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Microstructural characterizations of Cu processed by ECAP from 4 to 24 passes

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Abstract. The microstructures of pure Cu processed by equal channel angular pressing (ECAP) from 4 to 24 passes were investigated. It was found that the microstructures of Cu samples with a small number of ECAP passes (4-8) were not inhomogeneous and the fraction of high-angle grain boundary (HAGB) was low (25~43%). While for the samples with many number of ECAP passes (12-24), the grains became more equiaxed-like and the GB misorientations exhibited double-peak distribution with high fraction (51~64%) of HAGB. It was dislocation cells formed in large grains of the few-pass samples, but subgrains in the many-pass samples. These characterizations suggested that ultrafine-grained (UFG) microstructures in the few-pass samples were not fully accomplished, while it was obtained after many passes (>12). It is believed that dynamic recovery during processing for many passes was attributed to the formation of UFG microstructures.

Introduction

The technique of ECAP has ability to apply high shear strain (~1) into metal rods pass by pass without reduction of cross-section area [1]. As a result, a large grain can be decomposed into several small ones by a series of dislocation activities [1-3]. Thus, this method can be used to refine grain size in order to obtain materials with high strength and good ductility.

By checking the available documented results, many metals processed by ECAP for 4-10 passes at room temperature (RT) were commonly referred to as UFG materials [3-8], which might be due to their small grain sizes. However, there are also some investigations on microstructures indicating that the metals processed by ECAP with no more than 10 passes should be characterized in terms of *deformation structure* rather than being classified as “UFG” materials [9-11]. One strong experimental evidence to support this understanding was that the analysis of GB misorientation in ECAP Cu after 8 or less passes gave low fraction (30~40%) of HAGB (>15º), regardless of whether transmission electron microscopy (TEM) or electron back-scatter diffraction (EBSD) techniques was used [9-11]. Besides, some authors further indicated that the small grain size (0.2~0.3 μm) of ECAP Cu with similar passes should correspond to the spacing between boundaries including slightly misoriented subgrains, when measured under TEM [9]. Therefore, the conclusion was drawn that the microstructures consisting of low angle and high angle boundaries were still similar to the structures observed after other deformation processed [9-11].

In general, no matter whether a microstructure is a UFG structure or not, several important characteristics should be taken into account overall, including grain size and its distribution, the distribution of GB misorientation and the fraction of HAGB, grain shape and its aspect ratio etc. Therefore, the purpose of this work is to distinguish the microstructures of ECAP Cu with 4-24 passes and evaluate their characteristics.

Experimental

The rods (Φ 10 mm×85 mm) of commercial pure Cu (99.97%) were processed by ECAP from 4 to 24 passes (with route Bc). The ECAP processes were performed at room temperature, using a die with two channels intersecting at an angle of 90º. The detailed procedures can be found in reference [12]. Samples for microstructural characterization were cut from the as-processed rods perpendicular to its longitudinal axis (*X* plane [12]). EBSD measurements were performed on a LEO-Supra 35 FEG-SEM. Two different areas 12.5 × 12.5 μm^2 in size were scanned by a step of 50 nm for each sample. The raw data were processed by HKL-Channel 5. Due to the spatial resolution of the EBSD system, misorientations of less than 2º were not identified. For measuring grain size, the method of equivalent circle diameter was used. TEM observations were performed on JEM-2000FX II, operating at 200 Kv.

Results and discussion

Fig. 1 presents the EBSD images of Cu after ECAP deformation for 4-24 passes. It is clear that though ultrafine grains are created somewhere in the few-pass samples, there are still many regions in size of several microns, in which boundaries with small angle (<15º) are prevalent, as shown in Figs. 1(a) and (b). With increasing ECAP passes up to 12 or more, the microstructures become homogeneous together with smaller grains, as shown in Figs. 1(c)-(f). Furthermore, the grain shape also becomes more equiaxed-like, as implied by a decrease in average grain aspect ratio from 1.57, 1.17 to 1.20 for the 8, 16 and 24-pass samples, respectively.

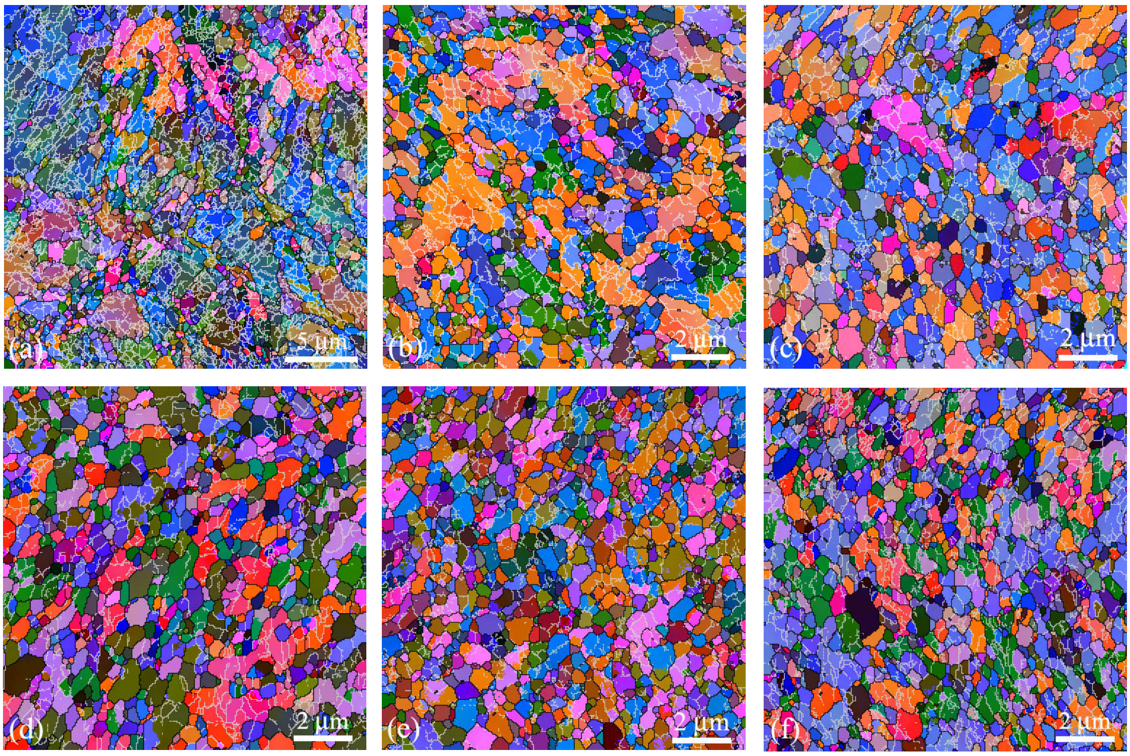

Fig.1 EBSD images of Cu after ECAP deformation for (a) 4, (b) 8, (c) 12, (d) 16, (e) 20, (f) 24 passes. Thick black lines mark boundaries with misorientation >15º (HAGB), while thin gray lines mark boundaries with misorientation <15º.

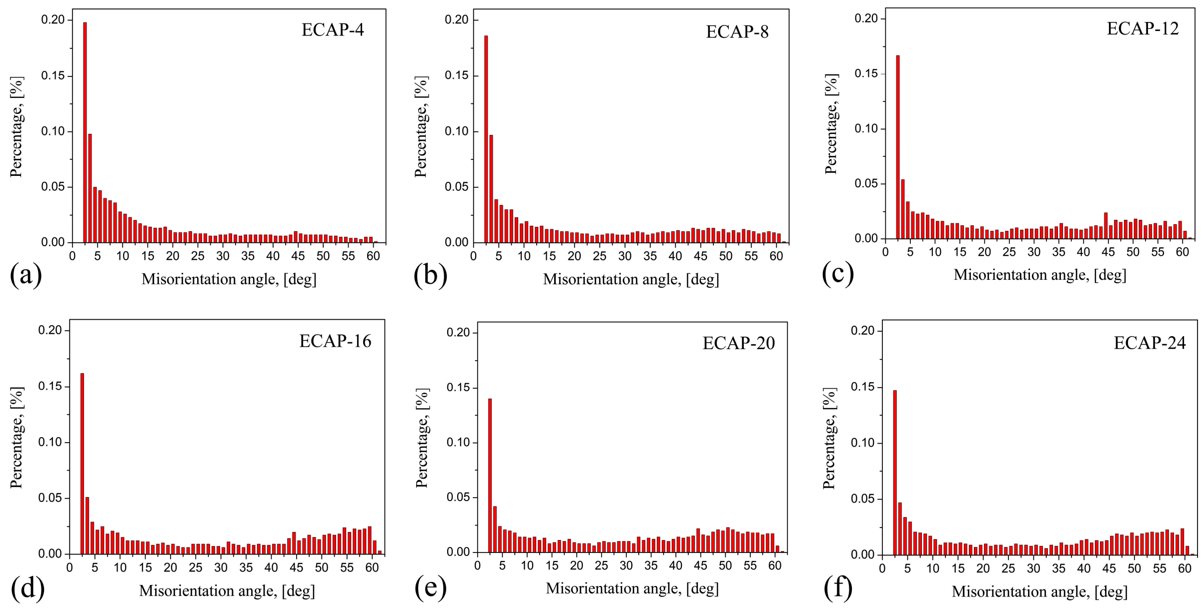

Fig.2 The distribution of GB misorientations of ECAP Cu: (a) 4, (b) 8, (c) 12, (d) 16, (e) 20, (f) 24 passes.

Fig. 2 shows the distribution of GB misorientations of ECAP Cu. It can be seen that the low angle boundaries dominate most fraction of boundaries in the few-pass sample, as shown in Figs. 2(a)-(c). The fractions of HAGB are only 33.7% and 42.8% for the 4 and 8-pass samples, respectively. By contrast, the numbers are increased up to 57.9% and 62.4% when the number of ECAP passes is 16 and 24, respectively. Moreover, Figs. 2(d)-(f) show that small peaks appear in the high angle region (50º ~60º), which suggests that more low angle boundaries have transformed into HAGB with increasing number of ECAP passes.

The average grain size and the fraction of HAGB with increasing ECAP passes are collected in Fig. 3. It can be seen that the average grain size continuously decreases from 510 nm for the 4-pass sample to 345 nm for the 24-pass sample. While the fraction of HAGB increases to a high level (~60%) after 16 passes, the rate of decrease gradually slows down. The highest fraction of HAGB that can be obtained in ECAP Cu with a similar number of passes is likely to be about 70%, as reported in previous investigations [13,14], suggesting that the amount of HAGB cannot be increased without limit under condition of severe plastic deformation.

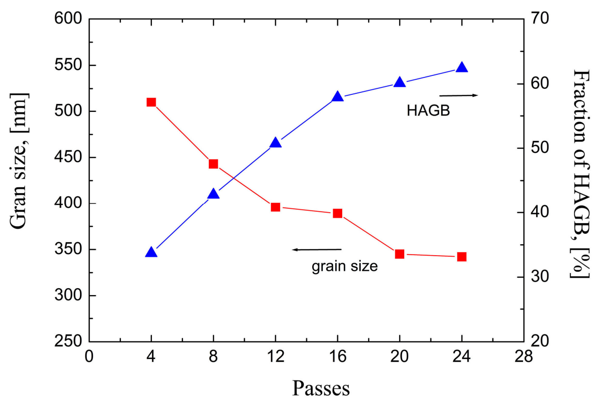

Fig.3 Average grain size and the fraction of HAGB vs. ECAP passes. All data were obtained from EBSD.

From the results presented above, the formation of UFG microstructures is not fully accomplished after 8 or less ECAP passes, because of the presence of many large regions with micron size and high fraction of low angle boundaries (~60%). TEM observations confirm that there are many dislocation cells divided by slightly misoriented boundaries in large-sized grains, as shown in Fig. 4(a). Therefore, more ECAP passes are needed in order to make these cells become grains. The increase in the fraction of HAGB in the many-pass samples and the decrease in the average grain size imply that more cells have transformed into real grains after many passes. This process is mainly involved in the evolution of boundaries from dislocation boundaries to GBs. According to the theory of grain subdivision, strain-induced dislocations arrange themselves into various dislocation configurations, such as the geometrically necessary boundary, polygonized dislocation wall and dense dislocation wall etc [15]. With increasing strain, polygonized dislocation walls (with misorientation <1°) are first converted into partially transformed boundaries (1~5°), then into conventional GBs [16]. All these transformations take place via the process of generation, arrangement and dissociation of dislocations - a kind of dynamic dislocation recovery under plastic deformation. Fig. 4(b) shows the microstructures taken from the 24-pass sample. Subgrains with free of dislocations but not dislocation cells can be seen, indicating that massive dislocation annihilations have occurred. It is believed that a strong dislocation recovery during ECAP deformation for many passes is favorable for increasing the fraction of HAGB and decreasing grain size.

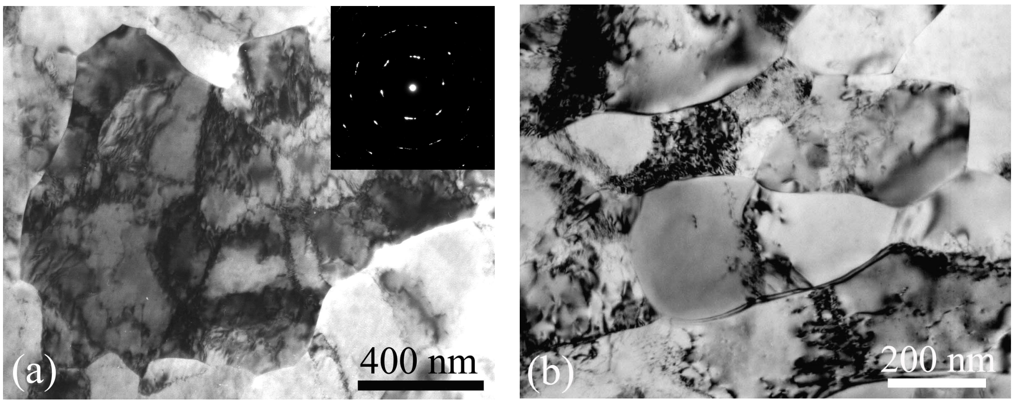

Fig.4 TEM micrographs of ECAP Cu, showing different sub-microstructures: (a) 8, (b) 24 passes. The inset of (a) is the corresponding SAD pattern with zone axis [011].

Summary

According to the present experimental results, UFG microstructures are not fully accomplished due to the high fraction of low angle boundaries and large regions with micron size when the number of ECAP passes is 8 or less. In order to obtain genuine UFG microstructures, more ECAP passes are needed. After 16 or more passes, really homogeneous UFG microstructures with average grain size of ~350 nm and high fraction of HAGB (>60%) can be achieved in Cu. The increase in the fraction of HAGB is attributed to dislocations recovery during ECAP process.

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