THREE ULTRASONIC DEVICES FOR THE ELASTIC MODULI DETERMINATION AT HIGH TEMPERATURES

C. Gondard, M.-H. Nadal, C. Hermerel

CEA DRMN/MOS/MFC
F- 21120 Is-Sur-Tille
France

INTRODUCTION

The aim of this paper is the characterization of materials submitted to high temperatures (up to 3000°C). In this field, some mechanical tests are available the experiments involve a lot of critical measurements which depends on the applied strength and on the temperature. An alternative solution is an ultrasonic method which advantages are a non destructive mechanism and a possible determination of the elastic moduli for a very weak strength to deformation ratio [1]. In that field, to determine the material behavior at high temperatures by a non destructive method, we propose three different devices. Two of them are based on contact measurement with delay-lines working up to 1000°C and up to 3000°C respectively. The last device, which is composed of a laser excitation and an interferometric detection, constitutes a non contact method especially useful for materials submitted to temperature neir their melting point. This paper is divided into three parts. The first one presents the principle of the elastic moduli measurement. The second one describes the different experimental set-up and the last part gives some results on metallic and organic samples.

PRINCIPLE OF THE ELASTIC MODULI MEASUREMENT

Elastic Moduli Versus Ultrasonic Velocities

The isotropic material characterization consists in determining the Young modulus (Y), the shear modulus (G) and the Poisson's ratio (ν). These material parameters depend on the shear and the longitudinal velocities (v_S and v_L respectively) which variations versus the temperature θ can be measured. The elastic moduli and Poisson's ratio can be expressed as :

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$$Y(\theta)=\rho(\theta)\,v_S^2(\theta)\,\frac{3v_S^2(\theta)-4v_L^2(\theta)}{v_S^2(\theta)-v_L^2(\theta)},\quad G(\theta)=\rho(\theta)v_S^2(\theta),\quad \upsilon(\theta)=\frac{1}{2}\frac{v_L^2(\theta)-2v_S^2(\theta)}{v_L^2(\theta)-v_S^2(\theta)} \quad (1)$$

The density is always a function of the temperature so that

$$\rho(\theta)=\frac{\rho_0}{(1+\alpha(\theta)\theta)^3} \quad (2)$$

which depends on the linear dilatation coefficient $\alpha(\theta)$ of the material.

The principle of the experiment is based on the time of flight measurement (t) of the acoustic wave of velocity $v(\theta)$ through the sample along the distance $l(\theta)$:

$$v(\theta) = l(\theta)/t \quad \text{and} \quad l(\theta) = l_0(1 + \alpha(\theta)\,\theta) \quad (3)$$

where l_0 is the sample thickness at ambient temperature (and pressure).

Using a time of flight measurement method of the ultrasonic wave (corresponding to the longitudinal or the shear mode), it is assumed that the different pulse echoes which are analyzed keep the same time profile. This involves to study materials with low dispersion. However, high attenuation of the acoustic waves can be observed, which depends on the grain size or porosity of the material. That is why, experiments are matched to sample thickness of some millimeters.

Material Characterization in Temperature by a Pulse Echo Technique

The principle of the experiment is described on the Figures 1.a-b. An oven is used so that the main heating region is located on the sample. This one is located between two wave-guides. At the end of each one are connected two transducers with the same ultrasonic characteristics. The two wave-guides are used to ensure the propagation of the waves [2,3] through the sample and also to undergo the temperature gradient. Therefore, the thermal resistance of the wave-guides is sufficient to keep the transducers near the room temperature [4,5].

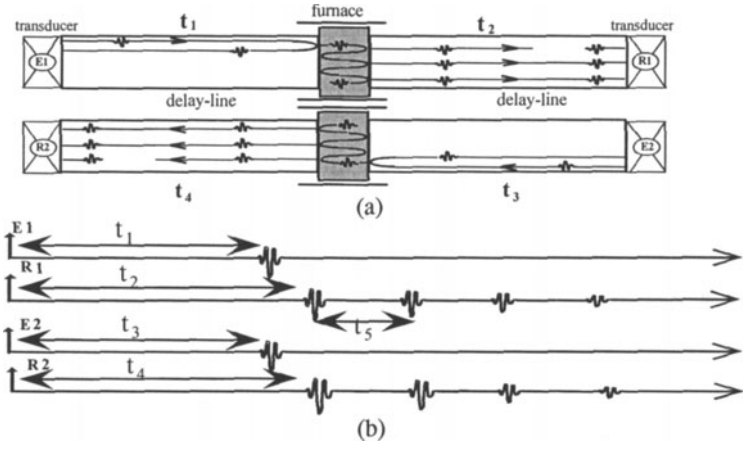

Figure 1. (a) Propagation of the ultrasonic waves in the delay-line/material/delay-line structure, (b) echograms detected by each transducer.

First, an acoustic pulse is generated by the transducer noted « 1 » (Fig. 1.a) and propagates through the wave-guides and then reaches the sample. Then, since the acoustic impedance of the wave-guide and of the sample is different, the acoustic wave is partially reflected back to the transducer « 1 ». A pulse echo is then detected at the time t_1 (Fig. 1.b). The transmitted wave crosses the sample and finally reaches the transducer noticed « 2 » at the time t_2. Then, by exciting the transducer « 2 », another couple of times of flight is detected at the times t_3 and t_4 (Fig. 1.b). The time of flight (t) of the ultrasonic waves in the sample is simply given by two different expressions :

$$t = \frac{1}{2}[\, t_2 + t_4 - (t_3 + t_1)\,] \qquad (4.a)$$

$$t = \frac{1}{2} t_5 \qquad (4.b)$$

The experiment is validated if the times of flight t_2 and t_4 are equal. If this condition is not fulfilled, it means that the temperature has changed between the two couples of acquisitions. In case of equivalent wave-guides and identical temperature gradient, the times t_1 and t_3 are also equal. This condition is not necessary. At the contrary, this method allows to ignore the temperature gradient and an eventual asymmetry of the heating without involving errors on the time of flight measurement.

Equation (4.a) allows the determination of (t) even if any reflection in the sample does not occur. This is particularly suitable to the characterization of material with a strong attenuation. The equation (4.b) corresponds to the available detection of multiple echoes in the material and leads to the best accuracy of the time of flight measurement and therefore of the elastic moduli.

This principle is based on the simple analysis of ray-path along the normal of the planar transducer. Then, the divergence of the acoustic beam is neglected. For a typical frequency of 5 MHz, with a 13 mm aperture, the near-field distance in Inconel is about 33 mm in the longitudinal mode (66 mm in the transversal mode respectively). Moreover, to protect the transducers against the temperature, the wave-guides have 330 mm length. For an incident longitudinal wave, it can be demonstrated that in such conditions, the divergence involves the generation of a lot of spurious echoes which amplitude has the same order of magnitude as the main echo corresponding to the direct path. Then, the A-Scan analysis are very difficult because the reflection echoes in the sample overlap with the spurious signals.

In order to avoid this mode conversion, the wave-guide has been screwed to create a grating surface. Then, when the acoustic beam reaches the surface of the wave-guide by divergence, the incident energy is not reflected in a particular direction but in all space, so that any pulse echo resolved in time can be created. Finally, the acoustic signal is mainly constituted by the echoes corresponding to the direct path, with a low noise due to the spurious echoes dispersion.

EXPERIMENTAL SET-UP

Contact Devices With Delay-Lines

The experimental set-up is based on the principle of measurement described in Figure 1.a. Cylindrical samples of 4.5 mm to 10 mm width and 23 mm diameter are

used. The 13 mm diameter transducers are 5 MHz from PANAMETRICS, in the longitudinal or shear mode. A computer controls the data record of the numerical oscilloscope. The temperature acquisition step is 25°C and the sampling rate is 500 MHz. Signal processing is performed for filtering the echoes and for an automatic time of flight measurement. For the first experimental device, we use two similar Inconel delay-lines which length is 330 mm, whereas in the second one, a more compact structure is realized with graphite delay-lines of 75 mm length. The screw steps of the delay-lines are fitted on each case with regard to the kind of transmitter used. All the experiments are performed with an inert atmosphere (Argon) to reduce the material oxidation. The coupling between the delay-lines and the sample is critical in this temperature range. Some special greases and graphite bonding are used.

Heating Kinetics

One of the most important differences between the two contact devices is the kinetic of heating. The first one, using a resistive oven involves a temperature rise about 5 hours to reach 1000°C. The free temperature decay takes 15 hours when the signal acquisitions are performed (Fig.2). The rise and the decay of the temperature are not controlled step by step. The rf heating is more efficient to rise the temperature in a few seconds up to 3000°C. The controlled temperature gradient depends on the oven power and on the matched coupling between the rf ring and the material. The temperature is measured by thermocouples in the first device and by a double spectrum pyrometer in the second set-up.

The advantage of the second device consists in two characteristics : the maximum of temperature is higher and the kinetic allow to reduce the effects of the temperature treatment undergone by the material. This last remark is particularly important with regard to some materials which are very sensitive to microstructure changes versus the temperature.

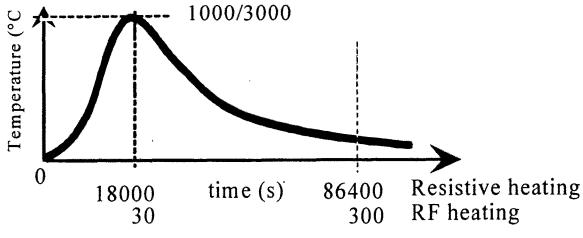

Figure 2. Heating kinetic comparing the resistive and the rf heating.

Non Contact Device - Laser Ultrasounds

The experimental set-up is composed of a Q-switched Nd:Yag laser operating at the wavelength of 1.064 μm and of a Mach-Zehnder heterodyne interferometer [6] (Figure 3). The probe is sensitive to out-of-plane displacement and its large bandwidth extends from 20 kHz to 30 MHz with a sensitivity less than 10^{-6} nm/$\sqrt{\text{Hz}}$ on mirror-like surfaces and about 5.10^{-5} nm/$\sqrt{\text{Hz}}$ on scattering surfaces. The laser beam is focused on the sample surface by a spherical lens to form a circular spot which is not entirely gaussian in space. The optical power density is adjusted in order to ensure a thermoelastic generation mechanism. The experiments are performed in a transmission mode (generation and detection on opposite sides).

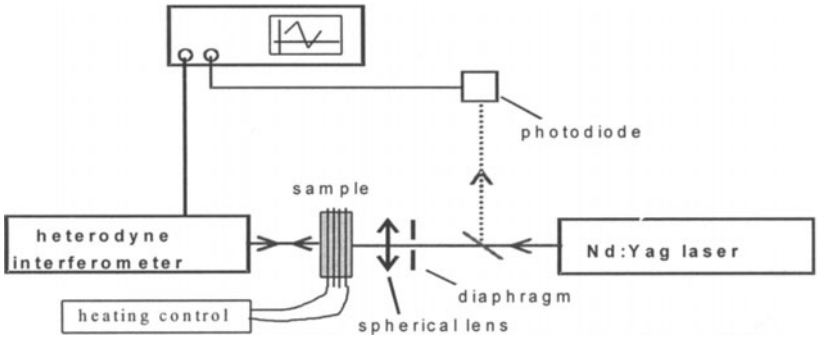

Figure 3. The Laser-ultrasound device, composed by a Q-switched Laser generation and a heterodyne-interferometer detection.

The temperature rise is performed by a heating wire. The data acquisitions are performed during the temperature rise, step by step (25°C). Some measurements have been also performed since the cooling to check the reversibility of the material behavior. The entire experiment takes a few minutes to perform the data acquisitions of materials between the ambient and 200°C. This experimental set-up is especially suitable to characterize some material of low melting temperature. In that case, a contact device would introduce a too important pressure on the sample and therefore a critical error on the evaluation of the distance passed through the ultrasonic waves [7,8].

EXPERIMENTAL RESULTS

Ultrasonic Velocity and Elastic Moduli Determination up to 600°C for Aluminum

The melting temperature of this aluminum alloy is about 650°C. The determination of the velocity decrease versus the temperature is given in the Figure 4. Because of the approach of the material melting temperature, the shear velocity decrease is not linear. The shear mode collapses progressively. The negative slope of this curve is 19% between the ambient and 575°C and is twice as much as the evolution of the longitudinal velocity in the same temperature range.

The Young modulus decreases when the temperature increases and changes between 69,7±2,3 GPa at the ambient and 45,6±1,5 GPa at 575°C (-35%). The shear modulus has quite the same behavior : 25,8±0,9 GPa at the ambient and 16,4±0,6 GPa at 575°C (-36%). At the contrary, the Poisson's ratio increases with the temperature to 0,353±0,010 at the ambient towards 0,386±0,007 at 575°C (+10%).

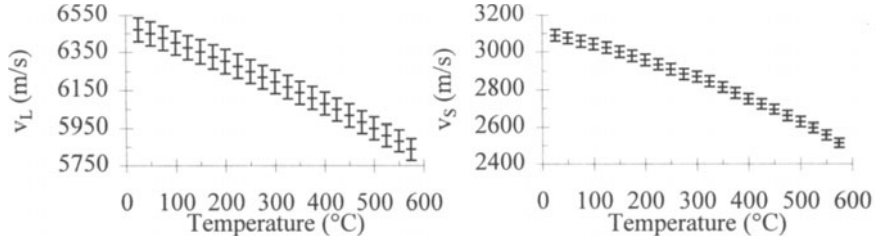

Figure 4. Evolution of the longitudinal and shear velocities between 20°C and 600°C.

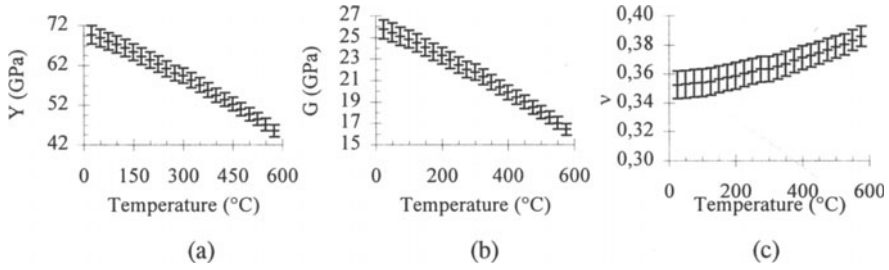

Fig. 5. Elastic moduli (a-b) and Poisson's ratio (c) variation of the tantalum versus the temperature

Elastic Moduli Determination up to 1000°C of Metallic and Organic Samples

The elastic moduli of tantalum have a quite linear decreasing whereas the temperature increases (Fig. 6). The slope of the Young modulus (shear modulus) variation is -3.1 10^{-2} GPa/°C (-1.3 10^{-2}GPa/°C). The slope of the increasing Poisson's ratio is 2.28 10^{-5}/°C. The relative uncertainty is 0.3 % at the ambient temperature and 1 % between 200°C and 900°C. It can be noticed that this method is able to measure accurately slight variations of the moduli in this temperature range. Data are not recorded between the ambient temperature and 200°C, because during the cooling, the coupling at the sample interface is not yet ensured.

The observed variation of elastic moduli of a graphite versus the temperature is inverted compared to a metallic sample behavior (Fig. 7) [9]. Then, the variation is given by a positive slope of 1.28 10^{-3} GPa/°C for the Young modulus and 9.1 10^{-4} GPa/°C for the shear modulus. The Poisson's ratio is quite constant up to 900°C. This corresponds to very slight variations, which is consistent with a refractory material usually used at much higher temperature.

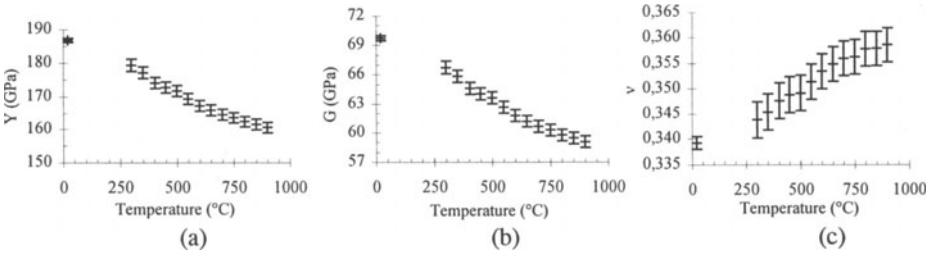

Fig. 6. Elastic moduli variation of the tantalum versus the temperature

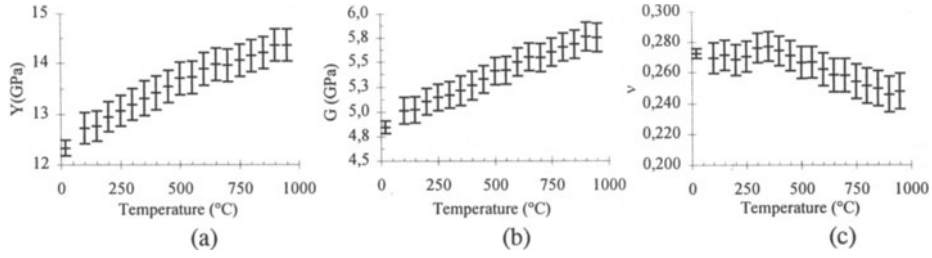

Fig. 7. Elastic moduli variations (a-b) and Poisson's ratio (c) of the isotropic graphite versus the temperature

Ultrasonic Signals up to 2500°C on a Graphite Sample

The preliminary experiments have been performed on the graphite sample already characterized up to 1000°C by the first contact device. First, we need to evaluate the difficulties due to the rf environment with regard to spurious effects on the detected signal. The frequency of the rf heating is less than 100 kHz and is therefore out of the bandwidth of the transducers. Nevertheless, the measurements are critical because of the difficulty of operating an accurate non perturbed and contactless temperature measurement. Moreover, the stabilization of the temperature is not yet controlled. This last point is under development.

The Figure 8 shows an example of ultrasonic signal (shear waves) measured at 2250°C on a graphite sample (10 mm thick). Multiple echoes are detected with a good signal-to-noise ratio in the sample and allow the shear wave velocity evaluation. The main signal is still disturbed by some spurious signals becoming from the divergence effects but they have a low amplitude. Dispersion phenomena are clearly point out.

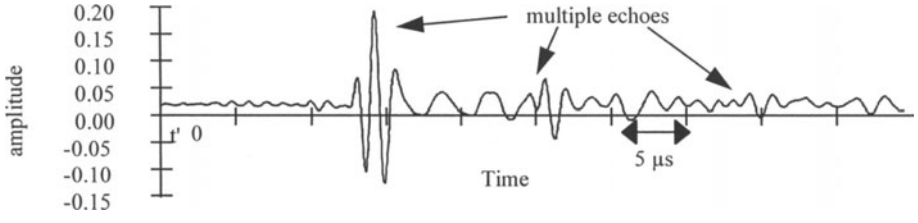

Fig. 8. Ultrasonic signal detected at 2500°C on a graphite sample

Pb Alloy Characterization up to 210°C (θ_f=250°C)

The Figure 9 shows the evolution of the ultrasonic signal versus the temperature when a Laser-ultrasonic system is used. The arrival of the Longitudinal waves (1L-precursor) and their multiple reflections (3L-5L) associated to the shear wave arrival (1T) are representative of an epicenter signal. Because of the low melting temperature of the Pb alloy, the deposited density of optical power governs the ablation regime of the ultrasounds generation. The time of flight of the waves increases with the temperature whereas the amplitude of the signal decreases. This last effect is significant of the Poisson's ratio decreasing [10].

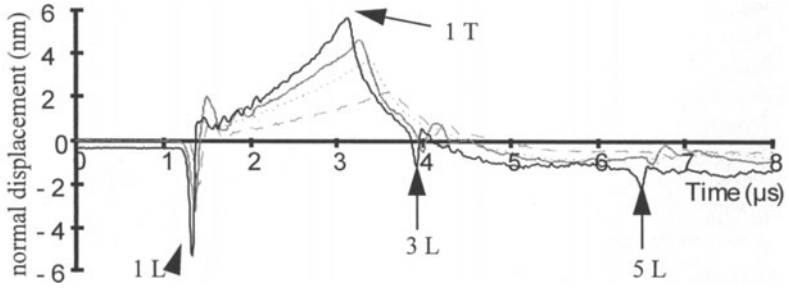

Fig. 9. Evolution of the laser ultrasonic signals versus the temperature.
—— 25°C —— 100°C ····· 150°C – – 200°C

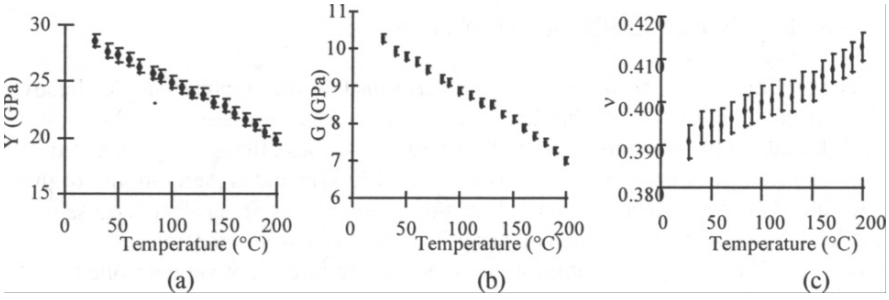

Fig. 10. Elastic moduli variation of a Pb alloy versus the temperature (a) Young modulus, (b) shear modulus (c) Poisson's ratio .

The Figure 10 presents the evolution of the elastic moduli of the Pb alloy. The decrease of the Young and the shear moduli is consequential (about 30% in this temperature range) whereas the Poisson's ratio increases.

CONCLUSION

In order to determine the material behavior at high temperatures by a non destructive method, we have proposed three different devices. Two of them are based on contact measurement with delay-lines working up to 1000°C and up to 3000°C respectively. The last device is composed by a laser excitation and an interferometric detection and then constitutes a non contact method especially useful for materials submitted to temperature near their melting point. In all cases, the principle of the method is to measure the velocity of the bulk waves in the solid, versus the temperature, which are linked to the elastic constants of the materials.

The use of a resistive or an rf heating involves different kinetics of temperature rise, which is particularly important with regard to the material behavior. This is applied to the characterization of an aluminum and a tantalum sample up to 600°C and 1000°C respectively. Moreover, the measurement of the ultrasonic velocity in graphite at 2245°C has been performed. The laser ultrasonic set-up has been used to characterize a lead alloy (melting temperature 250°C) for which the decreasing of 30% of the elastic moduli is determined up to 210°C. In all cases, the accuracy is in the order of the percent and the elastic constants are successfully compared to conventional other mechanical tests.

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