at high loads, which is also evident from Figure 6(b,c) that smooth surface is formed with slight delamination wear.

The wear rate with different loads for sample B1, B3 and B4 with increasing wt.% of ZrO$_2$ is shown in Figure 9(b). The value of wear rate initially increases due to initial wear then decreases due to formation of protective friction film and with increase in load wear rate decreases due to formation of fine ZrO$_2$ powder that has ball bearing effect, that reduces surface roughness because of its polishing action and thus reduces wear. The least value of wear rate of sample B3 at load 25N is $0.4734 \times 10^{-4}$ mm$^3$/Nm with 2% wt. of ZrO$_2$ and 2% wt. of MoS$_2$, which is also evident from Figure 6(a,c,d), results in formation of more smoother surface.

![Figure 9. Wear rate Vs Load for (a) B1, B2 and B3 (b) B1, B3 and B4.](image)

4. Conclusion

a) In this study, base matrix Fe-5Cu-3Sn, sintered at 1150°C, sintered densities are found more than green densities, as a result of liquid phase sintering, where liquefied Sn and Cu fills the pores by capillary action, thereby improving density after sintering process.

b) Highest microhardness value of 215HV is obtained for composite B4 with highest wt% of ZrO$_2$ in comparison to hardness of virgin sample about 190HV, due to presence of more harder phases formed by ZrO$_2$ with Fe matrix during sintering process.

c) Least value of COF (0.0421) is obtained for sample B4 with highest wt% of MoS$_2$ and wt% of ZrO$_2$ due to formation of solid lubricant layer at the interface, moreover due to polished surface provided by ZrO$_2$ particles as a result of their polishing effect on the surface during sliding motion.

d) Least value of wear rate $0.4011 \times 10^{-4}$ mm$^3$/Nm is shown by sample B4. This is due to higher hardness value of B4, as a result of harder phases that prevents wear, thus reduces wear rate.

g) Wear mechanisms are mostly adhesive wear, third body abrasion due to wear particles, delamination and microploughing in reinforced composites.

h) Thus best results for friction & wear are obtained for sample B4 with 2wt% of ZrO$_2$ & 2wt% of MoS$_2$.

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