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Combined laser ultrasonics, and Raman scattering in diamond anvil cell system operating in the transmission configuration

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Abstract. Recently it has been demonstrated that it is possible to detect both longitudinal and shear waves as well as measure their velocities for a non-transparent iron layer compressed in a diamond anvil cell (DAC) to pressures approaching 23 GPa by using laser ultrasonics (LU) technique. These experiments were conducted in the reflection configuration when point source and point receiver were located at the same side of the specimen. The purpose of the present study is to examine the generation and detection of the acoustical waves in the LU-DAC system operating in the transmission configuration. The experimental results obtained at 16.4 GPa presented in this article demonstrate that the LU-DAC technique can be applied for longitudinal wave velocity measurements in small specimens under high pressure in the transmission configuration.

1. Introduction

Understanding of the elastic behaviour of minerals under high pressure is a crucial factor for developing a model of the Earth's structure because most information about the Earth's interior comes from seismological data. More than fifty years ago, Birch proposed a simple empirical equation, Birch's law, that relates sound velocity to the density and mean atomic weight of the material the sound is passing through [1]. Because of this relation the velocity of sound can be measured in the Earth's interior as seismic waves travel from an earthquake on one side of the Earth to a seismograph on the other side. It can also be measured in the laboratory by sending ultrasound through samples at controlled pressures and temperatures [2]. Therefore, direct measurement of velocities and other elastic properties of minerals at elevated pressures and temperatures is the key to understanding the seismic information, allowing us to translate it into quantities such as chemical composition, mineralogy, temperature, and preferred orientation of minerals [3,4]. Recently it has been demonstrated that it is possible to detect both longitudinal and shear waves as well as measure their velocities for a non-transparent iron layer compressed in a DAC to pressures approaching 23 GPa by using LU in a point-source–point-receiver [5-7] or line-source–point-receiver [8] configurations. These experiments were conducted in the reflection configuration when point source and point receiver were located at the same side of the specimen. The purpose of the present study is to examine the generation and detection of the acoustical waves in the LU-DAC system operating in the transmission configuration.

2. Method

An experimental LU-DAC setup operating in the transmission configuration is shown in (figure 1). Schematics of the sample assemblage and of the sound wave trajectories in our LU experiments in a DAC are shown in figure 2.

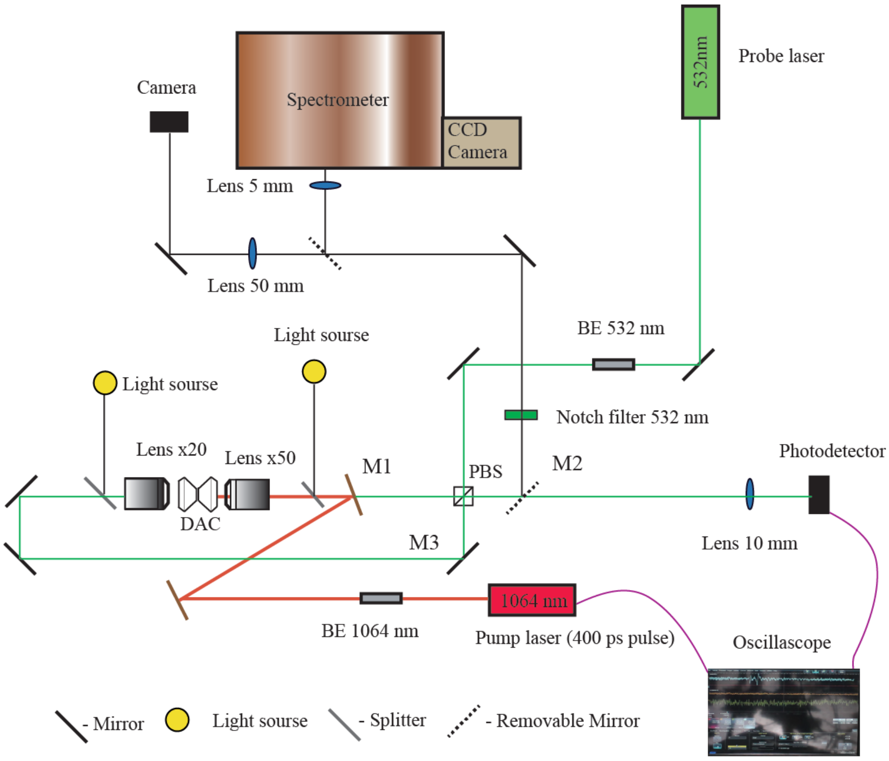

Figure 1. The sketch of the LU-DAC system operating in the transmission configuration. It consists of several components: (1) LU-DAC system (probe and pump lasers, photodetector, and oscilloscope); (2) the spectrometer for measuring Raman scattering for pressure determination; (3) the motorized sample stage; (5) double side high magnification imaging based on two long working distance infinity corrected objectives.

A flat iron specimen of about 150 μm in width is pressed between two diamond anvils (figure 2). The pump laser beam passes through a cylindrical lens in order to create a line-type source of material heating. The use of the beam expander (BE 1064 nm) allows shifting of the focal spot of the pump laser in z direction. The pump laser beam is focused by a Mitutoyo ×50 objective (Lensx50, figure1) with a working distance of 20 mm to a size of 3 μm on the sample surface. The position of the objective is fixed. The movement of the focal spot of the pump laser on the surface of the specimen in x direction in the x-z plane is achieved by rotating the M1 mirrors along the y axes. We do it remotely by using the compact piezo driven optical mount (NewPort, AG-M100L). The probe optical beam is from a continuous wave laser (Compass 3I5M, Diode-Pumped Laser, Coherent) with a maximum 150 mW power at 532 nm wavelength. For measurements of the surface acoustical waves, the photo-acoustic signals are detected by the photo deflection technique with a knife-screen, installed before the photo-detector [9]. We used a New Focus (model 1601) photo-detector with a 1 GHz bandwidth and a

2.5 GHz frequency bandwidth oscilloscope (WavePro 725ZI**,** LeCroy) to observe and record the signals. The use of the beam expander (BE 532 nm) allows shifting of the focal spot of the probe laser in z direction (perpendicular to the sample). The green light reflected from the specimen and passes through the polarized beam splitter (PBS). When the flip flop mirror (M2) is down, the reflected light after passing the PBS is focused by long distance focus lens (L5, f = 10 cm), on the sensor inside the Photodetector. For detecting acoustical waves inside the DAC only variation of the intensity of the reflected light is recorded. The LU-DAC system described above allows measuring velocities of the acoustical wave inside DAC in the transmission configuration, when the excitation and detection of the acoustical waves are on the opposite sides of the specimen in DAC (figure 2). In the transmission configuration, the probe laser beam is directed to the back side of the specimen in DAC through mirror (M3). The probe laser is focused on the sample by the objective, (Lens x20). Raman scattering system was used for determination of the pressure distribution over the specimen by measuring a shift of the diamond Raman line under pressure.

3. Results

The typical photoacoustic signal measured at 16.4 GPa is presented in figure 3. Following the notations introduced in [6,7], the L peak corresponds to the longitudinal wave (figure 2a). The peak marked as SL corresponds to skimming longitudinal wave [6]. As mentioned in [6], SL waves could combine, under certain conditions, with L waves leading to the so-called "head wave arrivals" (see SL-L peaks in figure 3).

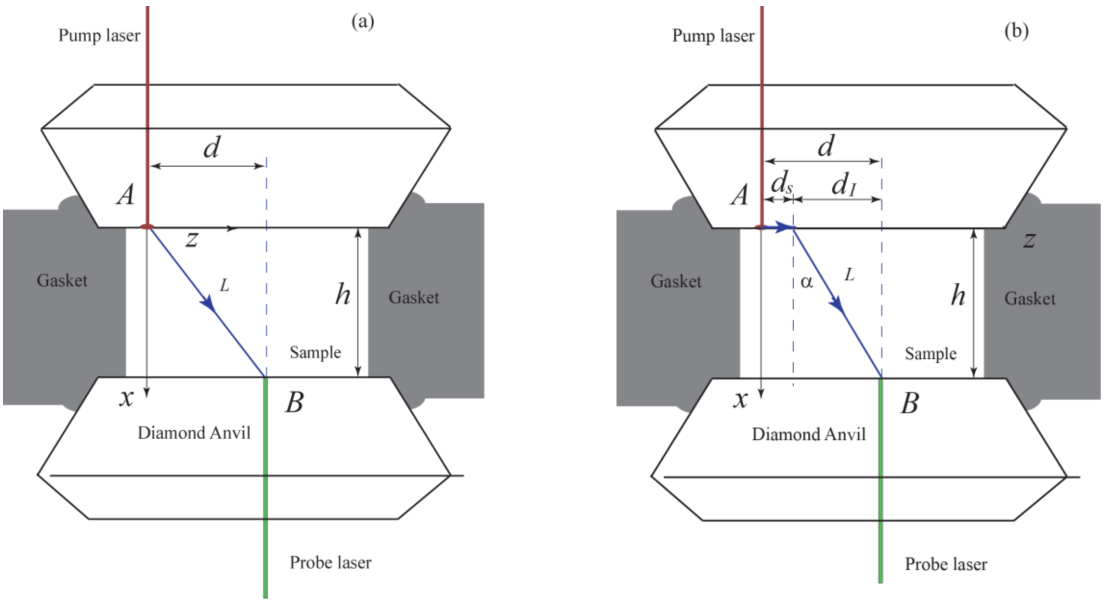

Figure 2. Sketch of the sound wave propagation in a flat iron specimen for the transmission configuration, where d is the distance between the points of the sound generation and detection; h is the thickness of the Fe-specimen. In the transmission case, the pump and probe lasers are incident to opposing side of the sample and the bulk wave travels through the material with no reflections. Excitation and detection of a number of different direct, reflected and refracted waves are possible using this technique.

As we can see (figure 3) only two waves have been detected (L and SL-L waves). Least square fits of two-parameters (sound velocity and sample thickness) to the experimental data $\tau(d)$ allowed us to determine the L-wave velocities in iron, and SL-L wave (figure 4). Time of flight of the L wave (τ_L) can be determined from a simple expression $\tau_L = 1/c_L\,(h^2 + d^2)^{1/2}$. Time of flight of *the SL-L* wave (τ_{SL})

has a simple form: $\tau_{SL} = \tau_I + \tau_S$, where $\tau_S = d_S/c_{SL}$, $\tau_I = h/\cos\alpha$, and α is the critical angle derived from the Snell's refraction law $\sin\alpha / c_L = 1/c_{SL}$.

Fitting of the L and SL-L wave arrivals at 16.4 GPa in the transmission configuration is shown in figure 4. Both the c_L and c_{SL} values of the correspondent waves in iron, determined in this work are in a good agreement with the results obtained earlier using the LU-DAC technique in the reflection configuration [6]. To our surprise the shear wave in iron was not detected in the transmission configuration.

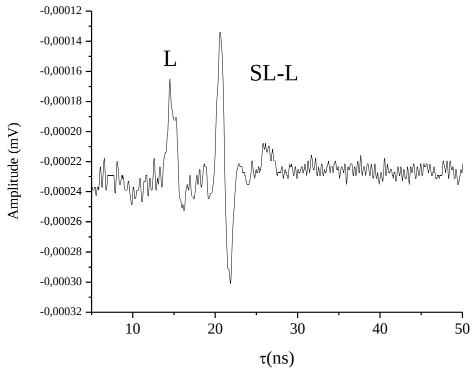

Figure 3. Photoacoustic signals measured at 16.4 GPa for $d = 41.5$.

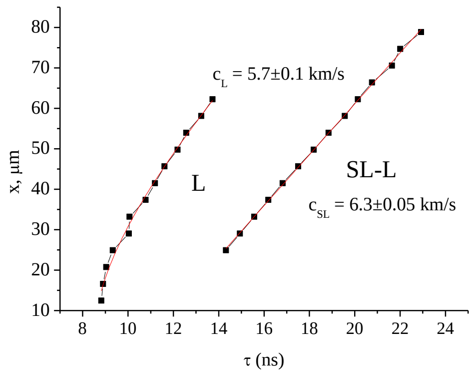

Figure 4. Fitting of the L and SL-L wave arrivals at 16.4 GPa.

4. Conclusion
The experimental results presented in this article demonstrate that the LU-DAC technique can be applied for sound velocity measurements in small iron specimens under high pressure in the transmission configuration. It was found that only longitudinal mode can be detected in this configuration.

Acknowledgement
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