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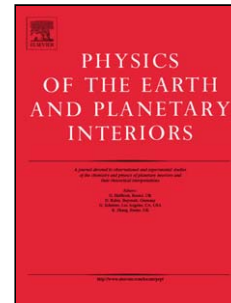
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# Sound velocity in iron carbide ( $\text{Fe}_3\text{C}$ ) at high pressure: implications for the carbon content of the Earth's inner core

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## Abstract.

We measured compressional sound velocities of  $\text{Fe}_3\text{C}$ -cohenite at high-pressure by inelastic x-ray scattering (IXS). We show that  $\text{Fe}_3\text{C}$  follows Birch's law for the longitudinal acoustic velocity  $V_P$ , namely a linear dependence between velocity and density. This dataset completes the previous sets recently established by Badro et al. (2007) for  $\text{FeO}$ ,  $\text{FeSi}$ ,  $\text{FeS}$ , and  $\text{FeS}_2$ , and provides new mineralogical constraints on the composition of Earth's core. Our results, combined with data already obtained for other iron alloys, are compared with seismic data. This suggests that a reduced carbon amount in the inner core could reasonably explain density and velocity differences between measurements made on pure iron and seismic models. This conclusion, however, depends on the remaining uncertainty on magnetic structure for a very low carbon content in the iron alloy. It does not preclude the incorporation of another light element in the inner core, such as silicon.

## 1. Introduction.

Light elements are assumed to be present in the Earth's core to account for the density and sound wave velocity discrepancies between seismological models such as PREM (Dziewonski and Anderson, 1981) and pure Fe or Fe-Ni alloys. Among these possible alloying elements, Si, O, H, S and C are considered to be the most likely additional components, as indicated by several lines of arguments such as cosmochemical abundances and high-pressure solubility (e.g., Birch, 1952; Poirier, 1994; Allègre et al., 1995; McDonough and Sun, 1995; Takafuji et al., 2005). The possibility that carbon might be an important constituent of the Earth's core has also been examined by Wood (1993). Carbon stability in carbides is indeed greatly enhanced at high-pressure, and extrapolations to inner core pressures indicate that  $\text{Fe}_3\text{C}$  is expected to crystallize rather than hcp-iron, which could make of  $\text{Fe}_3\text{C}$  a major phase in the Earth's inner core. A further constraint on the viability of  $\text{Fe}_3\text{C}$  as a major phase in the core can be obtained by comparison of its incompressibility with those derived from seismological data. However, these studies have led to date to different conclusions. Experimental high-pressure Equations of State (EoS) data indicate that  $\text{Fe}_3\text{C}$  could have a density at an average inner core pressure in excellent agreement with the density range determined from seismic data (Scott et al., 2001; Li et al., 2002). On the other hand, first-principle calculations predict values for the density and adiabatic incompressibility that differ significantly at core pressures and temperatures, so as to preclude  $\text{Fe}_3\text{C}$  as the major inner core-forming phase (Vočadlo et al., 2002).

In this work, we address the important question of the significance of  $\text{Fe}_3\text{C}$  in the inner core with measurements of propagation of aggregate sound velocity in  $\text{Fe}_3\text{C}$  at high-pressure, using the IXS technique. It has been shown that Birch's law (*i.e.* the linear dependence of longitudinal acoustic velocity as a function of density) holds for many iron alloys compounds, which provides a way to compare sound velocity properties of pure iron or iron alloyed compounds with seismological models for the Earth's core (Fiquet et al., 2001; Antonangeli et al., 2004; Badro et

al., 2007). The comparison of these mineral physics data with seismic data allow us to constrain the relative abundance of  $\text{Fe}_3\text{C}$  in the Earth's core, not only on the basis of density systematic but also using acoustic sound velocity measurements.

## 2. Experimental method.

Owing to third generation synchrotron light sources, it is now possible to use the IXS technique to study phonon excitations in a sample at high pressure in a diamond anvil cell (DAC). For instance, aggregate acoustic sound velocities have been reported for iron and iron-bearing compounds ( $\text{FeS}$ ,  $\text{FeS}_2$ ,  $\text{FeO}$  and  $\text{FeSi}$ ) at megabar pressures (Badro et al., 2007) and data have been published on single crystal of cobalt to 40 GPa (Antonangeli et al., 2004), demonstrating the capability of IXS to study the dynamics of systems under high pressure in the diamond-anvil cell. Following these previous experimental achievements, we have collected IXS spectra to 83 GPa on a polycrystalline sample of  $\text{Fe}_3\text{C}$  in the diamond-anvil cell. It must be noted that such experiments carried out on polycrystalline samples provide an average longitudinal acoustic velocity and its pressure (and density) dependence, which can be used to directly compare with aggregate properties sampled in seismic observations.  $\text{Fe}_3\text{C}$  samples of high purity have been synthesized from pure iron and graphite powder in a  $\text{MgO}$  capsule at 2 GPa and 1273 K, using a piston-cylinder apparatus. x-ray diffraction measurements and electron microprobe analyses confirmed that the structure and composition of the product matches that of cementite (space group  $\text{Pbnm}$ ,  $a = 4.518 \text{ \AA}$ ,  $b = 5.069 \text{ \AA}$  and  $c = 6.736 \text{ \AA}$  with  $Z = 4$ ; see Li et al., 2002).  $\text{Fe}_3\text{C}$  is referred to as cementite in metallurgy, where it is extensively mentioned as an important constituent of steels and cast irons. In nature,  $\text{Fe}_3\text{C}$  occurs as a minor constituent in iron meteorites and is named "cohenite". Some cohenite was also found as inclusions in garnets associated to a polycrystalline diamond aggregate from the Venetia kimberlite (Limpopo belt, South Africa) - see Jacob et al. (2004). The synthesized  $\text{Fe}_3\text{C}$  sample was loaded in Mao-Bell type diamond anvil cells or membrane-driven cell, equipped with diamond anvils with  $300 \text{ }\mu\text{m}$

flat culets or 150x300  $\mu\text{m}$  beveled culets. Neon was used as pressure transmitting medium, so as to avoid developing preferred orientation in the sample with increasing pressure. Diffraction pattern collected at each pressure step yield compression in excellent agreement with previously reported quasi-hydrostatic equation of states (Scott et al., 2001; Li et al., 2002). In addition, the samples were compressed and allowed to relax at room temperature for about 24 hours after each pressure increase. Measurements were performed at pressures up to 83 GPa.

Sound velocities were measured at high-pressure by very high resolution IXS at the beamline ID28 of the European Synchrotron Radiation Facility (ESRF). The instrument was operated in the Si (8,8,8) configuration, with an incident photon energy of 15.817 keV and a total instrumental energy resolution of 5.5 meV full width at half maximum (FWHM). The transverse dimensions of the focused x-ray beam of 25 x 60  $\mu\text{m}^2$  (horizontal x vertical, FWHM) were further reduced by slits at the highest pressures. The momentum transfer  $Q=2k_i \sin (\theta_s/2)$ , where  $k_i$  is the incident photon wave vector and  $\theta_s$  is the scattering angle, was selected by rotating the spectrometer around a vertical axis passing through the sample in the horizontal plane. The momentum resolution was set by slits in front of the analyzers to 0.25  $\text{nm}^{-1}$ . Further details of the experimental setup have been reported elsewhere (Krisch, 2003; Fiquet et al., 2004). The dispersion of longitudinal acoustic (LA) phonons was measured for 5 to 9 values of the momentum transfer between 4 and 12.5  $\text{nm}^{-1}$ . Typical IXS pattern are presented in Figure 1. The pattern are characterized by an elastic contribution, centered at zero energy, and an inelastic contribution from a longitudinal acoustic mode from  $\text{Fe}_3\text{C}$  and a transverse acoustic phonon from diamond visible at low  $Q$  values. Because of its higher sound velocities, longitudinal and transverse acoustic phonons from diamonds are detected at higher energies with respect to cohenite. The longitudinal acoustic phonon from  $\text{Fe}_3\text{C}$  can therefore unambiguously be identified as the excitation between the elastic line and the diamond phonons. A robust zero energy position is determined with several IXS scans stacked at each pressure step, including the scan of

the full elastic line. The phonons energy position  $E(Q)$  is then extracted by fitting a set of Lorentzian functions convoluted with the experimental resolution function of the IXS spectrometer, using a standard  $\chi^2$  minimization scheme. The dispersion curves can be well described by a sine function, which corresponds to the expression of the dynamical matrix limited to the first term in the expansion –nearest neighbor interaction- within the framework of the Born–von Karman lattice dynamics theory (see Ashcroft and Mermin, 1976). The average acoustic sound velocities can thus be simply fitted using the following equation:

$$E(meV) = 4.192 \times 10^{-4} V_p (m/s) Q_{\max} (nm^{-1}) \sin\left(\frac{\pi}{2} \frac{Q(nm^{-1})}{Q_{\max}(nm^{-1})}\right) \quad (1)$$

where  $V_p$  is the compressional sound velocity and  $Q_{\max}$  is the first Brillouin zone edge. Values for  $V_p$  are consequently derived from the sine fits made to the measured experimental dispersion, with  $Q_{\max}$  as a free parameter. Phonon dispersions are reported, along with their best sine fits, in Figure 2 for pressures up to 68 GPa. The observed increase of the phonon frequencies corresponds to an increase of the longitudinal wave velocity ( $V_p$ ) from 6100 to 9375 m.s<sup>-1</sup>.

At each pressure step (measured by ruby fluorescence), an angle dispersive x-ray diffraction pattern was collected in order to obtain directly the molar volume and hence, the density. It must be noticed that  $Q_{\max}$  parameters obtained in the fitting procedure show a good agreement with those inferred from the x-ray diffraction pattern recorded at each pressure increment. Sound velocities and corresponding densities of Fe<sub>3</sub>C obtained as described above, are reported in Table 1 and plotted in Figure 3, along with results obtained for other iron light-element alloys (Badro et al., 2007). As shown in Figure 3, the longitudinal velocity of Fe<sub>3</sub>C scales linearly with its density, thus evidencing again the validity of the Birch's law used in many previous studies (Fiquet et al., 2001; Antonangeli et al., 2004, 2005; Badro et al., 2007). Moreover, the validity of this well-known relation between longitudinal acoustic velocities and densities at high-temperature has

been strengthened by recent IXS sound velocity measurements carried out at high-pressure and temperature on a fcc Fe-Ni alloy (Kantor et al., 2007). This study does not indeed show any significant difference between room and high-temperature longitudinal acoustic velocities when normalized to density.

### 3. Results and discussion

We have further used this dataset to constrain the relative abundance of carbon that could possibly be alloyed in the solid iron core. We follow here the approach developed by Badro et al. (2007), where sound velocities were modeled using a simple composite mineral model, assuming that the average density  $\rho$  and the compressional velocity  $V$  of a two-component ideal solid are respectively given by:

$$\rho = x\rho_1 + (1-x)\rho_2 \quad (2)$$

and

$$V = \frac{V_1 \cdot V_2}{(1-x)V_1 + xV_2} \quad (3)$$

where  $x$  is the volume fraction of component 1. Forcing  $\rho = \rho_{\text{PREM}}$  and knowing  $\rho_1$  (iron density) from the thermal equation of state of Uchida et al. (2001) and the Birch's law presented above, we have a system of two equations and two unknown parameters that can be solved for  $x$  (the volume fraction of the alloyed light element) and  $\rho_2$  (the density of this alloy). Assuming that this simple expression for the sound velocity in an ideal solid holds for low light element contents, the most striking observation is that about 1 wt % carbon in the solid inner core could account for the difference between the measured compressional velocity and density, and the PREM inner core data. The set of equations (2) and (3) also yields a density of Fe<sub>3</sub>C component ( $\rho_2$ ) in a reasonable agreement with that predicted at inner core conditions from experiments or theoretical calculations (see Scott et al., 2001; Li et al., 2002 or Vočadlo et al., 2002). **At core densities, we calculated that shear velocity for such an alloy would be only marginally increased by such a carbon content. Calculated value would still be significantly higher**



than velocities indicated by the model PREM model, in agreement with theoretical results (Laio et al., 2000) which show that the shear modulus for  $\epsilon$ -iron at core pressures should be reduced by 70% when temperature is raised to core temperatures. It has also been speculated that partial melting could occur in the inner core, and dramatically reduce the composite shear velocities with respect to pure crystalline hcp-iron (see Singh et al., 2000).

High-pressure compression experiments confirmed that this phase could be preserved to 73 GPa, even at temperatures in excess of 1500 K (Scott et al., 2001). However, Vočadlo et al. (2002) reported a possible transition at 60 GPa toward a high-pressure non-magnetically ordered state. We observed an anomalous behaviour of the inelastic x-ray scattering dispersion curve recorded at pressures above 68 GPa, which prevented any further measurements at higher pressure. A IXS spectrum collected at 83 GPa (see Figure 2B) indeed displays a strong softening of the longitudinal acoustic branch used to determine  $V_P$  compressional velocity which could correspond to the second-order magnetic transitions proposed from theoretical calculations (Vočadlo et al., 2002). Lin et al. (2004) reported a magnetic collapse in  $\text{Fe}_3\text{C}$  at 25 GPa, based on x-ray emission spectroscopy measurements. Our observations, if consistent with such a transition, clearly place the transition pressure above 68 GPa at room temperature as indicated by theory (Vočadlo et al., 2002). As previously observed in the diffraction study of Scott et al. (2001), our own x-ray diffraction pattern do not indicate any major structural change. These data, however, do not have the resolution to resolve any subtle change such as small variation in the  $c/a$  ratio.

#### 4. Conclusions

To conclude, we show that 1 wt% of carbon in the inner core could reasonably explain the density and **compressional** velocity differences observed between average seismic models for the inner core and experiments. Such a result is obtained if carbon is considered as being the

unique light element alloyed to the iron inner core, in the frame of an ideal solid solution between pure iron and carbon. The magnetic transition at high-pressure proposed by theoretical calculations is here of central importance, since it will probably change the derivative of the elastic properties (in particular of the compressional P-wave velocity) with carbon content. It would be desirable to determine the transition pressure as a function of carbon content in future experiments, in particular in the range of few wt% in the alloy. In addition, there is no reason to consider that only one light element is present in the inner core. Badro et al. (2007) showed that silicon was likely to be the most abundant in the inner core, and that oxygen was probably incorporated in the liquid outer core (see Alfe et al., 2002). From a geochemical point of view, the sulfur content may be limited to 1-2 wt % in the core (Dreibus and Palme, 1996; McDonough and Sun, 1995), which would be strongly partitioned into the liquid outer core. For the inner core alloying light elements, we are thus left with silicon and carbon. Extensive experiments in the Fe-Si-C ternary system at high pressure and temperature would be critical for understanding the Earth inner core.

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**Figure captions:**

**Figure 1:** Representative IXS spectra of a polycrystalline  $\text{Fe}_3\text{C}$  sample at ambient pressure (left panel) and at 68 GPa (right panel) for selected momentum transfers  $Q$ . The experimental data are shown along with the best fit results of equation (1) to the data set (solid line) and the corresponding individual components (thin dotted line). The room pressure measurements were recorded out of the diamond-anvil cell. We show the quasi-elastic line and the longitudinal acoustic excitation for  $\text{Fe}_3\text{C}$  in this panel. At high pressure, the inelastic peaks only are shown for clarity reasons. Arrows indicate the LA phonon of  $\text{Fe}_3\text{C}$  on all panels, whereas LA and TA phonons of diamonds are also visible at low  $Q$  at high pressure (right panel).

**Figure 2:** (A) Typical dispersion curves for polycrystalline  $\text{Fe}_3\text{C}$  to 68 GPa. Data recorded at five to ten momentum transfers have been used in each dispersion curve to constrain  $V_P$  within an estimated error of 3%. The fitted values of  $Q_{\text{MAX}}$  are in very good agreement with those obtained from x-ray diffraction. (B) Experimental points and dispersion curve (solid line) obtained at a pressure of 83 GPa plotted along with lower pressure dispersion curves (dashed lines), showing a clear softening of the longitudinal acoustic branch at high  $Q$ .

**Figure 3:** Compressional P-wave velocities of Pnma  $\text{Fe}_3\text{C}$  as a function of density (solid squares), along with results obtained for other light elements alloyed with pure iron (**Badro et al., 2007**). As shown by the solid line, the experimental points move along a straight line. This linear relation between velocity and density is known as the Birch's law (**see Birch, 1952**). Solid squares:  $\text{FeS}_2$  pyrite, hollow stars:  $\text{FeSi}$ , solid stars:  $\text{FeO}$ , solid triangles: **phase IV FeS**, according to **Fei et al. (1995)**, hollow squares:  $\text{Fe}_3\text{C}$ , solid and hollow circles: pure iron IXS data and shock wave data respectively, solid and hollow diamonds: solid inner core and liquid outer core respectively.

**Table 1.** Pressure, density and compressional (P-wave) velocity data set for Fe<sub>3</sub>C cohenite.

Pressure (GPa)	Density	V <sub>P</sub> (m/s)
Ambient	7.679	6103 ± 413
18	8.203	6856 ± 321
31	8.542	7563 ± 241
50	9.029	8269 ± 228
68	9.414	9512 ± 251

**Table 2.** Birch's equations obtained for Fe<sub>3</sub>C cohenite, pure hcp iron and other alloys (from Badro et al., 2007) used to solve the system of equations (2) and (3).

Fe <sub>3</sub> C	$V_P = 1.90 \rho - 8671$
FeS <sub>2</sub>	$V_P = 3.00 \rho - 6977$
FeS phase IV	$V_P = 1.07 \rho - 1392$
FeO	$V_P = 1.67 \rho - 3285$
FeSi	$V_P = 1.82 \rho - 4169$
HCP iron	$V_P = 0.94 \rho - 1466$

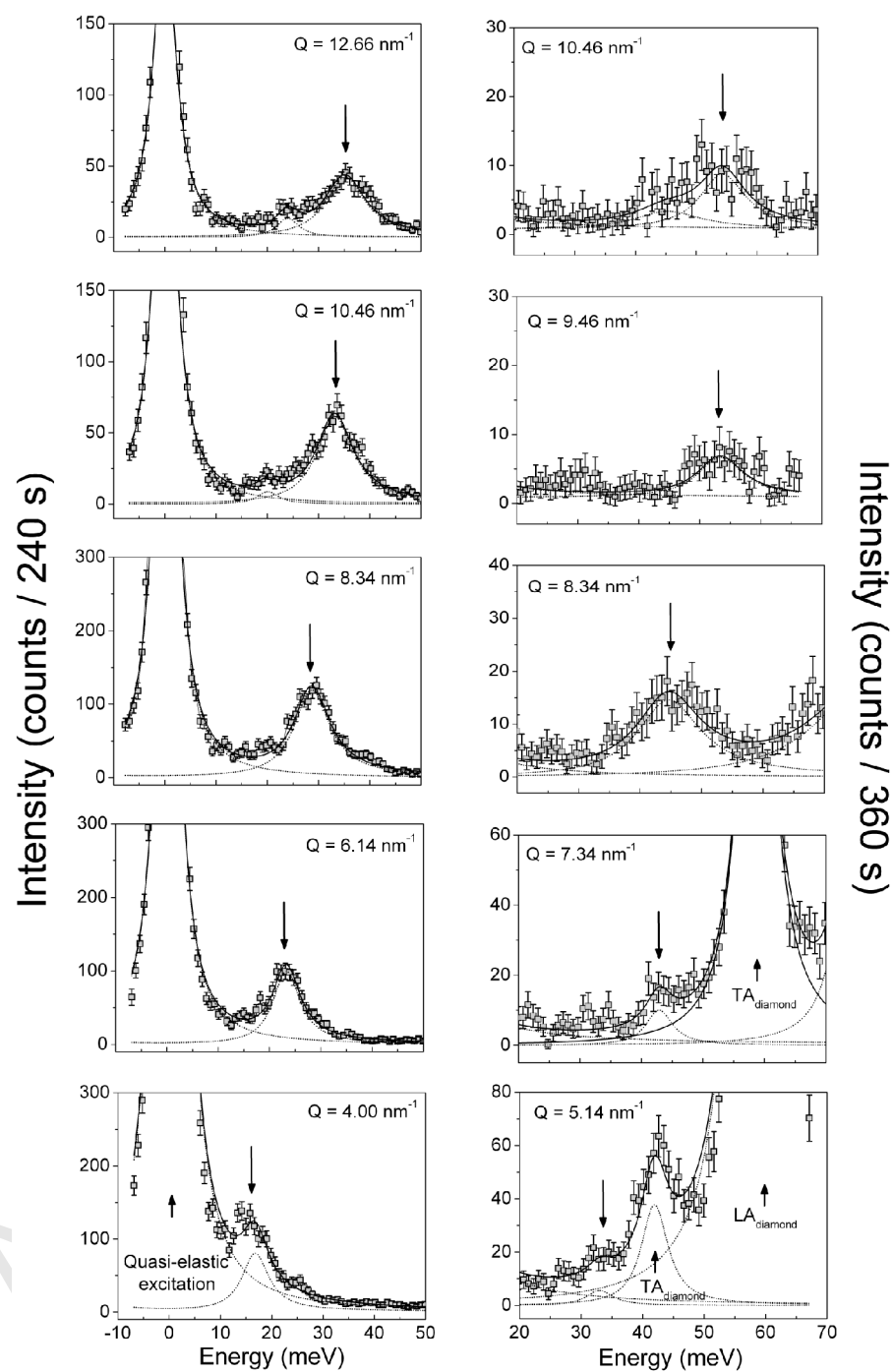


Figure 1.

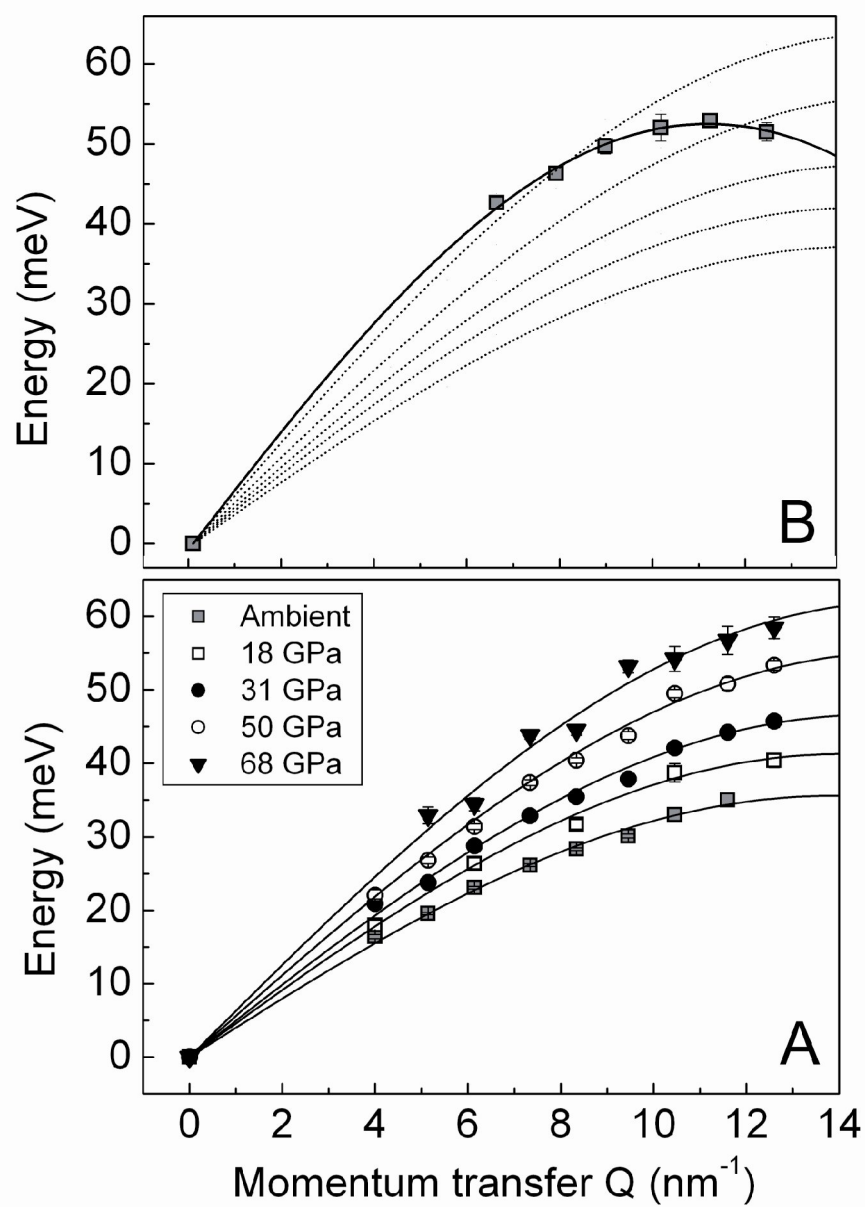


Figure 2.



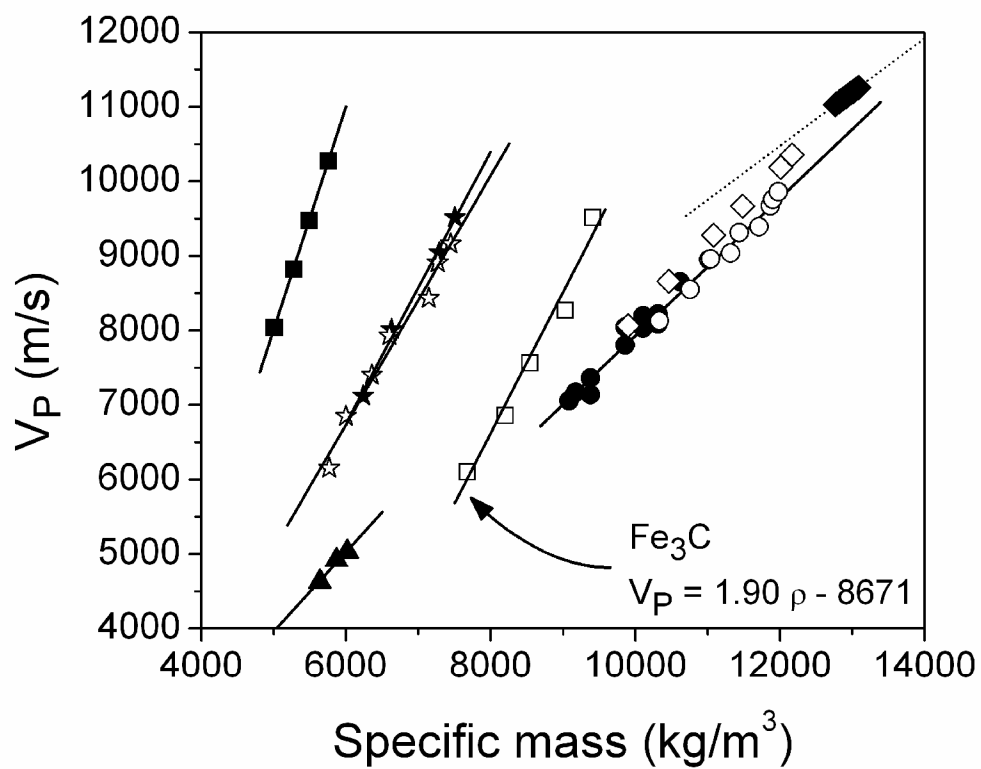


Figure 3.