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A NEW TECHNIQUE OF MEASUREMENT BY COUPLING OF A CYCLIC LOW FREQUENCY STRESS WITH AN ULTRASONIC WAVE

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Abstract.- By measurement of the low frequency internal friction spectrum, the different mechanisms which control the dislocation mobility, present very similar peaks. In the same way the microdeformation curves measured by the ultrasonic methods do not present great differences between different mechanisms.

To avoid this indetermination on the mechanisms, a new technique has been developed (1). It consists in measuring the attenuation $\Delta a$ and the relative change $\Delta t/t$ of propagation time of ultrasonic waves in a sample, with a permanent sinusoidal low frequency applied stress $\sigma$. Curves $\Delta a(\sigma)$ and $\Delta t/t(\sigma)$, each one measured during one cycle of the stress, are drawn as a function of time or temperature. These curves present different shapes and different evolutions depending on the involved mechanism. Some experimental results obtained by this technique are presented at this conference in two other papers (2,3).

In this paper, the measuring device will be presented.

1. Introduction.- In order to make the above-described measurements, a special device has been developed. This device is composed of four distinct parts:

a) a high precision ultrasonic echometer which is able to measure automatically the attenuation and the relative change of propagation time with a sensitivity of $10^{-4}$ dB/µs and $10^{-6}$ respectively, and with a measurement repetition rate of 10 kHz.

b) a mechanical system which allows to generate a compression stress of any waveform to the sample. Sinusoidal stresses can be generated between .001 and 2 Hz with amplitudes from 2 to 50 kg/cm².

c) a cryogenic system which allows to regulate the temperature of the sample between 4K and 300K.

d) an analog system of data acquisition which draws the curves $\Delta a(\sigma)$ and $\Delta t/t(\sigma)$, and also the curves $a(t)$, $\Delta t/t(t)$ and $T(t)$. The curves $\Delta a(\sigma)$ and $\Delta t/t(\sigma)$ are then treated on a Hewlett-Packard 9835 desktop computer.

This installation is constructed in such a way that it can be used during irradiation experiments. A magnetic field can also be applied to the sample.
2. The Ultrasonic Echometer.- An ultrasonic echometer working with a technique of echoes has been developed. The principle of the generator is presented in figure 2. A burst of ultrasounds (frequency $f_{US}$ between 4 and 40 MHz and duration $t_u$ from .5 to 2.5 $\mu$s) is emitted by a quartz transducer into the sample with a repetition rate from 1 to 10 kHz (figure 1). The same transducer receives the successive echoes coming from the sample. For each train of echoes, the attenuation $\alpha$ and the relative variation of propagation time $\Delta t/t$ are measured.

The generator (fig. 2) allows also to measure the absolute velocity of the ultrasounds with a high precision by the following technique: one or two bursts of ultrasounds are alternatively emitted into the sample. The time $t_{abs}$ between the two bursts is manually adjusted to obtain the exact superposition of the echoes coming from the two emitted bursts. This time $t_{abs}$ represents then the absolute propagation time of the ultrasounds.

To measure the attenuation, a system of synchronized electronic gates (c) allows to select manually two different echoes i and j (figure 3: $t_1$ and $t_2$ adjustable from 2.5 to 100 $\mu$s, $t_p$ adjustable from .3 to 5 $\mu$s). The peak amplitude (d) of the two selected echoes is measured and memorized in analog form. Then a logarithmic differential amplifier measures the attenuation in decibels. This signal is filtered by a low-pass filter and automatically calibrated in dB/$\mu$s (1 volt per dB/$\mu$s). Two separated circuits shift automatically the analog zero in order to plot very small variations of the attenuation.

The maximum sensitivity of the attenuation measurements is about $10^{-4}$ dB/$\mu$s.

To measure the relative variation of the propagation time, a one-shot pulse (b)
**Fig. 3: Principle of the attenuation measurement**

**Fig. 4: Principle of the measurement of the relative change of the ultrasonic time propagation**
Fig. 5: Principle of the stress device

of width $t_{cr}$ (between 0 and 100 μs) can be generated by steps of one nanosecond (fig.4). This pulse has high stability, better than 20 ps.

The emission of the burst of ultrasounds (a) is synchronized on the positive-going edge of the pulse (b). The width $t_{cr}$ of the pulse (b) is adjusted manually at the beginning of the experiment, so the negative-going edge of the pulse is exactly centered in the middle of the $n^{th}$ echo. A second one-shot pulse (c) is then generated, which begins at the negative-going edge of the first pulse and ends at the following zero-crossing of the oscillations of the $n^{th}$ echo. The width $\Delta t$ of this second pulse is proportionnal to the relative variation of the propagation time of the ultrasounds and is measured by a time-amplitude converter (d). The signal of the converter is memorized in analog form, then calibrated to obtain $10$ mV per ns. It is then filtered by a low-pass filter and transformed into the value of relative variation of time ($1$ volt for a variation of $10^{-2}$ of $\Delta t/t_{cr}$). Two separated circuits shift automatically the analog zero in order to plot very small variations of the relative change of propagation time.

The maximum sensitivity obtained on $\Delta t/t$ is about $10^{-6}$. To obtain such precision, it was necessary to stabilize the temperature of the electronic circuits at $70^{\circ}C$, with a stability better than $1^{\circ}C$.

During the measurements as a function of temperature, the variations of the ultrasonic velocity are relatively high and the time-amplitude converter is quickly out of range ($\Delta t < 100$ ns or $\Delta t > 300$ ns). To avoid this disadvantage, a system allows to follow automatically these variations by adding or subtracting 50 ns
Fig. 6: Examples of the two types of stress programs

Fig. 7: Example of experimental results showing an opening of the Δ\(t/t(\sigma)\) cycle

steps to the one-shot pulse (b) when the time-amplitude converter reaches one of the ends of its range.

3. The Stress Device.- The mechanical system is represented in figure 5. A compression stress can be applied to the sample (2) by two steel pieces (1). A motor of very low inertia (10) controls, through a train of gear-wheels (9) and micrometric screws (8), the displacement of a plate (6). This displacement is transformed in a strength on the sample by a linear calibrated spring (4). At the mobile extremity of the spring, a strength-cell (5) measures the stress on the sample. The mobile plate (6) is bound to the fixed plates by two steel bellows (7), which allows to make the preliminary vacuum inside the installation before work in a helium or nitrogen atmosphere (cryogenic fluids).

The electronic system of stress regulation allows to generate two types of stress programs:

a) linear stress ramps with stages (fig.6a) by a closed-loop regulation of the motor velocity through a tachymetric dynamo.

b) stresses of any wave form by a second close-loop regulation allowing to follow an analog stress reference, for example sinusoidal stresses (fig.6b).

4. Treatment of the Results.- The \(\Delta\alpha(\sigma)\) and \(\Delta t/t(\sigma)\) curves are drawn on a plotter at different temperatures. These results have to be treated in order to eliminate the electronic noise on the analog \(\Delta\alpha\) and \(\Delta t/t\) values due to the high sensitivity of these measurements, and also in order to eliminate the drift on \(\Delta\alpha\) and \(\Delta t/t\) values,
due to the fact that the absolute attenuation and velocity of the ultrasounds can vary quickly during an increase or a decrease of the temperature, which induces an opening of the $\Delta a(\sigma)$ and $\Delta t/t(\sigma)$ cycles (fig. 7).

This treatment is made by a program developed on a Hewlett-Packard 9835 calculator.

The experimental curves are digitized, which allows to eliminate the electronic noise. Then the program calculates the necessary corrections to close the $\Delta a(\sigma)$ and $\Delta t/t(\sigma)$ cycles, on the assumption that the drift due to the temperature is linear with the time on one cycle of the stress. Then the curves, automatically scaled, are drawn on a digital plotter. Examples of such results can be found in two other papers (2,3).

5. Conclusion.- A new measurement technique has been developed, which allows to plot at different temperatures the attenuation and the relative variation of propagation time of ultrasonic waves in a sample, as a function of a low frequency cyclic applied stress.

By this technique, $\Delta a(\sigma)$ and $\Delta t/t(\sigma)$ curves have been experimentally obtained on metal samples. They present different shapes and different evolutions, which seems to be characteristic of the microscopic mechanism controlling the dislocation mobility, as it is shown in two other papers (2,3).

References

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