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THE MICROSTRUCTURE AND MICROTTEXTURE OF PURE ALUMINIUM DURING RECOVERY

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Abstract

The evolution of microstructure and microtexture during the recovery of high purity aluminium has been investigated. Material with a large starting grain size was deformed to a reduction of 20% by cold rolling and samples were subsequently isothermally annealed at temperatures of 275°, 300° and 325°C. The size, shape and misorientations of the subgrains were investigated using backscattered electron microscopy and electron backscattered patterns in a JEOL 6300 SEM. It was found that although individual misorientations between subgrains were in the range 2-5°, the orientation spread in the central region of a grain was less than 10° and there was little evidence of any significant overall orientation gradient. During annealing, the misorientation spread and the mean misorientation decreased slightly. The results are discussed in terms of a model in which local misorientations decrease during annealing. At later stages of annealing, some recrystallization occurred and from a knowledge of the substructure it was possible to determine the relative mobilities of the subgrain boundaries and the recrystallizing grain boundaries.

Introduction

Many measurements of the change of subgrain size (D) with time (t) at constant temperature reveal kinetics of the form

$$D^n - D_0^n = kt \quad (1)$$

where n is a constant, k is a temperature dependent rate constant and D_0 is the subgrain size at $t=0$.

In many cases the value of the exponent n in equation 1 is found to be 2 (parabolic

growth), which is of a similar form to the predicted kinetics for normal grain growth. Work in which an exponent of 2 has been reported includes Smith and Dillamore (1970) on high purity iron, Sandstrom et al (1978) on 4N aluminium and Al-1%Mn, Varma and Willets (1984) and Varma et al (1988) on 4N aluminium, and Varma (1986) on Al-0.2%Mg.

However, there are several other investigations of subgrain growth in aluminium in which the subgrain growth rate decreases much more rapidly than implied by equation 1. For example Furu and Nes (1992) reported an exponent of ~ 4 in commercial purity (99.5%) aluminium. Other investigations of high purity ($> 99.995\%$) aluminium (Beck et al 1959 and Sandstrom et al 1987) have found growth kinetics which are more consistent with a logarithmic relationship of the form

$$\log D = k t \quad (2)$$

The number of experimental investigations of subgrain growth kinetics is rather small. In addition to this there is often considerable scatter of the data. It should also be noted that the amount of subgrain growth which is actually measured is usually very small. In the investigations quoted above, the ratio D/D_0 is rarely greater than ~ 2 . The combination of scatter and limited data range makes it very difficult to place much confidence in the quoted values of the exponent n , or indeed to determine whether the data fit equation 1 with a very high exponent n or whether they fit equation 2.

Experimental methods

Aluminium of 99.998% purity was cold rolled and annealed to produce a large ($600\mu\text{m}$) grain size. The material was then cold rolled 25% and carefully cut into specimens of dimension $\sim 2 \times 2 \times 2 \text{cm}$. Specimens were annealed at times of between 1 minute and 12 hours at temperatures of 250, 300 and 325°C . Anneals of less than 1hr were carried out in a salt bath and longer anneals were carried out in a muffle furnace.

The long transverse sections of the specimens were metallographically ground and mechanically polished prior to a final electropolish. The specimens were examined in a JEOL 6300 SEM using backscattered electrons, and grain and subgrain orientations were measured in the microscope using electron backscattered patterns. The semi-automated method of analysis involved measurement on the diffraction pattern of the position of three poles. Most of the orientation measurements were, as discussed below, all made on a single grain, and in order to minimise error in the orientation determination, the same three poles were always used. It is estimated that using this procedure, the relative orientations of the subgrains could be determined to an accuracy of $< 0.3^\circ$.

Subgrain growth kinetics

In order to avoid regions of inhomogeneous deformation near grain boundaries, only the central regions of the grains were analyzed. The subgrains were generally equiaxed, and

some alignment was detected. A typical microstructure is shown in figure 1.

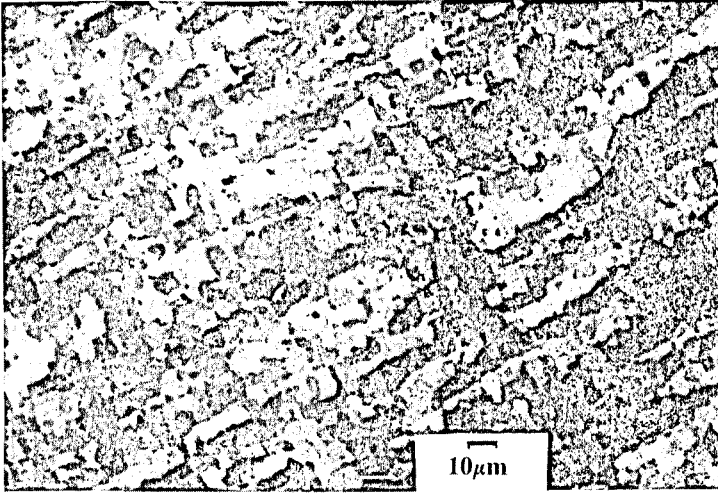


Figure 1. Backscattered electron micrograph of subgrains after 8hrs at 300°C

For the specimens deformed at 250°C and 325°C, measurements of subgrain size were made of ~100 subgrains in each of ~5 grains. Measurements were taken in two orthogonal directions in order to minimise the effects of any shape anisotropy. The orientations of adjacent subgrains were also determined.

For the specimen annealed at 300°C, all measurements were made in the central region of a single large grain in one specimen. After examination, the specimen was given a further anneal, lightly repolished and the original grain was then identified and re-examined in the SEM. This procedure was adopted in order to minimise any systematic errors or scatter which might arise from examining grains of different orientation. Measurements of subgrain size in two orthogonal directions were made and subgrain orientations were determined by five line scans each of approximately 25 subgrain diameters in two orthogonal directions.

The subgrain growth kinetics at the three temperatures are shown in figure 2. Although there is in all cases an initially rapid increase in subgrain size, the rate of subgrain growth drops rapidly, and analysis of the data suggests a reasonable fit to equation 1 with $n \sim 4.5$.

Theory suggests that if the subgrain boundary energy remains constant, then the rate of growth of a subgrain of radius R in an array of boundary energy γ_s is given by

$$dR/dt = V_s = \alpha M_s \gamma_s / R \quad (3)$$

where M_s is the boundary mobility and α is a constant of the order of unity.

The low angle boundary mobility may be determined from this relationship if we measure

the growth rate (dR/dt) at a particular value of subgrain size (R), and if the low angle boundary energy (γ) is also determined. This was done by measuring dR/dt in the later stages of growth when the growth rates are essentially linear. The boundary energies at the points of measurement were determined from the mean subgrain misorientations such as given in figure 4, using the Read-Shockley relationship between boundary energy and misorientation, and hence M_s was determined at each temperature. A plot of $\ln(M_s)$ against $1/T$ was, as shown in figure 3, approximately a straight line, the slope of which gave an activation energy for subgrain growth of $\sim 130\text{kJ/mol}$, which is reasonably close to that for self diffusion in aluminium.

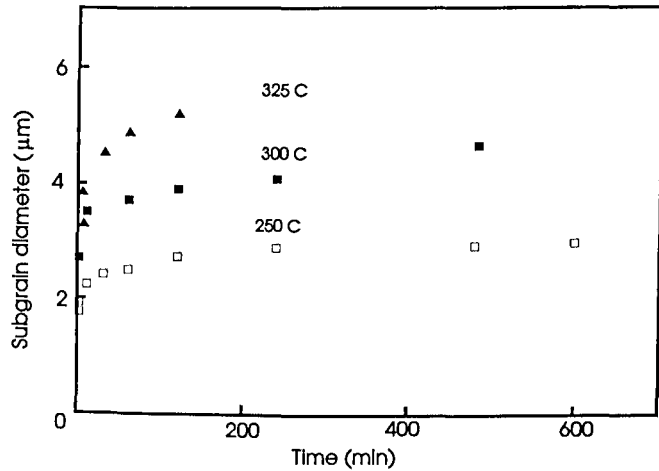


Figure 2. Subgrain growth kinetics

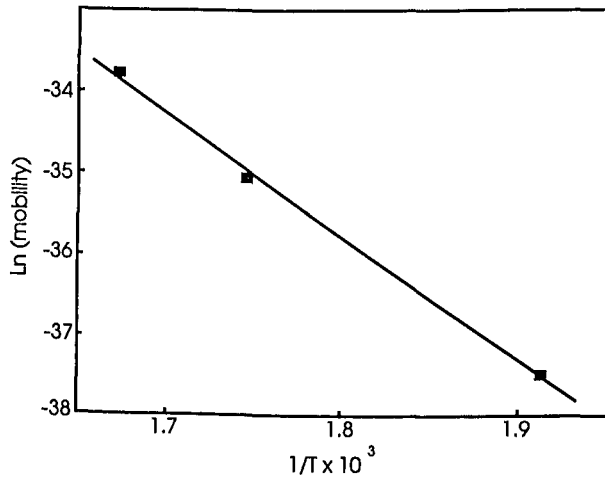


Figure 3. The temperature dependence of subgrain boundary mobility.

Subgrain misorientation

The subgrain misorientations were measured, and figure 4 shows both the mean misorientation as defined by the angle/axis pairs of adjacent subgrains, and the orientation spread, which was measured as the maximum misorientation between all the subgrains measured. As the measurements were for linear scans of 25 subgrains, the scan length was $\sim 70\text{-}100\mu\text{m}$ and from figure 4, it is seen that the initial overall orientation gradient is small, and of the order of $10^{-1} \text{ }^\circ\mu\text{m}^{-1}$. This is much lower than found in more highly deformed aluminium (Orsund et al 1989). The subgrain misorientations are also considerably lower than found for more highly strained aluminium.

From figure 4, it may be seen that there is a small decrease in both the orientation spread and the mean subgrain misorientation during subgrain growth. The change in misorientation is, like the subgrain growth rate, most rapid during the early stages of the anneal.

Furu and Nes (1992) have measured the changes of both subgrain size and misorientation in highly deformed commercially pure aluminium. They found that as subgrain growth proceeded, the average subgrain misorientation increased. Their result is consistent with there being **orientation gradients** in their specimens, and the presence of these was confirmed experimentally.

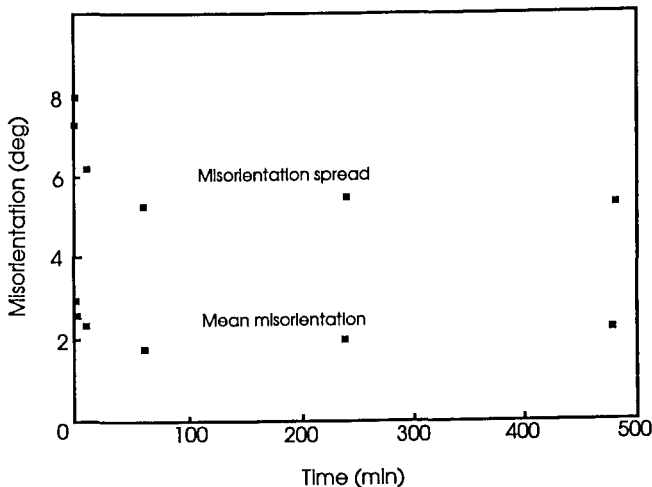


Figure 4. The mean misorientation of adjacent subgrains and the orientation spread for the specimen annealed at 300°C .

It has been argued (Humphreys 1992) that in a microstructure with a distribution of boundary energies and no overall orientation gradient, then boundary migration should lead to a decrease in the total area of high energy boundary. This behaviour has been observed in Fe-Si polycrystals of initially random orientation (Watanabe et al 1989), and has been shown to occur in both Monte-Carlo (Grest et al 1985) and network computer

simulations (Humphreys 1992), the latter simulations showing a correlation with the subgrain growth kinetics. We therefore interpret the experimental results above, in terms of an energy driven reduction in boundary energy, which, for low angle boundaries means lower misorientation. It is tentatively concluded that the kinetics of subgrain growth are affected by the change in boundary character which may result in changes of both energy and mobility, although a quantitative correlation is not yet possible.

In order to illustrate semi-quantitatively, the complexities of the problem we have run a vertex computer simulation (Humphreys 1992) with an initial microstructure containing five generic types of grain boundary:

1. Random high angle boundaries of constant energy and mobility.
2. Low energy boundaries with low mobility (typified by LAGBs or $\Sigma 3$ twins)
3. Low energy boundaries with high mobility (typified by low Σ boundaries in pure metals)
4. High energy boundaries with low mobility
5. High energy boundaries with high mobility

High or low energies or mobilities were given values of 5 or 1/5 respectively times those of the random boundaries. It was found that the distribution of boundary types changed during grain growth as shown in figure 5.

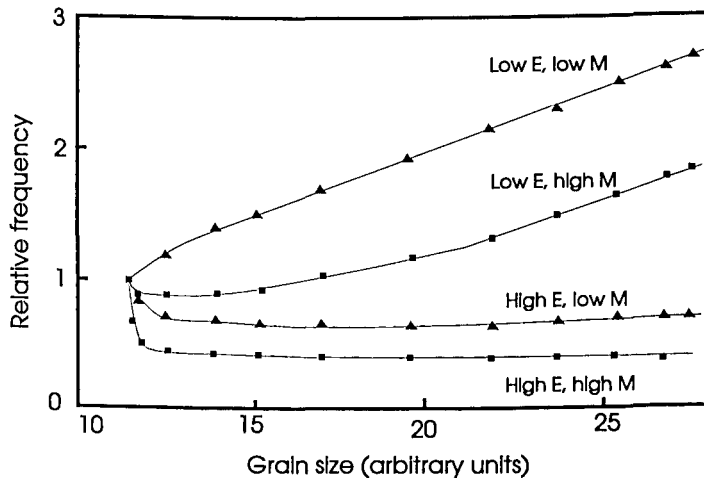


Figure 5. Computer simulation of the change in relative frequency of boundary type during grain growth. The frequencies of boundaries with high or low energy (E) or mobility (M) are measured relative to those for "random" boundaries (type 1).

It is seen that there is a tendency for the proportion of low energy boundaries to increase and for high energy boundaries to decrease during grain growth. However, the boundary mobility plays a significant role, and it is interesting that the largest increase is predicted for low energy/low mobility boundaries such as LAGBs and $\Sigma 3$ twin boundaries, which

is in accord with the experimental results of figure.

Mobilities of low and high angle boundaries

It is of interest to compare the mobilities of the low angle boundaries determined above, with the mobility of high angle boundaries, as information on the mobilities of low and high angle boundaries in deformed metals are poorly known. We have carried out some preliminary studies as follows on the specimen annealed at 300°C. During the later stages of subgrain growth experiments described above, some recrystallization occurred in the large grain in which subgrain growth was being measured, and the approximate velocity (V_b) of the high angle boundaries in this grain was measured. The driving force for recrystallization in the specimen is provided by the subgrains of radius R and energy γ_s , and is given approximately by $3\gamma_s/2R$, and hence the velocity of a high angle boundary of mobility M_b is given by

$$V_b = 3 M_b \gamma_s / 2R \quad (4)$$

and using equations 3 and 4 the relative boundary mobilities are given as

$$M_b/M_s = \beta V_b/V_s \quad (5)$$

where $\beta = 2\alpha/3$.

If, as an approximation we take $\beta=1$, then we see that the relative boundary mobilities are given by the ratio of the high angle and low angle boundaries in the same recovered grain. Our measurements of the boundary velocities at 300°C, gave a value of $M_b/M_s \sim 25$. Although this is a single measurement and too much reliance should not be placed on this preliminary result, this type of approach is in principle capable of yielding useful information about boundary mobilities under conditions of recrystallization now that better methods of quantitatively characterising subgrain structures are available.

Conclusions

1. Simultaneous measurement of subgrain growth and misorientation is necessary in order to interpret subgrain growth kinetics.
2. The present work confirms earlier investigations in aluminium which determined a subgrain growth exponent considerably greater than 2.
3. There is some experimental evidence that in the absence of a misorientation gradient, there is a decrease in subgrain misorientation during growth, and this is in general agreement with computer simulations of subgrain growth.
4. The mobilities of low angle boundaries were found to be approximately 1/25 that of high angle boundaries at 300°C

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