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Microstructure and mechanical properties of ultrafine grained copper processes by multiple isothermal forging

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Микроструктура и механические свойства ультрамелкозернистой меди, полученной всесторонней изотермической ковкой

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Microstructure and mechanical properties of ultrafine grained (UFG) Cu processed by multiple isothermal forging (MIF) are studied and compared to those after processing by equal channel angular pressing (ECAP). It is shown that when the final temperature of MIF and the temperature of ECAP are the same, similar microstructures and mechanical properties are obtained in this material. In general, MIF allows for processing UFG materials with grain sizes slightly exceeding those obtainable by ECAP, but in samples having significantly larger dimensions.

Keywords: ultrafine grained material, multiple isothermal forging, equal channel angular pressing, microstructure, mechanical properties

Изучены микроструктура и механические свойств ультрамелкозернистой (УМЗ) меди, полученной всесторонней изотермической ковкой (ВИК), проведено их сопоставление с характеристиками после деформации раноканальным угловым прессованием (РКУП). Показано, что, когда конечная температура ВИК и температура РКУП совпадают, получаются примерно одинаковые микроструктуры и свойства. В целом, ВИК позволяет получать УМЗ материалы с размерами зерен, несколько превышающими размеры, достижимые при РКУП, но в образцах значительно больших размеров.

Ключевые слова: ультрамелкозернистый материал, всесторонняя изотермическая ковка, равноканальное угловое прессование, микроструктура, механические свойства

1. Introduction

In the latest two decades, a great interest has been given to the development of severe plastic deformation (SPD) methods to obtain bulk ultrafine grained (UFG) and nanostructured (NS) materials [1-18]. These methods are based on a grain refinement in bulk samples when applying extremely large strains without changing their geometry. Among a variety of SPD methods developed up to date, a particular attention is given to equal-channel angular pressing (ECAP) [1,4,7]. Although it is claimed that this method is capable to produce bulk UFG materials, normally its application is restricted to rod-type samples with the diameter up to 40 mm. A promising method to produce bulk UFG materials is multiple (or multi-step) isothermal forging (MIF) [8-16]. This method is based on the use of dynamic recrystallization during hot and warm deformation of metals. Deformation is done by repeating cycles of forging operations, each of which consists of a sequence of upsetting a sample along the three orthogonal axes followed by drawing. MIF with a stepwise reduction of temperature allows one to refine the grain size down to the submicrometer and nanometers range avoiding fracture. The studies have shown that MIF is a universal method allowing one to obtain bulky nanostructured semi-products out of various metals and alloys including hard-to-deform alloys and intermetallics.

The recent studies have shown that nanostructuring by ECAP and MIF are competitive methods in terms of the improvement of mechanical properties of materials. Both methods result in a significant increase of the strength of materials.

The first attempt of a comparative study of the structure and properties of UFG metals processed by different SPD methods has been undertaken in Ref. [19], in which the authors studied the microstructure, strength and plasticity of oxygen-free copper prepared by ECAP, MIF, twist extrusion and accumulative roll bonding. However, these studies are not enough for a rationalized preference for any of these methods in terms of an increase of a certain set of properties in samples of certain geometries.

In our recent paper [20] we studied the microstructure and mechanical properties on copper M1 subjected to ECAP by route B_C in three regimes: at room temperature, at 200°C and combined regimes consisting of deformation at 200°C for the first four passes followed by deformation at room temperature for the last four passes. The latter regime is some analogue of MIF regarding to the temperature decrement between deformation cycles and it was found to produce slightly smaller grain size and higher strength.

The aim of the present paper is to explore the microstructure and mechanical properties of the same copper subjected to MIF and compare the results to the characteristics of UFG Cu processed by ECAP.

2. Material and methods

For the studies copper of 99.9% purity classified as M1 in Russian standards was used. For the studies of microstructure evolution during forging at temperatures 100, 150, 175 and 200°C model cubic samples with 10 mm edge length were used. These samples were deformed at a specified temperature by 3 and 4 cycles of subsequent upsetting along the all three axes. Strain during each upsetting was about 40%. The foils for microstructure studies were prepared on the three orthogonal planes of the samples from their central parts. Microstructures were studied by TEM using the microscope JEM 2000 EX.

The large-scale processing was done on a cylinder-shaped sample with the diameter about 70 mm and length about 100 mm according to the whole MIF-procedure including the upsetting along the three axes and drawing operations to restore the original sample shape after each cycle [8-16]. The deformation started at T=400°C and finished at T≈180°C when cracks initiated on the sample surface. The final shape of the sample was nearly cylindrical with diameter about 80 mm and height about 75 mm. An 11 mm thick plate normal to the cylinder axis was cut from this sample near its mid. The microstructure formed after the MIF treatment was studied in the center of this plate and on a distance of 25 mm from the center by TEM using the same microscope.

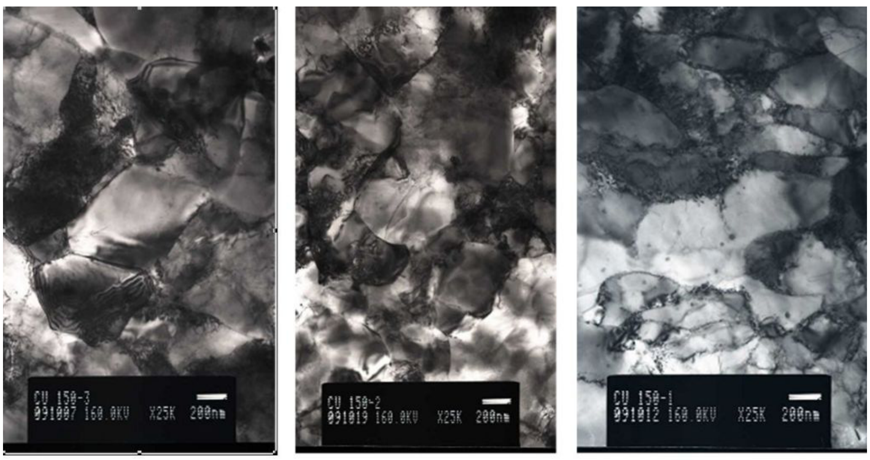
Mechanical properties were studied by tension at room temperature. Dogbone shaped samples with the gauge length 18 mm and thickness 2 mm were used for these studies.

3. Experimental results

Microstructure of model samples

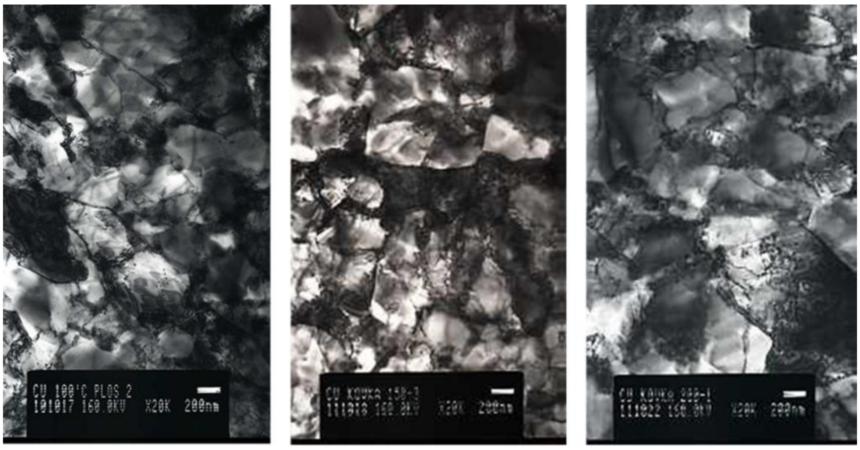
TEM studies have shown that the 3-cycles processing at each of the temperatures studied results in a formation of an equiaxed microstructure with the grain/subgrain size in the submicrometer range. Presented in Fig. 1 are the representative microstructures on three orthogonal planes of the sample deformed at 100°C.

Further straining does not change significantly the visible character of microstructure and the grain/subgrain size (Fig. 2). However, a comparison of the selected area diffraction patterns shows that they evolve from slightly



a) b) c)

Fig. 1. Representative microstructures observed by TEM on three orthogonal planes of the model samples deformed to 3 cycles of multiple forging at temperature 150°C.



a) b) c)

Fig. 2. Representative microstructures of the samples deformed to 4 cycles of multiple forging at temperatures 100°C (a), 150°C (b) and 200°C (c).

spread spots to ring patterns with largely scattered behavior with an increase of the strain (Fig. 3). This indicates that the amount of high-angle grain boundaries increases.

The grain/subgrain sizes measured after 3 and 4 cycles of MIF were approximately the same for each of the temperatures studied and constituted 390±40, 380±35, 435±45 and 410±40 nm for the temperatures 100, 150, 175 and 200°C, respectively. Considering not very large number of grains counted in the grain size determination by TEM, and, therefore, fairly large errors, one can say that the sizes of grains/subgrains at temperatures 100-200°C are approximately the same and amount about 400 nm.

a) b)

Fig. 3. Representative selected area electron diffraction patterns of the microstructures formed after 3 (a) and 4 (b) cycles of multiple forging at temperature 100°C.

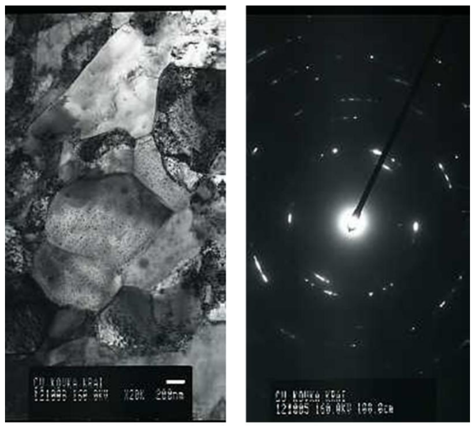
Microstructure and mechanical properties of the large-scale sample

Figs. 4 and 5 represent typical microstructures observed in the central and periphery parts of the large-scale sample processed by MIF, along with corresponding selected angle electron diffraction patterns.

As one can see, an equiaxed submicron-grained structure is formed in both regions of the sample studied. The average grain size is about 440±35 nm in the center and 580±35 nm in the periphery. Thus, the grain size obtained in the central part is approximately the same as in the centers of model samples, while in the periphery the grain size is higher. Nevertherless, it differs not very much so that one can consider that during MIF a relatively uniform microstructure with the grain size near 0.5 μm is formed.

a) b)

Fig. 4. Typical microstructure (a) and corresponding SAED pattern observed in the central part of large-scale sample of Cu subjected to MIF.



a) b)

Fig. 5. Typical microstructure (a) and corresponding SAED pattern observed in the periphery part of large-scale sample of Cu subjected to MIF.

The stress-strain curves obtained by tensile tests of three UFG samples are presented in Fig. 6. The average values of the mechanical characteristics obtained from these tests are the following: yield stress $\sigma_{0.2} \approx (290\pm2)$ MPa, ultimate tensile strength $\sigma_u \approx (335\pm2)$ MPa, total elongation $\delta \approx (20\pm3)$% and relative reduction $\psi \approx (93\pm1)$%.

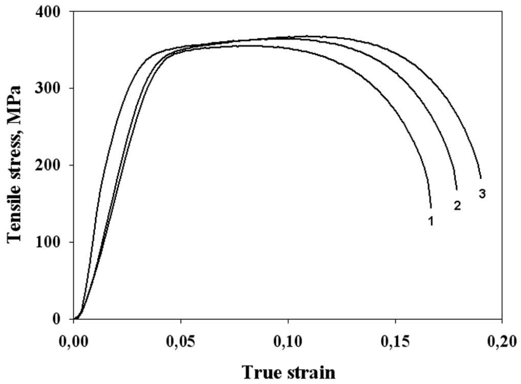


Fig. 6. Stress-strain curves of UFG Cu subjected to MIF processing (numbers on the curves correspond to the numbers of samples tested).

4. Discussion

Thus, the studies have shown that using MIF it is possible to process UFG copper with the grain size about 500 nm.

Although the studies show a slight difference between the size of ultrafine grains in the central and peripheric parts of the large-scale sample, one can say that MIF processing results in a relatively uniform microstructure.

In recent work [20] we have reported that ECAP processing at 200°C results in a somehow bimodal microstructure with the grain sizes around 400 nm and 700 nm the latter being constituted of recrystallized grains so that the average grain size is about 500 nm. Since the straining in MIF process stopped near 200 °C, the same grain size was obtained as by ECAP at this temperature.

The UFG copper processed by ECAP at 200°C had the following mechanical properties: $\sigma_y \approx (326\pm4)$ MPa, $\sigma_u \approx (348\pm2)$ MPa, $\delta \approx (24.4\pm1.8)$% [20]. As one can see, the mechanical characteristics obtained by ECAP at the temperature 200°C and by MIF with a similar final temperature are similar. That is, down to 200°C the two methods have nearly equal potential in the grain refinement and enhancement of mechanical properties. Down to this temperature, however, MIF has an advantage, since it allows for the processing large samples which have no principal size limitations. On the other hand, ECAP allows for achieving a smaller grain size due to the possibility of imparting large strains at lower temperatures. Deformation by MIF at such low temperatures require additional operations between the cycles like a removal of the outer layers when cracks appear.

5. Conclusions

The present study shows that multiple isothermal forging and equal channel angular pressing have similar potential in the grain refinement and the enhancement of mechanical properties of Cu if processing is carried out at similar temperatures. Due to the fact that deformation during ECAP is done in constraint conditions, its allows for straining the samples down to the room temperature and obtaining slightly smaller grain sizes than by MIF processing, which finished at elevated temperature of about 180°C. On the other hand, MIF allows for obtaining slightly larger but comparable grain sizes in much larger-scale samples. This proves that MIF processing has a high application potential.

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