RESEARCH ARTICLE

Sliding wear behavior of fully nanotwinned Cu alloys

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Abstract: Highly nanotwinned (NT) metals have advantages such as high strength, good ductility, favorable corrosion resistance, and thermal stability. It has been demonstrated that the introduction of high density NT microstructures can enhance the tribological properties of metals. However, the influence of the microstructure and the composition of NT alloys on the tribological behavior are not clear. In this work, the sliding wear behavior of fully NT materials, specifically Cu-Al and Cu-Ni alloys, are studied by a nanoscratch technique using a nanoindenter. The effects of microstructure and chemical composition on the wear properties are also studied. The results show that the chemical composition has an obvious influence on the wear resistance and microstructural deformation. For NT Cu-Al alloys, the hardness and sliding wear resistance improve with increased Al content from Cu-2wt.%Al to Cu-6wt.%Al. NT Cu-10wt.%Ni alloy shows even better wear resistance than Cu-6wt.%Al. The microstructural analysis shows that NT Cu alloys with higher wear resistance correspond to a smaller deformation-affected zone. The improvement of sliding wear properties of Cu-Al alloys with higher Al content may be ascribed to their decreased stacking fault energy. NT Cu-Ni alloy shows better wear resistance than Cu-Al alloy, this may be related to the formation of intermetallic compounds in Cu-Al system. This study broadens the knowledge about tribological properties of NT materials and provides a potential method to optimize their sliding wear resistance by altering the chemical composition of NT Cu alloys.

Keywords: nanotwinned alloys; thin film; stacking fault energy; tribology

1 Introduction

Highly nanotwinned (NT) metals have shown potential as an alternative to nanocrystalline (NC) metals, as they exhibit excellent properties, such as high strength, good ductility, favorable corrosion resistance, and thermal stability [1–5]. These properties of NT metals provide an impetus for their development and mechanical characterization for structural applications [6, 7]. However, in addition to the aforementioned properties, good tribological properties are also important for many engineering applications including both structural components and surface coatings [8].

The wear resistance of traditional engineering metals can often be simply related to hardness through the Archard equation, which states that the wear resistance is linearly proportional to the hardness [9]. It is also reported that hardness alone does not determine the wear resistance, as crack nucleation and propagation are also responsible for wear [10, 11]. The wear properties of NC metals have attracted some attention, since NC metals generally have high hardness [12]. It is reported that both the hardness and wear resistance of Ni increases as the grain size decreases from microcrystalline to nanocrystalline scale [13]. However, when the grain size is smaller than 12 nm,

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Hall-Petch hardening seems to breakdown and the reduction of the grain size no longer improves the wear resistance [14]. For example, Luo et al. [15] reported cyclic sliding experiments of Cu/Au multilayers and the formation of a microstructural vortex with increasing cycle number. For NT materials, the microstructural stability and hardness retention of multilayer Cu/Cu with nanoscale twins under fatigue loading and indentation has been demonstrated [16, 17]. Although the microstructural stability of NT materials is enhanced during cyclic deformation, the tribological properties of NT materials are still not well understood [16, 18, 19]. Zhang et al. [20] reported that the sliding wear properties of a Cu plate with a NT microstructural layer was better than those of equiaxed nanocrystalline Cu and coarse-grained Cu samples. Work by Singh et al. [8] confirmed that high-density NT Cu exhibited improved resistance to surface damage and to microstructural changes after an initial sliding pass; however, both low-density and high-density NT Cu exhibited similar surface hardness and microstructure after several sliding passes. These studies demonstrate that the introduction of high density NT microstructures can enhance the tribological properties of metals; however, details regarding the influence of the microstructure and the composition of materials have not yet been reported. To the best of our knowledge, no information is available in the literature concerning the tribological behavior of NT alloys.

In the present paper, the sliding wear behavior of fully NT materials, specifically Cu-Al and Cu-Ni alloys, is studied using a nanoindenter, which is a convenient method to assess the wear properties of materials [21]. The microstructural deformation under the tracks was examined. The effect of chemical composition on the wear properties and microstructural deformation of these materials will be discussed in this paper.

2 Experimental details

Fully NT columnar films of Cu-2wt.%Al, Cu-4wt.%Al, Cu-6wt.%Al, and Cu-10wt.%Ni were synthesized by magnetron sputtering; detailed information on these materials has been published in the literature [22]. The microstructural characterization of the as-deposited films was conducted using scanning electron microscopy (SEM, JEOL JSM-7001) of focused-ion-beam prepared cross sections (SEM/FIB, NanoLab200, FEI Company, Hillsboro, OR, USA) and transmission electron microscopy (TEM, JEOL JEM-2100F). Cross-sectional TEM samples were generated by mounting a cross section of the film on silicon, then dimple grinding and ion milling using a Fischione Model 1050 TEM Mill. The grain width and twin thickness were determined from TEM images using bright field, dark field, and high-resolution images.

The nanoindentation and wear testing of the metals was performed on a Nanoindenter XP (MTS, MN, USA) equipped with a piezo-driven nanopositioning stage. The hardness and elastic modulus measurements were conducted using a Berkovich tip in sets of 20 indents, where all indents were 2 μm deep and 50 μm apart from the next. Reciprocating wear tests were conducted with a spherical diamond tip of 10 μm tip radius and 60° cone angle. The specimens were glued onto steel specimen holders using cyanoacrylate glue for the frictional sliding experiments. Scratch tests with a constant applied load of 5 mN were conducted at a speed of 10 μm/s over a length of 100 μm by moving the nanopositioning stage back and forth. The tip was cleaned after every experiment in order to avoid wear debris transfer between experiments. The depth and width of the wear tracks were obtained using SEM and surface profilometry (Ambios XP2 profilometer). Microstructure characterization after the scratch tests was performed using SEM and FIB microscopy to assess deformation-induced microstructural changes.

3 Results and discussion

Figure 1(a) presents a typical FIB cross-sectional image of the microstructure of an as-sputtered Cu-2wt.%Al alloy. Figures 1(b) and 1(c) show cross-sectional TEM images of Cu-Al alloys indicating the grain width and the twin thickness. The sample has a typical columnar grain structure, with twins within the columnar grains. The presence of NTs is confirmed by a selected-area electron diffraction (SAED) pattern shown in Fig. 1(b). The SAED pattern shows the typical double hexagon pattern of (110) zone axis oriented twinned grains.
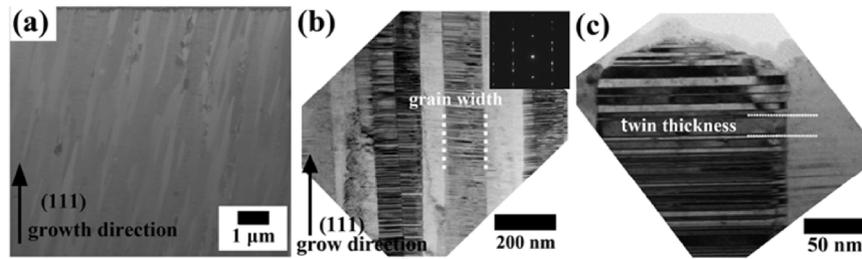
Fig. 1 (a) Representative FIB cross-sectional image and (b and c) cross-sectional TEM images of a NT Cu-2wt.%Al alloy. Inset is selected area electron diffraction (SAED) pattern.

Table 1 summarizes the measured grain widths and twin thicknesses (volume weighted average) as well as the hardness, modulus and friction coefficient for the tested NT alloy samples. It is known that the hardness of materials is one of the most important mechanical properties related to tribological properties [8, 23]. For NT Cu-Al alloys, the hardness increases as the Al content increases from 2wt.% to 6wt.%. This is consistent with tensile test results of NT Cu-Al alloys, which show an increased tensile strength with higher Al content [24]. The modulus of Cu alloy is higher than that of pure copper. A major contribution to this increased modulus arises from Al in solid solution. It is also shown that Cu-Ni alloys have a higher elastic modulus that is accompanied by a higher hardness compared with Cu-Al alloys.

While the friction coefficient for the different samples was obtained over the whole scratch length using the nanoindenter, we reported the values averaged over the sliding distance between 30–70 μm to account for the deceleration and acceleration of the stage at the ends of the tracks. The friction coefficient represents the friction between the NT alloys and the diamond nanoindenter tip [10]. Figure 2(a) illustrates the change of the friction coefficient as a function of cycle number with one cycle describing one back-and-forth pass. A

"steady-state" value is reached after approximately 40 back-and-forth passes in all four materials. The friction coefficient and wear resistance values shown in Table 1 are for 50 sliding cycles ($N = 50$).

The wear deformation during a sliding test can be assessed by the dimensionless wear coefficient [25], k, or wear resistance, R_w. Here, k is calculated according to the classical equation [9]:

$$k = VH / Px \qquad (1)$$

where V is the total amount of displaced material, which is calculated based on the depth and width of the track (shown in Table1), H is the hardness of the material (shown in Table 1), x is the total distance of travel (100 μm × 100), and P is the applied load (5 mN). Moreover, R_w is obtained by the following equation [26]:

$$R_w = H / k \qquad (2)$$

The calculated wear resistances of NT alloys are listed in Table 1. For NT Cu-Al alloys, the Cu-6wt.%Al is the most wear resistant alloy, followed by Cu-4wt.%Al and Cu-2wt.%Al. It should be noted that the wear resistance of the NT Cu-4wt.%Al alloy is lower than that of the NT Cu-6wt.%Al alloy, despite its smaller grain width and twin thickness. While it has been

Table 1 Summary of sliding tests results for Cu alloys with microstructure and composition.

Material	SFE* (mJ/m^2)	Grain width (nm)	Average twin thickness (nm)	Elastic modulus (GPa)	Hardness (GPa)	Friction coefficient (μ) ($N = 50$)	Volume removed (m^3) ($N = 50$)	Wear coefficient, K	Wear resistance, R_w (Pa)
Cu-2wt.%Al	37[27]	219 ± 7	10 ± 1	150 ± 50	3.1 ± 0.6	0.73	3.5 × 10^{-18}	2.91 × 10^{-3}	1.20 × 10^{10}
Cu-4wt.%Al	13[28]	111 ± 1	5 ± 1	145 ± 4	3.5 ± 0.1	0.15	3.5 × 10^{-18}	1.93 × 10^{-3}	1.81 × 10^{10}
Cu-6wt.%Al	6[28]	156 ± 7	13 ± 1	150 ± 20	3.8 ± 0.4	0.12	3.8 × 10^{-18}	1.52 × 10^{-3}	2.50 × 10^{10}
Cu-10wt.%Ni	47–74[31]	226 ± 7	20 ± 1	180 ± 20	3.8 ± 0.4	0.15	3.8 × 10^{-18}	0.93 × 10^{-3}	4.07 × 10^{10}

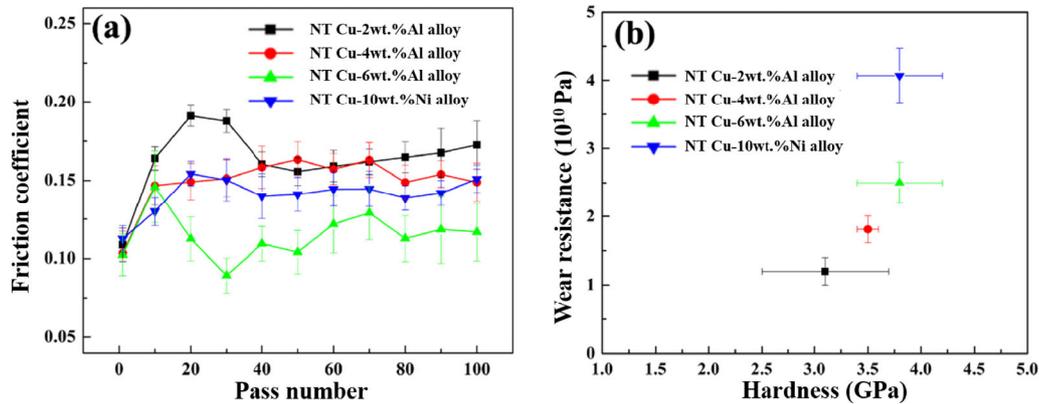* The SFE were obtained from previous reports [27, 28, 31].

Fig. 2 (a) Friction coefficient as a function of sliding pass number for different NT Cu alloys and (b) comparison of wear resistance and hardness for NT Cu alloys.

reported that tribological properties are enhanced by decreasing the grain size in Cu [13, 20], our results show that the wear resistance of Cu alloys is more dependent on the change of the chemical composition than on the microstructure. The sliding tests also demonstrated that the NT Cu-10wt.%Ni alloy is the most wear resistant alloy of the samples tested. The correlation between wear resistance and hardness is illustrated in Fig. 2(b), which compares the relative material performance. It can be seen that hardness is an important factor in assessing the wear resistance of NT alloys, since a higher hardness usually correlates with a better wear resistance. However, it seems that the wear resistance of these NT Cu alloys does not exactly follow the classical Archard equation, which predicts wear resistance to be linearly proportional to hardness [10]. This might be related to changes in the microstructure, since the hardness was determined on the as-deposited films, while the wear resistance was evaluated after 50 sliding cycles. Work by Hodge et al. [10] also shows that the wear resistance of amorphous alloys also do not follow the classical Archard equation, i.e. the wear resistance is not linearly proportional to the hardness. In that work, the discrepancy was suggested to be a result of different wear mechanisms operating in different materials [10].

The different wear behaviors of NT alloys can also be examined by studying the wear morphologies of the different materials after scratch tests. The microstructure for specimens after 50 cycles was investigated using SEM and FIB. Figure 3 shows top-down SEM views of wear tracks with the sliding

direction denoted by the arrow. In these images, surface plowing by asperities and a cracked transfer layer can be seen. The Cu-6wt.%Al alloy exhibits a narrower sliding track than the Cu-4wt.%Al alloy, followed by the Cu-2wt.%Al alloy. A narrower sliding track and less surface damage were also observed in the Cu-10wt.%Ni alloy. The surface damage observed by SEM is consistent with the wear resistance results. To further investigate the wear behavior, Fig. 4 presents the cross-sectional images perpendicular to the sliding direction after 50 cycles. Structural changes under the sliding track can be observed for all samples. While the material in the vicinity of the surface shows significant detwinning and an equiaxed grain structure, it is surrounded by the original microstructure. The areas of the deformation-affected zone (Fig. 4) for different Cu alloys were calculated using Image J software (Rasband, WS, US National Institutes of Health, Bethesda, Maryland, USA). The calculated areas of the deformation-affected zones below the sliding tracks for Cu-2wt.%Al, Cu-4wt.%Al, Cu-6wt.%Al, and Cu-10wt.%Ni are 1.06 μm^2, 0.88 μm^2, 0.62 μm^2, and 0.39 μm^2, respectively. It can be seen that Cu-10wt.%Ni has the smallest deformation-affected zone. For Cu-Al alloys, the size of the area decreases with higher Al content. It was shown that NT Cu alloys with higher wear resistance have a smaller deformation-affected zone, which suggests better microstructural stability. In other words, the wear resistance of NT Cu-Al alloys is more dependent on the microstructure evolution than on the initial microstructure. A similar result has been observed in
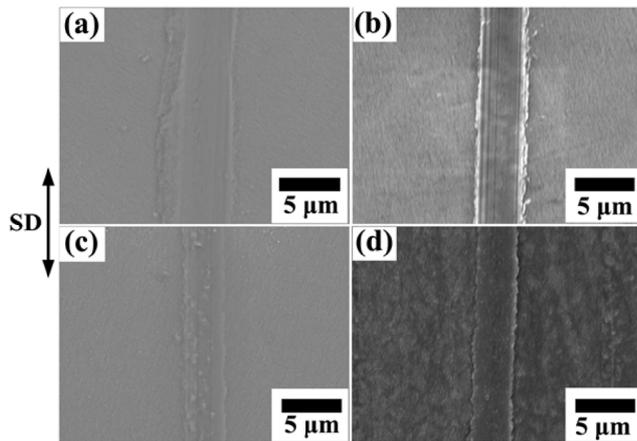
Fig. 3 Top view SEM micrographs of typical surface damage morphology in different NT Cu alloys after 100 sliding circles: (a) Cu-2wt.%Al alloy; (b) Cu-4wt.%Al alloy; (c) Cu-6wt.%Al alloy; (d) Cu-10wt.%Ni alloy. Double pointed arrow indicates the sliding direction (SD) for all samples.
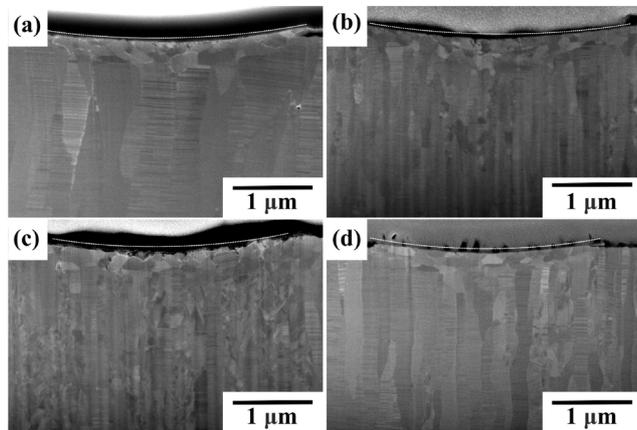

Fig. 4 FIB cross-sectional images (imaged at 52° tilt angle) perpendicular to the sliding direction of NT Cu alloys showing deformation-induced microstructure changes in the vicinity of the scratch surface after 50 sliding passes. (a) Cu-2wt.%Al alloy; (b) Cu-4wt.%Al alloy; (c) Cu-6wt.%Al alloy; (d) Cu-10wt.%Ni alloy. The position of the spherical indenter is indicated by the dashed line. In all cases, a clear boundary between the original micro-structure and the deformed region can be observed.

repeated frictional sliding tests for NT Cu, in which it was found that friction evolution as well as the local mechanical response are more strongly influenced by a local structural evolution during repeated sliding than by the initial structure [8].

This study has shown that the wear resistance of Cu-Al alloys increases with higher Al content and is accompanied by a smaller volume of changed microstructure. In addition to the improved hardness,

the enhanced wear properties and microstructural stability of Cu-Al alloys might be related to the difference of stacking fault energy (SFE). The SFE for NT Cu-Al alloys decreases with higher Al content as listed in Table 1 (Cu-2wt.%Al~ 37 mJ/m^2 [27]; Cu-4wt.%Al~ 13 mJ/m^2 [28]; Cu-6wt.%Al ~ 6 mJ/m^2 [28]). Generally, the lower the value of the SFE, the harder it is for cross-slip to occur [29]. Lowering the SFE can reduce the mobility of dislocations by restricting the cross-slip, leading to the dislocation process associated with grain boundary migration being highly restrained [30, 31]. Furthermore, the introduction of Al atoms may pin the potential moving planes, thereby decreasing the grain boundary energy and dislocation velocity, and then drag or suppress the grain boundary motion [30, 32]. For these reasons, the Cu-Al alloys with higher Al content might exhibit a smaller deformation-affected zone and increased wear resistance. Thus, the evolution of the microstructure under sliding contact is related to the SFE. However, it should be noted that the hardness and wear resistance of Cu-10wt.%Ni (SFE 47–74 mJ/m^2) [31] are both higher than those of the Cu-Al alloys. This significant difference in wear resistance compared to the Cu-Al NT alloys, despite the high SFE of Cu-10wt.%Ni, may be related to the formation of intermetallic compounds in the Cu-Al system [33] during sliding. This requires further study.

4   Conclusions

Four fully NT Cu alloys, Cu-2wt.%Al, Cu-4wt.%Al, Cu-6wt.%Al, and Cu-10wt.%Ni were tested using a nanoindenter to acquire hardness, elastic modulus, and sliding wear properties. The elastic modulus of Cu-Al alloys is about 150 GPa and the modulus of Cu-10wt.%Ni is 180 GPa. The hardness of Cu-Al alloys increases with the Al content. The hardness of Cu-10wt.%Ni is the same as Cu-6wt.%Al, which is 3.8 GPa. Their wear resistance, in an ascending order, is Cu-2wt.%Al, Cu-4wt.%Al, Cu-6wt.%Al, and Cu-10wt.%Ni. This suggests that the wear response of fully NT Cu alloys is mainly dependent on chemical composition. Microstructural analysis after the sliding wear tests showed that NT Cu alloys with higher wear resistance correspond to a smaller area of the

deformation-affected zone under the indentation surface. The enhanced wear properties and microstructural stability of Cu-Al alloys may be ascribed to the decrease of the SFE. Overall, NT Cu-Ni showed the best tribological properties, thus further highlighting the effect of composition. This investigation suggests that the chemical composition has a larger effect on the wear behavior than initial microstructure, including grain width and twin thickness, at least for the following fully NT Cu alloys: Cu-2wt.%Al, Cu-4wt.%Al, Cu-6wt.%Al, and Cu-10wt.%Ni.

Acknowledgements

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