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DISLOCATION MOTION IN PURE ALUMINIUM AT 0.5 T_f : ANALYSIS FROM INTERNAL FRICTION MEASUREMENTS

M.L. NO, C. ESNOUF, J. SAN JUAN* AND G. FANTOZZI
 GEMPPM- INSA de Lyon Bât. 502, 69621 Villeurbanne Cedex, France
 *Depto de Fisica del estado Solido, Facultad de Ciencias, U.P.V. Aptdo 644, Bilbao, Spain

Resumé : Une étude des phénomènes de relaxation à moyenne et haute températures a été entreprise dans l'aluminium de haute pureté (99,9999%). Ces relaxations sont au nombre de trois : deux relaxations P_1' et P_1 autour de 400K à une fréquence de 1 hertz et une relaxation à haute température près du point de fusion. Après une analyse des mécanismes possibles, les deux premières sont interprétées en termes de mouvement de dislocations crantées; la dernière est expliquée par le mouvement de dislocations gouverné par la création des crans thermiques.

Summary : A study of internal friction in medium and high temperatures range has been performed on high purity 99,9999% aluminium. Three relaxations have been detected : two notified P_1' and P_1 around 400K when the frequency of vibration is about one hertz and the third is observed at high temperature near the melting point. After a review of possible mechanisms, the two first are interpreted in terms of jogged dislocation motion and the last by the dislocation motion governed by the thermal jog nucleation.

I.- INTRODUCTION

Several groups of workers study actually the dislocations and subboundaries behaviour of f.c.c. metals, specially aluminium, in the temperature range around 0.5 T_m and more (T_m : melting temperature). Old results were obtained with the internal friction technique by Kê /1/ who observes a damping peak at 558K with a frequency of vibration of 1Hz. These last years and with the same experimental method, Woigard et al /2,3,4/ has investigated three relaxations P_1 , P_2 , P_3 in single and polycrystalline aluminium. The activation energy of P_1 peak is less than self-diffusion energy H_v while P_2 and P_3 peaks have an activation energy near H_v (H_v : activation energy for self-diffusion). Recently, these authors say that the heating rate during the peak description seems important and in some cases, the appearance of "pseudo peaks" is possible when the heating rate is too high /4/. If the internal friction spectra are described by discrete temperature increases (maintenance of 4 hours at each temperature), the peaks activation energy are different from previous ones. For instance, in the case of aluminium, the activation energies are very similar and near of 0.65 H_v for the relaxations. Also, Bonetti et al. /5,6,7/ observe the Kê peak is modified by annealing above 820K where another relaxation appears called K_2 situated at a temperature higher than the Kê peak one. They observe also a high temperature peak at about 800K for a vibration frequency of 80Hz. In the same temperature range than K_1 and K_2 peaks one, an extrinsic grain boundary sliding occurs by absorption and climbing of dislocations /7/ but the same authors had previously /6/ shown that these two relaxations are correlated with the dislocation rearrangements leading to cellular structures during in-situ creep.

All previously described results concern only usual pure metals (less or equal to 99.999% purity). In high purity aluminium (99,9999%) the results seem different enough : the relaxation strength is very high (one order of magnitude higher

than the P_1 relaxation observed by Woignard) /8/. This relaxation has a satellite called P_1' /9/ situated at 40K below and also no P_2 peak is observed in aluminium. At last, in the same purity aluminium, Kê et al. /10/ observe a relaxation assimilated to Kê peak although its temperature is different enough (70K lower about). An important decrease of this relaxation is observed when the grain size exceeds the sample size and another peak takes place if a small plastic deformation is made. Kê et al. support the idea that these relaxations are due to three different mechanisms although their peaks temperatures are similar.

So, we can remark that the situation is not very clear and some new experiments on high purity metals are necessary to analyse the different mechanisms; then we have performed the internal friction measurements on coldworked and annealed samples and crept samples in order to confirm or eliminate the possible interpretations as cross-slip, kink nucleation due to Peierls forces on anomalous glide plane 100 and climb.

II.- SPECIMENS AND EXPERIMENTAL PROCEDURES :

In this present work, we have used a 99,9999% aluminium obtained by zone refined melting at CECM Vitry (France). The internal friction measurements are made on an inverted torsion pendulum working in a temperature range from 4K up to 750K while the frequency of the oscillation is about one hertz. The samples have a parallelepipedic shape : 50mm for length, 5mm for width and different thickness going to 0.35mm to 1.2mm according to the thermomechanical treatment. A forced oscillation pendulum is also used with a frequency range of 10^{-4} - 1Hz. Moreover the grain size is systematically measured before and after the internal friction measurements and a subtraction of internal friction background has been performed on the internal friction spectra in order to determine the exact behaviour of each relaxation /9/. A damping measurement is also possible during the creep of samples (in-situ creep).

III.- EXPERIMENTAL RESULTS

Previously, we have obtained numerous results concerning the P_1' and P_1 peaks /8,9,11/. They appear at about 400K after coldwork by rolling at low temperature, after deformation by rolling or torsion at room temperature, and after deformation by torsion at high temperature (up to 600K).

Their evolutions caused by annealing after plastic deformation are weak expected during the first heating. They are quite stable after an annealing of about 500K and keep their stability in height and temperature at least up to 880K. This behaviour seems to be the same for an initial grain size from 0.2mm to 3mm measured at room temperature. Besides, the P_1' and P_1 peaks temperatures are sensitive to thermomechanical conditions through the dislocation lengths probably and sensitive the oscillating amplitude /9/ (45K in the range 10^{-7} - 3.10^{-5}) and also sensitive to the background evolution. So, all discussions on these peaks must be made only after a subtraction of internal friction background and a strict control of the oscillating strain. As a function of oscillating amplitude, the P_1' and P_1 peaks heights go through a maximum and the P_1' and P_1 peaks temperature follows an identical evolution for all initial thermomechanical treatments /9/. Now to verify the possible existence of cross-slip mechanism as discussed below, we have recorded the internal friction when a bias stress σ_m is superimposed to the measurement stress σ_m such the condition σ_m is verified. The figure 1 shows that the influence of the bias stress does not exist.

Then in order to have a very characteristic dislocations structure, the samples are previously crept in the stationary stage at 473K with a slow creep rate (typically $10^{-7}s^{-1}$). Figure 2a presents the internal friction of a 13% crept sample. The P_1' , P_1 peaks have practically disappeared and a strong increase of damping is present in the high temperature range. Figure 2b shows the internal friction versus the frequency of oscillation. A high temperature peak is present (HTP) associated with a strong modulus decrease. Moreover the P_1 peak is present again for a 40% cold-rolling after this creep and the internal friction in the high temperature range is smaller and there is a significant decrease of the HT peak. In order to confirm

the P_1 disappearance due to creep, we have recorded the damping during a creep-test (fig.3) at the P_1 temperature. The P_1 peak disappears although the creep stage is still the primary stage.

III.- DISCUSSION

In literature some mechanisms have been proposed for explaining the dislocation behaviour at medium temperatures and based on the TEM in situ observations (12,13). They show the existence of cross-slip at nodes of subboundaries but the required resolved stress is high enough (of the order of the Orowan stress). Indeed a high local internal stress is present when the arrangement of dislocation network is disturbed. Such a mechanism should be present also in crept samples. Another argument against the cross-slip for P_1 peak interpretation is the non-influence of the static stress superimposed to the oscillating strain (fig.1). Indeed the cross-slip requires only one overcoming of the energetical barrier due to the recombination of partial dislocations which is required for the slip plane change. Consequently a bias stress greater than the oscillating stress should produce the cross-slip and the peak should be absent.

In polygonized and crept samples, very numerous subboundaries are created; they are essentially composed of three families of dislocations /12/. The subboundaries are built from two dislocations families having two different burgers vectors and a reaction produces another family with a 120° coplanar burgers vector. Near the equilibrium, the long range stresses in the material are minimized (Frank formula respected) and then one family of dislocations in the subboundary has a pure screw character /13/. During the motion of such a subboundary only two families can glide in a normal slip plane 111 (fig.4) and the last must move in anomalous plane 100, this glide can be thermally activated if Peierls forces on 100 planes are considered /13/ and the P_1 peak should be equivalent to a Bordoni peak since the dislocation motion is governed by the kink nucleation at a triple node (cf. Fig.4b) but this interpretation does not seem good because no appreciable peak is observed after creep except the HT peak (fig.2).

Finally taking in to account the activation energy for P_1 peak /8/, we must retain the model of jogged dislocation motion. The peak is present only for long jogged dislocations (as shown by the apparent activation volume value $v = 2000b^3$ /9/) (peak observed after plastic deformation). In crept samples, the dislocation are almost all in subboundaries and then they are short and very straight i.e. the jog density is too low and the P_1 peak disappears. But some thermal jogs can be produced with a corresponding energy given by $H_v + 2H_j$ (H_j : energy for jog formation). In aluminium, this quantity is equal to about 2.4eV /14/ which is just the activation energy for the HT peak. Indeed the HT peak is very high when the P_1 peak is low, which is in agreement with our hypothesis.

In conclusion, two typical structures must be considered : the deformation structure with a high density of "geometrical" jogs which are responsible for the P_1' and P_1 peaks and the polygonized structure with a low density of jogs which is only moving by vacancy and thermal jogs nucleation.

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Fig.1.- Internal friction spectra on rolled Al at 77K (grain size = 1 mm) when a bias stress of 0.2 and $5 \times 10^{-5} \mu$ is superimposed to the oscillating stress $\sigma_m = 9 \times 10^{-6} \mu$

Fig.2.- a) Internal friction spectrum on 13% creeped Al at 473K (grain size = 5mm) with $\sigma_m = 10^{-5} \mu$; b) Internal friction versus log(frequency) at 693K (HT peak)

Fig.3.- Internal friction (1) and deformation (2) during in situ-creep at 412K for an applied stress of 5,5 MPa.

Fig.4.- Schematic process of the sub-boundary movement : a) originated by cross-slip; b) by kink nucleation on {110} plane at a triple node/12,13/