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Indentation behavior of aluminum processed by equal channel angular extrusion: Effect of bending angle

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Microindentation has been used to characterize the indentation behavior of aluminum processed by ECAE (equal channel angular extrusion). The bending angles of the ECAE dies are 90°, 120° and 150°. Using dislocation dynamics, it is found that the initial dislocation density increases with the decrease in the bending angle at the macroscopic yielding during the indentation. For the same indentation load, the plastic energy dissipated in the indentation increases with increasing bending angle.

Keywords: ECAE; Bending angle; Indentation; Stress–strain relation

In the last decade, there has been considerable interest in severe plastic deformation (SPD), which can be used to refine the microstructure of metallic alloys. Of the existing SPD processes, equal channel angular extrusion (ECAE) is one of the most versatile and potent techniques for fabricating ultrafine-grained materials in large quantities. In ECAE, a rod-shaped sample is extruded repetitively through a die with equal channels [1–5]. It has been demonstrated that the ECAE process is effective with aluminum alloys in producing equiaxed grains of submicron sizes [6]. The as-extruded aluminum alloys can be formed into domes [7] or rolled into thin sheets without a significant loss in superplastic behavior [8]. Different techniques, including tension and compression tests [9–13] and indentation tests [14–16], have been used to characterize the mechanical behavior of ECAE-processed Al under static and dynamic loading conditions.

A previous differential scanning calorimetric study of ECAE-processed Al over the temperature range from ambient to melting has shown that there was only a peak related to the growth of grains [17]. This suggests that the ECAE-processed Al had probably gone through the stages of recovery and recrystallization and had been in the stage of grain growth during the ECAE processing at room temperature. Thus, it is expected that the evolution of the microstructure in ECAE-processed Al depends on the geometrical configuration of the ECAE die, including the bending angle.

Microindentation tests have been used to obtain the indentation stress–indentation strain relation [16,18], from which important deformation parameters can be determined such as the thermal component of flow stress, the immobilization rate of dislocations, the remobilization probability of immobile dislocations and the initial dislocation density [18]. The evaluation of the initial dislocation density is significant since it enables assessment of the deformation severity in the ECAE-processed materials without using transmission electron microscopy to characterize the dislocation structure. Once all of the deformation parameters have been determined, the total dislocation density can be calculated to describe local plastic deformation as a function of plastic strain. Furthermore, from the loading–unloading curve, the plastic energy dissipated into the specimen in the indentation can be calculated [14,15,18] and associated with the dislocation density introduced via ECAE. This work highlights the effect of the bending angle of the ECAE die on the deformation severity in ECAE-processed Al from the viewpoint of dislocation dynamics.

Pure (99+%) aluminum rods of 12 mm in diameter were purchased from Alfa Aesar, MA, and machined into rods of 8 mm in diameter × 50.8 mm in length. All the machined Al rods were annealed at 773 K for 12 h and furnace-cooled to room temperature. The ECAE die consisted of two split blocks of tool steel (H-13),

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which were held together to form an internal channel of equal circular cross section (diameter 8 mm). These two channels are joined in an L-shape configuration. Three ECAE dies were used in which the bending angles between the two channels were 90°, 120° and 150°. The surface of the internal channel was lubricated with a mixture of MoS_2 powder and mechanical pump oil to reduce the friction between the die wall and the Al rod. A successive extrusion was carried out by rotating the rod 180° after the first extrusion. All the extrusions were performed at room temperature.

Microindentation tests were performed at room temperature, using a spherical indenter on a Micro-Combi Tester (CSM Instruments, Needham, MA). Four spherical diamond indenters with radii of 0.01, 0.05, 0.1 and 0.2 mm were used. The indentation loads were 80–240 mN for the 0.01 mm indenter, 300–1300 mN for the 0.05 mm indenter, 700–1800 mN for the 0.1 mm indenter and 1000–5000 mN for the 0.2 mm indenter. Both the loading time and unloading time were 30 s without an intermediate pause during the indentation, and constant loading and unloading rates were used in each indentation test. Prior to full indentation, a preload of 5 mN was applied to the indenter in order to maintain contact between the indenter and the surface of the sample and also to avoid the effect of impact. The loading–unloading curves were recorded, and from these the plastic energy dissipated in the indentation was calculated. After completely removing the indentation load, the impression size, $2a$ in diameter, was measured under a LOM, and was used to calculate the indentation stress and strain.

Following the method proposed by Tabor [19], Yang et al. [16,18] recently constructed the indentation stress–indentation strain curves for Al, ECAE-processed Al and cold-rolled Al. They observed similar work-hardening behavior to the tensile deformation of Al. Their results suggest that Tabor's approach [19] can be extended to analyze highly cold-worked materials. Extending Tabor's approach [19] to the indentation of the cold-worked Al by a rigid spherical indenter, one can calculate the indentation stress σ_{ind} and indentation strain ε_{ind} as

$$\sigma_{\text{ind}} = F/\pi a^2, \tag{1}$$

$$\varepsilon_{\text{ind}} = 0.18a/r, \tag{2}$$

where F is the indentation force applied to the indenter and r is the radius of the indenter.

Figure 1 shows the indentation stress–indentation strain curves of the ECAP-processed Al; the data for each curve were calculated from the results of the indentation tests using four different indenter sizes. All three curves are almost parallel to each other. It requires the highest indentation stress to produce the same indentation strain in the Al ECAE-processed with the 90° ECAE die, while it requires the least indentation stress in the ECAE-processed Al with the 150° ECAE die. This is probably due to the formation of dislocations in the ECAE-processed Al samples during the extrusion. As observed in the finite element simulation of the ECAE process [20], the plastic strain in ECAE-processed Al increases as the bending angle decreases. The ECAE extrusion produces the highest dislocation density in the ECAE-processed Al using the 90° ECAE die. This introduces the highest resistance to the penetration of the indenter.

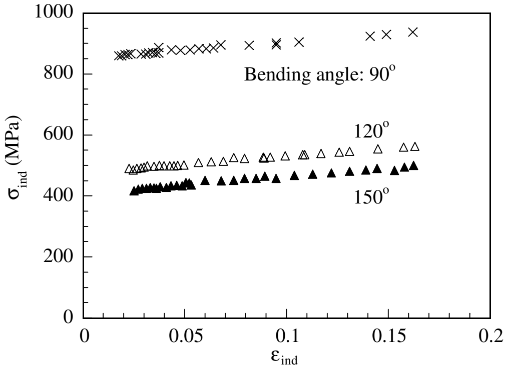

Figure 1. Dependence of the indentation stress on the indentation strain for the indentation of the ECAE-processed Al.

Using the dislocation dynamics and the relation between tensile stress and tensile strain as proposed by Bergström [21], one can express the relation between the indentation stress and the indentation strain as [16,18]

$$\sigma_{\text{ind}} = \sigma^*_{\text{ind}} + \alpha\mu b[(U/\Omega)(1 - e^{-\Omega\varepsilon_{\text{ind}}}) + \rho_0 e^{-\Omega\varepsilon_{\text{ind}}}]^{1/2} \tag{3}$$

where σ^*_{ind} is the thermal component of the 'indentation flow stress' (about three times the thermal component of the flow stress in tensile test), α a geometrical constant, μ the shear modulus, b the Burgers vector, U the immobilization rate of dislocations in the indentation deformation zone, Ω the remobilization probability of immobile dislocations in the indentation deformation zone, and ρ_0 the initial dislocation density at the macroscopic yielding during the indentation (at nearly zero indentation strain). The indentation stress–indentation strain behavior is then computer-simulated with four unknown parameters of $\sigma^*_{\text{ind}}, U, \Omega$ and ρ_0 using the non-linear curve-fitting software Peakfit from Jandel Scientific. In the simulation, the following parameters are used: $\alpha = 0.4$ [22], $\mu = 27$ GPa, $b = 0.2893$ nm. The Burgers vector b is calculated from the lattice constant of Al. Table 1 lists the simulation results.

A comparison of the simulation results with those for annealed Al [16] shows several interesting aspects regarding the severity of the plastic deformation in the ECAE-processed Al, which was created during the extrusion. First, the annealed Al has $\sigma^*_{\text{ind}} = 42.23$ MPa and $\Omega = 0.107$. Both parameters are supposed to be identical to those for the ECAE-processed Al since they are thermally dependent parameters. Secondly, the immobilization rate of dislocations for the annealed Al

Table 1. Deformation parameters for the ECAE-processed aluminum

Bending angle (degrees)	U/Ω (10^{12} cm^{-2})	Ω	σ^*_{ind} (MPa)	ρ_0 (10^{12} cm^{-2})
90	8.50	0.16	42.32	6.32
120	4.91	0.16	42.32	1.84
150	4.38	0.16	42.32	1.27