INTERNAL FRICTION AT MEDIUM TEMPERATURES IN HIGH PURITY ALUMINIUM

M. Nó, C. Esnouf, G. Thollet, J. San Juan, Gilbert Fantozzi

► To cite this version:

M. Nó, C. Esnouf, G. Thollet, J. San Juan, Gilbert Fantozzi. INTERNAL FRICTION AT MEDIUM TEMPERATURES IN HIGH PURITY ALUMINIUM. Journal de Physique Colloques, 1987, 48 (C8), pp.C8-161-C8-166. 10.1051/jphyscol:1987821 . jpa-00227125

HAL Id: jpa-00227125

<https://hal.science/jpa-00227125>

Submitted on 1 Jan 1987

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.

INTERNAL FRICTION AT MEDIUM TEMPERATURES IN HIGH PURITY ALUMINIUM

M.L. NÓ, C. ESNOUF*, G. THOLLET*, J. SAN JUAN and G. FANTOZZI*

Dpto de Física del Estado Sólido, Facultad de Ciencias,
 Universidad del País Vasco, Aptdo 644, SP-48009 Bilbao, Spain
 *GEMPPM-LA 341, INSA, Bât. 502, F-69621 Villeurbanne Cedex,
 France

Resumé.— Afin de déterminer le mécanisme qui est à l'origine des relaxations P_1 et P_1' nous avons comparé les résultats expérimentaux de frottement intérieur et de microscopie électronique d'un Al 99.9999%, obtenus autour de $0.5T_m$, avec les prévisions théoriques des différents modèles proposés dans la littérature.

Abstract.— In order to determine which mechanism is at the origin of the P_1 and P_1' relaxations, the internal friction and electron microscopy experimental results of 99.9999% Al around $0.5T_m$ have been compared to the theoretical forecasts of different proposed models.

1.- INTRODUCTION

Under certain experimental conditions we can observe in the highest purity aluminium (99.9999%) the P_1 and P_1' relaxations around $0.5T_m$ (at 1Hz) /1,2/. In a lower purity aluminium (99.9999%+10ppm Cu or Ag) we can observe a relaxation at higher temperatures which could correspond to P_1 relaxation of Al 99.9999% (6N)/3/. Another kind of results, which we will discuss later on, have been obtained by Kê et al. on 6N Al /4/.

In this paper we will try and analyse which model of all those proposed up to now coincides with the experimental behaviour of P_1 and P_1' relaxations.

2.- EXPERIMENTAL CONDITIONS

In this work, we have used a 6N Al refined by zone melting. Some samples had been doped with 10ppm of Cu or Ag. All of them have been produced by CECM of Vitry/Seine, France. These samples have undergone various thermo-mechanical treatments in order to create different microstructures : - Cold rolled at room temperature (RT) and at 77K. - Torsional deformation at RT and at 77K. - Creep at 200°C and 300°C. - Annealing at 600K.

Internal Friction (IF) spectra have been taken with an inverted torsion pendulum oscillating around 1 Hz with $2 \times 10^{-7} < \epsilon_m < 5 \times 10^{-5}$ and $6\text{K} < T < 730\text{K}$. We can also superimpose a static torsional stress (σ_s) upon the oscillating stress (σ_m).

The microstructures of the samples has been analysed before and after applying these thermo-mechanical treatments, thanks to a JEOL-200CX ($\Delta V_{\text{work}}=160\text{kv}$). The study focuses on those parts of the samples where the thickness is over 6000Å so that they are representative of the bulk.

3.- EXPERIMENTAL RESULTS

* The samples deformed by torsion or cold-rolling at RT up to 50% show two relaxations P_1 and P_1' which are stable after a first annealing at 600K (Fig.1 curve a). These relaxations are not affected by a static stress (σ_s) superimposed upon the oscillating stress (σ_m) and whose order of magnitude is up to 10 times σ_m (Fig.2) /1,2,5/. The strengths of these relaxations show a maximum versus the oscillating amplitude (fig.3) /1,2,5/ while at the same time the peak shifts towards low temperatures. The activation energy of the P_1 relaxation was measured by two different ways : a) In a low frequency pendulum /3/. b) By microcreep at various temperatures, using Klam's technique /2/.

We obtained the following results : $H_{P_1} = 1.1\text{eV}$, $H_{P_1'} = 0.8\text{eV}$.

By microcreep measurements and for different applied stress we have determined the activation volume of the process : $v_{P_1} \approx v_{P_1'} \approx 1000b^3$.

The observation of the samples by Transmission Electron Microscopy indicates that the dislocations are organised in cells formed by tangled dislocations (Fig.1a).

* The samples which have undergone creep through tension or torsion at 200°C or 300°C do not show P_1 and P_1' relaxations ; however they show a strong background towards high temperatures (Fig.1 curve b) which corresponds to the low temperature side of a relaxation placed near the melting temperature/2,5/. In this case, the samples show a polygonized microstructure (Fig.1b). The size of the subgrains is about 30 μm and the subboundaries are generally formed by families of dislocations that do not verify Frank's law.

* A deformation of at least 20% by torsion at RT, or a similar deformation by rolling at RT causes the reappearance of P_1 and P_1' relaxations. A later creep makes them disappear again /2,6/.

We could then conclude that P_1 and P_1' relaxations are associated to a deformation cells microstructure, while their disappearance is linked to a polygonized microstructure.

Experimental results on 99.9999%Al+10ppm of Cu or Ag have been reported in /3/ and could be summarized as follows :

There is only one relaxation which occurs at higher temperature ($T(\text{Al}+\text{Ag})=526\text{K}$, $T(\text{Al}+\text{Cu})=565\text{K}$) than the P_1 relaxation, whose activation energy is 1.27eV for Al+Ag and 1.54eV for Al+Cu. On the other hand a superimposed static stress does not affect internal friction spectra, and the internal friction maximum decreases versus oscillating amplitude. Furthermore, it is important to remark that the relaxation disappears by creep.

4.- DISCUSSION

In this section, we will try to bring together all the models proposed in order to account for the relaxations that appear around $0.5T_m$ in aluminium and other f.c.c. metals. We will also take into account all the models proposed to explain creep in stage II, since the temperature range in which such stage occurs, as well as the activation energy and the activation volume either coincide or are close to those of P_1 and P_1' relaxation. So, we will analyse each model predictions, and will compare them to our experimental results.

a) Mechanisms connected with boundaries

Ké et al. /4/ have worked on Al 6N and they have noticed that in the case of polycrystals there is a relaxation at 480K which they identify with Ké's peak and they attribute to the intercrystalline sliding at boundaries. If the grain size ϕ verifies $\phi > e$ (e being the sample's thickness), then the peak is replaced by another that they call "macrocrystalline peak" ($T \approx 500\text{K}$), attributable to a mechanism in which polygonization and boundaries play important roles. A 0.4% deformation on the previous sample makes the macrocrystalline peak disappear, and makes the P_1 peak appear at 450K.

We have previously proved /2,6/ that the background and P_1 relaxation depend on the thermomechanical treatments and the oscillation amplitude. In this way we can obtain variations up to 60K in the peak temperature, depending on the experimental conditions. This makes us think that the three peaks mentioned by Ké et al./4/ that have never been noticed at the same time, are the same P_1 relaxation. Furthermore, we have proved that both the appearance or disappearance of P_1 and P_1' , as well as their intensity are independent from the grain size. This makes us think that P_1 and P_1' can in no way be interpreted as a mechanism associated only with the boundaries as was formerly proposed by Woigard /7/, who noticed P_1 , P_2 and P_3 relaxations in 5N Al both in monocrystals and polycrystals.

b) Cross-slip on planes $\{111\}$

Among the models proposed to account for cross-slip Friedel-Escaig's stands out /8/. Let us start from a constriction which has nucleated on a fault ribbon

spread on the primary slip plane of a f.c.c. crystal. Cross-slipping on another plane occurs when the two halves of the constriction are separated along the line, allowing some dislocation length in between to be bowed out and to dissociate in the cross-slip plane as soon as the halves are brought apart (Fig.4). For some critical separation, AB, the configuration energy goes through a maximum, beyond which A and B are pushed apart under the work of stress, making the whole dislocation cross-slip. The activation energy falls between U_C and $2U_C$ (U_C being the constriction energy). If we consider that U_C is of order of a jog creation energy ($U_C=0.42\text{eV}$) we obtain that the activation energy of this process is very inferior to that of P_1 , P'_1 and P relaxations, so this process can not explain these relaxations.

The model proposed by Caillard /9/ and later generalised by Morris /10/ (Fig.5) assumes that in the case of three dislocations which come to an intersection, their corresponding burgers vectors are coplanar. This model explained before in /5/ was proposed to explain the extraction of dislocations out of the polygonization walls being these formed by three families and we think that it could also be generalised to the deformation cells every time we have three coplanar vectors. Caillard shows that the stress that this mechanism needs to take place is very high (approximately 25% lower than Orowan's stress). Such stress can not be justified by means of the stress applied in IF measurements; thus we consider that this process can not be responsible for P_1 and P'_1 relaxations. On the other hand, the fact that the applied static stress superimposed upon the oscillation stress does not affect P_1 and P'_1 relaxations, means that this mechanism does not explain these relaxations /2,5,6/. Any applied static stress higher than the oscillation stress should produce the cross-slip and then, internal friction should disappear. Furthermore, the activation energy for this process is weak if we consider that the constriction nucleation energy is more or less the same as the jog formation energy ($U_C=0.42\text{eV}$).

Later on, Morris et al. /10/ observed that the extraction in the case of the three dislocations being on the same plane as the burgers vector, can take place through a simple slipping mechanism that needs climbing. We therefore think that in that case it is the climbing that controls the extraction mechanism.

c) Slip on non-compact planes

The experiments carried out by Carrard /11/ on monocrystals deformed by creep or tension along $[112]$ directions, show that in the case of creep, the slipping on $\{100\}$ can be noticeable from 180°C onwards for $10^{-7}\text{s}^{-1} < \dot{\epsilon} < 10^{-5}\text{s}^{-1}$, whereas in the case of tension tests, it does not appear until 279°C for $\dot{\epsilon} = 12 \times 10^{-5}\text{s}^{-1}$.

There are several models proposed to account for slipping on non-compact planes. Among these, the appropriate one to explain the experimental results obtained up to this moment is glide on non-compact planes of dissociated dislocations on $\{111\}$. Such mechanism proposed by Friedel and later improved by Escaig /12/ assumes a dissociated dislocation on a plane (111) that joins together along a critical length AB, producing double kinks on a non-compact plane, and then it dissociates again on a plane (111) (Fig.6). This mechanism needs to create four constrictions and two dissociated segments on a non-compact plane. So, the activation energy will be: $2U_K = 4U_C + 2U_R$, U_K being kink nucleation energy, U_C constriction nucleation energy and U_R the energy needed to create a dissociated segment AA' or BB'. If we consider the fact that U_C can be compared with the jog creation energy, and that U_K is between 0.9 and 1.13eV /11/, the result is $1.3\text{eV} < 2U_K < 1.7\text{eV}$. The activation energy seems to be slightly high compared with the activation energy of P_1 and P'_1 relaxations, noticed on 6N Al. On the other hand, the activation volume expected for this mechanism is between $350b^3 < v < 700b^3$ /11/, a value which is slightly low compared with the activation volume measured in our micro-creep tests $900b^3 < v < 1600b^3$ /1/. We must also point out that such mechanism should give an IF peak similar to Bordoni's. Therefore we could expect non linear effects like those described by Esnouf /13/ for Bordoni relaxation. Thus, in the case of weak internal stress, both the internal friction peak strength δ_M and its correspondent temperature must have a maximum versus σ_m . So, δ_M and T_M increase as Paré's condition is not fulfilled, in such a way that if we apply a static stress superimposed upon the oscillating stress, the strength of the relaxation will increase. Our experimental results show a maximum δ_M versus σ_m but on the contrary T_M decreases with a non linear dependency versus σ_m , while σ_s does not affect P_1 and P'_1 relaxations, as opposed to the theoretical predictions.

Al samples doped with 10ppm of Cu or Ag have values for the activation energy obtained for P relaxation and IF results /3/ that can be explained through Friedel-Escaig's mechanism. However the study will be incomplete until the microstructure of the samples is thoroughly analysed and internal friction is studied in detail in order to precise the influence of the applied tension. As we will see later on, the results obtained up to now also coincide with a dislocation glide mechanism controlled by jog climb.

If, instead of considering free dislocations we considered a subboundary with three families of dislocations verifying Frank's law, then one of the three families is screw. Under stress, mixed dislocations can glide on two planes $\{111\}$ while screw dislocations can do it on $\{100\}$ or on $\{110\}$ after a Friedel-Escaig mechanism in which only one kink will be nucleated since node O is able to glide freely (Fig.7)/15/. The activation energy expected will be : $0.65\text{eV} < U_K < 0.85\text{eV}$, which is too low to account for P_1 relaxation. Moreover, we have noticed through our experiments that P_1 relaxation disappears from polygonized samples, which seems to indicate that this mechanism is not the cause of P_1 relaxation. On the other hand, it seems unlikely that in the case of non polygonized samples with a tangled dislocation structure there is a high number of screw dislocations that cause a relaxation as intense as P_1 or P_1' . Nevertheless a more thorough analysis of the microstructure would be advisable to verify this point.

d) Slip controlled by pipe-diffusion

Let us suppose there is a screw or mixed dislocation with a sessile jog. The dislocation cannot glide unless the jog climbs thanks to a non conservative movement which provides vacancies created either in the bulk or near the dislocation (Fig.8)!. In this process, the activation energy is around the auto-diffusion energy ($H_v=1.5\text{eV}$) or the pipe -diffusion ($H_d=1.1\text{eV}$), and it depends slightly on the type of dislocation /14/. In such conditions H_v is too high to be responsible for the relaxations we are dealing with. However, relaxations P_1 and P_1' can be explained in terms of pipe-diffusion. In this mechanism, a static stress superimposed upon σ_m cannot affect the relaxation because it will only change the oscillation centre of the dislocation line. This prediction agrees with the experimental results. On the other hand, from the value σ_m we could expect a decrease in the internal friction maximum versus the oscillating stress. For high stress, the number of vacancies absorbed or emitted during half cycle of the oscillating stress is limited by the number of vacancies, N_v , that may be created near a jog at a given temperature. Therefore, the number of jumps N that the dislocation may have, amount that must be increased with oscillating stress, will be limited by this process. Thus, the energy dissipated in a cycle (at a given temperature) stays the same whereas the elastic energy increases with stress and as a consequence the maximum of internal friction decreases with stress. This behaviour agrees with the experimental results (see Fig.3)/2,6/.

On the other hand, we could expect that in the case of polygonized samples, the dislocations are more straight (the polygonization walls are flat to minimize the energy) and therefore there will hardly be any deformation jogs and so, they would have to be created. In this case the activation energy would correspond to that of a double jog nucleation, which is very high /5/.

In this way, this mechanism seems capable of accounting for all results concerning relaxations P_1 and P_1' in 6N Al. For 6N Al doped with Ag or Cu, the experimental results obtained in internal friction can also be explained in terms of this mechanism. Indeed, in this case, the impurities will tend to place on the jogs modifying the climb energy of the jog. Therefore the activation energy of the process must be slightly higher than that of pipe-diffusion, in agreement with the experimental results.

5.- CONCLUSIONS

Considering our experimental results and the models proposed so far, we can conclude that the only mechanism capable of accounting for P_1 and P_1' relaxations in 6N Al is dislocation glide (screw for P_1 , mixed for P_1') controlled by jog climb and vacancy pipe-diffusion. However, for 6N Al doped with impurities, we need some supplementary internal friction tests and a characterization of the samples microstructure in order to state if the observed P relaxation is the same as P_1 relaxation which has shifted towards high temperatures due to the presence of impurities, or

if this relaxation is caused by another kind of process.

REFERENCES

- 1.- M.L.Nó, J.San Juan, C.Esnouf, G.Fantozzi, A.Bernalte; J. de Phys.44(1983)C9-751
- 2.- M.L.Nó; Thesis University of Lyon (France) (1985)
- 3.- C.Esnouf, A.Kulik, J.F.Theumann, M.L.Nó; This conference
- 4.- T.S.Ké, P.Cui, S.C.Yan, Q.Huang; Phys.Stat.Sol.(a)86 (1984) 593
- 5.- M.L.Nó, C.Esnouf, J.San Juan, G.Fantozzi; J. de Phys. 46 (1985) C10-347
- 6.- M.L.Nó, C.Esnouf, J.San Juan, G.Fantozzi; in press.
- 7.- J.Woigard; Phil.Mag. 33 (1976) 623
- 8.- B.Escaig; J. de Phys. 35 (1974) C7-151
- 9.- D.Caillard, J.L.Martin; Acta Met. 31 (1983) 813
- 10.- M.A.Morris, J.L.Martin; Acta Met 32 (1984) 549
- 11.- M .Carrard; Thesis University of Lausanne (1985)
- 12.- B.Escaig; Phys. Stat. Sol. 28 (1968) 463
- 13.- C.Esnouf; Thesis University of Lyon (1978)
- 14.- M.L.Nó, C.Esnouf, J.San Juan, G.Fantozzi; Scripta Met. 21 (1987) 213
- 15.- D.Caillard; Phil. Mag. A51 (1985) 157

Fig.1.-
Internal Friction spectra for different microstructural states and their corresponding transmission electron micrographies ($\epsilon_m = 8 \times 10^{-6}$).

- a) samples deformed by rolling or torsion at RT or at 77K.
b) Samples deformed by creep at 200°C or at 300°C.

Fig.2.- σ_s influence on Internal Friction spectrum. $\sigma_m = 2 \times 10^{-6} \mu$, a) $\sigma_s = 0$, b) $\sigma_s = 10^{-5} \mu$, c) $\sigma_s = 2 \times 10^{-5} \mu$, d) $\sigma_s = 4 \times 10^{-5} \mu$

Fig.3.- Evolution of the P_1 and P_1' relaxations strength versus σ_m for different thermomechanical treatments /2/.

Fig.4.- Cross-slip in the Escaig's model.

Fig.5.- Cross-slip mechanism at the nodes of sub-boundaries : we can see the cross-slip of dislocation 3 /9/.

Fig.6.- Friedel-Escaig's mechanism /11/.

Fig.7.- Glide motion in P_1 , P_2 , and P_3 planes due to nucleation and migration of a kink on the $\{100\}$ plane (P_1) /15/.

Fig.8.- Glide controlled by pipe-diffusion: 1) Initial state. 2) Loop of dislocation under stress. 3) Final state after creating and emitting a vacancy out of a jog.