

## Microstructure and mechanical behavior of severely deformed f.c.c. metals

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**Abstract.** The correlation between the microstructure and the mechanical behavior of ultrafine-grained face centered cubic (f.c.c.) metals processed by equal-channel angular pressing (ECAP) was studied. It was found that the maximum value of the yield strength obtained at high strains is determined by the shear modulus and the saturation value of the dislocation density according to the Taylor equation. It was also revealed that the value of the parameter  $\alpha$  in this equation decreases with decreasing stacking fault energy, indicating the effect of different geometrical arrangements of dislocations in the grain boundaries. In addition, it was shown that for ECAP processed Cu, the ductility decreases with increasing strain but at extremely high strains the ductility is partially restored due to a recovery of the grain boundary structure.

### Introduction

Severe plastic deformation (SPD) is an effective tool for producing bulk ultrafine-grained metals. One of the most common SPD methods is equal channel angular pressing (ECAP) that results in a homogeneous ultrafine-grained microstructure of the workpiece [1]. The ultrafine-grained materials produced by ECAP have a very high strength owing to their small grain size and high dislocation density [2]. Because of their practical importance, ultrafine-grained face centered cubic (f.c.c.) metals formed by SPD 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]. At room temperature, these two quantities reach their saturation values after about 4-10 ECAP passes which corresponds to an imposed strain value of  $\sim 4-10$  [4,5]. It has been reported also that simultaneously the yield strength increases with increasing strain and saturates at high strains [4-6]. Furthermore, the increase of the strength is generally accompanied by a reduction of ductility in the SPD-processed metals [4]. At the same time, after very large deformation by ECAP (above a strain value of 16), the ductility at room temperature was partially regained [4,7]. The goal of this paper is to study the correlation between the microstructure and the mechanical behavior of different ECAP-processed f.c.c. metals such as pure Al, Ni, Cu and Au. 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. The restoration of ductility at high strains is correlated to the changes in the dislocation structure in the grain boundaries.

### Experimental materials and procedures

High-purity (4N) Al, Au and Ni were processed by ECAP at room temperature. All samples were processed using a 90° ECAP die and following route B<sub>C</sub>, i.e. the sample was rotated along its longitudinal axis by 90° in the same direction after each pass. One pass corresponds to an equivalent strain of ~1. It was shown earlier that in these experiments the microstructural parameters and the yield strength tend to saturation [5]. The Al and Au samples were processed for 4 passes which corresponds to the saturation state [5,8] having grain sizes of about 1200 [9] and 460 nm [8], respectively. The Ni sample did not achieve the saturation state even after 8 ECAP passes at room temperature, therefore a disk with diameter of 10 mm and thickness of ~0.3 mm was cut from the sample processed by 8 ECAP passes and further deformed by high pressure torsion (HPT) under a pressure of 6 GPa for a total of 5 complete revolutions [2]. Additionally, Al – 1 wt.% Mg (Al1Mg) and Al – 3 wt.% Mg (Al3Mg) samples were processed by ECAP for 8 passes following route B<sub>C</sub> which corresponds to the saturation state of these materials [5]. Oxygen-free copper (99.98%) samples were processed in a 90° ECAP die following route B<sub>C</sub> up to 25 passes for studying the changes of yield strength and ductility as a function of number of ECAP passes. The yield strength and the ductility of the SPD-processed specimens were determined by tensile test. Further details of the preparation of the samples and the measurements of the mechanical properties have been given elsewhere [5,8,10]. The microstructure was 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 [11]. From the fitting parameters, the area-weighted mean crystallite size,  $\langle x \rangle_{area}$  and the dislocation density,  $\rho$  were determined [11].

### Results and discussion

Previous studies [3,5] revealed that for ECAP-processed f.c.c. metals, the dislocation density and the yield strength showed similar development in the function of strain, moreover both quantities saturate approximately at the same strain. This suggests that it is worthwhile studying the relationship between the yield strength measured by mechanical tests and the total dislocation density determined by X-ray line profile analysis. The relationship between the dislocation density and yield strength for plastically-deformed metals is generally characterized by the Taylor-equation:

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

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 ( $M = 3$  for untextured polycrystalline materials). Taking the value of  $\alpha = 0.33$ , the saturation values of the yield strength ( $\sigma_{Taylor}$ ) calculated from the maximum dislocation densities using eq. (1) are compared with the strength values ( $\sigma_{measured}$ ) obtained from mechanical tests in Fig. 1a. The size of the symbols represents the error of data. It is apparent that the values of  $\sigma_{Taylor}$  are in relatively good agreement with that of  $\sigma_{measured}$ . This indicates that in f.c.c. metals processed by SPD, the saturation value of the yield strength is basically determined by the interactions between dislocations, the majority of which is stored in the grain boundaries. Careful investigations show, however, that for metals having low stacking fault energy, i.e. for Cu and Au (see Fig. 1a), the value of  $\sigma_{Taylor}$  is somewhat higher than that of  $\sigma_{measured}$ . The difference between the values of  $\sigma_{Taylor}$  and  $\sigma_{measured}$  can be attributed to the fact that the value of  $\alpha$  for these metals may differ from 0.33 selected here. The real value of  $\alpha$  was calculated from eq. (1) substituting  $\sigma_{Taylor}$  by the value of  $\sigma_{measured}$  and modified values of  $\alpha$  are plotted as a function of the stacking fault energy ( $\gamma$ ) in Fig. 1b. It can be seen that the

real value of  $\alpha$  chosen for different metals decreases with decreasing  $\gamma$ . Hernández Olivares and Gil Sevillano [12] have shown that the value of  $\alpha$  increases from about 0.2 to 0.35 when the dislocation clustering increases, i.e. the dislocation structure develops from thick cell walls to sharp boundaries. For metals having low stacking fault energies, the higher degree of dislocation dissociation impedes the formation of sharp boundaries resulting in relatively low value of  $\alpha$ . Therefore, the decrease of  $\alpha$  with decreasing  $\gamma$  shown in Fig. 1b can be regarded as the result of a changing geometrical arrangement of dislocations with stacking fault energy.

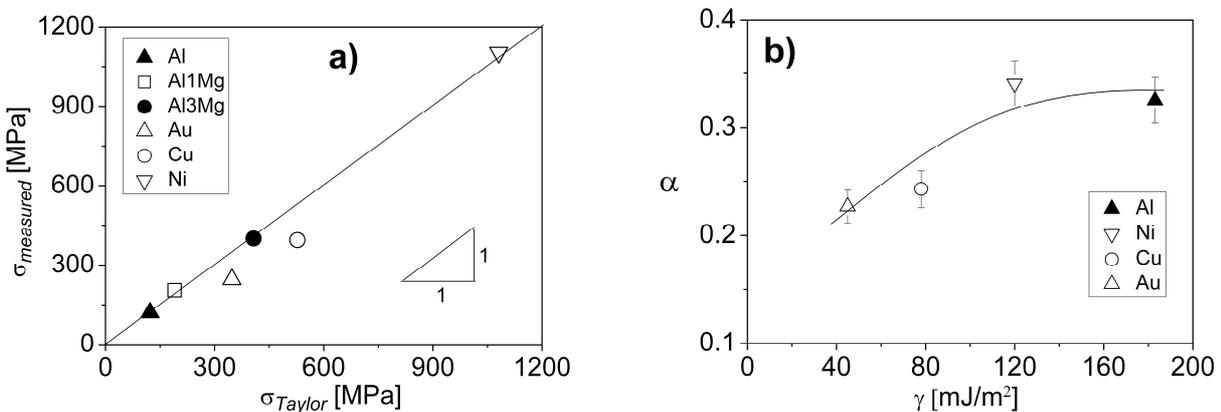


Figure 1: (a) The saturation values of the yield strength obtained from mechanical tests ( $\sigma_{measured}$ ) versus the values determined from the Taylor-equation ( $\sigma_{Taylor}$ ) for pure f.c.c. metals and Al-Mg alloys. (b) The value of  $\alpha$  as a function of the stacking fault energy ( $\gamma$ ) for pure f.c.c. metals.

Figure 2 shows the development of several characteristics of Cu during ECAP. The yield strength and the maximum elongation as a function of the number of ECAP passes for pure Cu are plotted in Fig. 2a and the dislocation density and the crystallite size versus the number of ECAP passes are shown in Fig. 2b. 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 which is accompanied by a decrease of the dislocation density in the boundaries (see Fig. 2b). Moreover, after 25 passes the crystallite size increases slightly. This structural recovery at large strains is accompanied by a decrease in the grain boundary thickness and an increase in the misorientation between the neighboring grains (compare Figs. 3a and b). The larger fraction of high-angle grain boundaries facilitates grain boundary sliding which improves ductility [7]. In spite of the decrease of the dislocation density, the high strength was preserved even after 25 passes which can be explained by the increase of the value of  $\alpha$  from 0.24 to 0.29 (calculated from eq. (1)) corresponding to the thinner grain boundaries.

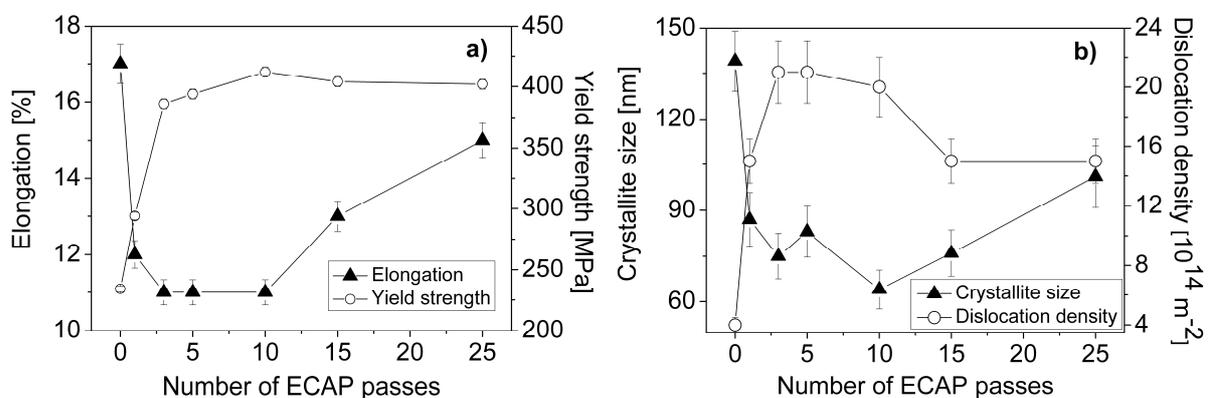


Figure 2: The yield strength and the maximum elongation (a) and also the dislocation density and the crystallite size (b) as a function of the number ECAP passes for pure Cu.

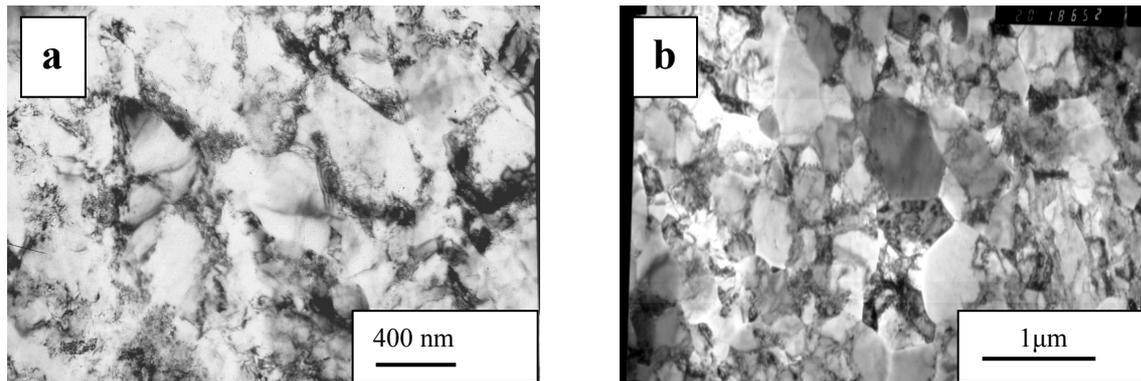


Figure 3: TEM images of the microstructure of Cu processed by ECAP for 5 (a) and 25 (b) passes.

### Summary

The saturation yield strength of f.c.c. metals achieved by severe plastic deformation is correlated to the maximum value of the dislocation density according to the Taylor equation. It is shown that the value of  $\alpha$  in the Taylor relationship decreases with decreasing stacking fault energy as a result of a decrease of clustering of dislocations in the grain boundaries. When pure Cu is processed by ECAP to extremely large strains, the ductility is partially restored due to the transformation of grain boundaries into thinner high-angle boundaries.

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