Post-dynamic recrystallization of the Cu-0.6Cr alloy

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Abstract. The results of the studying the transformation of structural parameters in the samples of the Cu-0.6Cr alloy during high-temperature free upsetting and subsequent cooling at different rates with the use of different media: liquid nitrogen, water and air are presented. Using EBSD and X-ray diffraction (XRD) analysis, we demonstrate that a decrease in the cooling rate of the samples of the Cu-0.6Cr alloy after a large deformation of ε~2 at a temperature of 800-850 °C promotes the formation of smaller structural fragments, an increase in the dislocation density and a reduction in the fraction of high-angle boundaries.

1. Introduction
The processes of dynamic polygonization and recrystallization during high-temperature deformation processing have a decisive effect on the type of structure being formed. They participate in structural transformations and the formation of properties in different classes of functional materials. There are a number of papers investigating the kinetics of these processes in various materials and under different deformation procedures [1-9]. Analysis of these papers shows that the predominant condition for the nucleation of recrystallized grains is the non-equilibrium state of grain boundaries, and recrystallization occurs via the mechanism of splitting of high-angle boundaries [1,2]. As a result, in the process of high-temperature deformation, the fraction of recrystallized grains increases and, correspondingly, the structure becomes refined. There are publications investigating the effects of deformation temperature and strain rate in a low-alloyed Cu-Cr-Zr alloy for electrical engineering applications on the polygonization and recrystallization processes in the structure. Using EBSD and TEM, it was found that an increase in strain rate leads to an increase in the volume of recrystallized grains [10].

With the development of modern techniques of severe plastic deformation (SPD), which provide active refinement of the structure to an ultrafine range under large strains, an important factor is the study of the effect of deformation and post-deformation processing parameters on the recrystallization processes, structural changes and a set of properties in metals. Therefore, the aim of the present work is to study the effect of large deformation (ε~2) by free upsetting of the samples of the Cu-0.6Cr alloy at a temperature of 800-850 °C and subsequent cooling at different rates on the transformation of the initial structure.

2. Material and experimental procedures
The Cu-0.6%Cr alloy in the form of cylindrical billets with a diameter of 20 mm and a height of 40 mm was subjected to upsetting by 85-90% at a temperature of 800-850 °C. Preliminary, the billets of
chromium bronze had been exposed to a temperature of 1000 °C for 1 hour with subsequent water quenching. The deformation was performed on a hydraulic press with a force of 6 GPa and a strain rate of 4 mm/s. Temperature was controlled using a Testo830-T1 pyrometer. At the beginning of the deformation, the temperature of the samples was 850±5 °C, and after the deformation was finished, the temperature was not less than 800 °C. As a result of the deformation, round samples with a diameter of 56 mm and a height of 5 mm were produced. In order to study the effect of the cooling rate of the samples on the changes in structural parameters, cooling was performed in three media: in liquid nitrogen, in water and in air.

Structural studies of the cross-section of the samples were performed using EBSD and XRD analysis. Scanning electron microscopy with electron backscatter diffraction (EBSD) was performed employing a TESCAN MIRA 3 LMH field emission microscope equipped with an Oxford Instruments HKL Channel 5 system. The scanning steps were 0.1 and 0.4 μm. Grain misorientation maps were constructed, grain and subgrain size were estimated, a quantitative analysis of the low- and high-angle boundaries was conducted. Diffraction patterns were taken with a Rigaku Ultima IV diffractometer in the Bragg-Brentano geometry using CuKα radiation generated at a voltage of 40 kV and a current of 40 mA. X-ray patterns were obtained within a range from 35° to 145° in the continuous scanning regime with a rate of 0.25 °/sec.

3. Results
As a result of the upsetping at a high temperature and subsequent quenching in different media, 3 states with different types of structures were obtained. To characterize the structural state and determine the grain misorientations, we performed an EBSD analysis with identification of the size of grains and subgrains. It can be seen from the bar charts of grain misorientation distribution (figure 1) that after quenching in nitrogen, grains with misorientation angles above 15° prevail.
Figure 1. Maps and bar charts of grain misorientation distribution: (a) upsetting 800 °C + cooling in liquid nitrogen, (b) upsetting 800 °C + cooling in water, (c) upsetting 800°C + cooling in air.

A high fraction of boundaries, over 20%, refers to misorientation angles of 58-60°, which correspond to twin boundaries. The fraction of twin boundaries decreases with decreasing cooling rate, as shown by the results of the study of the water-quenched condition. At the same time, the fraction of medium-angle boundaries increases and the fraction of high-angle boundaries decreases with decreasing cooling rate (figure 2).

Figure 2. Variation in the fraction of low-, medium- and high-angle boundaries.

The data on the size of grains and subgrains forming in the process of hot deformation and subsequent cooling are presented in table 1.

Table 1. Average sizes of grains and subgrains in the Cu-0.6Cr alloy.

| Condition | Average subgrain size, µm | Average grain size, µm |
|-----------|---------------------------|-----------------------|
| Upsetting 800 °C quenching in nitrogen | 5.0±0.3 | 7.5±0.4 |
| Upsetting 800 °C quenching in water | 3.1±0.3 | 6.5±0.4 |
| Upsetting 800 °C quenching in air | 1.1±0.2 | 1.7±0.2 |

The XRD results indicate that with an increase in the cooling time (cooling in air), the lattice parameter decreases (table 2), which is related to the process of decomposition of the supersaturated solid solution. The lattice parameter after quenching in nitrogen is 3.618(4) Å, and after cooling in air the lattice parameter is restored to a value of 3.616 (4) Å. The character of solid solution
decomposition affects the dislocation density, since dispersed particles are effective stoppers for dislocation propagation.

### Table 2. XRD analysis data for the Cu-0.6Cr alloy.

| Process Description                      | a, Å    | CRS, nm | ε, %   | Dislocation density, m⁻² |
|-----------------------------------------|---------|---------|--------|--------------------------|
| Upsetting 800 °C quenching in nitrogen   | 3.618(4)| 142±7   | 0.020  | 1.9*10¹³                  |
| Upsetting 800 °C quenching in water      | 3.617(6)| 155±7   | 0.023  | 2.1*10¹³                  |
| Upsetting 800 °C quenching in air        | 3.616(4)| 83±6    | 0.042  | 6.8*10¹³                  |

### 4. Conclusions

1. Large deformation (ε~2) by free upsetting of the samples of the Cu-0.6Cr alloy at a temperature of 800-850 °C and subsequent cooling in different media leads to the formation of the following structural parameters:

   - During cooling in liquid nitrogen, a grain type of structure is formed with an average grain size of 7.5±0.4μm, a low dislocation density (1.9*10¹³ m⁻²) and a high fraction of growth twins. This condition corresponds to the stage of finished dynamic recrystallization, the fraction of high-angle boundaries is over 60%.

   - During cooling in water, the fractions of medium- and high-angle boundaries are practically the same – 36% and 38%. The fraction of twin boundaries decreases. However, the dislocation density is close to the level of dislocation density after cooling in liquid nitrogen ~2.1*10¹³ m⁻². This condition corresponds to the stage of unfinished dynamic recrystallization. The average grain size is 6.5±0.4 μm.

   - Cooling in air leads to the formation of a predominantly subgrain type of structure. In this case, the process of post-dynamic recrystallization occurs. The average size of the grain-subgrain structure is 1.7±0.2 μm, the fraction of high-angle boundaries is not more than 23%. Active decomposition of the supersaturated solid solution takes place. The dispersed particles of the second phases, formed as a result of the exposure to high temperature, are efficient barriers to the dislocation propagation in the grain interiors. As a consequence, the dislocation density grows to 6.8*10¹³ m⁻².

2. A decrease in the cooling rate of the Cu-0.6Cr alloy samples after large deformation at a temperature of 800-850 °C promotes the formation of smaller structural fragments, an increase in the dislocation density, and a reduction in the fraction of high-angle boundaries.

### Acknowledgments

The study was supported by the Russian Science Foundation (project №19-19-00432).

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