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Effect of Mg addition on microstructure and mechanical properties of aluminum

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Abstract

The effect of Mg addition on the microstructural evolution and mechanical properties of high-purity aluminum was studied over a wide range of strain, up to ∼8. The high strains were achieved by applying the equal-channel angular pressing technique. The stress–strain relationship was related to the evolution of the microstructure investigated by transmission electron microscopy and X-ray diffraction peak profile analysis. In the early stages of plastic deformation the interaction between the dislocations and the Mg solute atoms results in an increase of the flow stress with temperature. The stable microstructure is developed at higher strains owing to the Mg addition resulting in the saturation of the proof stress at higher strains in Al–Mg alloys.

Keywords: Al–Mg alloy; Equal-channel angular pressing (ECAP); Microstructure; Dislocations

1. Introduction

Severe plastic deformation (SPD) is an effective tool for producing bulk ultrafine-grained (submicron-grain-sized or nanostructured) metals [1]. One of the most common SPD methods is the equal channel angular pressing (ECAP) technique [2,3]. The intense plastic straining during the ECAP procedure provides an opportunity to investigate several properties of metals, such as ductility or hardening, over an exceptionally wide range of strain [3,4].

It is well known that the addition of Mg influences the stacking-fault energy and thus the strength, the recovery and the recrystallization characteristics of Al [5–7]. In addition, the presence of Mg solute atoms may lead to the appearance of plastic instabilities resulting in the well-known serrated yielding or Portevin-Le Châtelier (PLC) effect [8].

In this paper, the effect of a Mg addition on the mechanical properties and on the microstructural evolution of high-purity (4N) aluminum is investigated over a wide strain range. The proof stress and the microstructure of pure Al and Al–Mg alloys are compared after compression testing and ECAP deformation.

2. Experimental materials and procedures

The experiments were conducted on high-purity (4N) Al and an Al–3 wt.% Mg alloy (Al3Mg). Before deformation, Al was annealed for 30 min at 400 °C and the alloy for 1 h at 500 °C. The samples were machined for compression tests in the form of cylinders 5 mm in diameters and 7 mm high. The compressive testing was conducted using an MTS machine operating with constant grip velocity (initial strain rate was $10^{-3}\,s^{-1}$) at 20, 80, 120, 160 and 200 °C. The samples were hold for 10 min at the testing temperature prior to deformation. It should be noted that in this temperature range the Al3Mg specimen remains stable solid solution, even if this period lasts for 2 h, i.e. in these experiments the holding time has no effect on the stress–strain behavior of the alloy. The maximum strain in the compression test was about 0.2 to retain the homogeneous deformation by limiting the bulge of the specimen. Additional Al and Al3Mg samples were deformed at room temperature by ECAP up to 8 passes using a 90° die following route B_C [2,3]. It can be shown that

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an imposed strain of ∼1 is introduced on each passage of the sample through the ECAP die [4]. The 0.2% proof stress of the ECAP deformed specimens was measured at room temperature by compression test. The compression axis was parallel to the output channel axis of the last ECAP pass.

The microstructure of both Al and Al–Mg specimens deformed at room temperature was investigated by X-ray diffraction peak profile analysis. The X-ray diffraction profiles were measured on the cross-section perpendicular to the axis of compression or the output channel of the last ECAP pass. In order to avoid machining effects, a layer of about 100 μm thickness was removed from the specimen surface by chemical etching before the experiments. The X-ray diffraction experiments were performed by a special double-crystal diffractometer (Nonius FR591). The instrumental broadening ($\Delta 2\Theta = 0.006^\circ$) was negligible compared to the measured peak broadening ($\Delta 2\Theta = 0.1$–0.3°) therefore instrumental correction was not performed. The diffractometer was operated at 40 kV and 70 mA with a rotating Cu anode (wavelength $\lambda = 0.15406$ nm). The $K\alpha_2$ peak of the Cu radiation was eliminated by a 0.16 mm slit placed between the source and the Ge monochromator. The profiles were recorded by a linear position-sensitive gas-flow detector (OED 50 Braun, Münich). The peak profiles were evaluated by the Multiple Whole Profile (MWP) fitting procedure described in detail elsewhere [9]. In this method, the Fourier transform of the experimental profiles are fitted by the product of the theoretical Fourier transforms of size and strain peak profiles. The theoretical functions used in the fitting are calculated on the basis of a microstructural model in which the size of spherical crystallites has a log-normal distribution and the lattice strains are caused by dislocations [10,11]. From the fitting parameters, the area-weighted mean crystallite size, $\langle x \rangle_{area}$, and the dislocation density, ρ, can be determined [9]. Selected samples were examined using a JEOL-200CX transmission electron microscope (TEM) operating at 200 kV. The TEM foils were taken from the centre of the cross section perpendicular to the axis of compression or the output channel of the last ECAP pass.

3. Experimental results

3.1. The effect of Mg addition on the mechanical properties

The stress–strain (σ–ε) curves obtained at different temperatures for the annealed pure Al and Al3Mg samples are shown in Fig. 1. It can be seen that for pure Al (Fig. 1(a)) at a given strain, the flow stress decreases with increasing temperature which may be explained by the enhanced thermal activation of dislocations at higher temperatures. By contrast, with the addition of 3 wt.% Mg (Fig. 1(b)) in the early stage of plastic deformation (the strain less than 0.03), the flow stress tends to increase with increasing testing temperature, but this tendency gradually disappears with increasing strain. At least up to $\varepsilon \approx 0.17$, the value of σ obtained at 80 °C is larger than at 20 °C. The corresponding upper strains for 120, 160 and 200 °C are ∼0.13, ∼0.06 and ∼0.03, respectively. The temperature dependence of the 0.2% proof stress, $\sigma_{0.2}$, for Al and Al3Mg samples is shown in Fig. 2 in the conventional $\ln \sigma_{0.2}$–$1/T$ plot and this demonstrates the strengthening effect of the Mg addition at the onset of plastic deformation.

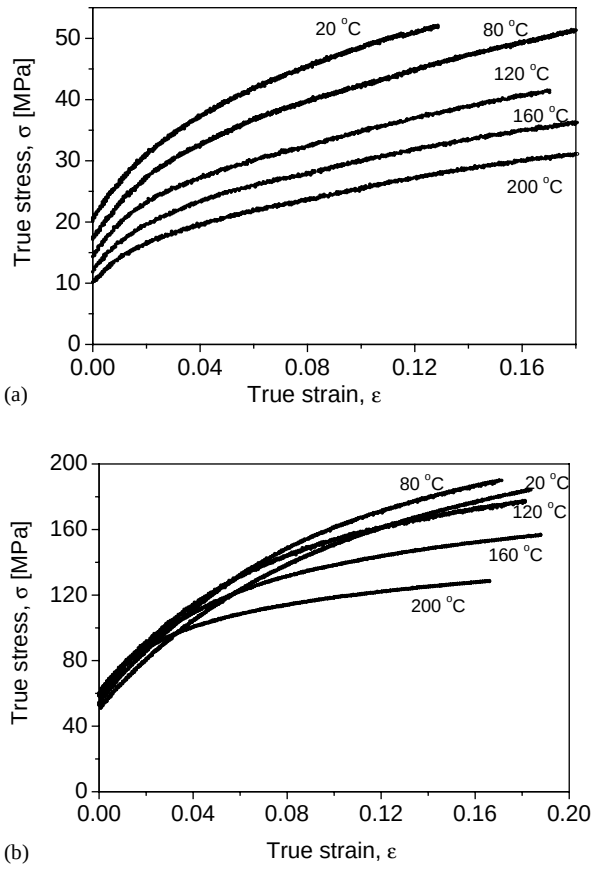

Fig. 1. Stress–strain curves obtained at different temperatures: (a) for pure Al and (b) for Al3Mg.

The proof stress of the Al and Al3Mg specimens at room temperature for some selected strain values are shown in

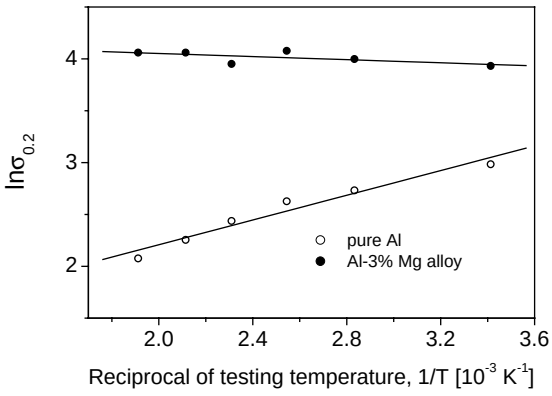

Fig. 2. The temperature dependence of the proof stress, $\sigma_{0.2}$, for Al (a) and for Al3Mg (b).

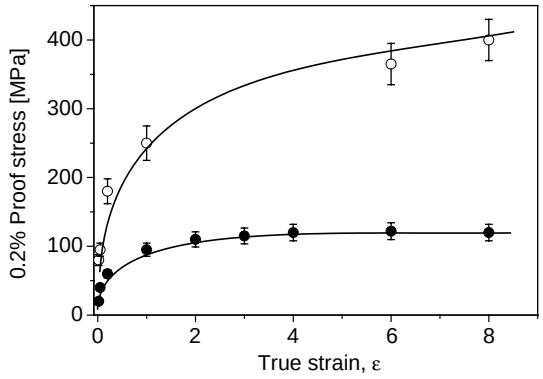

Fig. 3. The proof stress vs. true strain at room temperature for some selected strain values for pure Al (a) and for Al3Mg (b).

Fig. 3. For strains lower than 0.2, the proof stress was determined as the flow stress in the true stress versus true strain plots obtained by compression tests. At a given strain, the addition of 3 wt.% Mg increases the proof stress by a factor between 2 and 4. Furthermore, in the case of pure Al the proof stress saturated at strain $\varepsilon \approx 2$, while for Al3Mg it increased up to $\varepsilon \approx 6$.

3.2. The effect of Mg addition on the deformed microstructures

3.2.1. TEM investigations

Figs. 4 and 5 show the microstructures of Al and Al3Mg samples, respectively, deformed to different strains at room temperature. The TEM micrographs in Figs. 4(a and b) and 5(a and b) were taken in the $\langle 1\,1\,0 \rangle$ direction. These TEM micrographs demonstrate that the addition of Mg strongly influences the evolution of the dislocation structure during deformation. For pure Al, a cellular structure is formed in the very early stages ($\varepsilon < 0.05$) of deformation and this leads to a stable microstructure with an average grain-size of ~1 μm during ECAP. By contrast, in the Al3Mg alloy there is a random non-cellular microstructure even at relatively high strains of $\varepsilon \approx 0.2$. Furthermore, the stable structure established during ECAP in this alloy has a much smaller average grain size of ~0.2 μm. Earlier TEM results [2,3] showed that a stable grain structure is established after ~4 passes in pure Al whereas in the Al3Mg alloy a stable microstructure is reached only after ~8 passes when using route B_C in ECAP [12]. More details of the microstructures achieved by ECAP are given in other reports [2,12].

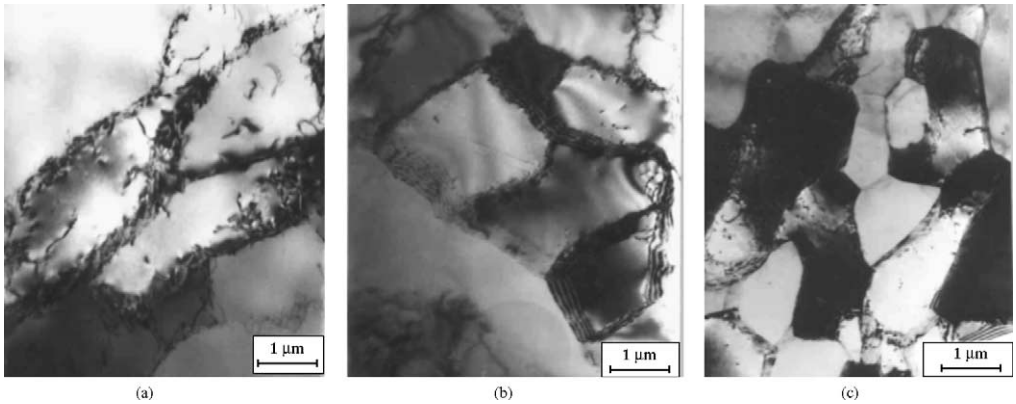

Fig. 4. Microstructure of Al after deformation at room temperature, (a) $\varepsilon \approx 0.05$, (b) $\varepsilon \approx 0.2$ (by compression) and (c) $\varepsilon \approx 8$ (by ECAP).

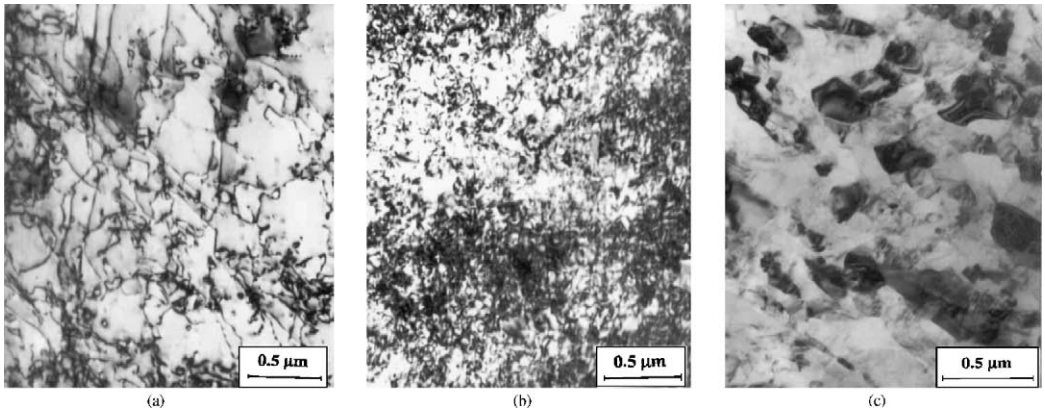

Fig. 5. Microstructure of Al3Mg after deformation at room temperature, (a) $\varepsilon \approx 0.05$, (b) $\varepsilon \approx 0.2$ (by compression) and (c) $\varepsilon \approx 8$ (by ECAP).

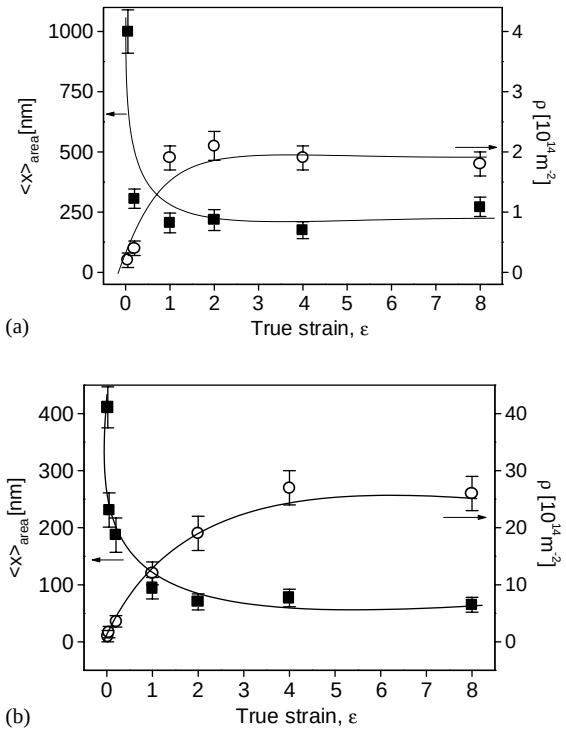

Fig. 6. The area-weighted mean crystallite size, $\langle x \rangle_{area}$ (solid squares) and the dislocation density, ρ (open circles) as a function of strain for Al (a) and Al3Mg (b).

3.2.2. *X-ray peak profile analysis*

Fig. 6 shows the mean crystallite size, $\langle x \rangle_{area}$, and the dislocation density, ρ, as a function of strain for both pure Al and the Al3Mg alloy. The mean crystallite size determined by X-rays is lower than the grain size observed in the TEM images which has been already reported for SPD materials previously [9,13]. The grains in SPD materials are divided into subgrains and/or dislocation cells which are separated from each other by low angle grain boundaries. The crystallite size in SPD metals obtained by X-ray diffraction is equivalent to the mean size of domains which scatter X-rays coherently. Consequently, X-ray diffraction makes a difference between the dislocation cells which are separated from each other by small misorientation, typically under 1–2°. At the same time it was found previously [9,13] that these dislocation cells can only be observed separately by electron microscopy if highly magnified TEM images are studied very carefully. The usual TEM investigation of SPD metals gives the grain size which is higher than the dislocation cell size obtained by X-ray diffraction peak profile analysis. To obtain a detailed image of the microstructure of SPD materials, the simultaneous application of X-ray diffraction and TEM is necessary. Fig. 6 shows that for both Al and Al3Mg the mean crystallite size decreases, while the dislocation density increases with increasing strain. It can be seen clearly that the addition of 3 wt.% Mg influences strongly the values of these parameters at a given strain. For example, at $\varepsilon = 8$ the crystallite size decreases and the dislocation density increases by factors of 4 and 14, respectively, when 3 wt.% Mg is added to Al. Furthermore, while in the case of pure Al the dislocation density saturated relatively quickly after a true strain of $\varepsilon = 1$, in the Al3Mg alloy the dislocation density increased up to a much higher strain, $\varepsilon = 4$.

4. Discussion

The results obtained from this investigation show that the addition of Mg to the aluminum matrix has significant effects on the mechanical properties and microstructural evolution over a wide range of strain. It is well established that the interaction between dislocations and solute atoms in Al–Mg alloys leads to solid solution strengthening by reducing the dislocation mobility. The results shown in Figs. 1(b) and 2 lead to the conclusion that, in the temperature range between 20 and 200 °C at the beginning of plastic deformation, the strengthening due to dislocation–solute interaction is dominant relative to the softening effect of thermal activation. By increasing the testing temperature, the diffusivity of the Mg solute to mobile dislocations, and thus the retarding influence of the solute atoms, is enhanced. This explains the slight increase of the flow stress (including also $\sigma_{0.2}$) at a given small strain with increasing temperature despite the softening effect of thermal activation. At higher strains, however, corresponding to higher dislocation densities, the recovery effect of thermal activation becomes stronger so that the flow stress of the Al–Mg alloy, as in pure Al, decreases with increasing temperature at a given strain.

Results of both mechanical testing and microstructural investigations of samples deformed at room temperature (Figs. 3–6) show clearly a lower recovery rate even at high strains in the Al3Mg alloy, where the saturated stress, dislocation density, and the stable microstructure are established at much higher strains than in pure Al. As a consequence of a lower recovery rate, the addition of Mg strongly increases the dislocation density (see Fig. 6) and accordingly increases the proof stress over a wide range of strain (see Fig. 3). Concerning the strengthening mechanisms, the Mg solute atoms may increase the strength of Al in two ways: (i) directly by pinning the dislocations and impeding their motion (solute–dislocation interaction) and (ii) indirectly by hindering the annihilation of dislocations during deformation, leading to an increase in the dislocation density (dislocation–dislocation interaction). In the initial stages of plastic deformation, where the dislocation density is relatively low, the solute–dislocation interaction is unambiguously the main strengthening process, as was discussed above.

It was shown previously [14,15] that, at a given temperature in the range between 20 and 200 °C, the stress–strain relationship of pure Al can be well described by a new em-

pirical constitutive equation over a wide range of strain (up to ∼8). The relevance of this new relationship has also been supported by numerical calculations taking into account the main dislocation mechanisms of plastic deformation [14]. In the case of Al3Mg alloy, however, the curve fitted to the measured data at low and medium strains deviates from the experimental stress values at high strains ($\varepsilon > 1$). It can be supposed that at high strains, because of the extra high dislocation density, the character of dislocation–dislocation interactions may change by the effect of a solute–dislocation interaction leading to a failure of the new constitutive relationship when extended to high strains for Al3Mg alloy.

5. Summary

The strength and the microstructure developed during deformation in Al are strongly influenced by the interaction between the Mg solute atoms and the dislocations over a wide range of strain. At the beginning of plastic deformation, the solute–dislocation interaction is the main strengthening process, resulting in the increase of the flow stress with increasing temperature. Mechanical testing and microstructural investigations reveal that the Mg addition has a strong influence on the recovery properties of Al. As a consequence of lower recovery, the addition of Mg strongly increases the dislocation density, leading to an increase of the proof stress over a wide range of strain. It is found that in both Al and Al3Mg the dislocation density tends to saturation and a stable microstructure is formed at high strains. The Mg addition, however, increases the strain corresponding to the formation of a stable microstructure and the saturated strength value.

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