Ultrafine Grain Evolution in a Cu–Cr–Zr Alloy during Warm Multidirectional Forging

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Keywords: Cu-Cr-Zr alloy; dynamic recrystallization; submicrocrystalline structure.

Abstract. The microstructure evolution and the deformation behavior of a Cu-0.3%Cr-0.5%Zr alloy subjected to multidirectional forging at a temperature of 673 K under a strain rate of about 10^{-3} s^{-1} were studied. Following a rapid increase in the flow stress during straining to about 1, the strain hardening gradually decreases, leading to a steady-state flow behavior at total strain above 2. The multidirectional forging led to the development of ultrafine grained microstructures with mean grain sizes of 0.9 μm and 0.64 μm in the solution treated and aged samples, respectively. The presence of second phase precipitates promoted the grain refinement. After processing to a total strain of 4, the fractions of ultrafine grains (D < 2 μm) comprised 0.36 and 0.59 in the solution treated and aged samples, respectively.

Introduction

The grain refinement can significantly enhance the mechanical properties of metallic materials according to Hall – Petch relationship [1-3]. One of the promising methods for grain refinement and strengthening of various metallic materials including copper and its alloys is severe plastic deformation (SPD) [4-7]. Recently, several techniques, such as equal channel angular pressing, high pressure torsion, accumulative roll bonding and multidirectional forging, have been proposed to provide large plastic strain [8]. Multidirectional forging (MDF) is a simple technique to obtain large plastic deformation, which does not require any specific equipment [9]. An application of multidirectional forging as method of SPD makes it possible to analyze the deformation behavior of materials during processing. The formation of new grains in metallic materials during large plastic deformation is commonly discussed in terms of dynamic recrystallization (DRX) [9, 10]. Since the DRX grain size depends on the deformation conditions, a significant grain refinement during severe plastic deformation is possible by decreasing the deformation temperature [11-13]. The grain refinement at low to moderate deformation temperatures results from the gradual evolution of strain-induced subgrains [9, 11]. Progressive increase in the misorientations between the strain-induced subgrains during plastic deformation leads to the transformation of the subgrains to the new fine grains. However, regularities of DRX during cold-to-warm working have not been studied in sufficient details. The aim of present work is to clarify the effects of dispersed precipitates on the deformation behavior and the microstructure evolution in a Cu-Cr-Zr alloy during multidirectional forging at 673 K.

Experimental Procedure

A Cu–0.3wt.%Cr–0.5wt.%Zr alloy was used as the starting material. The specimens were subjected to a solution treatment (ST) at 1193K for 30 min with subsequent water quenching. Several specimens after solution treatment were aged (ST+AT) at 723 K for 1 h. The multidirectional forging was carried out at 673 K with a strain rate of about 10^{-3} s^{-1}. Samples for multidirectional forging were machined with starting dimension of 25 mm × 20 mm × 16 mm. The loading axis during multidirectional forging was changed through 90° from pass to pass. The true strain applied at each pass was 0.4. The deformed samples were quenched in water and then reheated to 673 K within 20 min in each compression pass. The multidirectional forging temperature was automatically controlled with an accuracy of ±2°. Microstructural observations were performed on the sections parallel to the forging axis in the final pass, using a Quanta 600 FEG scanning electron microscope (SEM) equipped with an electron back scattering diffraction (EBSD) analyzer incorporating an orientation imaging microscopy (OIM) system. The SEM specimens were mechanically polished on 1000 grit SiC paper and then electropolished 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 1 μm for specimens after multidirectional forging to a total strain of 0.4 and 60 nm for specimens after multidirectional forging to strains of 2 and 4. The obtained EBSD maps were subjected to clean-up procedure setting a minimal confidence index of 0.1. The mean grain size was measured on OIM image as an average distance between high-angle boundaries with misorientations of $\theta \geq 15°$.

Results and Discussion

Deformation behavior. Figure 1 shows a series of the stress (σ) vs cumulative strain (Σε) curves for the Cu–Cr–Zr alloy after solution treatment (ST) and solution followed by aging treatment (ST+AT). The shape of σ–Σε curves is quite identical in spite of the difference in the initial state. The flow stress increases more than twofold after the first pass. Then, the strain hardening gradually decreases to almost zero, resulting in steady-state deformation behavior at cumulative strains above 2. Therefore, the envelope flow curves are similar to those associated with dynamic recovery. There is no visible difference between unloading and reloading stresses for all deformation passes. Therefore, static recrystallization does not affect the microstructure evolution in the Cu–Cr–Zr alloy during multidirectional forging (MDF).

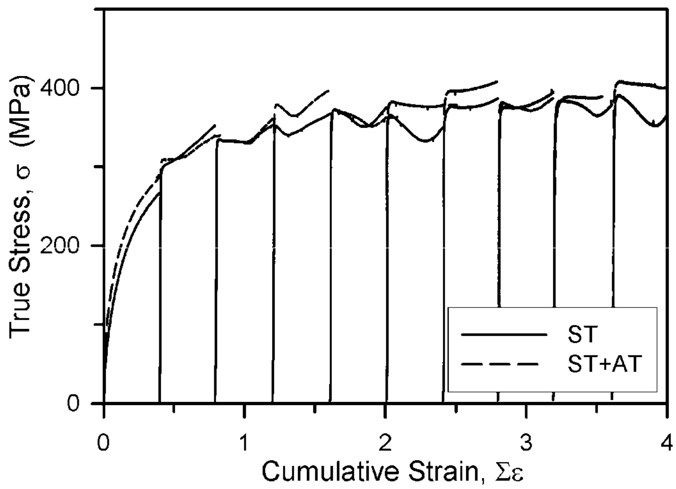

Fig. 1. True stress (σ) vs cumulative strain (Σε) curves for the Cu – Cr – Zr alloy after solution treatment (ST) and solution followed by aging treatment (ST+AT). The multidirectional forging was carried out at 673K.

Microstructure evolution. Typical deformation microstructures developed in the ST and ST+AT alloys during multidirectional forging at 673 K are shown in Fig. 2. Commonly, the multidirectional forging to a strain of about 0.4 leads to the appearance of a large number of the strain-induced subboundaries with low-angle misorientations (θ < 15°), which appear as deformation microbands (DMBs) and/or dense dislocation walls (DDWs) crossing over the original grains (Fig. 2). An increase of the total strain to about 2 leads to the formation of new ultrafine grains. The new grains are characterized by nearly equiaxed shape, with a mean grain size below 1 μm. The formation of the new ultrafine grains occurs nearby the initial grain boundaries as well as along several DMBs/DDWs. The change in the forging direction from pass to pass assists the development of frequently crossed DMBs/DDWs, and therefore, promotes the rapid evolution of the new ultrafine grains at the DMBs/DDWs intersections [11-13].

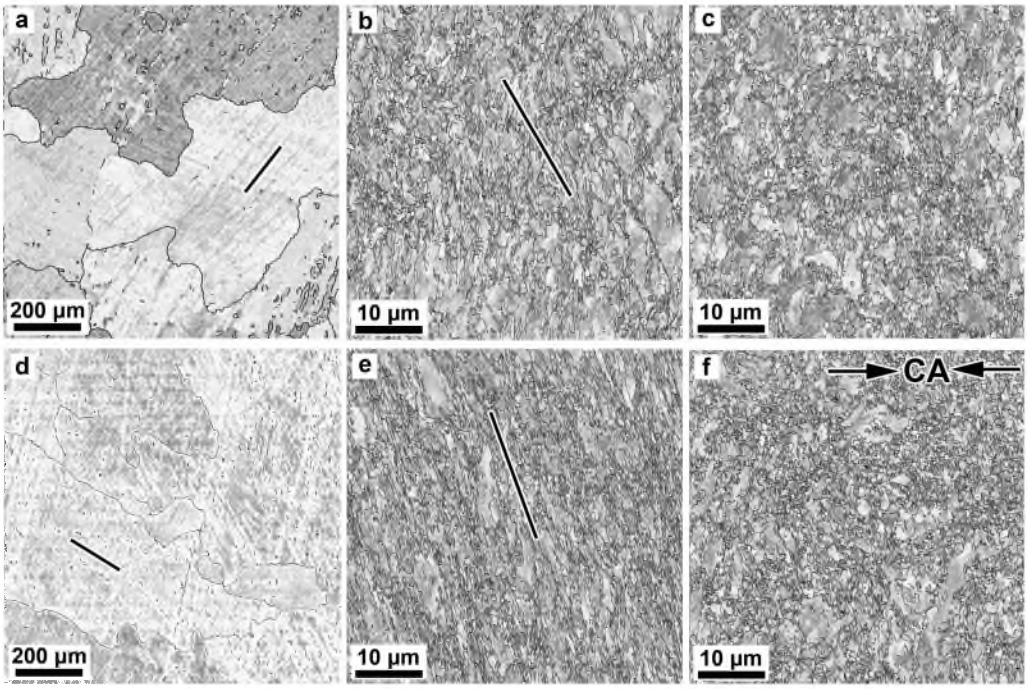

Fig. 2. Typical microstructures developed in the Cu-Cr-Zr alloys during MDF at 673 K to total strains of ε~0.4 (a, d), ε~2 (b, e), and ε~4 (c, f). The deformation microstructures in the initially solution treated (ST) samples are shown in (a, b, c); and those in the solution and aging treated (ST+AT) samples are shown in (d, e, f). The white and black lines indicate the low- and high-angle boundaries, respectively. The thick black stretches in a, b, d, e indicate the lines, along which the misorientation profiles were obtained (s. Fig. 5).

The initial state of the Cu-Cr-Zr alloy has a significant influence on the grain refinement during the warm MDF. After the MDF to a total strain of ε ~ 4, the ST+AT sample consists of almost uniform ultrafine grained structure, whereas the microstructure of the ST sample is rather heterogeneous (Fig. 2). In addition to the ultrafine equiaxed grains, the final microstructure of the ST sample contains relatively large irregular grains with an average size of ~5μm, which evidently represent the reminders of original grains. Such grains include the great number strain-induced boundaries with low-angle misorientations. The corresponding grain size distributions are characterized by sharp peaks for the grains with sizes less than 2 μm (Fig. 3), which can be considered as DRX grains. The fraction of these DRX grains comprises 0.59 in the finally developed microstructure of ST+AT sample, while that of 0.36 was obtained in the ST sample.

Kinetics of dynamic recrystallization. The kinetics of dynamic recrystallization in Cu–Cr–Zr alloy during the MDF at 673 K are illustrated by Fig. 4, which shows the strain effect on the fraction of high-angel boundaries (HABs) and the recrystallized fraction. The latter was evaluated as the area fraction of ultrafine grains with a size below 2 μm (s. Fig. 3). In the ST sample, the fraction of HAB almost linearly increases to 0.4 with straining to 2. It can be associated with progressive formation of DMBs. Then, the HAB fraction gradually approaches 0.45 at a large strain of 4. On the other hand, the rapid increase in the HAB fraction takes place in ST+AT sample in the strain range of $1.2 < \varepsilon < 2$, where the HAB fraction increases to above 0.5. Correspondingly, the fast development of the DRX grains takes place in the same strain range, leading the DRX fraction to 0.36 and 0.59 in the ST and ST+AT samples, respectively (Fig. 4).

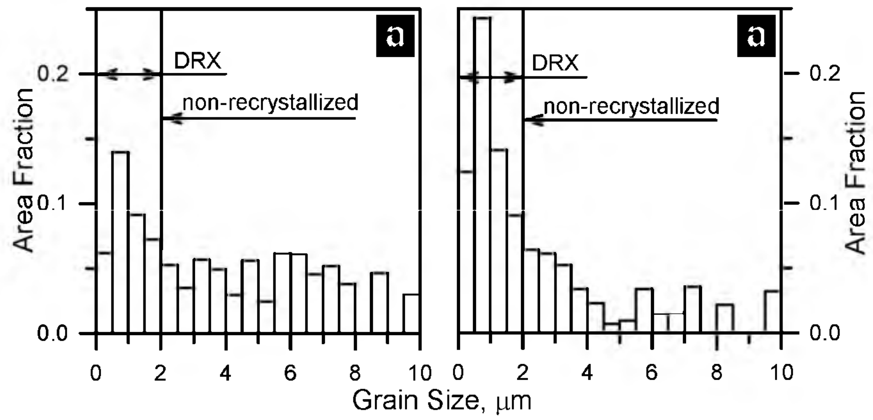

Fig. 3. The grain size distributions in the ST (a) and ST+AT (b) samples of the Cu-Cr-Zr alloy subjected to MDF to a total strain of ε ~ 4.

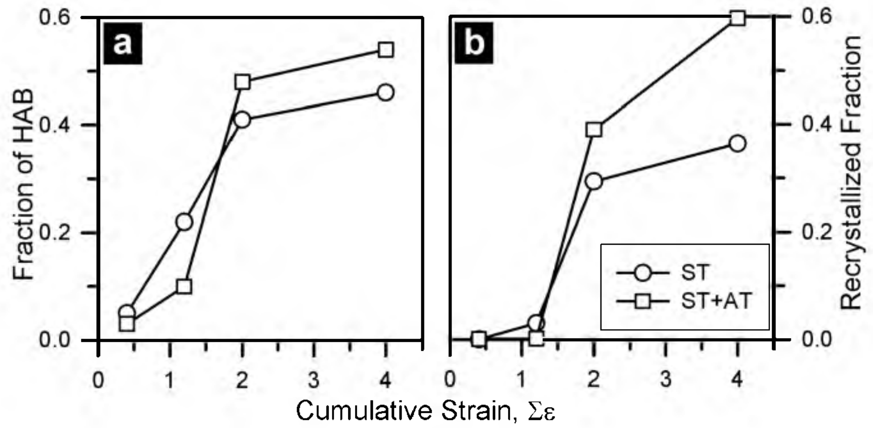

Fig. 4. The strain effects on (a) the fraction of high-angle boundaries (HAB) and (b) the recrystallized fraction in the ST and ST+AT alloys subjected to MDF at 673 K.

Figure 5 shows the misorientation profiles in the ST and ST+AT Cu–Cr–Zr alloys subjected to MDF to total strains of 0.4 and 2. These profiles were obtained along the lines indicated in Fig. 2. The multidirectional forging to a total strain of 0.4 brings about the evolution of DMBs and DDWs inside the original grains. At a strain of 0.4 the density of the DMBs/DDWs in the ST+AT sample is higher than in the ST sample. However, the misorientations of the DMBs/DDWs developed in the ST samples are larger than in the ST+AT ones. This may lead to fast appearance of HABs and new ultrafine grains in the ST samples upon further MDF. It's clearly seen in Fig. 5 that the density and misorientations of DMBs/DDWs in both the ST and ST+AT samples increase significantly during subsequent straining. After MDF to a relatively large strain of 2 the density of high-angle misorientations in ST+AT samples is higher than in the ST samples. This might result from high density of DDWs evolved in the ST+AT samples at preceding strains. These DMBs and DDWs serve as nucleation sites for ultrafine DRX grains during the MDF [6, 11]. Therefore, the high density of DDWs in the ST+AT samples results in superior kinetic of the DRX development.

The formation of new ultrafine grains strongly depends on the initial state as schematically shown in Fig. 6. The localization of deformation in the ST sample results in the development of sharp DMBs. The variation of slip systems within these DMBs results in local lattice rotations, leading to the appearance of new fine grains along the DMBs at relatively small strains. However, such pronounced deformation heterogeneity retards the development of the new grains and slows down the DRX kinetics in large strains. On the other hand, the uniform distribution of finely dispersed particle in the ST+AT sample promotes more homogeneous deformation, which is characterized by a high density of uniformly distributed DDW. These DDWs accelerate of the formation of new DRX grains with increase in strain above 1.2.

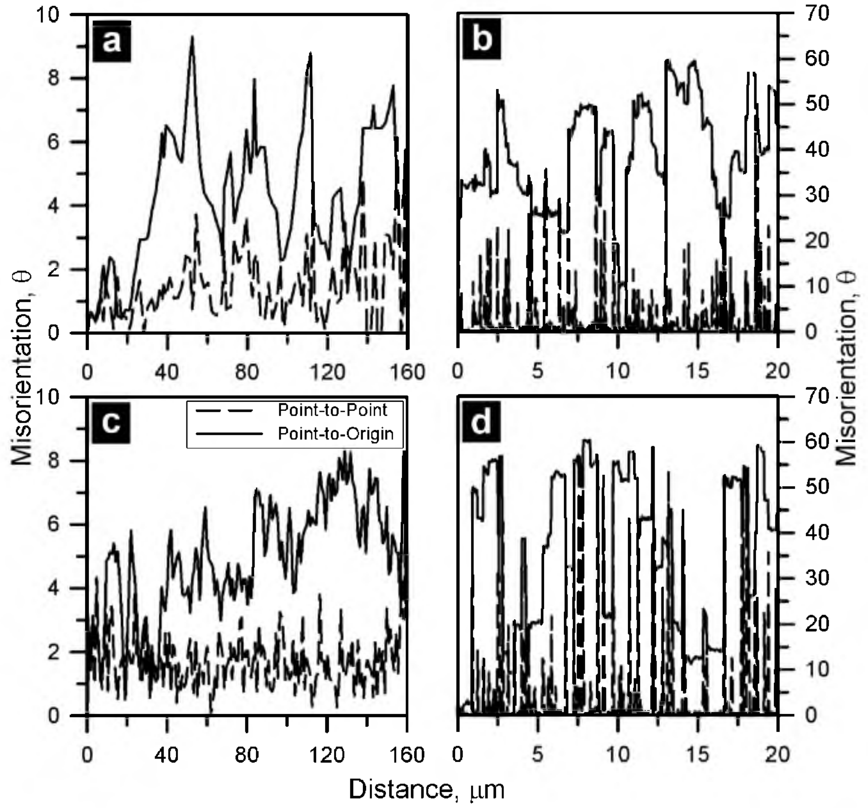

Fig. 5. Misorientation profiles (along the black lines in Fig. 2) in the ST (a, b) and ST+AT (c, d) Cu-Cr-Zr samples subjected to MDF to strains of ε ~ 0.4 (a, c) and ε~2 (b, d).

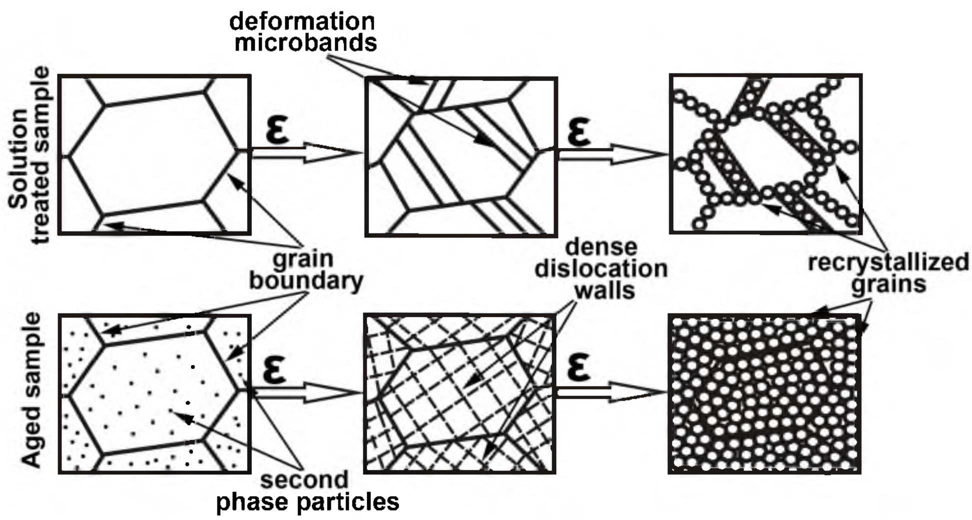

Fig. 6. Schematic illustration of the microstructure evolution in the Cu-Cr-Zr alloy during MDF at 673 K.

Summary

Multidirectional forging at a temperature of 673 K to a total strain of 4 leads to the formation of submicrocrystalline structure with a mean grains sizes of 0.9 μm and 0.64 μm in the ST and ST+AT samples, respectively. The volume fractions of the fine grains with a grain size below 2 μm comprise 0.36 and 0.59 in the samples initially subjected to solution and aging treatments, respectively. The formation of the new fine grains results from the evolution of strain-induced subboundaries appearing in the form of deformation microbands and dense dislocation walls, which gradually transform to the grain boundaries with increase in the strain. The presence of dispersed particles in the age treated samples promotes the development of homogeneous deformation substructures at an early deformation, and therefore, accelerates the fine grain formation at large strains.

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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