Journal of Alloys and Compounds 483 (2009) 271–274

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

journal homepage: www.elsevier.com/locate/jallcom

# Correlation between microstructure and mechanical properties of severely deformed metals

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ARTICLE INFO

*Article history:*
Received 30 August 2007
Received in revised form 4 July 2008
Accepted 14 July 2008
Available online 13 December 2008

*Keywords:*
Metals and alloys (A)
Dislocations and disclinations (C)
Mechanical properties (C)
Microstructure (C)
X-ray diffraction (D)

ABSTRACT

There is a correlation between the microstructure and the mechanical behavior of ultrafine-grained face centered cubic (fcc) metals processed by equal-channel angular pressing (ECAP). It is shown that the saturation yield strength is related to the maximum dislocation density according to the Taylor equation and, in addition, the value of the parameter $\alpha$ in the Taylor equation is strongly affected by the stacking fault energy because of different geometrical arrangements of dislocations within the grains. It is also demonstrated that the ductility of Cu processed by ECAP decreases with increasing strain but at extremely high strains the ductility is partially restored due to the recovery of the microstructure.



## 1. Introduction

Equal channel angular pressing (ECAP) is an effective tool for producing bulk ultrafine-grained (UFG) metals [1]. Because of their practical importance, UFG face centered cubic (fcc) metals formed by ECAP have been studied extensively [2–4]. It has been shown that, with increasing strain, the average dislocation density increases while the individual crystallite size becomes smaller [5,6]. At room temperature, these two quantities reach their saturation values after about 4–10 ECAP passes which, for an ECAP die having an internal channel angle of 90°, corresponds to an imposed strain value of ~4–10 [4–6]. It has been also demonstrated that simultaneously the yield strength increases with increasing strain and saturates at high strains [4,5,7,8]. Furthermore, the increase in the strength is generally accompanied by a reduction of ductility in ECAP-processed metals [4]. Different strategies were elaborated for achieving a combination of high strength and good ductility in bulk UFG metals [9]. One of the simplest way to improve the ductility is the application of a well-controlled heat-treatment after severe plastic deformation [10]. This thermomechanical procedure results in a bimodal grain structure with micrometre-sized grains embedded inside a matrix of ultrafine grains. The matrix grains impart high strength, while the large grains produced pronounced strain hardening to sustain the uniform deformation to large strains [10]. It was also observed in some materials that very large deformation by ECAP (above a strain value of ~16) might also lead to an increase in the ductility at room temperature [4,11].

The objective of the present report is to examine the correlation between the microstructure and the mechanical behavior of different fcc metals such as pure Al, Ni, Cu, Au and Ag processed by ECAP. The microstructural characteristics determining the saturation yield strength are specified. The influence of the number of ECAP passes on the yield strength and the ductility is investigated for pure Cu up to an extremely high strain value of ~29.

## 2. Experimental materials and procedures

High-purity (99.99%) Al, Ni, Au and Ag were processed by ECAP at room temperature using a 90° ECAP die and following route $B_C$ (i.e. the sample was rotated about its longitudinal axis by 90° in the same direction after each pass [12]). For a die with an angle of 90° between the two parts of the channel, one pass corresponds to an equivalent strain close to ~1 [13]. It was shown earlier that after several passes the microstructural parameters and the yield strength tend to saturate [5]. In this report, only the characteristic parameters corresponding to the saturation states are presented.

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doi:10.1016/j.jallcom.2008.07.200

The Al and Au samples reached a saturation state after 4 passes [5,14] having grain sizes of about 1200 nm [15] and 460 nm [14], respectively. The Ag specimen reached a maximum dislocation density after 8 ECAP passes with a grain size of about 200 nm. In these experiments the Ni samples failed during ECAP above 6 passes, therefore the microstructure and the yield strength obtained after 6 passes are used in this study. Oxygen-free copper (99.98% purity) samples were processed to a saturation state after 5 passes but the processing was continued up to 25 passes in order to study the effect of extremely large strains on the microstructure and the mechanical properties. For the die configuration used in this study, 25 passes correspond to a strain of about 29. The yield strength and the ductility were determined by tensile testing after ECAP. Details of the preparation of the samples and the measurements of the mechanical properties were given elsewhere [5,14,16].

Microstructures were investigated by analyzing X-ray diffraction line profiles. The X-ray diffraction experiments were performed using a special high-resolution diffractometer (Nonius FR591). The diffractometer was operated at 40 kV and 70 mA using a rotating Cu anode (CuK$\alpha_1$ radiation: $\lambda$ = 0.15406 nm). The line profiles were evaluated by the extended Convolutional Multiple Whole Profile (eCMWP) fitting procedure described elsewhere [17]. From the fitting parameters, the area-weighted mean domain size, the dislocation density ($\rho$) and the twin density ($\beta$) were determined [17]. The microstructure of Ag processed by 8 ECAP passes was also studied by Philips CM-20 transmission electron microscope operating at 200 kV. The TEM sample was mechanically thinned to about 50 μm, and then the sample, cooled to LN2 temperature, was thinned with 6 kV Ar$^+$ ions from both sides till perforation. Finally, the thin damaged layer was removed by 2 keV Ar$^+$ ions. This procedure minimized the thermal load during the preparation of the TEM sample.

## 3. Results and discussion

Previous studies [3,5] revealed that for fcc metals processed by ECAP the dislocation density and the yield strength show similar developments as a function of strain and both quantities saturate approximately at the same strain. This suggests that it is worthwhile studying the relationship between the saturation values of these quantities. The maximum dislocation density ($\rho_{max}$) determined by X-ray line profile analysis and the corresponding saturation yield strength ($\sigma_{max}$) measured by mechanical tests for different fcc metals are listed for these five metals in Table 1. The relationship between the dislocation density ($\rho$) and yield strength ($\sigma$) for plastically deformed metals is generally characterized by the Taylor equation:

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

where $\sigma_0$ is the friction stress, $\alpha$ is a constant depending on the arrangement of dislocations, $G$ is the shear modulus, $b$ is the length of the Burgers vector and $M$ is the Taylor factor (a value of $M$ = 3 was used in this study). The value of $\alpha M$ is of the order of 1 and therefore the validity of Eq. (1) for the saturation state achieved by ECAP may be checked by plotting the value of $\sigma_{max} - \sigma_0$ versus the product of $Gb\rho_{max}^{1/2}$ for different fcc metals. This plot for the five fcc metals is shown in Fig. 1a.

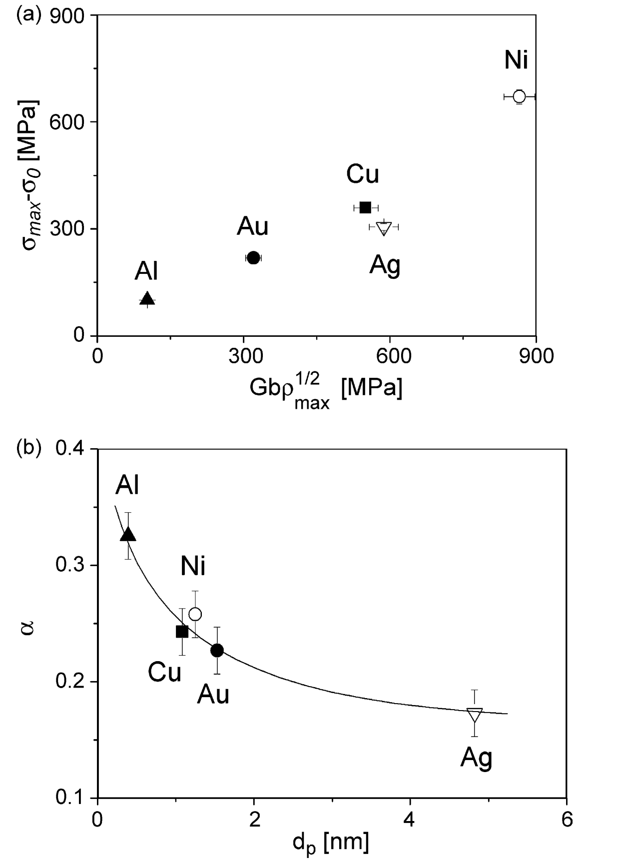


**Fig. 1.** (a) The saturation values of the yield strength reduced by the friction stress ($\sigma_{max} - \sigma_0$) versus the product of $Gb\rho_{max}^{1/2}$ for ECAP processed pure fcc metals where $G$ is the shear modulus, $b$ is the length of the Burgers vector and $\rho_{max}$ is the maximum value of the dislocation density. (b) The value of $\alpha$ in the Taylor equation as a function of the equilibrium splitting distance of partials in dissociated dislocations ($d_p$) for pure fcc metals.

The good correlation between these two quantities in Fig. 1a indicates 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 Fig. 1a is attributed to the difference between the values of $\alpha$ for the different metals. Thus, the value of $\alpha$ was calculated from Eq. (1) by using the experimental values of $\sigma_{max}$ and $\rho_{max}$, as recorded in the last column of Table 1. It is apparent that the value of $\alpha$ is highest for Al and lowest for Ag. It was shown previously that the value of $\alpha$ depends on the arrangement of dislocations [18,19]. Hernández Olivares and Gil Sevillano [18] showed that the more clustered the dislocation structure, the higher the value of $\alpha$. For example, in the case of a uniform random distribution of dislocations the value of $\alpha$ is about 0.15 while for sharp dislocation walls $\alpha$ is 0.37. The

**Table 1**
The stacking fault energy ($\gamma$), the maximum dislocation density ($\rho_{max}$), the twin density ($\beta$), the friction stress ($\sigma_0$), the saturation yield strength ($\sigma_{max}$), the equilibrium splitting distance of partials in extended dislocations ($d_p$) and the value of $\alpha$ calculated from Eq. (1) using the experimental values of $\sigma_{max}$, $\sigma_0$, and $\rho_{max}$.

| Material | $\gamma$ [mJ/m$^2$] | $\rho_{max}$ [$10^{14}$ m$^{-2}$] | $\beta$ [%] | $\sigma_0$ [MPa] | $\sigma_{max}$ [MPa] | $d_p$ [nm] | $\alpha$ |
|---|---|---|---|---|---|---|---|
| Ag, 8ECAP | 16 [22] | 46 ± 5 | 0.9 ± 0.1 | 29 ± 3 | 330 ± 10 | 4.8 | 0.17 ± 0.02 |
| Au, 4ECAP | 45 [21] | 17 ± 2 [14] | 0.28 ± 0.04 | 27 ± 3 [2] | 245 ± 7 | 1.5 | 0.23 ± 0.02 |
| Cu, 5ECAP | 78 [21] | 21 ± 2 [23] | 0.07 ± 0.03 | 35 ± 4 [2] | 394 ± 10 | 1.2 | 0.24 ± 0.02 |
| Ni, 6ECAP | 125 [21] | 18 ± 2 [unpub.] | 0.02 ± 0.02 | 60 ± 5 [2] | 730 ± 20 | 1.3 | 0.26 ± 0.02 |
| Al, 4ECAP | 166 [21] | 1.9 ± 0.2 [24] | 0.00 ± 0.02 | 20 ± 2 [2] | 120 ± 4 | 0.4 | 0.33 ± 0.02 |

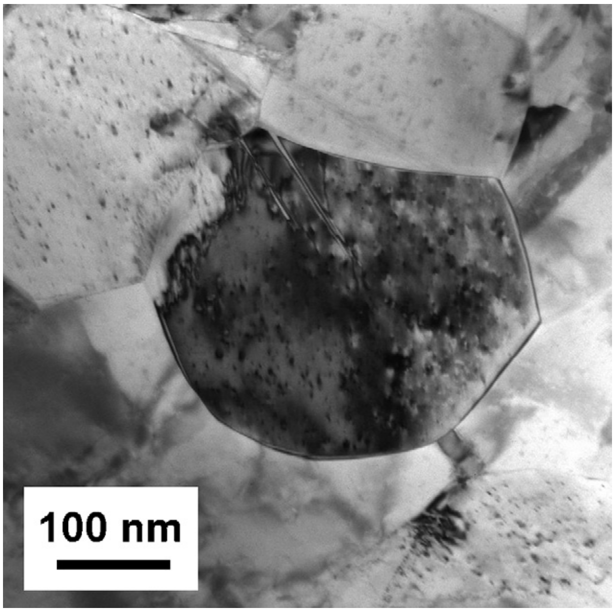


**Fig. 2.** Bright field (BF) TEM image of the microstructure of Ag processed by ECAP for 8 passes.

dissociation of lattice dislocations into partials in fcc metals has a strong effect on the arrangement of dislocations. The equilibrium splitting distance between the partials of dissociated dislocations ($d_p$) was determined from the shear modulus and the stacking fault energy ($\gamma$) according to conventional calculations [20] and these values are also listed in Table 1 where the values of $\gamma$ are taken from available data [21,22]. The values of $\alpha$ are plotted as a function of $d_p$ in Fig. 1b and it can be seen that the values decrease with increasing $d_p$. Thus, the higher the value of $d_p$ the higher the degree of dislocation dissociation which impedes the formation of sharp boundaries resulting in a relatively low value of $\alpha$.

Fig. 2 shows a TEM micrograph taken on the cross-section of an Ag sample processed through 8 ECAP passes. It can be seen that within the grains the dislocations have uniform distributions instead of arranging into cell walls or subgrain boundaries. However, for Cu or Al where the dislocation-dissociation is smaller, the majority of dislocations lie in the walls or subgrain boundaries [4]. Table 1 shows also that the value of the twin density, $\beta$, determined by X-ray line profile analysis increases with decreasing stacking fault energy. This trend is reasonable because the twin boundary energy also changes in the same way as $\gamma$, as shown elsewhere [21].

Fig. 3 gives the development of several mechanical and microstructural characteristics of Cu during ECAP. The yield strength and the maximum elongation are plotted in Fig. 3a as a function of the number of ECAP passes for pure Cu and the dislocation density and the domain size are shown in Fig. 3b versus the number of ECAP passes. The experimental results confirm that up to 10 passes the yield strength increases while the ductility decreases with increasing strain as a consequence of the accumulation of dislocations in the grain/subgrain boundaries. After 15 passes the ductility is partially restored and this is accompanied by a decrease in the dislocation density. Moreover, after 25 passes the recovery leads to increasing domain size which is accompanied by a further increase in ductility. This structural recovery at large strains is accompanied by a decrease in the grain boundary thickness corresponding to an evolution from non-equilibrium boundaries to a more equilibrated structure [25,26] and by an increase in the misorientation between neighboring grains as demonstrated by

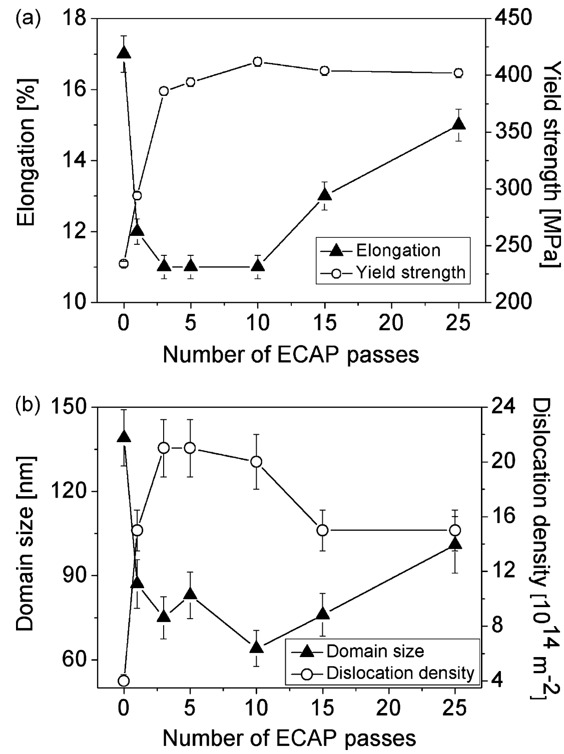


**Fig. 3.** The yield strength and the maximum elongation (a) and also the dislocation density and the domain size (b) as a function of the number ECAP passes for pure Cu.

data presented elsewhere [16]. Thus, the larger fraction of high-angle grain boundaries facilitates grain boundary sliding and this improves the ductility [11].

The value of $\alpha$ was calculated from Eq. (1) and plotted as a function of the number of ECAP passes in Fig. 4. The increase in the value of $\alpha$ with increasing strain is explained by the thinning of the grain boundaries and the evolution to a more equilibrated structure. The higher value of $\alpha$ leads to the restoration of high strength after 25 passes despite the corresponding decrease in the dislocation density.

## 4. Summary

The saturation yield strength of fcc metals processed by ECAP is correlated to the maximum value of the dislocation density according to the Taylor equation. It is shown that the value of $\alpha$

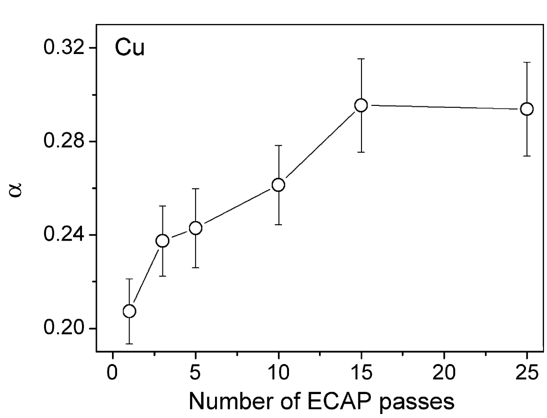


**Fig. 4.** The value of $\alpha$ in the Taylor equation as a function of the number of ECAP passes for Cu processed by ECAP.

in the Taylor relationship decreases with an increasing equilibrium splitting distance of the partials in dissociated dislocations as a result of a decrease of clustering of dislocations within the grains. When pure Cu is processed by ECAP to extremely large strains, the ductility is partially restored due to a transformation of grain boundaries into high-angle boundaries having a more equilibrated structure.

## Acknowledgements

This work was supported in part by the Hungarian Scientific Research Fund, OTKA, Grant Nos. K67692 and K71594 (JG and NQC) and in part by the National Science Foundation of the United States under Grant No. DMR-0243331 (TGL). In addition, JG is grateful for the support of a Bolyai János Research Scholarship of the Hungarian Academy of Sciences. The authors are obliged to Andrea Jakab for the preparation of the TEM sample.

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