Wear properties of copper and copper composites powders consolidated by high-pressure torsion

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Abstract: The wear characteristics of Cu and Cu-SiC composite microsize powders consolidated by cold compaction combined with sintering or high-pressure torsion (HPT) were investigated. The HPT processed (HPTed) samples with bimodal and trimodal microstructures and fine Cu grains and SiC particle sizes have superior hardness, reasonable ductility level, and high wear resistance. The wear mass loss and coefficient of friction of HPTed samples were remarkably lower than that of cold-compacted and sintered samples as well as that of micro and nano Cu and Cu-SiC composites from previous studies. The sample fabrication method has an apparent influence on the wear mechanism. The wear mechanism was converted from adhesive, delamination, three-body mechanism, grooves (take off the SiC particles), and cracks into abrasive wear after HPT. Oxidization can be considered a dominant wear mechanism in all cases. The worn surface morphology and analysis support the relationship between wear mechanism and characteristics.

Keywords: Cu-SiC composite powders; high-pressure torsion (HPT); fine and coarse grains; wear characteristics; worn surface morphology

1 Introduction

The low wear resistance of pure Cu limits its usage in different industrial applications. The improvement of pure Cu hardness and wear characteristics can be performed by different methods including the fabrication of Cu composites [1, 2]. Formation of Cu metal matrix composites (MMCs) improves the mechanical properties of Cu at low and high temperatures [2]. The advantages of particulate MMCs [3] have motivated different studies to improve the mechanical properties of pure Cu reinforced with ceramic particles [4–9]. Although powder metallurgy has many advantages [10], it suffers from various problems [4, 5], which have led to the search for new manufacturing methods for Cu MMCs.

The influence of grain and particle sizes of metals

and MMCs on their wear properties is an important research topic. Understanding the effects of decreasing grain and particle sizes on the wear properties of metals and MMCs manufactured by different methods (e.g., high-pressure torsion (HPT)) was the motivation behind previous works [11–22]. However, contradictory results between the improvement [15–19, 20, 22] and deterioration [21] of wear characteristics of HPT processed (HPTed) materials were obtained.

No previous investigation has focused on the influence of HPT on the wear features of microsize MMC powders. Therefore, a new study on the effect of HPT of Cu and Cu composites on their wear characteristics is required. Present work aims to first, explore the influence of producing Cu and Cu MMCs with a microstructure that gathers between fine and coarse grain and particle sizes on their wear and

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frictional properties. Second, it explores the influence of the sample manufacturing process on morphologies and analyses of worn Cu, Cu MMC, and Tungsten carbide (WC) ball surfaces. Finally, the present results are compared with the wear characteristics of as-cast Cu and previous works of micro, ultrafine grain (UFG), and nanometer microstructures Cu and Cu MMCs.

2 Experimental work

Cu and Cu-SiC MMCs were fabricated by cold compaction combined with sintering or HPT of Cu and SiC powders, as shown in Fig. 1(a) and previously discussed in Ref. [23]. Solid as-cast pure Cu samples were machined and used for further comparison in the present study. Observations of the microstructure and characteristics of different samples were presented in previous work [23]. Microstructure characteristics

(grain and particle sizes), and physical and mechanical properties of the cases are listed in Table 1 [23].

Dry sliding ball-on-flat reciprocating wear tests at sliding distances of 500–2500 m and applied loads of 5–30 N were conducted for all conditions, as shown in Fig. 1(b). The wear test conditions, parameters, and determination of the wear mass loss have been discussed previously [20]. The wear test was repeated five times for each condition and the average wear mass loss value was used. The influence of SiC% and the applied load under different sliding distance on wear mass loss was plotted in the form of the grid pattern using OriginPro8 and a so-called wear map. The wear mass loss values in the wear map were recorded by following a regular grid pattern with placement/plotting of the SiC% and applied load values along X- and Y-axis, respectively. Worn surface morphologies and analysis were conducted by scanning electron microscope (SEM).

Fig. 1 Macrographs of (a) Cu and Cu-SiC composites preparation flow chart and as-cast Cu sample with the position of the wear test, and (b) the ball-on-flat surface reciprocating sliding wear test.

Table 1 Summary of microstructure, relative density, and mechanical properties parameters of the different samples [23].

Parameter	Sample									
	Cu as-cast	Cu cold compacted	Cu-10%SiC cold compacted	Cu-20%SiC cold compacted	Cu sintered	Cu-10%SiC sintered	Cu-20%SiC sintered	Cu HPT	Cu-10%SiC HPT	Cu-20%SiC HPT
SiC particle size rang (μm)	–	–	56–12.6	56–12.6	–	56–12.6	56–12.6	–	0.128–0.248	0.071–0.104
SiC average particle size (μm)	–	–	25.5	25.5	–	25.5	25.5	–	0.137	0.078
Cu grain size rang (μm)	30.21–12.34	67.94–15.02	49.99–12.34	46.04–11.42	59.29–7.98	44.96–4.43	44.68–3.61	2.25–0.05	2.12–0.04	0.76–0.03
Cu average grain size (μm)	27.61	29.5	25.9	24.2	26.1	22.5	21	0.95	0.9	0.3
Percent of UFG and nano Cu grain sizes (%)	–	–	–	–	–	–	–	54	62	100
Relative density (ρ) (%)	100	97.8	96.4	94.6	98.9	98.4	95.5	99.7	99.6	99.4
Microhardness, Hv	68	63	106	119	84.5	122.3	138.6	190.9	276.1	328.5
Elongation (%)	–	–	–	–	16.9	9.1	7.9	12.9	7.8	7

3 Results and discussion

3.1 Wear properties

3.1.1 Wear mass loss

The wear mass loss curves shown in Fig. 2 indicate

50%–775% and 100%–900% increase in wear mass loss with increasing of load and distance, respectively. On another hand, the increase in SiC% decreased the mass loss owing to the increase in hardness [4–6, 9, 13, 14, 23]. The wear mass loss of different samples decreased by 27%–85% after the sintering process under

Fig. 2 The effect of the applied load on the wear mass loss of the as-cast Cu, cold compacted, sintered, and HPTed Cu and Cu-SiC samples under different sliding distances (a) 500, (b) 1,500, and (c) 2,500 m.

different conditions owing to the 34%–17% increase in microhardness, as shown in Table 1. This increase in microhardness after sintering is attributed to the decrease in void content; hence, bonding improved because of Cu diffusion [23]. Interestingly, the wear mass loss increased with the increase in the load at different sliding distances. However, the percentage increase in mass loss (in the form of the slope of wear mass loss curves) becomes clearer under applied loads from 20 to 30 N and increasing distance. The increase in sliding distance with higher applied load contributes to severe wear, which is more obvious in cold-compacted and sintered samples with lower hardness than in HTPed samples. The wear mass loss decreased at a higher rate after HPT than after the sintering process. The higher wear resistance obtained after HPT processing compared with cold compaction and sintering is attributed to the increase in hardness [24] achieved through the formation of a fully-dense microstructure with fine Cu grains and SiC particles, as shown in Table 1.

The high capability of HPT in improving wear resistance compared with other processing methods was also proved by drawing wear mass loss maps. The wear maps clarified the correlation between the sample manufacturing process and wear mass loss under different loads, SiC content, and distance of 2500 m, as shown in Fig. 3. The mass loss gradually decreased with the increase in SiC content for different cases. The mass loss ranged from 90 to 20 mg after cold compaction and sintering, which is 94.4% and 75% higher than the HPTed case.

The decrease in wear mass loss after HPT processing observed in Figs. 2 and 3 is the result of Cu grain refinement and SiC particle fragmentation, as shown in Figs. 4(j)–4(h). The influence of fabrication methods on the Cu grain and SiC particle sizes is reflected in their microstructures, as shown in Fig. 4 and Table 1. Interestingly, HPTed Cu samples with grain size of 2.16–0.05, 2.12–0.04, and 0.76–0.03 μm ; and SiC particle size of 0.128–0.248 and 0.071–0.104 μm enables them to have multigrain-size microstructures.

Fig. 3 Wear mass loss map of (a) cold compacted, (b) sintered, and (c) HPTed Cu and Cu-SiC composite samples under different applied load and sliding distances of 2,500 m.

Fig. 4 OM photomicrographs of the microstructure of (a) Cu as-cast, (b) Cu, (c) Cu-10, and (d) 20%SiC cold compacted samples, (e) Cu, (f) Cu-10, and (g) 20%SiC sintered samples, and EBSD color-coded orientation map images of (h) Cu, (i) Cu-10, and (j) 20%SiC HPTed samples.

These types of microstructures (bimodal or trimodal) improve the wear resistance of Cu MMCs compared with those with only micro- or nanomatrix grains and reinforcement particle sizes [6, 9, 21]. The high wear properties of the HPTed samples are also proved by comparing them with previous works [4, 6, 7, 9, 11–14, 17, 19, 25–27] as follows.

Figure 5 shows the comparison between the hardness and wear mass loss of Cu and Cu MMCs in the

present work and Cu and Cu MMCs samples with microsize microstructure. It noted that the hardness and wear resistance of HPTed Cu and Cu MMCs were higher than those with matrix and reinforcement with sizes in the range 10–75 μm and hardness values in range 80–140 Hv [4, 6, 7, 9, 13, 14, 25]. This observation can be explained by the effects of the grain and particle refinement, which contribute to improving hardness, and hence reduced wear mass loss.

Fig. 5 Comparison between (a) hardness and wear mass loss of HPTed (b) Cu and (c) Cu-SiC samples with previous works concerned with fabrication of micro size Cu and Cu composites samples.

The other part of the comparison is concerned with the comparison between the results of the present work and the Cu and Cu MMCs with UFG and nanosize microstructure [11, 12, 14, 17, 19, 26–29], as shown in Fig. 6. It noted that HPT processing reduces the wear mass loss of Cu by 55%–100% compared

with that of Cu and Cu alloy with 50–800 nm grain sizes [11, 12, 14, 17, 19]. Thus, the Cu produced with a mixed microstructure of large and fine grain sizes (bimodal microstructure) is effective in improving wear resistance. Grain refinement contributes to the improvement of hardness, as shown in Figs. 5(a)

and 6(a) and Table 1.

Although grain refinement increases the hardness and wear properties, it simultaneously decreases the ductility of samples [11, 12, 14, 17, 26–29]. The ductility decrease can be attributed to the increase in the grain boundary area [23]. The grain boundary area increase hinders dislocation movements which could accumulate within the grains that contribute to the enhancement

of strength (hardness). Similar observations of the ductility decrease combined with grain refinement down to UFG or nanograin sizes owing to the effect of the increase in grain boundary area were previously noted in Refs. [30, 31]. Moreover, sliding during the wear test increased the strain hardening of the worn surface, increasing the possibility of crack formation and damage. Thus, the combination of hardness and

Fig. 6 Comparison between (a) hardness and wear mass loss of HPTed (b) Cu and (c) Cu-SiC samples with previous works concerned with fabrication of UFG and nanosize Cu and Cu composites samples.

ductility through the formation of a bimodal microstructure is needed to improve wear resistance, i.e., a bimodal microstructure with a mixture of fine grains (that increase the hardness) and large grains (that improve ductility). Interestingly, the formation of such a microstructure conserves 80% of HPTed sample ductility, as indicated in Table 1, which reduces the degree of delamination and crack formation, resulting in reduced wear mass loss similar to that noted previously in Res. [29, 32].

The HPTed Cu MMCs have 25%–100% higher wear resistance than that of the MMC with ultrafine and nanomatrix grains and reinforcement sizes [11, 12, 14, 26–28], as summarized in Fig. 6(c). The high wear properties of HPTed Cu MMCs compared with MMCs with nanomatrix grains and reinforcement sizes [11, 12, 14, 26–28] are because of the following reasons. The HPTed Cu composites were fabricated using microsize SiC particles that enable the HPTed Cu MMC samples to acquire SiC% (reaching 20%) higher than 2.04%–18% achieved previously in Refs. [11, 12, 14, 26–28]. Furthermore, the degree of agglomeration decreased with a noticeable homogenized distribution of reinforcement, starting with microsize SiC particles compared with that noted in the case of MMCs fabricated using UF and nanosize materials [22, 23]. Finally, similar to the Cu samples, the formation of trimodal microstructure enhanced hardness by 300% and retained approximately 85% of the ductility of Cu MMC samples after HPT processing for improved wear resistance. The effects of combining high hardness with acceptable ductility level, which improve the mechanical and wear properties of the material, have been noted in previous works [29, 32–34].

The cold compaction of pure Cu powder was not effective in enhancing the wear resistance of pure Cu compared with as-cast Cu. The wear resistance of as-cast Cu was 54%–82% higher than that of cold-compacted Cu under different conditions owing to the low microhardness of cold-compacted samples. The hardness and wear resistance of cold-compacted Cu samples increased after sintering and HPT processing, with reduced wear mass loss of 10%–30% and 87%–74%, respectively, compared with as-cast Cu samples. Therefore, sintering and HPTed processing of Cu powder can produce Cu samples with superior wear properties than that of solid Cu samples.

3.1.2 Friction properties

Figures 7(a)–7(d) show the curves of the coefficient of friction (COF) of Cu samples. The COF of the as-cast Cu sample increased to its maximum value at 200 m,

Fig. 7 Variation of COF under different applied loads and sliding distances of 2,500 of (a) as-cast, (b) cold compacted, (c) sintered, and (d) HPTed Cu samples.

then oscillated around the maximum value as shown in Fig. 7(a). COF values of different Cu samples exhibit the same behavior, as shown in Fig. 7. Interestingly, the variation range of COF values of HPTed Cu samples decreased because of the increased hardness due to grain refinement [13, 17, 18, 20, 29]. The behavior of COF of Cu MMCs processed by different methods was similar to that noted in the case of Cu samples, but with smaller variation range owing to the higher hardness of Cu MMCs, as shown in Fig. 8 and previously noted in Refs. [8, 13, 14, 27, 28, 35].

The average COF decreased by 5%–40% for all cases with increasing applied load, as shown in Fig. 9 and previously noted in Refs. [6, 7, 8, 17, 21, 35]. On the other hand, the increasing distance increased COF by 1%–30% owing to the deterioration of sample surfaces [7]. The average COF values of Cu and Cu composites decreased after sintering and HPT processing. The magnitude of the decrease in COF after HPT was greater than that of sintered samples. The COF decreased by 20%–40% and 50% after sintering of cold-compacted Cu and composite samples, respectively. On the other

Fig. 8 Variation of COF under different applied loads and sliding distances of 500 m (a–c) and 2,500 m (d–f) of (a and d) cold compacted, (b and e) sintered, and (c and f) HPTed Cu-20%SiC samples.

Fig. 9 The effect of the applied load on the average value of the COF of the as-cast Cu, cold compacted, sintered, and HPTed Cu and Cu-SiC samples under different sliding distances of (a) 500, (b) 1,500, and (c) 2,500 m.

hand, the COF of HPTed samples were 50%–60% lower than those of sintered samples. The COF decrease after sintering and HPT processing is attributed to the

increase in hardness. However, the COF of HPTed samples was the lowest owing to its high efficiency in grain size decrease, which increased hardness more obviously compared with other methods, as previously noted in SPD of different alloys [17, 18, 20]. Moreover, the samples that contained a mixture of different grain sizes retained a good level of ductility to decrease the degree of damage, and hence the COF.

3.1.3 Worn surface morphology of copper, copper composites samples, and WC ball

It can be noted that delamination and adhesion were the dominant wear mechanisms of cold-compacted Cu samples, with delamination wear increasing with distance, as shown in Fig. 10. The delamination of the surface layer of the sample can be explained as follows. During the sliding of the WC ball due to Cu softening, Cu particles were detached from the sample surface and adhered to the ball surface. Then, as the ball continuous to slide, the Cu particles were returned/re-transferred to the sample surface, forming the delaminated layer (SEM observation of the WC of the ball surface confirmed the results shown in Fig. 10(g)). Furthermore, the worn surface of cold-compacted MMC samples is also characterized by grooves (due to take off the SiC shown by yellow arrows) and cracks (indicated by red arrows), as shown in Fig. 10. The take off of SiC particles can be explained by low bonding between the matrix and SiC particles, which contributes to the presence of the three-body wear mechanism between the sample surface, WC ball, and SiC particles. This type of the mechanism increases the wear mass loss of cold-compacted composites compared with other materials, as shown in Figs. 2 and 3. The degree of adhesive and delamination wear increased with load, as shown in Fig. 10 and noted previously in Refs. [4, 6, 7, 9]. Oxygen was clearly detected in different cold-compacted samples, as shown in Figs. 11(a) and 11(d), which means that oxidation should be considered as a possible wear mechanism of cold-compacted samples, as previously noted in Ref. [6].

The surface of the wear test ball was not affected by the wear test with cold-compacted Cu MMC sample. Small localized areas with adhesion and detachment of Cu on the ball surface were observed, as shown in Fig. 10(g). The transfer and adhesion of Cu onto the

Fig. 10 SEM photograph of the worn surface morphology of Cu (a and d), Cu-10 (b and e), and 20%SiC (c and f) cold compacted samples corresponding to a sliding distance of 2,500 m and applied loads of 5 N (a–c) and 30 N (d–f), and (g–h) the corresponding WC wear surface morphology after the sliding against Cu-20%SiC cold compacted sample under sliding distance of 2,500 m and an applied load of 30 N under different magnifications.

Fig. 11 EDS analysis of worn surface of (a–c) Cu and (d–f) Cu-20%SiC cold compacted (a and d), sintered (b and e), and HPTed samples (c and f) corresponding to a sliding distance of 2,500 m and an applied load of 30 N

WC ball surface hindered the ball from sliding against the cold-compacted samples, which confirms the high COF of cold-compacted samples as shown in Figs. 7–9. High magnification of the WC ball surface revealed a polished surface with adhesion of Cu in a dark color, as shown in Fig. 10(h). The ball surface was also free of plastic deformation bands or scratches.

The wear mechanism of the sintered Cu sample is a combination of adhesive, abrasive, and low degree of delamination with increasing adhesive wear, applied load, and sliding distance, as shown in Fig. 12. The worn surface of the sintered Cu composite sample was free of grooves and cracks observed in cold-compacted samples. These observations confirm the capability of the sintering process in improving wear resistance. Oxygen was found in the analysis of worn sintered

samples, as shown in Fig. 11, which makes oxidation the wear mechanism of sintered samples. Moreover, tungsten (W) with percentage of 11.84% was found in the analysis of the surface of worn sintered Cu MMC samples. This observation is attributed to the increase in bonding; thus, the hardness of the Cu-SiC after the sintering process makes it capable of removing a significant amount from the WC ball. After the sintering process, the wear of the WC ball becomes clear with an area of 1.23 mm², as indicated in Figs. 12(g)–12(h). The ball used in testing the sintered Cu MMCs has a rough surface with the presence of voids, as shown in Fig. 12(h), and confirmed by W transfer to the wear sample by EDS of sintered Cu MMCs.

Figure 13 shows the worn surface morphology of the HPTed samples under applied loads of 5 and 30 N

Fig. 12 SEM photograph of the worn surface morphology of Cu (a and d), Cu-10 (b and e) and 20%SiC (c and f) sintered samples corresponding to a sliding distance of 2,500 m and applied loads of 5 N (a–c) and 30 N (d–f), and (g–h) the corresponding WC wear surface morphology after the sliding against Cu-20%SiC sintered sample under sliding distance of 2,500 m and an applied load of 30 N under different magnifications.

Fig. 13 SEM photograph of the worn surface morphology of Cu (a and d), Cu-10 (b and e), and 20%SiC (c and f) HPTed samples corresponding to a sliding distance of 2,500 m and applied loads of 5 N (a–c) and 30 N (d–f), and (g–h) the corresponding WC wear surface morphology after the sliding against Cu-20%SiC HPTed sample under sliding distance of 2,500 m and an applied load of 30 N under different magnifications.

and 2,500 m. The worn surface morphology of the HPTed samples can be characterized by abrasive wear, which is considered the main wear mechanism of HPTed samples. It is also noted that the worn surface of HPTed samples has a more homogenized distribution of SiC particles than in other cases, which contributes to the increase in wear resistance. Interestingly, no grooves and cracks were found in the HPTed Cu-SiC case owing to its strong bonding, as shown in Fig. 13. The formation of microstructures with multiple grain and particle sizes is the main reason for the change of the wear mechanism to abrasive wear after HPT. The refinement of Cu grain size with the fragmentation of SiC particles increased the hardness, and thus transformed the wear mechanism into abrasive wear [4, 9, 17–20, 25–27, 29]. Furthermore, the microstructure with multiple grain and particle sizes [34] decreased the degree of wear compared with that in the nanocomposites [9, 11, 27, 28, 35].

The analysis of HPTed Cu and Cu MMC samples indicate the presence of W, as noted in Fig. 11. Moreover, the amount of W in HPTed Cu MMC sample surface was two times that in the sintered sample shown in Fig. 11(e). The high W content on the surface of HPTed samples is due to their higher hardness. Furthermore, oxygen was found, as shown in Figs. 11(c) and 11(f), which makes oxidation a wear mechanism in all samples in the present work. Severe wear with a large area of 2 mm² was noted in the case of the WC ball after wear test of HPTed MMCs, as shown in Figs. 13(g) and 13(h). Therefore, HPTed samples have a higher effect on increasing the wear of the WC ball owing to its high hardness, which transfers a large amount of W onto the sample surface, as shown in Fig. 11(f).

Figure 14 shows SEM photo micrographs and EDS of the as-cast Cu sample after wear test under applied loads of 5 N and 30 N and sliding distance of 2,500 m.

Fig. 14 SEM photograph and EDS analysis of worn surface of the as-cast Cu corresponding to sliding distance of 2,500 m and applied loads of (a–b) 5 N and (c–d) 30 N.

Fig. 14 (Continued)

The dominant wear mechanisms in as-cast Cu samples under different conditions were delamination and adhesive wear. It was also observed that the wear in worn cold-compacted Cu was more severe than that of as-cast Cu sample, as shown in Figs. 10(a) and 10(d), and Figs. 14(a) and 14(c), respectively. The high wear resistance of as-cast Cu sample relative to cold-compacted Cu is due to its higher hardness. On the other hand, the wear of as-cast Cu samples under applied loads of 5 N and 30 N and sliding distances of 2,500 m was greater than that of Cu HPTed samples under same conditions, as shown in Figs. 14(a) and 14(b). Therefore, the HPTed Cu samples have higher wear resistance compared with as-cast, cold-compacted, and sintered samples. The EDS analysis confirmed oxidation as the wear mechanism of as-cast sample. There was no evidence of the presence of W on the as-cast Cu sample surface.

Therefore, the wear mechanisms can be summarized as grooves (due to the SiC particle take off) that lead to three-body wear mechanism, delamination, and

cracks in the cold-compacted samples. With the increase of bonding and thus hardness after the sintering process, the wear mechanism changed to a lower degree of delamination, with the presence of adhesive and abrasive wear mechanisms. Finally, after HPT processing, the wear mechanism was abrasive wear owing to the superior hardness of HPTed samples. Interestingly, for all cases, oxidation was main wear mechanism as indicated by EDS under all wear conditions.

4 Conclusions

In the current research, it can conclude that:

(1) HPT of Cu and Cu MMCs with microsize powders improves their wear resistance. The formation of a microstructure with different grain and particle sizes decreases the wear mass loss after HPT by 70%–97% compared with samples fabricated by other methods.

(2) HPTed samples have higher wear resistance (by

25%–100%) than Cu and Cu MMCs consisting of large and fine matrix grains and reinforcement particles in the present and previous works.

(3) The COF of the cold-compacted Cu and Cu composites decreased by 2%–40% and 32%–60% after sintering and HPT, respectively, because of the change in material processing method, and thus the hardness increased.

(4) The sample fabrication method has an obvious influence on the wear mechanism.

(5) The SEM photo micrographs and EDS patterns of the wear samples and WC ball confirm the wear mass loss, friction, and wear mechanism results.

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