DISLOCATION SUBSTRUCTURES OF ALUMINIUM-MAGNESIUM ALLOYS DURING THERMOMECHANICAL PROCESSING

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ABSTRACT: The dislocation substructures during thermomechanical processing have been investigated using four high purity aluminium-magnesium alloys, Al-0.13%Mg, Al-1%Mg, Al-3%Mg and Al-5%Mg. The experimental materials were deformed in a plane strain compression testing machine at 385°C and constant strain rates of 2.5/s and 25/s to strains of 1 and 1.2. A decreasing strain-rate from 25/s to 2.5/s was also applied. Transmission Electron Microscopy (TEM) was used to investigate internal dislocation density (ρ_i), subgrain size (δ) and misorientation across subgrain boundaries (θ). Experimental results show that with increasing magnesium content, internal dislocation density and misorientation across subgrain boundaries increase while subgrain size decreases. A relationship of $\rho_i^{1/2}\delta = c_2$ with $c_2 = 5$ for Al-0.13%Mg and 10 for the other three alloys has been obtained from the present results.

Keywords: *high purity Al-Mg alloys, internal dislocation density, subgrain size, misorientation across subgrain boundaries, TEM*

1. INTRODUCTION

The dislocation substructures are the key features of the fine scale internal state during hot deformation, which determine hot strength and subsequent recrystallisation behaviour. Dislocation substructures can be described by three internal state variables, *i.e.* internal dislocation density (ρ_i), subgrain size (δ) and misorientation across subgrain boundaries (θ). These three variables are the essential parameters for physically based modelling of hot deformation and subsequent recrystallisation behaviour [1], which is now the most powerful tool to understand materials behaviour during hot deformation and to aid control of thermo-mechanical processing. The internal state variables have been investigated extensively for many years, but there is still much work required for a complete understanding of their evolution, especially during thermo-mechanical processing. The present work is to investigate dislocation substructures of some high purity aluminium-magnesium alloys during hot deformation at constant and changing strain rate.

High purity aluminium-magnesium alloys with several magnesium contents were used in the present research, as aluminium alloys with different content of magnesium are the most important matrix materials of a commercial series of aluminium alloys. Plane strain compression testing has been used to simulate the industrial rolling process. The microstructural evolution of the alloys is discussed in relation to previously obtained results on a commercial purity Al-1%Mg alloy.

2. EXPERIMENTAL

Experimental materials supplied by Alcan International Limited are high purity Al-0.13%Mg, Al-1%Mg, Al-3%Mg and Al-5%Mg. The materials were deformed at 385°C and at constant equivalent strain rates of 2.5/s and 25/s in a plane strain compression testing machine. A decreasing strain-rate deformation from 25/s to 2.5/s at strain of 1 was also carried out in order to simulate condition in industrial thermo-mechanical processing. Details of experimental alloys and mechanical tests have been given in a parallel paper [2].

The specimens for TEM-observations to investigate deformed structures were produced by electro-thinning in a solution of 25% nitric acid+75% methanol at -30°C and 10-15 volts. All three internal state variables were determined from micrographs taken by TEM. Internal dislocation density (ρ_i), mean subgrain size (δ) and misorientation across subgrain boundaries (θ) were determined using surface intersection point counting, the linear intercept method with parallel test lines, and Kikuchi patterns obtained in TEM, respectively. Each average value given in the paper is the mean from three to five different areas in each of three TEM foils.

3. RESULTS AND DISCUSSION

3.1 Effect of magnesium content

The dislocation substructures in specimens of the four Al-Mg alloys deformed at 385°C and 2.5/s to strain of 1 are shown in fig.1. In all specimens, subgrain structures have been well established. In lower magnesium alloys, equiaxed subgrains are homogeneously distributed, while in higher magnesium alloys equiaxed subgrains remain within higher contrast bands that are so-called microbands [3]. The misorientation across microband boundaries is higher than that across equiaxed subgrain boundaries. The frequency (or density) of microbands appearing in the TEM increases with increasing magnesium content. Fig.2 shows quantitative results of internal dislocation density (ρ_i), subgrain size (δ) and misorientation across subgrain (and microband) boundaries (θ) determined from TEM micrographs such as those shown in figs.1. With increasing magnesium content, internal dislocation density increases, while subgrain size decreases. Overall, misorientation increases slightly with increasing magnesium content, but the trend is within the confidence limits of measurements.

Flow stress is related to internal dislocation density, subgrain size and friction stress due to solution hardening (σ_f) as

$$\sigma = \alpha MGb\rho_i^{1/2} + \alpha' MGb/\delta + \sigma_f \tag{1}$$

where α and α' are constants, M ($\approx$3) is the Taylor factor, G ($\approx$20.5 GPa at T=385°C) is the shear modulus, b (=0.286 nm) is the value of Burgers vector. Assuming $\sigma_f \approx$25MPa for Al-1%Mg and that the internal dislocation term is larger than the subgrain size term in equation (1) by a factor of 3 [4], α = 0.26 and α' = 0.83. The value of α = 0.26 is larger than that expected in a commercial Al-1%Mg alloy (α = 0.18) [6].

If stress is related only to internal dislocation density, the following equation can be written

$$\sigma = c_1 Gb\rho_i^{1/2} \tag{2}$$

where c_1 is a materials constant. The constant c_1 is approximately 3.6 for Al-0.13%Mg and 2 for the other three alloys. The value of c_1 for the alloys of ≥1%Mg is consistent with those obtained from tension, compression and torsion tests in different Al-Mg alloys [5]. However, c_1 is expected to be lower in pure aluminium than in Al-Mg alloy [5]. The higher present value of c_1, *i.e.* lower value of internal dislocation density, in Al-0.13%Mg may arise from annihilation by interaction between dislocations of opposite signs and/or accumulation of dislocations into subgrain boundaries during the period between the end of deformation and quenching. Assuming that the low measured internal dislocation density in Al-0.13%Mg only arises from transfer to subgrain boundaries, the expected increase in misorientation of tilted subgrain boundaries would be about 0.2° calculated in terms of the following equation

$$\rho_b = 2\theta / b\delta \tag{3}$$

where ρ_b is the dislocation density of subgrain boundaries. This increase is consistent with the high value of misorientation for Al-0.13%Mg in figure 2 (although within the confidence interval). It also leads to the implication that the total stored energy only decreases slightly due to re-arrangement of dislocation into subgrain boundaries, which gives a relative higher growth rate of recrystallisation nuclei [2].

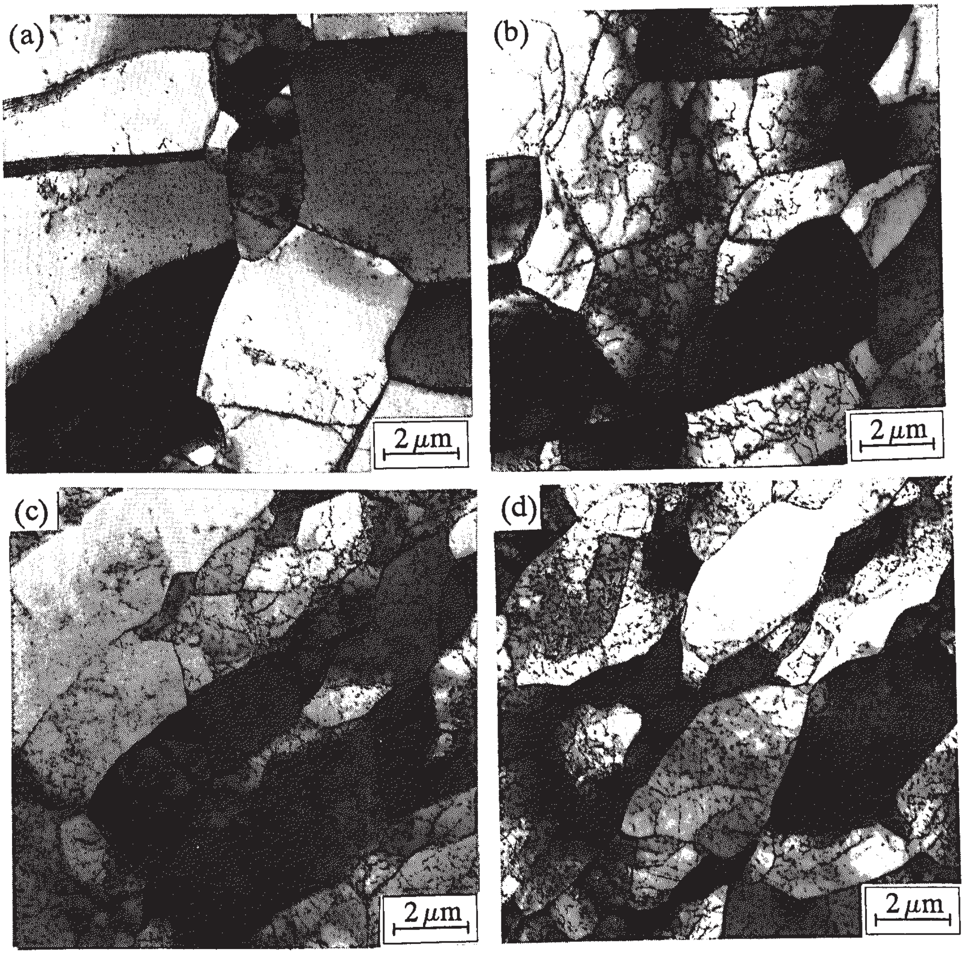

Figure 1 TEM micrographs taken from the specimens deformed at 385°C and 2.5/s to strain of 1 of the alloys (a) Al-0.13%Mg, (b) Al-1%Mg, (c) Al-3%Mg and (d) Al-5%Mg.

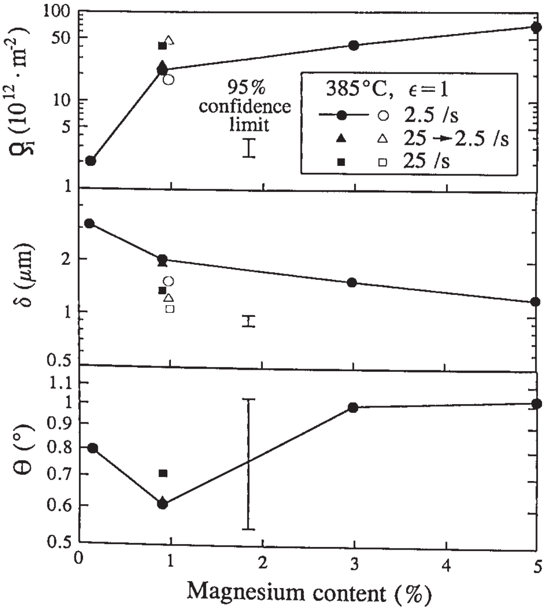

Figure 2 internal dislocation density (ρ_i), subgrain size (δ) and misorientation across subgrain boundaries (θ) determined from TEM micrographs. (solid symbols for high purity alloys and open symbols for commercial purity alloy)

3.2 Effect of deformation conditions

The effect of deformation conditions on dislocation substructures were investigated in the Al-1%Mg specimens deformed at 385°C and at constant strain-rates of 2.5/s and 25/s to strains of 1 and 1.2, as well as in the specimens deformed at decreasing strain-rate from 25/s to 2.5/s at a strain of 1 to strains of 1 and 1.2. Fig.3 shows TEM results. Equiaxed subgrains appear in all specimens, but their mean size decreases while internal dislocation density and misorientation across subgrain boundaries increase with increasing strain-rate (also see fig.2). The same dislocation structures are observed in the specimens deformed at constant strain rate of 2.5/s and decreasing strain-rate from 25/s to 2.5/s at a strain of 1 for the two different deformation strains, indicating no transient behaviour of dislocation substructures appearing under the changing deformation conditions. Some previous data obtained on a commercial Al-1%Mg alloy deformed under the same conditions are also given in fig.2. Comparing the values for high purity Al-1%Mg and commercial Al-1%Mg shows that internal dislocation density is nearly the same in both alloys, but subgrain size in high purity alloy is larger for constant strain rate deformation. The smaller subgrain size in commercial Al-1%Mg indicates that particles $Al_6(MnFe)$ [7,8] or Mn in solution hinder migration of subgrain boundaries, *i.e.* growth of subgrains. The mean misorientation across subgrain boundaries in the commercial alloy, which is about 4° [8,9], is much higher than in the high purity alloy, which, as mentioned above, arises from different microband behaviour. A transient behaviour of dislocation substructures, *i.e.* different internal state variables between constant strain-rate and decreasing strain-rate at a strain of 1, where the change in strain-rate is just finished, has been observed in the commercial purity Al-1%Mg alloy [8,9]. This indicates that impurities in Al-1%Mg alloys have a significant effect on microband behaviour, which in turn influence recrystallisation behaviour significantly [1,10]. In addition, the internal dislocation densities for the high purity specimens deformed at constant strain rate of 2.5/s and decreasing strain rate to a strain of 1.2 are $2.9 \cdot 10^{13}$ m^{-2} and $3.0 \cdot 10^{13}$ m^{-2}, which are similar to those to strain of 1 (fig.2). The subgrain sizes are 2.06 μm and 2.02 μm, which are also nearly the same as those at a strain of 1. These results indicate that at a strain of 1 internal dislocation density and subgrain size have reached steady state.

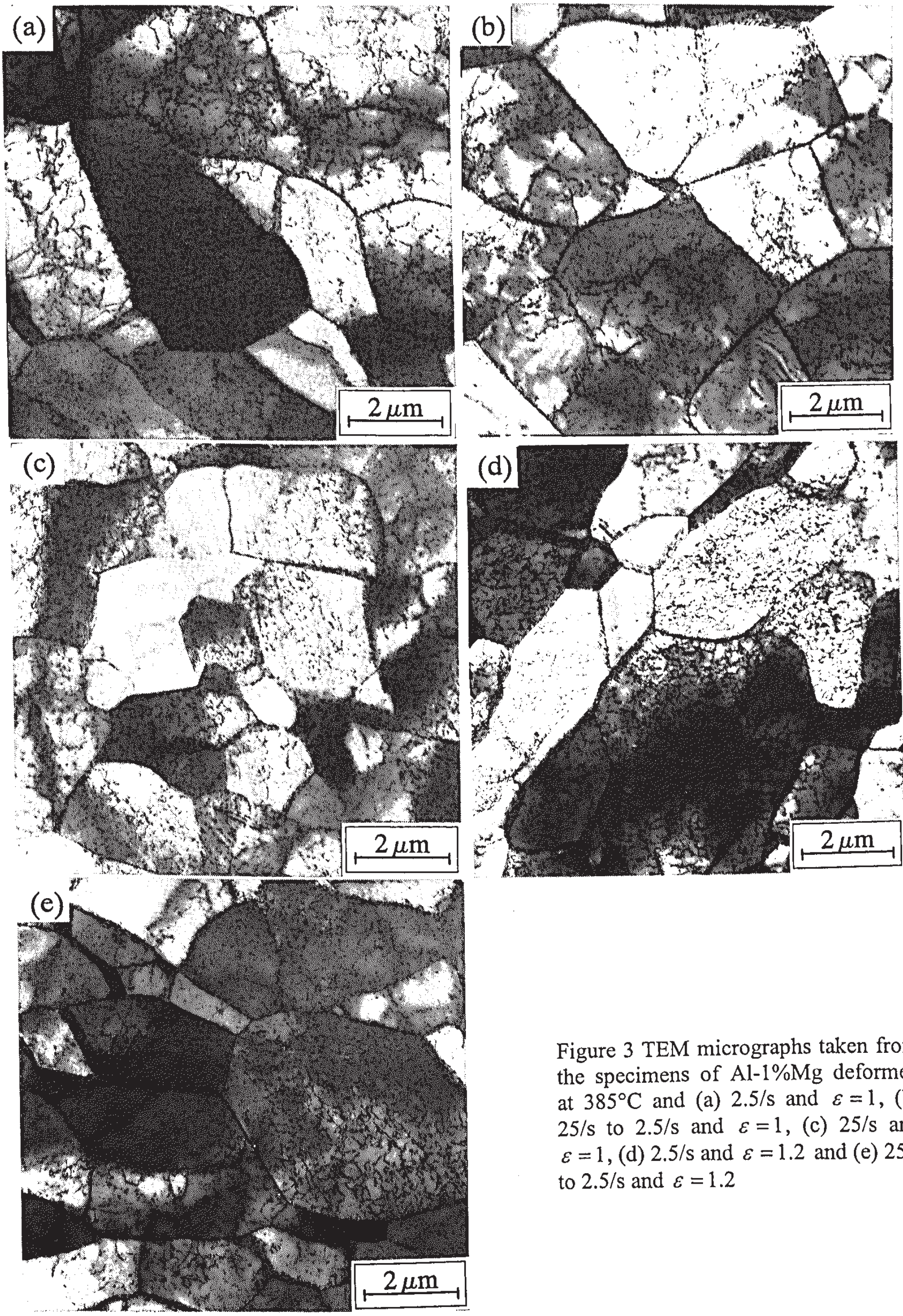

Figure 3 TEM micrographs taken from the specimens of Al-1%Mg deformed at 385°C and (a) 2.5/s and $\varepsilon = 1$, (b) 25/s to 2.5/s and $\varepsilon = 1$, (c) 25/s and $\varepsilon = 1$, (d) 2.5/s and $\varepsilon = 1.2$ and (e) 25/s to 2.5/s and $\varepsilon = 1.2$

3.3 Relationship between internal state variables

For steady state, there exists a relationship between internal dislocation density and subgrain size [1,4], and the following equation can be written,

$$\rho_i^{1/2}\delta = c_2 \qquad (3)$$

where c_2 is a constant (between 7 and 30 [1,4]). From the present results, c_2 is approximately 5 for Al-0.13%Mg and 10 for the other three alloys. As discussed in the last section, the smaller value for Al-0.13% may arise from higher recovery rate which allows some static recovery during the period between the end of deformation and quenching. Using the same correction described in the last section to eliminate the effect of static recovery, $c_2 \approx 10$, which is the same as that for the other alloys. The value of c_2 is slightly smaller than those obtained from tension, compression and torsion tests at lower strain-rates, but larger than previous results obtained on a commercial Al-1%Mg alloy deformed in the plane strain compression testing machine under the same conditions, which arise, as discussed above, from the effect of particles or impurities on migration of subgrain boundaries.

4. CONCLUSIONS

1. Internal dislocation density increases, while subgrain size decreases, with increasing magnesium content and increasing strain rate. Misorientation between subgrains increases slightly with increasing magnesium content and strain rate.
2. Internal dislocation densities in commercial and high purity Al-1%Mg alloys are nearly the same, but subgrain size is lower and especially misorientation across subgrain boundaries is much higher in commercial Al-1%Mg than in high purity Al-1%Mg alloy.
3. A relationship between internal dislocation density and subgrain size ($\rho_i^{1/2}\delta = c_2$) is valid with $c_2 = 5$ for the high purity Al-0.13%Mg and 10 for the other alloys. The low value for Al-0.13%Mg is considered to arise from recovery between the end of deformation and quenching.

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