A low-to-high friction transition in gradient nano-grained Cu and Cu–Ag alloys

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Abstract: A unique low-to-high friction transition is observed during unlubricated sliding in metals with a gradient nano-grained (GNG) surface layer. After persisting in the low-friction state (0.2–0.4) for tens of thousands of cycles, the coefficients of friction in the GNG copper (Cu) and copper-silver (Cu–5Ag) alloy start to increase, eventually reaching a high level (0.6–0.8). By monitoring the worn surface morphology evolution, wear-induced damage accumulation, and worn subsurface structure evolution during sliding, we found that the low-to-high friction transition is strongly correlated with distinct microstructural instabilities induced by vertical plastic deformation and wear-off of the stable nanograins in the subsurface layer. A very low wear loss of the GNG samples was achieved compared with the coarse-grained sample, especially during the low friction stage. Our results suggest that it is possible to postpone the initiation of low-to-high friction transitions and enhance the wear resistance in GNG metals by increasing the GNG structural stability against grain coarsening under high loading.

Keywords: gradient nano-grained metals; Cu and Cu–Ag alloy; low-to-high friction transition; subsurface plastic deformation; wear

1 Introduction

When metallic materials undergo dry sliding, the coefficient of friction (COF) is initially low (0.2–0.4), but it jumps to a steady-state value of 0.6–1.2 (high COF) after only several hundred cycles [1, 2]. Usually, it is intractable to preserve or prolong the intrinsic low friction state of metals during sliding, and the consequent high friction stage inevitably reduces the energy efficiency and makes these materials unusable in tribological applications. Among the reasons for the swift low-to-high friction transition, sliding-induced shear instability near the surface is the primary factor that triggers surface roughening

[3, 4] in the initial stage, distinct microstructure discontinuity from the underlying bulk metallic material [5–9], and subsequent subsurface cracking and delamination under tribological loading [10, 11]. For instance, there is a clear correlation between the friction transition and the increasing surface roughness in a tungsten–carbon tribo-system sliding for an initial 100 cycles [12]. Recently, the inevitable friction transitions were delayed so that they occurred after a longer cycles in either homogenous coarse-grained (CG) or nano-grained (NG) metals and alloys [13–15], while sliding-induced strain localization was significantly alleviated by decreasing the contact pressure and speed. Some researchers [7, 15]

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have proposed that such a transition in numerous tribo-systems corresponds to the deformation mechanisms transitioning from dislocation activity in the low-friction regime to grain boundary sliding under high friction, which is affected by the contact stress.

A similar strategy to reduce shear stress in numerous engineering applications is to apply lubricants that sit above the metallic surfaces [16–19]. A typical example of lubricant films is diamond-like carbon (DLC), which, when applied to tungsten under ambient conditions, can maintain a low COF (~ 0.2) for up to approximately 2,500 cycles, before increasing suddenly up to 0.5 under contact stress of 0.8 GPa [16]. The COF transition during dry sliding can be explained by surface roughening and the corresponding mechanical mixing between the sliding partners, which determines the reliability of tribo-systems. The aforementioned low-to-high friction transitions are either strongly dependent on the sliding conditions (contact pressure, sliding speed, etc.) or based on the performance of lubricants. However, there is still a lack of knowledge and understanding of the influence of a metallic material's worn subsurface microstructure during the friction transition.

Our recent work offers a different strategy to suppress sliding-induced strain localization in a Cu–5Ag alloy [20] by introducing a GNG surface layer, covering a graded variation in grain size from about 30 nm in the topmost surface to micrometers in the interior. As such, the GNG surface layer in the Cu–5Ag alloy yielded a remarkable reduction in COF from 0.64 for the CG sample to 0.29 under a load of 50 N. The stable gradient nanostructure was able to persist in the low-friction state for more than 30,000 cycles in a Cu–5Ag sample under high tribological loading. Similar COF reductions have been observed in GNG Cu [21] and steel [22]. Because the low friction state is one of the most significant considerations for tribological applications [23], it is important to determine if there is a low-to-high friction transition in the GNG metals during sliding, as well as to understand how to maintain a low friction state for a longer duration and the corresponding wear mechanisms. In this work, this

was examined in the GNG Cu and Cu–5Ag samples by extending the sliding period based on our previous experiments [20, 21] and adjusting the sliding conditions. The objective of the current study was to investigate the correlation between dynamic friction and wear behavior and subsurface structure evolution in GNG metals.

2 Experimental

The starting materials were commercial-purity Cu (99.97 wt%) and Cu–5Ag alloy bars (15 mm in diameter) with a CG structure (average grain size of 20 μm). Subsequently, the bars were subjected to a surface mechanical grinding treatment (SMGT) at liquid nitrogen temperature, giving rise to a GNG structure in the surface layer. Identical processing parameters were applied to the Cu and Cu–5Ag samples, and the details of the GNG structure can be found in Refs. [20, 21]. The micro-hardness gradient ranges from 2.9 GPa at the surface to 0.85 GPa in the CG matrix for the GNG Cu–5Ag sample. For the GNG Cu sample, the hardness decreases from 1.8 GPa at the surface to 0.65 GPa in the CG matrix.

For comparison, bulk nano-grained Cu and Cu–5Ag samples were prepared by means of dynamic plastic deformation (DPD) [20]. The deformed microstructure of the DPD Cu sample is composed of nanosized grains (~ 75 nm) mixed with approximately 33 vol% of nanotwinned regions (twin thickness $\lambda = 49$ nm) [24]. The DPD Cu–5Ag sample is characterized by nanograins (~ 60 nm) mixed with small fraction nanotwins (twin thickness $\lambda = 22$ nm). The hardness values for the NG Cu and Cu–5Ag samples are 1.53 and 2.4 GPa, respectively.

Friction tests were performed with an Optimol SRVIII reciprocating tribometer in a ball-on-plate contact configuration under unlubricated conditions at room temperature (25 °C) in air. The tungsten carbide cobalt (WC–Co) balls (10 mm in diameter) with a microhardness of 17.5 GPa were chosen as the counterfaces. The friction tests were carried out along the length direction of the SMGT bars at a sliding stroke of 1 mm, a sliding velocity of 0.01 m/s, and normal loads of 10–90 N. One sliding cycle is defined as two strokes (trace and retrace). The

surface morphologies and roughness of the worn samples were measured using an Olympus 4000 confocal laser scanning microscope (CLSM) with a height resolution along the Z axis of 10 nm. The profiles of the worn surfaces were measured using a MicroXAM three-dimensional (3D) surface profilometer system to determine the wear volumes. A reference surface was determined to quantify the volume of the wear scar, and the volume of material below the reference surface was taken as the wear volume.

Cross-sectional structural characterization of the worn subsurface layers was performed on an FEI Nano-SEM Nova 430 system operated at 15 kV and a JEOL-2010 TEM operated at a voltage of 200 kV. Cross-sectional TEM foils were prepared by cutting in the middle of the wear scars parallel to the sliding direction in a focused ion beam (FIB) (FEI Helios NanoLab DualBeam 650) system. A thin layer of platinum was deposited on the worn surface to protect against beam damage.

3 Results and discussion

The friction tests were performed for a prolonged period of up to tens of thousands of cycles in order to investigate the likely friction transition phenomenon occurring in the samples. Starting with the CG and NG structures, the measured COFs of the Cu and Cu–Ag samples increased immediately, bypassing the low-friction stage and reaching the steady-state high-friction stage (0.6–0.8) after several hundred to thousand cycles (Fig. 1). In contrast, both the GNG

Cu and Cu–Ag samples remained in the low-friction state after longer sliding cycles. Taking the GNG Cu–Ag as an example, the sample persisted in the low friction state for 32,000 cycles and the COF increased gradually from 0.29 to 0.63 (comparable to the steady-state values in the NG and CG samples) at 45,000 cycles (Fig. 1(a)). The low-to-high friction transition of the COF required a much longer sliding period (~13,000 cycles) for the GNG Cu–Ag sample. A similar COF variation (Fig. 1(b)) was observed in the GNG Cu sample, but with a shorter low-friction duration (approximately 10,000 cycles). Here, the low-friction duration is defined as the number of sliding cycles that the materials can endure until a stable and low COF starts to rise. Such unusual COF variations have not been observed before in metallic materials with homogeneous structures under similar sliding conditions [13, 14].

Figure 2 shows the surface and subsurface morphology features in the GNG Cu–5Ag sample corresponding to the low-to-high transition process of the COF under tribological loading. First, the sliding surface remained smooth after sliding for 9,000 cycles in the low-friction stage (COF = 0.3), and the measured roughness R_a was 0.02 μm parallel to the sliding direction (Figs. 2(a) and 2(d)), equal to that of the original surface from our previous paper [20]. Cracks were not identified in the subsurface layer after sliding for 9,000 cycles (Fig. 2(g)). When the COF increases to ~0.4 upon further sliding for 35,350 cycles, micro-sized pile-ups are generated, leading to a rough surface ($R_a = 0.11 \mu\text{m}$) in the transition

Fig. 1 Plot of COF as a function of sliding cycles for the CG, NG, and GNG samples sliding against WC–Co balls with a slide stroke of 1 mm and velocity of 0.01 m/s: (a) Cu–5Ag samples at a load of 50 N; (b) Cu samples at a load of 30 N.

Fig. 2 (a–c) Surface SEM images, (d–f) 3D confocal laser microscopy profiles, and subsurface SEM images for the GNG Cu–5Ag samples sliding after different sliding cycles with different COF values: (a, d, g) 9,000 cycles, COF = 0.3; (b, e, h) 35,350 cycles, COF = 0.4; (c, f, i) 37,500 cycles, COF = 0.5. A load of 50 N, a slide stroke of 1 mm, and a velocity of 0.01 m/s were applied for each sample. Double-ended arrows indicate the sliding directions. Sliding surfaces are outlined by dash-dotted lines.

stage (Figs. 2(b) and 2(e)). In addition, cracks propagate roughly parallel to the sliding direction in the subsurface layer at an average depth of 1.5 μm (Fig. 2(h)). This type of sliding-induced damage with long laminar cracks has also been reported in other tribo-systems [25, 26]. When the COF reached ~ 0.5 after 37,500 cycles, the surface roughness increased substantially to 0.2 μm due to cracking in the contact surface (Figs. 2(c) and 2(f)). Meanwhile, several cracks were generated in the subsurface layer (Fig. 2(i)), which eventually leads to the detachment of wear particles. Correspondingly, the wear loss of the GNG Cu–5Ag sample during the low friction stage was decreased by almost one order of magnitude compared to the CG sample (Fig. 3). In addition, the measured wear volumes of the GNG sample increased slightly during the low friction stage (0–28,000 cycles) and increased markedly after the initiation of the low-to-high friction transition (above 30,000 cycles). These results demonstrate that surface roughening as well as subsurface cracking corresponds exactly to the transition from low-COF to high-COF in the

GNG Cu–5Ag sample, which is accompanied by noticeably increased wear loss in the friction transition regime. It should also be noted that the wear volumes of the GNG samples are much smaller than those of the CG and NG samples during the entire sliding cycle at a load of 50 N, indicating a significantly enhanced wear resistance compared with the CG sample.

Fig. 3 Variation of wear volume with sliding cycles for the CG, NG, and GNG Cu–5Ag samples sliding against WC–Co balls under a load of 50 N, a slide stroke of 1 mm, and a velocity of 0.01 m/s.

The worn subsurface microstructure evolution for the GNG Cu–5Ag sample in different COF regimes is shown in Fig. 4. Specifically, after sliding for 9,000 cycles (COF = 0.3, Fig. 4(a)), no obvious structural changes and Cu oxides were detected in the topmost surface layer (Fig. 4(d)), consistent with our previous investigation [20]. Grain coarsening occurs at a depth of 0.5–10 μm below the topmost layer (Figs. 4(a) and 5(a)), and accommodates massive plastic deformation induced by sliding. A dynamic saturated grain size (~ 200 nm) was reached at a depth of approximately 1.5 μm . At a depth of 5 μm , some grains grow preferentially at the expense of others, resulting in submicrometer-sized grains surrounded by nano-grains (Fig. 5(a)).

After sliding for 36,000 cycles, the COF enters the low-to-high friction transition regime, and the corresponding subsurface structure shows a different scenario. Repeated tribological loading induces vertical plastic flow in the grain coarsening layer. Extensive plastic deformation can no longer be accommodated homogeneously in this layer, which starts to evolve into the topmost NG layer (Fig. 4(b)). Inhomogeneous vertical plastic flow leads to different NG layer thicknesses, ranging from approximately

0.5 μm to several microns. The grain size was reduced to as small as 20 nm, and no ring of Cu oxide was identified in the NG layer (Fig. 4(e)). In the high-friction regime (COF = 0.6), there is a sharp boundary between the NG and grain coarsening layers (Fig. 4(c)). The NG layer consists of Cu and Cu oxides, which provide the signature for the formation of the tribolayer (Fig. 4(f)). Such a nano-grained tribolayer has frequently been observed in tribo-systems with high COFs [23]. Figure 4(g) demonstrates that the tribolayer with an average size of 15 nm was above the grain coarsening layer. Unlike the situation in the low-friction stage, the grain coarsening at different depths exhibits a continuous and homogeneous process (Fig. 5(b)). The grain size within the grain coarsening layer is less than 100 nm, indicative of the wear-off of the saturated coarsened grains (~ 200 nm in size) formed in the low-friction stage. During COF elevation, laminar cracks were frequently observed at a depth of 1–2 μm , which corresponds to the peeling-off of the area of saturated coarsened grains (Figs. 2(h) and 2(i)).

The measured grain size profiles indicate that the low-to-high COF transition is strongly related

Fig. 4 (a–c) Typical TEM bright-field images of the subsurface microstructure in the GNG Cu–5Ag samples after sliding for 9,000, 36,000, and 54,000 cycles at a load of 50 N, respectively. (d–f) Corresponding electron diffraction patterns in the topmost surface layers (as indicated in (a–c)). (g) Variation of the mean grain sizes along with the depth in the GNG samples before and after sliding for 9,000, 36,000, and 54,000 cycles. Sliding surfaces are outlined by dash-dotted lines. A slide stroke of 1 mm and a velocity of 0.01 m/s were applied for each sample.

Fig. 5 Typical TEM images of the subsurface microstructure at a depth of 5 μm below the surface in the GNG Cu–5Ag samples after sliding for (a) 9,000 and (b) 54,000 cycles at a load of 50 N.

to the disappearance of stable NGs in the topmost layer, which results in wear-off of the grain coarsening layer in the GNG Cu–5Ag samples. This is in accordance with the depth of the observed subsurface cracks below the sliding surfaces (Fig. 2(h)) and wear loss data (Fig. 3). Previous investigations demonstrated that the COF elevation in the CG and NG samples stems from sliding-induced surface roughening and/or formation of a delaminating tribolayer [20]. Here, Cu oxides were not detected at the beginning of the COF elevation in the GNG sample (Figs. 4(b) and 4(e)), indicating that surface roughening initiates the COF elevation. Afterwards, repeated sliding on the roughened surface generates local discontinuities and cracks, forming a delaminating nanostructured tribolayer. In fact, after repeated sliding for tens of thousands of cycles, the plastic deformation accommodation ability is significantly reduced in the surface layer, and vertical plastic flow involves the topmost NGs layer as well as the grain coarsening layer, ultimately leading to the detachment of the tribolayers. A similar subsurface structure evolution and cracking were revealed in the GNG Cu sample (Fig. 6). The major difference from the GNG Cu–5Ag sample is that dynamic recrystallization (DRX) occurs underneath the grain coarsening layer. The DRX grain size obviously increases with the increasing sliding cycles as a result of accumulated friction

heat.

To explore the nature of the low-to-high friction transition, we considered the hardness of the sample as an important factor, and selected some metals and alloys with different hardnesses for comparison. COFs of various metals and alloys with homogeneous structures, including the CG and NG samples, were measured under the counterface of WC–Co balls by means of the same friction tests in a load range from 10 to 90 N. Including the COF data for materials under the same tribological conditions as those reported in Refs. [27–32] (Fig. 7(a)), all of these metals and alloys exhibited high steady-state COFs without undergoing a low and stable friction stage, nearly independent of the initial hardness. For instance, the steady-state COFs of Cu–Al alloys remain unchanged (~ 0.7) with increasing hardness ranging from 0.7 to 2.3 GPa [30]. However, significant mitigations in COFs for pure Cu (~ 0.37) and Cu–5Ag alloy (~ 0.29) were achieved during sliding of a submillimeter-thick GNG layer, compared to their CG and NG counterparts. Clearly, this is a stable low friction stage lasting for tens of thousands of sliding cycles, rather than a short-lived low friction stage that only occurs during the running-in stage of the tribo-pairs during dry sliding. As described above, the low-to-high friction transition is closely related to the stability of the subsurface GNG structure during sliding. What other factors might affect the friction transition? Figure 7(b) plots the load dependence of the low-friction duration for the GNG metals. The low-friction duration shortens with an increasing sliding load for both the GNG Cu and Cu–5Ag samples. For example, the low-friction duration for the GNG Cu–5Ag samples decreased from 4.2×10^4 to 1.8×10^4 cycles with a load from 30 to 100 N. The maximum Hertzian contact stress was estimated to be ~ 1.5 GPa under the lowest load of 30 N, which far exceeds the yield strength of the GNG surface layer. This implies that plastic deformation is completely imposed in the surface layers for all sliding conditions. It is interesting to note that the low-friction duration of the GNG samples is reminiscent of the low-cycle fatigue in cyclic loading owing to two fundamental characteristics: comparable fatigue lifetime (10^4

Fig. 6 Typical cross-sectional SEM images of the subsurface microstructure in the GNG Cu samples after sliding for (a) 6,000 [21], (b) 12,000, and (c) 15,000 cycles at a load of 30 N, respectively. (d) Typical TEM image in the topmost surface layer as indicated in (c). (e) Corresponding electron diffraction pattern in the selected area indicated in (d). The worn surface is outlined by dash-dotted lines.

cycles [33]) and plastic deformation applied in each cycle.

In short, the initiation of the low-to-high friction transition in GNG metals strongly depends on the subsurface structure stability as well as the sliding conditions. First, as shown in Fig. 7(b), the GNG Cu–5Ag sample exhibits a much longer low-friction duration compared to the GNG Cu samples. This can be attributed to the more stable NGs surface layer and grain coarsening layer in the GNG Cu–5Ag sample relative to Cu [21], due to the pinning effect of grain boundary migration by alloying Ag. For copper, the instability of the NGs in the topmost surface and adjacent coarsened

grains as well as dynamically recrystallized grains (Figs. 6(a) and 6(c)) resulted in a shorter low-friction stage. Second, it is understandable that large tribological loading or high contact stress is prone to cause shear deformation incompatibility, structural instability, and wear loss in the subsurface layer in the GNG sample, resulting in a short low-friction duration. This effect is pronounced in homogenous NG materials subjected to high loading because the massive plastic deformation cannot be well accommodated via grain boundary sliding and/or dislocation activity [7, 15]. It is worth mentioning that the GNG metals exhibit enhanced capacity-low friction and wear synergy (Fig. 7(b)) in contrast to

Fig. 7 (a) Variation of COFs with initial hardness for various types of metals and alloys, including the CG, NG, and GNG samples. The sliding load varied from 10 to 90 N and the WC ball was applied as the counterface for each sample. (b) Low-friction coating duration as a function of sliding load in the GNG Cu and Cu–5Ag samples. Data from the literature for graphene and DLC coating tribo-systems were added for comparison.

lubricant films including DLC and graphene [16, 34].

4 Conclusions

In summary, there exists a unique low-to-high friction transition in the GNG Cu and Cu–5Ag alloys. By tracking the worn surface morphology evolution, wear-induced damage accumulation, and worn subsurface structure evolution during sliding, the low-to-high friction transition is found to be intimately related to distinct microstructural instabilities induced by vertical plastic deformation and wear-off of the stable nano-grains in the subsurface layer. The initiation of this transition in GNG metals strongly depends on the stability of the subsurface structure against grain coarsening as well as the sliding conditions. Our results shed new light on how to develop gradient nanostructured metals and alloys with unprecedented low-friction durations and high wear resistances.

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