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Microstructure and yield strength of severely deformed silver

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High-purity silver was processed by equal-channel angular pressing (ECAP) to eight passes, giving an average grain size of ~250 nm. The densities of dislocations and twins were measured and compared with reported values for other face-centered cubic metals processed by ECAP. The results show the saturation yield strength correlates to the dislocation density according to the Taylor equation, and that the value of the geometrical parameter α in the Taylor equation is strongly influenced by the stacking fault energy.

Keywords: Equal-channel angular pressing; Severe plastic deformation; Stacking fault energy; Silver; Taylor equation

Equal-channel angular pressing (ECAP) is an effective tool for producing bulk ultrafine-grained metals [1]. Because of the practical importance of these materials, the properties of ultrafine-grained face-centered cubic (fcc) metals formed by ECAP have been extensively investigated [2–6]. These experiments show that the average dislocation density increases with increasing strain while the individual crystallite size becomes smaller [7]. At room temperature, these two quantities reach saturation values after about 4–8 ECAP passes which, for an ECAP die having an internal channel angle of 90°, corresponds to an imposed strain of ~4–8 [8]. It has been demonstrated also that simultaneously the yield strength increases with increasing strain and saturates after ~4–8 passes [5,7,9,10]. The microstructure and the yield strength of ECAP-processed fcc metals with medium (e.g. Cu) or relatively high (e.g. Al, Ni) stacking fault energies (SFEs) have been studied in numerous papers. However, only very limited information is available for materials having low SFE such as Au [11], and no data are available to date documenting the microstructures in severely deformed Ag.

The present investigation was initiated to examine the microstructural characteristics of high-purity silver after processing by ECAP where this metal has an exceptionally low SFE, variously reported as ~22 mJ m^{-2} [12] and ~16 mJ m^{-2} [13]. The results obtained for the defect densities in this study are compared with those reported for other fcc metals processed by ECAP in order to critically evaluate the influence of the SFE on the relationship between the dislocation density and the yield strength.

High-purity 99.99% Ag billets having lengths of ~70 mm and diameters of ~10 mm were homogenized for 60 min at a temperature of 741 K (corresponding to $0.6T_m$, where T_m is the absolute melting point of Ag). The billets were then pressed through different passes in ECAP at room temperature using a pressing velocity of 1 mm s^{-1} and a die with an internal channel angle of 90°. The pressing was conducted using route B_c in which the billet is rotated about its longitudinal axis by 90° in the same direction after each pass [14]. For this die configuration, one pass corresponds to an equivalent strain of ~1 [8]. Based on tests conducted up to 16 passes, it was concluded that the saturation state of the microstructure was achieved after testing through eight passes. Accordingly, in the following, results were

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obtained on a sample of Ag processed by ECAP for a total of eight passes.

The microstructure of the material was examined after ECAP on a transverse section perpendicular to the axis of the billet. An X-ray diffraction line profile analysis was performed using a special high-resolution diffractometer (Nonius FR591) with Cu $K\alpha_1$ radiation ($\lambda = 0.15406$ nm). The line profiles were evaluated using the extended convolutional multiple whole profile (eCMWP) fitting procedure [15]. The dislocation density (ρ) and the twin fault probability (β), equal to the frequency of {1 1 1} twin faults along a ⟨1 1 1⟩ direction, were determined from these fitting parameters. The microstructure after ECAP was also studied using atomic force microscopy (AFM) and by transmission electron microscopy (TEM) with a Philips CM-20 operating at 200 kV. The TEM sample was mechanically thinned to about 50 μm, cooled to liquid nitrogen temperature and then thinned with 6 kV Ar^+ ions from both sides until perforation. Finally, a thin damaged layer was removed using 2 keV Ar^+ ions. The yield strength was determined by uniaxial compression in the direction of the longitudinal axis of the as-pressed billet.

Figure 1 shows typical AFM and TEM images after eight passes of ECAP. It is apparent that processing by ECAP produces a relatively homogenous grain structure with an average grain size of the order of ~250 nm. Similar grain sizes were recorded in both AFM and TEM and this confirms that the ion beam imparts no significant damage to the material. It is apparent from the TEM micrograph in Figure 1b that, in addition to grain boundaries and dislocations, the structure contains twins as a consequence of the very low twin fault energy of Ag (~8 mJ m^{-2} [13]). The more highly magnified micrograph in Figure 1c shows that the dislocations have reasonably uniform distributions within the grains. Earlier studies of Cu and Al after ECAP showed the dislocations were arranged into dense structures to lower their strain energies [5,7]. The different dislocation structure observed in Ag after ECAP is attributed to the higher degree of dissociation into partials due to the exceptionally low SFE. This means that cross-slip and/or climb of highly dissociated dislocations is difficult in Ag and this hinders the rearranging of these dislocations into walls or subgrain boundaries. Based on the TEM images, a relatively high dislocation density of the order of $\sim 50 \times 10^{14}$ m^{-2} may be estimated for the dislocation structure of Ag after ECAP.

Table 1 gives the values of the dislocation density (ρ) and twin probability (β) determined by X-ray line profile analysis. Thus, in Ag processed through eight ECAP passes the total dislocation density determined by X-ray line profile analysis ($\sim 46 \times 10^{14}$ m^{-2}) is in good agreement with the dislocation density in the grain interiors estimated from the TEM images. For comparison purposes, Table 1 shows also the reported dislocation densities and twin probabilities of other fcc metals processed by ECAP to the saturation condition [11,17,18]: an exception is Ni where the value of ρ is taken from unpublished data of the present authors. Figure 2 gives the twin fault probability plotted against the twin fault energy and it shows there is a sharp increase in the twin probability in the saturated condition with decreasing twin fault energy.

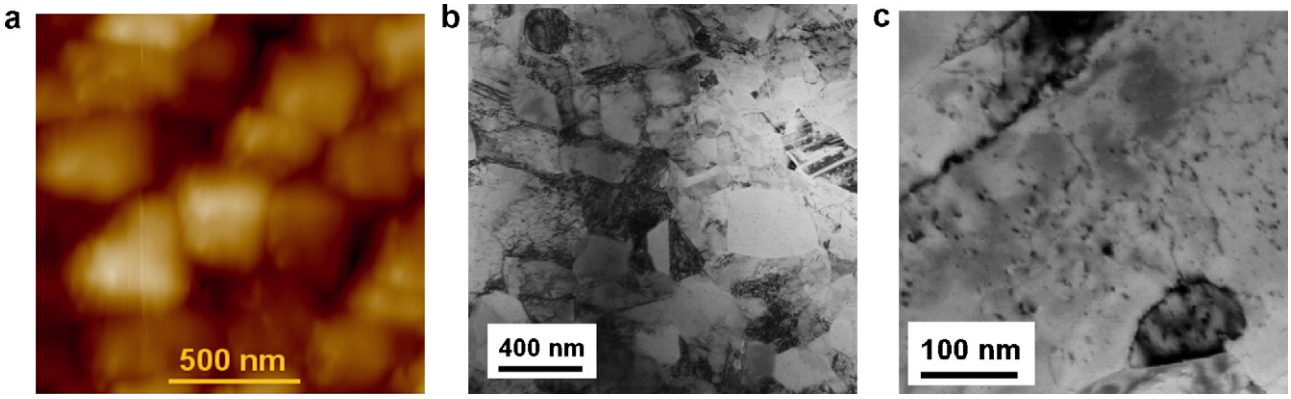

Figure 1. (a) AFM and (b, c) TEM images at two different magnifications of the microstructure of Ag processed by ECAP for eight passes.

Table 1. The stacking fault energy (γ), the twin fault energy (γ_T), the shear modulus (G), the maximum dislocation density (ρ), the twin fault probability (β), the friction stress (σ_0), the saturation yield strength (σ_Y), the equilibrium splitting distance of partials in extended dislocations (d_p) and the value of α calculated from Eq. (1) using the experimental values of σ_Y, σ_0 and ρ

Metal	γ (mJ m^{-2})	γ_T (mJ m^{-2})	G (GPa)	ρ (10^{14} m^{-2})	β (%)	σ_0 (MPa)	σ_Y (MPa)	d_p (nm)	α
Ag, 8ECAP	22 [12]	8 [13]	30 [16]	46 ± 5	0.9 ± 0.1	29 ± 3	330 ± 10	3.5	0.17 ± 0.02
	16 [13]							4.8	
Au, 4ECAP	45 [12]	15 [13]	27 [16]	17 ± 2 [11]	0.28 ± 0.04	27 ± 3 [6]	245 ± 7	1.5	0.22 ± 0.02
	32 [13]							2.2	
Cu, 5ECAP	78 [12]	24 [13]	47 [16]	21 ± 2 [17]	0.07 ± 0.03	35 ± 4 [6]	394 ± 10	1.2	0.22 ± 0.02
	45 [13]							2.1	
Ni, 6ECAP	125 [13]	43 [13]	82 [16]	18 ± 2 [unpub.]	0.02 ± 0.02	60 ± 5 [6]	730 ± 20	1.3	0.27 ± 0.02
Al, 4ECAP	166 [13]	75 [13]	26 [16]	1.9 ± 0.2 [18]	0.00 ± 0.02	20 ± 2 [6]	120 ± 4	0.4	0.32 ± 0.02

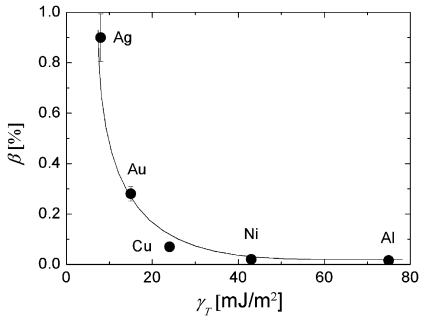

Figure 2. The twin fault probability determined in the saturated condition for fcc metals as a function of the twin fault energy.

Earlier studies showed that the dislocation density and the yield strength of fcc metals exhibited similar variations with strain, and both quantities saturate at approximately the same strain when processing by ECAP [4,7]. Table 1 includes the maximum dislocation density determined by X-ray line profile analysis and the corresponding saturation yield strength measured by mechanical testing for different fcc metals. It is well known that the relationship between the dislocation density (ρ) and the yield strength (σ_Y) for plastically deformed metals generally follows the Taylor relationship, which is given by

$$\sigma_Y = \sigma_0 + \alpha M^T G b \rho^{1/2}, \quad (1)$$

where σ_0 is the friction stress, α is a geometrical constant depending on the arrangement of dislocations, G is the shear modulus, b is the length of the Burgers vector and M^T is the Taylor factor. Since the value of αM^T is of the order of 1, it is possible to check the validity of Eq. (1) for the saturation state achieved in ECAP by plotting the value of $(\sigma_Y - \sigma_0)$ against the product of $Gb\rho^{1/2}$ for different fcc metals, as shown in Figure 3. It is important to note that the errors on the individual datum points in Figure 3 are very small, and are smaller than the sizes of the individual symbols when no error bars are indicated.

The good correlation between these two quantities in Figure 3 demonstrates that in fcc metals processed by ECAP the saturation value of the yield strength is essentially determined by the interactions between dislocations. The scattering of the datum points in Figure 3 is attributed to the difference between the values of α for the different metals. Therefore, the values of α in Table 1 were calculated from Eq. (1) by using the experimental values of σ_Y and ρ. In the calculation of parameter α, the value of M^T was estimated as the average of the Taylor factors for the 111, 200, 220, 311, 331 and 420 orientations weighted by the strength of the orientations. The strength of an *hkl* orientation was determined as the product of its multiplicity and the ratio of the experimental integrated intensity of the *hkl* reflection and the theoretical intensity characterizing a texture-free polycrystal. The individual Taylor factors of the different orientations were determined from Figure 4b in an earlier report [19]. The M^T values for Al, Ni, Cu, Au and Ag were obtained as 3.04, 2.90, 2.98, 3.12 and 3.08, respectively. These values deviate only slightly from the value characterizing the random case (3.06), thereby indicating that the texture in the studied samples is relatively weak.

Comparing the values of α for all fcc metals in Table 1, it is apparent that α is highest for Al and lowest for Ag. In practice, the value of α depends upon the arrangement of dislocations in the material [20,21]. For example, it has been shown that the value of α increases from ~0.15 to ~0.37 when dislocation clustering increases and the dislocation structure evolves from a uniform random distribution through thick cell walls to sharp boundaries [20].

The dissociation of lattice dislocations into partials in fcc metals has a strong effect on the arrangement of dislocations. In the formation of dense dislocation structures, as in walls or subgrain boundaries, an important role is played by cross-slip. Thus, the higher the degree of dissociation, so the more difficult the occurrence of cross-slip. The degree of dislocation dissociation is characterized by the equilibrium splitting distance (d_p) between the partials of the dissociated dislocations. Assuming the dislocation before dissociation is of a mixed type with half-edge and half-screw character, the splitting distance between the partials may be expressed by the shear modulus, G, the Burgers vector, b, and the stacking fault energy, γ, through the expression [22]:

$$d_p = 0.031 \frac{Gb^2}{\gamma}. \quad (2)$$

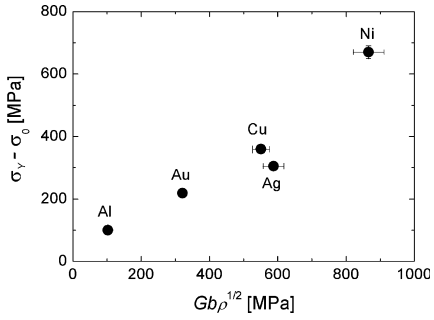

Figure 3. The saturation values of the yield strength reduced by the friction stress $(\sigma_Y - \sigma_0)$ vs. the product of $Gb\rho^{1/2}$ for fcc metals processed by ECAP.

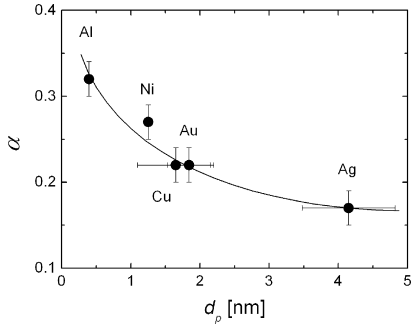

Figure 4. The value of α in the Taylor equation as a function of the equilibrium splitting distance (d_p) of partials in dissociated dislocations for fcc metals.

Table 1 lists the different values of γ reported in the literature and the values of d_p obtained from application of Eq. (2). It should be noted that these values differ slightly from those documented in an earlier report where data were collected from several different authors [23]. To reduce the uncertainty in the d_p values, the same relationship (Eq. (2)) was used for all materials and the same value of 45° was assumed for the angle between the line and Burgers vectors of dissociated lattice dislocations. The values of α against d_p are plotted in Figure 4 where the uncertainty in d_p due to the different values of γ is incorporated in the error bars. It is apparent from this plot that α decreases with increasing equilibrium splitting distance. These results are readily understood when it is noted that the higher values of d_p lead to higher degrees of dislocation dissociation and that this impedes the formation of sharp boundaries and gives relatively low values of α. As a consequence, Ag has the lowest value of α. It should be noted that, beside the interaction between dislocations, the relatively high frequency of twins in Ag is expected to make an additional contribution to the strength. However, if this hardening effect is taken into account, the Taylor-type contribution to the total yield strength will be reduced and this will lead to an even smaller value of α for Ag, providing additional confirmation that the higher splitting distance is accompanied by a smaller value of α.

The extremely low value of the stacking fault energy in Ag leads to a very high saturation density of dislocations in a reasonably uniform distribution, as shown by the TEM images of Figure 1. It is known that the distribution of dislocations can be characterized by the so-called dislocation arrangement parameter, M, determined also by X-ray line profile analysis [21]. The higher the screening of the strain fields of dislocations, the smaller the value of M. In the saturation state achieved by ECAP, the following M values were obtained: 2.0 ± 0.2 for Al, Ni and Cu, 1.4 ± 0.1 for Au and 1.2 ± 0.1 for Ag It is interesting to note that, in spite of the rather random distribution of dislocations, the Ag sample has the smallest value of M. This apparent dichotomy is attributed to the extremely high density of dislocations with opposite Burgers vectors, which is a direct consequence of the restricted cross-slip in Ag. Thus, dislocations with opposite signs are very close to each other but cannot annihilate due to the difficulty of cross-slip. The screening of the strain fields of such dislocation arrangements may be more effective than the dense structures that develop in metals with medium or high SFEs.

In summary, the microstructural characteristics of pure 99.99% silver were studied after processing by ECAP for eight passes at room temperature. It was found that the refined grain size is ~250 nm and a high density of twins is formed during ECAP in addition to the introduction of a high dislocation density. Within the ultrafine grains produced by ECAP, the dislocations have reasonably uniform distributions instead of arranging into cell walls or subgrain boundaries. This contrasts with other fcc metals which have medium or high SFEs. An analysis shows that the saturation yield strength of fcc metals processed by ECAP correlates with the maximum value of the dislocation density according to the Taylor relationship. It is shown also that the value of the geometric constant in the Taylor equation, α, decreases with an increasing splitting distance of the partials in dissociated dislocations due to the decreased degree of clustering of dislocations within the grains.

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