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Evaluation of Elastic Properties of Iron in Diamond Anvil Cell by Laser Ultrasonics Technique

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Abstract. We propose an improved algorithm for the determination of shear wave velocity in iron under high pressure within the frame of an earlier proposed laser ultrasonics technique [1]. The modification is based on the observation that the signal identified at short propagation distances as a TT wave (the wave that is excited as the shear (T) wave at the diamond/iron interface and is reflected by the iron/diamond interface as a T wave) splits into two signals, a TT wave and another wave, which may be associated with a head wave, beginning from some threshold distance. This finding allows us to improve the accuracy of shear wave velocity determination. Statistical errors of the sample thickness and of the longitudinal and shear velocities measurements were found to be less than 2%.

1. Introduction
Understanding the elastic behavior 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. Measurement of the elastic properties of small specimens under high pressure and high temperature (HPHT) conditions is also important for synthesis of new superhard and functional phases [2]. The conventional way to measure elastic properties of non-transparent materials at high pressures is to use an ultrasonic interferometer either in a diamond anvil cell (DAC) [3] operating up to 10 GPa or in a large-volume press with a limited pressure range up to 20 GPa [4]. Another method is to use impulsive stimulated light scattering (ISLS) operating up to 115 GPa, but not allowing simultaneous determination of shear and longitudinal velocities of the isotropic solid [5]. Recently it was demonstrated that using laser ultrasonics (LU) in diamond anvil cells (LU-DAC) makes it possible to measure both longitudinal and shear velocities of a non-transparent iron layer in DAC at pressures up to 23 GPa [1]. The main advantage of the LU-DAC point-source-point-receiver technique for non-transparent amorphous solids and melts is that it does not require any additional data (such as thickness of the specimen in DAC) to determine the elastic properties of the iron at high pressures. We chose to use iron for two reasons. First, iron is a material in which acoustical waves are easily excited by a short laser pulse. Success in acousto-optic detection of sound waves in iron under high pressure
[1] opens the way to study elastic properties of other functional materials, such as superhard materials synthesized under HPHT conditions. A thin layer of iron can also be used as a transducer for opto-acoustical sound excitation and acousto-optical sound detection in DAC loaded with different non-transparent material. Second, the study of acoustical wave propagation in iron under high pressure has a direct application in geophysics [6]. In Ref. [1], five from the total number of detected opto-acousto-optic signals were described in detail. Among them there were the signals associated with LL, LT/TL and TT waves. It was found that, in order to determine shear velocity in iron under pressure, two peaks LT/TL (longitudinal-transverse/transverse-longitudinal wave) and TT should be fitted simultaneously. In this report, it will be demonstrated that the signal, which starts as TT wave, splits into two signals starting from some threshold distance between the pump and probe laser beams: TT wave and another wave which might be associated with a so called head wave. This finding allows us to improve accuracy of shear wave velocity determination. We will also demonstrate that in addition to the five waves described in Ref. [1] a lateral longitudinal wave and head waves can be detected. Measurements conducted at a pressure of 10.9 GPa will be used as an illustration of the application of LU-DAC technique for measuring velocities of longitudinal and shear waves in iron.

2. Experimental methods

Recently, Decremps et al [7] proposed combining the DAC device with the technique of picosecond laser ultrasonics in plane wave configuration to measure sound velocities under high pressure. An advantage of the picosecond laser ultrasonics is that it allows remote measurements of time of flight of the longitudinal wave inside the DAC.

![Figure 1. Sketch of sound waves propagation in the DAC. The following notations are introduced in the sketch: h is the thickness of the layer; d is the distance between the pump and a probe laser beams; x and z are Cartesian coordinates.](image)

When an ultra-short laser pulse, known as a “pump pulse,” is focused onto an opaque surface the optical absorption results in a thermal expansion that launches an elastic strain pulse. This strain pulse mainly consists of longitudinal acoustic waves that propagate directly into the bulk. Each time the strain pulse meets the sample surface it can be detected by a delayed laser pulse through the influence on its reflectivity [8]. Recent advances include the generation of picosecond shear waves using anisotropic materials [9] or small (~1 µm) optical spot sizes [10], and generation of terahertz acoustic waves in solids [11] and up to ~ 10 GHz in liquids [12]. The picosecond ultrasonic technique is now used widely for measuring elastic properties of submicron samples [13]. The disadvantage of the picosecond laser ultrasonics in plane wave configuration [7] is that the thickness of the specimen must...
be measured independently of longitudinal acoustic wave velocity and the equation-of-state must be
determined (i.e., using x-rays) to calculate the bulk and shear elastic moduli of a specimen. To
overcome this problem, using laser ultrasonics in diamond anvil cells (LU-DAC) in a point-source-
point-receiver configuration was proposed [1].

![Figure 2. Sketch of the experimental set-up.](image)

It was demonstrated that a point-source-point-receiver configuration allows detection and
measurements of both longitudinal and shear velocities of the non-transparent iron layer in DAC at
pressures up to 23 GPa [1]. A sketch of the LU-DAC experiment in point-source-point-receiver
configuration is shown in figure 1. The main idea of the LU-DAC technique is to determine
the specimen thickness in DAC by separating pump and probe lasers (figure 1). A pump laser beam
generates an acoustic pulse on the interface between specimen and diamond (point A in figure 1). In the
experiment, the reflectivity variations induced by the acoustic pulses reflected from the surface of the
specimen in DAC or propagated along the diamond/iron interface are detected by a probe laser (point D
in figure 1). Such an experimental set-up is typical to many LU all-optical pump/probe systems [14].

The time of flight, $\tau$, of the bulk sound wave from the point of excitation (point A, figure 1) to
the point of detection (point D, figure 1) separated at diamond/iron interface by a distance $d$ can be
described by a simple equation

$$c_\alpha^2 \tau_\alpha^2 = d^2 + 4h^2 \quad \text{where } a = L, T$$

where $c$ is sound velocity. The sound velocity and thickness $h$ of the sample under pressure can then be
obtained by fitting experimental data in a $(\tau, d)$ coordinate system.

In this study, the Nd:YAG laser (with a pulse width of 0.5 ns at a repetition frequency of 1 kHz, of
70 mW power at $\lambda=1064$ nm) was used for acoustic wave excitation. The light from the pump laser is
focused by a x20 objective with a working distance of 17 mm on the top surface of the sample inside
the DAC. A green laser (532-nm) with a power of 150 mW is used as a probe laser. An oscilloscope
(“Le Croy” 7300A, 3 GHz frequency bandwidth) was used to record the signals. The micro-mechanical
system measuring the displacement $d$ of the focal spot of the pump laser relative to that of the probe
laser on the diamond/iron interface has been calibrated through preliminary optical measurements. In
figure 2, mirror I redirects light reflected from the top surface of the sample in the DAC to the photo
detector “New Focus” with a bandwidth of 1 GHz through a lens with a focal distance of 10 cm. The
filter (HT-532 nm, HR 1064 nm) is used to cut the light from the pump laser. The imaging system for
visualization of probe and pump laser spots on the sample surface inside the DAC consists of an illuminating lamp, a set of splitters, and a CCD camera.

A symmetrical DAC [1] with a pair of type I brilliant-cut diamond anvils of 0.25-0.3 carat and a culet of 300-500 μm was used in these experiments. A stainless steel or Re gasket 250 μm thick was first indented to 25-40 μm, and then a hole of 80-150 μm diameter was drilled to serve as the sample chamber. Ruby chips distributed around the gasket hole perimeter served as pressure sensors. An adjustment made with four screws allows us to achieve less than 10% variation of the pressure inside the sample, 10.9 ± 1.0 GPa.

3. Results and Discussion

LU DAC experiments demonstrate that besides two skimming (lateral) and three bulk, which were described in Chigarev et al [1], multiple head waves (see definition in Choi and Dahl [15]) and weaker skimming waves can also be detected in iron under high pressure in the DAC. For example, a pattern of waves observed at 10.9 GPa is shown in figure 3. To measure the acoustic pulse arrival time, τ, the positions of the pulse peaks were determined. In figure 3, peaks SL_D and ST_D can be assigned to longitudinal and shear skimming bulk waves in diamond [16]. Linear fitting of the experimental data gives 18.58 ± 0.50 km/s and 12.86 ± 0.16 km/s for velocities of SL_D and ST_D waves respectively. We also marked a small dip in the signal that appears after the arrivals of SL_D and ST_D waves in figure 3. Linear fitting of the dip arrivals shows that it is associated with a wave traveling at a velocity of 5.69 ± 0.22 km/s, indicating that the dip can be assigned to the skimming longitudinal wave in iron (SL_Fe).

The next set of peaks can be attributed to the so-called LL wave that is excited as the longitudinal wave at the diamond/iron interface and is reflected by the iron/diamond interface as the longitudinal wave. Fitting of the LL peaks using equation (1) provides the thickness of the iron layer, h = 35.78 ± 0.32 μm, and the velocity of the longitudinal wave in iron: c_L = 6.17 ± 0.12 km/s. The value of the longitudinal velocity determined from fitting LL peak is slightly higher than that determined from fitting the SL_Fe dip. The value of the longitudinal wave determined from fitting LL peak is expected to be more accurate than that from SL_Fe dip because the latter is weak and can be traced only for a relatively short distance d.

**Figure 3.** The signals measured at different distances d. The step of the scan is 7.4 μm. The top signal was measured at d = 43.6 μm. Pressure was 10.9 GPa.

**Figure 4.** Fitting of the SL_D, ST_D, SL_Fe, LL, LT/TL and SL_Fe-TT wave arrivals at 10.9 GPa. Thickness of the sample is taken from LL measurement to fit LT and TT peaks.
The fourth peak is due to LT/TL acoustic mode conversion at the rear surface of the iron layer. Fitting of the LT peak with thickness $h$ and velocity of longitudinal wave $c_L$ obtained from fitting LL peak allows us to determine the shear velocity of the iron at 10.8 GPa: $c_T = 3.18 \pm 0.02$ km/s.

The fifth peak (along the TT - SL$_{Fe}$-TT direction in figure 3) was attributed to the arrival of the transverse-transverse (TT) wave [1]. Fitting all fifth peaks in figure 3 gives a shear wave velocity of $c_T = 3.08 \pm 0.25$ km/s in iron and $h = 33.60 \pm 0.56$ µm for thickness of the layer. The velocity of the shear wave in iron obtained from the fifth and LT peaks fitting are in agreement within experimental error, however, the difference in thicknesses is high and exceeds 10%; $h=35.78 \pm 0.32$ µm (from LT and LL signals fits) and $h=33.60 \pm 0.56$ µm (from fifth signal fits). The statistical error of the fifth peaks fitting (0.25 km/s) is also higher than that for the LT peaks fitting (0.02 km/s). To make the fitting self-consistent it was proposed that the velocity of the TT waves be determined by simultaneously fitting fifth and LT waves with fixed thickness obtained from LL fitting [1]. Analysis of the fitting of the fifth peaks revealed that with increasing pressure, the accuracy of fitting of the TT velocity diminishes. Moreover, subsequent repetition of the measurements conducted in Ref. [1] leads us to conclude that the discrepancy between the LT and TT fitting is intrinsic. Detailed analysis of the peaks associated in Chigarev et al [1] with fifth peak (figure 3) provides another explanation of the discrepancy in shear wave velocity determination based on LT and fifth peaks—the signal associated with the TT wave at short distances splits into two signals with increasing distance $d$ (starting from some threshold distance, see figure 3). An additional split peak in figure 3 is marked by SL$_{Fe}$-TT.

Linear fitting of the SL$_{Fe}$-TT arrival (figure 4) indicates that the wave associated with this peak propagates with a velocity of 5.92 ± 0.07, close to the velocity of the longitudinal velocity of iron or SL$_{Fe}$. Therefore, this signal is likely related to the arrival of the shear head wave emitted by the skimming longitudinal wave in iron and reflected at the iron/diamond interface SL$_{Fe}$-TT (head waves [15]) and the arrival of the type SL$_{Fe}$-TT-SL$_{Fe}$.

If we fit only the TT peaks without including SL$_{Fe}$-TT peaks, using thickness of the iron layer obtained from the fitting of the LL peaks, then shear velocity in iron appears to be 3.21 ± 0.03 km/s, which is close to that from the LT peaks fitting for the shear wave: $c_T = 3.18 \pm 0.02$ km/s. Consideration of the LT and TT peaks fitting suggests that when determining the shear wave in iron under high pressure (a) peaks associated with the TT wave should be identified and (b) both LT and TT peaks should be fitted with fixed sample thickness determined from the fitting of LL peak.

4. Conclusions
As a result of this study we introduce a modification in the algorithm proposed in Chigarev et al [1] for the determination of the shear wave in iron using the LU-DAC technique. The modification is based on the finding that the signal associated with a TT wave at short propagation distances splits into two signals with increasing distance $d$. One signal is assigned to the TT wave. The other signal is likely associated with the head wave. This finding allows us to improve the accuracy of shear wave velocity determination. The statistical error of the sample thickness and the longitudinal and shear velocity determination was found to be less than 2%.

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