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Orientation Design for Enhancing Deformation Twinning in Cu Single Crystal Subjected to Equal Channel Angular Pressing**

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Slip and twinning are two major plastic deformation modes in crystalline materials.[1] In general, slip is often the predominant deformation mode and twinning is extremely difficult to occur in face-centered-cubic (FCC) crystals under conventional loading conditions.[1] In coarse-grained (CG) FCC polycrystals, for example Cu, deformation twins are indeed uncommon unless subjected to high strain rate and/or low-temperature conditions[2–6] because the existence of a large number 24 of slip systems in FCC structure makes slip a very efficient deformation mechanism. Recently, deformation twins were frequently observed in deformation induced ultrafine-grained or nanograined FCC crystals.[7–10] For instance, profuse deformation twins were found in nanocrystalline (nc) Al powder,[8] nc Al film[7] and nc Cu fabricated by severe plastic deformation methods.[9,10] In those cases, deformation twinning is an integral part of process leading to structural refinement. Those experimental results indicate that the process of deformation twinning often competes with slip under plastic deformation, and that high strain rate, low temperature, and small grain size are three major contributing factors for the occurrence of deformation twinning in FCC crystals.[2-10] The corresponding mechanism can be attributed to a constraint effect on slip under those conditions, so that twinning becomes an active deformation mode.[2–10]

Equal channel angular pressing (ECAP) has been utilized to fabricate ultrafine-grained materials world wide for a decade.[11,12] The prominent feature of ECAP is that intense shear strain is imposed along a well-defined plane around the intersection of the input and output channels.[13,14] It means that the plastic deformation of the materials along the other directions will be severely restricted by the channel wall. Therefore, the special deformation mode of ECAP may provide a possibility to effectively suppress the activity of slip systems for some specially oriented single crystals even at low strain rate and at room temperature (RT). On the other hand, FCC crystal has the definite twinning plane and direction, i.e. {111} and ⟨ 112 ⟩, respectively.[15–17] Then it gives rise to an interesting question: if a twinning system in a FCC single crystal, i.e., (111)[112], is just placed meeting one of the macroscopic shear deformation of ECAP, whether the twinning becomes active or not for the plastic deformation even at low strain rate and at RT? Obviously, the corresponding experimental results will be beneficial for better understanding of the fundamental competition mechanisms between slip and twinning in FCC crystals. In the current research, we show that deformation twinning can be significantly enhanced even in copper single crystal when the special orientation is designed to meet the macroscopic shear deformation of ECAP.

Experimental Design and Procedures

The crystallographic orientation of the Cu single crystal in the current study was designed with (111) plane perpendicular to the intersection plane of ECAP and with($\bar{1}\,\bar{1}\,2$) plane parallel to the intersection plane, as demonstrated in Figure 1. In this case, the twinning system will acquire the maximum resolved shear stress.[14] The initial crystallographic orientation of the single crystal was determined using the electron backscatter diffraction (EBSD) method. The billet with a length of 50 mm and 10 mm in diameter was cut from a large copper single crystal. Then it was processed for only one-pass in a right angle die at RT and an extrusion rate of 5mm/min and lubrication of MoS_2. After ECAP, the microstructural characterizations were performed using transmission electron microscope (TEM; JEM-2000FXII) and scanning electron microscope (SEM: Cambridge S-360). Thin foils for TEM and the samples for SEM were focused on the ID-ED plane in the center of the pressed rods. The prepared thin foils were then first mechanically ground to ~ 50 μm thick and finally thinned by a twin-jet polishing method at RT. The samples for SEM experiments were then mechanically ground using the abrasive paper and finally electro-polishing.

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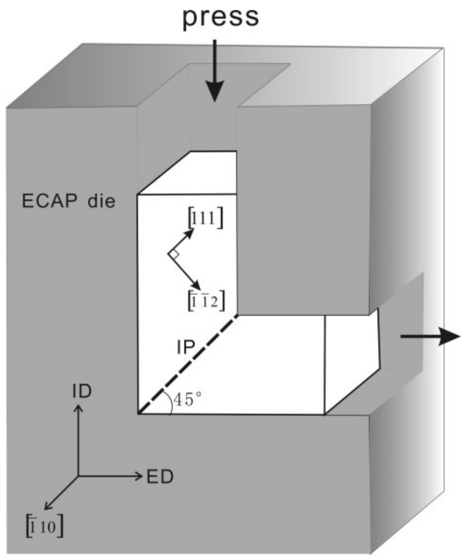

Fig. 1. Schematic illustration of the specially designed copper single crystal. (ID for insert direction, ED for extrusion direction)

Result and Discussion

SEM observations revealed that dense shear bands were formed on the shear flow plane (just the ($\bar{1}$ 10) crystallographic plane) of the copper single crystal after pressing. Those shear bands have a width range from 10 to 50 μm and make an angle of about 20° with respect to the extrusion direction. The area of the shear regions occupies about 10 % of the total area. The present experimental observations are very similar to the investigations by Paul et al.[5,6] in Cu single crystal strained by the method of channel-die. Figures 2(a) and (b) display a typical shear band, which consists of two types of microstructures. The Region I in Figure 2(b) stands for the area inside the shear band with very fine banding structures along the shear direction. The Region II in Figure 2(b) represents the area outside the shear bands with coarse ribbon structures, where stored high density of dislocations, having an interaction angle of about 70° with respect to the shear band. According to the initial orientation, it is easy to calculate that the shear bands were formed approximately aligning along the initial [112] crystallographic direction, as shown in Figures 2(a) and (b). Figure 2(c) shows the microstructures at the boundary of the shear band. It can be seen that many parallel banding structures with a width of sub-micrometers were formed. The corresponding selected area diffraction (SAD) pattern indicates that plentiful deformation twins exist inside those banding structures. The microstructures in the center of the shear band are presented in Figure 2(d), many deformation twins distribute desultorily and discontinuously in this area, as marked by the arrows in the figure. The results above clearly demonstrate that the deformation twins have been widely activated during the ECAP deformation of the current Cu single crystal even strained at RT.

Extensive observations by TEM revealed that the deformation twins in the present crystal can be categorized into three types according to their locations and morphologies. The Type I twins are located along the shear band as demonstrated in Figure 3(a) and Figure 2(d). Those profuse twins can be found in the interior of shear bands and have a length of several hundreds of nanometers and a width below 100 nm. The Type II twins, very few of needle-like ones, were often observed in the vicinity of shear bands but outside it, as presented in Figure 3(b). Their length is from several hundred of nanometers to several micrometers. Figure 3(c) shows a typical [011] high-resolution TEM (HR-TEM) image of Type I deformation twins. It can be found that the deformation twin front has a pedestal-shape. Many Shockley partial dislocations can be recognized at the twin front, as indicated by the white arrows. The Type III twins are abundant and very fine. They were widely found within the fine banding structures or within the Type I twins in the interior of shear bands, as indicated by arrows in Figures 4(a) and (b). Most Type III twins are in a scale of tens of nanometers except for few of them with a length larger than 100 nm, as clearly shown by the dark field image in Figure 4(b). Figure 4(c) is a typical HRTEM image of Type III twins with a width of several nanometers. Some stacking faults and partial dislocation activities can be seen in the vicinity of twin boundaries, as marked by the white arrows in the figure, indicating that the formation processes of those twins are related to the partial dislocation mediated process.

The internal twins are those formed within the already twinned section of the crystal, i.e. within the primary twin. The internal twins were only observed in those metals with

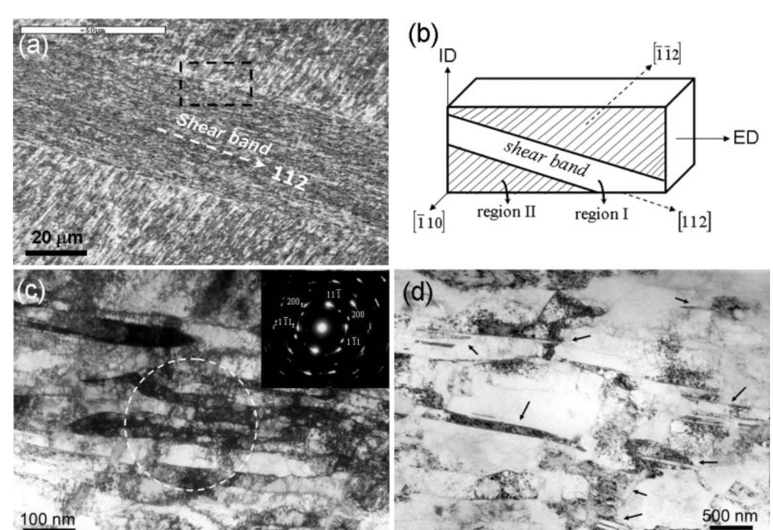

Fig. 2. (a) A typical SEM morphology of the shear bands formed after extrusion; (b) illustration of the location of shear bands in the ECAPed crystal; (c) TEM image of the area marked by square in (a) and the corresponding SAD; (d) typical microstructural morphology in the center of shear band.

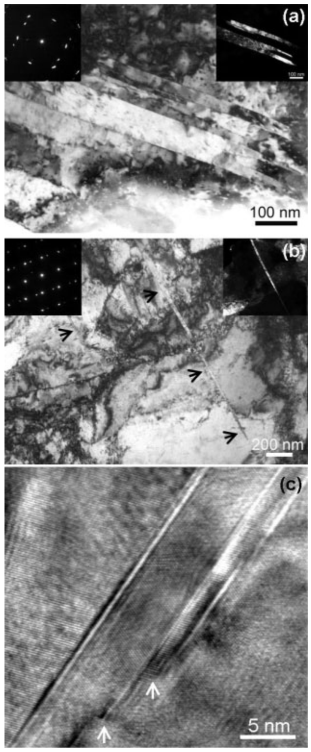

Fig. 3. TEM micrographs of deformation twins in current Cu single crystal: (a) Type I twin, located within shear band and along shear direction; (b) Type II twin, located outside shear band with a lenticular shape. The SAD patterns with zone axis [110] are shown in the left upper and the corresponding dark field images are in the right upper. (c) a typical ⎹ 011 ⎸ HRTEM image of Type I twin.

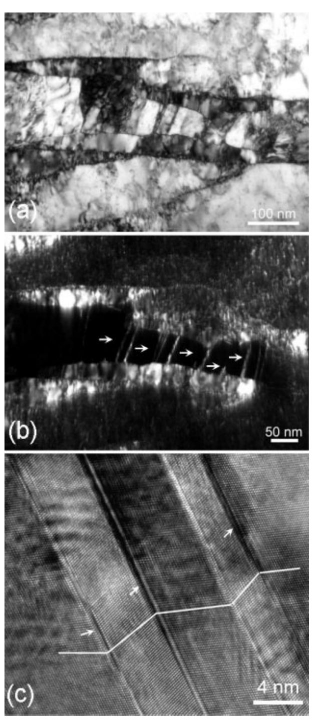

Fig. 4. (a) Bright-field TEM micrograph of Type III twin and (b) the corresponding dark field image; (c) a typical ⎹ 011 ⎸ HRTEM image of Type III twin. Some stacking faults and partial dislocations can be seen in the vicinity of twin boundary.

low stacking fault energy, such as austenitic steels[18] and β-phase titanium alloys.[19] But our research reveals that the internal twins widely exist in the present Cu specimen. Figure 5 shows a typical example of an internal twin, in which the primary twins and the internal twins are indicated by the large arrows and small arrows, respectively. It can be seen that very fine Type III twins (internal twins) often nucleated within the twinned Type I twin (primary twin) section. The corresponding selected area diffraction pattern demonstrates that the intersection angle of the primary twin and internal twin is about 70°. The formation of internal twin is mainly due to a cooperative activation of two twinning modes within the same crystal volume.[20] In the present experiment, the Cu single crystal was specially designed with one twinning system just match one of the macroscopic shear deformation during ECAP, therefore this twinning system was easily activated during extrusion. Besides, the Cu single crystal has undergone a crystallographic orientation rigid body rotation process during deformation.[14] At the initial stage, the specially selected twinning system activates; but after rigid body rotation, another twinning system (get an angle of about 70° with respect to the specially selected twinning system) acquired the maximum resolved shear stress would be activated. Therefore two twinning systems can be cooperatively activated during the process of extrusion. Due to the procedures above, it is not difficult to understand why many internal twins were found in the current Cu single crystal.

These experimental results demonstrate that Cu single crystal can deform by twinning at a very low strain rate and at RT during ECAP when a proper crystallographic orientation was specially selected with respect to the ECAP die. However, many other experiments also processed a series of copper single crystals with random orientations at RT and at low strain rate by ECAP[10,21] or other deformation methods,[22-25] only a few of deformation twins were observed. The generation of abundant deformation twins in the current Cu single crystal at low strain rate and at RT may mainly be due to the following reasons. Firstly, the special deformation

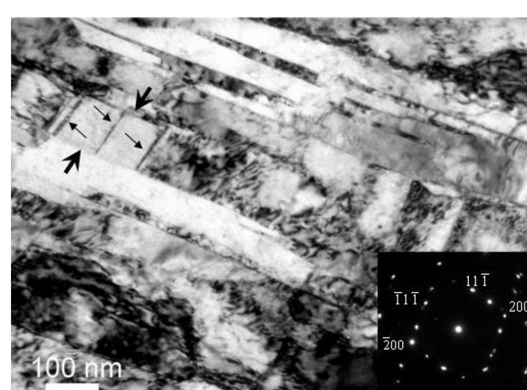

Fig. 5. A typical TEM image of internal twin. The primary twins and the internal twins are marked by large arrows and small arrows, respectively.

mode of ECAP can exert a considerable constraint on slip systems during extrusion although Cu crystal has many independent slip systems. Secondly, we designed the orientation of the Cu single crystal purposefully to make one of twinning systems just meet one of the macroscopic shear deformation of ECAP, leading to a higher level of resolved shear stress on the twinning system than those on most slip systems, which can also assist the constraint process of slip during extrusion. In addition, the microstructures were severely refined during ECAP, which can also apply a confining effect on the dislocation slip activities. Therefore, the slip activities can be suppressed to some extent under this deformation condition, and twinning would become an active deformation mode during extrusion. In terms of the micro-scale twinning mechanism, it is possible that the three types of deformation twins may nucleate via two different mechanisms. The Type I and Type II twins appear in a large scale and with a needle-like shape, which is believed to be triggered via the well known pole mechanism [10,25] because needle-like or lenticular shape is the typical configuration for the deformation twin formed by such mechanism. The twinning mechanism can be also confirmed by the corresponding HR-TEM observation, as shown in Figure 3(c). The Type III twin grows within the fine shear bands with a scale of submicro- or nano-meters. It is suggested that the Type III twin was induced by grain size effect and nucleated thought the partial dislocation mediated process, as observed by HR-TEM shown in Figure 4(c) and also as predicted by the molecular dynamic simulation previously.[26,27]

Conclusion

In summary, FCC crystals, for example Cu, have abundant (24) slip systems, which lead themselves very easy to slip, exhibiting superior plastic deformation ability without the assistance of twinning under conventional loading conditions. The special deformation mode of ECAP can successfully stimulate the formation of profuse deformation twins via suppressing the activity of some slip systems for the specially designed Cu single crystals at low strain rate and at RT after only one-pass ECAP. The results of extensive TEM observations show that the three types of deformation twins may be activated via two different mechanisms, i.e. pole-type, and partial-dislocations emitted from grain boundaries. Furthermore, slip is the dominant deformation mode in FCC crystals, but twinning is quite active even at low strain rate and at RT under the current deformation.

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