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Activation parameters and deformation mechanisms of ultrafine-grained copper under tension at moderate temperatures

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ABSTRACT

Mechanical properties, characteristic features of deformation behavior, the activation energy for plastic deformation, strain rate sensitivity and activation volume of ultrafine-grained (UFG) and conventional coarse-grained (CG) copper have been studied by tension in the temperature interval of 293–573 K and in the strain rate interval of 1.3×10^{-2}–3.0×10^{-5} s^{-1}. It is found that both the properties and the activation parameters differ significantly in UFG and CG copper suggesting the different deformation mechanisms. Plastic flow is shown to be controlled by grain boundary diffusion in the case of UFG copper. Considering the thermal activation analysis data and deformation relief appearing on the pre-polished surface of the test samples, the significant contribution of grain boundary sliding to the overall deformation during plastic flow of UFG copper at moderate temperatures is supposed.

1. Introduction

It is well known that the decrease of grain size of a polycrystalline material results in the increase of the yield stress according to the Hall–Petch relation. Therefore great efforts are launched lately to develop ways of highest possible refinement of grain structure in order to obtain maximal strength. The methods of severe plastic deformation are of particular interest since they enable production of bulk ultrafine-grained (UFG, grain size is equal to 100–1000 nm) samples free of residual porosity and contaminations [1]. Refining the grain size through severe plastic deformation increases the ultimate and yield stresses. However, ductility drops significantly, restricting the applications of these UFG materials [2]. To improve their mechanical properties, understanding the deformation mechanisms is required.

The strain rate sensitivity, m, the activation energy for plastic deformation, Q, and the activation volume, ν, are the values which can reveal the acting deformation mechanisms. To measure them, the strain rate jump during single-axis tension or compression tests [3–5] or temperature jump during creep tests [6] is generally carried out. Nanoindentation and stress relaxation experiments are also used to obtain the activation volume [7]. However, it is well-known that UFG stricture produced by severe plastic deformation exhibits low thermal stability [1,8]. So unwanted structural changes may take place during sometimes long-continued tests at even moderate temperatures. They make difficult reliable measurement of the above values. To reduce the effect of structural changes during deformation on the measurement of m, Q and ν, one suggests using the yield stress σ_y obtained by single-axis tensile tests at different temperatures and strain rates. Since σ_y is measured just after the onset of the test, it is minimally effected by the change of UFG structure.

Thus in the present work, the deformation behavior of UFG material has been investigated, the activation energy for plastic deformation and the activation volume have been measured in the interval of moderate temperatures to judge on the main deformation mechanisms involved in the plastic deformation. Coarse grained recrystallized counterpart has been studied also for comparison.

2. Experimental

The material used in this study was copper (99.7 wt%). UFG structure was formed by equal-channel angular pressing (ECAP) at room temperature. The ECAP treatment was carried out in the Institute of Physics of Advanced Materials (Prof. Valiev, Ufa, Russia). Cylindrical rods 100 mm in length and 16 mm in diameter were pressed for 16 times trough the round channels crossing at

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the angle of 90° following the B_c route [1,8]. To obtain coarse-grained recrystallized counterpart, the UFG samples were annealed in vacuum ($P=10^{-2}$ Pa) at 723 K for 7200 s.

Flat dog-bone tensile samples with the gauge size of $10\times3\times0.5$ mm^3 were electro-discharge machined in the longitudinal section of the ECAP-processed billet. Tensile tests were carried out using a constant strain rate test machine in the temperature interval of 293–573 K and in the strain rate interval of 1.3×10^{-2}–3.0×10^{-5} s^{-1}. The heating rate for the tests was about 20 K/min. The yield stress σ_y was taken to be the stress corresponding to plastic strain of 0.2% and the ultimate stress σ_u met the maximum on the stress–strain curves.

The microstructure of UFG copper prior to the tests was studied using transmission electron microscopy (TEM) on a JEM 2100 microscope operated at 200 kV and EBSD. Thin foils were prepared by jet electropolishing in an electrolyte of the following composition: CH_3COOH:CrO_3:H_2O=68:12:20 at 293 K at a voltage of 25 V. Observations were made in both bright and dark field imaging modes. The size of the element of grain/subgrain structure was measured in a dark field mode as a maximal distance between boundaries in a highlighted area.

Samples for EBSD studies were mechanically polished and then electropolished using the above mentioned electrolyte at room temperature. The EBSD utilized a Quanta 600 FEG scanning electron microscope operated at 30 kV. The dimension of the scanning areas was 15×40 μm^2. A hexagonal scanning grid with the step size of 50 nm was applied. In order to check structure evolution during the tension, the structure of the samples tested at 373 and 573 K at strain rates of 3.0×10^{-5} and 3.3×10^{-3} s^{-1}, respectively, was studied also. Because the grain size rose during the tests, 25×25 and 50×50 μm^2 scan dimensions with 200 and 250 nm step sizes, respectively, were selected. Kikuchi pattern for each point of the scan formed by backscattered electrons was captured and then analyzed and indexed by the system software (TSL OIM data collection). The accuracy of the misorientation determination is ≈0.6° in the case of field emission electron gun [9]. The data obtained were processed using TSL OIM analysis software. The EBSD study involved standard clean-up procedures, as follows: grain dilatation with a grain tolerance angle of 5°; a minimum grain size of two pixels. It was assumed that the grain/subgrain was a region containing more than one scan point inside which the disorientation angle between the neighboring points did not exceed the tolerance angle. The tolerance angle was fixed at 15° and 2° for grains and subgrains, respectively. The grain/subgrain size was defined as the diameter of a circle having the same area as the area of grain/subgrain. The all structural parameters obtained by EBSD were averaged out using the following formula:

$$\langle d\rangle=\frac{\sum_{i=1}^{N}S_i\,d_i}{\sum_{i=1}^{N}S_i} \tag{1}$$

where S_i and d_i are the square and the diameter of the i-th grain, respectively. This formula takes into account the area covering by grains of the definite size so it is more significant in the case of UFG materials processed by ECAP having a wide grain size distribution. Boundaries with misorientation less than 2° were out of consideration. Low angle boundaries were regarded to have misorientation in the range of 2–15°. Boundaries with misorientation angle greater than 15° were considered as high angle grain boundaries.

It is well-known that the activation energy for plastic deformation under constant stress can be expressed as [10]

$$Q=R\left[\frac{\partial\ln\dot{\varepsilon}}{\partial(1/T)}\right]_{\sigma} \tag{2}$$

where σ is the stress, $\dot{\varepsilon}$ is the strain rate, R is the gas constant, T is the test temperature. To calculate the activation energy using the experimental data on the yield stress one should differentiate the expression (2) with respect to stress [11]:

$$Q=R\left[\frac{\partial\ln\dot{\varepsilon}}{\partial\ln\sigma_y}\right]_T\left[\frac{\partial\ln\sigma_y}{\partial(1/T)}\right]_{\dot{\varepsilon}}=\frac{R}{m}\left[\frac{\partial\ln\sigma_y}{\partial(1/T)}\right]_{\dot{\varepsilon}} \tag{3}$$

where stress is set at the yield stress, m is the strain rate sensitivity, $m=\partial\ln\sigma_y/\partial\ln\dot{\varepsilon}$.

The activation volume of UFG copper was calculated using the formula [12]:

$$\upsilon=MkT\left[\frac{\partial\ln\dot{\varepsilon}}{\partial\sigma_y}\right] \tag{4}$$

where k is the Boltzmann constant, M is the Taylor factor ($\sqrt{3}<M<3$). We used the value $M=3$ to obtain the upper limit of the activation volume.

3. Results

3.1. Structural features

TEM studies reveal that UFG structure with high density of defects (dislocations and dislocation aggregations) forms in copper as a result of ECAP (Fig. 1). Boundaries of the elements of grain/subgrain structure are fuzzy. Extinction contours are observed inside the elements suggesting the presence of high internal stresses. The shape of the elements is either equiaxed or slightly elongated. Their size obtained from dark field images varies in the interval of 0.3–1.0 μm. In the most cases they misorientated with a low angle relative to each other.

Fig. 2 a shows the fragment of EBSD subgrain map. One can see two types of subgrains, namely, equiaxed or slightly elongated small subgrains less than 1 μm in size and lager ones which are considerably elongated along the shear direction. The area fractions of the small and large subgrains are the same. Subgrain size varies from 0.1 to 3.7 μm, the average subgrain size is 1.3 μm (Fig. 3a). The subgrain size distribution obtained by EBSD is shifted to the larger size relative to the respective distribution of the elements of grain/subgrain structure obtained by TEM. It is related to the fact that dark field TEM analysis reveals fragments having

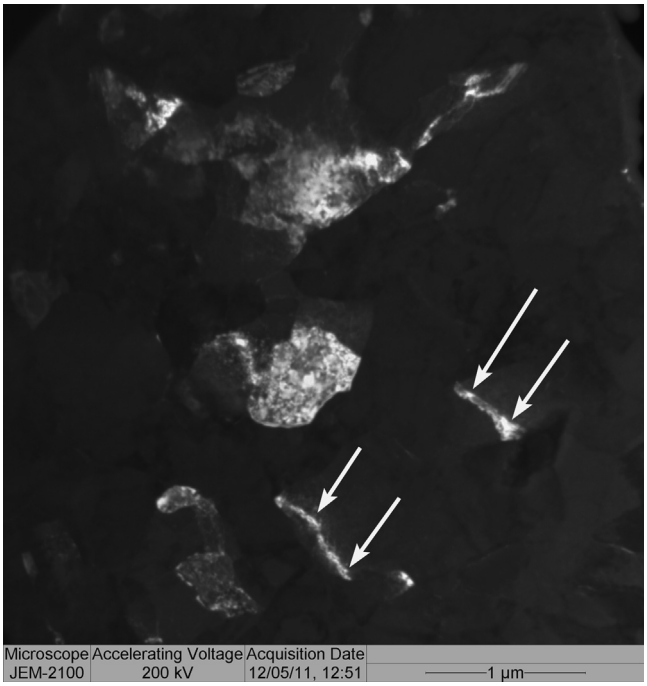

Fig. 1. Microstructure of UFG copper (TEM dark field image). The arrows mark extinction contours.