RECOVERY AND DIFFUSION ON GRAIN BOUNDARIES IN ALUMINIUM
J. Kwieciski, J. Wyrzykowski

To cite this version:
J. Kwieciski, J. Wyrzykowski. RECOVERY AND DIFFUSION ON GRAIN BOUNDARIES IN ALUMINIUM. Journal de Physique Colloques, 1990, 51 (C1), pp.C1-557-C1-562. <10.1051/jphyscol:1990187>. <jpa-00230355>

HAL Id: jpa-00230355
https://hal.archives-ouvertes.fr/jpa-00230355
Submitted on 1 Jan 1990

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L’archive ouverte pluridisciplinaire HAL, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d’enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.
RECOVERY AND DIFFUSION ON GRAIN BOUNDARIES IN ALUMINIUM

J. Kwieciński and J.W. Wyrżykowski

Institute of Materials Science and Engineering, Warsaw University of Technology, Narbutta 85, 02-524 Warsaw, Poland

Abstract - Investigations of recovery and diffusion on Grain Boundaries (GB) in polycrystalline aluminium at low temperatures were performed using the dislocation spreading method. The kinetics of isothermal GB recovery was determined. The results obtained show that the logarithmic law of recovery obtains, which is characteristics of recovery of mechanical properties. The distribution functions of GB diffusivity were determined and they served as a basis for calculating the parameters of GB self-diffusion: preexponential factor and activation energy. It was found that GB self-diffusion is the rate controlling factor of the recovery process. The effect of thermo-mechanical treatment on both the kinetics of GB recovery and the distributions of GB diffusion parameters was presented. The distributions of GB diffusion parameters can be used as GB characteristics of polycrystal.

1. INTRODUCTION

Recovery of the microstructure of polycrystals, apart from intra-grain mechanisms, can also take place by means of annihilation of point defects [1] and dislocations [2] on Grain Boundaries (GBs), as well as by means of ordering of GB structure [3]. Annihilation in GBs which leads to the decrease of the total density of defects is a thermally activated mechanism controlled by grain boundary diffusion.

The method that was employed in the present work consisted in studying GB dislocation spreading. It allows to determine the kinetics of recovery on GBs in polycrystals [2] and to study GB diffusion [4,5]. If a sliding lattice dislocation encounters a GB, then their interaction results in a formation of the so-called Trapped Lattice Dislocation (TLD). At elevated temperatures (which in the case of aluminium is also room temperature, as it exceeds 0.3 Tm) TLDs undergo dissociation into Extrinsic Grain Boundary Dislocations (EGBDs) and as a result the stress fields of TLDs are relaxed with the kinetics dependent on temperature and time [4-9]. The process during which the dissociation products spread out along GB is usually called EGBD spreading. Because EGBD spreading is a mechanism by means of which GBs can act as microstructural sinks for lattice dislocations, hence, spreading is
one of the mechanisms of recovery. Spreading of dislocations in GBs manifests itself by an increase of the width of dislocation image, which can be observed by electron microscopy, when TLD contrast fading can be seen. The rate of spreading depends on the structure and chemical composition of GBs, which means that this parameter is a characteristic GB property [10]. Dislocation spreading in GBs involves mass transport along GB [6,7] and can be used to study GB diffusion [4,8]. Investigations of dislocation spreading give information about local diffusion. In the case of aluminium it is specially useful because of a lack of an appropriate tracer.

The aim of this paper is to show how the spreading method can be used for determining the kinetics of recovery process on GBs and for investigations of GB self-diffusion in polycrystalline aluminium. Furthermore, the kinetics of GB recovery and distribution functions of GB diffusional parameters depend on the thermo-mechanical treatment and as such may form GB characteristics for polycrystalline materials.

2. MATERIAL

Investigations were performed on polycrystalline 4N aluminium whose chemical composition is presented in Table I. The detailed description of the material is presented in ref. [11]. In order to determine the influence of thermo-mechanical treatment on GB characteristics in polycrystal the material was prepared by means of hydrostatic extrusion and subsequent recrystallization annealing at two temperatures: 578 K (state S) and 623 K (state G) in one hour. This yielded two Polycrystals S and G with mean grain sizes of 4 and 22 μm, respectively. In earlier investigations it was established that Polycrystal S is characterized by a higher fraction of GBs with ordered structure than Polycrystal G [10].

<table>
<thead>
<tr>
<th>Sl</th>
<th>Fe</th>
<th>Zn</th>
<th>Cu</th>
<th>Ba</th>
<th>S</th>
<th>P</th>
<th>Ca</th>
<th>Cr</th>
<th>Mn</th>
<th>Pb</th>
<th>K</th>
<th>V</th>
</tr>
</thead>
<tbody>
<tr>
<td>24</td>
<td>24</td>
<td>21</td>
<td>19</td>
<td>16</td>
<td>12</td>
<td>5</td>
<td>2</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td></td>
</tr>
</tbody>
</table>

3. EXPERIMENTAL

Two methods can be used to determine the spreading kinetics of dislocations on GBs. The first is based on investigations of spreading when annealing is conducted in isothermal conditions and this method was used in the present work. The second is based on isochronal annealings at various temperatures [4].

Spreading was observed on thin foils by means of the Philips electron microscope with an accelerating voltage of 100 kV. The observations in Polycrystal G were performed at four temperatures: 293, 343, 373 and 403 K, and in Polycrystal S at 293, 373 and 403 K. In this work the experimental procedure was applied which is described in detail in ref. [2].

In order to characterize the process of recovery on GBs in a given sample, changes in the number of GBs with and free of TLDs were determined as a function of annealing time at a given temperature. For each measuring time-temperature point the fraction \( X \) of GBs free of TLDs was determined after observing 3 to 5 foils and at least 50 boundaries.

Regardless of the spreading model [6,7,12] the spreading time \( t_d \) at temperature \( T \) is inversely proportional to the GB diffusion coefficient \( D_b \)

\[
\frac{A}{T} \quad t_d = \frac{1}{\delta D_b} \quad (1)
\]
where \( A \) is a constant and it depends on the model assumptions, \( \delta \) is the grain boundary width. According to the model of Łojkowski and Grabski [12]

\[
A = \frac{C k Sm^B}{G V}
\]

where \( C \) is the numerical constant, \( V \) is the atomic volume, \( G \) is the shear modulus, \( S_m \) is the dislocation core width when TLD contrast disappears during TEM observations. According to Johansson and Thölen [8] \( S_m \) is equal to three extinction distances and this was confirmed by Varin and Romanowska [13]. The value of constant \( C = 20 \) was assumed after ref. [13].

4. RESULTS AND DISCUSSION

Dependence of the fraction of GBs with TLDs \((1 - X)\) on the time of annealing at 293 up to 1500 h for Polycrystals G and S is presented at Fig.1.

Fig.1, 2. Curves of isothermal recovery on GBs in polycrystalline aluminium for states S and G.

Fig.2 presents data for Polycrystal G at 293, 343, 373 and 403 K and for Polycrystal S at 293, 373 and 403 K, time up to 100 h. The experimental results of investigations concerning changes in the fraction of GBs with TLDs \((1 - X)\) as a function of time can be approximated by equation:

\[
(1 - X) = A - B \times \ln t_d
\]

where \( X \) - the fraction of GBs free from TLDs, \( t_d \) - recovery time in seconds, \( A \) and \( B \) - constants for a given temperature. The same dependence is regarded as characteristic of the recovery of mechanical properties and it is usually called the logarithmic recovery law [14].

Polycrystals G and S differ distinctly as far as the course of recovery is concerned (Figs. 1 and 2). The rate of recovery is much higher in Polycrystal G than in S. The rate of recovery in both polycrystals increases markedly with temperature. Recovery rate decreases with increasing extent of recovery.

On the basis of the obtained data the activation energy of the process was determined from the equation:

\[
\ln t_d = \ln C + \frac{Q_r}{RT}
\]

where \( C \) - constant for a given fraction, \( Q_r \) - recovery activation energy. Plotting the graph \( \ln t_d \) versus \( 1/T \) for a constant fraction, from the slope of the line the values of \( Q_r \) corresponding to fractions from the range 0.2 -
0.8 (1-X) covered by the measurements were obtained for both polycrystals. The values obtained are presented in Fig. 3.

![Fig. 3. Plot of activation energy of recovery $Q_r$ as a function of the fraction of GBs with TLDs in Polycrystals G and S](image)

Activation energy of recovery $Q_r$ changes linearly with the fraction of GBs with TLDs (1-X), the range of the changes being different for the two polycrystals. The linear dependence of activation energy on the fraction

$$Q_r = Q_0 - b (1-X)$$

(where $Q_0$, $b$ - constants) was observed. Such dependence is characteristic of recovery processes for which the logarithmic law obtains [14]. As $Q_r$ is dependent on recovery fraction, it is also a function of time, which means that $Q_r$ changes during recovery. The $Q_r$ values obtained reveal that recovery activation energy increases with the degree to which recovery has advanced. This means that recovery, being a thermally activated process, begins on more disordered boundaries, i.e. on boundaries with lower activation energy.

For each time-temperature point the diffusivity value was calculated using equation (1). Next the distribution functions of GB diffusivity were determined at different temperatures and they are presented in Fig. 4 and 5.

![Fig. 4, 5. Distribution functions of GB diffusivity in aluminium for Polycrystals G and S, respectively.](image)

The obtained distributions give the GB characteristics of diffusional properties in aluminium polycrystals and they depend on thermo-mechanical treatment. The results obtained show that the GB diffusivities at a given temperature differ from each other by two orders of the magnitude or even more. Furthermore, the values of GB diffusivities in Polycrystal G are higher in comparison with Polycrystal S. So the higher diffusional properties in the given aluminium one can obtain in the case of a higher recrystallization
temperature. The above results describe process of self-diffusion in aluminium and if one approximates these results to the temperature of 523 K they are in a good agreement with results of computer simulations of GB self-diffusion at 523 K in aluminium obtained by Biscondi [15].

It can be assumed that in log-normal coordinates the experimental points at constant temperature (Fig. 4 and 5) can be approximated by linear function. In this way the distribution functions of the GB diffusivity take a form of straight lines in the experimental area. The slope of these lines depends weakly on temperature for Polycrystal G but has a strong temperature dependence for Polycrystal S. This indicates that distribution of GB diffusion activation energies is more uniform for Polycrystal G than for Polycrystal S. This is also confirmed by a calculation of activation energies of GB diffusion. Taking values of the GB diffusivity corresponding to constant fraction (Fig. 4 and 5) the Arrhenius plot is obtained and further the parameters of GB self-diffusion are calculated. The procedure allows to obtain the distributions of GB self-diffusion parameters in polycrystals. The obtained parameters of GB self-diffusion: preexponential factor $\delta D_0$ and activation energy of GB self-diffusion $Q_b$ are given in Table II. These distributions are much more uniform in Polycrystal G than in S. The values of GB self-diffusion parameters are close one to the other in Polycrystal G while in Polycrystal S these values fall in a broader range.

Table II. Grain boundary self-diffusion parameters in aluminium obtained by the dislocation spreading method.

<table>
<thead>
<tr>
<th>Polycrystal</th>
<th>Fraction (1-X)</th>
<th>$Q_b$ [kJ/mol]</th>
<th>$\delta D_0$ $[m^3/s]$</th>
</tr>
</thead>
<tbody>
<tr>
<td>S (4 μm)</td>
<td>0.5</td>
<td>67.7</td>
<td>2.0$\times$10^{-14}</td>
</tr>
<tr>
<td></td>
<td>0.8</td>
<td>47.2</td>
<td>3.4$\times$10^{-18}</td>
</tr>
<tr>
<td>G (22 μm)</td>
<td>0.2</td>
<td>51.1</td>
<td>8.1$\times$10^{-19}</td>
</tr>
<tr>
<td></td>
<td>0.8</td>
<td>49.7</td>
<td>4.9$\times$10^{-18}</td>
</tr>
</tbody>
</table>

The determined values of recovery activation energy correspond to the values of GB self-diffusion activation energy and therefore indicate that GB self-diffusion is the rate controlling factor for recovery on GBs. Individual GBs in polycrystal differ in their diffusional properties which reflects the differences in the structure and leads to the differences in recovery kinetics. The Polycrystals G and S have different GB characteristics as a consequence of their different thermo-mechanical treatments, hence they differ in kinetics of the process.

5. CONCLUSIONS

1. The kinetics of recovery by annihilation of dislocations in GBs in aluminium can be determined by the dislocation spreading method and it is described by the logarithmic recovery law.
2. The rate of recovery on GBs depends on the GB characteristics of polycrystals.
3. The distributions of parameters of GB self-diffusion in aluminium: preexponential factor and activation energy may be obtained using the dislocation spreading method and they can be used as GB characteristics of polycrystal.
REFERENCES

3. M. W. Grabski, J. Physique, 46 (1985), C4-587
7. P. H. Pumphrey, H. Gleiter, Phil. Mag., 30 (1974), 593