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Effect of Initial Grain Size, Die Angle and Pass Sequence on the Formation of Ultrafine Grain Structure in Cu by ECAP

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Abstract

The mechanisms of microstructural evolution in copper have been investigated by ECAP, TEM and EBSD in order to characterize the microstructure after successive passes. The effect of initial grain size, die angle and pass sequences on the formation of ultrafine grain structure have been analysed. Texture evolution is discussed based on EBSD results. These experimental results are interpreted in terms of a preliminary model with four successive stages: homogeneous dislocation distribution; elongated sub-cell formation; elongated sub-grain formation; break-up of sub-grains into equiaxed units; sharpening of grain boundaries and final equiaxed ultra fine structure.

1. Introduction

Professor T. G. Langdon has made notable advances to our understanding of creep, superplasticity (e.g., [1-3]) and the creation of ultrafine grain sized materials through severe plastic deformation [4-6]. Equal Channel Angular Pressing (ECAP), the principal method of severe plastic deformation, has been established as an effective process for producing ultra-fine grain size (<1μm) metals and alloys [7-10]. The inner angle of intersection of the channels, Φ, and the outer angle of curve, ψ, are two important parameters that determine the amount of strain applied to the sample [11]. There are specific rotation schemes of the sample between consecutive passes that determine the final microstructure of the sample [12-14]. Most commonly, four rotation schemes have been used – routes A, B_A, B_C and C [15]. Route B_C has been established as the most effective route for producing an equiaxed [16-19] microstructure, though conflicting results have been recently reported (e.g., [20]).

2. Experimental Methods

Two ECAP dies were made out of H-13 tool steel with channel intersection angle of 90° and 102° respectively. The two piece design is unique since we used horizontal split unlike normal designs where vertical split is used. This enables replacement of the lower block with channels of different angles while the top piece is still reusable. The outer arc of curvature was a smooth 20°. The channel diameter was uniformly 0.95 cm except at the entry and exit points where it were made little broader to reduce friction. Commercially pure Cu (purity <99.9%) rods with grain sizes of 17μm and 31.6 μm were machined into billets with dimensions of 6.5 cm length and diameter a little less than the channel diameter. The sample was pressed using H- 13 tool steel plunger guided by a hydraulic press. TEM analysis was carried out on 200 kV JEOL (JEM-2010, LaB_6). A part of the TEM work was done on Philips CM30. Crystallographic orientation analysis (using EBSD experiments) was done with TSL set up on Stereoscan 360 (Cambridge Instruments).

3. Results and Discussion

3.1 Effect of initial grain size and die angle

The die angle has some influence on the shear strain, as shown in Figure 1(a). The shear strains were calculated using Equation 1 [21]. The die angle of 90° results in a strain that is 5% higher than the 102° die angle. The 102° angle die was initially used to decrease friction effects and minimize die damage during extrusion.

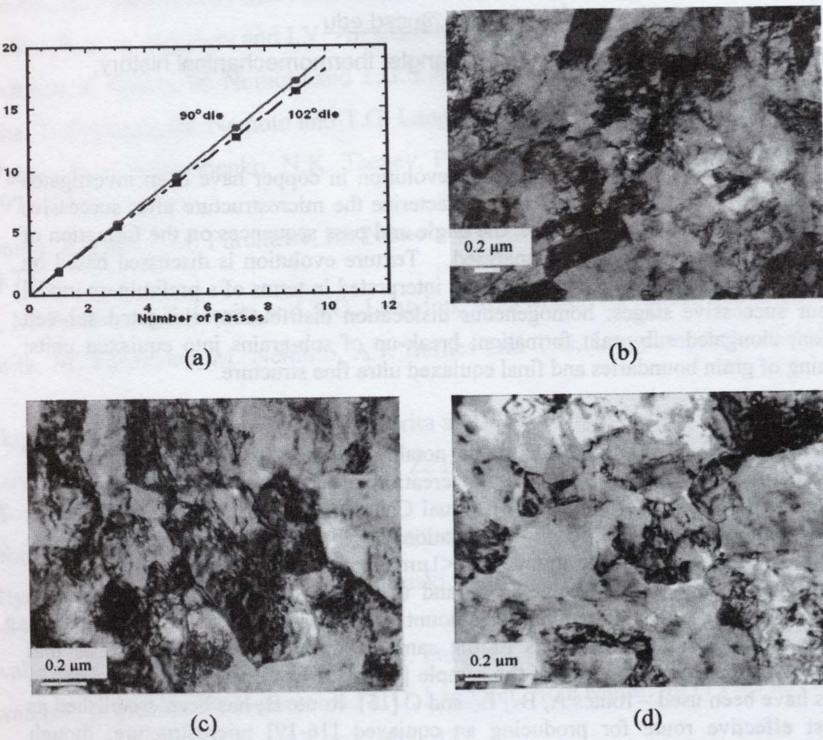


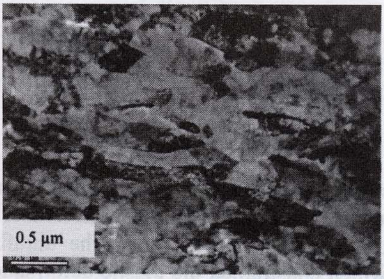
Figure 1: (a)Plot showing the effect of die angle on shear strain after different number of passes.TEM images of ECAP Cu: (b)102° die, 8 passes, initial grain sizes-31.6 μm (c) 90° die, 8 passes, intial grain sizes- 31.6 μm (d) 102° die, 8 passes, initial grain sizes- 17μm.

$$\varepsilon_N = \frac{N}{\sqrt{3}}\left[2\cot\left(\frac{\Phi}{2}+\frac{\Psi}{2}\right)+\Phi\,\mathrm{cosec}\left(\frac{\Phi}{2}+\frac{\Psi}{2}\right)\right] \quad (1)$$

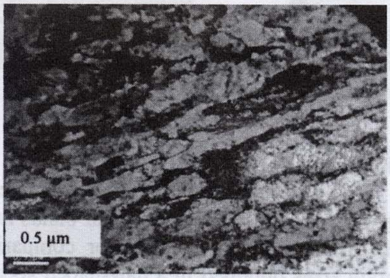
Indeed, the die angle had little effect on the microstructure produced. Two characteristic microstructures in the plane perpendicular to extrusion direction after eight passes are shown in Figure 1 (b) and (c). The grain sizes are approximately 0.5 μm and the grains are equiaxed in the plane. Similarly, the initial grain size shows little effect after eight passes. Figure 1 (b) and (d) show the microstructure of specimens subjected to eight passes (102° die) with initial grain sizes of 31.6 μm and 17μm respectively. The microstructure is analogous to the one shown in Figures 1 (b) and (c) for the same initial grain size of 31.6 μm and different die angles (90° and 102°).

3.2 Effect of pass sequence

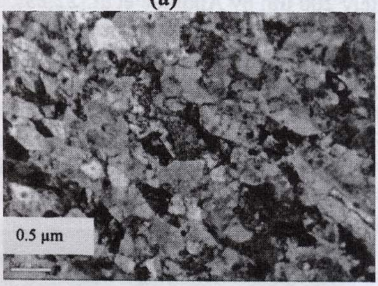
The evolution of microstructure, especially texture from the initial condition to eight passes, was found to be similar for samples produced from 90° and 102° dies. For 90° die, focus here is on comparison between 8 pass samples produced by different rotation schemes – routes B_C, A and C. As Figure 2 shows, routes A and C produced elongated grains while route B_C resulted in a more equiaxed microstructure.



(a)



(b)

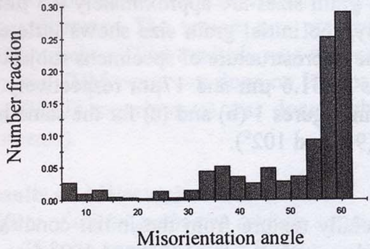


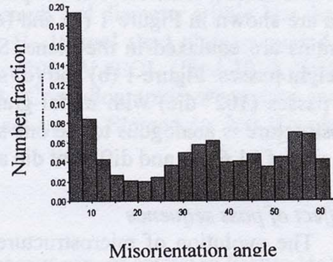
(c)

Figure 2: Bright field TEM images (sample parallel to extrusion axis) of samples produced from 90^0 die using three routes (8 passes); (a) route A- no rotation, (b) route C- 180^0 rotation route B_C- 90^0 rotation.

3.3 *Misorientation angle*

Figure 3 shows the misrorientation between adjacent grains as a function of the number of passes. For the initial material, Figure 3(a), the majority of the grain boundaries are high angle boundaries (30-60°). For two passes, the situation is dramatically changed: approximately 30% grains have angles lower than 12°. The remaining boundaries have orientations over a broad range. As the number of passes increase, the fraction of high angle grain boundaries increases, as shown in Figure 3(c).





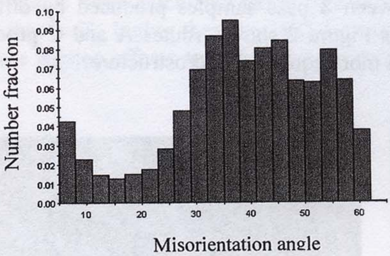
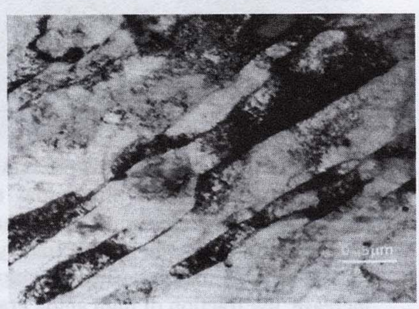


Figure 3: Distribution of misorientation angles for samples processed by 102^o die using route B_C: (a) Initial condition(grain size-17 μm) (b) 2 passes(c) 4 passes.

4. Evolution of Microstructure

The similarity between the microstructures produced by ECAP and the ones generated within adiabatic shear bands is striking, in spite of the significant differences in thermomechanical history. For comparison purposes, Figure 4(a) shows the ultrafine grain sizes obtained in a hat-shaped specimen which constrained the plastic deformation in a narrow region with thickness of approximately 200 μm by Andrade et al.[22,23]. The shear strain imparted dynamically was approximately equal to 4. A grain size of ~0.1 μm was produced. Figure 4(b) shows the ECAP structure with equivalent shear strain (two passes). The grain size is fairly similar, with a greater grain-boundary waviness observed after ECAP. This is indeed surprising, considering the major differences in strain rate (approximately 1 s^{-1} for ECAP and 104 s^{-1} for hat shaped specimen) and thermal history (successive thermal spikes after each pass for ECAP and adiabatic heating to T= 600 K for hat-shaped specimen).

(a)



(b)

Figure 4: TEM micrographs of Cu subjected to shear strain of 4; (a) hat-shaped specimen ($\dot{\gamma} \approx 10^4$ s^{-1}),(b) ECAP: 2 passes(~ $\dot{\gamma} \approx 1$ s^{-1})

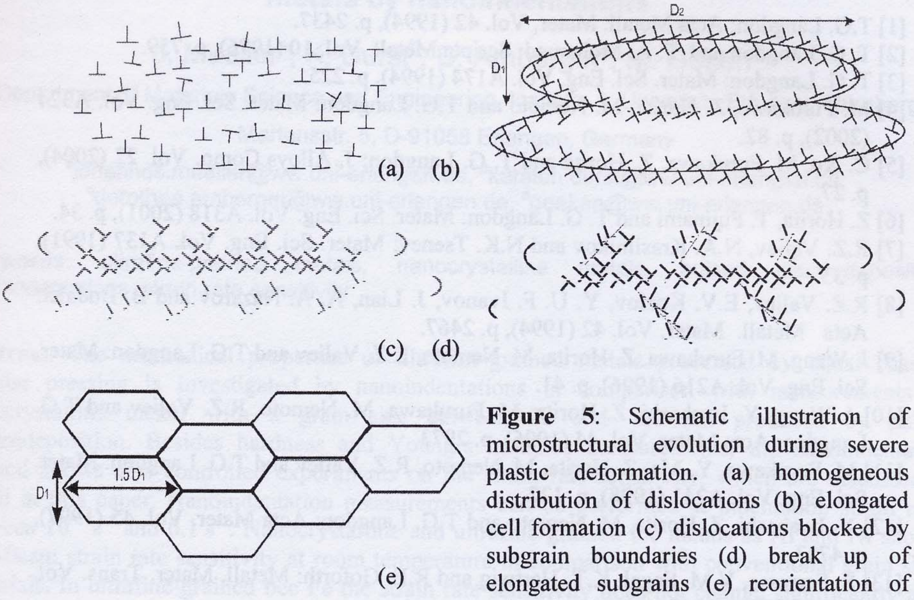


Figure 5: Schematic illustration of microstructural evolution during severe plastic deformation. (a) homogeneous distribution of dislocations (b) elongated cell formation (c) dislocations blocked by subgrain boundaries (d) break up of elongated subgrains (e) reorientation of subgrain boundaries and formation of ultrafine grain size.

A model for grain-size refinement was proposed by Meyers et al. [24,25] for adiabatic shear bands. The principal features of this model are shown in Figure 5. The sequence of events leading to the formation of the ultrafine grain size is self evident from Figs. 5(a-e). This model is entirely consistent with the misorientation evolution observed in Figure 3. A homogeneous dislocation distribution is replaced by energetically favorable subgrains. These subgrain walls present a barrier for dislocation propagation and their misorientation between adjacent grains increases gradually with plastic strain (Fig. 5c). Meyers et al.[25] showed that elongated sub-grains are preferred over equiaxed ones and calculated the optimum aspect ratio. The thickness of these oblate spheroids ultimately determines the grain size; upon subdivision, the equiaxed structure is formed. Thermal energy assists in creating “clean” boundaries. One of the differences between Fig. 4(a) and (b) is that the grain boundaries are more curved (indicating mobility) in the ECAP produced sample. It is to be noted that the deformation time for shear band formation is extremely low ($\sim 2.5 \times 10^{-5}$ s) and temperature as high as 600 K is reached in the deformation process. On the other hand, during ECAP deformation time is high (to the order of a few seconds) while the expected temperature rise is much lower as compared to the shear band temperature rise. A detailed analysis is being carried out.

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