Ultrasonics 54 (2014) 963–966

Contents lists available at ScienceDirect

Ultrasonics

journal homepage: www.elsevier.com/locate/ultras

Short Communication

Elastic characterization of platinum/rhodium alloy at high temperature by combined laser heating and laser ultrasonic techniques

K. Burgess [a], V. Prakapenka [b], E. Hellebrand [c], P.V. Zinin [a,*]

[a] *Hawaii Institute of Geophysics and Planetology, University of Hawaii, Honolulu, HI, USA*
[b] *Center for Advanced Radiation Sources, University of Chicago, IL, USA*
[c] *Department of Geology and Geophysics, University of Hawaii, Honolulu, HI, USA*

ARTICLE INFO

Article history:
Received 26 September 2013
Received in revised form 9 January 2014
Accepted 10 January 2014
Available online 22 January 2014

Keywords:
Laser ultrasonics
Surface acoustic waves
High temperature

ABSTRACT

We demonstrate an innovative pump–probe technique combined with laser heating to determine the velocity of a surface Rayleigh wave at high temperature. Laser ultrasonics in a point-source–point-receiver configuration was combined with laser heating to evaluate the elastic properties of micron size specimens. The measurements of the velocity of the surface Rayleigh wave (SRW) were conducted at 1070 K.

1. Introduction

There is widespread interest in the elastic properties of solids at elevated temperatures [1]. Much of the impetus for current research in this area has arisen from geophysical and geochemical studies of the Earth's mantle and core [2] and the need to understand the elastic properties of refractory materials and hard coatings [3]. Laser ultrasonics (LU) appears to be the most appropriate technique for determination of the acoustical properties of very small non-transparent solids and thin films at high temperatures [4,5]. Usually, these measurements are conducted inside a vacuum furnace [4,6], which makes it difficult to apply this technique to study the effect of high temperature on the elastic properties of small micron-sized specimens synthesized under high temperature. The most effective way to heat a small specimen is to use laser heating. In our study, we combined LU with advanced flat-top laser heating (LH) techniques [7,8] to study the behavior of the velocity of a Rayleigh wave (RW) in a PtRh alloy at high temperatures up to 1500 K. The choice of platinum based alloy was due to its very high chemical stability at high temperatures and its possible application as a transducer in laser ultrasonics measurements to study the acoustical properties of liquids and nanomaterials at high temperatures at ambient [9] and high pressures inside diamond anvil cells (DAC) [10,11].

* Corresponding author. Tel.: +1 8089569960.
E-mail address: zinin@soest.hawaii.edu (P.V. Zinin).

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http://dx.doi.org/10.1016/j.ultras.2014.01.011

2. Experiment

A sketch of the laser ultrasonics-laser heating (LU-LH) system is shown in Fig. 1. The system consists of five major components: (1) the LU-DAC system (probe and pump lasers, photo detector, and oscilloscope); (2) the laser heating system: a fiber laser (1064 nm) and a π-shaper, which is designed to allow precise control of the laser heating spot shape (e.g., gauss, flat-top, donut [8]) and size from 8 μm to ~100 μm, laser power is controlled by diode current remotely in the range from 2 to 100 W; (3) the spectrometer for measuring temperature of the sample (using a black-body radiation fit), Raman and fluorescence spectroscopy for pressure determination and sample characterization; (4) the motorized sample stage; (5) double side high magnification imaging based on two long working distance infinity corrected objectives.

In our configuration, the laser heating is applied to one side of the specimen (plate) and the velocity of the RW is measured on the opposite side of the specimen (Fig. 2). The experimental LU set-up is typical of many all-optical pump–probe systems [9]. We used a Nd:YAG laser from TEEM Photonics™, with a pulse width of 0.5 ns at a repetition frequency of 20 kHz, and 100 mW power at $\lambda = 1064$ nm as the pump laser for acoustic wave excitation and an oscilloscope ("Le Croy" 7300A, 3 GHz frequency band) for recording signals. The pump laser beam is focused by a Mitutoyo × 50 objective with a working distance of 20 mm to a size of 3 μm on the sample surface. We used a 532-nm, 150 mW Compass laser as a probe. The micro-mechanical system measuring the displacement d of the focal spot of the pump laser relative to that

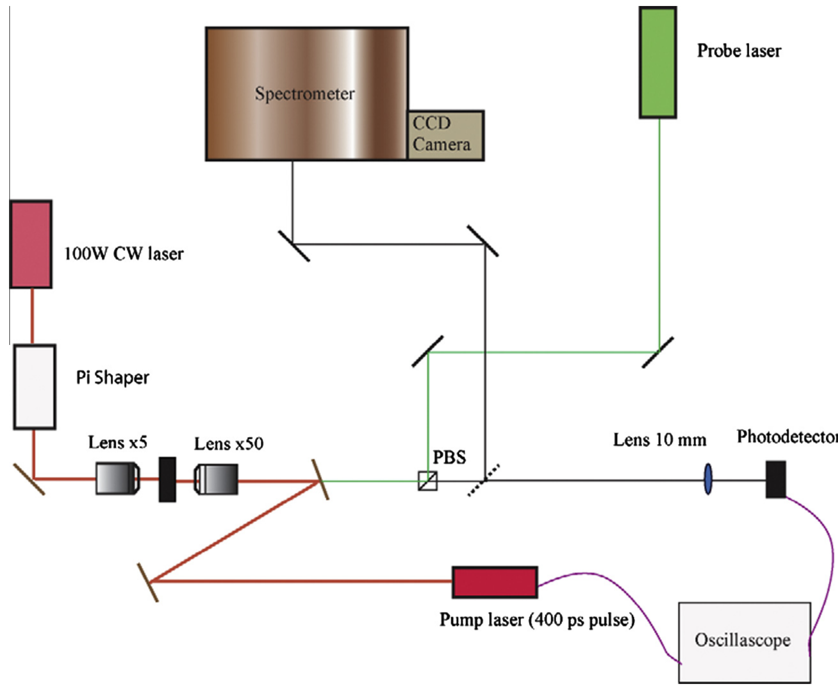

Fig. 1. (a) A sketch of the LU-LH system.

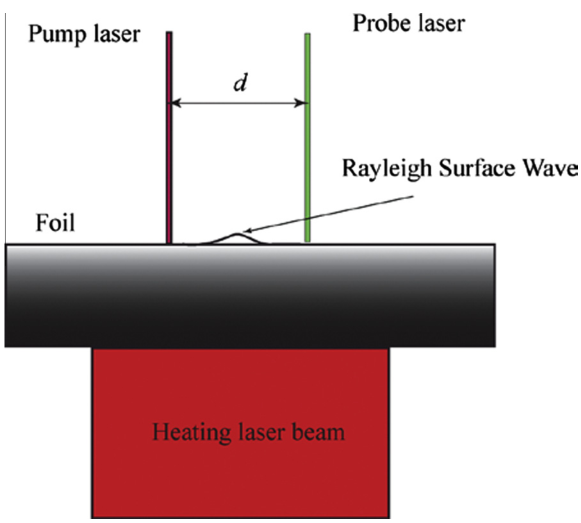

Fig. 2. A sketch of the SRW excitation and detection in the laser heated foil. Here, d is the distance between the pump and the probe laser beams.

of probe laser on the sample surface had been calibrated using preliminary optical measurements.

The elemental composition of the sample was determined using an electron microprobe analysis (EMPA) JEOL Hyperprobe JXA-8500F at the School of Ocean and Earth Science and Technology, University of Hawaii. In order to ensure a reliable result, a pure platinum powder was used as the standard for calibration of boron and carbon in the sample. The result obtained from the EMP measurements gives 18% rhodium (Rh) and 82% platinum.

3. Results

Fig. 3a shows signals detected by the probe laser in platinum at different distances at ambient temperature. The signals are dominated by pulses due to arrival of the surface Rayleigh wave (SRW) in the PtRh alloy, but the skimming longitudinal wave (SLW) was also detected (see insert in Fig. 3) up to $d = 30$ μm [12]. To measure the acoustic pulse arrival times, the positions of the peaks were determined as an average of the position of the maximum and minimum of the pulse. The velocities (V_R) of these waves are determined by using the arrival time (τ) and a simple equation (Fig. 3b): $d = \tau\ V_R$. The velocity of the Rayleigh wave in the platinum alloy was found to be 2.111 ± 0.040 km/s, and for the longitudinal velocity $V_L = 4.252 \pm 0.057$ km/s. The longitudinal wave velocity in pure platinum is $V_L = 3.260$ km/s, and the shear wave velocity is $V_S = 1.730$ km/s [13,14]. The Victorov's formula for the Rayleigh wave velocity can be used to estimate the value of the Rayleigh wave velocity in platinum, which is $V_R = 1.608$ km/s. For rhodium, we can estimate velocity of the SRW using data for velocities in the [100] direction, $V_L = 5.81$ km/s, and $V_S = 3.965$ km/s [15]. This gives an estimate for the Rayleigh wave velocity of Rh as $V_R = 3.5177$ km/s. If Pt and Rh contribute proportionally to the velocities of the SRW in Pt/Rh alloy then we would have $V_R = 0.82 * 1.608 + 0.18 * 3.51 = 1.988$ km/s for an 18% Rh platinum alloy. This is close to the value of the SRW, 2.111 km/s, which was obtained experimentally.

Fig. 4a shows signals detected by the probe laser in the PtRh alloy at different distances at 1070 ± 112 K. The main peak can be attributed to the SRW wave in the alloy. At high temperatures we were not able to determine pulses attributed to SLW (Fig. 4a). The velocities of SRWs are determined using arrival time τ and a simple linear equation (Fig. 4b). The velocities of the SRWs in PtRh alloy at high temperature were found to be 1.751 ± 0.074 km/s at 1070 ± 112 K.

4. Discussion

The penetration depth of the SRW at 2 GHz in the PtRh alloy, is around 1 μm. The temperature gradient, ΔT, over the thickness of $h = 1$ μm is equal to power per unit area transported by the alloy divided by the coefficient of the conductivity of the alloy. If the