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Ultrasonic Interferometry at High Pressures and Temperatures: From the Bell Telephone Laboratories to National Synchrotron Facilities

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Abstract

Laboratory experiments utilizing ultrasonic interferometric techniques to measure sound velocities of minerals at elevated pressures and temperatures have progressed over the past 3/4 century and recently achieved conditions approaching those of the Earth's lower mantle. This progress has included the development of dual mode transducers, designing new interferometers, *in situ* pressure determination in multi-anvil, high-pressure apparatus, and integration of these experiments at national synchrotron X-radiation facilities in the U.S., Japan and Europe.

Keywords

Ultrasonic Interferometry, Synchrotrons, Piezoelectric Transducers, Mineral Physics, High Pressure, Wave Velocities

1. Introduction

Several technological developments during World War II made possible measurements of sound velocities in solids using high-frequency ultrasonic methods; many of these grew out of work at the Radiation Laboratory at the Massachusetts Institute of Technology and comparable laboratories in England. In particular, the wartime development of pulsed circuits in conjunction with piezoelectric transducers made pulsed ultrasonic measurements practical.

Following the invention of piezoelectric transducers in the early 1950s at the Bell Telephone Laboratories in Murray Hill, New Jersey, Warren Mason established a physical acoustics group there; he recruited a talented group of young scientists to develop new techniques for conducting Megahertz-frequency ultrasonic interferometric experiments to measure elastic wave velocities in solids and

liquids, including both the pulse superposition and phase comparison methods [1] [2]. Among these new experimentalists were H. J. (Hal) McSkimin, Paul Andreatch and Emmanuel Papakakis (joining D. B. Fraser and L. C. LeCraw); the group also included theoreticians such as R. N. Thurston and K. Brugger who formulated new approaches to treat third-order elastic moduli [3]. Mason also edited a book series on Physical Acoustics (Vols I to XVIII from 1964 to 1988); e.g., [4] which included important experimental papers (by McSkimin; Anderson and Liebermann; Truell, Elbaum and Hikata;) as well as theoretical papers (by Thurston and Brugger; Alers). These books edited by Mason are required reading for students in mineral physics, in addition to the book on Ultrasonic Methods in Solid-State Physics by Truell, Elbaum and Chick [5]. The concept of ultrasonic interferometric methods and experimental implementations can be found easily in the above-mentioned references.

2. Early Years of Ultrasonic Interferometry at University Laboratories

Following the development of these ultrasonic interferometry techniques at the Bell Labs, several U.S. mineral physics[1] labs began in the 1960s to utilize them to measure sound velocities of minerals at elevated pressures and temperatures (mostly at pressures below 10 kbar/1 GPa and temperatures less a few hundred K): Francis Birch (and later Richard O'Connell) at Harvard, Orson Anderson (and later Edward Schreiber) at the Lamont Geological Observatory of Columbia University (and later at UCLA), Gerhard Barsch (and later Earl Graham) at Penn State, Murli Manghnani at the University of Hawaii, Hartmut Spetzler at Caltech (and later at the University of Colorado), Gene Simmons at MIT, Thomas Ahrens at the Rensselaer Institute of Technology, and Syun-iti Akimoto at the University of Tokyo.

Over the subsequent 75 years, these ultrasonic techniques have continued to develop and the velocity measurements have been conducted at ever-increasing pressures and temperatures, now reaching conditions of the Earth's lower mantle. The primary purpose of this paper is to review the evolution of the techniques of ultrasonic interferometry over this period; this is an extension and expansion of our earlier papers [6]-[9]. An earlier version was presented in the FORCE[2] series of seminars at Arizona State University. With the encouragement of Ms. Sarry Sun to submit a review for a special issue of the International Journal of Geosciences on Earth Structure and Dynamics, I have chosen to focus on developments in our laboratory at Stony Brook, including developments at the synchrotrons of

[1]According to Robert Hazen, "mineral physics is the study of mineralogical problems through the application of condensed matter physics". In reality, mineral physicists use not only physics but also solid-state chemistry. In addition, they study not only minerals but all materials related to natural minerals (e.g., structural analogs, but also glasses, melts, and fluids). Although he never used the term, Francis Birch is widely acknowledged as the "father of mineral physics". Orson Anderson often cited the German physicist Eduard Grüneisen as the person who first introduced the term mineral physics.
[2]FORCE (Facility for Open Research in a Compressed Environment) at the Arizona State University is a state-of-the-art high-pressure research facility enabling experiments on materials under extreme conditions.

the U.S. Department of Energy at Brookhaven National Laboratory and the Argonne National Laboratory, and our collaborations with colleagues in Australia, Japan, and Europe.

Both pulse superposition and phase comparison provide the travel times of the acoustic waves by overlapping the echoes from the ends of the specimen; with the advent of dual-mode transducers, it became possible to obtain the travel time of both compressional (P) and shear (S) waves simultaneously.

During the early 1980s at the Australian National University (ANU), Ian Jackson and Herbert Niesler designed and built a new ultrasonic interferometer capable of precise and accurate measurements of travel times of the 10 - 100 MHz compressional and shear waves through specimens of sub-cm dimensions. They collaborated with Don Weidner to develop a procedure to correct elastic wave travel-times measured by ultrasonic interferometry for the phase shifts arising from propagation of the elastic waves through the thin bond-coupling transducer to the crystals. This new ultrasonic technique was first applied in measurements of the pressure dependence of wave velocities in a 3 GPa liquid-medium piston-cylinder apparatus (a capability originality developed in George Kennedy's laboratory at UCLA) [10].

During the subsequent 35 years, my laboratory at Stony Brook has maintained an active collaboration with the laboratory of Ian Jackson in Canberra. We capitalized on ultrasonic techniques developed in Jackson's lab at the ANU and synthesis techniques to fabricate polycrystalline specimens of high-pressure phases of mantle minerals developed in my lab at Stony Brook (see **Figure 1**).



Figure 1. Sally Rigden from the ANU with Gabriel Gwanmesia, from Delaware State University, and the author in High Pressure Laboratory at Stony Brook in 1991. Polycrystalline specimens of high-pressure phases were synthesized at Stony Brook and their sound velocities measured at the ANU during visits by Gwanmesia and the author.

In 1994, Baosheng Li (then a graduate student of mine and now a Professor in the Department of Geosciences at Stony Brook) and Jackson (my first graduate student and subsequently the Director of the Research School of Earth Sciences at the ANU) adopted these new ultrasonic techniques to measure sound velocities in situ in a 1000-ton uniaxial split-cylinder apparatus (USCA-1000) which consists of a 1000-ton Kennedy-Getting type hydraulic press modified for use with a Walker-type multi-anvil module (see **Figure 2**) [11]. This pioneering development provided the opportunity to conduct sound velocity experiments to pressures of 10 GPa and was exploited over the next few years by many graduate students and postdocs [12]-[15].

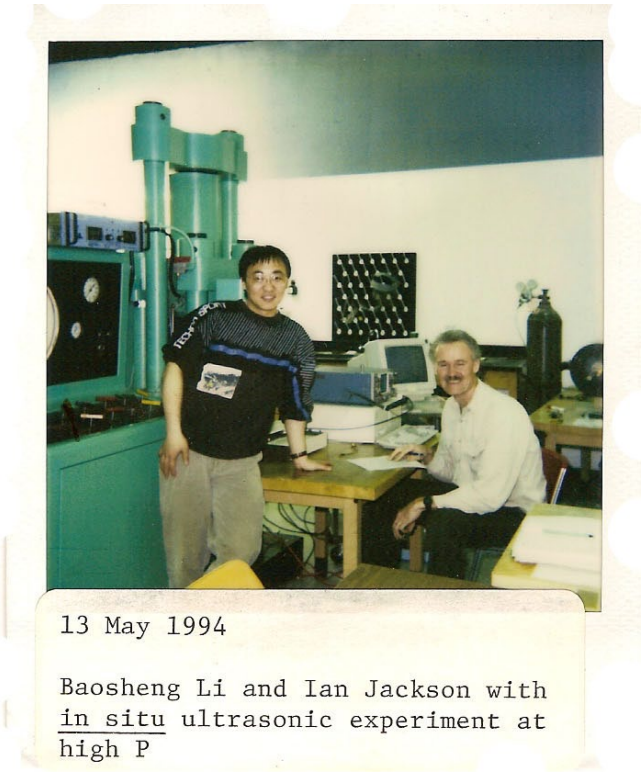


Figure 2. Baosheng Li and Ian Jackson in the Stony Brook High Pressure Laboratory. Ian spent 6 weeks in our laboratory in 1994 working with Baosheng on interfacing the ANU ultrasonic interferometry system with the 1000-ton split-cylinder apparatus of the Kennedy-Getting type.

As part of her doctoral research at the ANU, Jennifer Kung spent six months at Stony Brook on the synthesis of polycrystalline specimens of $ScAlO_3$-perovskite with the aid of Gabriel Gwanmesia. When she was a postdoctoral research associate at Stony Brook from 2000-2005, Jennifer measured the high-temperature velocities of orthoenstatite in Jackson's lab (**Figure 3**) [16].

In the most recent collaboration between the ANU and Stony Brook, Ting Chen and her colleagues measured the sound velocities of polycrystalline coesite at high pressure and temperature in Baosheng Li's laboratory using a specimen hot-pressed by the author in a girdle—anvil apparatus in 1975, and so very "well-

aged", Ting Chen *et al.* [17].



Figure 3. Jennifer Kung with high-temperature, ultrasonic apparatus in laboratory of Ian Jackson at the RSES at the ANU. Circa 2011.

3. Synchrotron X-Radiation Facilities

Many recent ultrasonic studies of the sound velocities of minerals at high pressures and temperatures have been conducted at the synchrotron X-radiation facilities of the U.S. Department of Energy, including the National Synchrotron Light Source (NSLS) at the Brookhaven National Laboratory and the Advanced Photon Source (APS) at the Argonne National Laboratory; and, also, at SPring-8 in Japan[3] and at PETRA in Germany and at the ESRF in France.

The principal advantage of conducting these ultrasonic experiments at a synchrotron facility is that the X-ray beam provides a direct image of the specimen under high pressure and temperature; With Xradiography, the specimen dimensions are provided, whereas X-ray diffraction establishes the crystal structure and unit-cell volume; thus, the crystal structure of the specimen can be verified, and its length obtained at elevated pressures and temperatures. These length changes of the specimen at high P and T are needed to convert the observed travel times

[3]SPring-8 is a large synchrotron radiation facility located in Harima Science Park City, Hyogo Prefecture, Japan. The name "SPring-8" is derived from **S**uper **P**hoton **ring**-**8** GeV (8 GeV, or 8 giga electron volts, being the energy of electron beam circulating in the storage ring); Spring-8 is managed by RIKEN and the Japan Synchrotron Radiation Research Institute (JASRI). SPring-8 delivers the most powerful synchrotron radiation currently available. Consisting of narrow, powerful beams of electromagnetic radiation, synchrotron radiation is produced when electron beams, accelerated to nearly the speed of light, are forced to travel in a curved path by a magnetic field. The research conducted at SPring-8 includes nanotechnology, biotechnology and industrial applications. SPring-8 was opened in 1997 to users from academia, research institutes, governmental agencies and industry, domestic and international. Any user whose application is accepted may use the facility.

of the acoustic waves to velocities.

One example of the usefulness of synchrotron facilities is represented by the work of Xuebing Wang, Ting Chen and Xintong Qi (all graduate students in the laboratory of Baosheng Li), who developed a new method for *in-situ* pressure determination in multi-anvil, high-pressure apparatus using an acoustic travel time approach within the framework of acoustoelasticity [18]. The ultrasonic travel times of polycrystalline Al_2O_3 were calibrated against the NaCl pressure scale up to 15 GPa and 1500 K in a Kawai-type double stage multi-anvil apparatus in conjunction with synchrotron X-radiation, thus providing a convenient and reliable gauge for pressure determination at ambient and high temperatures.

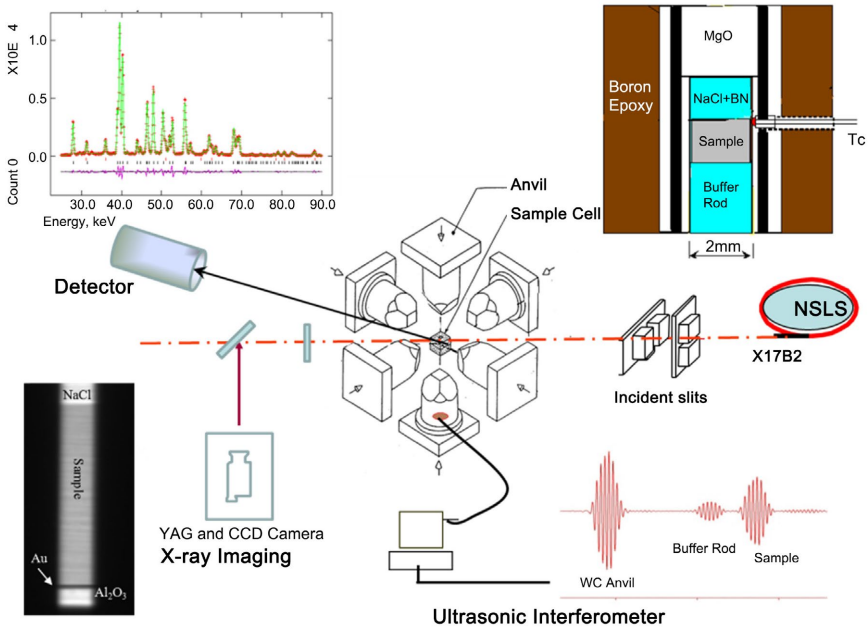


Figure 4. Schematic of installation of DIA-type apparatus (SAM-85) on X17B at NSLS.

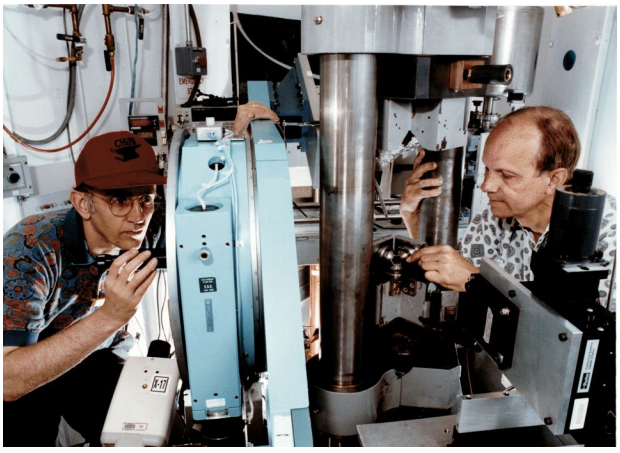


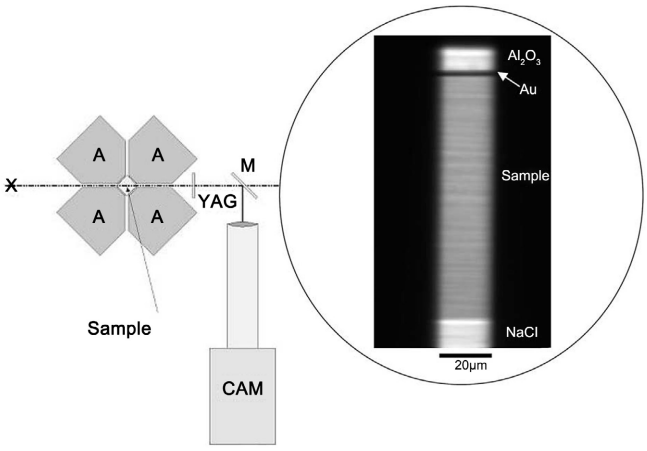
Figure 5. Don Weidner and Mike Vaughan performing ultrasonic experiments with SAM-85 installed on the superconducting wiggler beamline (X17B) at the NSLS.

Baosheng Li led the exploitation of these new opportunities by adapting ultra-

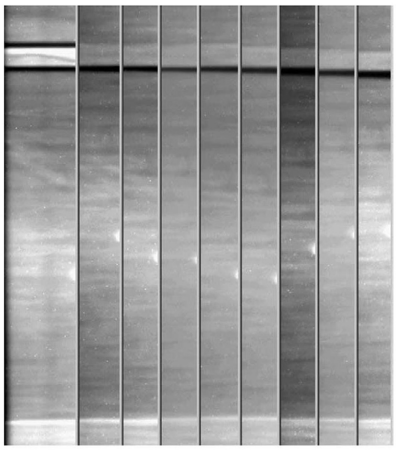
sonic interferometric techniques for use in a DIA-type cubic-anvil apparatus (SAM-85) and installed on the superconducting wiggler beamline (X17B) at the National Synchrotron Light Source (NSLS) of the Brookhaven National Laboratory (BNL)-see details in [7] and [19] (see **Figure 4**). Over a decade later, Don Weidner and colleagues pursued similar ultrasonic experiments (see **Figure 5**).

These developments have enabled P and S wave velocities and X-ray measurements to be performed simultaneously to pressures of 8 GPa and temperatures of 1600 K on both single-crystal [12] and polycrystalline specimens [6] [13] [14].

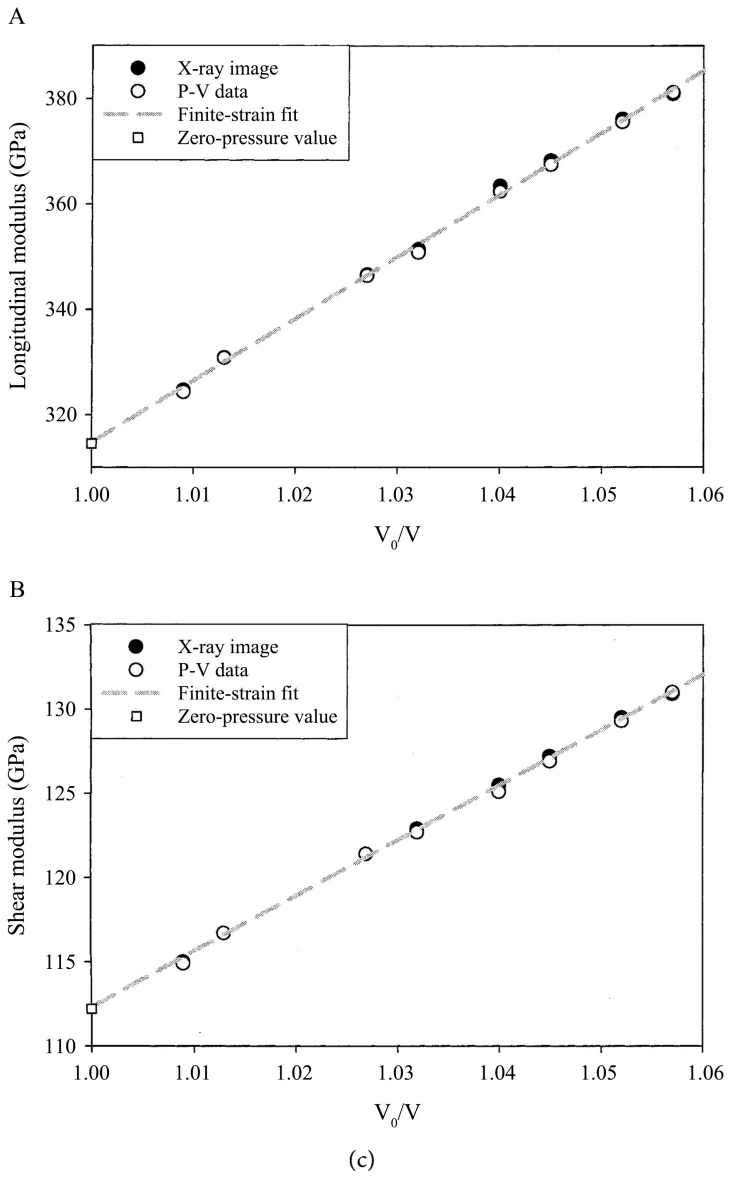
Incorporation of X-radiography to the existing setup allowed us to monitor sample lengths directly at elevated pressure and temperature (see **Figure 6(a)** and **Figure 6(b)**) [7] and [19]. By combining the ultrasonic determination of travel times and the measurement of specimen volumes and lengths by the techniques of X-ray diffraction and radiographic imaging, the fitting process for velocities can be conducted without knowledge of the cell pressure by using V_O/V of the specimen as the independent variable. In this manner, it is possible to eliminate the systematic errors introduced by the pressure scale in the observed elastic parameters.



(a)



(b)



(c)

Figure 6. (a) X-radiographic image system. The X-rays (X) pass through the gaps between the anvils (A) in SAM-85 and into the sample chamber. A typical cell assembly image is shown in the right panel; (from top to bottom) the Al_2O_3 acoustic buffer rod, a 2-micron thick gold foil inserted between buffer rod and specimen, a NaCl disk behind the specimen; (b) A series of X-ray images recorded at pressures from ambient to 9.0 GPa (indicated by the NaCl pressure scale). The images shown here are the same as in (a) (from top to bottom: Al_2O_3 buffer rod, gold foil, specimen and NaCl). (c) (A and B) Calculated longitudinal and shear elastic moduli of (Mg0:83, Fe0:17)O as a function of V0/V at pressures. The open circles (PV data) represent the moduli calculated using the specimen lengths converted from X-ray diffraction data. The solid circles represent the moduli calculated through using the specimen lengths observed from the X-radiographic imaging technique. The long-dashed lines represent the finite strain fits.

The elasticity of ferropericlase with a potential mantle composition of (Mg 0:83, Fe0 0:17)O was determined pressures of 9 GPa (NaCl pressure scale) at room temperature (Figure 6(c)).

If these results are compared with those for MgO, calculations show that the P and S wave velocities of a perovskite plus ferropericlase phase assemblage with a pyrolite composition at the top of the lower mantle (660 km depth) are lowered by 0.8% and 2.3%, respectively, when compared with those calculated using the elastic properties of endmember MgO. Consequently, the magnitudes of the calculated wave velocity jumps across the 660 km discontinuity are reduced by about 11% for P wave and 20% for S wave, if this discontinuity is considered as a phase transformation boundary only (ringwoodite > perovskite + ferropericlase).

At the Advanced Photon Source (APS) of the Argonne National Laboratory (ANL), Baosheng Li later collaborated with Yanbin Wang (former graduate student at Stony Brook and now on the faculty of the University of Chicago) to conduct ultrasonic experiments in the multi-anvil (MAA), split-sphere apparatus (T-25) installed on the GeoSoilEnviro (GSECARS) beamline (13-ID-D) (see **Figure 7** and **Figure 8**); with this Kawai-type apparatus, these experiments can be conducted to pressures of 30 GPa and temperatures of 3000 K [20]. See reports of experiments using these facilities in the papers by Stony Brook graduate students Sibo Chen [21] and Siheng Wang [22], and postdoc Zhicheng Jing [23].

In Zhicheng Jing *et al.*, [24] (see also [23]), sound velocities of Fe and Fe-S liquids were determined by combining the ultrasonic measurements and synchrotron X-ray techniques under high pressure-temperature conditions from 1 to 8 GPa and 1573 K to 1973 K using the T-25 apparatus at the APS. A new lunar structure model incorporating available geophysical observations points to a smaller core radius; their model suggests a top-down solidification scenario for the evolution of the lunar core (see **Figure 9** below).

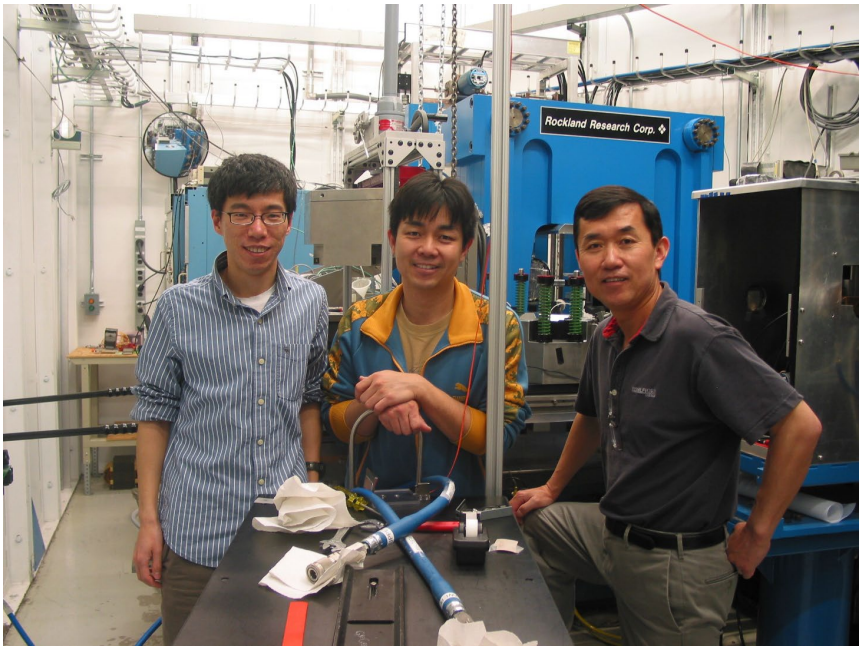


Figure 7. Zhicheng Jing and Tony Yu with Yanbin Wang (L to R) with T-25 split-sphere apparatus on GSECARS sector of the APS.



Figure 8. Baosheng Li inserting ultrasonics cell assembly in T-25 split-sphere apparatus at the GSECARS beamline of APS.

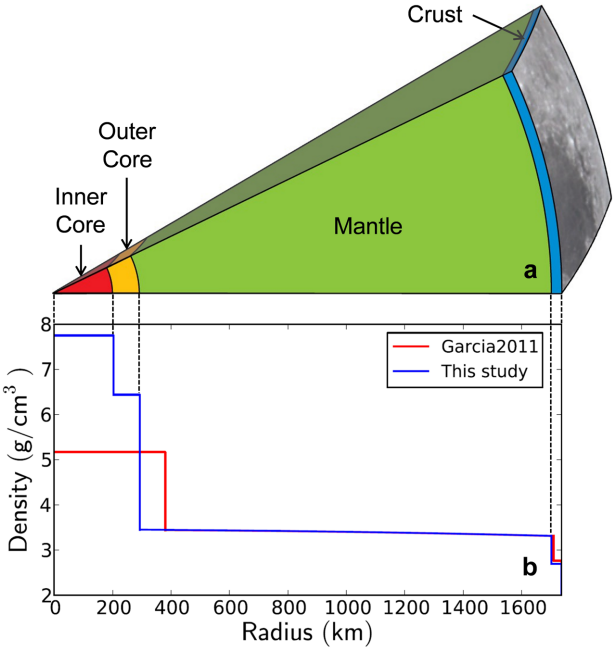


Figure 9. Preferred internal structure model of the Moon. (a) Illustration of a four-layer structure model of the Moon, consisting of an inner core, an outer core, a mantle, and a crust. (b) The density profile of the Moon as a function of radius from this study is shown in blue. For comparison, the model from Garcia *et al.* [25] (2011) is shown in red. From [24].

Significant advances in the utilization of ultrasonic interferometry have also emerged from installation of multi-anvil apparatus on the beamlines at the Spring-8 synchrotron radiation facility in Japan.[4] Ultrasonic measurements at simultaneous high pressures and high temperatures were conducted with a 1500-ton Kawai-type multi-anvil apparatus (SPEED-1500) located at the beamline BL04B1 in SPring-8 (see Figure 10 and Figure 11).

[4]See note 3.

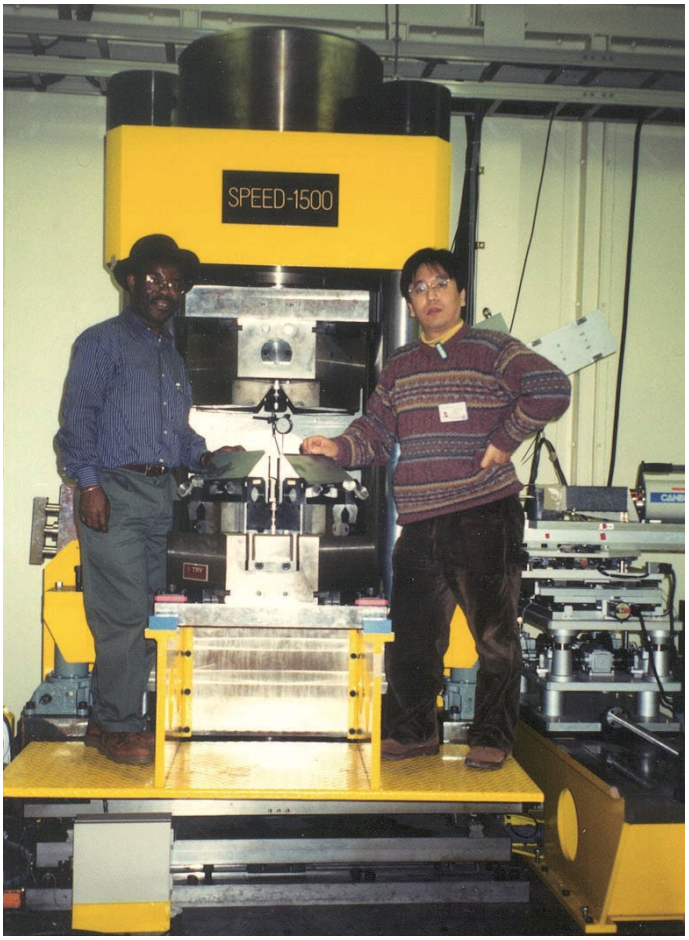


Figure 10. Gabriel Gwanmesia with Wataru Utsumi with SPEED-1500 apparatus at Spring-8.

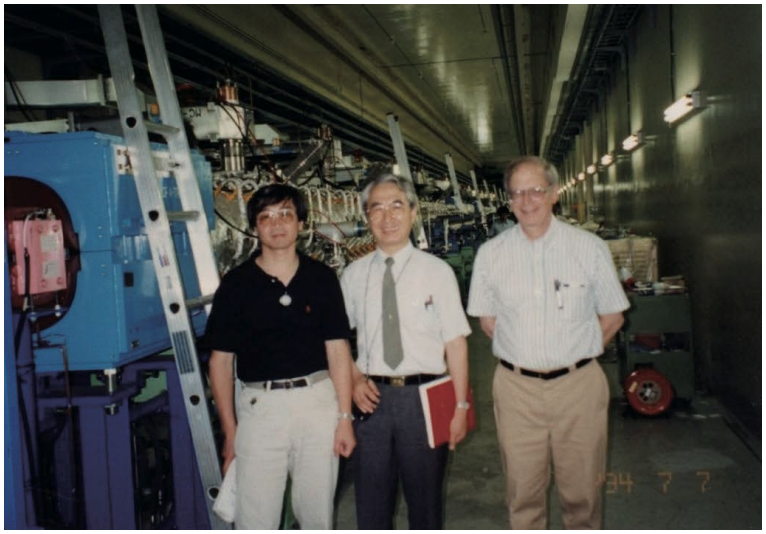


Figure 11. Toshi Sasaki, Shigeho Sueno and Charlie Prewitt at Spring-8 in 1994.

Many of these experiments have been performed by members of the research group of Tetsuo Irifune at the Geodynamics Research Center (GRC) of Ehime

University in Matsuyama, including Steeve Greeaux, Yuji Higo and Toru Inoue (now at Hiroshima University); the latter two have served postdoctoral stints in the research group of Baosheng Li at Stony Brook. Ultrasonic measurements at simultaneous high pressures and high temperatures were also conducted with a 1500-ton Kawai-type multi-anvil apparatus (SPEED-1500) located at the beamline BL04B1 in SPring-8; by Liu., Irifune, Gréaux, Arimoto, Shinmei and Higo [26] and Xu, Gréaux, Inoue., Noda, Gao, & Li [27].

In the Higo *et al.* [28] paper, a new pulse-echo interferometry system was described for measurements of sound velocity at simultaneous high pressure and temperature corresponding to those of the Earth's lower mantle; in **Figure 12**, these new data are compared with the results of earlier studies. This system allows precise measurements of the elastic velocities of minerals under the pressures and temperatures corresponding to the lower mantle for the first time, which should greatly contribute to our understanding of mineralogy of the whole mantle.

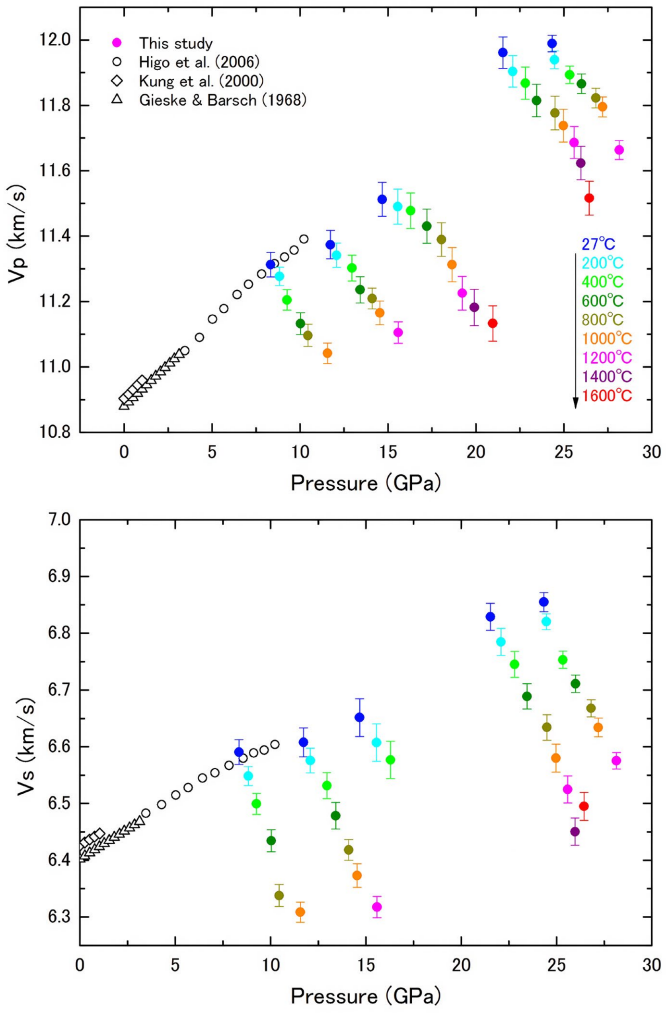


Figure 12. Longitudinal and shear wave velocities of alpha-Al203 alumina at high pressure and temperature obtained in the study of Higo, Irifune and Funakoshi [28] compared with previous studies.

In Europe, ultrasonics experiments have been conducted on the PETRA beamlines at the Deutsches Elektronen-Synchrotron (DESY) in Germany. A recent paper by Neri, Man, Chantel, Farla, Bauer, Linhardt, Boffa-Ballaran and Frost [29] (2024) describes the development of a dual travel time method for pulse-echo ultrasonic systems installed at the Bayerisches Geoinstitut (BGI) and on the large volume press (LVP) located in the end-station of the high-energy wiggler beamline P61 (called P61B) on the PETRA beamlines at the Deutsches Elektronen-Synchrotron (DESY) in Germany; although this study did not use synchrotron radiation, it closely followed the approach of Xuebing Wang *et al.* [18].

The current status of laboratory experiments to utilize ultrasonic techniques to measure the sound velocities of materials at elevated pressures and temperatures is illustrated in **Figure 13**.

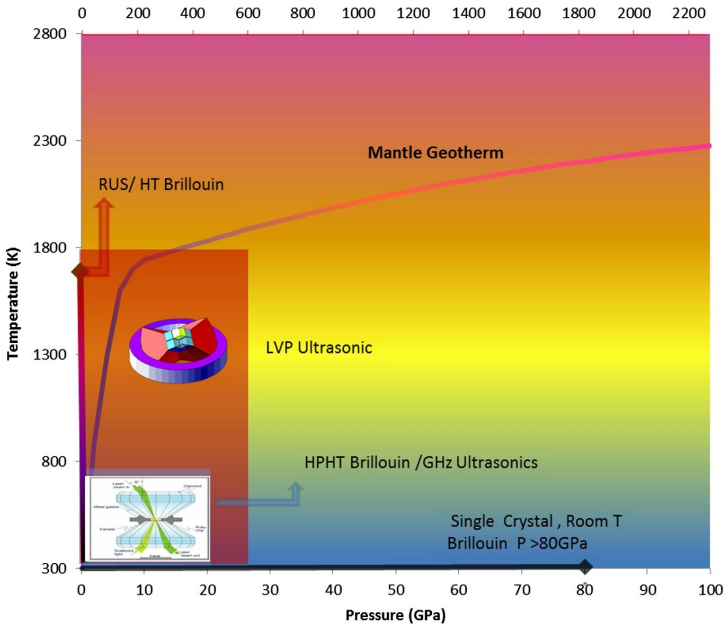


Figure 13. Mantle geotherm and various experimental techniques to use ultrasonics to explore those pressures and temperatures in the laboratory. To date, the ultrasonic experiments of Higo *et al.*, [28] extend to 1873 K at 27 GPa, slightly beyond the temperature range in the figure.

The evolution of these techniques to higher pressures and temperatures can be illustrated by the ultrasonic studies of hematite over the past six decades.

In 1968 in my first experimental paper, we measured the sound velocities of polycrystalline hematite to 0.3 GPa at room temperature (Liebermann and Schreiber [30]). These experiments utilized single-mode P and S wave transducers, thus requiring the data from 2 different experiments to be combined to calculate the specimen length changes required to calculate the wave velocities.

Twenty years later, Liebermann and Maasch [31] measured the sound velocities of single crystal hematite to 3 GPa and low temperatures across the Morin transition at 261 K.

More recently, Ran Wang and Baosheng Li [32] measured the sound velocities

of the same polycrystalline specimen from our 1968 study to 13 GPa at room T. Also, in 2025, Yongtao Zou and colleagues [33] measured the sound velocities of polycrystalline hematite to 6.5 GPa and 1100 K; Yongtao Zou, who was previously a post-doctoral researcher at Stony Brook, used a hematite specimen from our laboratory.

See relationship of mineral physics studies to other fields of research in the Earth sciences (Figure 14).

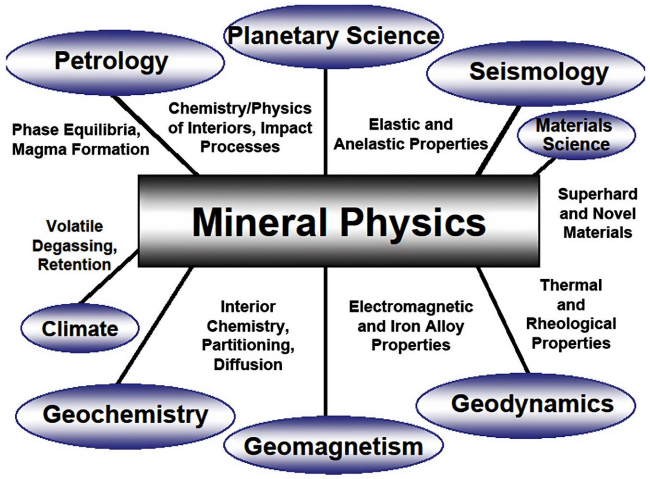


Figure 14. Relationship of mineral physics to other fields of research on the Earth's interior.

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Conflicts of Interest

The author declares no conflicts of interest regarding the publication of this paper.

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