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Original Article

Microstructural and sliding wear behavior of SiC-particle reinforced copper matrix composites fabricated by sintering and sinter-forging processes

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ABSTRACT

Cu and Cu/SiC$_p$ composite compacts were prepared through sintering and sinter-forging processes. Influence of SiC particles and fabrication type on the tribological behavior of pure Cu and Cu/SiC$_p$ composites was investigated. Dry sliding wear tests represented that the sinter-forged Cu composite compacts with 60 vol.% SiC exhibit the lowest wear loss compared to other compacts. Moreover, the results indicated that applying compressive force during sintering process of Cu and Cu/SiC$_p$ compacts has a significant effect on reducing and eliminating porosities and achieving to higher bulk density. Therefore, wear loss of the Cu and Cu/SiC$_p$ compacts produced through sinter-forging process was improved significantly compared to conventionally sintered Cu and Cu/SiC$_p$ composite compacts.

1. Introduction

Owing to high electrical and thermal conductivity, low coefficient of thermal expansion (CTE), superior corrosion and oxidation resistance, good ductility and high melting point, metallic copper is one of the most commonly used structural and functional metals which can be utilized for engineering applications [1–3]. Nevertheless, due to its nature, there are some drawbacks, such as, low yield strength, weak creep resistance and low hardness, which can suppress applications of pure copper [1,3,4]. To overcome those limitations, ceramic particulate reinforcement dispersion, like SiC can improve high-temperature mechanical and tribological properties of copper because of high strength, superior wear resistance and high modulus of SiC reinforcement [3,5]. Cu/SiC composites

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combine both the superior ductility and toughness of copper and high strength and high modulus of SiC reinforcements [4,6]. SiC particulate-reinforced copper metal matrix composites (Cu/SiC$_p$ MMCs) potentially can be employed for usages in high temperature structural applications, such as brakes and other severe frictional applications [7], electronic packaging [3,8], electrical contacts [3], resistance welding electrodes [3,9] and high performance switches [4]. The most common routes for particle-reinforced Cu matrix composites include casting and powder metallurgy methods [8–10]. Because of low agglomeration, low segregation and good wettability, powder metallurgy (PM) is preferred to casting [9]. Final processes to achieve a high densification rate of compacts in PM method involve hot isostatic pressing (HIP) or hot pressing (HP). Similar to conventional sintering, isotropic shrinkage can cause densification during isostatic pressing, whereas the shrinkage just in one direction can occur in hot pressing process, which generates shear strain inside compact. This created shear strain during hot pressing increases more effectively the densification than isostatic pressing, which does not generate shear strain. Sinter-forging process is different from hot pressing process in that powder compact is not forced via the walls of the die [11–13]. In sinter-forging, the initial powder is compacted under a uniaxial compressive load without any lateral restriction, i.e. there is no die wall to restrict shear deformation, which can cause densification of compact during sinter-forging, and moreover, shear and radial strain can eliminate flaws and improve densification of compact [11]. The sinter-forging process has been developed by Wakai et al. in 1986 for consolidation of fine-grained yttria-stabilized tetragonal zirconia (YSTZ) ceramic based-materials [14]. The sinter-forging process uses the super-plasticity property of ceramic materials to eliminate large pores, which can be created during sintering. Due to simultaneous deformation and densification of the ceramic compacts during sinter-forging, high density ceramic material can be obtained with a minimum value of grain growth [11–13]. This process has been used to fabricate the ceramic materials like Al_2O_3 [11,12] and their composites. Tjong and Lau [9] studied the tribological behavior of copper and its composites reinforced with SiC particles (5–20%), which were prepared by hot isostatic pressing (HIP) process. Their work represented that the addition of SiC particulates to copper matrix decreases the strain localization range in the subsurface region conducting to volume loss reduction. Kennedy et al. [7] investigated the tribological characteristics of several Cu/SiC$_p$ composites, which were synthesized from copper-coated SiC particles. Their results indicated the interface bond strength worth between matrix and reinforcement particulates, using an intermediate coating on SiC particles resulted in wear rate reduction of Cu/SiC particulate composites. In this study, Cu and its composites reinforced with SiC particles were fabricated by conventional sintering and sinter-forging process, and compared the physical and mechanical properties of the samples like density, hardness and wear resistance.

2. Experimental materials and methods

2.1. Materials

The as-received powder material for the composite matrix was pure atomized and spherical copper powder with an average particle size of 45 μm. SiC reinforcement particulates with a diameter of <10 μm were irregular and angular in shape, both powders from Sigma–Aldrich, Germany with high purity of 99.9%. The morphology of both powder particles is shown in Fig. 1(a) and (b).

2.2. Methods

The metal matrix composites studied in this work, were based-on pure copper reinforced with 20, 40 and 60 vol.% SiC particles. Cu and Cu/SiC$_p$ composites were fabricated through the sintering and sinter-forging processes. For this purpose, Cu and SiC powders were mixed using an attritor to produce composite powders including 20, 40 and 60 vol.% of SiC particulates. Then the composite powders were compacted under a uniaxial stress of 250 MPa at ambient temperature. Compacted powders were sintered at different conditions; all the samples were presintered in an inert atmosphere. Afterwards, on the presintered specimens were performed the sintering and sinter-forging processes to densify Cu and Cu with 20, 40 and 60 vol.% SiC, separately. The samples had disc-shaped with dimension of ϕ15 mm × 5 mm. Table 1 summarizes the various sintering and sinter-forging conditions which used to fabricate Cu and Cu/SiC$_p$ compacts.

The density of Cu and Cu/SiC$_p$ composite compacts was determined according Archimedes' method. In this technique, density is determined by measuring the difference between the specimen weight in air and when it is suspended in distilled water at room temperature. Hardness of both pure copper and composites were determined using a Vickers tester under an applied load of 0.245 N. Dry sliding wear

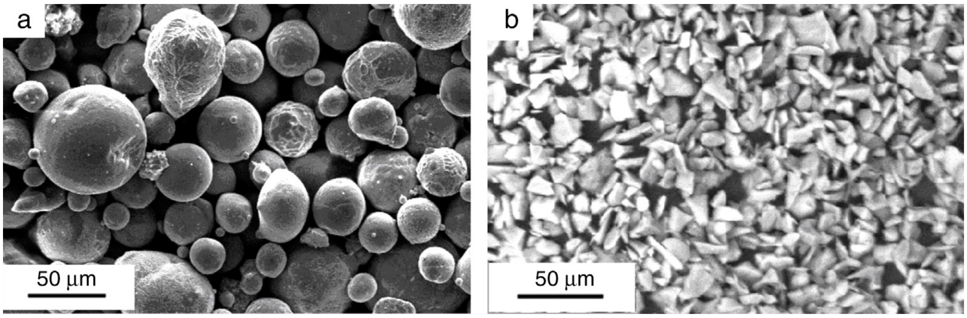

Fig. 1 – SEM micrographs of as-received powders of: (a) Cu, and (b) SiC.

Table 1 – Sintering and sinter-forging conditions for the fabrication of Cu and Cu/SiC$_p$ compacts.

Composition of compacts	Sintering temperature and time	Sinter-forging temperature (°C)	Sinter-forging time (h)	Sinter-forging stress (MPa)
Cu	900 °C, 3 h	600–700	1–3	25–100
Cu-20 vol.% SiC	950 °C, 3 h	750–850	2–6	50–100
Cu-40 vol.% SiC	1000 °C, 3 h	800–900	3–7	75–175
Cu-60 vol.% SiC	1050 °C, 3 h	850–950	4–8	175–250

measurements were performed on pure copper and its composites with 20, 40 and 60 vol.% SiC using the pin-on disc tester at room temperature with relative humidity of 50–60% corresponding to ASTM G99. Cylindrical pins with 5 mm in diameter and 50 mm in length were made from hardened steel with a hardness of HRC 60 and low surface roughness ($R_a < 0.3\,\mu$m) were used as the counterface material. The disc was rotated at 18 mms^{-1}, and the normal loads were 15, 35 and 55 N. The duration and sliding distance of wear test was 2 h and 132 m; respectively. The weight loss of pins and specimens was measured after 30, 70, 100 and 132 m of sliding wear distance in an analytical balance of 0.0001 × g precision. The weight loss was converted to volume loss value. Microstructures were observed with a scanning electron microscope (SEM).

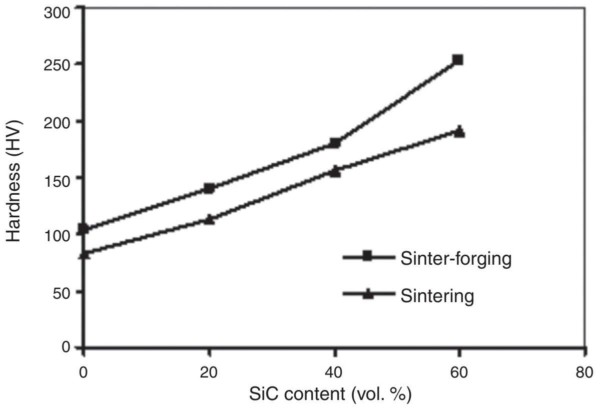

Fig. 3 – Effect of the fabrication type and SiC content on the hardness of Cu and Cu/SiC$_p$.

3. Results and discussion

3.1. *Relative density and hardness of sintered and sinter-forged Cu and Cu/SiCp compacts*

The effect of SiC content and the type of fabrication on the relative density of Cu and Cu/SiC$_p$ compacts is represented in Fig. 2.

Relative density is defined as the (final density of the compact)/(theoretical density of the compact) × 100. Final density of each sintered and sinter-forged Cu & Cu/SiC$_p$ compact was measured through Archimedes method. Theoretical density is defined as the bulk density of fully dense compact without any porosity. As represented, the sinter-forged compacts have the higher density than the sintered compacts. During the sinter forging process two different densification mechanisms can be considered to predict densification behavior of the materials; which are either stress-assisted diffusional mechanisms alone or both stress-assisted diffusional and strain-controlled pore elimination mechanisms [11–13,15,16]. In fact, for the pure copper and the composites including small amounts of SiC particles, both stress-assisted diffusional and strain controlled pore closure mechanism are responsible for the densification behavior of the samples. In addition, High compressive stress, especially during early stages of the sintering process can restrict the expansion of the compacts and effectively help to prevent porosity formation and weakening of the ceramic powder-metal matrix bond [17]. The shear deformation during the sinter-forging brings together the opposite surfaces of large pores and fragments them into smaller pores. In other words, relative density increasing, and as a result, the porosity decreasing are due to the strain-controlled healing of the sintering flaws [11,12,18]. For all the samples, the sintering temperature was higher than the temperature used for sinter-forging process. These results confirm that in the sinter-forging process, stress assisted diffusion and plastic flow induced pore closure mechanisms have a significant effect on the increasing densification rate in pure metallic and metal matrix composite samples. Moreover, with increasing SiC content, the density of compacts decreases, since the sintering capability of the compacts decreases with increasing SiC ceramic particles. Hence, higher density achievement of sintered and sinter-forged Cu and Cu/SiC$_p$ demands high sintering time and temperature.

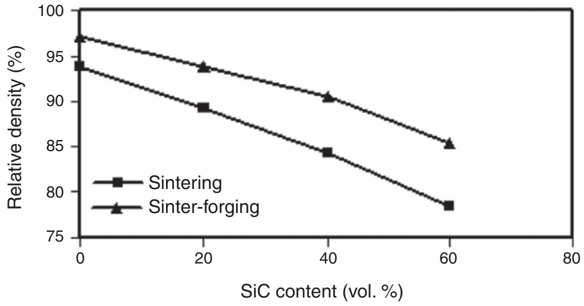

Fig. 2 – Effect of the fabrication type and SiC content on the relative density of Cu and Cu/SiC$_p$ compacts.

The effect of SiC content on the hardness of Cu and Cu/SiC$_p$ compacts is shown in Fig. 3.

As shown, with increasing of SiC particle content, the hardness of the compacts increases. Because of higher hardness value of SiC ceramic particles compared to the soft Cu matrix, the hardness of the compacts increases. As it can be seen, with increasing relative density, hardness of Cu and Cu/SiC$_p$ samples increases, which is a direct effect of decreasing in the percentage of porosities in the microstructure. Comparing the results reported in this work with the same work reported by

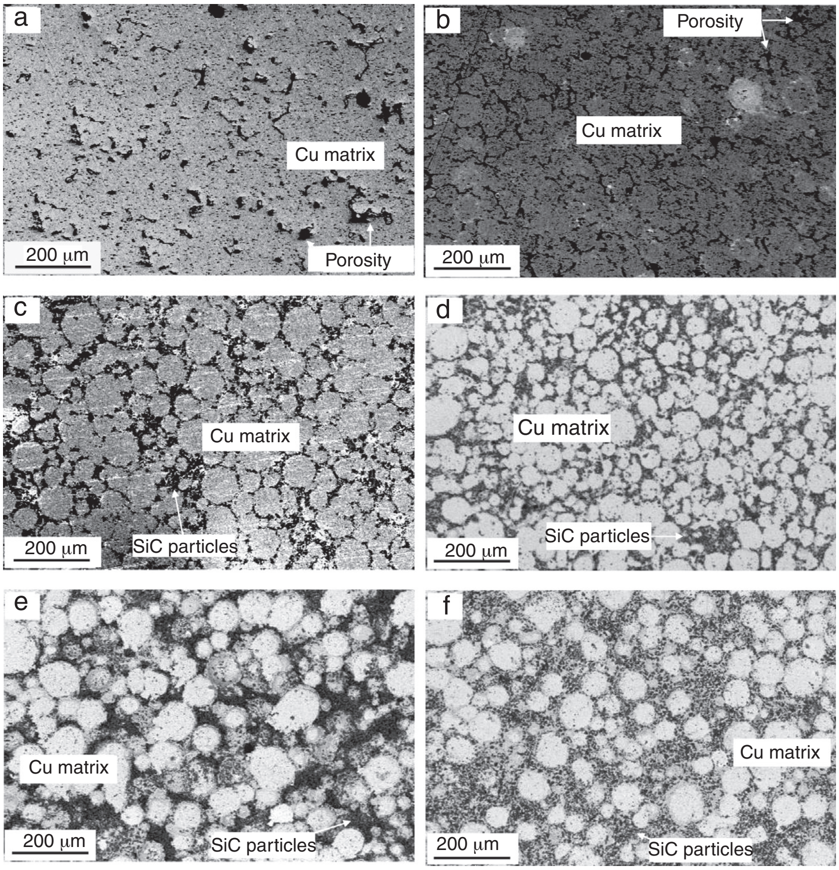

Fig. 4 – SEM micrographs of the sintered and sinter-forged Cu (a and b), Cu-20 vol.%SiC (c and d), and Cu-40 vol.%SiC (e and f) compacts, respectively.

other researchers [9,10], it can be concluded that sinter forging is a convenient process by which, relatively dense samples of copper and Cu/SiC$_p$ composites with reasonable mechanical properties can be obtained. In other words, sinter-forging process is more effective than conventional sintering process for the fabrication of Cu and Cu/SiC$_p$ composites and by this procedure, the higher density can be achieved.

3.2. *Surface morphology of conventionally sintered and sinter-forged composites*

Fig. 4(a), (c), and (e) represent the SEM micrographs of the surface morphology of Cu, Cu-20 vol.% SiC, and Cu-40 vol.% SiC composite samples, which were prepared through the conventional sintering method. The corresponding surface morphology of the samples prepared by the sinter-forging method is presented in Fig. 4(b), (d), and (f).

By comparing the SEM micrograph of a conventionally sintered Cu sample (Fig. 4(a)) with that of a sinter-forged one (Fig. 4(b)) leads to the conclusion that the sinter-forged sample exhibits a fine surface morphology with a lower porosity. The low porosity was also observed for the sinter-forged Cu-20 vol.% SiC (Fig. 4(d)) and Cu-40 vol.% SiC (Fig. 4(f)) when compared to that observed for conventionally prepared samples of the same compositions (Fig 4(c) and (e)). In addition, the composites, which were prepared through the sinter-forging method exhibit less grain growth compared to those prepared by the conventional sintering method. The low grain growth in the samples obtained via the sinter-forging method can further contribute to a decrease in the sintering temperature because of applying the compressive load during the sinter-forging process of Cu and Cu/SiC$_p$ compacts [11,13].

3.3. *Influence of the applied load and sliding distance on the wear behavior of sintered and sinter-forged Cu and Cu/SiC$_p$ compacts*

The influence of the applied normal load on the wear loss of sinter-forged Cu and Cu/SiC$_p$ compacts after the sliding distance of 132 m and sliding velocity of 18 mms^{-1} is shown in Fig. 5(a).

As it can be observed in Fig. 5(a), with increasing the applied normal load during sliding wear tests, the weight loss of pure copper and Cu/SiC$_p$ compacts increases [19,20]. Applied load affects the wear rate of compacts significantly and is the most dominating factor controlling the wear behavior. By increasing the applied load, plastic deformation on the subsurface due to increased penetration depth of counterface can occur. When applied load exceeds form critical load, mild to severe wear can take place, which normally can be characterized by large metallic debris particle creation, which can adhere to counter body and subsurface area [19].

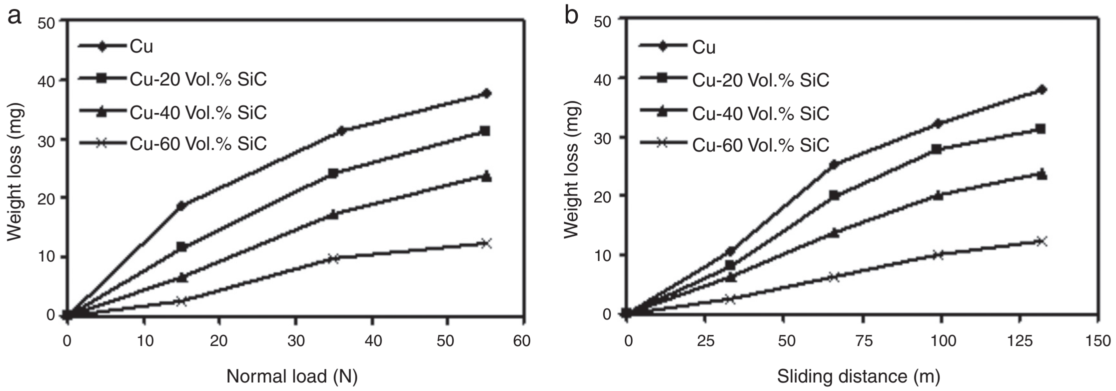

Fig. 5 – The influence of (a) normal load and (b) sliding distance on the weight loss of sinter-forged Cu and Cu/SiC$_p$ compacts at the sliding distance of 132 m and sliding velocity of 18 mms^{-1}.

Fig. 5(b) represents the effect of sliding distance on the wear loss of the sinter-forged Cu and Cu/SiC$_p$ compacts at the normal load of 55 N and the sliding velocity of 18 mms^{-1}. Pure Cu matrix represents a high weight loss during dry sliding wear as expected. It can be seen from Fig. 5(b), that the addition of 20 vol.% SiC to copper is very effective to reduce its weight loss. It can be explained that SiC particle addition increases the hardness of copper considerably and suppresses the wear loss of the compact [1,9]. The weight loss of 20 vol.% SiC$_p$/Cu compacts increases slightly with increasing the sliding distance. As the volume content of SiC reached to 40 vol.% and above, the weight loss shows few changes with increasing the sliding distance [1,9].

Fig. 6 shows the volume loss versus SiC content for the specimens that produced through conventional sintering and sinter-forging processes.

Fig. 6 clearly indicates that Cu/SiC$_p$ composite samples exhibit a lower wear volume loss compared to pure copper. In addition, dry wear resistance of Cu compacts increases with the increasing SiC volume content because of high hardness of SiC particulate ceramic reinforcement, which corresponds to the well-known wear law of Archard, that indicates the wear volume loss of a material is inversely related to its hardness and decreases by increasing of the bulk hardness of the compact in a constant applied load and sliding distance [1,9,21,22]:

$$W_V = \frac{KPL}{H_V} \tag{1}$$

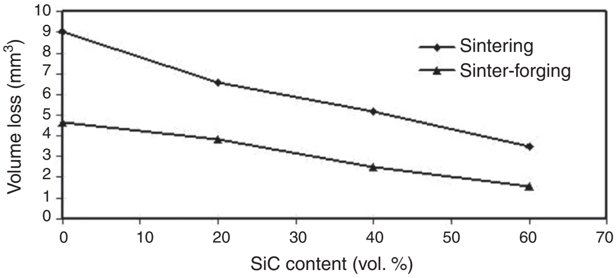

Fig. 6 – Variation of the volume loss versus SiC content for the specimens, which were tested at an applied load of 55 N, a sliding velocity of 18 mms^{-1}, and a sliding distance of 132 m.

where, W_V is the wear volume loss, K is the wear coefficient, P is the normal load, L is the sliding distance and H_V is the Vickers hardness. It can be observed that the variation tendency of the volume loss is in accordance with Archard's wear equation. When the unreinforced Cu samples abraded against hardened steel counterpart, the counterbody enters and sliced deeply the surface of Cu compact, which cause the material removal during the sliding wear and as a result, the continuous grooves and a significantly micro cutting on the worn surface of unreinforced Cu compact can be observed [23].

In addition, Kennedy et al. [7] demonstrated that the main wear mechanism of particulate-reinforced metal matrix composites is delamination wear of soft metallic matrix by plastic deformation, which conducts to the crack nucleation in the soft and ductile matrix. Then this phenomena followed by the propagation and connection of the created cracks results in material removal in the form of delamination platelets or thin sheets, which corresponds to the present work for ductile Cu matrix. High wear resistance of SiC-particulate Cu matrix composite materials ascribed to the hard SiC particles presence in Cu matrix. During the sliding wear tests, hard SiC particles can bear the contact load between the two surfaces, which results in soft Cu matrix protection from wear [7]. Due to high hardness and wear resistance of SiC particles in soft Cu matrix, these hard and wear resistant particles diminish extremely the plastic strain on the worn surfaces of Cu/SiC$_p$ composites [7,23]. Moreover, the wear resistance of sinter-forged compacts is higher than the sintered ones, which is associated with the higher density, lower porosity as well as higher hardness of the sinter-forged compacts. Because of radial and axial strain created during sinter-forging, which resulted in achieved higher density and lower porosity, the mechanical properties such as wear resistance can improve.

3.4. *Morphology of the worn surface of the sintered and sinter-forged Cu and Cu/SiC$_p$ compacts*

Fig. 7 represents SEM micrograph of the worn surface of (a) delamination cracks of the sintered Cu and (b) shear wedges because of the plastic flow after sliding wear at an applied load of 55 N, sliding velocity of 18 mms^{-1} and a sliding distance of 132 m; respectively. Ductile Cu demonstrates basically weak

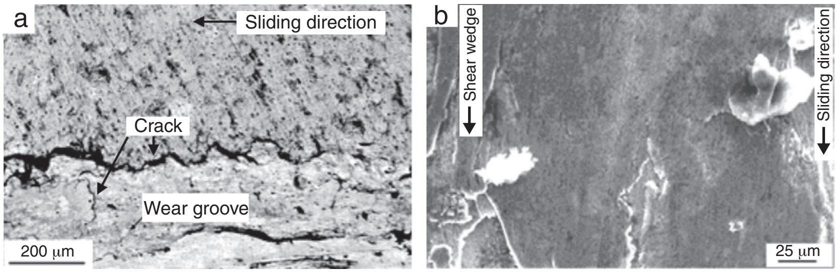

Fig. 7 – SEM images of the worn surface: (a) wear cracks of the sintered Cu and (b) shear wedges due to the plastic flow after sliding wear at the load of 55 N and sliding velocity of 18 mms^{-1}.

wear resistance due to its low hardness nature. Hence, material removal in the form of wear debris is generally appeared on Cu surface during wear against a counterpart body. Wear debris formation mechanism is by the delamination cracks distributed close to Cu subsurface. These delamination cracks are associated with the locally plastic deformation [9]. In addition, since pure Cu is much softer than the steel counterpart, therefore plastic strain localization in the subsurface area can cause delamination crack formation and extreme delamination of Cu surface layers which conducts to a higher volume wear loss, as shown in Fig. 7(a).

Because of the severe surface deformation and flow of soft copper during sliding wear tests, worn surfaces of pure Cu exhibit shear wedge pattern [21], as represented in Fig. 7(b), which can be attributed to soft, ductile and low hardness nature of pure copper matrix.

Fig. 8 (a–f) show the SEM micrographs of worn surfaces at the applied normal load of 55 N for the sintered and sinter-forged Cu, Cu-20 vol.% SiC and Cu-40 vol.% SiC, respectively. The worn surface varies for Cu and Cu/SiC$_p$ compacts and depends on the density, porosity and hardness of the compacts and other parameters, such as, applied normal wear load and sliding velocity. For instance, sliding contact at high loads and high velocities usually leads to severe wear of the surface. Worn surface of the sintered Cu compact is smooth and almost deep and uniform because of high counterbody penetration in Cu matrix. In addition, microcrack and shear wedges due to plastic flow close to subsurface region can be appeared. For the sinter-forged Cu compact, the worn surface is rougher than Cu sintered compact and worn surface is not deep and uniform as sintered Cu compact. This can be attributed to the higher density and hardness of the sinter-forged compacts compared to the sintered Cu compact. Similar to the sintered Cu compact, micro-crack and layer detachment of the surface can be appeared, as shown in Fig. 8(b). For both sintered and sinter-forged Cu-20 vol.% SiC compact, the worn surface is rougher than the worn surface of the sintered and sinter-forged pure copper compact, which can be attributed to the presence of hard and irregular SiC ceramic particle reinforcement. As a result, the removal of soft copper matrix and the wear loss is less than pure copper compact, as shown in Fig 8(c and d). Sintered Cu-20 vol.% SiC compact has uniform and deeper worn surface compared to sinter-forged Cu-20 vol.% SiC compact, which can contribute to its lower density and hardness. For both compacts, wear grooves without an overlay of wear particles or a tribolayer are present on worn surface. The dark-colored spots, which were the original positions of SiC particles can be seen as shown in Fig 8(c and d). By increasing the volume content of SiC particles to 40 vol.%, the worn surface, even becomes rougher than Cu-20 vol.% SiC compact as observed in Fig 8(e and f) and the volume loss decreases compared to Cu-20 vol.% SiC compact. Sintered Cu-20 vol.% SiC compact has uniform and deeper worn surface compared to the sinter-forged Cu-20 vol.% SiC compact, which can contribute to its lower density and hardness. For both compacts, wear grooves and dark-colored region, which were SiC particles place can be seen, besides the concentration of dark-colored area is more than Cu-20 vol.% SiC, as shown in Fig 8(e and f). Moreover, Fig 8(e and f) represent the agglomeration state of wear debris on the worn surface of Cu-40 vol.% SiC, which produced by linking and mixing of the fine worn particles.

High wear resistance of Cu/SiC$_p$ compacts is related to the presence of high SiC ceramic particle content. The ceramic particles have high load bearing capacity, which protect the matrix from the destructive action of the abrasives by reducing the depth of penetration of the abrasives and provide a more effective barrier to subsurface shear by the motion of the hardened steel counterface [24]. Other reason for wear resistance increasing of Cu/SiC$_p$ composites by increasing of SiC content especially at low applied wear loads and velocity can be contributed to the formation of mechanically mixed layer. Mechanically mixed layer are created by the production and breaking of wear particles, mixing and agglomeration, compaction of mixture and wear debris formation between the worn surface and counterpart body during sliding wear. This layer is normally harder than bulk material, and postpones the severe wear occurrence and suppresses the wear deformation extent in subsurface area of the worn surfaces [25,26].

It is clear from Fig 8(a–f) that there was considerable difference between the wear of the sintered and sinter-forged Cu and Cu/SiC$_p$ compacts. The difference in wear behavior of sintered and sinter-forged Cu and Cu/SiC$_p$ compacts depends on the strength of the bond between Cu matrix and SiC ceramic reinforcement. The sintered compacts have deeper and more uniform wear tracks than the sinter-forged compacts. This could be due to a more removed of SiC particles from the surface, related to the higher porosity, lower density and lower hardness of the sintered Cu and Cu/SiC$_p$ compacts compared to the sinter-forged Cu and Cu/SiC$_p$ compacts.

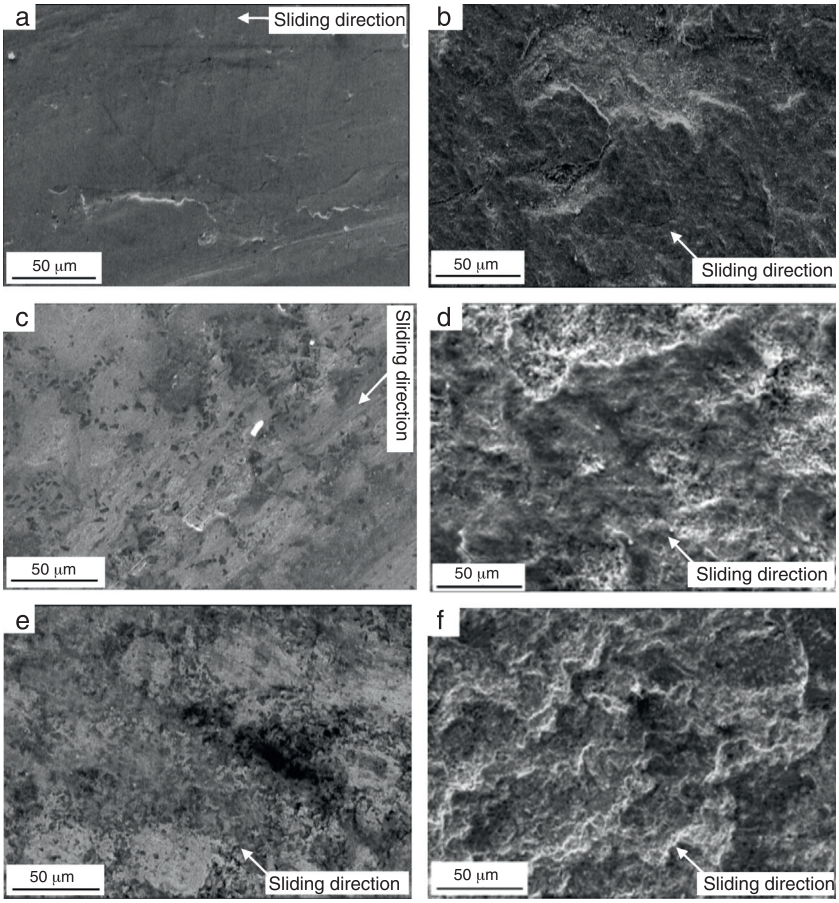

Fig. 8 – SEM micrograph of worn surfaces at the applied normal load of 55 N for the sintered (a, c, e) and sinter-forged (b, d, f) Cu, Cu-20 vol. %, and Cu-40 vol.% SiC; respectively.

4. Conclusions

This investigation provides perceptions into microstructural and sliding wear behavior of Cu and Cu/SiC$_p$ compacts produced through conventional sintering and sinter-forging in powder metallurgy (PM) route. The main conclusions of the present study can be drawn as follows:

- Due to imposed compressive stress during sinter-forging higher density, lower porosity, and higher hardness were achieved compared to the conventional sintering process.
- Sinter-forged Cu and Cu/SiC$_p$ composite compacts exhibit lower wear volume loss than sintered Cu and Cu/SiC$_p$ compacts.

Conflicts of interest

The authors declare no conflicts of interest.

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