ESRF	**Experiment title:** Sound velocity of hcp iron at very high pressure by inelastic x-ray scattering	**Experiment number**: ES-222
Beamline: ID28-ID27	**Date of experiment**: from: 29.10.2014 to: 04.11.2014 (ID28)	**Date of report**: 06 March 2015
Shifts: ID-28:18 ID-27:3	**Local contact(s)**: L. Paolasini (ID-28) G. Garbarino (ID-27)	*Received at ESRF:*

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Report:

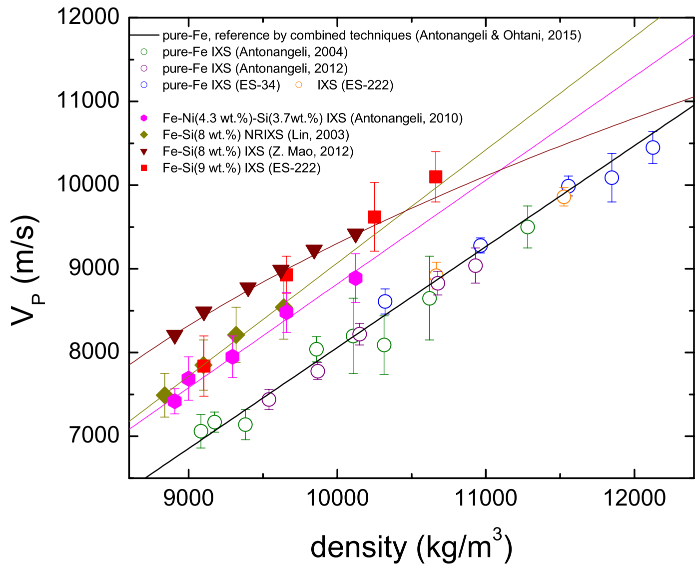
We performed sound velocity and density measurements on polycrystalline iron in the hcp phase at 79, and 127 GPa, complementing data collected in previously allocated beamtime (experimental report ES-34 and Figure 1). Unfortunately diamonds blew while collecting diffraction at ~190 GPa, before collection of IXS data. As backup we collected data on Fe-Si(9wt.%), determining both density and sound velocity in the hcp phase at 54, 80, 113 and 139 GPa (Figure 1).

In both cases IXS measurements have been performed on polycrystalline samples compressed in a diamond anvil cell (DAC) using the Si(9,9,9) instrument configuration. The Si(9,9,9) configuration has been proven to enhance the contrast between sample and diamonds phonons with respect to data obtained using the Si(8,8,8) configurations as typically done for powders in DAC (the widths of diamond phonons are defined by the energy resolution of the spectrometer), still providing enough flux to collect good-quality data in reasonable amount of time (~300 s per point for pure Fe, ~500-600 s per point for Fe-Si(9wt.%)). Spectra have been collected in transmission geometry, with the x-ray beam impinging on the sample through the diamonds, along the main compression axis of the cell, and hence probing exchange momenta q perpendicular to the cell-axis. The x-ray beam was focused at sample position down to 12x7 μm^2 (horizontal x vertical, FWHM) by optics in Kirkpatrick-Baez configuration. Momentum resolution was set to 0.28 and 0.84 nm^{-1} in the horizontal and vertical plane. A vacuum chamber was used to minimize the quasi-elastic scattering contribution from air. At each investigated pressure point, we mapped the aggregate longitudinal acoustic phonon dispersion throughout the entire first Brillouin zone collecting 8 to 9 spectra in the 3-12.5 nm^{-1} range. The energy positions of the phonons were extracted by fitting a set of Lorentzian functions convolved with the experimental resolution function to the IXS spectra, utilizing a standard χ^2 minimization routine. We then derived the aggregate compressional sound velocity V_P from a sinus fit to the phonon dispersion, with error bars between ±1 and ±2% for pure Fe samples and between ±2 and ±4% for Fe-Si(9wt.%).

For both samples we collected 2D diffraction patterns at all pressures on ID27. Tacking advantage of the 3x3 μm^2 beam, we collected data over the entire sample surface, monitoring pressure gradients. Collected diffraction pattern will be analyzed to determine sample texture as well.

Pressures were generated by symmetric type MAO DAC, equipped with FIB custom cut 300/100/40 µm bevelled anvils and composite Re/c-BN gaskets. Diamonds were pre-aligned and oriented to select the fastest transverse acoustic phonon of the diamond in the scattering plane (*i.e.* at the highest energies) and to minimize its intensity. The focused beam of 12x7 μm FWHM at sample position granted collection of clean spectra on specimens of ~30 μm in diameter. Such a small beam also permitted to probe phonons across moderate pressure gradients (as determined from the fine diffraction mesh), while the composite gasket ensured relatively thick samples (8 to 12 μm at the highest pressure), and hence good IXS signal. Pressure was measured off line by collecting Raman spectra at the tip of the diamonds, and crosscheck after collection of sample diffraction by using literature equation of state (for iron) and unpublished equation of state for Fe-Si(9wt.%) (Y. Fei, private communication).

The measured sound velocities for both pure-Fe and Fe-Si(9wt.%) are plotted as a function of the measured density in Figure 1, together with previous IXS determination from ID-28 experiments and literature data.



<u>Figure 1</u>: Compressional sound velocity V_P as a function of density. Open circles are IXS data for pure-Fe at high pressure and ambient temperature: green circles data from [1], purple circles data from [2], blue circles are data from ES-34, orange are data collected during this beamtime. The solid black line is a recently established reference for the Vp-ρ relation obtained combining determinations by various techniques (ultrasonics, IXS, ISLS, picosecond acoustics) [3]. Magenta hexagons are IXS determinations on Fe-Ni(4.3wt.%)-Si(3.7wt.%) [4]. Dark yellow diamonds are NRIXS results on Fe-Si(8wt.%)[5]. Brown upside-down triangles are IXS results on Fe-Si(8wt.%)[6]. Red squares are IXS data collected on Fe-Si(9wt.%) during this beamtime.

The data on pure-Fe well compare with previous IXS determination on ID28 [1-2, ES-34] and are within error bars with a recently established reference for the V_p-ρ relation obtained combining determinations by various techniques (ultrasonics, IXS, ISLS, picosecond acoustics) [3]. Preliminary results obtained on Fe-Si(9wt.%) better agree with NRIXS measurements [5] at low pressure (54 GPa), but seem to be in closer agreement with IXS results [6] at higher pressure (80 to 140 GPa) (Figure. 1), but further data are needed to differentiate between the two proposed trends [5,6].

References

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