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2014 IOP Conf. Ser.: Mater. Sci. Eng. 63 012097

(http://iopscience.iop.org/1757-899X/63/1/012097)

IOP Conf. Series: Materials Science and Engineering **63** (2014) 012097 doi:10.1088/1757-899X/63/1/012097

Effect of multidirectional forging and equal channel angular pressing on ultrafine grain formation in a Cu-Cr-Zr alloy

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Abstract. The microstructure evolution was investigated in a Cu-0.3%Cr-0.5%Zr alloy subjected to large plastic deformation at temperature of 400 °C. Two methods of large plastic deformation, i.e., equal channel angular pressing (ECAP) and multidirectional forging (MDF) were used. The large plastic deformations resulted in the development of new ultrafine grains. The formation of new ultrafine grains occurred as a result of continuous reaction, i.e., progressive increase in the misorientations of deformation subboundaries. The faster kinetics of microstructure evolution was observed during MDF as compared to ECAP. The MDF to a total strain of 4 resulted in the formation of uniform ultrafine grained structure, while ECAP to the same strain led to the heterogeneous microstructure consisting of new ultrafine grains and coarse remnants of original grains. Corresponding area fractions of ultrafine grains comprised 0.23 and 0.59 in the samples subjected to ECAP and MDF, respectively.

1. Introduction

The formation of ultrafine grained structure during large plastic deformation is one of the most discussed topics nowadays. Since ultrafine grained structure provides high yield strength in accordance with Hall-Petch relationship [1], obtaining ultrafine grained metals and alloys has a great practical potential for engineering application. The most effective methods to produce ultrafine grained structures are based on large plastic deformations, for example equal channel angular pressing (ECAP), multidirectional forging (MDF), accumulative roll bonding (ARB), high pressure torsion (HPT) and etc. [2]. The development of ultrafine grained structures during large plastic deformation is usually considered as process of dynamic recrystallization (DRX) [3]. There are two types of dynamic recrystallization: discontinuous dynamic recrystallization (dDRX) and continuous dynamic recrystallization (cDRX) [3]. At low-to-moderate temperature of large plastic deformation the major mechanism of microstructure evolution is cDRX. The cDRX includes the development of a large number of strain-induced low-angle subboundaries at an early stage of deformation and then the progressive increase in the misorientations among the subgrains leading to the formation of ultrafine grains surrounded by high-angle boundaries at large strains. The new grain formation is strongly affected by starting materials and deformation conditions, such as the temperature, the strain rate and etc. [4-6]. The presence of fine dispersoids in the starting materials as well as reducing the initial grain size affects remarkably the kinetics of ultrafine grain formation during large strain plastic deformation [7-9]. On the other hand, a decrease of processing temperature slows down the kinetics of cDRX although results in smaller grain size evolved at sufficiently large strains [10]. However, despite of numerous investigations devoted to the influence of starting material and processing conditions on the kinetics of grain refinement, the effect of the processing methods on the ultrafine grained structure formation has not been studied in sufficient detail. The aim of present work is to clarify the effect of methods of large plastic deformations on the microstructure evolution in a Cu-Cr-Zr alloy.

2. Experimental Procedure

A copper alloy with the following chemical composition, Cu–0.3wt.%Cr–0.5wt.%Zr, was studied. The specimens were subjected to a solution treatment at 920 °C for 30 min with subsequent aging treatment at 450 °C for 1 h (peak aged conditions). The multidirectional forging (MDF) and the equal channel angular pressing (ECAP) were chosen as methods of large plastic deformation. The plastic deformations were carried out under isothermal conditions at 400 °C to various total strains up to 4. The dies were preheated using high temperature furnaces and the temperature was controlled automatically with accuracy of ± 2°. The samples with starting dimensions of 25 mm × 20 mm × 16 mm were machined for MDF. The MDF was carried out with consequent change in the deformation axis through 90° from pass to pass. The true strain applied in each pass was about 0.4 and a strain rate was approximately 10^{-3} s^{-1}. After each forging pass, the samples were water quenched and then reheated to the test temperature during 15 min. The billets of 14 mm × 14 mm × 90 mm were subjected to ECAP via route B_C (90° anticlockwise rotation of the specimens after each pass) at a strain rate of 1 s^{-1}. The samples for ECAP were preheated during 30 min. A die angle of 90° was chosen that resulted in a true strain of about 1 at each pass. The deformed samples were quenched in water after complete ECAP cycle, i.e. after reaching the desired total strain.

The microstructure of deformed samples was examined by a Quanta 600 FEG scanning electron microscope (SEM) equipped with an electron backscattering diffraction (EBSD) analyzer incorporating an orientation imaging microscopy (OIM) and a Jeol JEM-2100 transmission electron microscope. The strain hardening was measured by means of microhardness tests. The microhardness tests were carried out using a WOLPERT 420MVD with a load of 300 g and holding time of 10 s. The microstructural investigations were performed on the sections parallel to the final forging axis for the MDF samples and on the Y plane or flow plane parallel to the side face at the point of exit from the die [11] for the ECAP samples. The samples for SEM observations were mechanically polished on 1000 grit SiC paper with subsequent electropolishing using an electrolyte of HNO_3:CH_3OH=1:3 at room temperature with a voltage of 10 V. The step size for EBSD scanning was 100 nm for the samples strained to a total strain of around 1, and 60 nm for the samples processed to total strains of 2 and 4. An average grain size was estimated by linear intercept method as a distance between high-angle boundaries on the obtained EBSD maps. The fraction of ultrafine grains (UFG) was obtained using OIM software (EDAX TSL, version 5.2).

3. Results and Discussion

3.1 Strain hardening and microstructure evolution

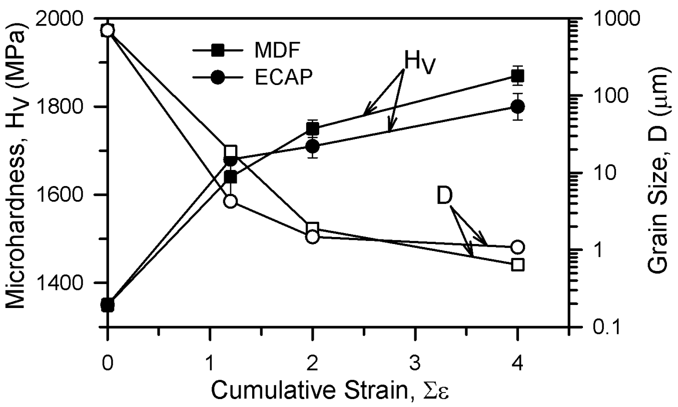

Figure 1. Effect of large plastic deformation on the microhardness (H_V) and the grain size (D) in a Cu–Cr–Zr alloy.

The effect of large plastic deformation on the microhardness is shown in figure 1. It is apparent that the large plastic deformation has a significant influence on the microhardness irrespective of the processing method. The microhardness increases from 1350 MPa in the initial state to above 1600 MPa after processing to a total stain around of 1. Then the strain hardening increment decreases leading to gradual increase in the microhardness upon further straining. Note here that at the relatively small strains around 1, the microhardness of samples processed by ECAP is a little bit higher than that after MDF. On the other hand, the opposite effect of the processing method on the hardening is observed at larger strains. Namely, the MDF samples exhibit higher microhardness than ECAP ones. This divergence increases with increasing the total strain. After processing to a strain of 4, the microhardness comprises 1870 MPa and 1800 MPa in the MDF and ECAP samples, respectively. The strain dependence of grain size correlates with the strain hardening. Commonly, the average grain size decreases with increase in the total strain (figure 1). At relatively small strains of about 1, the grain refinement is more pronounced after ECAP as compared to MDF, the corresponding average grain sizes comprise 4 μm and 18 μm. Further processing to a total strain of 4 is accompanied by a gradual decrease of the grain size to 1.1 μm and 0.6 μm after ECAP and MDF, respectively.

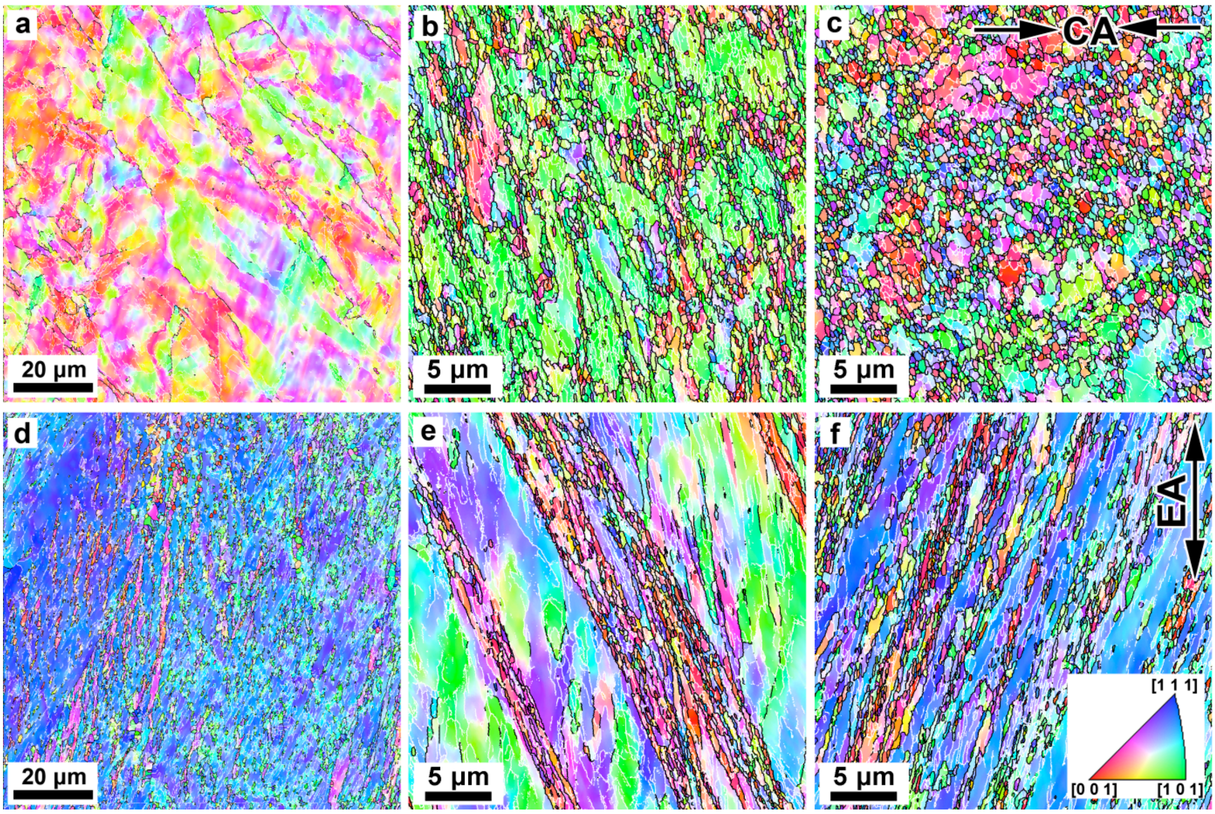

Figure 2. Deformation microstructures in a Cu–Cr–Zr alloy subjected to multidirectional forging (a-c) and equal channel angular pressing (d-f) at temperature of 400°C to a total strain of 1.2 (a) 1 (d); 2 (b, e); 4 (c, f). The white and black lines indicate the low- and high-angle boundaries, respectively. The inverse pole figures are shown for the last compression axis (CA) and the extrusion axis (EA).

The deformation microstructures evolved in a Cu–Cr–Zr alloy during large plastic deformation are shown in figure 2. The deformation to a relatively small strain of about 1 brings about the development of a great number of low-angle strain-induced subboundaries after both the MDF and ECAP processing (figures 2a and 2d). Note here that there is a difference in the deformation microstructures even after small strains. The number of the strain-induced high-angle boundaries is larger in the ECAP samples than in the MDF ones. An increase in the total strain to about 2 leads to the formation of new ultrafine grains (figures 2b and 2e). A remarkable difference between microstructures evolved during the MDF and ECAP can be seen in figure 2. The sharp deformation microbands develop during ECAP resulting in the formation of a bimodal microstructure (figure 2e). The deformation microbands consist of a great number of the new ultrafine grains with an average size below 1 μm. These grains are surrounded by moderate-to-high angle boundaries. Between the deformation microbands, there are areas of original grains consisting of dislocation subgrains with low-angle boundaries. The spacing of these remnants of original grains is about of 10 μm. An increase in the total strain to 4 by ECAP does not affect remarkably the formation of new ultrafine grains, although the average size of original grain remnants becomes less than 5 μm (figure 2f). On the other hand, the MDF results in the homogeneous microstructure evolution (figure 2b). The formation of new ultrafine grains occurs nearby the initial grain boundaries. Such homogeneous formation of microstructure during MDF may be associated with the changes in the forging axis from pass to pass. As a result, the MDF leads to the development of ultrafine grained structure at a strain of 4. The new ultrafine grains are characterized by almost equiaxed shape. Besides the new ultrafine grains, the microstructure of MDF samples includes small remainders of the original grains (figure 2c).

The fine substructures evolved during MDF and ECAP to strains of 1 to 4 are presented in figure 3. The MDF to a relatively small strain of about 1.2 results in the development of large number of strain-induced subboundaries with low-angle misorientation crossing over the original grains (figure 3a). Further MDF is accompanied by an increase in the subboundary misorientations leading to the formation of relatively equiaxed subgrains surrounded by moderate-to-high angle boundaries (figure 3b). The fine microstructure after MDF to a strain of 4 consists of largely misorientated grains/subgrains (figure 3c). On the other hand, the different fine microstructures are evolved during the ECAP. The first pass of ECAP leads to the evolution of the spatial network of the planar low-angle boundaries (figure 3d). The main distinctive feature of the ECAP samples can be clearly observed after the second pass. The sharp deformation microbands, which include a large number of new ultrafine grains, appear in the deformation microstructure (figure 3e). The ring-like diffraction pattern that obtained from the deformation microband region indicates high-angle misorientations between grains within the microband. In contrast, the single spot diffraction pattern taken from neighboring area suggests low-angle misorientations among dislocation cell substructure (figure 3e). The fraction of ultrafine grained structures gradually increases upon further processing (figure 3f).

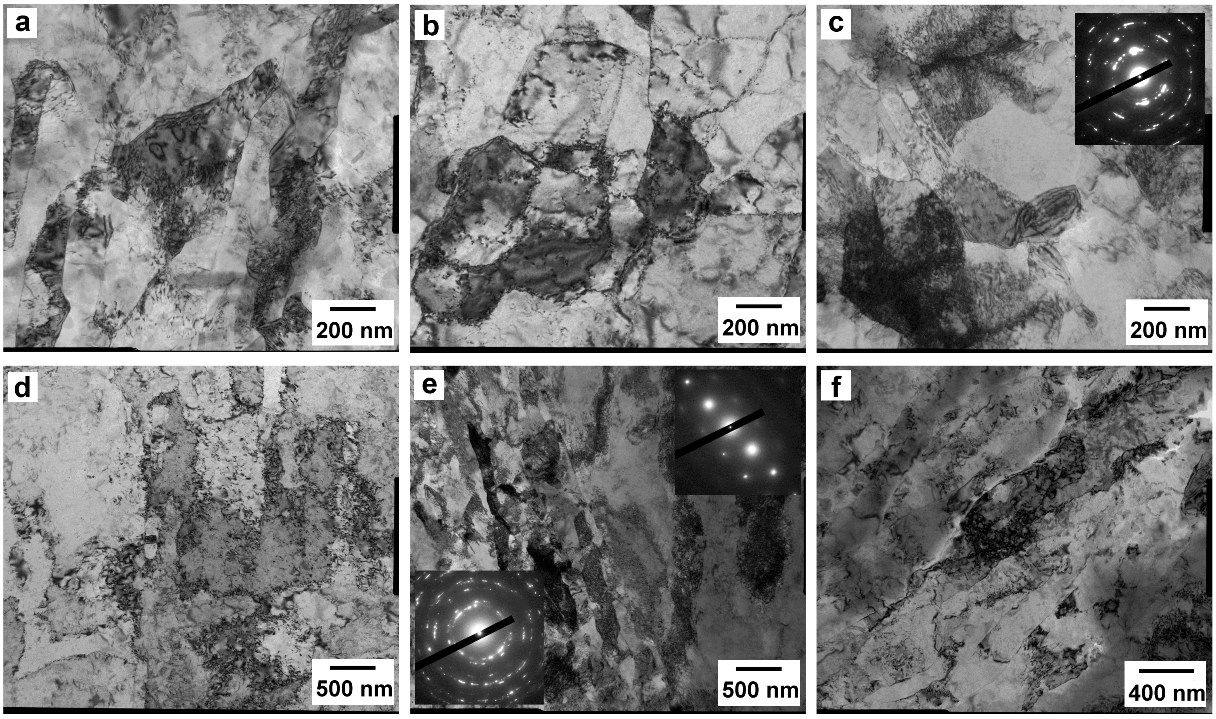

Figure 3. Fine deformation substructure in a Cu–Cr–Zr alloy subjected to multidirectional forging (a-c) and equal channel angular pressing (d-f) at temperature of 400 °C to a total strain of 1.2 (a), 1 (d); 2 (b, e); 4 (c, f). The size of selected-area diffraction in (c, e) was 1.4 μm.

Detailed investigation of microstructure evolution suggests that the method of large plastic deformation has a significant effect on the development of new ultrafine grains (figure 4). It is clearly seen in figure 4 that there is remarkable difference in the grain size distributions in the samples processed by the MDF and ECAP. The deformation by the MDF to relatively small strain of about 2 leads to the appearance of a peak against grains with a size less than 2 μm, whereas any remarkable peaks on the grain size distribution do not appear after the ECAP to the same strain (figures 4a and 4b). Further MDF to a strain of 4 is accompanied with a significant increase in the area fraction of these ultrafine grains (figure 4c). The ECAP to a large strain of 4 also leads to the remarkable increase in the ultrafine grain fraction, which is clearly revealed in figure 4 by a peak for grain sizes below 2 μm (figure 4d). However, the peak corresponding to the ultrafine grains ($D \leq 2$ μm) in the MDF sample is almost three times higher than that evolved in the ECAP sample.

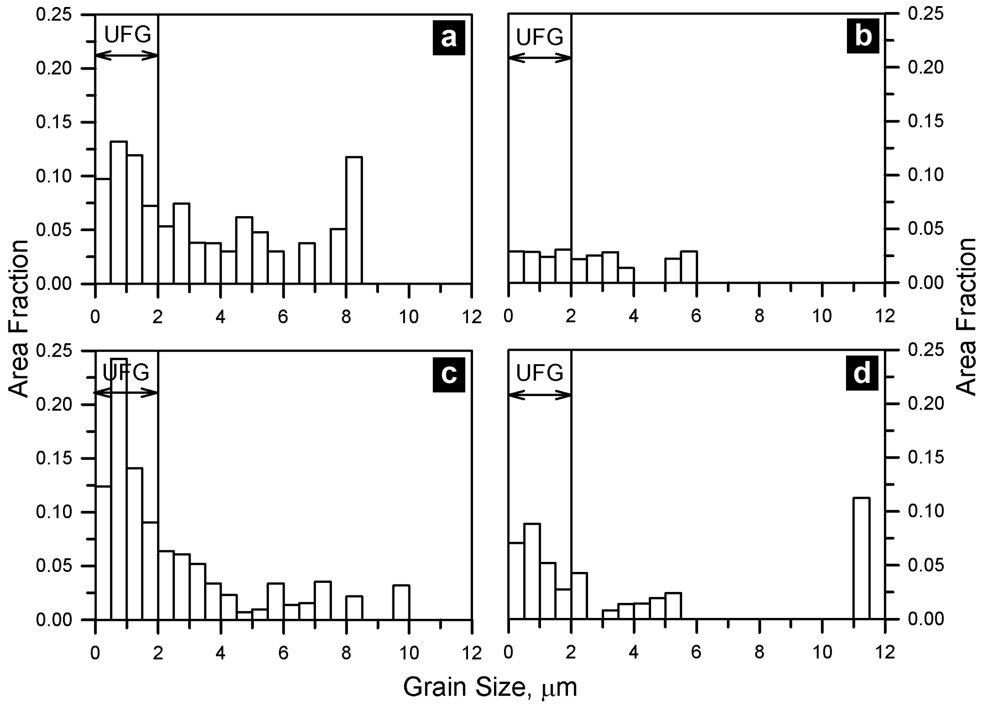

Figure 4. Grain size distributions for Cu–Cr–Zr alloy processed by multidirectional forging (MDF) (a, c) and equal channel angular pressing (ECAP) (b, d) to total strains of 2 (a, b) and 4 (c, d).

3.2. Kinetics of continuous dynamic recrystallization

The kinetics of continuous dynamic recrystallization during large plastic deformation can be represented by strain dependencies of the high-angle boundaries fraction and/or the fraction of ultrafine grains (figure 5). The rapid increases in the high-angle boundary fraction to 0.36 takes place after the first pass of ECAP. Then, the fraction of high-angle boundaries gradually approaches 0.49 with increase in the strain to 4. In contrast, the fraction of ultrafine grains almost linearly increases to 0.23 during the ECAP to total strain of 4. This behavior can be associated with the formation of bimodal microstructure, i.e. the deformation microbands comprising a large number of new ultrafine grains and the work hardened areas of initial grains involving low-misoriented dislocation substructures.

On the other hand, the fast development of high-angle boundaries during the MDF occurs in the strain range of 1.2 to 2, when the high-angle boundary fraction increases from 0.1 to 0.48 (figure 5). Then, the fraction of high-angle boundaries gradually increases to 0.54 during subsequent MDF to the total strain of 4. The strain effect on the ultrafine grain fraction is similar to the strain dependence of high-angle boundary fraction. An abrupt increase in the ultrafine grain fraction from 0.001 to 0.42 occurs with increase in the strain from 1.2 to 2. This rapid increase in the area fraction of ultrafine grains can be explained by the formation of numerous low-angle strain-induced subboundaries at an early stage of deformation, the misorientations of which progressively increase with straining, leading to homogeneous development of many ultrafine grains throughout the sample. Further MDF to a strain of 4 is accompanied by a gradual increase in the ultrafine grain fraction to 0.59.

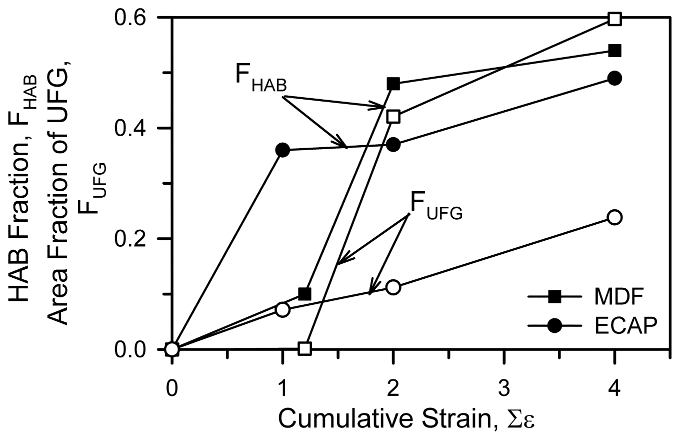

Figure 5. The strain dependencies of the high – angle boundary fraction (F_{HAB}) and the ultrafine grain fraction (F_{UFG}) in a Cu–Cr–Zr alloy subjected to large plastic deformations by MDF and ECAP.

4. Summary

The multidirectional forging and the equal channel angular pressing to a strain of 4 lead to the development of ultrafine grained structure in a Cu-0.3%Cr-0.5%Zr alloy with an average grain size of 0.6 μm and 1.1 μm, respectively. The multidirectional forging is characterized by the faster kinetics of grain refinement as compared to equal channel angular pressing. The fractions of ultrafine grains with a size below 2 μm comprises 0.59 and 0.23, respectively, in the samples after multidirectional forging and equal channel angular pressing to a strain of 4. The change in the deformation axis during multidirectional forging from pass to pass promotes the formation of uniform network of strain-induced subboundaries and, hence, results in the homogeneous microstructure evolution. In contrast, the deformation localization leading to microband development results in the heterogeneous microstructure. In this case, the new ultrafine grains readily appear in the deformation microbands, whereas coarse remnants of initial grains are hard to refine.

5. Acknowledgements

The financial support received from the Ministry of Science and Education, Russia under grant No. 14.513.11.0106 is gratefully acknowledged. The authors are grateful to the personnel of the Joint Research Centre, Belgorod State University, for their assistance with instrumental analysis.

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