Physics of the Earth and Planetary Interiors 174 (2009) 282–291

Contents lists available at ScienceDirect

Physics of the Earth and Planetary Interiors

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

Development of *in situ* Brillouin spectroscopy at high pressure and high temperature with synchrotron radiation and infrared laser heating system: Application to the Earth's deep interior

Motohiko Murakami[a,b,*], Yuki Asahara[c], Yasuo Ohishi[c], Naohisa Hirao[c], Kei Hirose[d,e]

[a] *Department of Earth and Planetary Materials Science, Graduate School of Science, Tohoku University, Sendai 980-8578, Japan*
[b] *Institute for Study of the Earth's Interior, Okayama University, Misasa, Tottori 682-0193, Japan*
[c] *Japan Synchrotron Radiation Research Institute, 1-1-1 Mikazuki-cho, Sayo-gun, Hyogo 679-5198, Japan*
[d] *Institute for Research on Earth Evolution, Japan Agency for Marine-Earth Science and Technology, 2-15 Natsushima-Cho, Yokosuka-city, Kanagawa 237-0061, Japan*
[e] *Department of Earth and Planetary Sciences, Tokyo Institute of Technology, 2-12-1 Ookayama, Meguro, Tokyo 152-8551, Japan*

ARTICLE INFO

Article history:
Received 9 October 2007
Received in revised form 16 April 2008
Accepted 23 July 2008

Keywords:
Earth's interior
Mineral physics
Sound velocities
High pressure and high temperature
Elasticity

ABSTRACT

Seismic wave velocity profiles in the Earth provide one of the strongest constraints on structure, mineralogy and elastic properties of the Earth's deep interior. Accurate sound velocity data of deep Earth materials under relevant high-pressure and high-temperature conditions, therefore, are essential for interpretation of seismic data. Such information can be directly obtained from Brillouin scattering measurement. Here we describe an *in situ* Brillouin scattering system for measurements at high pressure and high temperature using a laser heated diamond anvil cell and synchrotron radiation for sample characterization. The system has been used with single-crystal and polycrystalline materials, and with glass and fluid phase. It provided high quality sound velocity and elastic data with X-ray diffraction data at high pressure and/or high temperature. Those combined techniques can potentially offer the essential information for resolving many remaining issues in mineral physics.

1. Introduction

One of the longstanding challenges in mineral physics has been experimental determination of the reliable acoustic wave velocities of deep-Earth materials under relevant high-pressure and high-temperature conditions, because precise knowledge of sound velocity (elasticity) is essential for interpretation of seismic observations and development of global seismological models of Earth's interior. Constraints on mineralogy, chemical composition and thermal structure of the Earth's interior can be developed by using those experimental sound velocity data for this purpose.

Considerable effort has thus focused on the development of the measurement for use in high-pressure apparatuses (such as multi-anvil and diamond anvil cell (DAC) devices) to determine the sound velocities at high pressure and high temperature. Recently developed and improved techniques include ultrasonic interferometry (Abramson et al., 1999; Bassett et al., 2000; Chen et al., 1998; Higo et al., 2006; Kinoshita et al., 1979; Li et al., 1998, 1996; Liebermann, 2000; Liebermann and Li, 1998; Rigden et al., 1994; Sinelnikov et al., 1998; Yoneda, 1990; Yoneda and Morioka, 1992), impulsive stimulated scattering (Abramson et al., 1999, 1997; Brown et al., 1989; Zaug et al., 1993), inelastic X-ray scattering (Fiquet et al., 2004, 2001; Mao et al., 2001a), nuclear resonance inelastic X-ray scattering (Lubbers et al., 2000; Mao et al., 2001b; Shen et al., 2004) and Brillouin spectroscopy (Duffy et al., 1995; Sinogeikin and Bass, 2000, 2002; Sinogeikin et al., 1998; Zha et al., 1998, 1997, 2000). Using those techniques, substantial experimental data on elasticity of Earth's deep materials have accumulated at least to pressure and temperature conditions of the mantle transition zone. However, sound velocities under lower mantle conditions (above ~25 GPa in pressure and ~1900 K in temperature) have rare because of the experimental difficulties.

Diamond anvil cell high-pressure apparatus can generate extremely high static pressures to conditions approaching those of the inner-core (~300 GPa) (Mao et al., 1990, 1989), and the recent advancement of infrared laser heating technique in a DAC (Boehler, 1992; Shen et al., 1996) combined with synchrotron radiation have allowed us to explore successfully the phase equilibria in the lowermost-mantle and core materials under relevant pressure

---

[*] Corresponding author at: Department of Earth and Planetary Materials Science, Graduate School of Science, Tohoku University, Sendai 980-8578, Japan.
Tel.: +81 22 795 6662; fax: +81 22 795 6662.
E-mail address: motohiko@m.tains.tohoku.ac.jp (M. Murakami).

0031-9201/$ – see front matter © 2008 Elsevier B.V. All rights reserved.
doi:10.1016/j.pepi.2008.07.030

and temperature conditions (Dubrovinsky et al., 2007; Murakami et al., 2004).

Brillouin scattering measurement can also be conducted in the diamond anvil cell. With this method, the Brillouin frequency shift, related to the photon–phonon interactions between the focused optical probe and sample, is determined under high-pressure condition. Major advantages of this technique are that very large sample volume are not required and the results are not significantly affected by porosity, micro-cracks, inclusions in the sample, and the uncertainties of sample dimensions. In addition, this technique can be used with both glass materials (Zha et al., 1994), and liquid phases (Polian and Grimsditch, 1983). Brillouin spectroscopy with diamond anvil cell technique is, therefore, one of the most promising techniques to determine elastic properties of deep mantle materials. However, high quality Brillouin data generally require clear transparent, colorless, unstrained single-crystals. Most results so far have, therefore, been limited to conditions below ∼20 GPa and ∼1000 K. Recent advances in high-pressure Brillouin spectroscopy measurements extended drastically the upper pressure limit, approaching 180 GPa, which is outer-core pressure (Murakami et al., 2007a,b). Murakami et al. (2007a,b) have first reported aggregate shear wave velocities in polycrystalline $MgSiO_3$ perovskite and post-perovskite phases within their thermodynamic stability field. These samples were synthesized *in situ* in a DAC from gel starting materials by heating with an infrared laser up to pressures of 172 GPa at room temperature. These studies demonstrated the potential of laser annealing techniques. This method efficiently promotes the synthesis of polycrystalline sample and reduces deviatoric stresses on the sample, thus substantially improving the quality of Brillouin spectra of aggregate samples under extreme pressure conditions.

A combined diamond anvil cell system for Brillouin spectroscopy and synchrotron radiation enables the simultaneous measurements of sound velocities and X-ray diffraction at high pressure and high temperature. This information provides elastic properties such as bulk and shear moduli, and their pressure and temperature derivatives. The *in situ*, high-pressure–temperature X-ray diffraction data by synchrotron radiation is essential not only for obtaining the structural information (phase identification, lattice parameters, compressibility and density) of the sample but for providing the precise pressure values under high-temperature condition from the tiny sample in a DAC. Such a combined system is currently in operation in third-generation synchrotron radiation source of Advanced Photon Source at Argonne National Laboratory (Sinogeikin et al., 2006). However, simultaneous measurements under pressure and temperature condition of the lower mantle are still a technical challenge, especially heating. Application of resistive heating technique in a DAC (Sinogeikin et al., 2000), which is the conventionally used technique for Brillouin method, normally works well below 10 GPa and 1000 K for simultaneous measurements (Sinogeikin et al., 2006). Moreover, a major barrier for resistive heating, which generally reaches ∼1500 K at most, is to generate the higher temperatures of the lower mantle. High-temperature Brillouin scattering measurements with laser heating have been conducted at ambient pressure on single-crystals of MgO and Al_2O_3 (Sinogeikin et al., 2004). An infrared laser heating technique would be a favorable alternative, which can potentially generate over 3000 K at higher pressures (Knittle and Jeanloz, 1989; Shen and Lazor, 1995; Shen et al., 1998; Zerr and Boehler, 1993; Zerr et al., 1997).

In order to meet the requirements for simultaneous sound wave velocity measurements and sample characterization under lower mantle conditions, a Brillouin scattering measurement system that uses an infrared laser heating technique was recently installed at the BL10XU beamline station in the Japanese synchrotron facility of SPring-8. In this report, we show the details of the system together with some examples of results. On the basis of the examples of preliminary results, future prospects for applications to geophysical problems and problems that still need to be overcome will be discussed.

2. Experimental setup

2.1. Overview of the in situ Brillouin scattering measurement system at high pressure and high temperature

A system for *in situ* Brillouin scattering measurement at high pressure and high temperature has been installed at the BL10XU beam line station in the synchrotron facility of SPring-8. This system consists of three optical components used for Brillouin spectroscopy, X-ray diffraction and infrared laser heating (temperature measurement). For the simultaneous measurements, all optical probes for each of these three component must converged on the sample without optical and physical interference. The data from each component are extracted by simultaneous and independent detector/analysing systems for each, simultaneously. This system is shown in Fig. 1. Fig. 2 shows the whole view of this measurement system installed at SPring-8.

2.1.1. Brillouin scattering measurement system

The main component of Brillouin scattering system are a diode-pumped laser with a wavelength of 532 nm as a incident probe beam (Verdi V2, Coherent), a Sandercock-type six-pass tandem Fabry-Perot interferometer as an analyzer of the scattered light (TFP1, JRS Scientific Instruments) (Lindsay et al., 1981; Sandercock, 1982), and optics for incident beam positioning, and focus adjustments, and scattered light guides to the interferometer. The diode-pumped probe laser with its small line width (<5 MHz at maximum output power), low beam divergence (<0.5 mrad in full-angle divergence), and high pointing stability (<2 μrad/°C) is highly compatible with the Brillouin scattering measurement. The laser power of the incident probe beam (with a maximum output power of 2 W) is adjusted by the rotationally variable neutral density (ND) filter. High stability of the elastic peak (Rayleigh peak) position during the measurements was achieved with an appropriate amount of reference light that was split slightly (<1%) from the main probe beam. This reference light is continuously provided to the interferometer. A Sandercock-type six-pass tandem Fabry-Perot interferometer is equipped with automatic synchronization, ultra linear tandem scanning and a motor controlled mirror alignment system on the dynamic vibration isolation optical stage. The tandem system allows complete dynamic synchronization of two interferometers over a large scanning range. Detailed description of the Sandercock-type Fabry-Perot interferometer is described in elsewhere (Sandercock, 1982).

In this measurement system, a DAC mounted on the multiaxial stage with XYZθχαβ (Fig. 3) is placed on the corner with 150° angle of the pentagonally shaped optical bench for Brillouin measurement system (Fig. 1). The incident laser focused to ∼20 μm in diameter is introduced into the sample and the scattered light is analyzed by a Fabry-perot interferometer. All optical paths for Brillouin measurements are coordinated parallel or perpendicular to the optical bench. A symmetric ∼50° scattering geometry (which means the angle between incident laser and scattered light is ∼50°) is adopted in all experiments. Both sides of the sample image for Brillouin measurements can be observed through the charge coupled device (CCD) cameras from the outside of the experimental hutch.