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ScienceDirect
Trans. Nonferrous Met. Soc. China 22(2012) 819−824
Transactions of Nonferrous Metals Society of China
www.tnmsc.cn

Effect of stacking fault energy on mechanical properties of ultrafine-grain Cu and Cu–Al alloy processed by cold-rolling

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Received 13 April 2011; accepted 29 September 2011

Abstract: Cu, Cu−2.2%Al and Cu−4.5%Al with stacking fault energies (SFE) of 78, 35 and 7 mJ/m^2 respectively were processed by cold-rolling (CR) at liquid nitrogen temperature (77 K) after hot-rolling. X-ray diffraction measurements indicate that a decrease in SFE leads to a decrease in crystallite size but increase in microstrain, dislocation and twin densities of the CR processed samples. Tensile tests at room temperature indicate that as the stacking fault energy decreases, the strength and ductility increase. The results indicate that decreasing stacking fault energy is an optimum method to improve the ductility without loss of strength.

Key words: Cu; Cu alloys; cold-rolling; tensile tests; stacking fault energy

1 Introduction

Bulk ultrafine-grained (UFG) materials and nanocrystalline (NC) grains have been fabricated by severe plastic deformation (SPD) methods [1−4]. However, UFG materials produced by SPD usually have high strength but disappointingly low ductility [5]. How can the ductility be improved without loss of strength? More and more attention is paid to this research. ZHAO et al [6] reported that there was an optimum stacking fault energy that yields the best ductility in UFG Cu−Zn alloys. QU et al [7] investigated the influence of stacking fault energy (SFE) on the microstructural evolution during deformation and the corresponding mechanical properties of Cu−Al alloys processed by equal-channel angular pressing (ECAP). They found that the strength and uniform elongation were simultaneously improved by lowering the SFE [7]. SFE always plays a fundamental role in the description of the defect structure and could affect the grain refinement during SPD processing; as SFE determines the probability of cross slip, it is the possible mechanism of dynamic recovery along with dislocation climb [8−10]. Cold-rolling (CR) at liquid nitrogen temperature (LNT) is an effective and economic method among the SPD techniques to produce bulk UFG metals. In this study, the effect of stacking fault energy on the tensile properties of ultrafine-grained or nanostructured Cu–Al alloys prepared via cold-rolling (CR) at liquid nitrogen temperature was systematically investigated.

2 Experimental

Commercial copper (99.9% purity) was formed into plates with a thickness of 7.9 mm. Cu−2.2%Al (mass fraction) and Cu−4.5%Al alloys were prepared by induction vacuum melting at 800 °C for 2 h. Afterwards, Cu−2.2%Al and Cu−4.5%Al were processed by hot-rolling with a final thicknesses of 8.9 mm (Cu, Cu−2.2%Al and Cu−4.5%Al with SFE of about 78, 35 and 7 mJ/m^2, respectively) [11,12]. Before cold-rolling, copper plates were annealed in vacuum at 600 °C for 2 h and Cu−Al alloy plates were annealed in vacuum at 750−800 °C for 4 h, which eliminated some local flaws and made the composition of these materials more uniform. These materials were sliced into disks with a thickness of 6.9 mm for the cold-rolling process. The rolled sheets were soaked in liquid nitrogen for about 5 min and then cryogenically rolled to further reductions. Although the relocating time before rolling was minimized as far as possible, the actual rolling temperature was a bit higher than 77 K. These plates with 0.5 mm thickness were cold-rolled at liquid

Foundation item: Project (50874056) supported by the National Natural Science Foundation of China
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DOI: 10.1016/S1003-6326(11)61250-8

nitrogen temperature after being annealed in vacuum at 150 °C for 0.5 h. The thickness of the samples decreased from 0.5 to 0.2 mm. The total thickness reduction for these samples was above 95% after multiple rolling passes with a thickness reduction of 40 μm imposed in each separate pass.

Dog bone-shaped tensile samples with gauge size of 15 mm×10 mm were electro-discharge machined. Tensile tests were carried out at room temperature with a Shimazu Universal Tester machine at a strain rate of 1.0×10^{-4} s^{-1}.

X-ray diffraction (XRD) measurements of the CR samples were carried out on the rolling plane using an X-ray diffractometer equipped with a Cu target operating at 1.2 kV. Cu K_α radiation was selected at the goniometer receiving slit section, the divergence and anti-scattering slits were set at 0.5°and 0.5°, respectively, and the width of the receiving slit was 0.3 mm. A series of θ−2θ scans were performed to provide a record of the XRD patterns at room temperature. Pure Cu sheet (99.95% purity) annealed at 400 °C in vacuum was used as an XRD peak-broadening reference for both the crystallite size and the microstrain calculation. The XRD results provide a representation of the defect structure information of the CR samples. Specimens prepared from longitudinal sections were etched in a solution of 15 g $FeCl_3$+5 mL HCl+100 mL H_2O for Cu−Zn alloys.

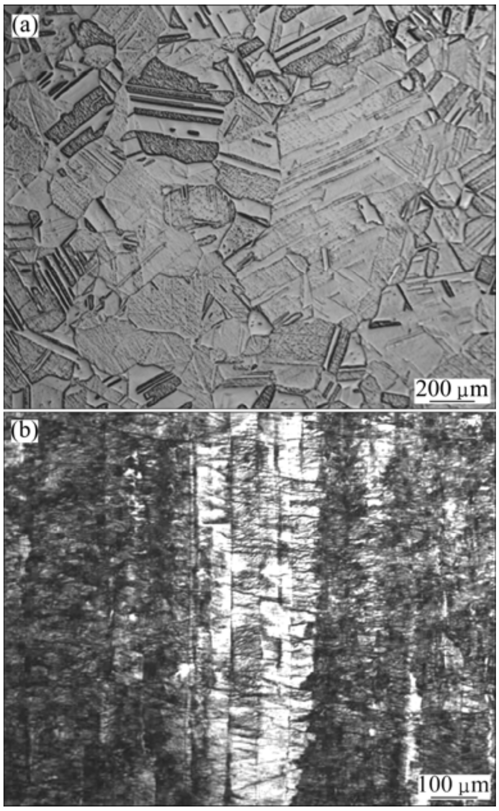

Fig. 1 Optical micrographs of Cu−4.5%Al before (a) and after (b) rolling

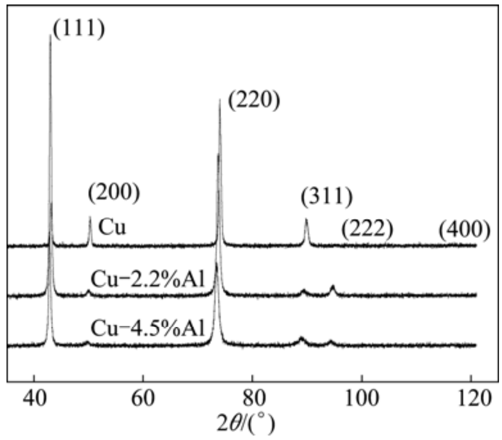

Fig. 2 XRD patterns for Cu, Cu−2.2%Al and Cu−4.5%Al after multiple passes cold-rolling

3 Results and discussion

Figure 1 shows the optical micrographs of Cu−4.5%Al alloy by cold-rolling. A large number of annealing twins distribute in the sample before cold-rolling. The optical images of the structure indicate that the grain sizes are clearly reduced and elongated in the cold-rolling direction to a reduction of about 95%.

The XRD patterns for CR samples with different stacking fault energies are shown in Fig. 2, which indicates an increase in peak broadening with a decrease of stacking fault energies. The X-ray peak broadening is due to the combined effect of the structural refinement (finer crystallite size) and residual strain [13]. At the same time, an increase in the lattice parameter due to the presence of Al solute leads to a shift in the XRD peaks towards lower diffraction angles. In addition, it is seen that the as-rolled samples exhibit an evident ⟨110⟩ texture.

The average crystallite size was measured from the X-ray diffraction-line broadening. Because of the existence of {111} and {110} textures, it is possible in practice to calculate the crystallite sizes (d_{111} and d_{200}) and the microstrains $(\langle\varepsilon\rangle^2_{111})^{1/2}$ and $(\langle\varepsilon\rangle^2_{200})^{1/2}$ using the pairs of (111)–(222) and (200)–(400) reflections [6]. Dislocation density ρ can be represented in terms of crystallite size d and microstrain $(\langle\varepsilon\rangle^2)^{1/2}$ as [14−16]:

$$\rho=\frac{2\sqrt{3}\left(\langle\varepsilon\rangle^2\right)^{1/2}}{d_{\mathrm{XRD}}b} \tag{1}$$

where b is the Burgers vector equal to $b=(\sqrt{2}/2)a$ for the FCC Cu alloy and a is the lattice parameter. The twin density β, defined as the probability of finding a twin boundary between any two neighboring {111} planes, is calculated as [16−18]:

$$\beta = \frac{\Delta C \cdot G \cdot (2\theta)_{111} - \Delta C \cdot G \cdot (2\theta)_{200}}{11 \tan \theta_{111} + 14.6 \tan \theta_{200}} \tag{2}$$

where $\Delta C{\cdot}G{\cdot}(2\theta)_{111}$ and $\Delta C{\cdot}G{\cdot}(2\theta)_{200}$ are the angular deviations of the gravity center from the peak maximum of the {111} and {200} XRD peaks, respectively. The average crystallite size (d_{111} and d_{200}), the microstrains ($(\langle\varepsilon\rangle^2{}_{111})^{1/2}$ and $(\langle\varepsilon\rangle^2{}_{200})^{1/2}$), the dislocation density ρ and the twin density β are illustrated in Figs. 3 and 4, respectively.

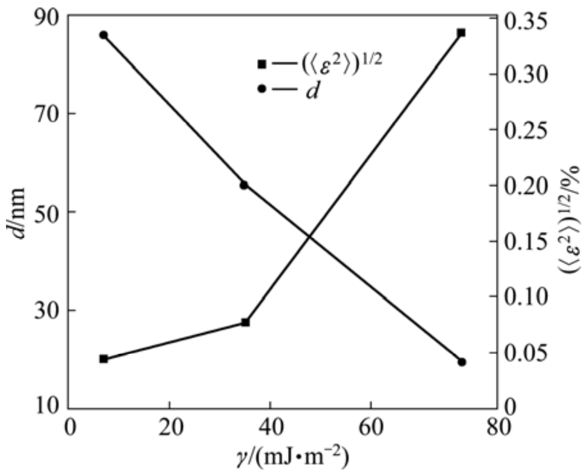

Fig. 3 XRD-measured crystallite size and microstrains of multiple passes cold-rolling processed Cu, Cu−2.2%Al and Cu−4.5%Al

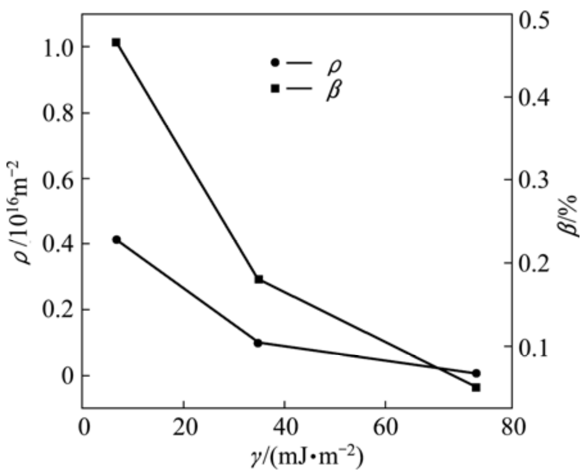

Fig. 4 Dislocation density ρ and twin density β in multiple passes cold-rolling processed Cu, Cu−2.2%Al and Cu−4.5%Al

Figure 3 shows that as the stacking fault energy decreases, the average crystallite size decreases and the mean microstrain increases. Lowering SFE increases the partial dislocation storage and enhances the grain refinement [14]. In addition, lower SFEs lead to both the increase of dislocations density and twin density. This can be explained qualitatively that a lower SFE promotes the deformation twinning and dislocation density, which promotes the refinement [6]. Figure 4 shows the influence of SFEs on the dislocation density and twin density. It shows that the rolling process gradually increases the dislocation density and the twin density. There is a simple explanation for this result. First, the reduction in the SFE promotes the splitting of the full dislocations into two partials containing a wide stacking fault ribbon; second, a lower SFE also promotes the formation of deformation twins in the cold-rolling process [19]. Dislocation nucleation and slip is a common plastic deformation mechanism in FCC metals, but the activation of the full dislocations is higher than that of the twinning partial dislocations, below which the critical stress or a critical temperature exists [20]. Twinning partial is prevalent at low temperatures or at high strain rates [21]. ZHAO et al [21] found that high density nano-scale twins were produced by means of dynamic plastic deformation at liquid nitrogen temperature in Cu. Others found that deformation twinning preferred to occur in FCC metals when deformed at low temperature [22,23]. So, there are high density nano-scale twins in alloys with low stacking fault energies at high strain rates and at low deformation temperature. As stated above, deformation twins and stacking faults play a key role in grain refinement in CR [24].

Figure 5 shows the tensile mechanical behavior of the UFG Cu, Cu−2.2%Al and Cu−4.5%Al samples. As shown in Fig. 5, the yield strength and the ultimate strength increase with decreasing stacking fault energy. The Cu−4.5%Al has the highest strength and the best ductility. Both uniform elongation (UE) and elongation at fracture are simultaneously increased by lowering the SFE. It seems to indicate that reducing the SFE results in enhanced strengthening and improved ductility by CR at liquid nitrogen temperature.

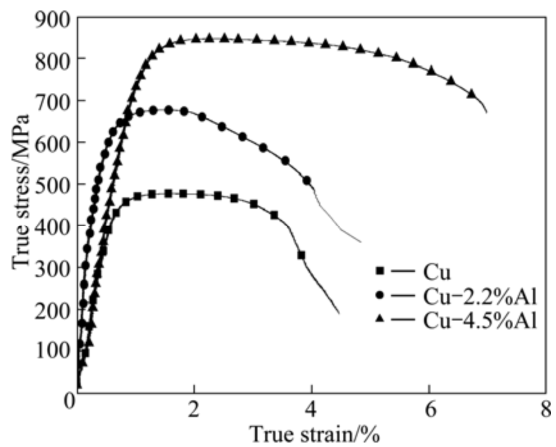

Fig. 5 True stress—strain curves of UFG Cu, Cu−2.2%Al and Cu−4.5%Al alloys

Crystallite size strengthening and dislocation–dislocation interactions, twin strengthening and solid solution strengthening are contributed to the strength of Cu−Al alloys [9]. Solid solution strengthening contribution increases linearly with $c^{2/3}$, where c is the molar concentration of the alloy [25]. For the CR samples with refined grains, the strengthening is mainly caused by dislocation-dislocation interactions, yield strength (σ_y) can be correlated to the dislocation density (ρ) following the Taylor equation:

$$\sigma_y = \sigma_0 + \alpha MGb\sqrt{\rho} \tag{3}$$

where σ_0 is the friction stress, α is a constant, M is the Taylor factor and G is the shear modulus [26].

Figure 6 shows the relationship between the yield stress of Cu−Al alloys with different molar fraction of Al and theories of strengthening including solid solution strengthening and dislocation-dislocation interactions in the same process. Such strength differences originate from the presence of twin densities in the cold-rolling samples. The coherent twin boundary (TB) which can serve as an effective sink and source of dislocations is a special kind of internal interface with low energy, as well as a barrier against the dislocation transmission [27]. So, twining strengthening and grain refinement play an important role in strengthening.

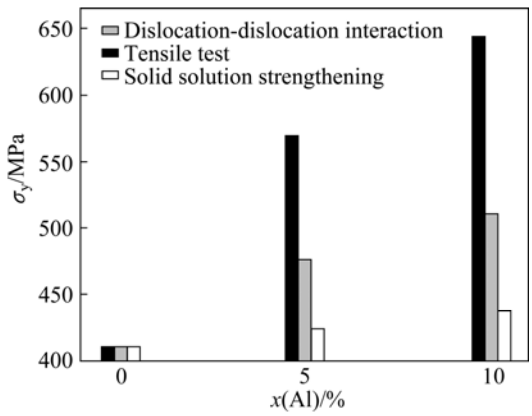

Fig. 6 Variation of σ_y for Cu−Al alloys with different Al concentration to different strengthening mechanisms

It is known that uniform elongation in tension can be determined using the CONSIDÉRÉ criterion:

$$(\frac{\partial \sigma}{\partial \varepsilon})_{\dot{\varepsilon}} \le \sigma \tag{4}$$

where σ is the true stress, ε is the true strain and $\dot{\varepsilon}$ is the strain rate [28]. So, the strain hardening rate (SHR) Θ can be defined by $\Theta = (\partial\sigma/\partial\varepsilon)_{\dot{\varepsilon}}$ [29]. This indicates that the ability of work hardening by the accumulation of dislocations can be regained consequently leading to a larger uniform elongation [7]. The UFG or NC alloys lose the strain hardening quickly once yielding occurs [30].

Figure 7 shows that the Cu−4.5%Al is qualified as the best propensity for plastic instability in the early stage of plastic deformation. The highest twin density β is found in the Cu−4.5%Al sample. TBs are much more hardenable than the conventional high-angle grain boundaries as TBs gradually lose coherence during plastic flow [27]. So, it indicates that twin boundaries block the propagation of dislocation slip and decrease the slip barrier spacing, which has a great effect on the improvement of SHR [31]. The high rate of strain hardening in Cu−4.5%Al can be correlated to the highest twin density. The average crystallite size decreases but the microstrain, dislocation density and twin density in the cold-rolling samples increase with decreasing SFE. The better ductility of UFG brass samples is derived from higher Θ , which is caused by its low SFE and high twin density [16]. Smaller crystallite size and higher twin density play a major role in the higher strength of the UFG brass. It provides an explanation for the Cu−4.5%Al alloy sample with the highest strength and best ductility.

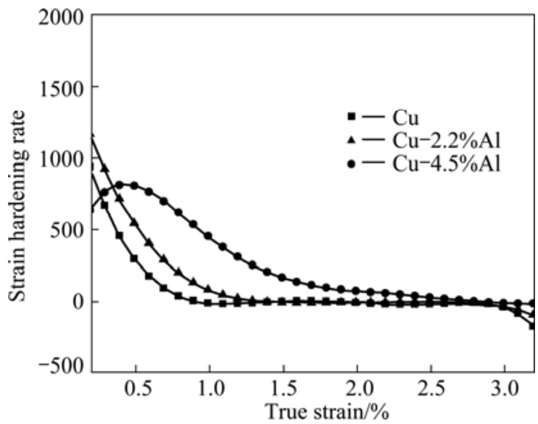

Fig. 7 Relationship of strain hardening rate and true strain

4 Conclusions

1) By means of cold-rolling at liquid nitrogen temperature, bulk ultrafine-grained or nano-grained Cu and Cu-alloy samples with different stacking fault energy are prepared.

2) X-ray diffraction measurements indicate that lower SFEs lead to the decrease in crystallite size but increase in microstrain, dislocation and twin densities in the cold-rolled samples.

3) Tensile tests results show that a decrease of SFE leads to both increasing strength and ductility in the materials. Cu−4.5%Al alloy has the best uniform elongation and ductility on account of the highest SHR.

4) It is an effective method to enhance the strength and the ductility which increases twinning by lowering SFE during CR at liquid nitrogen temperature.

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层错能对冷轧超细晶铜及铜铝合金力学性能的影响

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摘　要：在液氮温度下(77 K)采用冷轧技术制备层错能分别为 78、35 和 7 mJ/m^2 的铜、Cu−2.2%Al 和 Cu−4.5%Al 铜合金。X 射线衍射分析表明，冷轧后样品的晶粒尺寸随着层错能的降低而降低，但微观应变、位错密度和孪晶密度则升高。室温拉伸实验表明，随着层错能的降低，铜合金的强度和塑性呈增加，说明降低层错能是一种能够同时提高冷轧铜合金强度和塑性的方法。

关键词：铜；铜合金；冷轧；拉伸试验；层错能

(Edited by FANG Jing-hua)