

High-temperature internal friction in aluminium studied by isothermal mechanical spectrometry

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Abstract

Isothermal mechanical spectrometry experiments (10^{-4} Hz to 100 Hz) were carried out on strain hardened aluminum samples after recovery annealing between 300 and 700 K. For lower annealing temperatures (up to 480 K), internal friction spectra exhibit only a low-frequency background. Above 480 K, a relaxation peak stands out progressively against the background. The peak corresponds to the traditional intermediate temperature peak (ITP). Its height increases for annealing below 580 K and decreases above that temperature: the relaxation parameters undergo large changes after successive annealings. The peak is attributed to dislocation segment motion in dislocation pile-ups created by strain hardening and modified by recovery annealing.

1. Introduction

A large volume of data on high-temperature relaxation in aluminium has been published [1–4], but large differences are observed. Some workers attribute the observed peaks to a direct or indirect grain boundary relaxation whilst others believe that these relaxation peaks are due to network dislocation motion. It has been shown [5] that, in aluminium, relaxation effects are very sensitive to a large number of experimental parameters, such as sample purity, strain amplitude, rate of temperature change and previous thermomechanical treatments. This may explain the differences observed between the previous results and indicates that the classical anisothermal internal friction method is not convenient for a rigorous study of the high-temperature relaxation in aluminium. Since it is now possible to describe internal friction spectra *vs.* frequency at a fixed temperature with great accuracy, experiments were carried out with different purity aluminium samples to obtain precise relaxation peak parameters. Results obtained during successive annealing of strongly rolled 3N polycrystalline aluminium are presented in this paper.

2. Experimental method

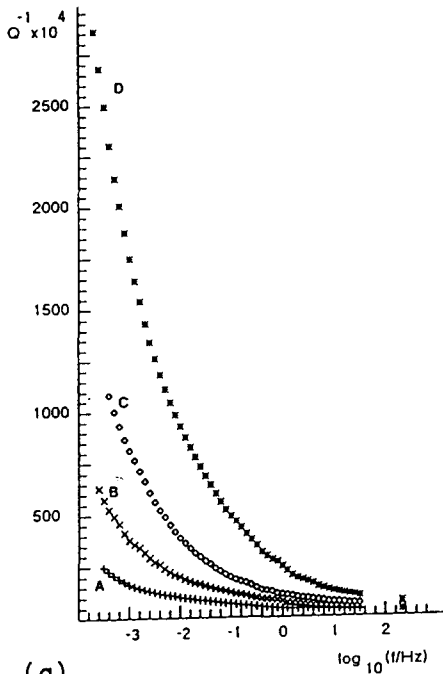
Samples were flat bars of 3N aluminium (60 mm × 5 mm × 1 mm) 67% cold rolled after high-temperature annealing. Internal friction measurements were carried

out with a forced torsion pendulum [6] in a vacuum of 10^{-5} Torr. The temperature was stabilized for 30 h before the beginning of each isothermal experiment and held constant during frequency sweeps between 160 and 10^{-4} Hz. Ten discrete frequencies per decade were used. For each measurement temperature, Q^{-1} was determined for three maximal vibration amplitudes: 5×10^{-6} , 10^{-5} and 2×10^{-5} . The annealing and measurement temperatures were the same, if not otherwise specified.

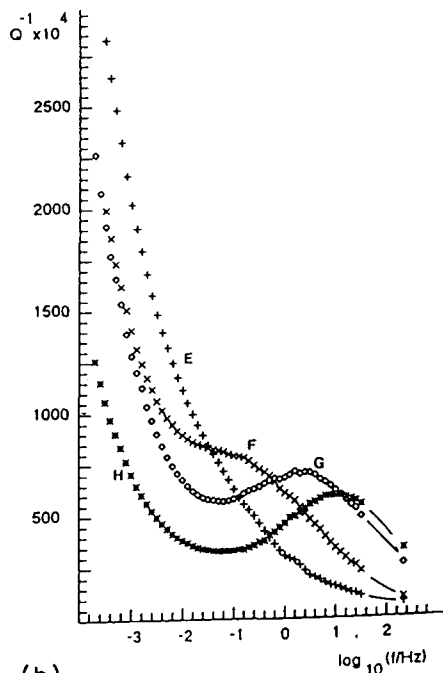
3. Experimental results

Figure 1 shows the internal friction spectra obtained after annealing at increasing temperatures. For annealing temperatures lower than 480 K, a low-frequency background is observed (Fig. 1(a)). A relaxation peak stands out progressively against the background (Fig. 1(b)) for annealing temperatures above 480 K, and the background decreases.

After background subtraction, it is possible to describe the evolution of the relaxation peak. Figure 2 shows that the peak height increases for measurements carried out after annealing below 577 K; it decreases for measurements carried out after annealing above that temperature. For all these experiments, the measurement temperature was the same as the annealing temperature. If measurements were carried out at lower temperature after high-temperature annealing, the evolution of the peak frequency shown in Fig. 3 was



(a)



(b)

Fig. 1. Internal friction measured after annealing (30 h) at different temperatures. The measurement temperatures are the same as the annealing temperatures. $\epsilon_M = 10^{-5}$. A, 327 K; B, 363 K; C, 400 K; D, 474 K; E, 489 K; F, 528 K; G, 577 K; H, 660 K.

observed. The peak shifts towards low frequency after high-temperature annealing. This is shown in Fig. 4 which gives the temperature and frequency peak positions after annealing in the range 501–659 K.

The activation parameters depend strongly on the annealing temperature as shown in Table 1.

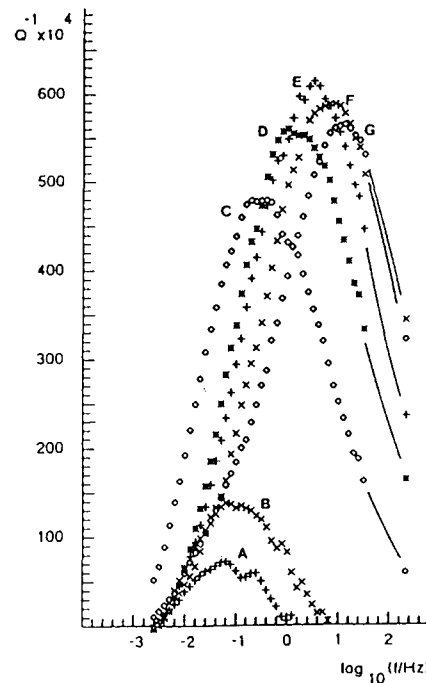


Fig. 2. Peaks obtained after background subtraction. Temperatures are the same for measurement and annealing. $\epsilon_M = 10^{-5}$. A, 489 K; B, 502 K; C, 528 K; D, 550 K; E, 577 K; F, 611 K; G, 660 K.

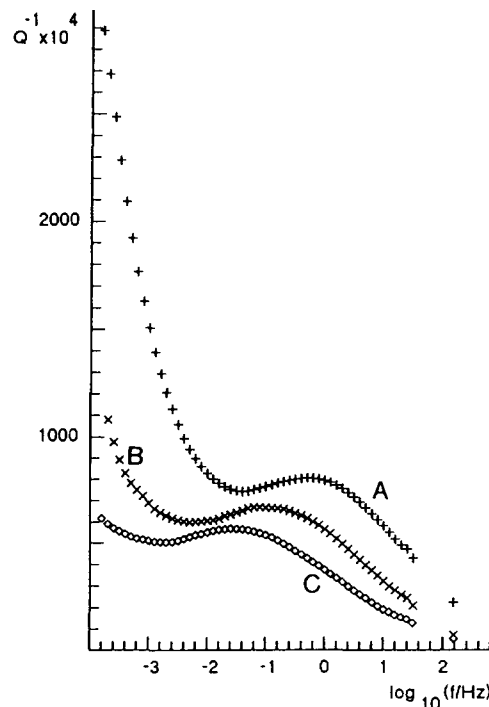


Fig. 3. Internal friction spectra obtained at the measurement temperature of 570 K, after annealing at 570 (A), 618 K (B) and 659 K (C).

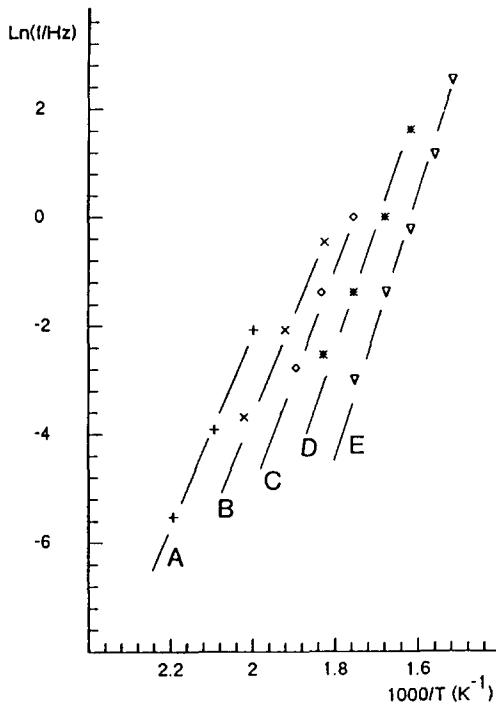


Fig. 4. Arrhenius plots obtained after annealing at 501 K (A), 548 K (B), 570 K (C), 618 K (D) and 659 K (E).

TABLE 1. Activation parameters as a function of annealing temperature

| Annealing temperature (K) | Peak temperature at 1 Hz (T_p) (K) | T_p/T_M^a | Activation energy (eV) | τ_0 (s) |
|---------------------------|--|-------------|------------------------|---------------------|
| 501 | 538 | 0.58 | 1.5 | 10^{-15} |
| 548 | 555 | 0.60 | 1.45 | 2×10^{-14} |
| 570 | 568 | 0.61 | 1.7 | 10^{-16} |
| 618 | 588 | 0.63 | 1.7 | 10^{-15} |
| 659 | 613 | 0.66 | 2 | 10^{-17} |

^a T_M , melting temperature.

The peak temperature at 1 Hz increases from 538 to 613 K for an annealing temperature increase from 501 to 659 K. The activation energy increases from 1.5 to 2 eV and τ_0 decreases from 10^{-15} to 10^{-17} s.

4. Discussion and conclusion

The frequency (or temperature) position of the intermediate temperature peak (ITP) in aluminium is not fixed, but strongly dependent on the thermomechanical treatment. To describe this peak at 1 Hz with a classical anisothermal method, a temperature of 660 K is required. We have shown that changes in the relaxation parameters occur below this temperature. Low-frequency isothermal mechanical spectrometry is the only method that can be used to obtain valuable results on ITP in aluminium.

Because of the observed evolution of the internal friction spectra, *i.e.* the absence of a peak after low-temperature annealing, the increase in the peak with annealing temperature and the variation of the activation parameters, it is difficult to attribute the peak to grain boundary relaxation. This evolution can be correlated with the variation of the dislocation network during annealing.

However, the value measured for the activation energy of the main relaxation mechanism is too high for a dislocation mechanism in aluminium. The peak width corresponds to a stretched exponential relaxation function Kohlrausch, WILLIAMS, WATTS model (K.W.W.) [7, 8], rather than to a simple exponential. With the model of Ngai and White [9], the activation energy and the logarithm of the limit relaxation time must be multiplied by a factor of less than unity, giving true values which are lower than apparent values. Further isothermal experiments are necessary to provide quantitative information.

References

- 1 A. Rivière, *Thèse Doctorat*, Poitiers, 1984.
- 2 M.L. No, C. Esnouf, J. San Juan and G. Fantozze, *Acta Metall.*, 36 (1988) 837.
- 3 K. Iwasaki, *Phys. Status Solidi A*, 42 (1983) 115.
- 4 T.S. Kê and A.W. Zhu, *Phys. Status Solidi A*, 113 (1989) K195.
- 5 A. Rivière, J. Woïrgard and J. de Fouquet, *J. Phys.*, 46 (1985) C10-343.
- 6 J. Woïrgard, Y. Sarrazin and H. Chaumet, *Rev. Sci. Instrum.*, 48 (1977) 1322.
- 7 R. Kohlrausch, *Amm. Phys. (Leipzig)*, 12 (1847) 393.
- 8 G. Williams and J.C. Watts, *Trans. Faraday Soc.*, 66 (1970) 80.
- 9 K.L. Ngai and C.T. White, *Phys. Rev. B*, 20 (1979) 2475.