Combined *in situ* Synchrotron X-Ray Diffraction and Ultrasonic Interferometry Study of ε-FeSi at High Pressure and Temperature

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Presentation preference (please check one): Oral ( ) Poster (CHECK!!)

1. Introduction

It has long been accepted that the core of the Earth, as well as those of other planetary bodies, is predominantly made up of metallic iron (or an iron-nickel alloy), but several studies have shown that metallic Fe is too dense to be the sole element in the Earth's core; particularly the solid inner core [i.e. 1, 2]. This indicates that there must be some amount of one or more light elements in the core, and in order to assess which elements these might be, and in what possible proportions, we must study the physical properties of iron-light element alloys under extreme conditions and compare them with the information gained from seismic studies on the Earth's interior. This study focuses on one such alloy: ε-FeSi.

2. Experimental

The starting FeSi material was purchased in powdered form from Alfa Aesar. Powder diffraction conducted on this starting powder revealed that the powder was pure homogeneous ε-FeSi in composition. This powder was then vigorously ground by hand in using an agate mortar and pestle for approximately 30 minutes; the resulting fine powder had an average grain size on the micron scale.

This fine powder was then tightly packed into a gold capsule, dried at 150°C for 2 hours, and then the capsule was pressure sealed to ensure no readsorption of moisture. This capsule was then placed inside a standard 14/8 octahedral cell assembly and prepared for a hot-pressing experiment in a Walker-type 1000-ton uniaxial split-cylinder apparatus. The sample was hot-pressed at 700°C and 7 GPa for exactly one hour.

The hot-pressed sample was analyzed using beamline X17B2 at the National Synchrotron Light Source at Brookhaven National Laboratory to check for any possible heterogeneity in composition or grain size, none of which were found. The *in situ* experiment was conducted in the DDIA-type SAM85 press installed at beamline X17B2 at the NSLS. A schematic of the experimental setup is shown in Figure 1. The cell assembly used in the experiment is shown in Figure 2. The ultrasonic measurements were conducted using a dual-mode transducer that was capable of generating frequencies from 20 to 70 MHz.

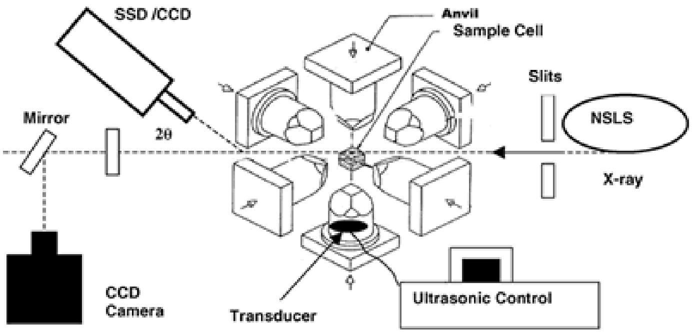

Fig.1. Schematic diagram of the experimental setup used at beamline X17B2 at the NSLS for this study. (After [3]).

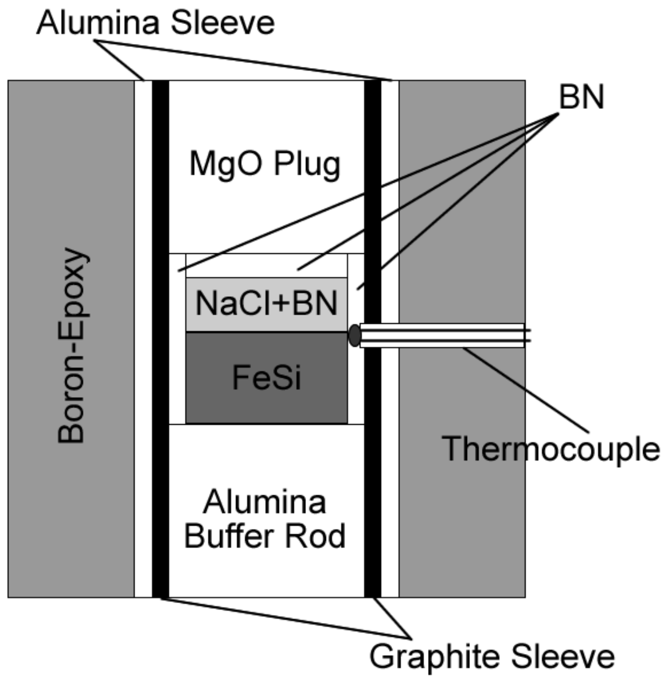

Fig.2. Schematic diagram of the experimental cell assembly used in this study. The NaCl + BN is a powdered mixture in 10:1 proportions to prevent significant grain growth during the experiment. A 1μm-thick disc of Au foil is placed above and below the sample and at the bottom of the buffer rod to smooth all contact surfaces.

The cell assembly was placed inside the SAM85 press, and brought up to the maximum oil pressure of 60 tons at room T, stopping every five tons along the way to collect data. Then, 10 cycles of data were collected by heating, cooling, and decreasing the pressure. At each desired set of conditions, an X-Ray image of the sample was taken using the CCD Camera shown in Figure 1, ultrasonic data was collected, and an X-Ray diffraction pattern for both the NaCl pressure calibrant and the FeSi sample were collected. A typical sample X-Ray image is shown in Figure 3.

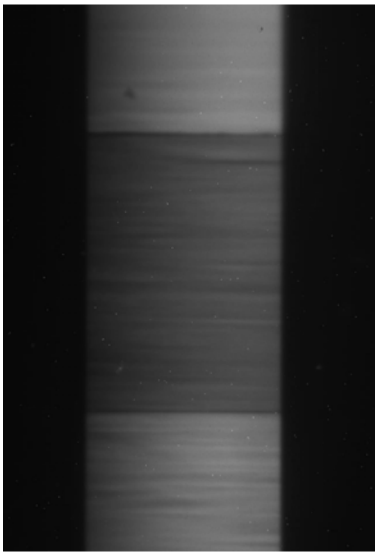

Fig.3. X-Ray image of the sample collected at 15 tons of oil pressure during initial cold compression. Black areas to left and right are the anvils. Dark gray area in the center is the FeSi sample. Light area above the sample is the NaCl, and the light area below is the buffer rod. Dark lines at top and bottom of sample are the Au foil discs.

3. Results and discussion

Over 70 data sets were collected during this experiment, each one at a unique set of temperature and pressure conditions following a "saw-tooth pattern" typical of this type of experiment. The conditions under which each data set was collected is shown in Figure 4. Note that in all of the following plots, the pressure used is that obtained via cell refinement of the NaCl pressure calibrant.

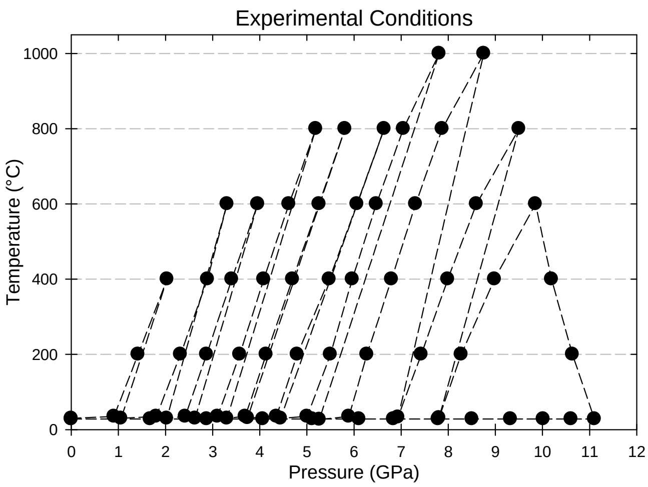

Fig.4. Plot showing all of the conditions at which data were collected for this study.

By determining the length of the sample in pixels at the end of the experiment when the press is opened, and then measuring the absolute length of the sample after the experiment, we can calibrate the pixel to length ratio, and thereby determine the absolute length of the sample at all P-T conditions (Figure 5). We use the pulse echo overlap (PEO) technique to determine the two-way travel times of P and S waves going through the sample; the results along cold compression are shown in Figure 6. From the lengths and travel times, we can then directly obtain the P and S wave velocities for the solid FeSi sample. We use the X-Ray data to determine the cell volume of the sample, which can give us a handle on the compressibility of the sample material through equation of state analysis. More details about the experiment, as well as the results of the bulk and shear moduli, and their pressure and temperature dependence derived from these data, will be presented at the workshop.

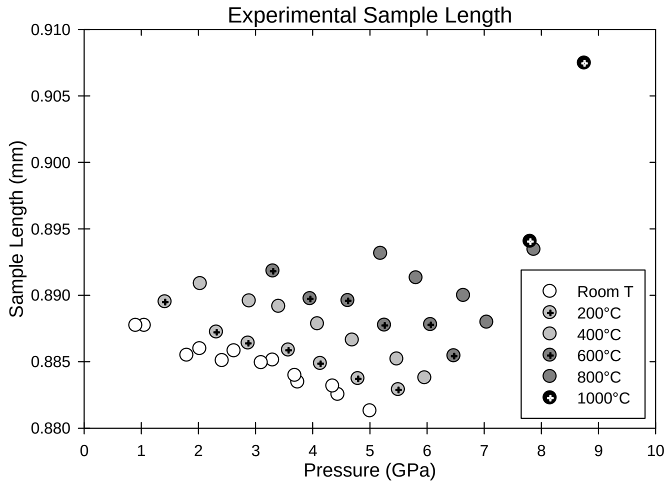

Fig.5. Plot of the sample length obtained at each data point as a function of pressure. Data is organized in sets by T.

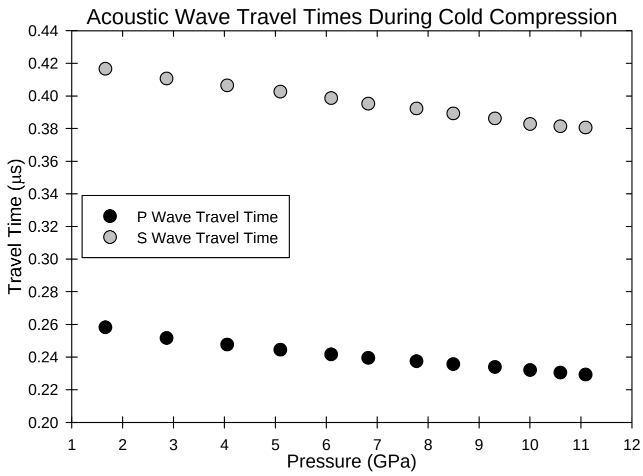

Fig.6. Plot of P and S vave 2-way travel times in the FeSi Sample as a function of pressure during cold compression.

References

[1] Jephcoat, A. & Olson, P. (1987) Nature 325: 332-335.
[2] Mao, H.K., et al. (1998) Nature 396: 741-743.
[3] Li, B., Kung, J. & Liebermann, R.C. (2004) PEPI 143-44: 559-574.