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## STUDY OF DISLOCATION DYNAMIC BY ACOUSTIC SPECTROSCOPY

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**Abstract :** To study the dynamic properties of dislocations three new techniques are presented : the forced vibration pendulum, the ultrasonic coupling method, the continuous wave acoustic microscope.

### 1. Introduction

In order to study the dynamic properties of structural defects acoustic spectroscopy methods have been used in a very large frequency range ( $10^{-4}$  to  $10^9$  Hz). To improve the experimental analysis and to increase the range of application three new techniques have been developed and will be presented in this paper.

In the low frequency range ( $10^{-1}$  -  $10^5$  Hz) classical acoustic methods consist to measure the logarithm decrement  $\delta$  of the free vibration of a specimen previously excited at its resonant frequency. The Internal Friction (IF) is then given by  $IF = \delta/\pi$ .

As the frequency is approximately constant the acoustic spectrum has to be measured as a function of the temperature. Fig. 1(a) presents the mechanical relaxation process discovered by

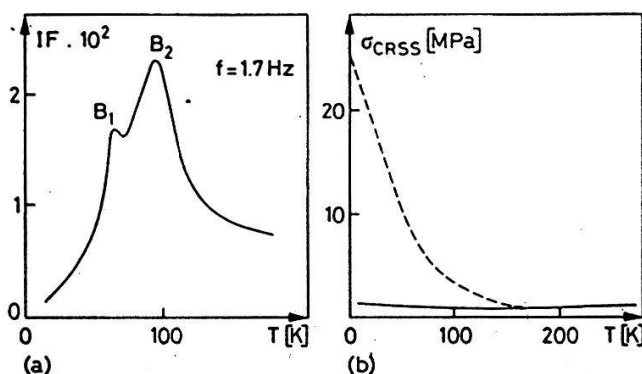


Fig. 1. (a) The Bordoni peak measured in 6N aluminium; (b) the critical resolved shear stress measured in pure aluminium. The dotted line represents the value of  $\sigma_{CRSS}$  which is theoretically expected from the Bordoni peak measurement.

Bordoni [1] in plastically deformed f.c.c. metals and attributed by Seeger [2,3] to the kink pair formation mechanism on dislocation (KPF). Nevertheless the discrepancy between the low temperature value of the critical resolved shear stress as obtained from the Bordoni Relaxation and from mechanical testing leads to some doubt about the interpretation of this Bordoni Relaxation (Fig. 1(b)) [3].

Fig. 1(a) corresponds to a typical results as obtained by classical IF measurements where the damping is measured as a function of the temperature. This technique allows to put in evidence different relaxation mechanisms, to measure the corresponding relaxation parameters (relaxation energy and attempt frequency) and, changing the experimental conditions, to attribute these relaxation effects to different type of defects (point defects, dislocations, grain boundaries, ..). Nevertheless, using this experimental technique to study the dynamic properties of defects, two type of difficulties appear:

(a) When the spectrum is measured as a function of the temperature, interferences appear between pure relaxation effects and every kind of other temperature dependent effects as annealing of defects, phase change,.. Moreover the relaxation mechanism itself could present a non trivial temperature dependent behaviour.

(b) Due to the similarities between different relaxation mechanisms an accurate interpretation of these peaks is not easy to do. Considering for example, the different mechanisms controlling the mobility of dislocations only non linear properties (secondary properties) are able to give informations on the real mechanism.

In the following two experimental technics are presented to solve these two problems.

## **2. Forced vibration pendulum**

Already a long time ago it was proposed to measure IF through the phase change between stress and strain using forced vibrations. The difficulties came from the high precision required to study IF in metals. Following different suggestions proposed by Woirgard new types of forced vibration pendulums have been

constructed. Fig. 2 presents a result obtained by D'Anna in Nb. Fig. 2(a) shows that the precision obtained with our forced pendulum is similar to the one obtained in classical free vibration pendulum. Moreover on Fig. 2(b), the IF is measured in isothermal conditions and the high temperature peak ( $\gamma$  peak) is observed as a function of the frequency.

From the Arrhenius plot an accurate value of the activation energy can be deduced [ $E = 0.62$  eV]. Moreover the forced vibration frequencies being very small ( $10^{-4}$  -  $10^1$  Hz), the temperature of measurement is also small. This allows to measure relaxation effects before the annealing of the defects.

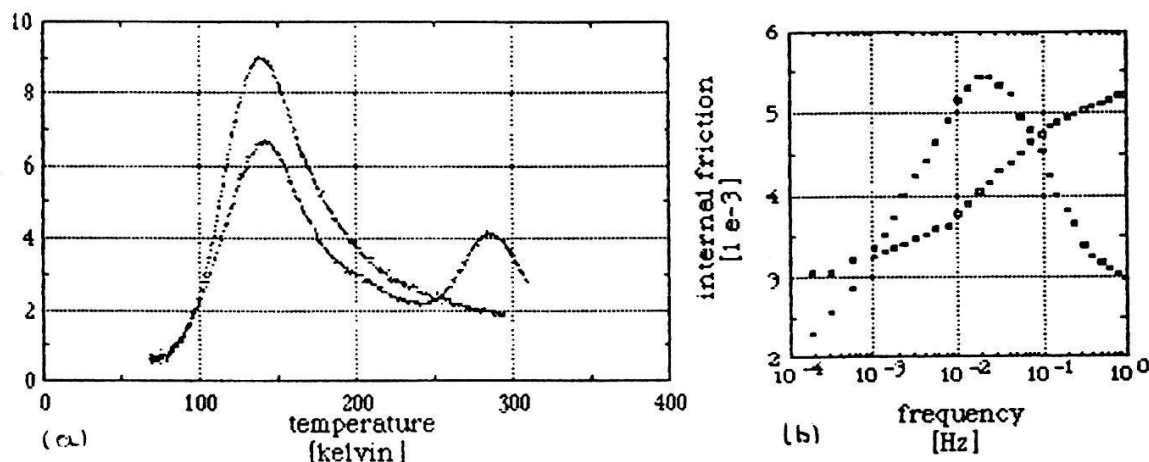


Fig. 2. (a) The hydrogen cold-worked peak (CWP-H) and the  $\gamma$  peak (higher temperature) measured by forced vibration; (b) The  $\gamma$  peak measured by forced vibration at 250 K versus the frequency.

### 3. Interpretation of the IF peaks by the ultrasonic coupling method [4,5]

By measurement of the low-frequency IF of f.c.c. and b.c.c. pure metals, several IF relaxation peaks have been observed. Some of them have been attributed to intrinsic mechanism (KPF). Other have been attributed to interaction mechanisms between dislocations and point defects. From IF measurements, it is not easy to distinguish between these different mechanisms. Ultrasonic experiments by the coupling method have been performed in order to obtain results qualitatively different when different relaxation mechanisms operate.

The principle of the ultrasonic coupling method consists in measuring variations of the attenuation,  $\alpha$ , and the velocity,  $v$ ,

of ultrasonic waves in a sample subjected to a permanent sinusoidal low-frequency applied stress (Fig. 3a). The closed curves  $\Delta\alpha(\sigma)$  [Fig. 3b] and  $\Delta v/v(\sigma)$  measured during one cycle of the low-frequency stress, are drawn for different temperatures. The shape of these curves and their evolution are characteristic for each mechanism controlling the dislocation motions. For this reason these curves have been called the signature of the dislocation motion mechanism.

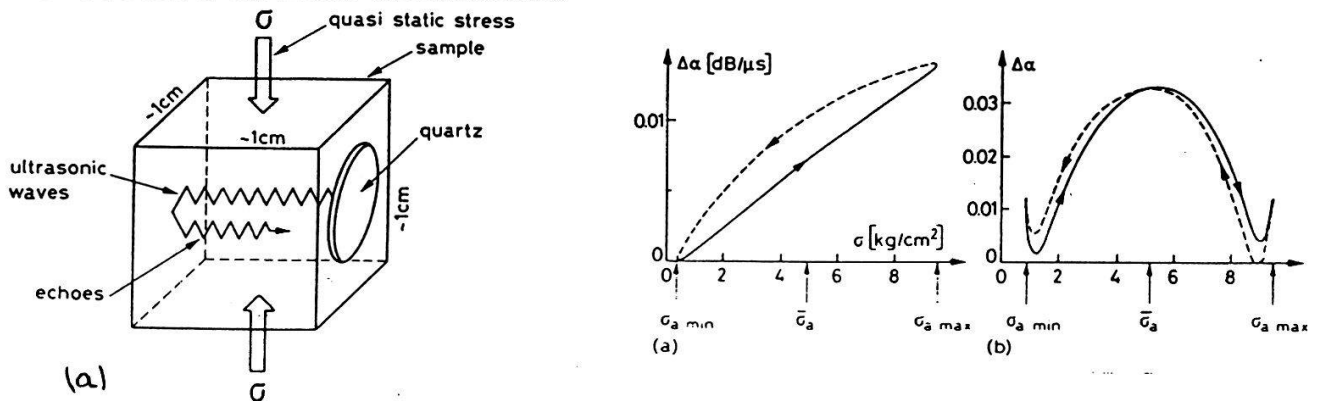


Fig. 3 (a). The principle of the measurement device using the two-wave acoustic coupling method; (b) Two examples of signature obtained in 5-N aluminium.

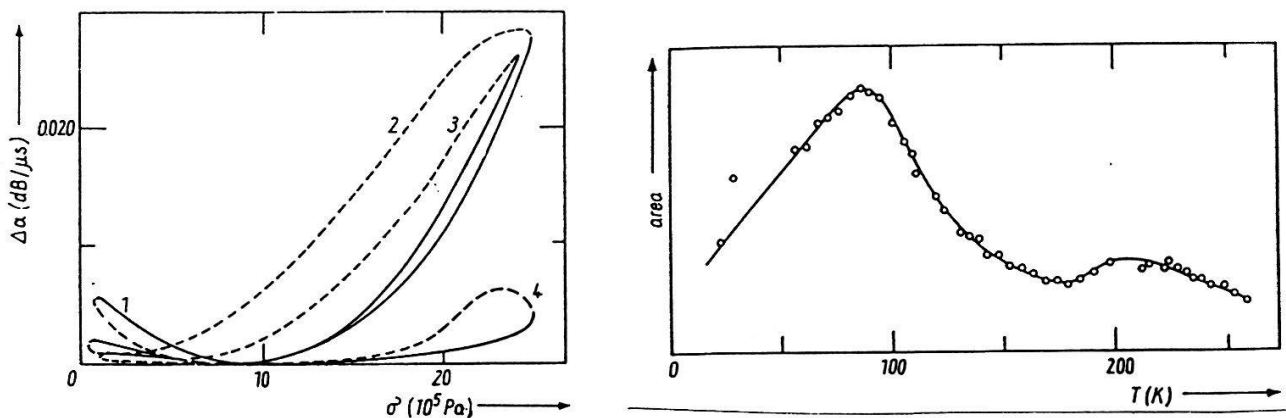


Fig. 4 (a). The BR signatures measured at different temperatures [(4) 39K, (2) 85K, (3) 217K]. The frequency of the applied stress is 0.02 Hz and the frequency of the ultrasounds is 8.5 MHz. (b) The surface of the BR signatures as a function of the measurement temperature.

The signature measured in a sample of 6 N aluminium in the temperature range of the Bordoni relaxation are shown in Fig. 4a. Bujard [5] has shown that in the case of the KPF mechanism, the enclosed area of the signature is proportional to the mechanical energy loss in the sample. Thus plotting these areas versus the temperature, Fig. 4b has been obtained. This graph shows a peak with a maximum at about 80 K, which is the attempt temperature for

the Bordoni peak at 0.02 Hz. Furthermore, using the KPF mechanisms, the shape of the signature has been calculated and clearly the observed shapes are very similar to the calculated one.

The results strongly support the interpretation of the BR by the KPF model.

#### **4. Continuous wave acoustic microscope [6]**

In order to study the anelastic properties of thin films or of small samples a new technique has been developed using ultrasonic waves. This technique is very similar to conventional acoustic microscopy. Conventional acoustic microscope use very short acoustic pulses. But in order to preserve the pulse shape ultrasonic receivers must have wide bandwidth, what in turn raises the problem with poor signal to noise ratio. Using continuous wave (CW) an acoustic microscope has been developed by Kulik on the basis of a network analyzer. It conserves the advantages of the short pulse spectroscopy, but has an excellent signal to noise ratio. It allows to perform quantitative measurements of velocity and attenuation of the local surface acoustic wave by measuring the reflectance as a function of :

- the frequency  $f$  of the ultrasounds. The fast Fourier transform (FFT) calculation gives a time domain resolution sufficient to separate the different reflections (Equivalent short pulse spectroscopy),
- the height  $z$  of the lens (CW  $V(z)$  curve). Velocity and attenuation of the surface acoustic wave are directly obtained by FFT.

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