**Introduction**

Ultra-fine grain refinement attracts attention since the grain size less than one micrometer is needed for materials for micro-machines. In recent years, severe plastic deformation (SPD) processes [1,2] have been developed as a method of the ultra-fine grain refinement. However, it is not clear how the ultra-fine grain refinement occurs during SPD [3]. The purpose of this study is to characterize the deformation structure of Cu processed by Accumulative Roll-Bonding (ARB) [1]. We will discuss the ultra-fine grain refinement by observing microstructural evolution of single crystal caused by SPD.

**Experimental**

The starting metals of ARB were single crystals of pure Cu (99.99%) grown by the Bridgman method. The single crystals having the (100) sheet-plane and the [001] rolling-direction were used. The dimensions of the sheet were 2 mm thickness, 50 mm width and 120 mm length. The single crystals were first rolled to 50% reduction and provided for ARB without lubrication at room temperature. Including the second rolling without bonding in the number of ARB cycles, ARB was carried out to various cycles up to 9 cycles. The deformation microstructure was characterized with a transmission electron microscope (TEM) and a field emission type scanning electron microscope (FE-SEM).

**Results and Discussion**

Figure 1 shows the TEM micrograph of Cu ARB processed by 9 cycles. Lamellar-boundary structure elongated to the rolling direction (RD) [4,5] are seen in Figure 1. The mean spacing of the lamellar boundaries was 320 nm along the normal direction, ND. Many dislocations inside the elongated grains are tangled as shown in Figure 1.

In the present study, the Cu single crystal with no grain boundary was used for rolling and ARB. After the ARB process of 9 cycles, we can expect 256 bonded layers with 4 µm thickness in the crystal. However, the spacing of the lamellar boundaries in Figure 1 was much shorter than this value. This shows the occurrence of grain subdivision to form new grains during the ARB process.

![Fig. 1 TEM micrograph showing the microstructure of Cu ARB processed by 9 cycles.](image)

Fig. 1 TEM micrograph showing the microstructure of Cu ARB processed by 9 cycles.

Orientations of grains in the ARB processed specimens were measured by electron back-scattering diffraction patterns. The variation of the orientations is expressed by the misorientation angle $\theta$ with respect to the orientation at a certain position. Figure 2 shows the change of the misorientation angle $\theta$ as a function of the distance $d$ along the ND direction. Figure 2 (a) shows the result after the ARB of 1 cycle. The variation of $\theta$ shown in Figure 2 (a) is gradual. Although dislocations to change local orientations in the ARB processed crystal are generated, high-angle grain boundaries with $\theta$-15deg. are not formed at this stage. Figure 2 (b) shows the result after the ARB of 4 cycles. In Figure 2 (b), the change of $\theta$ more than 15deg. occurs in a very short distance and this shows the generation of high-angle boundaries.
Regions with almost constant $\theta$ between steep changes of $\theta$ correspond to new grains formed by ARB. At $d=13\ \mu$m in Figure 2 (b), we can find the new grain with the size of about 1 $\mu$m. After the ARB of 9 cycles (Figure 2 (c)), the mean size of new grains is smaller than that after the ARB of 4 cycles. This show the occurrence of grain refinement with the increase in the ARB cycles.

Fig. 2  The relationship between the misorientation angle and the distance along ND after the ARB of (a) 1 cycle, (b) 4 cycles and (c) 9 cycles.

From Figure 2, the grain refinement can be understood as the change in the $\theta$-$d$ relationship. That is to say, initial plastic deformation causes the gradual change of $\theta$ along $d$. As the amount of plastic deformation increases, the change $\Delta \theta$ during the increment of the distance $\Delta d$ increases. When the ratio $\Delta \theta/\Delta \delta$ reaches a critical value, the $\theta$-$d$ relationship drastically changes and a new grain is formed at the location. In this case, a criterion to form a new grain is given by [6]

$$\Delta \theta/\Delta \delta = \rho b$$ (1)

where $\rho$ is the critical dislocation density to form the new grain during SPD and $b=2.56 \times 10^{-10}$ m is the amount of the Burgers vector of Cu. Evaluating the maximum value of $\Delta \theta/\Delta \delta$ from the $\theta$-$d$ relationship obtained by the present experiments, $\rho$ is given as $7 \times 10^{14}$ m$^{-2}$. This value of $\rho$ resonably corresponds to the maximum dislocation density observed in severely deformed metals and alloys. This suggests the validity of the criterion of (1).

Conclusions

Single crystals of Cu were subjected to the ARB process at room temperature up to 9 cycles. Grain refinement occurred after the ARB process. Dislocations to change local orientations are generated during the initial cycles of ARB. When the dislocation density reaches a critical value of about $7 \times 10^{14}$ m$^{-2}$, new grains and high-angle grain boundaries are formed during the subsequent cycles of ARB.

Future work

Single crystals are effective specimens to understand the mechanism of the grain refinement by SPD. Using the single-crystal specimens, the 3D structural change caused by SPD will be investigated. The mechanism of the grain refinement will be formulated with a dislocation model considering both edge and screw dislocations.

Reference


Publications

Not yet.