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Pressure-induced magnetic transition and sound velocities of Fe_3C: Implications for carbon in the Earth's inner core

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[1] We have carried out nuclear resonant scattering measurements on ^{57}Fe-enriched Fe_3C between 1 bar and 50 GPa at 300 K. Synchrotron Mössbauer spectra reveal a pressure-induced magnetic transition in Fe_3C between 4.3 and 6.5 GPa. On the basis of our nuclear resonant inelastic X-ray scattering spectra and existing equation-of-state data, we have derived the compressional wave velocity V_P and shear wave velocity V_S for the high-pressure nonmagnetic phase, which can be expressed as functions of density (ρ): V_P(km/s) = $-3.99 + 1.29\rho$(g/cm^3) and V_S(km/s) = $1.45 + 0.24\rho$(g/cm^3). The addition of carbon to iron-nickel alloy brings density, V_P and V_S closer to seismic observations, supporting carbon as a principal light element in the Earth's inner core. **Citation:** Gao, L., et al. (2008), Pressure-induced magnetic transition and sound velocities of Fe_3C: Implications for carbon in the Earth's inner core, *Geophys. Res. Lett.*, *35*, L17306, doi:10.1029/2008GL034817.

1. Introduction

[2] Carbon is one of the candidate light elements for the Earth's core [*Li and Fei*, 2007, and references therein]. *Wood* [1993] proposed that for most conceivable sulfur to carbon ratios, Fe_3C could be the major inner core component rather than iron-nickel alloy, and may account for the density deficit in the inner core. A critical test for this hypothesis is to compare the density and sound velocities of Fe_3C with those of the inner core under corresponding pressure and temperature conditions.

[3] Upon compression, Fe_3C transforms into a non-magnetic phase. The effects of the pressure-induced magnetic transition on the density and the compressibility of Fe_3C remain controversial. Within experimental uncertainties, X-ray diffraction measurements at 300 K do not show any discontinuity in the compression curve of Fe_3C up to 73 GPa [*Scott et al.*, 2001; *Li et al.*, 2002]. On the other hand, an *ab initio* study predicted a pressure-induced magnetic transition at 60 GPa and 0 K and found the high-pressure nonmagnetic phase less compressible than the low-pressure magnetic counterpart [*Vočadlo et al.*, 2002]. Experimental measurements at 300 K confirmed the occurrence of a magnetic transition under high pressure, but indicated different transition pressures for X-ray emission spectroscopy (XES) (25 GPa) [*Lin et al.*, 2004a] versus for X-ray magnetic circular dichroism (XMCD) (9 GPa) [*Duman et al.*, 2005] measurements. Synchrotron Mössbauer spectroscopy (SMS) is an established technique to investigate the electronic configuration and magnetic ordering of iron in iron-bearing phases [e.g., *Alp et al.*, 1995; *Sturhahn and Jackson*, 2007, and references therein]. SMS measurements can provide an independent constraint on the pressure of the magnetic transition, and allow a reassessment of the compressibility of Fe_3C.

[4] On the V_P versus density plot, the inner core has a different slope from pure iron at 300 K, although the two linear trends overlap within the inner core density range [*Lin et al.*, 2003; *Mao et al.*, 2004]. In contrast, the linear trends of V_S versus density of the inner core and pure iron at 300 K are nearly parallel, but with a considerable offset of ~2 km/sec. The mismatch in density and velocities can not be explained by the addition of nickel but may reflect the effect of temperature and/or the presence of light elements such as carbon. To date, the only velocity data on Fe_3C have been calculated from ultrasonic measurements on porous samples at 1 bar and 300 K [*Dodd et al.*, 2003]. Nuclear resonant inelastic X-ray scattering (NRIXS) is a newly-developed technique for measuring the partial phonon density of state (PDoS) of iron in iron-rich alloys [e.g., *Sturhahn and Jackson*, 2007]. Combining high-pressure PDoS with equation of state (EoS) data, compressional and shear velocities of compressed Fe_3C can be derived.

2. Experimental Procedure

[5] An Fe_3C sample was synthesized from a mixture of 94.45% ^{57}Fe-enriched iron (Cambridge Isotope Laboratories Inc., # FLM-1812-0) and graphite powder (Sigma-Aldrich, # 282863) at an atomic ratio of Fe:C = 2.922:1. The mixture was packed in an MgO capsule and equilibrated at 3 GPa and 1373 K for 19 hours, using the multi-anvil apparatus at the University of Illinois. Conventional Mössbauer measurements at ambient condition confirmed that the run product is pure Fe_3C, with a magnetic hyperfine field of 21.8 (±2.7) T, in accordance with previous results [e.g., *Ron and Mathalone*, 1971]. The lattice parameters of the ortho-

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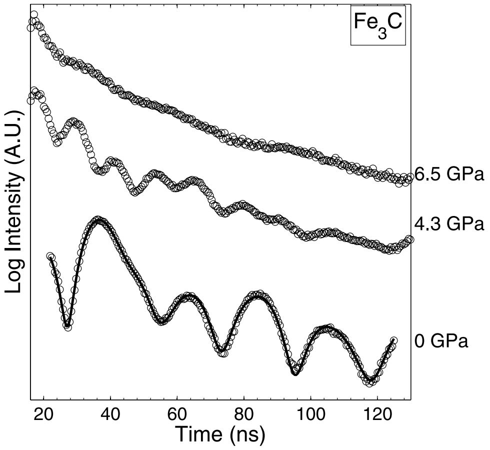

Figure 1. Synchrotron Mössbauer spectra of Fe$_3$C (open circles) and fitting results for the 1-bar data (solid curve). The loss of fast oscillations between 4.3 and 6.5 GPa indicates the occurrence of a pressure-induced magnetic transition.

rhombic Fe$_3$C phase (space group Pnma, #62) are $a = 5.080$ Å, $b = 6.758$ Å, $c = 4.514$ Å, based on a high-resolution X-ray diffraction pattern collected at 11-BM of the Advanced Photon Source (APS), Argonne National Laboratory.

[6] SMS and NRIXS experiments were performed at the undulator beamline 3-ID-B of the APS. The focused X-ray beam was less than 10 μm in diameter and has an energy resolution of 1.0 meV. SMS measurements were conducted in a symmetrical diamond-anvil cell (DAC) with a Re gasket and methanol-ethanol-water (16:3:1 by volume) pressure medium, which has been shown to maintain hydrostaticity up to 15.4 GPa [*Fujishiro et al.*, 1982]. An avalanche photodiode detector (APD) was placed along the X-ray beam path to collect SMS signals. Typical collection time for each SMS spectrum was 30 minutes. The CONUSS program package [*Sturhahn*, 2000] was used to extract magnetic hyperfine field parameters from synchrotron Mössbauer spectra.

[7] NRIXS measurements were carried out on a piece of polycrystalline Fe$_3$C sample in a panoramic DAC using flat diamonds with 300-μm culet size, a Be gasket, NaCl pressure medium and ruby balls as the pressure marker [*Mao et al.*, 1978]. Three APDs were used to collect the delayed fluorescence radiation from three directions perpendicular to the X-ray beam. The diameter of the sample chamber was kept within 50–70 μm to limit pressure gradients and self-absorption of the inelastic scattering signals by the sample. Each NRIXS spectrum was collected over an energy range of -70 to $+90$ meV around the ^{57}Fe nuclear resonance energy of 14.4125 keV in steps of 0.2 meV. The collection time was typically 4 hours for each pressure. Partial PDoS was derived from NRIXS data using the program PHOENIX [*Sturhahn*, 2000].

3. Results and Discussion

3.1. Magnetic Transition and Core Density Deficit

[8] At 1 bar and 300 K, Fe$_3$C is ferromagnetic. The SMS spectrum at 1 bar can be fitted assuming one iron site with a hyperfine field of 20.0(±2.4) T (Figure 1), consistent with the known value of ~20.5 T [e.g., *Ron and Mathalone*, 1971], and with the conventional Mössbauer measurements on the same sample. A loss of magnetism was observed between 4.3 and 6.5 GPa, as indicated by the disappearance of fast oscillations in the SMS spectra (Figure 1).

[9] This magnetic transition pressure is lower than the XES results showing a gradual reduction in the satellite intensity between 1 bar and ~25 GPa [*Lin et al.*, 2004a], and the XMCD results showing a continuous decrease of integrated intensity between ~6 and ~15 GPa [*Duman et al.*, 2005]. The discrepancy in transition pressure may reflect the uncertainties introduced in data evaluation and pressure calibration.

[10] Within the experimental uncertainties, the magnetic transition between 4.3 GPa and 6.5 GPa appears invisible in the compression curve of Fe$_3$C [*Scott et al.*, 2001; *Li et al.*, 2002], therefore we can use the set of V_0, K_0 and K' values from *Scott et al.* [2001] to estimate the density of Fe$_3$C at core pressures. For direct comparison with previous studies, we calculated the density of Fe$_3$C at an average inner core pressure of 338 GPa and a likely inner core temperature of 5300 K, using the third-order Birch-Murnaghan EoS and estimated thermal expansion coefficients at 338 GPa following the method described by *Wood* [1993] and *Vočadlo et al.* [2002]. Our results suggest that Fe$_3$C is 2.4% lighter than the inner core at these pressure and temperature conditions, similar to the previous result of 2.6% [*Lin et al.*, 2004a], but less than 3.8% [*Vočadlo et al.*, 2002] and more than 0.9% [*Wood et al.*, 2004] (Table 1). The density of Fe at 338 GPa and 5300 K is 2.9% higher than that of the inner core (Table 1). Indeed, Fe$_3$C alone cannot reproduce

Table 1. EoS Parameters and Densities of HCP-Fe and Fe$_3$C

Reference	V_0 (Å^3)	K (GPa)	K'	α ($\times 10^{-5}$ K^{-1})[a]	ρ (g/cm^3)[b]
		Fe$_3$C			
Scott et al. [2001]	155.26	175.00	5.2	0.385	12.54
Lin et al. [2004a]	148.00	288.00	4.0	0.448	12.52
Vočadlo et al. [2002][c]	143.40	316.62	4.3	0.542	12.35
Wood [1993]	154.82	174.00	5.1	0.335	12.74
		Fe			
Mao et al. [1990]	22.2	166.00	5.5	0.69 – 1.25	13.22
Isaak and Anderson [2003]					

[a] Thermal expansion coefficient α of Fe$_3$C at 338 GPa is estimated based on $\alpha_0 = 4.1 \times 10^{-5}$ K^{-1} for the high-temperature paramagnetic phase at 1 bar and 480–600 K [*Wood et al.*, 2004] following the method described by *Wood* [1993] and *Vočadlo et al.* [2002]. Thermal effect on α is ignored.

[b] Calculated values at 338 GPa and 5300 K; see text for details.

[c] The reference temperature of V_0, K, K' is 0 K as given by *Vočadlo et al.* [2002] and 300 K in other EoS's.

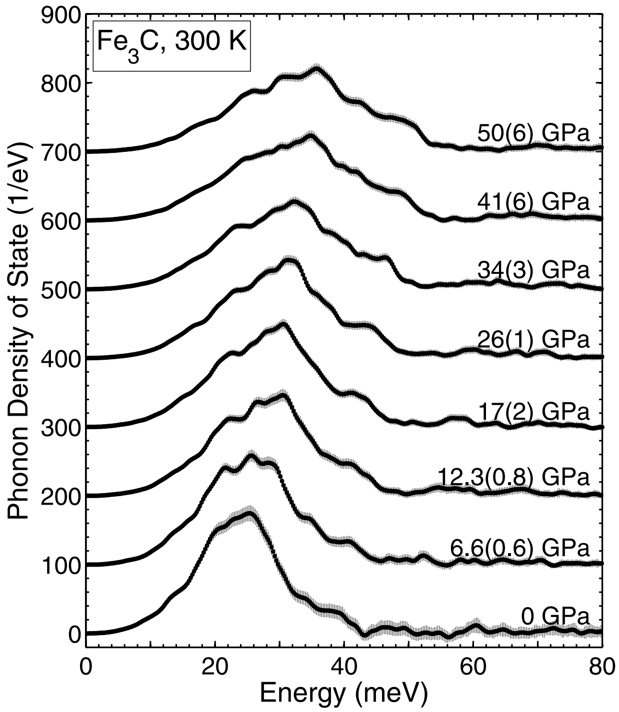

Figure 2. Fe partial phonon density of states (PDoS) of Fe$_3$C extracted from NRIXS spectra between 1 bar and 50 GPa, at 300 K. High-pressure spectra are shifted vertically for clarity.

the inner core density. To account for the density deficit in the inner core, ~3 wt.% carbon (equivalent to about 50% Fe$_3$C) is needed. This estimate is sensitive to the EoS parameters and the core temperature and needs to be revised when more accurate data become available.

3.2. Sound Velocities, Poisson's Ratio and Birch's Law

[11] From the NRIXS spectra collected at 300 K and up to 50 GPa, we derived the partial PDoS of Fe in Fe$_3$C (Table 2 and Figure 2). At each pressure, the Debye velocity (V_D) of Fe$_3$C is extracted from a parabolic fitting of the low-energy portion of the PDoS between 3.0 and 12.0 meV [*Hu et al.*, 2003]. The following relations allow us to calculate aggregate compressional velocities V_P, shear wave velocity V_S, shear modulus G and Poisson's ratio ν from V_D and EoS parameters:

$$\frac{3}{V_D^3} = \frac{1}{V_P^3} + \frac{2}{V_S^3} \tag{1}$$

$$\frac{K_S}{\rho} = V_P^2 - \frac{4}{3} V_S^2 \tag{2}$$

$$\frac{G}{\rho} = V_S^2 \tag{3}$$

$$\nu = \frac{3K_S - 2G}{2(3K_S + G)} = \frac{\left(\frac{V_P}{V_S}\right)^2 - 2}{2\left(\left(\frac{V_P}{V_S}\right)^2 - 1\right)} \tag{4}$$

[12] V_P, V_S and ν of Fe$_3$C are calculated using the EoS parameters of Fe$_3$C at 300 K [*Scott et al.*, 2001]. The difference between the adiabatic bulk modulus K_S and the isothermal bulk modulus K_T at 300 K (<2%, corresponding to <1% difference in V_P) is within experimental uncertainties, hence ignored in the calculation.

[13] Compared with porosity-corrected values from ultrasonic measurements [*Dodd et al.*, 2003], our V_P at ambient condition is 11–15% higher. The ultrasonic results appear questionable, as the same study also gives a much smaller bulk modulus K_0 (105–125 GPa) than determined by X-ray diffraction measurements [*Scott et al.*, 2001; *Li et al.*, 2002], and a Poisson's ratio of 0.22–0.27, smaller than the known values of various Fe-rich alloys (0.27–0.37) [*Mao et al.*, 2001; *Lin et al.*, 2003, 2004b, 2005; *Mao et al.*, 2004].

[14] V_P and V_S of Fe$_3$C do not increase smoothly with density (Figure 3). The V_P of the low-pressure magnetic phase plots slightly below the linear trend of the high-pressure non-magnetic phase, whereas the V_S of the magnetic phase

Table 2. Compressional Wave Velocity V_P, Shear Wave Velocity V_S, Isothermal Bulk Modulus K_T, Shear Modulus G and Poisson's Ratio ν of Fe$_3$C at 300 K

P (GPa)	ρ (g/cm^3)[a]	V_P (km/s)	V_S (km/s)	K_T (GPa)	G (GPa)	ν
			Fe$_3$C [Dodd et al., 2003]			
0	7.68(0)	5.33–5.14	3.01–3.08	105–125	69–72	0.22–0.27
			^{57}Fe$_3$C (This Study)			
0	7.83(0)	5.89(5)	3.05(7)	175(4)	73(3)	0.32(1)
16.6(6)	8.10(4)	6.43(9)	3.4(1)	209(9)	95(6)	0.30(1)
12.3(8)	8.31(5)	6.7(1)	3.48(6)	239(12)	101(2)	0.32(1)
17(2)	8.47(9)	6.9(2)	3.50(6)	263(20)	104(3)	0.33(2)
26(1)	8.75(7)	7.3(1)	3.60(6)	310(17)	113(3)	0.34(2)
34(3)	9.0(1)	7.6(2)	3.66(6)	352(30)	120(2)	0.35(1)
41(6)	9.2(2)	7.7(3)	3.60(6)	388(48)	119(3)	0.36(2)
50(6)	9.4(2)	8.1(3)	3.77(6)	435(50)	133(3)	0.36(2)

[a] The density of ^{57}Fe$_3$C is higher than that of natural Fe$_3$C used by *Dodd et al.*'s [2003] study. Numbers in the parentheses are uncertainties in the last digit(s). Contributions to uncertainties include: pressure - the pressure differences between different rubies and before and after NRIXS measurements; density - pressure and EoS parameters; V_P, V_S and G - pressure, EoS, and PDoS parabolic fitting parameters; K_T - pressure and EoS parameters; ν-V_P and V_S.

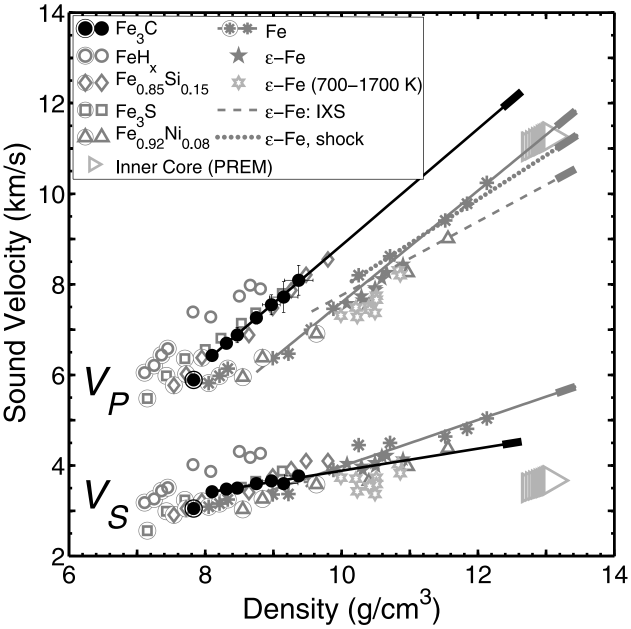

Figure 3. Comparison of V_P and V_S versus density between Fe_3C and other Fe-rich alloys. Density of $^{57}Fe_3C$ is used for all NRIXS data. All data are at 300 K unless otherwise indicated. Magnetic phases are marked by outer circles. Long solid lines are linear fits to the data of the non-magnetic phases of Fe_3C (black) and ϵ-Fe (gray). Short solid bars mark the density of Fe_3C (black) and ϵ-Fe (gray) at inner core pressures and 5300 K (see text and Table 2 for details). Data sources: Fe_3C (this study); FeH_x [*Mao et al.*, 2004]; $Fe_{0.85}Si_{0.15}$ [*Lin et al.*, 2003]; Fe_3S [*Lin et al.*, 2004a]; $Fe_{0.92}Ni_{0.08}$ [*Lin et al.*, 2003]; ϵ-Fe (asterisk) [*Mao et al.*, 2001]; ϵ-Fe (star) and ϵ-Fe (700–1700 K) [*Lin et al.*, 2005]; ϵ-Fe, shock [*Brown and McQueen*, 1986]; ϵ-Fe, IXS [*Fiquet et al.*, 2001].

plots well below the linear trend of the non-magnetic phase, reflecting a significant increase in shear modulus across the magnetic transition boundary. The effects of the magnetic transition on the sound velocities of Fe_3C are similar to FeH_x, but different from pure iron, Fe-Ni, Fe_3S, and $Fe_{0.85}Si_{0.15}$, of which the V_P and V_S decrease upon magnetic transition, due to an increase in density and/or decrease in shear modulus.

[15] For the non-magnetic phase of Fe_3C, V_P increases linearly with density (ρ): V_P(km/s) = −3.99 + 1.29ρ(g/cm^3) (Figure 3). *Birch* [1961] found that at pressures above a few kilobars (when most cracks are closed), the principal factors determining V_P are the density and the mean atomic mass M: V_P(km/s) = $a + b \cdot \rho$(g/cm^3), where the constant a(km/s) generally decreases with increasing M, and $b = 3.05$ ((km $\cdot$ cm^3)/(s $\cdot$ g)) for a large number of mantle minerals. Like other iron-rich alloys at 300 K, the linear relation between the V_P and ρ of Fe_3C is consistent with Birch's law. The mean atomic mass of Fe_3C (44.9) is smaller than that of Fe_3S (49.9) and $Fe_{0.85}Si_{0.15}$ (51.7), yet at a given ρ, V_P of Fe_3C is similar to or lower than that of Fe_3S and $Fe_{0.85}Si_{0.15}$, contrary to expected from Birch's law. At 300 K, the Birch's law slope of the non-magnetic Fe_3C is similar to that of pure iron, but significantly steeper than PREM (Figure 3). Extrapolated to the inner core pressures, V_P of Fe_3C is $\sim$ 10% higher than the PREM value. The presence of carbon can counter the effect of nickel to bring a closer match between the V_P of Fe-Ni alloy and the inner core.

[16] V_S of the non-magnetic phase of Fe_3C also increases linearly with density: V_S(km/s) = 1.45 + 0.24 ρ(g/cm^3). Interestingly, the slope is smaller than that of pure iron at 300 K. As a result, the extrapolated V_S of Fe_3C at inner core pressure is 1.5 km/s lower than that of pure iron, and considerably closer to the PREM value (Figure 3). Although the presence of other light elements can not be ruled out, carbon may hold the key to explaining the low shear velocity of the inner core.

[17] Poisson's ratio is an important seismic observation that provides an additional constraint on models of core composition. The observed value of the inner core is 0.44 [*Dziewonski and Anderson*, 1981]. At 300 K, the measured Poisson's ratios of iron, Fe-Ni alloys, FeH_x, Fe_3S and $Fe_{0.85}Si_{0.15}$ are smaller than 0.35 and show little pressure dependence [*Mao et al.*, 2001; *Lin et al.*, 2003, 2004b, 2005; *Mao et al.*, 2004]. The Poisson's ratio of the non-magnetic Fe_3C at 300 K falls into a comparable range to other iron-rich alloys, but gradually increases from 0.30 at 6.6 GPa to 0.36 at 50 GPa (Table 2), approaching the PREM value.

[18] Recent NRIXS measurements on iron revealed a significant reduction of V_P and V_S at high temperature, and a deviation of the V_P-density relation from Birch's law [*Lin et al.*, 2005]. In order to provide a stringent test for the hypothesis of a carbon-rich inner core, the effects of temperature on the sound velocities of Fe_3C need to be investigated.

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