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Low-temperature mechanical loss measurements of 5N and 6N lead

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Abstract

Measurements of the dynamic modulus and internal friction of 5N and 6N lead were performed after low-temperature plastic deformation. Low-temperature deformation induces an important decrease in the low-temperature modulus. This result is associated with lubrication dislocation motion due to intrinsic point defects. Finally, the evolution of the relaxation peak observed when the annealing temperature is increased demonstrates that Bordoni relaxation due to kink pair formation occurs in lead at a very low temperature compared with other f.c.c. metals.

1. Introduction

Experimental and theoretical arguments indicate that the problem of Peierls stress in f.c.c. metals is due to the existence of several types of kink pair formation (KPF) mechanism, presenting very different activation energies [1]. In order to verify whether different microscopic KPF mechanisms occur in lead, measurements of the internal friction and dynamic modulus have been performed in 5N and 6N lead samples. It was expected that a lubrication mechanism would occur at very low temperatures and Bordoni relaxation would be observed.

2. Experimental details

The internal friction and resonance frequency were obtained by a flexural vibration technique of thin clamped beams [2]. The damping of the free vibrations was measured using a discrete Fourier transform method. The vibrations of the sample were electrostatically excited by an electrode in a vacuum of 10^{-6} Torr and detection was realized using the same electrode. To reduce parasitic interactions with the sample holder, two particular sample shapes were used. The first was a thin cantilevered beam partially notched in the middle of its free end [3]. The second had a particular geometry which allowed flexural vibration to be converted into torsional deformation of the material. This technique allowed a deformation amplitude between 10^{-7} and 10^{-5} and a frequency range from 100 Hz to several kilohertz to be measured. The temperature could be varied between 20 and 350 K. An automatic data acquisition system enabled relevant parameters to be recorded directly: the frequency F, damping IF and absolute temperature T. It should be recalled that the relative elastic modulus variations are proportional to the square of the relative frequency variations [4].

The samples were prepared from highest purity monocrystalline (99.999%) and polycrystalline (99.9999%) rod. After shaping, the beams were annealed for 4 h in a high-vacuum furnace at a temperature of 540 K. Finally, the thin cantilevered beam was deformed by 5% by rolling at room temperature.

3. Results

Measurements in 5N lead were performed using the first specimen shape; a linear increase in temperature, at a rate of 1 K min^{-1}, was used with a deformation amplitude of 10^{-7}. Figure 1(a) presents the modulus variations $(F/F_0)^2$, where $F_0=232$ Hz is the value of the modulus measured at 22 K in the reference state. Figure 1(b) shows the internal friction IF as a function of the temperature T. Curves a in Figs. 1(a) and 1(b) represent the reference state of the sample, before cold working. Curves b, c, d, e, f and g in Figs. 1(a) and 1(b) correspond to different annealing temperatures. The annealing time at each temperature is equal to 1 min.

After plastic deformation at 22 K, obtained by flexion of the two free ends in opposite directions, an important decrease in the modulus (curve b in Fig. 1(a)) is observed. This softening can be annealed by increasing

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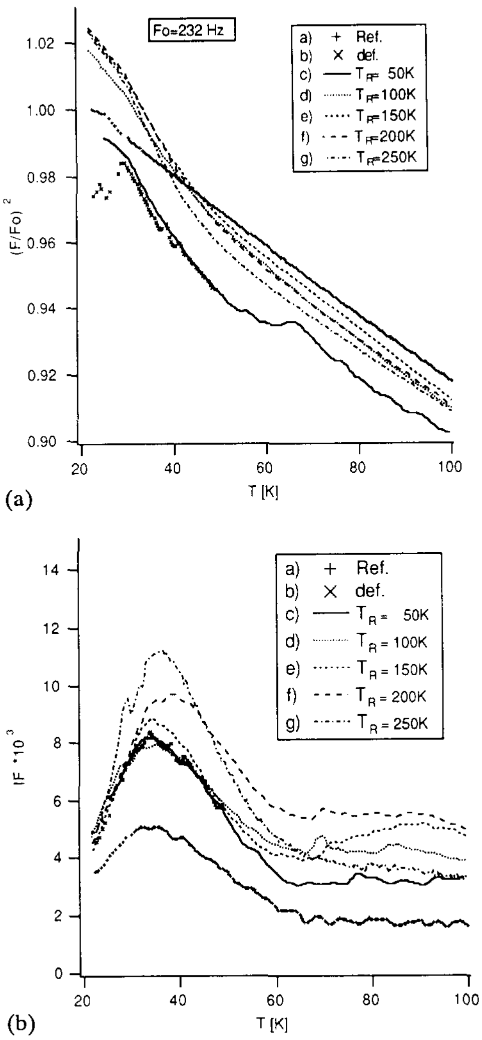

Fig. 1. Measurement of the modulus (a) and damping (b) in 5N lead in the reference state (curve a), cold worked at 22 K (curve b) and after annealing for 1 min at various temperatures (curves c, d, e, f and g).

the temperature of the sample as shown in Fig. 1(a): the modulus at 22 K increases with increasing annealing temperature (curves b, c, d and e) and remains constant after annealing at 150 K (curves e, f and g).

After cold working, the internal friction at 22 K is greater than before cold working (curve b in Fig. 1(b)). With an increase in the annealing temperature, this value remains approximately constant (curves c, d, e, f and g). However, the initial slope of the internal friction increases with increasing annealing temperature.

A relaxation peak and modulus defect appear at about 35 K. The height of the internal friction peak is constant up to an annealing temperature of 150 K (curves b, c and d). At higher annealing temperatures, the relaxation peak increases up to 250 K (curves e, f, and g in Fig. 1(b)). For an annealing temperature of 250 K, we obtain the greatest modulus defect and the highest relaxation peak (curve g in Figs. 1(a) and 1(b)). For higher annealing temperature, the relaxation peak and modulus defect decrease as shown in Figs. 2(a) and 2(b).

The first measurements of the modulus variations and internal friction in 6N lead for the second specimen shape are reported in Figs. 3(a) and 3(b). The same temperature programme was performed, but with a deformation amplitude of 2×10^{-5} and a frequency F_0 equal to 668 Hz. Curves a represent the reference state. Curves b, c and d correspond to different annealing temperatures (1 min) as before.

After cold working at 22 K, an important decrease in the modulus is observed (curve b in Fig. 3(a)). At the same time, the internal friction at 22 K is strongly increased and the relaxation peak at 38 K is not well marked (curve b in Fig. 3(b)). The relaxation peak develops as the annealing temperature is increased, with a simultaneous decrease in the internal friction

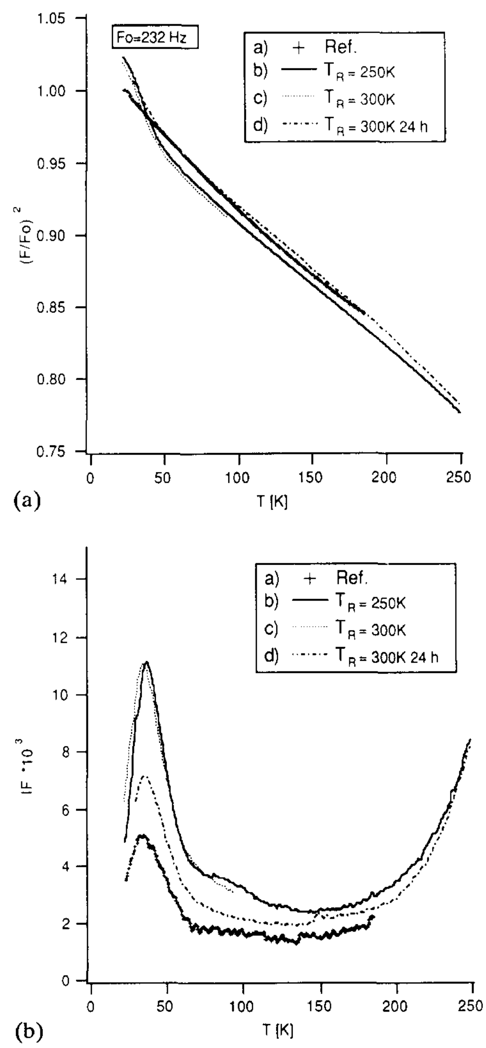

Fig. 2. Measurement of the modulus (a) and damping (b) in 5N lead, in the reference state (curve a) and at higher annealing temperatures (curves b, c and d). These results follow those shown in Figs. 1(a) and 1(b).

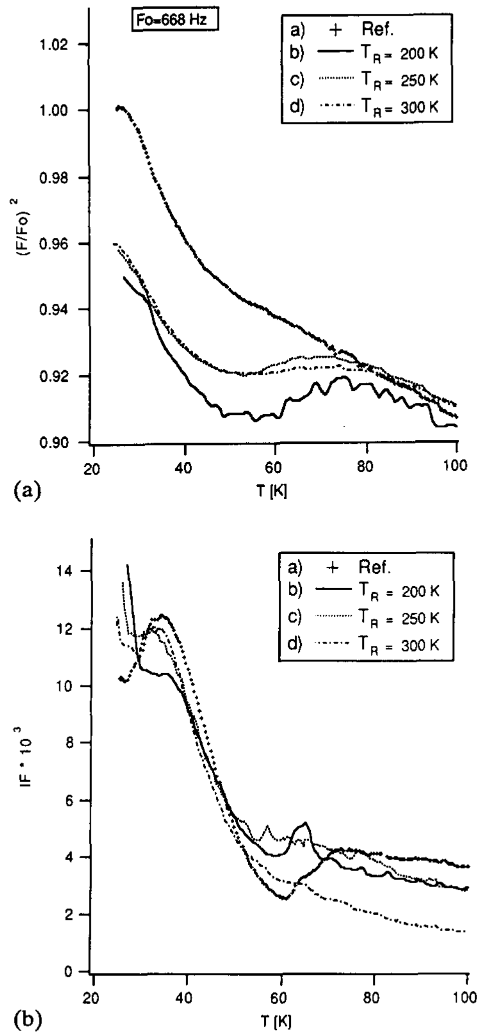

Fig. 3. Measurement of the modulus (a) and damping (b) in 6N lead. Curves a represent the reference state; curves b, c and d correspond to different annealing temperatures. These results were obtained using the second specimen shape. The sample is excited in a flexural mode, but material deformation takes place in a torsional mode.

from that at 22 K (curves b, c and d in Fig. 3(b)). The softening of the modulus at 22 K, induced by cold working, is also partially annealed at these temperatures (curves b, c and d in Fig. 3(a)), and a modulus increase (hardening) appears at 60 K.

4. Discussion

The effects observed in these cold-working experiments are very similar to those obtained by Lauzier *et al.* [5, 6] in very pure aluminium. After low-temperature plastic deformation, an important softening is induced: the modulus decreases and the damping increases. This behaviour has been attributed to lubrication of dislocation motion due to interaction of the dislocations with vacancy-type intrinsic point defects created during cold working; this leads to a short-circuit KPF mechanism [1]. Softening disappears during annealing.

A sudden strong increase in the modulus (hardening) is also observed for both samples. It takes place at a temperature of 28 K for the first sample (curve b in Fig. 1(a) and at 70 K for the second sample (curve b in Fig. 3(a)). These values are very different. This may be due to the fact that intrinsic point defect recovery in lead is strongly dependent on the initial defect densities, as shown by electrical resistivity recovery spectra performed in lead [7]. For a high initial density, only substage II appears in the vicinity of 70 K. In contrast with low initial defect densities, several substages II are present between 5 and 150 K.

Hardening of the modulus at 70 K is observed in curves c and d in Fig. 3(a) and is not well understood. It seems that weak plastic deformation occurs during cooling of the sample. The reason may be thermal dilatation due to the special geometry of the sample or the very high measurement amplitude used in this case.

Finally, the observation that the height of the relaxation peak increases when the low-temperature softening of the modulus is annealed demonstrates that Bordoni relaxation occurs [6]: the dislocation segments, which no longer participate in the lubrication mechanism (responsible for the low-temperature softening), contribute to the classical KPF mechanism by increasing the relaxation strength. An estimation of the activation energy gives a value of 0.04 eV with an exponential factor of 6.6×10^8 Hz. In comparison with Bordoni relaxation values in other f.c.c. metals these are much lower, but are of the same order of magnitude as those obtained by Wagner and Stangler [8].

5. Conclusions

After low-temperature plastic deformation, an important decrease in the low-temperature modulus is observed in 5N and 6N lead, as obtained previously in aluminium. This result is associated with lubrication of dislocation motion due to intrinsic point defects. Annealing of this softening occurs at different temperatures, depending on the cold working of the two samples. Finally, the evolution of the relaxation peak observed as a function of the annealing temperature demonstrates that Bordoni relaxation due to KPF occurs in lead at very low temperatures compared with other f.c.c. metals. In the future, measurements by the coupling method will be made to determine whether the measured signatures after low-temperature plastic deformation are effectively those associated with a short-circuit KPF mechanism, which has been called "the lubrication mechanism" of KPF [1].

Acknowledgments

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