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EFFECT OF IMPURITIES ON THE INTERNAL FRICTION SPECTRUM OF MAGNESIUM AFTER PLASTIC DEFORMATION

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Abstract.- We have studied the impurity effect on the internal friction spectrum, with two different samples magnesium 4N and magnesium 6N containing zinc and iron :
- for the sample 4N we observe a broad peak depending on the oscillation strain amplitude : this peak shifts towards low temperature when the amplitude increases.
- for the sample 6N containing zinc and iron, the internal friction spectrum is different form the one of the 6N sample previously mentioned. After annealing at 250 K we observe, at low strain amplitude, besides the B_1 and B_2 peaks, two other peaks situated around 100 and 150 K. But at a high strain amplitude we find the two peaks B_1 and B_2 as in the 6N sample.

After annealing at 500 K the B_1 and B_2 peak are not observed but a large peak appears which shifts towards low temperature with increasing ε_m. This peak is specific on thermomechanical unpinning of dislocations.

1. Introduction.- The internal friction spectrum of a high purity magnesium after plastic deformation is very similar to the one observed in f.c.c. metals. Indeed, the broad peak observed for the less pure materials [1-2] is replaced by several peaks in the case of pure magnesium [3-4] . There are :

a) two peaks situated around 40 and 80 K labelled B_1 and B_2 respectively. They are stable during annealing.

b) two peaks situated around 105 and 220 K, called P_1 and P_2 respectively and removed during temperature cycling.

c) a peak at 15 K or an important decrease of internal friction between 10 and 30 K after annealing up to 300 K

d) a stable peak near 420 K

We have shown elsewhere that the two peaks B_1 and B_2 have the characteristics of the Bordoni relaxation [5,6] i.e. :

- they are relatively stable during annealing
- their maximum temperature depend weakly on the microstructural state and obeys the Arrhenius relation.
- their relaxation strength presents a maximum as a function of stress behaviour.

In this paper we have studied the impurity effect on the internal friction

2. Experimental results.- Three differents polycrystalline samples have been used :
- a high purity magnesium noted Mg_1 with one at.ppm of impurity (99,9999 %).
- a less pure magnesium noted Mg_2 with < 100 at.ppm of impurity (99,99 %).
- a high purity magnesium noted Mg_3 containing zinc and iron (20 at ppm zinc +25 ± 13 at ppm iron).

After drawing, the samples were annealed for two hours at 300°C under argon atmosphere (grain size ≃ 1 mm). The internal friction have been performed with an inverted torsion pendulum oscillating at a frequency of about 1 Hz, with a maximum strain amplitude equal to $4 10^{-5}$. The specimens have been deformed in situ by torsion of 2 % at 10 K then the internal friction was recorded between 10 K and 500during linear heating at a rate of 60 K/h. Fig.1, 2 and 3 show the internal friction spectra as a function of temperatrue between 10 and 250 K, for the three samples Mg_1 and Mg2 and Mg_3.

Immediately, after plastic deformation at 10 K (fig.1), we observe :
- for the Mg_1 sample (curve 1) : two small peaks B_1 and B_2 situated around 40 and 80 K, and an important peak P_1 situated around 105 K. Furthermore we observe a peak P_2 situated about 220 K.
- for the Mg_3 sample (curve 3) : there are a relatively large peak at about 40 K and a peak (may be P_I) at about 105 K.
- for the Mg_2 sample (curve 2) : there is a large maximum situated between 30 and 120 K.

It must be noted that the internal friction background is important for Mg_2 and Mg_3 samples.

After heating up to 250 K (fig.2) we observe essentially :
- for Mg_1 sample, two peaks B_1 and B_2 at about 40 and 80 K and a small peak Π_1 at about 15 K (curve 1).
- for Mg_3 sample (curve 3) two small peaks (may be B_1 and B_2) at about 40 and 65 K, but the internal friction background is relatively strong.
- for Mg_2 sample (curve 2) always a large maximum is present between 40 and 120 K and also the internal friction background is very important (we can compare the internal friction at 150 K for the three samples).

Fig. 3 presents the internal friction spectra after annealing at 400 K for the three samples ; one can note :
- for the Mg_1 sample (curve 1), the two peaks B_1 and B_2 have disappeared and an important decrease of the internal friction is noticed between 10 K and 30 K, the pic Π_1 being annealed.
- for the Mg_2 and Mg_3 samples, we observe a relatively large maximum at about 40 K (for Mg_3 ; curve 3) and 25 K (for Mg_2, curve 2). The internal friction background is more elevated for the Mg_2 sample than Mg_3 and especially an increase of the internal friction background is observed at about 150 K for Mg_2 sample.

We have shown elsewhere [6] that the internal friction background is very sensitive to the oscillating strain amplitude ε_m .In order to eliminate the internal background effect we have measured the internal friction at a small amplitude (some 10^{-6}). The fig. 4 shows the internal friction spectrum of the Mg_3 sample after plastic deformation at 10 K (2 %) and annealing at 250 K.

On this figure one can note that besides the B_1 and B_2 peaks, two other peaks situated around 100 K and 145 K appear. One peak seems to shift to lower temperature with increasing the strain amplitude.

After heating upto 500 K (the B_1 and B_2 peaks have been already disappeared) we have studied the variation of the internal friction as a function of the strain amplitude (ε_m) for different temperatures (Fig. 5). The curves $\delta = f(\varepsilon_m)$ shift towards low temperature with increasing the measurement temperature (curve 1 to 4) and the internal friction maximum has about the same value.

3. Discussion.- Wa have shown elsewhere that the B_1 and B_2 peaks observed in a high purity magnesium after plastic deformation have the same features as the Bordoni relaxation in the f.c.c. metals.

For a less pure magnesium, the internal friction spectrum is constituted of a large maximum between 40 and 120 K (fig. 1 and 2). This maximum has been observed by several authors in a less pure magnesium [1,2]. In a magnesium 6N containing zinc and iron, at a low measurement amplitude, we observe besides the Bordoni peakssome other peaks (Fig.4). We can consider then, the large maximum observed in a less pure magnesium can be the superposition of two or more peaks. This idea has been already proposed by Tsui and Sack. This maximum shifts towards lower temperature when measurement amplitude ε_m increases [6] . Thus, this maximum can be attributed to a thermally activated breakaway process and we try to analyse the results by the T.G.L. depinning model.

Teutonico et al [7] have developed a model for thermally activated breakaway of a dislocations with only one pinned point at its middle. This dislocation have one or two equilibrium position according to the value of the applied stress or the dislocation loop length 2l.

- for the loop length smaller than critical length $l_c = \frac{2Gb^4}{E_o}$ only the pinned position of dislocation is possible. (E_o : activation energy for the depinning).
- for the loop length larger than l_c , there are two equilibrium position of dislocation ; one corresponding to pinned position and another one corresponding to the depinning position.

In the case of a high frequency or a low temperature ($E_o > kT$) and considering an exponential distribution of the loop lengths, Lücke et al [8] lead to a simplified form of the internal friction as a function of a parameter τ determined by the applied stress, the temperature and the frequency.

$$\Delta\tau = \Lambda L^2 \left(\frac{1+\tau}{\tau 3} \right) \exp -\left(\frac{1}{\tau}\right)$$

$$\tau = \frac{L}{l_o} = \frac{\sigma_o}{\sigma_{t_1,L}}$$

L : the average loop length of dislocation.

l_o : the critical loop length and

$$\sigma_{T_1,L} = \sigma_1 \{1 - \left[\frac{kT}{E_o} \ln\left[\left(\frac{\nu_1\sigma_1}{8\nu\sigma_o}\right) \frac{kT}{E_o}\right]\right]^{1/2}\} \quad \text{(I)}$$

$\sigma_1 \simeq \frac{E_o}{b^2L}$, (ν_1 : the attempt frequency ; b^2L : activation volume).

For a given internal friction we have :

$$\sigma_m = \alpha \frac{E_o}{b^2L} \left(1 - \frac{T}{T_c}\right)^{1/2} \quad \text{(II)}$$

with α = 0,36 at the maximum. T_c is a constant and has a classical value as [9] $T_c \simeq E_o/25\ k$.

For calculating T_c and E_o the curves of measurement amplitude ε_m has been plotted as a function of $T^{1/2}$ on the figure 6 for a Mg_3 sample after annealing at 500 K. From this figure, considering the relation (II) we can get :

T_c = 75 K and as $T_c \simeq E_o/25\ k$, we have :

$E_o = 0,16 \pm 0,02$ eV ; $b^2L \simeq 200\ b^3$

Using an Arrhenius relation $f = f_o \exp. \frac{-E}{kT}$ for the results obtained by frequency change we get ($\varepsilon_m = 2.10^{-5}$)

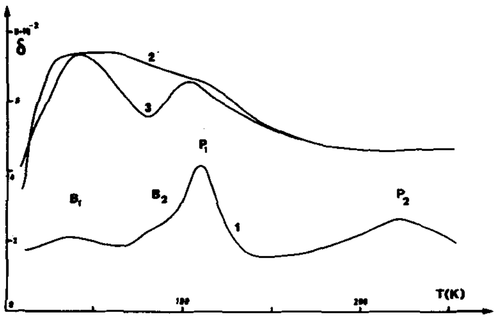


Fig. 1 : Internal friction spectrum for three samples immediately after plastic deformation of 2% at 10 K.
Mg_1 (curve 1, $\varepsilon_m = 410^{-5}$)
Mg_2 (curve 2, $\varepsilon_m = 210^{-5}$)
Mg_3 (curve 3, $\varepsilon_m = 210^{-5}$)

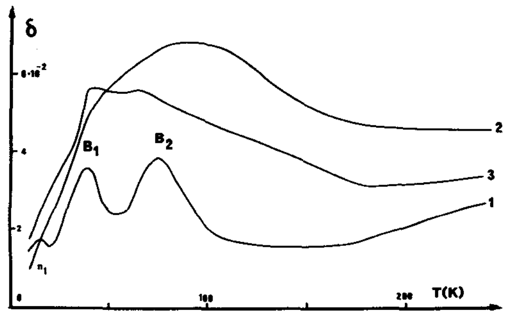


Fig.2 : Internal friction spectrum after plastic deformation of 2% at 10 K and heating up to 250 K
Mg_1 (curve 1)
Mg_2 (curve 2)
Mg_3 (curve 3)

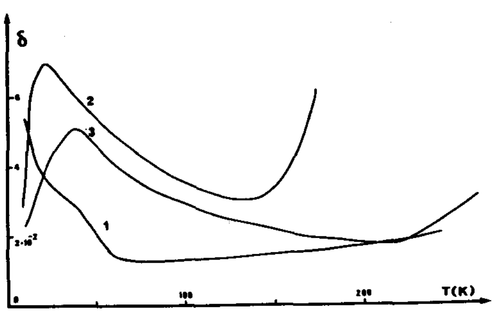


Fig. 3 : Internal friction spectrum after plastic deformation of 2% at 10 K and annealing at 400 K
Mg_1 (curve 1)
Mg_2 (curve 2)
Mg_3 (curve 3)

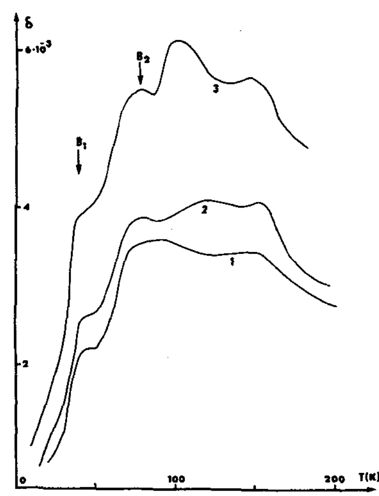
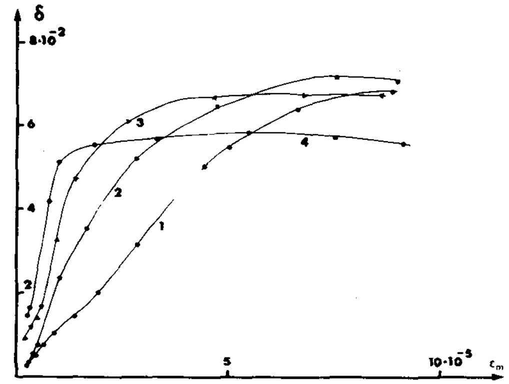
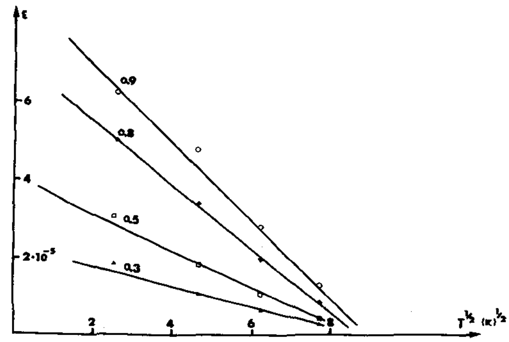


Fig.4 : Internal friction spectrum after plastic deformation of 2% at 10 K and annealing at 250 K for a Mg_3 sample with
$\varepsilon_m = 3.5\ 10^{-6}$ (1)
$\varepsilon_m = 5.8\ 10^{-8}$ (2)
$\varepsilon_m = 1.3610^{-5}$ (3)



<u>Fig.5</u> : Internal friction as a function of ε_m for a Mg_3 sample, after plastic deformation of 2% at 10K and annealing at 500 K



<u>Fig. 6</u> : The variation of measurement amplitude as a function of $T^{1/2}$ (the T.G.L. model).