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Submicron SiCp/Cu composite was fabricated by means of powder metallurgy route followed by hot extrusion, and then its morphology and mechanical properties were investigated. The results showed that densification of the composite was achieved by this fabricating process when the content of submicron SiC particle was below 5 vol.%. Furthermore, the friction and wear properties of all the submicron SiCp/Cu samples were carefully investigated with lubricant of 20# machinery oil. It was found that 5 vol.% SiCp/Cu samples exhibit excellent wear properties. This phenomenon can be explained by the following fact that a new kind of tribolayer is formed, in which submicron SiC particles are well dispersed during the wear test. In addition, the wear mechanism under lubricated condition was also discussed.

Keywords: metal-matrix composite; tribophysics; wear testing; lubricated wear including scuffing

1. Introduction

Copper-based metal matrix composites (CMMCs) have received great attention because of their improved wear resistance, high electrical and thermal conductivities, and low coefficient of thermal expansion. These properties made CMMCs to be attractive materials for a wide range of applications, such as make-and-break electric switches, heat exchangers, and sliding contact materials.[1–17] It is well known that SiC particles have the advantage of high hardness and electric conductivity compared with the other reinforcement particles. Therefore, CMMCs reinforced by SiC particles have attracted the attention of many researchers.[11–17] The production of CMMCs involves powder metallurgy (PM) and ingot casting. The ingot casting technique received a great deal of attention for their low manufacturing cost, but it also suffered from the following drawbacks: poor wettability between molten copper and ceramic particles and nonuniform distribution of the particles due to the agglomeration.[1] PM route could minimize these drawbacks. However, the composites fabricated by regular PM processing had the disadvantage of high porosity, which would dramatically decrease the strength of composites. The recently developing hot isostatic pressing (HIP) could eliminate all the drawbacks mentioned above,[11–13] but the complicated process and the high cost of the HIP technique make it difficult for wide industrial applications. In order to find out an

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effective method which has both low cost and good mechanical properties, Zhan et al. have developed a complex process. This complex process comprises the regular PM route followed by hot extrusion.[16]

For the potential application of CMMCs reinforced by SiC particle, the tribological and friction properties are especially important and have been studied by a number of authors.[12–16] However, this paper mainly focuses on the friction and wear properties under unlubricated conditions. Xue [18] reported the SiC whisker and molybdenum particle-reinforced aluminum matrix composites under lubrication, an increase of 50% in load-carrying capacity was found. The friction and wear behaviors of the SiC particle-reinforced CMMCs under lubricated conditions are still not reported. Moreover, the sizes of SiC particles mentioned above are all larger than 10 μm, and submicron SiC particle-reinforced CMMCs are still not taken into consideration.

The purpose of this article is to understand the effect of submicron SiC particle on the friction and wear properties of CMMC, and three aspects of this problem are addressed. First, submicron SiCp/Cu was fabricated by using the PM route followed by hot extrusion. Second, the mechanical and physical properties were tested. Third, particular attention was paid on the effect of load on friction, and on wear of the resulting composites under oil-lubricated condition. Finally, the formation and behavior of tribolayer were analyzed. These topics discussed in this paper will provide some important insights for researchers in the field of surface modification.

2. Experimental details

2.1. Composite preparation

Surface-clean Cu powders (6 ~ 10 μm in diameter) and SiC particles (130 nm in diameter) were prepared. The composites with 0.5, 1, 1.5, 5, and 10 vol.% SiC were fabricated by means of the PM route followed by hot extrusion as follows. Firstly, the Cu and SiC powders were well mixed mechanically. Secondly, the mixed powders were compacted in a dye with a pressure of 400 MPa. Thirdly, the compacted disk-like samples with 50 mm in diameter and five mm in thickness were sintered under a reducing atmosphere at 1223 K for six hours and the atmospheric pressure was 1.5 atm. Finally, extrusion at 673 K was applied to produce bars with a diameter of 10 mm, and the extrusion ratio was 10:1.

2.2. Mechanical and physical properties

The ultimate strength and percentage elongation of specimens were determined by a WDW-200 tensile tester. Microhardness of the composite was determined by a HV-1000 Vickers tester under an applied load of 20 g before and after the wear test. The in-depth microhardness profile was tested on the cross section.

2.3. Microstructure observation

The morphologies of the specimens were characterized by a scanning electron microscope (JXA-840A) equipped with energy dispersive X-ray analysis (AN10000).

2.4. Friction and wear test

Friction and wear tests were conducted under laboratory conditions by a block-on-ring MM1000 wear tester system, as shown in Figure 1. The sliding ring was made of 5140 steel

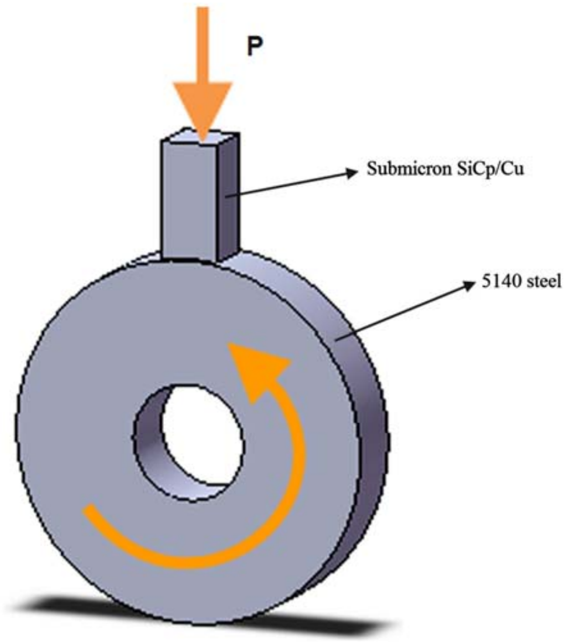

Figure 1. Schematic setup of block-on-ring pair.

with hardness of HRC 40. The dimension of the test specimens was $8 \times 8 \times 20$ mm, and the square faces (8×8 mm) of the specimen with one side parallel to the sliding direction were put in contact with the slider. The tests were carried out at a sliding velocity of 10 m min^{-1} under constant applied load levels of 150–1200 N and a lubricated condition with 20# machinery oil (20 drops each minute). The sliding distance was about 2200 m. The weight loss of the specimens was measured in an analytical balance of $0.0001 \times$ g precision. The specimens were well cleaned before and after the wear tests. For comparison, the wear tests of pure Cu, 0.5 vol.% SiC, 1.0 vol.% SiC, 1.5 vol.% SiC, 5.0 vol.% SiCp/Cu composites, and C51100 bronze were performed.

3. Results and discussion

3.1. Relative density, mechanical properties, micrograph, and fractograph

The relative density of SiCp/Cu was determined by Archimedes' method. According to this method, relative density was calculated by the ratio of tested density and theoretical density. The values of the relative density of the SiCp/Cu composites with reinforcement content are shown in Table 1. It can be seen that the relative density is close to the theoretical value at low reinforcement content, that is 98.5%. When increasing the reinforcement concentration to 5 vol.%, the relative density decreases to 95.4%. The relative density of 10 vol.% SiCp/Cu composite reduces to 92.9%. Figure 2 shows the morphology of 5 vol.% SiCp/Cu composite. There are no dimples on the surface of the specimen. This indicates that densification is achieved during the hot extrusion process. For the relative high surface energy of the submicron SiC particles, they exist in an agglomeration state in Cu matrix. It can be seen that it is difficult to soften Cu to flow through the agglomerate and rigid submicron SiC particles by the PM route. And the agglomeration of SiC particles becomes severe with the increment of

Table 1. Values of relative density and porosity% of SiCp/Cu composites.

Material composition	Relative density	Porosity (%)
0.5 vol.% SiCp/Cu	0.985	1.5
1.0 vol.% SiCp/Cu	0.981	1.9
1.5 vol.% SiCp/Cu	0.974	2.6
5.0 vol.% SiCp/Cu	0.954	4.6
10 vol.% SiCp/Cu	0.929	7.1

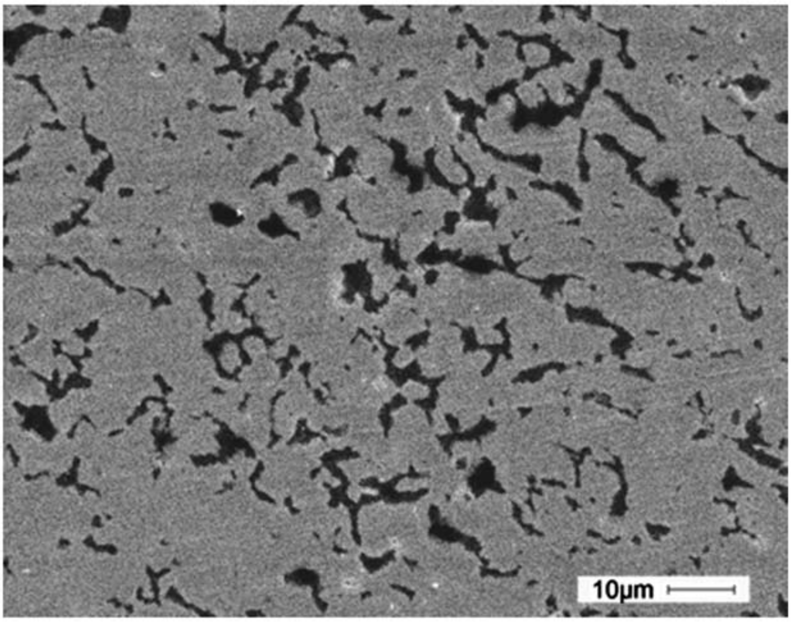

Figure 2. Microstructure of Cu-5 vol.% SiC composite.

Table 2. Mechanical properties of submicron SiCp/Cu.

Material composition	Ultimate strength (MPa)	Percentage elongation (%)	Vickers microhardness (HV)
0.5 vol.% SiCp/Cu	208	42	65
1.0 vol.% SiCp/Cu	210	38	70
1.5 vol.% SiCp/Cu	225	36	75
5.0 vol.% SiCp/Cu	187	8.6	82
10 vol.% SiCp/Cu	95	2.1	85

the SiC content. So the porosity of the composites increases rapidly with the increment of the SiC particles. The agglomerated SiC particles form a strip-like shape as shown in Figure 2, which is due to the effect of the hot extrusion process.

The mechanical properties of samples are shown in Table 2. The microhardness of composites is improved considerably and the ductility decreases with the increasing content of the SiC particles. The ultimate strength of the composites increases with the content of SiC,

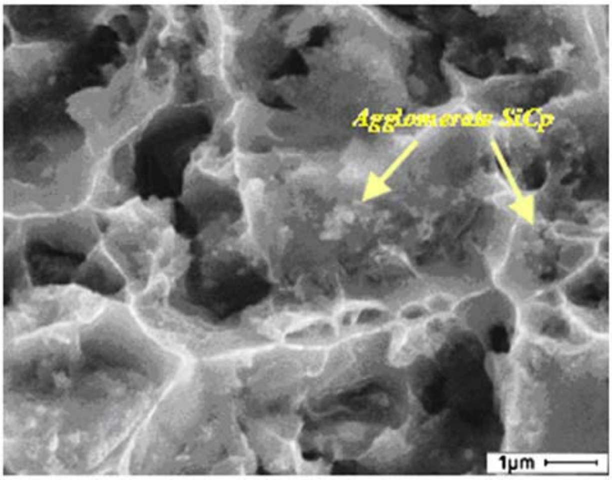

Figure 3. Fractograph of 5 vol.% SiCp/Cu composite after tensile test.

increasing between 0.5 ~ 1.5 vol.%, and then decreases with further more additives of the SiC particles. When the content of SiC increases to 10 vol.%, the ultimate strength of the composites is 50% less than that of 5 vol.% SiCp/Cu composite. Associated with Table 1, these indicate that the relative density of composites decides the mechanical properties such as yield strength and elongation. As the volume content of submicron SiC increases from 0.5 to 1.5 vol.%, the ultimate strength is enhanced because of the well-dispersed submicron SiC particles. The ultimate strength and elongation will decrease with further more addition of the SiC particles because of the agglomeration of the submicron SiC.

Table 2 shows the good integrated mechanical properties of the 5 vol.% SiCp/Cu. Therefore, the 5 vol.% SiCp/Cu is most concerned in this paper. Figure 3 shows the fractograph of 5 vol.% SiCp/Cu composite. The agglomeration of the submicron SiC particles can be observed. In addition, the nature of failure appears to be ductile and intergranular.

3.2. Friction and wear

Figure 4 shows the friction coefficient tested under different applied loads. The friction coefficients are very low ($\mu = 0.05$–0.07), when the applied loads are less than 600 N. While the applied load increases to 1200 N, the friction coefficient rises to a rather high standard ($\mu = 0.25$–0.34) and is unstable. It is clear that there is a transition of wear regime when the applied load increases from 600 to 1200 N.

For all the applied loads, it is found that the friction coefficient can be characterized by two stages. At the initial stage, the friction coefficient is controlled by surface roughness and the formation of a tribolayer, the friction coefficient is high and unstable. At the second stage, the friction and wear are controlled by the nature of the tribolayer, the friction coefficient decreases and exhibits a steady state. Such friction regimes have been explained in previous papers.[19,20]

In this study, wear rate was defined as the total weight loss divided by the total sliding distance. Figure 5 summarizes the variation of wear rate with loads for pure copper, C51100 bronze, and submicron SiCp/Cu samples. Obviously, for all the tested materials, the wear rate increases with the increment of the applied load. Pure copper exhibits an extremely high

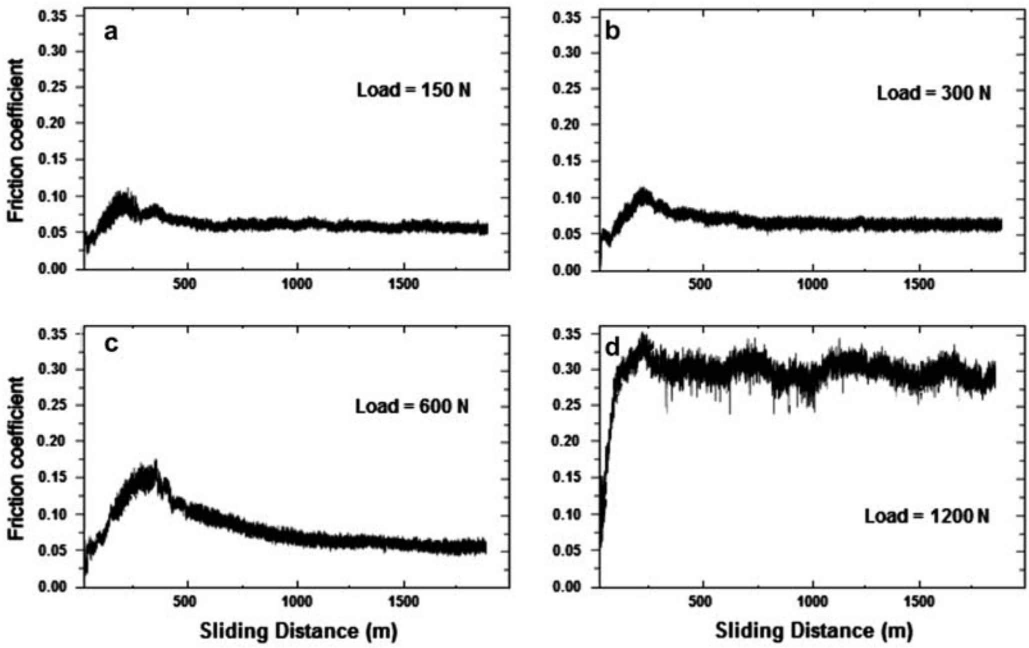

Figure 4. Friction coefficients of 5 vol.% SiCp/Cu composites investigated in the sliding wear test.

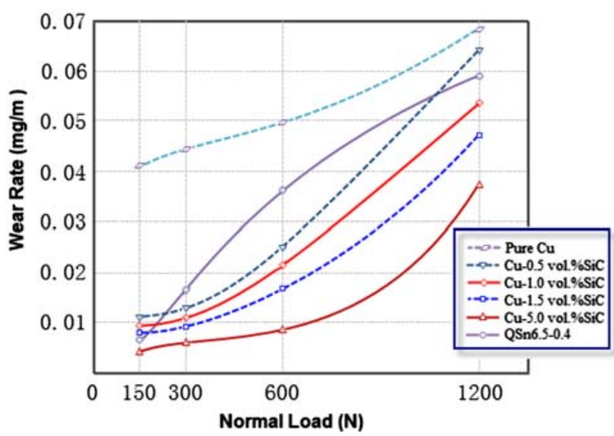

Figure 5. Wear rate against 40Cr under different applied loads.

weight rate during wear sliding as expected. The wear rate of submicron SiCp/Cu decreases significantly. For instance, the wear rate of pure Cu and 0.5 vol.% SiCp/Cu is 0.041 and 0.011 mg m^{-1}, respectively, when the applied load is 150 N. This may be due to the fact that hardness of composites is improved with the additions of SiC particles, and the addition of SiC particles can effectively restrain the flow of soft Cu matrix. Apparently, there is a trend that the wear rate decreases with the increasing content of the SiC particles, which is a well-accepted law. The wear rate of pure Cu and C51100 bronze is approximated as linearly increasing with the increase of applied load. However, the wear rate of the SiCp/Cu shows a slow increase when the applied load is lower (150–600 N), while there is an obvious acceleration when the applied load is higher (600–1200 N).

3.3. Morphologies of the worn surfaces

It can be clearly seen from Figure 5 that the 5.0 vol.% SiCp/Cu composite presents an excellent wear resistance. Figure 6 reveals the morphologies of the worn surfaces of pure Cu and five vol.% SiCp/Cu composites with different applied loads. For comparison, the worn surface of the original state of the 5 vol.% SiCp/Cu composite specimens is also presented. Figure 6(a) shows that the submicron SiC particles exist in an agglomeration state in Cu

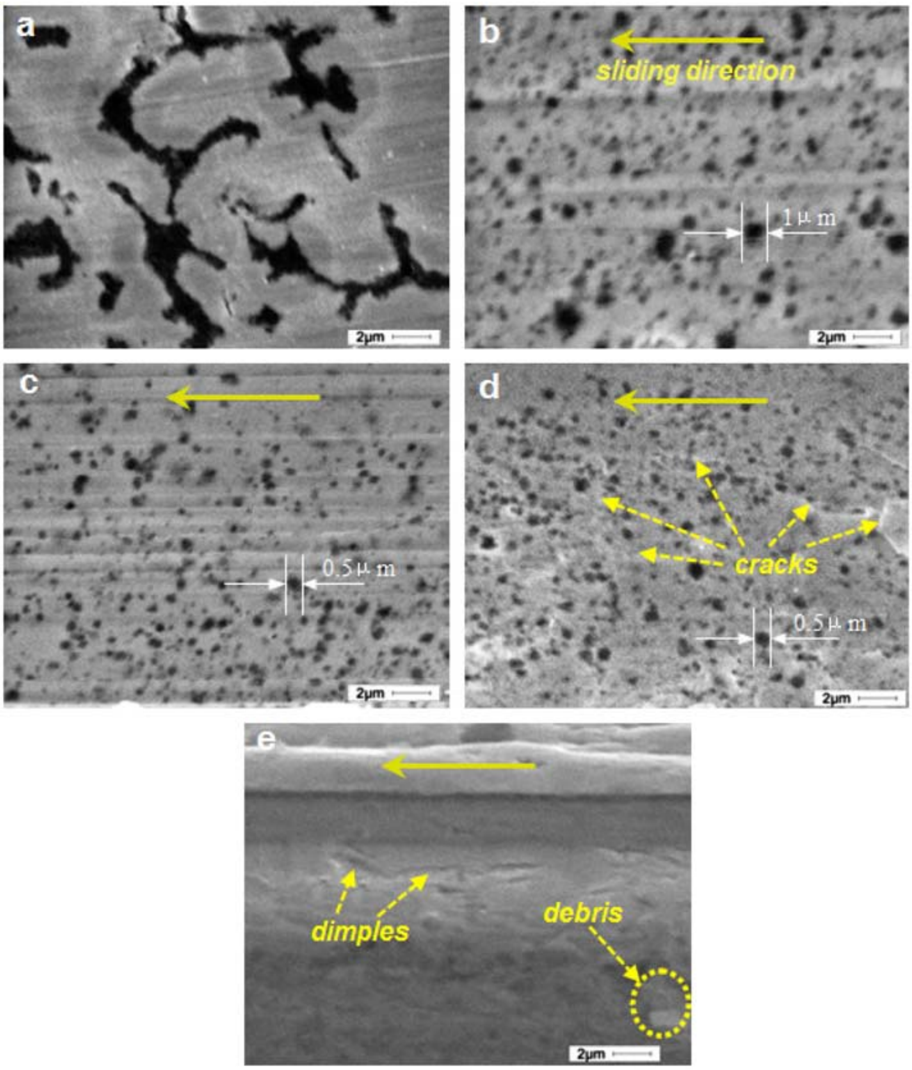

Figure 6. (a). SEM microstructure of five vol.% SiCp/Cu samples in original state, (b) worn surface of five vol.% SiCp/Cu samples under the load of 300 N (c) worn surface of five vol.% SiCp/Cu samples under the load of 600 N, (d) worn surface of five vol.% SiCp/Cu samples under the load of 1200 N, and (e) worn surface of pure Cu under the load of 300 N.

matrix. It can be seen that the degree of agglomeration has reduced remarkably in Figure 6 (b–d). These indicate that the wear behavior between the composite and the counterpart results in the formation of a kind of tribolayer, in which the submicron SiC particles are well dispersed. The formation of this type of well-dispersed tribolayer may be due to the friction-stirring effect of the counterpart. However, the morphologies of the tribolayers are different with the applied loads. The dimension of agglomerate SiC particles is about one μm when the applied load is 300 N (Figure 4(b)), while it is about 0.5 μm when the applied load is 600 and 1200 N (Figure 4(c) and (d)). It can be concluded that the dispersing process of agglomerate SiC particles will stop when the applied load is above 600 N.

The smooth polished surface with submicron grooves is shown in Figure 6(b) and (c), and the size of wear debris is very small (0.3–0.4 μm). The formation of this kind of the smooth polished surface can be explained by the following reasons:

(1) The thickness of lubricant film is thicker than the size of wear debris, so the wear debris is easily transferred from the worn surface with the flow of lubricant. It is difficult for the submicron debris to congregate on the worn surface of the composites or the 5140 steel counterpart.
(2) The debris contains submicron SiC particle because the size of wear debris is larger than SiC particle, so the hardness of debris is also high. The debonded SiC particles with sharp edge and the delaminated wear debris plough over the surface of composites, which causes the formation of submicron grooves.

Figure 6(d) shows the worn surface of composites under the load of 1200 N. It can be seen that microcracks spread over the worn surface, and the closed microcracks indicate the formation of new wear debris. The size of debris is much larger than the debris formed under the applied load of 300 and 600 N. These indicate that the tribolayer cannot bear such high applied load and cracks.

Continuous and deep grooves, plastic dimples, and wear debris are observed on the wear track of pure Cu (Figure 6(e)), which indicates severe plastic deformation. The investigation of the collected debris from pure copper shows that fine pits (2–3 μm) form and congregate together. These indicate that adhesive wear occurs on the pure Cu surface.

The EDAX analyses conducted on the worn surface and counterpart are shown in Table 3. There is no Fe found on the worn surface of composites, and no Cu found on the counterpart under the load of 300 and 600 N. That is to say, very few materials are transferred during the wear process. The addition of SiC particles can reduce the contact of Cu with Fe to a considerable degree. Thus the occurrence of adhesive wear is restrained. Because the size of the debonded and delaminated debris of the SiC particle is at the level of submicron, the plough phenomenon does not happen on both the worn surface and the counterpart steel.

Table 3. Elemental composition of the worn surface and counterpart in Figure 6.

Element	Si (wt.%)	Cu (wt.%)	Cr (wt.%)	Mn (wt.%)	Fe (wt.%)
Figure 6(b) Content	7.18	91.01	–	–	–
Figure 6(c) Content	8.07	90.05	–	–	–
Figure 6(d) Content	7.35	83.76	–	–	7.37
Figure 6(b) Counterpart	0.31	–	0.71	0.48	96.46
Figure 6(c)Counterpart	0.32	–	0.73	0.47	97.02
Figure 6(d) Counterpart	1.12	10.34	0.45	0.16	86.25

While at the load of 1200 N, there are considerable materials transferring on both the worn surface of composites and the counterpart. The percentage of SiC in the worn surface is much higher than that inside the composite. This indicates that many of the debonded submicron SiC particles have re-implanted into the worn surface.

EDAX analyses indicate that the tribolayer presents an excellent wear performance at the load of 300 and 600 N. It adheres neither to the worn surface of composite nor to the counterpart. In both conditions, the wear mechanism is delaminating wear. When the load increases to 1200 N, the adhesive wear occurs. Therefore, with the increment of load, the wear mechanism of composite has a trend of transition from delaminating wear to adhesive wear.

3.4. Microhardness depth profiles

The distribution of the microhardness of the worn surface in depth direction is shown in Figure 7. Apparently, the composite shows the evidence of work-hardening due to plastic strain localization in the subsurface region. The microhardness on the worn surface increases with the increment of load. When the applied load rises from 600 to 1200 N, the microhardness of worn surface is almost similar. The microhardness of the worn surface does not increase any more. This indicates that the microhardness reaches to its maximum value. The maximum value is about 312 HV. Emge et al. [21] found nanocrystalline tribolayers in copper. The reason for the great increment of microhardness may be the effect of both work-hardening and nanocrystallization. It also can be seen that the depth of deformation hardening increases with the increment of applied load. Conclusion can be drawn on that the tribological behavior of submicron SiCp/Cu is highly different from that of micrometer SiCp/Cu [9,11,13].

3.5. Wear mechanism

When the applied load is less than 600 N, the smooth polished worn surface, the low wear rate, the small size (0.3–0.4 μm) wear debris and the low-stable friction coefficient ($\mu = 0.05 \sim 0.07$) indicate that the wear mechanism of the composite is delaminating wear. When the load rises to 1200 N, the severe plastic deformation, the high wear rate, the big size (2–3 μm) wear debris and the highly unstable friction coefficient ($\mu = 0.25 \sim 0.34$) indicate that

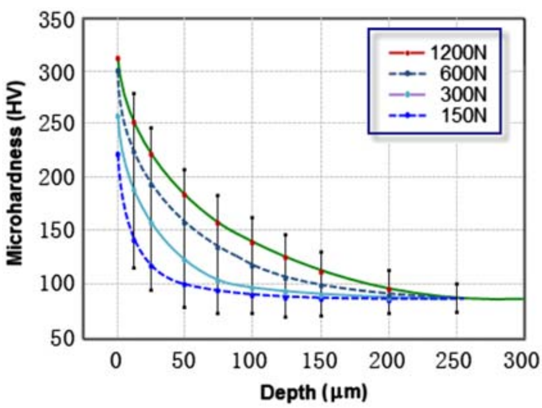

Figure 7. Vickers microhardness depth profiles of worn surfaces of 5 vol.% SiCp/Cu composite at different applied loads.

the wear mechanism of composite is adhesive wear. The transition from delaminating wear to adhesive wear results in the acceleration of wear rate (Figure 5). According to the above facts, it can be concluded that the behavior of tribolayer plays particularly an important role in the friction and wear behavior of the submicron SiCp/Cu in this study. The wear mechanism of submicron SiCp/Cu can be shown as the following stages when the applied load is below 600 N:

(1) *Before the formation of tribolayer (the first stage)*: The hard asperities of 5140 steel surface act as cutting tools to scratch the composite surface and form rough grooves. The increasing hardness of the work hardened wear debris rolling between the mating surfaces causes a plowing action on the surface of submicron SiCp/Cu. During the work-hardening process, the agglomerate SiC particles of composite surface are split into smaller blocks due to the flow of surface material along the sliding direction and the transverse direction. At the same time, the subsurface of composite also experiences this procedure, the rapidly increased coefficient (Figure 4), the high wear rate and the delamination of larger dimension wear debris can be observed in this stage.
(2) *Formation of tribolayer (the second stage)*: With the process of wear behavior, the surface of 5140 steel counterpart becomes smooth gradually. The debris produced by the first stage delaminates from the worn surface. Subsequently the subsurface of the composite is exposed. The exposed subsurface proceeds to experience the work-hardening process, and the dispersion of agglomerate SiC particles continues. When the microhardness of worn surface reaches its peak value (220 HV at the applied load of 150 N, 260 HV at the applied load of 300 N, 312 HV at the applied load of 600 and 1200 N, respectively), the dispersedness of SiC particles stops. Thus, a tribolayer with well-dispersed submicron SiC particles forms. This stage can be characterized as the slowly declined coefficient, the still high wear rate and the delamination of larger dimension wear debris.
(3) *After the formation of tribolayer (the third stage)*: In this stage, the friction and wear behavior go into a stable state. Tribolayer with a smooth polished surface forms. EDAX shows that the content of SiC in the worn surface is much higher than that inside of composite, which is because the dimension of submicron SiC particle is so small that it is much easier for the SiC particle to penetrate into the worn surface than to debond from it. The typical signal of this stage is the coefficient wave in a very narrow scope and the value of coefficient is very low ($\mu = 0.05 \sim 0.07$). The dimension of wear debris is very small (about 0.3–0.4 μm) and the wear rate is not changed in this stage. The smooth surface and the submicron debris indicate that the wear mechanism is a delaminate wear.

As to the wear mechanism of submicron SiCp/Cu, when the applied load exceeds 1200 N, it only experiences two stages because of the heavy applied load. The first stage of the wear behavior is highly similar to the first stage of the wear behavior mentioned above. The difference consists in the fact that the plastic deformation and the effect of work-hardening are more severe. In the second stage, the tribolayer is also formed. However, it will easily rupture under such high applied load soon after the formation of the tribolayer. Because of the high contact stress between the composite and the steel counterpart, the segregate effect of SiC between composite and the counterpart declines to a neglectful degree. The adhesive wear occurs in all the stages. The coefficient rises to a considerably high degree and does not decline, as shown in Figure 4(d). The wear rate also keeps up at a rather high level.

4. Conclusion

(1) Submicron SiCp/Cu was fabricated by means of the PM route followed by hot extrusion. The 5 vol.% SiCp/Cu exhibits good integrated mechanical properties. The ultimate strength of 10 vol.% SiCp/Cu is 50% less than that of 5 vol.% SiCp/Cu. The submicron SiC particles exist in agglomeration state in composites.
(2) Compared with pure Cu and C51100 bronze, 5 vol.% SiCp/Cu exhibits a good wear resistance. The wear rate of C51100 bronze is 1.6, 2.4, 4.4, and 1.3 times than that of 5 vol.% SiCp/Cu at the load of 150, 300, 600, and 1200 N, respectively.
(3) During the sliding wear test, the worn surface of composites forms a kind of tribolayer in which the submicron is well dispersed. This tribolayer exhibits an excellent wear performance. The wear mechanism of composite is a delaminating wear when the applied load is below 600 N, and the wear mechanism is an adhesive wear when the applied load exceeds 1200 N.

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