The sound velocity measurements of Fe$_3$S

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**ABSTRACT**

We measured the sound velocity of Fe$_3$S at room temperature up to 85 GPa employing inelastic X-ray scattering to better constrain the constitution of the inner core. The density of Fe$_3$S was also determined by X-ray diffraction under the same conditions. The relation of the P-wave velocity ($v_P$) and density ($\rho$) of Fe$_3$S follows Birch's law, $v_P$(m/s) = 1.14(5) × ρ(kg/m$^3$) – 2580(410). Based on Birch’s law determined here for Fe$_3$S and that for ε-Fe reported previously, we found that sulfur decreases both density and compressional velocity of hcp-Fe at the core pressure and 300 K.

**Keywords:** Fe$_3$S, inner core, sound velocity, Birch’s law, inelastic X-ray scattering

**INTRODUCTION**

The Earth’s core has been studied seismologically, and seismic wave velocities and the density of the core are important observable properties (e.g., Birch 1964; Dubrovinsky et al. 2000). According to these studies, the inner core is less dense than pure iron, and it has been accepted that the inner core is comprised of iron and light elements (e.g., Poirier 1994). Sound velocity data are more accurate than density in seismology and can provide important constraints on the structure and composition of the inner core (e.g., Badro et al. 2007; Cao et al. 2005; Song and Helmberger 1998). In spite of their importance, the sound velocity data of the core materials at high pressure are still very limited due to technical difficulties although many experiments to constrain the density of the inner core have been carried out using in situ X-ray diffraction (e.g., Chen et al. 2007).

Sulfur has been suggested as one of the most plausible light elements in the Earth’s core (e.g., Campbell et al. 2007; Seagle et al. 2006). The S content in the core is estimated to be at least a few weight percent (Hillgren et al. 2000; McDonough 2003). In previous work, phase relationships in the Fe–FeS system under high pressures were investigated (e.g., Fei et al. 2000; Kamada et al. 2010). Kamada et al. (2010) confirmed that Fe$_3$S is stable with ε-Fe up to 220 GPa, and Fe$_3$S is, therefore, one of the candidates of the inner-core materials. The compression behavior of Fe$_3$S was investigated up to 80 GPa (Seagle et al. 2006; Chen et al. 2007). Chen et al. (2007) estimated the S content to be 12.5–20.7 at% (7.9–13.0 wt%) in the outer core and 2.2–6.2 at% (1.4–3.7 wt%) in the inner core. Other studies have investigated the sound velocities, $v_P$ of iron and iron–light element alloys (e.g., Antonangeli et al. 2004; Badro et al. 2007; Fiquet et al. 2001). Although sound velocities of FeS and FeS$_2$ have been measured according to inelastic X-ray scattering (IXS), there are very limited data for Fe$_3$S. FeS and FeS$_2$ may not be appropriate for the inner core materials because the Fe–S system has several iron-rich intermediates such as Fe$_3$S$_2$, FeS, and Fe$_3$S coexisting with Fe and high-pressure polymorphs in FeS at high pressure (Fei et al. 1995). Only Fe$_3$S coexists with ε-Fe under the core conditions as a subsolidus phase (Kamada et al. 2010, 2012). Therefore, it is essential to study $v_P$ of Fe$_3$S to understand the seismic and chemical properties of the Earth’s core. Lin et al. (2004) studied $v_P$ of Fe$_3$S up to 57 GPa employing nuclear resonance inelastic X-ray scattering (NIS) and found that it follows Birch’s law, which suggest the linear relationship between density and sound velocity (Birch 1961). They also reported that the sound velocity of Fe$_3$S changes significantly across the magnetic transition occurred at approximately 21 GPa. However the application of the results to the Earth’s core was not made due to a narrow pressure interval of their NIS measurements. In this research, we measured $v_P$ of Fe$_3$S based on inelastic X-ray scattering method up to 85 GPa.

**EXPERIMENTAL METHOD**

The starting material of Fe$_3$S was synthesized by a multi-anvil press from a mixture of powdered Fe (99.9% purity) and FeS (99.9% purity) with the ratio of Fe:S = 85:15 (wt%). The synthesis conditions were 21 GPa and temperatures between 1263 and 1553 K. The sample was then reduced to 21 GPa at room temperature and then heated above the melting temperature at 1533 K for 5 min to homogenize the mixture. The temperature was then lowered to 1413 K and kept at that temperature for 30 min to crystallize Fe$_3$S from the Fe–S liquid. The temperature was then reduced to 1263 K and kept at that temperature for 1 h for the growth of the Fe$_3$S crystals. The chemical composition of the recovered sample was determined to be Fe$_{3+x}$S$_{1-x}$ (in at%) using a scanning electron microscope with energy-dispersive X-ray spectroscopy and its backscattered image is shown in Figure 1. A small fragment of the recovered sample was used for inelastic X-ray scattering (IXS) experiments at high pressures.

A symmetric diamond-anvil cell was used to generate high pressure. The culet