Microstructure and mechanical characteristics of gradient structured Cu and Cu alloys processed by surface mechanical attrition treatment

XZ Hu, LP Cheng, HL Chen, Z Yin, Z Zhang, BP Shu, YL Gong and XK Zhu*
Faculty of Materials Science and Engineering, Kunming University of Science and Technology, Kunming, Yunnan 650093, China
E-mail: xk_zhu@hotmail.com

Abstract: Cu-Al-Zn alloys with different stacking fault energy (SFE) were processed by surface mechanical attrition treatment (SMAT) at cryogenic temperature (CT), mechanical properties of gradient structured Cu-Al-Zn alloys were investigated in this study. Al and Zn content in alloys, which result in the decrease of SFE, can contribute to the increase in strength. Cu-4.5wt%Al-14.3wt%Zn alloy with the lower SFE shows that the strength increased, the ductility did not decrease significantly with increasing processing time, and the strength can be improved by a thicker gradient structure (GS) layer. The better combination of strength and ductility was achieved in Cu-4.5wt%Al-14.3wt%Zn alloy with lower SFE.

1 Introduction
In the traditional engineering materials, strength and plasticity are incompatible. There has been increasing interest in severe plastic deformation (SPD) processing in metals over the past decades[1, 2], such as cold rolling (CR) [3-5], equal-channel angular pressing (ECAP) [6, 7], and high-pressure torsion (HPT) [8, 9]. The primary reason for this interest is previous experimental evidence that the strength of materials significantly increases with decreasing grain sizes according to the well-known Hall-Petch relation[10]. An extremely high strength and hardness has been achieved in bulk ultrafine grained (UFG) materials at the expense of plasticity condition compared with coarse grained counterpart. In the previous studies, which demonstrated that the strength and ductility increase simultaneously when the grain sizes is less than 100nm [11]. Our expectation is to obtain high strength and good plasticity from this new discovery. Some early reports found that strength and plasticity can be improved simultaneously in Cu–Al alloys and Cu–Zn alloys by decreasing the stacking fault energy (SFE) [12, 13]. However, it was not enough investigated yet in Cu–Al–Zn alloys with different SFE.

In this work, mechanical properties of gradient structured Cu-Al-Zn alloys with different SFE under surface mechanical attrition treatment (SMAT) was systematically investigated, which can realize gradient structure in Cu-Al-Zn alloys. Metals with gradient structure (GS) exhibits a superior
mechanical properties compared with coarse grained materials[14]. In this paper, we found that the enhancement of the mechanical properties is different from that of Cu–Al alloys and Cu–Zn alloys. The possible deformation mechanisms in Cu–Al–Zn alloys were discussed accordingly.

2 Experimental procedure
The materials used in the experiments are Cu-4.5wt%Al-14.3wt%Zn alloy and Cu-1.86%Al-23.89wt%Zn alloys with different SFEs of 7 and 10 mJ/m²[15], respectively. Cu–Al–Zn alloys were rolling to plate with a thickness of 3 mm and annealed in vacuum at 1023k for 2h. SMAT was performed on two surface of these samples. The experiment is under vacuum at cryogenic temperature (CT). Uniaxial tensile tests were carried out on a Shimadzu Universal Tester at room temperature with a maximum load of 10KN and at a constant strain rate of 1.0×10⁻⁴ s⁻¹. Hardness measurements were carried out on a HX-1 Vickers hardness testing machine. The hardness of all samples was performed on the cross-sectional of the samples with a load of 50 g and a loading time of 15 s. The detailed microstructure characteristic of GS samples were analyzed by means of electron backscattered diffraction (EBSD) observations, which was carried out on a Zeiss Auriga, with a field emission scanning electron microscope, the operating voltage is 15KV, and the original EBSD data were analyzed by the channel5 software.

3 Results and Discussion
The engineering stress–strain curves of Cu–Al–Zn sample are given in Figure 1. The mechanical properties, which including yield strength (YS) σ₀.², ultimate tensile strength (UTS) 𝜎𝑈𝑇𝑆 and uniform elongation (UE) 𝜀ᴜ are shown in Figure 1. The YS and UTS of the Cu–Al–Zn samples increased with the processing time increasing. Compared with the Cu-1.86wt%Al-23.89wt%Zn alloys, the Cu-4.5wt%Al-14.3wt%Zn alloys with a lower SFE exhibits a better strength–ductility combination with different SMAT time.

![Figure 1](image1.png)

**Figure 1.** Engineering stress–strain curves of annealed Cu–Al–Zn samples and processed by SMAT at CT with different time, (a) Cu-4.5wt%Al-14.3wt%Zn samples, and (b) Cu-1.86wt%Al-23.89wt%Zn samples.

The normalized work hardening rate against the true strain are shown in Fig. 2. It is obvious that the annealed samples have the higher normalized work hardening rate than the GS samples. The normalized work hardening rate decreases gradually with the increasing processing time. The work hardening rate behavior of the Cu-4.5wt%Al-14.3wt%Zn alloy originate from its unique microstructure, which is given and discussed in the following sections.
Fig. 3 shows the hardness profiles of Cu–Al–Zn samples. The average hardness of the annealed samples is ~1325 MPa and ~1072 MPa, respectively. Hardness has a substantial increase after the SMAT process and reaches the highest value at the topmost surface layer. The maximum hardness value is approximately 1.6 and 1.8 times higher than annealed samples. The hardness of GS samples decreased from the topmost surface layer to central matrix and remained in a constant at a certain depth. The thickness of the GS layer of the Cu-1.86wt%Al-23.89wt%Zn samples have apparent increment with the time increasing, which is unlike that the thickness of Cu-4.5wt%Al-14.3wt%Zn. The maximum value can be explained as the largest strains and strain rates occurred in the topmost surface layer during the cryogenic SMAT process [16].

![Figure 2](image1.png)

**Figure 2.** The normalized work hardening rate against the true strain of Cu–Al–Zn samples. (a) Cu-4.5wt%Al-14.3wt%Zn samples, and (b) Cu-1.86wt%Al-23.89wt%Zn samples.

![Figure 3](image2.png)

**Figure 3** The hardness profiles as a function of the depth for Cu–Al–Zn samples. (a) Cu-4.5wt%Al-14.3wt%Zn samples, and (b) Cu-1.86wt%Al-23.89wt%Zn samples.

Fig. 4 shows EBSD mappings of annealed Cu-1.86wt%Al-23.89wt%Zn samples, and the red lines in Figure 4 (b) represents twin. The significant annealing twin can be seen in annealed Cu-1.86wt%Al-23.89wt%Zn samples and the grain size of the whole sample is nearly homogeneous. EBSD mappings of Cu-1.86wt%Al-23.89wt%Zn samples (SMAT for 5, 30 min) is shown in Fig. 5. The grain size of Cu-1.86wt%Al-23.89wt%Zn alloys after SMAT increased from the surface to the center and the grain size gradually tended to be homogeneous. The thickness of the GS layer increased with the processing time from 5min to 30min, twin of surface layer decreased gradually with the time increasing.
Figure 4. EBSD images of annealed Cu-1.86wt%Al-23.89wt%Zn samples: (a) Orientation map. (b) Boundary misorientation (2° ≤ θ < 15°) map.

Figure 5. EBSD images of GS samples from surface to center: (a) Orientation map, and (b) Boundary misorientation (2° ≤ θ < 15°) map of the Cu-1.86wt%Al-23.89wt%Zn SMAT-5min sample. (c) Orientation map, and (d) Boundary misorientation (2° ≤ θ < 15°) map of the Cu-1.86wt%Al-23.89wt%Zn SMAT-30 min sample.

3.1 Effect of GS layer on mechanical behaviors

The hardness has an obvious increase from the CG core region to the treated surface layer, and the thickness of the GS layer with the high SFE samples is larger than that of the lower SFE samples under the same time. The hardness of Cu-4.5wt%Al-14.3wt%Zn samples is always higher than that of Cu-1.86wt%Al-23.89wt%Zn samples at the same depth and time, Zhu et al. demonstrated that the yield strength increases with the thickness of GS layer increasing [17]. Cu-4.5wt%Al-14.3wt%Zn alloys have a higher hardness and smaller grain size on the surface layer compared with Cu-1.86wt%Al-23.89wt%Zn alloys. Therefore, thicker GS layer and smaller grain size give rise to higher yield strength.

The annealed Cu-Al-Zn sample with lower hardness is easier to deform, which can produce a thicker deformation layer when processed by SMAT. However, Cu-4.5wt%Al-14.3wt%Zn samples processed by 5min has reached a higher hardness value, which is difficult to produce further deformation. Cu-4.5wt%Al-14.3wt%Zn samples need more energy to produce further deformation, and Cu-1.86wt%Al-23.89wt%Zn samples produce greater deformation extent for the same energy. Therefore, the GS layer of the higher SFE sample is thicker than that of the low SFE under the same time. Thicker GS layer of
Cu-4.5wt%Al-14.3wt%Zn alloys and smaller grain size of Cu-4.5wt%Al-14.3wt%Zn alloys both can result in higher yield strength.

The ductility of Cu-4.5wt%Al-14.3wt%Zn alloys have no significant decrease compared to Cu-1.86wt%Al-23.89wt%Zn alloys with the process time increasing, which can attribute the gradient structure[18]. In GS samples, plastic deformation occurs first in the coarse-grain region as the tensile strain increasing, coarse-grain region is surrounded and constrained by GS layer, which stay in elastic deformation. The GS layer thickness of Cu-4.5wt%Al-14.3wt%Zn alloys almost remain unchanged with increasing processing time, and the ductility have no significant decrease. Therefore, the thicker coarse-grain region may contribute to plastic strain and improve the ductility[18]. There exists an optimum thickness of GS layer for the superior combination of strength and ductility in Cu-Al-Zn alloys with low SFE., the strength increases continually and ductility remains the same with the time increasing. The GS obtained by SMAT provides an effective way for high strength and good ductility.

3.2 Effect of SFE
SFE is a several material parameter and has influence on microstructure and mechanical properties, which can transform the dominant plastic deformation mechanism from dislocation slip to deformation twinning by lowering SFE[13, 19]. Local softening can lead to plastic instability, which is caused by dislocation annihilation through cross-slip and climb [11, 20]. Unlike the enhancement in strength, the improvement in ductility results from the increase in the strain hardening rate with decreasing SFE. As mentioned previously, restraining dynamic recovery can effectively improve the strain hardening rate, and the increase of strain hardening rate can delay the onset of necking and enhance the tensile ductility[21, 22]. With the decreasing of SFE, it is difficult to make cross-slip and climb of dislocations, which can effectively inhibit dislocation annihilation or dynamic recovery [23].

Twins density is also increased with decreasing SFE, as mentioned previously, Wang et al. [24] indicated that the increase of twins can improve the ductility by a decrease of SFE. Annealing twin is also usually found, meanwhile, there is a bump at the true strain of 0.1 nearby, which is likely to be twins, a series of previous studies demonstrated that there exists twins [25, 26]. An et al. [12] indicate that there exists an optimal combination of strength and ductility in Cu-Al alloy with the lowest SFE. Therefore, the work hardening rate of Cu-4.5wt%Al-14.3wt%Zn samples is higher than Cu-4.5wt%Al-14.3wt%Zn samples under the same processing time, which is derived from lower SFE and more twins.

4 Conclusion
Cu-Al-Zn samples with different stacking fault energy were processed by surface mechanical attrition treatment at cryogenic temperature, which have a significant increase in yield strength and a slight decrease in ductility compared with annealed Cu-Al-Zn samples. Thicker GS layer of Cu-4.5wt%Al-14.3wt%Zn alloys and smaller grain size of Cu-4.5wt%Al-14.3wt%Zn alloys both can result in higher yield strength. The work hardening rate of Cu-4.5wt%Al-14.3wt%Zn samples is higher than Cu-4.5wt%Al-14.3wt%Zn samples, which is derived from a lower SFE. There exists an optimum thickness of GS layer for the superior combination of strength and ductility in Cu-4.5wt%Al-14.3wt%Zn alloys.

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