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DISLOCATION SEGMENT DISTRIBUTION EFFECTS ON DYNAMIC MODULUS AND INTERNAL FRICTION

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Résumé - Module dynamique et frottement intérieur mesurés directement pendant la déformation permettent d'analyser l'évolution de la distribution des segments de dislocations. On a obtenu ainsi des indications sur les transformations de structure qui accompagnent soit le passage entre le processus de fluage déterminé par l'énergie de self-diffusion massive ou par l'énergie inférieure, soit le passage entre conditions de déchargement et rechargement.

Abstract - By means of analyses of the dislocation segment distributions obtained from dynamic modulus and internal friction measurements made directly during deformation, indications have been obtained on the dislocation structure changes accompanying both the passage from lower to bulk self-diffusion activation energy creep processes as well as the transition occurring by unloading and reloading.

Introduction - In previous papers /1,2,3/ it has been shown that from dynamic modulus (M_d) measured directly during deformation together with internal friction coefficient (Q^{-1}), deformation rate (\dot{\varepsilon}) and eventually dislocation density (\rho) it is possible to obtain indications on the evolution of the distribution function \phi(\ell) /4,5,6/ giving the density per d\ell of dislocation segments in the range \ell+\ell+d\ell. In particular results of previous researches concern the vibration frequency which is effective in the thermally activated processes of link overcoming by the dislocation segments, the average area swept out per elementary slip act by each dislocation segment and the contribution to dynamic modulus and to deformation of the long length tails of the distribution functions.

Taking advantage of those results further problems are considered here, i.e. the questions of the transition mechanism responsible for the passage from 87 KJ/mol to 125-142 KJ/mol as dominating activation energies for stationary creep above or below ~ 425 K in Al /7/, and of the structure changes in unloading and reloading.

Experimental - Rectangular sheets of AI 99.99%, 1x10^{-3}m thick, 3x10^{-3}m wide, with lengths ranging from 1 to 1.5x10^{-2}m, were used for the experiments, with grain sizes ~ 10^{-3} m.

Internal friction and dynamic modulus were measured directly during constant load deformation by means of an inverted pendulum, as already described by us and by other authors /1,8/. Loads were chosen so as to have at various temperatures comparable deformation rates, in the range 10^{-4} - 10^{-6}sec^{-1}. Torsional frequencies ranged from 5 to 10 Hz. The amplitudes were smaller than 10^{-5}. Errors relating to internal friction coefficients were ~ 1%, to dynamic modulus 1-2%.

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The resonance frequencies served to determine the torsion modulus: the modulus changes were determined by differential measurements, with reference samples non-deformed and with equal lengths.

TEM observations were made after some deformations at some temperatures. For TEM the specimens were prepared by means of electrolytic polishing.

Results - As already discussed in previous papers /1,2/ internal friction and dynamic modulus show behaviour with correspondences which may be expressed by the relation

\[ Q^{-1}_b + Q^{-1}_v + Q^{-1}_d = Q^{-1}_0 + C \varepsilon + \beta \Delta M \]  

(1)

where \( Q^{-1}_b \) is the background contribution taken as a constant and equal to \( Q^{-1}_0 \) before deformation, \( Q^{-1}_v = C \varepsilon \) is the internal friction component due to viscous deformation, which resulted negligible, \( Q^{-1}_d \) the component depending on dislocation damping. Last \( Q^{-1}_d \) is considered proportional to \( \Delta M = (M_d - M_0)/M_0 \) (corresponding to a modulus defect) through the coefficient \( \beta \) which expresses the amount of relaxation (for S.L.S., \( \beta < 1 \) if relaxation prevails).

The diagrams in Fig. 1(a)-b)-c) illustrates the said behaviour for some temperatures in evidence.

At the various temperatures effect of unloading during creep have also been examined. Such unloading effects during deformation can be better followed through the variations of \( Q^{-1}_d \), instead of those of the modulus defect which becomes small by increasing deformation, with large relative errors. Of course the variations of \( Q^{-1}_d \) can be translated to those of \( \Delta M \) through the ratio \( \beta \), which is assumed to remain constant as during deformation.

On unloading \( Q^{-1}_d (\beta \Delta M) \) shows a different behaviour at different temperatures, as visible in Fig. 2(a)-b)-c). At lower temperatures, e.g. room temperature and 423K, unloading is accompanied by rapid large \( Q^{-1} \) decreases, followed by smaller, progressive additional decreases: on reloading previous \( Q^{-1} \) values are restored progressively.
At the higher temperatures instead unloading is accompanied by rapid $Q^{-1}$ increases, followed by a progressive regression to the values before unloading: apart from a small polygonization peak, reloading causes a similar behaviour.

In various papers /9,10,11,12/ it has been assumed that the variations of $Q^{-1}$ and $M_d$ in the medium temperature range, from ~450K to ~750K, are due to dislocation relaxation. The decreases of $M_d$ occur in a broad temperature range, owing to a broad spectrum of relaxation times. The phenomena can be better localized by considering the $Q^{-1}$ and $M_d$ behaviour together, as shown in Fig. 3. Full relaxation can be assumed to occur after the $K_2$ peak, i.e. at $T_c$.

At lower temperatures the modulus defects can be interpreted with reference to two extreme cases, i.e. partial relaxation of all the dislocations or complete relaxation of part of all the dislocations. Since $\beta < 1$ (Fig. 1) the second case is admitted; so the fraction $\xi$ of the relaxed dislocations has to be taken into consideration and this is obtained by considering $\xi$ equal to the ratio $\Delta M_{Td}/\Delta M_{Tc}$ where $\Delta M_{Td}$ is the modulus decrease from room temperature up to the deformation temperature $T_d$, $\Delta M_{Tc}$ the modulus decrease up to $T_c$, both referring to conditions before deformation.

Finally a TEM image is given in Fig. 4 illustrating dislocation structures occurring before the $Q^{-1}$, $\Delta M$ peak experienced during deformation at 423K; such structures are of interest for the discussion.

![Fig. 3](image1.png) ![Fig. 4](image2.png)  
Fig. 3 - Internal friction coefficient ($Q^{-1}$) and dynamic modulus ($M_d$) vs temperature before deformation; $T_c$ temperature of full dislocation relaxation. Fig. 4 - TEM image of an Al specimen deformed just before the $Q^{-1}$ peak during creep at 423K (ref. Fig. 1b). Fig. 5a) - Activation energies characteristic of stationary creep of Al at various temperatures (ref. /7,13/). Fig. 5b) - Reference model for the passage from stage $H_1$ to stage $H_2$ in Fig. 5a).

Discussion - First the transition between the two medium temperature processes governed by different activation energies is discussed.

As an introduction to the problem well known experimental results from /13/, subsequently specified in /7/, are recalled. These results are illustrated by the diagram in Fig. 5a). The transition between the two processes characterized by $H_1 = 87$ KJ/mol and $H_2 = 125-142$ KJ/mol might be understood in terms of two temperature dependent rate processes as sketched in Fig. 5b). Assuming that the two processes act in series larger rates will be dominant. According to this scheme, transition between each other dominating process will occur at temperature $T_t$, being

$$\dot{\varepsilon}_{01} \exp(-H_1/RT_t) = \dot{\varepsilon}_{02} \exp(-H_2/RT_t)$$

Introduction of the experimentally known quantities, i.e. $H_1 = 87$ KJ/mol, $H_2 = 142$ KJ/mol, $T_t = 423K$ leads to the pre-exponential ratio

$$\dot{\varepsilon}_{01}/\dot{\varepsilon}_{02} = 10^{-5}$$
The deformation rate can be expressed by
\[ \dot{\varepsilon} = \int_{-\infty}^{\infty} b - \frac{\sigma - Gb}{\rho} \frac{ab}{\sigma} \frac{kT}{\phi(x)} \exp \left(-\frac{H}{kT}\right) \phi(x) dx \] (4a)
i.e.
\[ \dot{\varepsilon}_e = \int_{-\infty}^{\infty} b - \frac{\sigma - Gb}{\rho} \frac{ab}{\sigma} \frac{kT}{\phi(x)} \phi(x) dx \] (4b)

where \( \nu_d \) and \( H \) are vibration frequency and activation energy for the thermally activated dislocation motion, \( S = \bar{x}^2 \) is the average area swept out in each elementary slip per dislocation segment, thus expressed as a function of \( \bar{x}^2 \) through the proportionality coefficient \( c \), \( \sigma_e \) the effective stress which is taken equal to \( \sigma - Gb/\rho \) (\( \sigma \) applied stress, \( \alpha \) strength constant taken equal to 1), \( a = A/\sigma_e \) expresses the proportionality of activation area \( A \) to effective stress.

According to 4b) the possibility that the pre-exponential ratio in (3) depends on different \( \nu_d \)'s is considered first. The lower energy \( H_1 = 87 \text{KJ/mol} \) has been related to pipe diffusion along the dislocations /7,14/, whereas the higher energy \( H_2 \) is related to bulk self-diffusion. So it may be reasonable to assume that the vibration frequency effective in pipe diffusion is the one of attack per dislocation segment of length \( \bar{x} \), i.e. \( \nu_d = \nu_D/\bar{x} \), whereas \( \nu_d = \nu_D \) for lattice self-diffusion, \( \nu_D \) being the Debye frequency. Since relations (4a–b) refer to elementary processes the activation energy for lattice self-diffusion will be introduced into (4a) as higher energy, i.e. \( H_2 = 125 \text{KJ/mol} /15/ \).

To check the effects of the said differences in \( \nu_d \) it is worth considering also the following relations, regarding the dynamic modulus and dislocation density both given as a function of the dislocation segment distribution function:
\[ \Delta G/G = \xi \int_{-\infty}^{\infty} \eta \bar{x}^3 \phi(x) dx = \Delta M \quad (5); \]
\[ \rho = \int_{-\infty}^{\infty} \bar{x} \phi(x) dx \quad (6) \]

Following previous considerations /3/ \( \phi(x) = a \bar{x}^2 \exp(-B \bar{x}^2) \) will be used as distribution function. Introduction of this function into (5) and (6) allows us to obtain \( A \) and \( B \) as a function of \( \sigma \) and \( \Delta M \) assuming \( \Delta M = \Delta G/G \). Results of relating calculations are expressed by diagrams of \( \varepsilon^* = \varepsilon(\Delta M/\eta)^{-1} \) as a function of \( \rho^* = \rho(\Delta M/\xi)^{-1} \).

Fig. 6a)-b)-c) Diagrams of \( \varepsilon^* = \varepsilon(\Delta M/\eta)^{-1} \) vs \( \rho^* = \rho(\Delta M/\xi)^{-1} \) calculated by solving eqs. (4a),(5),(6). In Fig. 6a) \( T = 423 \text{K} \); diagram 1, \( \nu_d = \nu_D/\bar{x} \), \( H = 87 \text{KJ/mol} \), \( c = 4\pi \); diagram 2, \( \nu_d = \nu_D \), \( H = 125 \text{KJ/mol} \), \( c = 4\pi \); diagram 3, \( \nu_d = \nu_D \), \( H = 87 \text{KJ/mol} \), \( c = 4\pi \). In Fig. 6b) \( T = 297 \text{K} \); diagram 1, \( \nu_d = \nu_D/\bar{x} \), \( H = 87 \text{KJ/mol} \), \( c = 4\pi \); diagram 2, \( \nu_d = \nu_D \), \( H = 87 \text{KJ/mol} \), \( c = 4\pi \). In Fig. 6c) \( T = 423 \text{KJ/mol} \); diagram 1, \( \nu_d = \nu_D \), \( c = 1 \); diagram 2, \( \nu_d = \nu_D \), \( c = 10^{-3} \); diagram 3, \( \nu_d = \nu_D/5 \), \( c = 10^{-3} \). The dotted lines show experimental \( \varepsilon^* \) for the deformations \( \varepsilon \) indicated.

According to the assumption in question the experimental creep rates at \( T = 423 \text{K} \) should be consistent with \( \varepsilon \) values calculated by introducing into (4a) either \( \nu_d1 \) and \( H_1 \) or \( \nu_d2 \) and \( H_2 \). Diagrams so calculated are drawn in Fig. 6a); they are consistent with the experimental data represented by faint lines, eventually with small changes in c (causing ordinate shift) or of \( \xi \) (causing shift of both ordinates and abscissae) for
fitting to the dislocation densities experienced case by case.

However, if the same $v_d = \nu b / \lambda$ is used at the lower temperatures, e.g. at room temperature, diagram 1 of Fig. 6b) is obtained, displaced towards low $\dot{\varepsilon}^*$ values, whereas with $\nu_d = \nu D$ good correspondence with the experimental deformation rate is obtained (diagram 2 in Fig. 6b). Large increases of the other terms, besides $v_d$, that contribute to $\dot{\varepsilon}^*$ in 4b), are unlikely: so the explanation based on the reduced attack frequency is abandoned. In this respect it is also noticed that, in a theoretical treatment, Granato et al. /16/ conclude that the effective frequencies can differ from those of attack, tending to $v_D$ for strong pinning points.

Of course if $\nu_d = \nu D$ is maintained with $H_1$, diagrams calculated at 423K will result displaced towards deformation rates higher than those experienced, as shown in Fig.6a); i.e. there occurs the inverse of the situation considered previously with reference to room temperature, with the difference that now decreases of other terms contributing to $\dot{\varepsilon}^*$ in 4b), seem reasonable, in particular of the c coefficient since the ratio of S over $\dot{\varepsilon}$ should change with different dislocation structures. For example if the moving dislocations find their pinning points in grooves of dislocations, as in the case of Fig. 4, the c ratio will be $a / \lambda$ where $a$ is the distance between dislocations in the groove.

This $a / \lambda$ ratio may be of the order of $10^{-3}$ larger than the ratio between the pre-exponentials in (3). Further adjustments can be obtained by considering different distribution functions (see also /3,5,6/) smaller internal back-stresses (with minor contributions) as well as effective frequency decreases for weak pinning points. Curves for different c's, down to the range of the experimental deformation rates are drawn in Fig. 6c).

According to this view the transition between the stages characterized by the two activation energies $H_1 - 2$ should depend on variations of $\dot{\varepsilon}_{01}$ mainly dependent on the dislocation structure, so that above $T_1$ the $H_2$ process prevails. A detailed analysis of the processes governed by two energies is not considered here: mention is only made to paper /17/ where contributions of g.b. and lattice diffusion are treated with particular reference to the transfer of glide from one grain to the other across the grain boundary: results of a previous work /2/ are also recalled according to which $\nu_d$, $H_1$ and $\nu_d$, $H_2$ refer respectively to the conditions before and after the $Q^{-1}, \Delta M$ peak encountered during creep at the temperature in evidence.

Discussing now the unloading and reloading effects reference is made to the experimental results given in Fig. 2a)-b)-c).

The phenomena are analyzed by assuming that the dislocation density $\rho$ does not change on unloading and reloading, but only the dislocation segment distribution. This assumption is based on the fact that reloading causes the recovery of the same relaxation conditions present before unloading.

The variations of the distribution function may thus be followed by solving the system of equations (5) and (6) for constant $\rho$ (the $\rho$ has been taken corresponding for each temperature and $\Delta M$ (before unloading) to the maximum of the diagrams of Fig.6a).

The structure evolution can be followed through the average segment length

$$\bar{\lambda} = \int_0^\infty \phi(\lambda) d\lambda / \int_0^\infty \phi(\lambda) d\lambda$$

(7)

The diagrams reported in Fig. 7 have been obtained in this way.

At the lower temperatures the increases in $\bar{\lambda}$ are understood considering that pinning points which are overcome under load become effective again after unloading.
Reloading causes new unpinning without relevant variations in the dislocation density. So the evolution on reloading appears different from the one after the first loading (see for ex. /1/), when increases of the dislocation density by multiplication play a fundamental role.

At higher temperatures, instead, dislocations, which are pushed against links or other pinning sites by the load, become free when the load drops, thus acquiring longer free lengths and giving greater contributions to the pertaining reversible deformation; they then return to the lengths corresponding to the average net of pinning distances by rearrangement processes driven by the internal stresses. Reloading has a similar effect, first of unpinning and subsequent pushing the dislocations against stronger links, in part due to the development of new subboundaries.

References

17/ E. Bonetti, A. Cavallini, E. Evangelista and P. Gondi, this Conference.