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AUTOMATIC SYSTEM FOR MEASUREMENTS OF INTERNAL FRICTION AND ELASTIC MODULI**M. WELLER and E. TÖRÖK****Max-Planck-Institut für Metallforschung, Institut für Werkstoffwissenschaften, D-7000 Stuttgart, F.R.G.***Institut Straumann AG, CH-4437 Waldenburg, Switzerland*

Résumé - On a développé un système universel et automatique, avec un barreau vibrant en résonance, qui est contrôlé par un microordinateur, pour mesurer le frottement interne et les modules d'élasticité dans une gamme de températures étendues de 10 K à 1300 K. La décroissance des oscillations (100 Hz - 30 KHz) est détectée par une méthode électrostatique avec modulation en fréquence et stockée en appliquant une conversion A/D (12 bit) rapide. Cela rend possible des mesures de frottement interne dans une gamme de $Q^{-1} = 10^{-6}$ jusqu'à 10^{-1} . Des exemples expérimentaux sont présentés pour montrer la puissance et l'universalité du système.

Abstract - A versatile computer controlled automatic resonant bar system has been developed for measurement of internal friction and elastic moduli in a wide temperature range of 10K to 1300K. The decaying oscillations (100 Hz to 30 KHz) are detected electrostatically by the frequency modulation technique and stored using high speed buffered 12 Bit A/D conversion enabling internal friction to be measured in the range $Q^{-1} = 10^{-6}$ to 10^{-1} . Examples of measurement are presented to show the capability and versatility of the system.

I - INTRODUCTION

The resonant bar technique is well known for a long time as a classical method for the investigation of the elastic properties of solids. The resonance frequency of a sample excited to mechanical vibrations is one of its eigenfrequencies. These are determined only by the mode of oscillation (flexural, torsional or longitudinal), the appropriate elastic moduli, and the dimensions and density of the sample. By exciting the sample to distinct oscillations (at the different temperatures) the corresponding elastic moduli (Young's modulus E or shear modulus G) are obtained. Simultaneously the internal friction may be determined from the decay of the amplitude of the eigenvibrations. A variety of different types of apparatus is described in literature (see e.g. /1-3/).

In this paper we describe a versatile experimental set up which is especially characterized by a wide temperature range (10K to 1300K), high vacuum, and a variety of sample geometries (bars, wires or ribbons) which may be used. Also the newly developed, automatic computer controlled measuring system allows rapid and precise measurements of frequency and internal friction in a wide range ($Q^{-1} = 10^{-6}$ up to 10^{-1}).

II - MECHANICAL PART

1.) **Sample holder.** - Usually specimens with 40 or 50 mm length and rectangular cross section (5x1 mm) or wires with 1 mm diameter are excited to flexural vibrations in the free-free mode. This is shown schematically in Fig. 1 (front view) and Fig. 2 (lower part, side view). The sample, mounted horizontally, is supported directly at the nodes of oscillation by two thin wires (W_1, W_2) of 0.3 mm diameter, one nickel

and the other chromel acting *simultaneously* (together with the sample) as the thermocouple for temperature measurement. Each wire is guided through rollers mounted on a stainless steel frame (A1,A2) (Fig. 1 and 2) thus forming a loop around the sample. One end is electrically isolated and pulled up with a spring, the other end goes through plate B. The distance, a , between the two frames (A1) and (A2) can be adjusted according to the sample length, L , in order to match the distance of the nodes of oscillation.

A special sample holder is used for thin ribbons. The sample is clamped only at one end between two jaws which are mounted in part A1 and thus oscillates as a cantilever beam (vibrating reed). In this case temperature is measured by inserting another thermocouple (Philips thermocoax) into the jaws close to the sample.

2.) A view of the total mechanical arrangement is shown schematically in Fig. 2. The sample holder from Fig. 1 (P, A1, A2) is connected by four long rods R with plate B resting on the basal cube C which is mounted on a base plate BP. This vertical arrangement enables different tubes with furnaces or cryostats, to be positioned below the cube C depending on the required temperature range.

The *standard* arrangement as shown in Fig. 2 is designed for temperatures between 77K and about 850K. The sample holder is surrounded by the heating cage H (Philips thermocoax heater). S1 and S2 are heat shields containing holes for the supporting rods R, the thermocouple wires W1 and W2 and the carrier rod of the excitation electrode E. The entire assembly is surrounded by a double walled tube (T) with 72 mm inside diameter and 750 mm length which is mounted from below to the basal plate BP at flange F. This tube may be evacuated (for heating to higher temperatures) or filled with exchange gas (N_2 , He) when immersed into a dewar with liquid nitrogen for lowering the temperature to 77K. The electrode rod, E, (isolated at part I) may be moved up and down with the aid of micrometer M which is connected with the aid of a clutch and gear to the DC motor D.

For operation at *lower temperatures* the heater H and the outer parts of the shields S1, S2 are demounted. The surrounding tube T is then replaced by a liquid helium cryostat with 63 mm inside diameter (Leybold Heraeus GmbH, Köln) enabling temperatures from about 10K up to 650K.

A modified mechanical set up is used for *high-temperature* measurements in the range from RT up to 1300 K. The sample holder (P,A1,A2) and envelope tube T are constructed from high temperature materials (Inconel), and parts of the supporting rods from aluminium oxide. Instead of Ni/NiCr, Pt/PtRh thermocouple wires are used. Heating occurs by a furnace (Kanthal Co., 2 KW) positioned around the lower part of tube T. Radiation fins are positioned between plate BP and the furnace.

In all cases measurements are carried out in a vacuum of 10^{-5} to 10^{-6} mbar, which is also required for the high voltage excitation.

III - EXCITATION AND MEASURING SYSTEM

The basic principle of the system is represented schematically in Fig. 3. It is subdivided into the *excitation and detection* system (upper part) and the *computer controlled measuring system* (lower part).

Excitation and detection of the sample oscillations is based on the well known electrostatic principle (see e.g. /1-3/). A single electrode is used for excitation and pick up of the oscillations as was used by Bordoni /4/, Paré /5/ and others /6-8/. Amplitude detection occurs by using the frequency modulation (FM) and demodulation technique. The distance between the sample and the electrode varies with the change in temperature. This is kept constant by the geared DC motor driving a micrometer head connected to the electrode rod (see Fig. 2).

The vibrations are excited and maintained constant in a feed back system by an AC voltage in the range of 50-300 V, which is superimposed on a DC polarisation voltage (150-450 V). Particular modes of oscillation (fundamental vibration or higher tones) may be selected with the aid of a newly designed Chebyshev bandpass filter system with selectable centre frequency and bandwidth. Torsion oscillations of samples

(with rectangular cross section) may be excited by placing the electrode displaced sideways with respect to the main sample axis.

The measuring and control system is based on the standard IEEE 488-1978 (HP-IB) interface bus with a Hewlett-Packard (HP) series 200 computer as controller and with the usual peripheral units (floppy disc, plotter and printer).

The thermovoltage of the sample is monitored by a standard (high resolution) DVM (HP 3478A). Since the sample is supported directly by the thermocouple wires (W1, W2) the temperature is measured instantaneously at the sample. The temperatures T are calculated by comparison with a calibrated Pt-100 resistance and (corresponding) polynomial fits (accuracy $T = \pm 0.1$ to 0.3 K, depending on the temperature range). Temperature variation and control occurs by a temperature controller TC 8101 with power supply (Telbit GmbH, Stuttgart).

The frequency f is measured with an universal counter (Philis PM 6654) with a temperature stabilized quartz crystal. The resolution in the kHz-ranges is $\Delta f/f \approx 10^{-7}$.

The internal friction, Q^{-1} , is determined from the freely decaying amplitudes after switching off the excitation amplifier with a relay switch which is controlled by the universal interface (UI 488, Telbit).

The decaying oscillation is monitored with the aid of a rapid A/D converter with 12-bit (3 1/2 digit) resolution. For this purpose we use a HP 6942A multiprogrammer with a 69751A plug-in card enabling up to 3.3×10^4 readings per second and 4K-byte buffered memory (500 kHz and 64 kByte optional). Other technical solutions to digitize the amplitude decay are conceivable (e.g. transient recorders or storage oscilloscopes). The advantage of our system is that it is computer controlled and may thus be tailored to widely differing situations by software programming: (i) Digitizing of the decay curves may be adapted to the level of damping. High damping requires a high reading rate within a small number of oscillations, for low damping just the reverse is necessary. (ii) The digitized and stored decay data are accessible to multiple mathematical treatments, fitting procedures etc. to determine the internal friction, Q^{-1} , of the decaying oscillation, or its amplitude dependence.

Fig. 4 shows as an example the amplitude decay for a sample with high damping ($f = 3.06$ kHz). (It relates to the peak damping in Fig. 5 at ≈ 480 K, $Q^{-1} = 1.6 \times 10^{-2}$). The internal friction is determined by integrating over every positive and negative half-period of the oscillation followed by fit of an exponential decay. This method allows the internal friction within a wide range from up to $Q^{-1} = 10^{-1}$ down to 10^{-6} to be determined with high accuracy. Fig. 4 shows additionally a Fourier transform (FFT) of the decaying oscillation.

The elastic moduli of the sample are obtained from the theory of elastic vibrations (Bernoulli-Euler and Timoshenko beam theory). From flexural eigenvibrations of (isotropic) rectangular bars, with thickness h , width b length l , and density ρ , Young's modulus, E , is obtained from the frequency of the fundamental mode, f_1 , as given by Spinner and Tefft /9/

$$E = 0.94642 \rho l^4/h^2 (f_1)^2 F \quad (1)$$

The correction factor F depends on the shape and is given for a Poisson ratio of $\nu = 0.3$ as

$$F = 1 + 6.596(h/l)^2 + \dots \quad (1a)$$

For our standard sample dimensions, $b = 5$ mm, $h = 1$ mm the correction factor is $F \approx 1.0041$ for $l = 40$ mm, and $F = 1.0026$ for $l = 50$ mm. The nodes are positioned at $0.224 l$ and $0.776 l$.

From flexural vibrations of cylindrical specimens with diameter d , E is obtained from the frequency of the first mode /9/.

$$E = 1.261886 \rho l^4/h^2 (f_1)^2 F_1 \quad (2)$$

The correction factor is calculated in /9/ (with $d = 1$ mm and $\nu = 0.3$: $F_1 = 1.0021$ for $l = 50$ mm; $F_1 = 1.0036$ for $l = 40$ mm).

The *shear modulus*, G , of rectangular bars is derived from torsional resonant frequencies, f_n , as /9/

$$G = (2\pi f_n/n)^2 R \quad (2)$$

The shape factor may be approximated for $h/b \ll 1$ as /9,10/

$$R = (1+(b/h)^2)/(4-2.521 h/b) \quad (2a)$$

(For $b = 5$ mm and $h = 1$ mm, $R = 7.437$). The second torsion mode ($n = 2$) has its node positions at 0.25% and 0.75%, i.e. close to the node positions for the fundamental flexure mode. It may thus be excited in our apparatus with the same wire positions by sideward displacement of the electrode.

For further informations on elastic vibration of bars such as vibration frequencies, node positions, correction factors etc. we refer, e.g., to /1,3,9,11/.

IV - EXPERIMENTAL EXAMPLES

Figures 5 to 7 illustrate the applicability and versatility of the apparatus.

Fig. 5 shows the temperature dependence of the internal friction, Q^{-1} , and Young's modulus, E , for a ceramic sample, ZrO_2 doped with 3% Y_2O_3 (Y-TZP), from ref. /12/. The sample with dimensions of 40x5x1 mm was oscillating in flexure (fundamental mode, $f \approx 3.06$ kHz). One side was covered with silver paint. E was calculated with eq.(1).

Fig. 6 shows measurements with a thin amorphous $Ni_{35}Ti_{65}$ ribbon about 30 μ thick and 3 mm wide which was clamped at one end and oscillating as a cantilever beam with a free length of 7 mm. The peak at 240K is correlated with the presence of hydrogen /13/.

Finally, Fig. 7 demonstrates measurements up to 1273K obtained with the high temperature sample holder obtained on a stainless steel (316L) sample /14/. Two subsequent runs are shown in Fig. 7a for the internal friction, in Fig. 7b, for the elastic moduli, E and G . In the first run of Fig. 7a, the peak at 170K is interpreted as a Bordoni type relaxation, the second one at 340K is attributed to interstitial solute atoms, probably carbon /14/.

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Fig. 1 - Sample holder

Fig. 2 - Mechanical part (schematically).

Fig. 3 - Block diagram of the excitation and measuring system.

Fig. 4 - Amplitude decay and FFT for a sample vibrating at 3.06 kHz.

Fig. 5 - Q^{-1} and E vs. temperature for $ZrO_2-Y_2O_3$ ceramics ($f \approx 3.06$ kHz).

Fig. 6 - Q^{-1} and f^2 vs. temperature for an amorphous $Ni_{35}Ti_{65}$ ribbon. ($f \approx 270$ Hz).

Fig. 7 - Temperature dependence of the internal friction (a) and the elastic moduli (E, G) (b) for a stainless steel sample.