The effect of strain rate on tensile behavior and deformation mechanisms of ultrafine-grained aluminum

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Abstract. The effect of strain rate on strength, ductility and uniformity of deformation of ultrafine-grained aluminum processed by equal channel angular pressing through plastic flow at room temperature in a strain rate interval of \(1.0 \times 10^{-5}\) to \(8.6 \times 10^{-3}\) s\(^{-1}\) have been studied. The contribution of grain boundary sliding to the overall deformation (\(\eta\)) 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 \(\eta\) increase. Improving the ductility at low strain rates should be related to the increase in strain rate sensitivity caused by enhancing \(\eta\), 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 probably 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 known to be 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 some orders of magnitude higher than that for its coarse grained counterpart [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 0.31\(T_m\) for aluminum, 0.22\(T_m\) for copper and 0.17 \(T_m\) 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) study deformation behavior at room temperature of an UFG metal depending on the strain rate using aluminum as an example; (ii) 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; (iii) calculate GBS rate using a model and compare it with the experimental data.

2. Experimental

2.1. Mechanical testing

The material used in this study was pure aluminum (99.99%). The UFG structure was formed by equal-channel angular pressing (ECAP) at room temperature. The ECAP die channel had a square cross section with 10 mm sides. The internal angle ($\Phi$) between the die channels was 90°, and the angle ($\Psi$) 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. route [15].

Flat dog-bone tensile samples with a gauge size of 8×2.5×1 mm were electro-discharge machined in the longitudinal horizontal section of the UFG aluminum billet. 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 (Fig. 1). 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. A Quanta 200 3D Dual Beam with a tungsten hot cathode operated at 30 kV was used for SEM studies and FIB processing.

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

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

$$ m = \frac{\partial \lg \sigma}{\partial \lg \dot{\varepsilon}} \quad (1) $$

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

Fig. 1. A FIB-made circles on the sample surface to evaluate strain in a local area.
2.2. Measurement 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 of to the overall deformation of the whole sample was not estimated. The approach proposed by Langdon [16-18] was used. The sliding strain was estimated using the vertical component of the sliding vector v. v was visualized and measured in SEM on cross-sections of the surface prepared using FIB (Fig. 2). First, a platinum stripe 40×1.5×1.5 µm in size was deposited on the area of interest by means of the FIB assistant gas injection system to improve the SEM contrast and to prevent surface damage during the mill. Next, the cross-section of the interface platinum stripe - surface was constructed. Because one took a side view, a profile of the surface was clearly visible and measurable (Fig. 2). Three stripes in each studied local area were cross-sectioned. The number of v measurements depended on the relief: generally 8–10 on each cross-section. The sliding strain was calculated: [16-18]

\[ \varepsilon_{gbs} = kn \langle v \rangle, \]

where \( k \approx 1.5 \) is a constant, \( n \) is the number of boundaries with displacement per unit length and \( \langle v \rangle \) is the average value of v. The contribution of GBS to the overall deformation was expressed as \( \eta = \varepsilon_{gbs}/\varepsilon \), where \( \varepsilon \) is a strain in the studied local area.

The relative error of the \( \eta \) measurement was evaluated using the formula

\[ \delta = ((\Delta n/<n>)^2 + (\Delta v/<v>)^2 + (\Delta \varepsilon/\varepsilon)^2)^{1/2}, \]

where \( \Delta n \) is the absolute error of the \( n \) measurement, \( <n> \) is the simple average of \( n \), \( \Delta v \) is the absolute error of the \( v \) measurement and \( \Delta \varepsilon/\varepsilon \) is the relative error of the strain measured in a local area. \( \Delta \varepsilon/\varepsilon \) was 0.1; \( \Delta n \) and \( \Delta v \) were calculated according to standard statistical methods.

The GBS contribution to the overall deformation was calculated according to the model [18] using the following expression:

\[ \varepsilon_{gbs} = \frac{A'D_gGb}{k_BT} \left( \frac{b}{d} \right)^2 \left( \frac{\sigma}{G} \right)^2 \]  

(3)
where $A'$ is a dimensionless constant having an experimental value close to $\sim 10$, $D_b$ is the grain boundary diffusion coefficient, $G$ is the value of the shear modulus at the testing temperature, $b$ is the Burgers vector, $k_B$ is Boltzmann’s constant.

3. Results and discussion

3.1. Deformation behavior

The engineering stress – strain curves obtained for the UFG and coarse-grained aluminum at different strain rates are shown in Fig. 3. Apparently, the high values for the ultimate and yield stresses, the relatively low elongation up to failure, the modest value of the strain hardening, approach of the ultimate stress and the onset of the deformation localization at a low plastic strain are characteristic features of the deformation behavior of UFG aluminum under tension. These values are typical for UFG materials and related to the fact that dislocation density saturates due to the dynamic recovery or the annihilation of dislocations into the grain boundaries [19]. These features are most pronounced when testing the maximal strain rate. As the strain rate decreases, the difference between the ultimate and yield stresses increases, and the strain up to the onset of necking and the total elongation rises.

The distribution of plastic strain in the gage length of the test samples reveals that at $\dot{\varepsilon} = 8.6 \times 10^{-3} \text{ s}^{-1}$, practically the all strain is localized in a neck, while $3/4$ of the gage length remains strainless after the tension test. The decreased strain rate increases the uniform elongation and reduces strain in the neck. A maximal uniform elongation of $\approx 12–25\%$ is observed at $\dot{\varepsilon} = 1.0 \times 10^{-5} \text{ s}^{-1}$. Thus, the decreased strain rate of UFG aluminum decreases the tendency toward necking (Fig. 4).

Dependence of the ultimate and yield stresses on the strain rate in logarithmic coordinates are plotted in Fig. 5. As observed, the dependence is not linear. The strain rate sensitivity ($m$) varies from $0.02–0.04$ at high strain rates, rising to $0.08$ when decreasing the strain rate. The increase in $m$ may be related to the development of GBS and the enhancement of its contribution to the overall deformation. However, the $m$ value remains lower than that corresponding to GBS ($m_{\text{GBS}} = 0.5$).

Enhancing the uniformity of elongation and improving the ductility by decreasing the strain rate were demonstrated previously for UFG copper [20], aluminum [21,22] and aluminum alloys [23-25]. The effect should be related to the increased value of $m$ according to the Hart’s criterion [20]. However, there was no discussion in [20] regarding the physical reasons of the $m$ rising in UFG metals by decreasing the strain rate. In [24], it was assumed that the ductility rises due to the interplay of GBS and the shear banding in commercial aluminum alloy 6082. In turn, in shear bands, GBS occurs. However, the authors of [21] suppose that the $m$ value is rather low for the tension of UFG aluminum at room temperature, making GBS an implausible explanation for the enhanced ductility at low strain rates. When accounting for the above, the measurement of the contributions of GBS to the overall deformation will reveal the deformation mechanisms that act during the tension of UFG metals at different strain rates.

3.2. Deformation relief

The SEM investigation shows that the deformation relief arising on the polished surfaces of the tested samples during deformation depends on the local strain in the site of observation at all of the studied strain rates. As mentioned above, $3/4$ of the gage length of the sample remains strainless after the test at $8.6 \times 10^{-3} \text{ s}^{-1}$; and the other $1/4$ represents the neck. No deformation relief is apparent on the strainless part of the gage. In the local region strained to $10–20\%$, bands of localized deformation (BLD) appear on the surface. They are visible as relatively coarse surface folds perpendicular to the tensile direction that are $2–4 \ \mu\text{m}$ wide and several hundred microns in length. The distance between BLD is $10–20 \ \mu\text{m}$ or more (Fig. 6a).
The relief characteristic to GBS is observed inside BLD and the adjacent areas (see the area rounded by the white unbroken line oval in Fig. 6b). GBS relief, if any, is poorly expressed between BLD in this region (the area rounded by the white intermittent line oval in Fig. 6b). The difference between the BLD and the micro shear bands described in [24] should be noted. First, the scratches bend but do not break in BLD, suggesting BLD are not related to GBS. Second, they are different in size. Third, the number of the micro shear bands on a square unit rise when decreasing a stain rate and vice versa for BLD.

Fig. 3. Engineering stress – strain curves obtained for UFG and coarse-grained (CG) aluminum at different strain rates.

Fig. 4. Distribution of strain in the local areas on the gage length calculated as a distortion of the mark circles.

Fig. 5. The ultimate ($\sigma_u$) and yield ($\sigma_y$) stress versus strain rate obtained from the tensile tests. Note the greater slope of the dependence at lower strain rates.
The long BLD are divided into parts tens of microns in length in the regions locally strained more than 30%. The number and width of the BLD increase. They cover practically all of this part of the gage. The network of grain boundaries due to GBS is visible between BLD; the more strain, the more GBS will occur. Signs of intragrain dislocation slip, arising as long straight lines and progressing from boundary to boundary, can be observed inside grains coarser than 5 µm (Fig. 6c).

Deformation relief arising on the sample tested at $\dot{\varepsilon} = 10^{-5}$ s$^{-1}$ differs significantly from the relief described above. The network of boundaries induced by GBS is clearly observed in the uniform elongated gage length of the sample (outside neck, $\varepsilon \approx 8–20\%$, Fig. 7a). Here there are regions containing BLD and containing no traces of GBS (Fig. 7b, 7c). These regions have a size of $\approx 50$ µm in width and 100–150 µm in length in the most strained local areas ($\varepsilon \approx 20\%$, Fig. 7c). In the less strained local areas ($\varepsilon \approx 13\%$) these regions are bigger (Fig. 7d). In the necking areas ($\varepsilon > 40\%$) there are no regions without GBS-related relief.

The samples tested at $\dot{\varepsilon} = 1.0 \times 10^{-4}$ and $3.1 \times 10^{-5}$ s$^{-1}$ exhibit the same features of the deformation relief as that tested at $\dot{\varepsilon} = 1.0 \times 10^{-5}$ s$^{-1}$. The size of the BLD-regions corresponds generally to the strain in the local areas in the uniform elongated gage length, i.e. the more the local strain, the less area of the BLD-regions. This suggests the interrelation between the number and the size of the BLD-regions and plastic strain value in a local uniform elongated area.

3.3. Measurement of GBS contribution to the overall deformation

The dependence of the GBS contribution to the overall deformation ($\eta$) versus the strain in a local area is presented in Fig. 8. $\eta$ is the upper limit of the GBS contribution to the overall deformation of the whole sample because there are areas where BLD develops and GBS does not. For deformation at $\dot{\varepsilon} = 8.3 \times 10^{-3}$ s$^{-1}$, the most of the gage length remains strainless: $\eta = 0$. The contribution is measurable in the neck area only: $\eta = 19\%$ in the area locally strained to 34%. At $\dot{\varepsilon} = 1.0 \times 10^{-4}$ and $3.4 \times 10^{-5}$ s$^{-1}$, $\eta = 20\%$ in the area of necking. In the uniform elongated parts $\eta$ reaches 27 and 72%, respectively. At a strain rate of $\dot{\varepsilon} = 1.0 \times 10^{-5}$ s$^{-1}$ the maximal value of $\eta$ is close to the head of the test sample ($\eta = 41\%, \varepsilon = 10\%$), while in the necking area, it is close to the break surface ($\eta = 45\%, \varepsilon = 44\%$). In the uniform elongated part, $\eta$ decreases while remaining rather high, $\eta = 31\%$. Therefore, $\eta$ varies from 20 to 70\% in the interval of the studied strain rates. It was shown earlier that the contribution of GBS to the overall deformation under the tension of the UFG aluminum at room temperature and $\dot{\varepsilon} = 8.3 \times 10^{-3}$ s$^{-1}$ reached 24\% [14]. For UFG aluminum, $\eta$ reached 40–70\% under indentation [13]. $\eta = 25\%$ for the compression of UFG copper at room temperature. Evidently, the contribution of GBS to the overall deformation agrees with the experimental data of the other researchers.

As mentioned above, contradictory opinions exist in the literature regarding the role of GBS when enhancing the strain rate sensitivity and improving the ductility of the UFG metals by decreasing the strain rate. The present data suggest that the contribution of GBS to the overall deformation increases when decreasing the strain rate, reaching a significant value in local areas (up to 40–70\% including in the uniform elongated regions). Therefore, GBS is very important.

The data obtained demonstrates the growth of uniform elongation, contribution of GBS to the overall deformation in local areas and the decrease in number and area of BLD-regions (the latter means the increase of GBS to the overall deformation of the whole sample) by decreasing the strain rate.
3.4. **On the origin of the regions containing BLD**

It is reasonably to assume that the appearance of the regions containing BLD is related to the structural features of UFG aluminum. They have been studied in detail elsewhere [13,26-29]. Analysis of these works reveals relatively lengthy bands (tens of micrometers in width and more than 50 µm in length) having bigger grain size and lower fraction of high angle grain boundaries in comparison with those in the main volume fraction of the material. These bands may be the structural reason for the BLD-regions formation. In turn, in the BLD-regions GBS occurs only in the neck region and at lowest strain rate. The GBS contribution to the overall deformation of the whole sample decreases, possibly explaining the relatively low value of m. To improve the ductility of the UFG materials by increasing the strain rate sensitivity and the contribution of GBS to the overall deformation, a homogeneous grain structure is required. This conclusion is indirectly confirmed by the studies of GBS and m in...
Al-30Zn alloy [23]. The contribution of GBS to the overall deformation in the alloy was 40–60\% and $m\approx0.25$ at $10^{-4}$ s$^{-1}$. The essentially increased value of $m$ in the alloy relative to the pure aluminum with close values of $\eta$ may be attributed to the fact that in alloys, the grain structure is more homogeneous [11]. However, this suggestion requires further experimental validation.

Fig. 7. Deformation relief on the surface of the sample tensioned at $\dot{\varepsilon}=1.0\times10^{-5}$ s$^{-1}$. (a) $\varepsilon=16\%$. (b) $\varepsilon=10\%$. (c) $\varepsilon=21\%$. (d) $\varepsilon=13\%$. The areas where preferably BLD appears are surrounded by the white ovals.

3.5. Calculation of GBS contribution to the overall deformation

GBS rate was calculated for the tension of aluminum at room temperature at a strain rate of $\dot{\varepsilon}=1.0\times10^{-5}$ s$^{-1}$. $\eta$ was taken as 50\% under these conditions so experimentally obtained GBS rate was $\dot{\varepsilon}_{\text{GBS}}^\text{exp}=5.0\times10^{-6}$ s$^{-1}$. Material parameters used in the calculation are listed in Table 1.
Calculation by the expression (3) results in the GBS rate of $\epsilon_{\text{GBS}}^{\text{calc}} = 1.2 \times 10^{-9} \text{ s}^{-1}$. Thus, calculated GBS rate is $4.2 \times 10^3$ lower than the measured one. This discrepancy may be due to utilization of the grain boundary diffusion coefficient in ordinary coarse-grained metal. There is a number of works [3,4,30-32] suggesting the higher value of grain boundary diffusion coefficient in UFG metals relative to coarse-grained ones. The estimation of their ratio varies from $1.1 \times 10^6$ [26,27] at 398 K for nickel to $1.3 \times 10^3$ at 371 K for palladium [30]. Utilization of the enhanced by 3–4 orders of magnitude grain boundary diffusion coefficient results in a good agreement between calculated and experimentally measured GBS rate.

Fig. 8. The contribution of GBS to the overall deformation in the local area versus the strain in this area obtained for the samples tested at different strain rates.

Table 1. Values needed to calculate GBS rate [2].

| Material                                      | Aluminum                   |
|-----------------------------------------------|----------------------------|
| Pre-exponential for grain boundary diffusion, $\delta D_0$, m$^3$/s | $5.0 \times 10^{-14}$     |
| Activation energy of grain boundary diffusion $Q_b$, J/mol           | $8.4 \times 10^4$         |
| Calculated grain boundary diffusion coefficient, m$^3$/s             | $5.2 \times 10^{20}$      |
| Shear modulus G, Pa                                         | $2.5 \times 10^{10}$      |
| Burgers vector b, m                                    | $2.9 \times 10^{-10}$     |
| Test temperature, K                                    | 293                       |
| Grain size d, m                                         | $10^{-6}$                 |
| Stress $\sigma$, Pa                                      | $1.0 \times 10^8$        |
| Grain boundary width $\delta$, m                       | $1.0 \times 10^{-8}$      |

4. Conclusions
1. The mechanical behavior of UFG aluminum exhibits several characteristic features compared to the coarse grained counterpart: high values of ultimate and yield stresses, relatively low elongation up to failure, a modest value of strain hardening, approach of the ultimate stress and the onset of deformation localization with little plastic strain. The strength characteristics decrease, the strain hardening changes insignificantly, elongation up to failure and uniformity of deformation rise by decreasing the strain rate. The latter effect should be explained by the increased strain rate sensitivity; this sensitivity is due to the increased contribution of GBS to the overall deformation at a low strain rate.

2. The deformation relief study reveals that intragrain dislocation slip in relatively coarse grains and the grain boundary sliding of ultrafine grains are involved in the plastic flow of UFG aluminum. Direct measurements of the contribution of grain boundary sliding to the overall deformation in local areas show that it increases by decreasing the strain rate. The
The upper limit of the contribution reaches 45% in the necking area and 72% in the area of the uniform elongation.

3. The value for the strain rate sensitivity remains low, even at low strain rates, despite the significant contribution of grain boundary sliding to the overall deformation. This effect is most likely related to the presence of the worse grain refined areas where grain boundary sliding is limited.

4. Calculated GBS rate is in a good agreement with the experimentally measured value when using grain boundary diffusion coefficient in UFG aluminum being enhanced by 3–4 orders of magnitude relative to coarse grained counterpart.

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