Laser ultrasonic measurements in a diamond anvil cell on Fe and the KBr pressure medium

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Abstract. Laser ultrasonic technique has been successfully applied to a sample embedded in a transparent pressure transmitting medium to measure sound velocity at high pressures in a diamond anvil cell. Shear and longitudinal acoustic wave velocities are measured both for an opaque sample, Fe, and a transparent pressure transmitting medium, KBr, at pressures ranging from 0.3-24.8 GPa. The small sample size used in this experiment for sound velocity measurements suggests great potential for using this technique to evaluate the elastic properties of solids at pressures approaching 100 GPa and for other samples embedded in soft, transparent pressure transmitting media such as KBr or argon.

1. Introduction
Direct measurements of velocities and absorption of acoustic waves in minerals at elevated pressures and temperatures are key to understanding seismic information [1]. For samples compressed in a diamond anvil cell (DAC), the laser ultrasonics (LU) was first used to generate and detect surface acoustic waves [2] and later to measure the longitudinal sound velocities [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 [5,6] or line-source–point-receiver [7] configurations. These experiments were conducted without a pressure transmitting medium, which led to non-hydrostatic pressure conditions in the sample. Soft pressure transmitting media (PTM) are used in high pressure (HP) experiments in a DAC to eliminate or reduce pressure gradients in the sample because they could bias experimental data. The purpose of the present study is to examine the effect of a pressure transmitting medium (KBr) on the monitoring of acoustic waves in experiments with the LU-DAC.

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2. Method
A set of LU-DAC experiments in line-source–point-receiver configuration was conducted using KBr powder as a PTM (figure 1). We used KBr powder because it is very soft [8] and is considered one of the best pressure transmitting media [9]. Schematics of the sample assemblage and of the sound wave trajectories in our LU experiments in a DAC are shown in figure 1.

![Figure 1. Sketch of the sound wave propagation in a flat iron specimen embedded in the KBr pressure medium for the experimental configurations D/Fe (a) and KBr/Fe (b). Dashed line - bulk waves; solid line - a skimming bulk wave emitting a head wave; d - distance between the points of the sound generation and detection; $h_{Fe}$ - thickness of the Fe-specimen.](image)

A flat iron specimen of about 150 μm in width is pressed to the lower diamond anvil and separated from the upper one by a KBr layer (figure 1). The way the sample is loaded in DAC is similar to that used in impulsive stimulated light scattering [2] and gigahertz ultrasonic interferometry [10,11] in the DAC. The initial thickness of the iron specimen, $h_{Fe}$, was estimated to be about 15 μm. For the sample geometry presented in figure 1, the propagation time of the acoustic waves inside the Fe-specimen can be measured in two configurations: either when the pump and the probe laser beams are focused on the diamond/Fe interface (D/Fe configuration, figure 1a) or when the pump and the probe lasers are focused at the KBr/Fe interface (KBr/Fe configuration, figure 1b).

3. Results
The photoacoustic signals measured at 20.8 GPa are presented in figure 2. Following the notations introduced in [5-7], the peaks corresponding to the waves both excited and reflected from one of the interfaces as longitudinal (L) or transversal (T) are marked as LL or TT, respectively, where index “i” stands for the medium where the wave propagates (Fe, KBr, or D for diamond). The peaks marked as SL or ST correspond to the waves propagating as skimming L or T, respectively [12]. As mentioned in [7], skimming waves SL and ST could combine, under certain conditions, with LL or TT waves leading to the so-called “head wave arrivals” (see SL_D-LL_Fe, SL_Fe-TT_Fe and SL_KBr-LL_Fe peaks in figure 2).

In figures 3a and 3b the propagation times ($\tau$) of the acoustic waves, measured for different distances $d$ between the generation and detection points, are presented for the pressures at 9 and 20.8 GPa, respectively. A method of analysis of TTFe echoes in the Fe-specimen and LLKBr and TTKBr echoes in KBr can be found elsewhere [5-7]. Least square fits of two-parameters (sound velocity and sample thickness) to the experimental data $\tau(d)$ allowed us to determine the T-wave velocities in iron, $C_{TTFe}$, as well as the L- and T-wave velocities in KBr ($C_{LKBr}$ and $C_{TKBr}$) (figure 3c).
Figure 2. Photoacoustic signals measured at 20.8 GPa for \(d\) varying from 7.4 to 70.5 \(\mu m\) and from 4.0 to 70.6 \(\mu m\) in the configurations D/Fe (a) and KBr/Fe (b), respectively. In both measurements \(d\) was changed in increments of 3.7 \(\mu m\).

Due to overlapping of the LL\(_{Fe}\) and SL\(_{D-LLFe}\) signals (figure 3a), we could not determine the longitudinal wave velocity in Fe, \(C_{LFe}\), using the analysis method discussed in [5,6]. Nevertheless, we found that the velocity \(C_{LFe}\) could be obtained from \(\tau\)-values measured for the skimming longitudinal wave SL\(_{Fe}\) propagating in iron parallel to the interface with the diamond. These waves were detectable, however, only at pressures between 9 and 12 GPa (figure 3c).

Figure 3. Propagation time \(\tau\) of the acoustic signals measured for different generator-detector distances, \(d\), at 9 GPa (a) and 20.8 GPa (b). Squares and triangles represent the data for the configuration D/Fe and KBr/Fe, respectively. Solid curves represent the fit results.

Pressure dependences of the measured velocities (c): solid rhombi - \(C_{LFe}\), obtained from the SL\(_{Fe}\) wave; solid triangles - \(C_{TFe}\) from the TT\(_{Fe}\) wave; open rhombi marked (1) - \(C_{LKBr}\) from the SL\(_{KBr}\)-LL\(_{Fe}\) wave; open squares - \(C_{LKBr}\) from the LL\(_{KBr}\) wave; open rhombi marked (2) - \(C_{TKBr}\) from the ST\(_{KBr}\) wave; open triangles - \(C_{TKBr}\) from the TT\(_{KBr}\) wave.

Because the arrivals of the longitudinal LL\(_{KBr}\) and transversal TT\(_{KBr}\) waves could not be recognized at 20.8 GPa (figure 2b), the values of \(C_{TKBr}\) and \(C_{LKBr}\) were obtained from the skimming ST\(_{KBr}\) and mixed SL\(_{KBr}\)-LL\(_{Fe}\) waves, respectively. As shown in figure 3b, the signal SL\(_{D-LLFe}\) has been observed in both D/Fe and KBr/Fe experimental configurations.

Sound velocities of the acoustic waves measured in the present experiment at pressures up to 24.8 GPa are summarized in figure 3c. Both the \(C_{LFe}\) and \(C_{TFe}\) values of iron, determined in this work from the SL\(_{Fe}\) and TT\(_{Fe}\)-signals, respectively, are in a good agreement with the results obtained earlier using the LU-DAC technique without a PTM [5-7].
4. Conclusion
The experimental results presented in this article demonstrate that the LU-DAC technique can be applied for sound velocity measurements in small specimens compressed in soft pressure transmitting media to very high pressures. Moreover, our results show that this technique allows the determination of acoustical properties of both the specimen and the pressure medium.

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References
[1] Bass J D 2008 Phys. Earth Planet. Inter. 170 207
[2] Crowhurst J C, Abramson E H, Slutsky L J, Brown J M, Zaug J M and Harrell M D 2001 Phys. Rev. B 64 100103
[3] Villagran-Muniz M, Navarrete M and Mejia-Uriarte E V 2003 Rev. Sci. Instrum. 74 732
[4] Decremps F, Belliard L, Perrin B, and Gauthier M 2008 Phys. Rev. Lett. 100 035502
[5] Chigarev N, Zinin P, Ming L C, Amulele G, Bulou A and Gusev V 2008 Appl. Phys. Lett. 93 181905
[6] Chigarev N, Zinin P, Mounier D, Bulou A, Ming L C, Acosta T and Gusev V 2010 High Pressure Research 30 78
[7] Zinin P, Chigarev N, Mounier D, Bulou A, Ming L C, Acosta T and Gusev V 2010 J. Phys. Conf. Ser. 215 012053
[8] Singh S, Singh R K, Singh B P, Singhal S K and Chopra R 2000 Physica Status Solidi a-Applied Research 180 459
[9] Shen A H, Reichmann H J, Ghen G, Angel R J, Bassett W A and Spetzler H 1998 Geophys. Monogr. Ser. 101 71
[10] Jacobsen S D, Spetzler H A, Reichmann H J, Smyth J R, Mackwell S J, Angel R J and Bassett W A 2002 J. Phys.-Condes. Matter 14 11525
[11] Bassett W A, Reichmann H J, Angel R J, Spetzler H and Smyth J R 2000 Am. Miner. 85 283
[12] Beghi M G, Every A G, and Zinin P V 2004 Ultrasonic Nondestructive Evaluation: Engineering and Biological Material Characterization, ed T Kundu (Boca Raton: CRC Press)