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A Model for the Signatures Observed in 5N Lead by the Two Wave Acoustic Coupling Method

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Abstract: The ultrasonic low-frequency coupling method was used to study the relaxation processes in cold worked and annealed lead, in particular the Bordoni relaxation. New high-temperature signatures were observed in 5N lead on each temperature side of the high-frequency Bordoni peak. The signatures, measured in a temperature range where a non-negligible internal friction background is observed at low frequency, present a kind of "mirror symmetry" around the temperature of the high-frequency Bordoni peak. These signatures can be interpreted by a model assuming (i) that the high frequency motion of the dislocations is controlled by a thermally activated KPF (Kink Pair Formation) mechanism and (ii) that the low-frequency motion of the dislocations is controlled by a dislocation-point defects interaction, involving a LDWRF (Limited Displacement Without Restoring Force) mechanism. In this model, the KPF mechanism is responsible for the ultrasonic attenuation and for the mirror symmetry of the signatures. The LDWRF mechanism is responsible for the low-frequency internal friction background and for the characteristic shape of the signatures. An excellent correspondence between the experimental signatures and signatures numerically simulated with this model strongly suggests (i) that the Bordoni peak is due to a KPF mechanism and (ii) that the internal friction background is due to a LDWRF mechanism.

1. INTRODUCTION

Measurements performed in 5N lead in a temperature range between 50K and 250K, using low-frequency internal friction measurements (IF), ultrasonic attenuation measurements (US) and low-frequency ultrasounds coupling technique (US-LF), yield results (internal friction spectra and signatures) which can be coherently explained only if one introduces two dislocation motion mechanisms: the LDWRF (Limited Displacement Without Restoring Force) mechanism for the low-frequency motion, and the KPF (Kink Pair Formation) mechanism for the high-frequency motion.

2. EXPERIMENTAL TECHNIQUE

In order to study the dynamic properties of dislocations in lead after a low-temperature plastic deformation, a two wave acoustic coupling method was used. The samples were prepared from the highest purity mono- (99.999 %) and poly-crystalline (99.9999 %) lead rods. After shaping, these beams were annealed 4 hours in a high-vacuum furnace at a temperature of 540 K.

The *coupling technique* is a method for the study of dislocation dynamics [1]. The principle of this US-LF method consists of measuring the attenuation α and the velocity v of ultrasonic waves in a sample subjected to a permanent sinusoidal low-frequency applied stress σ . The closed curves $\Delta\alpha(\sigma)$ and $\Delta v(\sigma)$, measured during one cycle of the low-frequency stress, are plotted for different temperatures, maximum amplitudes of the low-frequency applied stress or times. The shapes of the curves $\Delta\alpha(\sigma)$ and $\Delta v/v(\sigma)$ and their time or temperature evolution are characteristic for each mechanism controlling the dislocation motion. For this reason, these curves have been called "signatures" of the dislocation motion mechanism. The technique allows one to measure automatically the attenuation and the relative change in propagation time of longitudinal ultrasonic waves with a sensitivity of 10^{-4} dB/ms and 10^{-6} respectively, between 4 K and 300 K. A mechanical system is able to generate a compressive stress of any waveform to the sample between 10^{-4} and 10^{-1} Hz with amplitudes from 0.1 to 50 MPa.

3. EXPERIMENTAL RESULTS IN 5N LEAD

Internal friction measurements performed in lead after cold working and annealing [2-3], obtained by a low-frequency (LF) flexural vibration technique and by a high-frequency (HF) ultrasonic attenuation technique, show that relaxation peaks appear at 40K and 150K respectively (Figs. 1a and 1b). The overall characteristics of these relaxation peaks correspond to the primary and secondary characteristics generally

associated with the Bordoni peak observed in the fcc metals [4-6], and it has been concluded that *the relaxation peaks observed in lead at low- and high-frequency are the Bordoni peaks* [2-3].

To study the mechanisms responsible for the Bordoni relaxation, experiments were performed using the US-LF coupling method. New high temperature signatures (Figs. 1c-1e) were observed on each temperature side of the high frequency Bordoni peak (150K). The *shapes* of these signatures are almost parallelograms. But, below and above the high-frequency Bordoni peak, the signatures turn clockwise and anti-clockwise respectively, which leads to a kind of "mirror-symmetry" with regard to the HF-Bordoni peak temperature. As it is shown in Fig. 1, in the temperature range (50k-250K) of these signatures, a non-negligible internal friction background is observed at low frequency in cold-worked and annealed lead (Fig. 1a).

Figure 1: Experimental results obtained from 5N lead after cold working and an anneal at 300K.

Figures 1a & 1b represent the internal friction measurements at 528 Hz and 18.5 MHz respectively. The peak relaxations observed at 40K and 150K respectively correspond to the Bordoni peak in lead.

Figures 1c to 1e show the corresponding experimental signatures measured at 76K, 176K and 209K respectively. The frequency of the low-frequency stress is 0.02Hz and the ultrasonic frequency is 18.5 MHz.

4. INTERNAL FRICTION BACKGROUND AND THE LDWRF MODEL

The low-frequency internal friction background observed between 50K and 250K seems to be athermal and hysteretic (does not depend on the temperature and the frequency), but depends on the thermomechanical treatments of the samples. In order to study it in more details, internal friction and frequency experiments as a function of the measurement amplitude have been performed at different temperatures [2]. Typical experimental results are displayed in Fig. 2. These results were obtained at a temperature of 150K in 5N lead after a cold working and an annealing at 350K.

The amplitude-dependant internal friction background presents two components: the first one is independent of the measurement amplitude at low amplitudes and the second one increases with the measurement amplitude at higher amplitudes. This increase of the internal friction background with the amplitude gives a

Figure 2: Internal friction as a function of the measurement amplitude in 5N lead after cold-working and annealing at 350K. The circles correspond to experimental data measured at 150K. The plain and dashed lines correspond to the theoretical results calculated with the LDWRF model and the breakaway Granato-Lücke models respectively.

Figure 3: Schematic representation of the motion of a dislocation inside a field of obstacle forces distributed on the glide plane under the effect of an applied stress, without strong pinning points (left) and with strong pinning points (right).

straight line if plotted in a Granato-Lücke plot, and can then be interpreted as a breakaway mechanism of the dislocations from immobile point obstacles segregated on the dislocation segments [2]. The Granato-Lücke breakaway model allows one to simulate exactly the experimental data, as shown in Fig. 2.

The part of the internal friction background which is independent of the measurement amplitude, at low amplitudes, is hysteretic and athermal. This background cannot be interpreted by the thermoelastic effect, because the measured IF (internal friction) values are largely higher than the theoretical thermoelastic IF values. But this kind of background can be perfectly explained using a dislocation-point defect interaction mechanism proposed by Gremaud [7-8] and called the "LDWRF" (Limited Displacement Without Restoring Force) mechanism.

If immobile point defects are distributed in the bulk and show a long range elastic interaction with the dislocations, one has to consider the existence, on the glide planes of the dislocations, of a field of forces of different intensities. When a dislocation is submitted to a given constant stress in the conditions represented in Fig. 3, the dislocation first moves freely through an average distance d . Then the dislocation begins to move inside the field of forces. Some of the smallest forces are crossed easily, but the higher forces cannot be jumped. The dislocation encounters more and more forces which cannot be overcome during its displacement and it is finally stopped. If the stress is reversed, there are no restoring forces acting on the dislocation, because no hard pinning points are considered here. The dislocation will begin to move in the opposite direction only when the applied stress becomes negative. When hard pinning points are considered (Fig. 3), the average displacement is controlled by the line tension or by the LDWRF mechanism. In this case, the average displacement $\bar{u}(\sigma)$ of the dislocation segment is typified by a hysteresis curve as shown in Fig. 4 [7-8].

Figure 4: The hysteresis curve of the average displacement versus the stress for a dislocation segment strongly pinned and submitted to a cyclic applied stress, when its motion is controlled by the LDWRF mechanism.

5. SIGNATURE MEASUREMENTS AND THE KPF MODEL

The new high-temperature signatures (Figs. 1c, 1d, 1e), which are obtained on above and below the high-frequency Bordoni peak (150K), are correlated with the hysteretic and athermal internal friction background observed in the low-frequency IF spectrum. In order to interpret these signatures, one assumes first that the hysteretic curve (Fig. 4) of the average displacement $\bar{u}(\sigma)$ of the dislocation segments, controlled by the LDWRF mechanism, is responsible for the shapes (parallelograms) of the signatures.

Figure 5: Motion of a dislocation inside a field of obstacle forces distributed on the glide plane under a low-frequency applied stress. The black points represent the unjumped strong forces and the white points the jumped weak forces. (a) Below the high-frequency Bordoni peak, the ultrasonic stress field can only induce small displacements of the geometrical kinks (kink chain model equations for the US attenuation on the left). (b) Above the high frequency Bordoni peak, the thermal activation allowed easy kink pair formations under the ultrasonic stress field (classical string model equations for the US attenuation on the right).

If one assumes now that a KPF mechanism is responsible for the HF-Bordoni peak, this KPF mechanism has also to be responsible for the "mirror symmetry" of the signatures. Indeed, the dislocation vibration generated by the low-amplitude ultrasonic stress is described by a geometrical kink chain model below the HF-Bordoni peak temperature (Fig. 5a), and by a classical string model above the HF-Bordoni peak temperature (Fig. 5b). This means that the ultrasonic stress field can only induce small displacements of the geometrical kinks at low temperature, and the ultrasonic attenuation $\alpha(\bar{u})$ decreases if the average dislocation displacement $\bar{u}(\sigma)$ due to the low-frequency applied stress increases (hardening of the kink chain). Above the HF-Bordoni peak, thermal activation leads to easy kink pair formation under the effect of the ultrasonic stress field, and the ultrasonic attenuation $\alpha(\bar{u})$ increases when the average displacement $\bar{u}(\sigma)$ of the dislocation due to the low-frequency applied stress increases. The combination of the effect of the high- and low-frequency stresses allows one to simulate the signatures. Figure 6 shows the experimental signatures obtained in the temperature range from 40K to 210K (a-e) and the corresponding signatures numerically simulated (f-j) by introducing $\bar{u}(\sigma)$ calculated from the LDWRF model inside $\alpha(\bar{u})$ calculated from the equation sets reported in Fig. 5.

For the simulation of the signatures, only one parameter has to be adjusted at each temperature: *the internal stress field*. In the case reported here (Fig. 6), one has to introduce a monotonical change of the internal stress field as a function of the temperature in order to fit exactly the experimental signatures. This means that this technique is also able to measure the temperature evolution of the internal stress field after thermomechanical treatments of the samples.

6. CONCLUSION

New signatures, measured in a temperature range where a non-negligible internal friction background is observed at low frequency, and presenting a kind of "mirror symmetry" around the temperature of the high frequency Bordoni peak, can be interpreted by a model assuming (i) that the high-frequency motion of the dislocations is controlled by a thermally activated KPF (Kink Pair Formation) mechanism and (ii) that the low-frequency motion of the dislocations is controlled by a dislocation-point defect interaction, involving a LDWRF (Limited Displacement Without Restoring Force) mechanism. The perfect correspondence between the experimental signatures and signatures numerically simulated with these models strongly suggests (i) that the Bordoni peak is due to a KPF mechanism and (ii) that the low-frequency amplitude independent internal friction background is due to a LDWRF mechanism.

Acknowledgments

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Figure 6: Experimental (figs. 6a - 6e) and simulated (figs. 6f - 6j) signatures obtained in 5N lead at different temperatures. The experimental signatures have been obtained after a cold working of 0.9% at 4K and a room temperature annealing.