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Relaxation Mechanisms in High Purity 99.999% Aluminium at Medium Temperatures

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Abstract. In this work we have studied the microstructural conditions for the appearance of the P_3 relaxation at about 650K in 99.999% Aluminium. The experimental behaviour of the P_3 peak allows us to conclude that it is linked to a process of dislocation mobility.

1. INTRODUCTION

The internal friction (IF) spectra in Al 5N single crystals was studied by Woïrgard et al. [1,2] between 300K and 700K and decomposed in a high temperature background plus three relaxation peaks named P_1 , P_2 and P_3 . These relaxations appear also in bicrystals and an annealing at 673K increases the P_2 and P_3 relaxations. In polycrystals Woïrgard et al. [1,2] observe so high the P_2 relaxation that the P_1 and P_3 relaxations are hidden. These results were confirmed by Bonetti et al. [3], who observe in Al 4N single crystals slightly deformed by flexion three relaxations K_1 , K_2 and H that increase with the deformation degree and do not disappear even after annealing at 813K during 12 hours. These relaxations appear also in polycrystals after primary recrystallization, being much more strength than in single crystals [3,4]. On the contrary, Kê et al. [5] do not observe any relaxation between 450K and 700K in Al 4N and Al 5N single crystals grown by zone melting. Nevertheless they observe a peak around 640K (named P_{365}) in similar single crystals grown by dynamic annealing [5-7], but this peak disappears after a 5% creep deformation at the temperature of the peak maximum, or after a 5% tensile deformation at room temperature followed by a 2h annealing at 873K [7]. In the case of polycrystals of Al 4N and Al 5N with bamboo microstructure, these authors observe a relaxation also at 640K when the sample has been deformed 3% and annealed at 830K. This relaxation is dependent on the oscillating amplitude [7] and disappears by tensile deformation [8]. Using another kind of internal friction measurements, stabilizing the temperature 4 hours before each measurement, Rivière et al [9-11] observe three relaxations P_1 , P_2 and P_3 that are amplitude independent and besides P_2 and P_3 do not disappear even after annealing at 800K.

A comparative and exhaustive study of the relaxations observed in the literature in Al6N, Al5N and Al4N at medium and high temperatures has been accomplished by N6 [12]. This study allows her to conclude that the high temperature component of the Kê peak, as well as the P_{365} , P_3 and H peaks are the same relaxation, that in the present work will be named as P_3 peak.

The aim of this work is to contribute with some supplementary results about the P_3 peak in order to understand its behaviour and to clarify the microscopic mechanism responsible for this relaxation.

2. EXPERIMENTAL CONDITIONS

In the present work we used 99.999% Aluminium (5N) "super raffinal" AIAG. The 5N Al supplied in 10mm plates was rolled until it was 1.1mm thick and annealed under primary vacuum for 1h at 500K in order to recrystallize the samples, that were subsequently aged for a long time at room temperature. In this condition the samples show a mixed microstructure of tangled dislocations and polygonization walls. In order to obtain a polygonized dislocation structure the samples were crept [12]. This microstructure disappears during heating until 700K at 100K/h, because the samples recrystallize. After this recrystallization, the samples do not show any polygonized structure and the grain size is about $\phi=2\text{mm}$. The study of the P_3 peak was carried out on these samples ($50\times5\times1\text{ mm}^3$). Internal friction was measured using an inverted torsion pendulum operating at about 1Hz with an oscillating amplitude of 4×10^{-6} . All IF spectra have been measured only on heating, at a heating rate of 100K/h.

3. RESULTS AND DISCUSSION

The internal friction spectrum of a recrystallized sample as described previously, corresponds to curve 1 in fig.1 and shows a P_2 relaxation about 550K and a relatively flat high temperature background.

Fig. 1. Internal friction after cumulative torsional deformation at room temperature. Before deformation (curve 1) and after a total deformation of $\pm 0.5\%$ (curve 2), $\pm 2\%$ (curve 3) and $\pm 8\%$ (curve 4).

In order to study the influence of deformation degree on the internal friction spectrum, the sample has been deformed in torsion at room temperature by cumulative steps of $\pm 0.5\%$, $\pm 1.5\%$, $\pm 2\%$, $\pm 4\%$ and successive steps of $\pm 4\%$ until a total deformation of $\pm 16\%$. For a better visual clarity we plot in Fig.1 (curves 2 to 4) only the internal friction spectra corresponding to the total deformation of $\pm 0.5\%$, $\pm 2\%$ and $\pm 8\%$. We observe that the high temperature background increases with the deformation percentage until a degree of 8%, and shifts the more and more towards low temperature. Simultaneously, a slight decrease of the P_2 relaxation can be observed. Further cumulative deformations by $\pm 4\%$ steps do not affect the background and the obtained internal friction spectra were almost superposed on the curve 4 of Fig.1.

Fig. 2. Internal friction spectra and modulus defect on the same sample previously deformed $\pm 16\%$ (see fig. 1). Curve 1: after a new $\pm 12\%$ torsional deformation performed at room temperature. The sample recrystallize again between 625 and 700 K. Curve 2: after recrystallization we observe the P_2 peak and the appearance of the P_3 peak.

Nevertheless, a further high deformation of $\pm 12\%$ performed in one step at room temperature gave rise to the recrystallization of the sample between 652K and 700K during the measurement heating run, as is shown in the internal friction spectrum and modulus defect of curves 1 in Fig.2. The curve 2 of Fig.2 corresponds to the first spectrum after recrystallization and shows two relaxations P_2 and P_3 that can be also seen in the associated modulus curve. These two relaxations P_2 and P_3 became stable during successive heating runs until 730K as is shown in Fig.3.

Fig. 3. Internal friction spectrum and modulus defect of stable P_2 and P_3 relaxation.

These results can be explained as follows. The original sample with a microstructure mainly with tangled dislocations presents the P_2 peak that shows a similar behaviour to the P_1 peak observed in Al 6N [13,14], and seems to be linked to a high jog density on dislocations [1,15,16]. The torsional deformation performed in successive steps increases the dislocation density, but during the measurement heating runs until 730K the dislocations reorganize in polygonization walls, like it has been also observed for the Al 6N [12,14]. This way, the high temperature background increases with the density of polygonized dislocations because in fact it is the low temperature side of a P_H peak that appears at higher temperature and attributed to the mobility of the subgrain boundaries [12,13,15,17]. At the same time we observe a slight decrease of the P_2 peak due the loosing of jogs by the dislocations during the polygonization process [12,16]. It should be remarked that after strong deformation, the internal friction spectrum (curve 1 of Fig. 1) shows the presence of the P_1 peak, that usually appears only in Al6N [12]. This effect is due to the increase of dislocation density and their depinning from the impurities during the deformation, that produces some non negligible dislocation density completely free of impurities. However, the recrystallization undergone (Fig.2) by the sample destroys completely the polygonized microstructure and the high temperature background recovers its original level (like in curve 1 of Fig.1) due to disappearance of the P_H peak. Nevertheless a high density tangled dislocation microstructure remains in the sample and the P_2 peak recovers its original strength. It is in this microstructural condition when the P_3 peak is clearly seen.

In order to characterize the behaviour of the P_3 relaxation we have studied its evolution with the oscillating amplitude. In Fig.4 we plot the internal friction spectra and modulus variation curves corresponding to the oscillating amplitudes of 4, 8, 16 and 32×10^{-6} . From Fig.4, it becomes evident that the P_3 peak shows a strong dependence on the oscillating amplitude. The previous results allows to conclude that the P_3 relaxation is due to some mechanism of dislocation mobility and the associated modulus defect curves (in Fig.4) suggest that this relaxation could be linked to the same dislocations responsible for the P_2 peak.

Fig. 4. Evolution of the P_3 relaxation as a function of the oscillating strain amplitude.
 Curve 1: $\varepsilon_m=4 \times 10^{-6}$. Curve 2: $\varepsilon_m=8 \times 10^{-6}$. Curve 3: $\varepsilon_m=16 \times 10^{-6}$. Curve 4: $\varepsilon_m=32 \times 10^{-6}$.

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References

- [1] J. Woïrgard, thesis University of Poitiers (France) (1974)
- [2] J. Woïrgard, J.P. Amirault, J. de Fouquet, ICIFUAS vol. 1, Springer-Verlag (1975) 392
- [3] E. Bonetti, E. Evangelista, P. Gondi, R. Tognato, *Phys. Stat. Sol. (a)* **39** (1977) 661
- [4] T.S. Kê, P. Cui, C.M. Su, *Phys. Stat. Sol. (a)* **84** (1984) 157
- [5] C.M. Su, T.S. Kê, *J. de Phys.* **46** (1985) 359
- [6] C.M. Su, T.S. Kê, *Phys. Stat. Sol. (a)* **94** (1986) 191
- [7] A. Rivière, thesis University of Poitiers (France) (1984)
- [8] A. Rivière, J.P. Amirault, J. Woïrgard, *J. de Phys.* **42** (1981) C5-439
- [9] A. Rivière, J. Woïrgard, J. de Fouquet, *J. de Phys.* **46** (1985) C10-343
- [10] E. Bonetti, E. Evangelista, R. Gondi, R. Tognato, *Il Nuovo Cimento* **33B** (1976) 408
- [11] C.M. Su, T.S. Kê, *Acta Met.* **37** (1989) 79
- [12] M.L. Nô, thesis University of Pais Vasco (Spain) (1990)
- [13] M.L. Nô, C. Esnouf, J. San Juan, G. Fantozzi, *Acta Met.* **36** (1988) 827-836 and 837-845
- [14] M.L. Nô, J. San Juan, C. Esnouf, *Materials Science and Engineering A* **113** (1989) 281-285
- [15] M.L. Nô, J. San Juan, *Materials Science and Engineering A* **164** (1983) 153-158
- [16] M.L. Nô, J. San Juan, J.I. Pérez-Landazabal, C. Esnouf, *Materials Science Forum* **119-121** (1993) 255-260
- [17] J. Friedel, Colloque de Frottement Intérieur IRSID (Saint-Germain-en-Laye) 13-14 Oct. 1970