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Journal of Alloys and Compounds 444–445 (2007) 265–267

Journal of ALLOYS AND COMPOUNDS

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Shear modulus determination versus temperature up to the melting point using a laser-ultrasonic device

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Received 28 June 2006; received in revised form 21 September 2006; accepted 7 October 2006

Abstract

Shear modulus versus temperature $\mu(T)$ is determined by an innovating laser-ultrasonic facility. Bulk and surface wave velocities are measured versus temperature and compared. The analysis of the Rayleigh-wave is related to the shear modulus leading to a high sensitivity measurement independent of the thermal expansion coefficient. This last point is of great interest especially to establish the behavior of materials including phase transition associated with high volume changes. This paper focuses on results up to the melting point on tin, pure aluminum and an aluminum alloy.

Keywords: Ultrasonics; Material characterization; Laser-ultrasonics; Shear modulus; Rayleigh-waves

1. Introduction

The shear modulus μ is one of the parameters needed to implement the constitutive relations. These mechanical properties can be measured for a solid or a liquid with different ultrasonic methods [1–3]. This paper deals with the determination of $\mu(T)$ based on the bulk or surface wave velocity measurements performed by an original laser-ultrasonics device [4]. Indeed, due to its non-contact characteristic, this technique has demonstrated its potential for high temperature measurement [5]. The present work focuses on tin and aluminum as first trials. It could be used on plutonium in the future. Indeed, a special effort was made to measure the Rayleigh-wave velocity to validate the $\mu(T)$ determination independent of the thermal expansion coefficient.

2. Experimental set-up

This study is devoted to the characterization of white tin or aluminum samples obtained by cold rolling ($T_m^{Sn} = 505$ K; $T_m^{Al} = 933$ K) in the temperature range [300–900] K. The experimental set-up is composed of three main components: a Q-switched Nd:YAG laser operating at the wavelength of 1.064 μm to generate ultrasound; a Mach–Zehnder heterodyne interferometer [4] to measure the normal component of the mechanical displacement and a furnace with infrared lamps to control the temperature of the sample (1–10 K/min—temperature regulation better than 1/10 K) (Fig. 1). A sample is introduced inside a quartz cell located in a greater quartz tank filled by inert gas (Ar) to avoid oxidation effects, especially around the melting point.

3. Results

The principle experimental challenge is to determine the times-of-flight, respectively, of the bulk (longitudinal and shear polarization) or surface wave in the solid, and of the longitudinal wave in the liquid. Measurements are performed in a transmission or reflection configuration at the epicenter.

Bulk and surface wave measurements have to be distinguished: unlike the bulk wave velocities measurements, the surface wave velocities analysis does not need to correct the propagation distance with the thermal expansion coefficient. This last point a focus of this study: the propagation distance is independent of the material characteristics and is determined using a precise and easy location of the generation and detection beams.

We define in the solid:

- the longitudinal wave velocity (V_{SL});
- the shear wave velocity (V_{SS});
- the Rayleigh-wave velocity (V_R);

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doi:10.1016/j.jallcom.2006.10.054

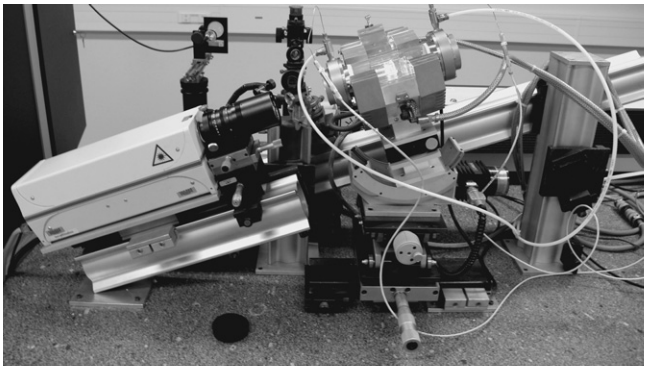

Fig. 1. Photograph of the experimental set-up.

and in the liquid:

- the longitudinal wave velocity (V_{LL}).

Considering isotropic solids, the bulk wave velocities and the Rayleigh-wave velocity are connected by the well-known Victorov relationship. Because of this, many of the results obtained here can be compared to those from direct surface measurements or via bulk results.

For tin (Fig. 2) these values of V_{SL} are in good agreement with the results of Nakano et al. [6], even if a difference exists around the melting point because of a shift in the measurement of the temperature (in the quartz cell and not in the sample). The shear wave attenuation, near the melting, leads to a crucial difficulty of the shear velocity measurement. The elastic moduli are deduced from the previous results by using the elasticity theory relations given for an isotropic solid. Furthermore, the density of the liquid tin is given by a linear function versus temperature.

For pure polycrystal metals, a $\mu(T)$ modeling is proposed, made of a continuous analytical expression over a large temperature range, up to the melting point (developed in Refs. [7,8]). Fig. 3 shows the comparison between some experimental data of the shear modulus versus temperature and the modeling. The drastic drop in $\mu(T)$ around $T/T_m = 1$ is described rather well by the modeling in a small temperature range.

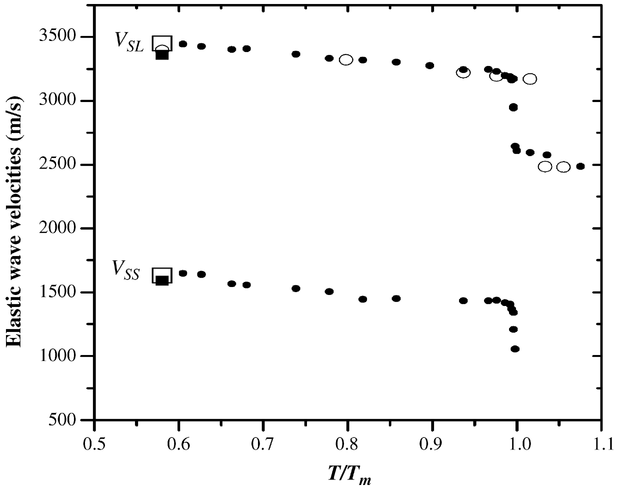

Fig. 2. Elastic velocities of tin vs. temperature up to the melting point, (●) this study, (○) [6].

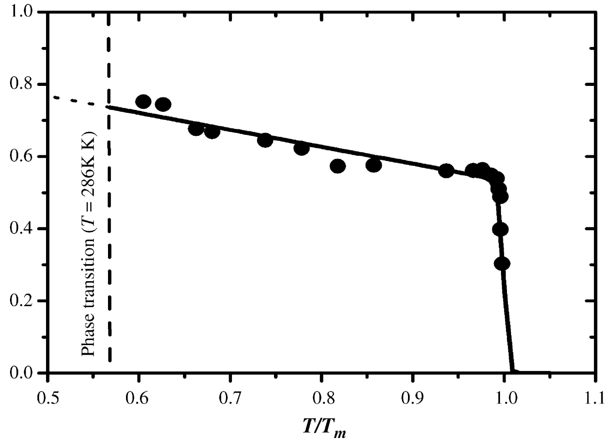

Fig. 3. Evolution of the shear modulus μ/μ_0 of tin vs. T/T_m up to the melting point: (●) experiment and (—) the model.

Concerning aluminum results, Fig. 4 shows the ultrasonic signals versus temperature. In that case, the study is focused on the Rayleigh-wave time-of-flight measurements. The ultrasonic signal is then composed of four main echoes. The first one corresponds to the head wave (longitudinal polarization noted P). The more efficient measurement corresponds to R_1 echo given by the Rayleigh-wave. This main echo is followed by two reflections due to the boundary conditions and noted R_2 and R_3.

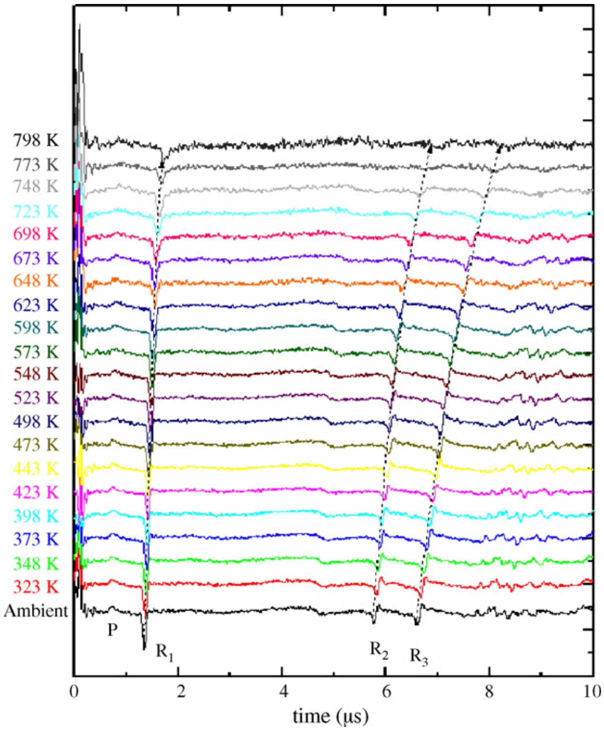

Fig. 4. Evolution of the ultrasonic signal vs. temperature. R_1 indicates the principal Rayleigh echo, R_2 and R_3 are reflections of R_1. P denotes head wave propagating with the longitudinal wave velocity.

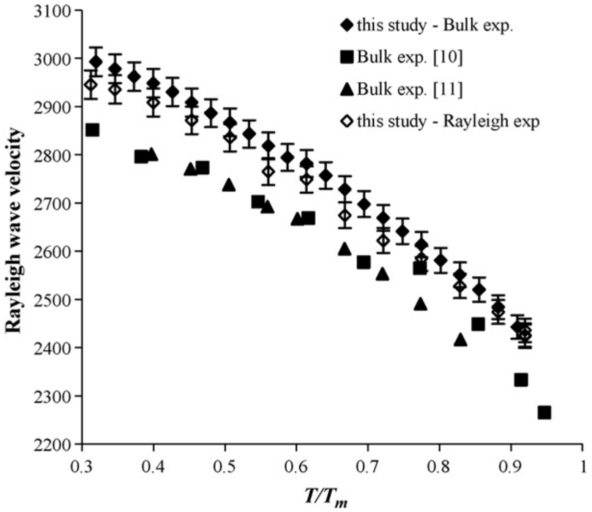

Fig. 5. Rayleigh-wave velocitiy evolution vs. temperature: comparison of direct Rayleigh-wave measurements and bulk measurements (our study and references [10,11]).

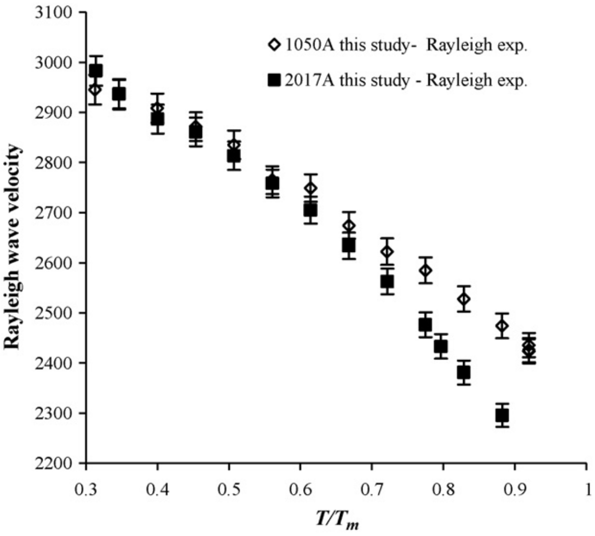

Fig. 6. Absolute comparison of Rayleigh-wave velocities of 1050A and 2017A.

By using the Rayleigh-wave time-of-flight measurements, we obtained a very accurate determination of the Rayleigh-wave velocity and therefore $\mu(T)$ for two main reasons: (i) sensitivity is high compared to bulk wave measurements and (ii) the Rayleigh-wave velocity determination is thermal expansion coefficient independent. Fig. 5 shows our results considering pure aluminum (1050A) characterization comparing bulk and Rayleigh-wave time-of-flight measurements (contact or non-contact ultrasonic method [9]) and literature (bulk measurements only [10,11]). First conclusion, bulk and surface wave measurements are in very good agreement, leading to the validation of the new concept of $\mu(T)$ determination. Secondly, the Rayleigh-wave velocity measurements are in good agreement with literature.

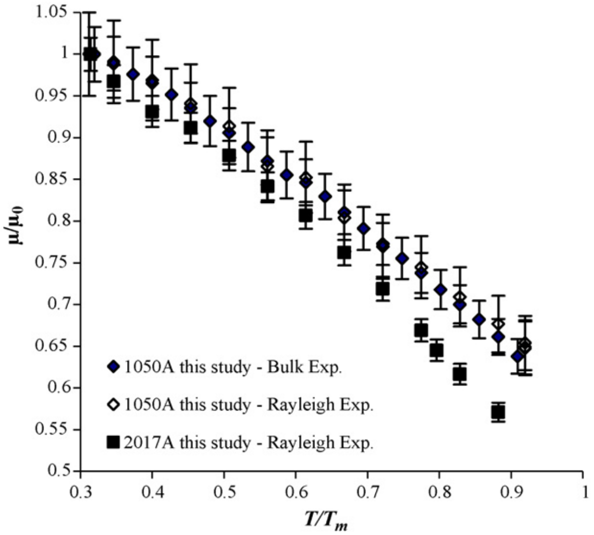

Fig. 7. Normalized shear modulus measurement comparing 1050A and 2017A.

Fig. 6 shows the absolute comparison of our surface wave measurements considering pure aluminum (1050A) and an aluminum alloy (2017A). Alloying appears to affect the result showing a split of $V_R(T)$ upon $T/T_m = 0.6$. The Rayleigh-wave of the alloy seems to fall more rapidly versus temperature than the pure metal. Therefore, the elasticity of the alloy decreases more rapidly versus temperature than the pure metal (Fig. 7).

4. Conclusion

This paper shows the high potential of laser-ultrasonics to study a solid to liquid metal behavior versus temperature. To our knowledge, $\mu(T)$ has been measured through this surface technique for the first time. Further work consists of two ways: performing Rayleigh-wave measurements around the melting point and for a metal exhibiting a phase transition. This method could then be applied to pure plutonium or plutonium alloys if the device can be implemented in a glove box.

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