INFLUENCE OF REMAINING IMPURITY
CONCENTRATION ON THE INTERNAL FRICTION
AFTER CYCLIC DEFORMATION IN PURE
ALUMINIUM

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INFLUENCE OF REMAINING IMPURITY CONCENTRATION ON THE INTERNAL FRICTION AFTER CYCLIC DEFORMATION IN PURE ALUMINIUM

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Abstract. - The characteristic internal friction peak of low temperature (78K) cyclic deformation in Aluminium has been studied. The peak can be explained by an interaction dislocation-dislocation loop [1]. The height of this peak depends on the type and the concentration of impurities and its evolution with annealing temperature can be understood by supposing a diffusion mechanism of dislocation loops and impurities.

1. Introduction. - The defects created by cold-working are not of the same nature and concentration after a one way or a cyclic deformation. As transmission electron microscopy observations revealed, the latter treatment creates a very dense arrangement of dislocations and a high number of small dislocation loops.

In Aluminium the internal friction spectrum measured at 1 Hz reveals two peaks between 80K and 300K, figure 1: The Bordoni peak B₂, at ~90K, and the peak Pₐ at ~ 250K, characteristic of a cyclic deformation (fatigue peak) [1,2].

2. Experiments. - In order to study the influence of the impurities on the internal friction spectrum after cyclic deformation, Aluminium samples of different origins and different impurity concentrations have been measured (table 1). The measurements have been carried out in an inverted torsion pendulum with a frequency of 1 Hz for 1 mm in diameter samples and 2.4 Hz for 2.5 mm in diameter samples respectively.

3. Results. - 3.1. Influence of Impurities. - On the figures 2 and 3 the evolution of the peak heights of B₂ and Pₐ and of the peak temperature of Pₐ as a function of...
Fig. 2: Peak heights $Q_{B_2}^{-1}$ and $Q_{PF}^{-1}$, frequency $f$ and $P_F$ - peak temperature $T_P$ as a function of annealing temperature $T_A$ in Al 5N.

- the peak appears after room temperature annealing
- its height is maximum after annealing at 380K
- the peak temperature increases with annealing temperature
- when the peak height of $P_F$ is increasing, the one of $B_2$ is decreasing.

Fig. 4: As figure 2, but measured in Al 6N 1.

On figures 4 and 5 the corresponding results are shown for the Al 6N 1 and Al 6N 2 samples (about 1 ppm impurity concentration). The most important differences are the following:
- there is no $P_F$ peak after room temperature annealing
- the peak height of $P_F$ is maximum after annealing at 420K
- the peak height of $P_F$ is small and the one of $B_2$ is big compared with those of the Al 5N samples.

Some of the results obtained with Al 5N 3 samples (15 ppm Si) are shown on figure 6 [5]. In the case of this type of Aluminium the maximum peak height is already obtained after one annealing below 340K.

3.2. Electron microscopical observations.- In order to have supplementary informations on the mechanism responsible for the peak $P_F$, similar treated specimen had been observed in a transmission electron microscope [6]. It appeared that a small part of the dislocation loops, created by the fatigue treatment, is highly mobile above 240K. In an impure Aluminium (e.g. 99.98%) no peak $P_F$ is measured, but also no mobile loops can be detected. The loops are mobile over more or less long periods of time depending on the concentration and on the element of impurity remaining in the metal. In Aluminium samples with a total impurity concentration of $\sim$5N and 1 ppm Si the loops remain mobile during several months whereas in a sample with 15 ppm Si (Al 5N 3) the loops become immobile after a few days.

In situ deformation experiments with fatigued specimen revealed a strong interaction between long dislocations and small dislocation loops. In the temperature domain of the $P_F$ maximum depinning as well as dragging processes have been observed [5].

![Fig. 6: Internal friction as a function of temperature after different annealing treatments in Al 5N 3 (15 ppm Si).](image1)

![Fig. 7: Internal friction as a function of temperature in Al 6N 2 deformed at 78K (a), with a preceding n-irradiation at 78K (b).](image2)

4. Discussion.- The interpretation of the fatigue-peak $P_F$ by an interaction between dislocations and dislocation loops has been confirmed by the appearance of such a peak when dislocations and loops were created separately [7].

On figure 7 the internal friction spectrum measured after a fast neutron-irradiation, followed by a tensile deformation, both at 80K, is compared with a spectrum measured with a specimen similarly treated but without irradiation. The
peak at 260K, which behaves as $P_F$, can be attributed to the presence of dislocation loops observed by Transmission Electron Microscopy inside similarly treated specimens.

According to the electron microscopy observation and the results after n-irradiation, an interaction dislocations with dislocation loops is proposed as the responsible mechanism for the peak $P_F$. The assumed model is based on a dragging of the loops perpendicular to the dislocation line \([1,4,7]\). If $\Lambda$ is the total density of dislocations, $N_1$ and $N_2$ the number of loops and impurities, respectively, at the dislocations per unity of length, and $L_0$ the initial length of a dislocation segment, then the height of peak $P_F$ is given by:

$$Q_F = \frac{g^2 \cdot \Lambda N_1}{12(N_2 + 1/L_0)^3}$$

($g^2 = 0.1$: orientation factor)

A general expression to describe the number of defects which have migrated to the dislocations is given by \([8]\):

$$N_i = K_i \cdot (D_i t)^{m_i}$$

with

- $K_i$: factor depending on the concentration and type of defects;
- $D_i$: diffusion coefficient of defect $i$;
- $t$: annealing time;
- $m_i$: a constant ($< 1$).

Although the diffusion of the loops is not isotropic, it is still reasonable to assume that it can be described by equation (2) (index $i=1$ for the loops; $i=2$ for the impurities).

The coefficients $C_i = K_i \cdot D_{oi}^{m_i}$, $m_i$ and the activation energies $E_i$ for the diffusion coefficients have been determined from isothermal annealing experiments with Al 5N 2 samples \([7]\). In that way the following values have been obtained:

- $E_1 = 0.65 \pm 0.05$ eV, $E_2 = 0.73 \pm 0.08$ eV
- $m_1 = 0.28 \pm 0.03$, $m_2 = 0.75 \pm 0.08$
- $C_1 = 4.35 \times 10^8$, $C_2 = 1.12 \times 10^{11}$

Assuming an annealing time of 30 min, the evolution of the peak height of $P_F$ can be calculated. The results shown on figure 8 are in good agreement with the measurements shown on figures 2 and 3.

The activation energy for the migration of the loops to the dislocations is not the one of a simple mechanism and $E_1$ is an effective activation energy, which can contain e.g. a depinning process or a shear mechanism of the dislocation loop to transform a sessile loop into a mobile one. Therefore the activation energy for the peak growth ($0.65$ eV) should be higher than the activation energy for the peak it-
self (0.43 eV).

The influence of the impurities can be double. On the one hand impurities can be nuclei for the formation of secondary defect (here dislocation loops \( g \)), so that in a purer metal less loops are created. In order to see if this simple assumption already describes the evolution of the peak height \( P_F \) in Al 6N samples, it was supposed that the density of the effective impurities is 10 times smaller than in the Al 5N 2 samples (e.g. Si atomes, see table 1). Using otherwise the same numerical values as those received for Al 5N 2, the peak height of \( P_F \) can be calculated at the different annealing temperatures. Again the results (figure 9) are in good agreement with the measurements (figures 5 and 6). In particular the shift of the maximum peak height to higher temperatures is predicted.

On the other hand at higher annealing temperatures, the impurities migrate to the dislocations, so that the latter are pinned at low temperatures and also at the peak \( P_F \) temperature. In the case of Aluminium the Si atomes seem to be very effective impurities. This can be seen from the results in Al 5N 3 (figure 6). Because of the high concentration of Si, many loops are present near the dislocations already after annealing to room temperature, consequently the peak \( P_F \) is high. At annealing temperatures still below 340K the Si atomes start to migrate to the dislocations and as a consequence the heights of the peaks \( P_F \) and \( B_2 \) decrease. In Al 5N 2 with only 1 ppm Si, this effect is therefore observed at temperatures above 380K.
References


Table 1: Impurity concentrations of Aluminium samples

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<th>Ca</th>
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<th>Si</th>
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<td>3</td>
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<td>1 mm</td>
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<tr>
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