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Tensile behavior and deformation mechanisms of ultrafine-grained aluminum processed using equal-channel angular pressing

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ABSTRACT

The structure and deformation behavior of ultrafine-grained aluminum processed by equal channel angular pressing through plastic flow at room temperature in a strain rate interval of 1.0×10^{-5}–8.6×10^{-3} s^{-1} have been studied. The contribution of grain boundary sliding to the overall deformation (η) was calculated using displacement of grains relative to each other in local areas of a gage length. The strength characteristics and tendency toward necking diminish when decreasing the strain rate, while elongation up to failure and η increase. Improving the ductility at low strain rates should be related to the increase in strain rate sensitivity caused by enhancing η, reaching 45% in the local areas of the neck and totaling 72% in the local areas of uniformly elongated portion. The relatively low value of the strain rate sensitivity (m=0.08) is due to the heterogeneity of grain structure in UFG aluminum.

1. Introduction

Refining grain structures is a promising way to enhance mechanical properties of materials. The techniques used to produce ultrafine-grained (UFG, $d<1000$ nm) and nanocrystalline ($d<100$ nm) grain sizes have been developed until recently. The methods involving severe plastic deformation are of particular interest because they enable the production of bulk billets free of residual porosity and contamination [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. To improve their mechanical properties, understanding and controlling the deformation mechanisms must be achieved by manipulating structural parameters or variations in the strain conditions.

Intragrain dislocation slip and twinning are the dominant mechanisms of plastic deformation for coarse grained metallic materials at moderate temperatures ($T<0.5T_m$, where T_m is the melting temperature). The role of diffusion-controlled mechanisms, such as grain boundary sliding (GBS) and diffusion creep, is negligible [2]. Concurrently, the direct and indirect data on the essential increase in grain boundary diffusivity in UFG metals are available in the literature. For example, the grain boundary diffusion coefficient in UFG nickel is several times higher than for its coarse grained counterparts [3,4]. The temperature interval of activation of some diffusion-controlled processes in UFG materials shifts to lower temperature due to the increase of grain boundary diffusivity [1,5,6].

The role of dislocation-related deformation mechanisms should be declined in UFG structures due to the difficulties during the generation and movement of the dislocation and to the increase of grain boundary diffusivity. In fact, computer simulation and experimental studies show that the dislocation slip changes to GBS [7], grain rotation [8], diffusion creep [9] and grain boundary migration (grain growth) [10] in nanocrystalline materials. The grain size in UFG materials processed using severe plastic deformation exceeds that in nanocrystalline materials. Nevertheless, GBS is experimentally observed in UFG FCC metals under tension, compression and indentation at room temperature [11–14], corresponding to $0.31T_m$ for aluminum, $0.22T_m$ for copper and $0.17T_m$ for nickel. However, the contribution of GBS to the overall deformation in these works was estimated under fixed strain conditions. Therefore, the effect of the test conditions on the role of the different deformation mechanisms has not been established. Consequently, the aims of the present work are as follows: (i) investigate the peculiarities of the UFG structure using aluminum as an example; (ii) study its deformation behavior at room temperature depending on the strain rate; (iii) establish the effect of the strain rate on the contribution of the GBS to the overall deformation and determine the role of this deformation mechanism under various strain conditions.

2. Experimental

2.1. Structure investigation and mechanical testing

The material used in this study was pure aluminum (99.99%). The UFG structure was formed by equal-channel angular pressing

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(ECAP) at room temperature. The ECAP die channel had a square cross section with 10 mm sides. The internal angle (Φ) between the die channels was 90°, and the angle (Ψ) subtended by the outer arc of curvature at the intersection between the two channel sections was 37°. The billet was pressed 8 times following the B_c route [15].

Flat dog-bone tensile samples with a gauge size of $8\times 2.5\times 1$ mm^3 were electro-discharge machined in the longitudinal horizontal section of the UFG aluminum billet. Because structural parameters may vary in the different parts of the billet [16,17], the structure of each test sample was studied using electron back-scatter diffraction analysis (EBSD) to identify and exclude samples with a badly refined structure. One of the flat surfaces of the test samples was ground to remove approximately 50 μm thickness of the surface layer and mechanically polished with a succession of abrasives concluding with a 1 μm diamond suspension. The final electropolishing was accomplished at 30 V for 10–20 s using a 10% $HClO_4$+90% CH_3OH solution cooled to 243 K.

To measure the local strain of a region in the sample, the surface was marked with a line of circles 50 μm in diameter and ~1 μm deep using a focused ion beam. The distance between the circles was 250 μm. The tensile test resulted in the distortion of the circles and increasing the distance between them, as easily measured by scanning electron microscopy (SEM), giving the nearby strain value. Knowledge of the local strain is important to evaluate the contribution of GBS to the overall deformation correctly because it differs significantly from the total elongation of the test sample due to the very pronounced localization of plastic deformation. The structure of the prepared samples was investigated using transmission electron microscopy (TEM), SEM and EBSD analysis.

The TEM foils were prepared by jet electropolishing in an electrolyte containing 6% $HClO_4$+94% CH_3OH at 243 K and 30 V. A Jeol 2100 microscope operated at 200 kV was used for conventional bright- and dark-field imaging. The size of the elements of the grain–subgrain structure was measured as a maximal distance between the nearest visible boundaries.

A Quanta 200 3D scanning electron microscope with a tungsten hot cathode operating at 30 kV was used for SEM studies and as an orientation probe during the EBSD investigations. The dimension of the EBSD scanned areas was 50×50 μm^2. A hexagonal scanning grid with a step size of 0.3 μm was applied. 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 so-called 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 the grain/subgrain. All of the structural parameters obtained by EBSD were averaged using the following formula:

$$\langle d\rangle = \frac{\sum_{i=1}^{N} S_i d_i}{\sum_{i=1}^{N} S_i} \quad (1)$$

where S_i and d_i are the area and the diameter of the i-th grain, respectively. This formula accounts for the area covered by grains of a definite size, making it more significant for UFG materials processed by ECAP with a wide grain size distribution. It should be noted that the calculation of (1) generally gives a higher average value than that calculated as the simple average. Boundaries with misorientation less than 2° were not considered. Low angle boundaries were regarded to have misorientation from 2° to 15°. Boundaries with a misorientation angle greater 15° were considered high angle grain boundaries.

The tensile tests were carried out using an Instron 3369 testing machine at room temperature. The initial strain rates were 8.6×10^{-3}, 1.0×10^{-4}, 3.1×10^{-5} and 1.0×10^{-5} s^{-1}. The yield stress σ_y was the stress corresponding to the plastic strain (ε=0.2%) and the ultimate stress (σ_u) met the maximum on the stress–strain curves.

The strain rate sensitivity m was calculated using the following formula:

$$m = \partial \ln \sigma / \partial \ln \dot{\varepsilon} \quad (2)$$

where σ is the ultimate or yield stress, and $\dot{\varepsilon}$ is the strain rate.

2.2. Calculation of GBS contribution to the overall deformation

The GBS contribution to the overall deformation (η) was measured in local areas containing visible signs of GBS. Because there were areas exhibiting no GBS-related deformation relief, the GBS contribution to the overall deformation of the whole sample was not estimated. The approach proposed by Langdon [18–20] was used. The occurrence of GBS will lead to the occurrence of offsets in three mutually perpendicular directions at any selected boundary. Fig. 1a shows the scheme of the sample's surface cross-section oriented along the tensile direction. Two orthogonal displacements lie in the plane of the cross-section (u and v) due to the occurrence of GBS between grains 1 and 2 under the action of an applied stress (σ). The third displacement (w) is perpendicular to the section and is not shown. The strain due to GBS can be

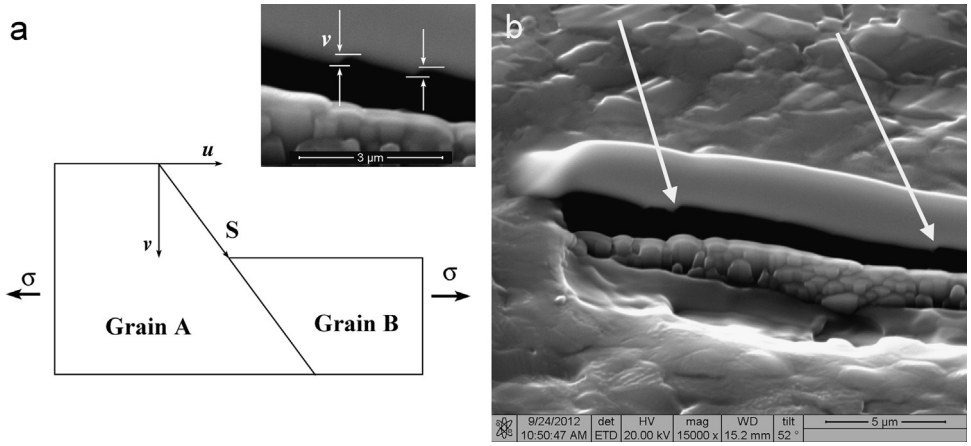

Fig. 1. (a) A two-dimensional scheme of GBS between grains A and B adapted from [20]. u and v denote the offsets of the sliding vector S. (Inset) v was measured using SEM. The tilt of the sample in the SEM is accounted for. (b) The cross-section of the interface "platinum stripe–surface" of the tested sample. The white arrows indicate steps on grain boundaries due to GBS.