ESRF	**Experiment title:** Sound velocity of hcp iron at very high pressure by inelastic x-ray scattering	**Experiment number**: ES-34
Beamline: ID28	**Date of experiment**: from: 03.07.2013 to: 08.07.2013	**Date of report**: 21 February 2013
Shifts: 15	**Local contact(s)**: M. Krisch, A. Bosak	*Received at ESRF:*

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Report:

We performed sound velocity and density measurements on polycrystalline iron in the hcp phase at 65, 97, 133, 153 and 173 GPa, obtaining high-quality data at pressures much higher than those so far obtained by inelastic x-ray scattering on the ID28 beamline (~110 GPa [1-3]) and comparable with the highest pressures reported in literature (~170 GPa [4,5]).

IXS measurements have been performed on polycrystalline iron 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 iron 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). 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.

By scanning the scattering angle at the elastic energy (*i.e.* q-scan at ΔE=0) we also collected diffraction pattern to directly derive the density and evaluate developed preferential alignment of the investigated samples. For the lowest and for the highset investigated pressures we also collected 2D diffraction patterns on ID27 (courtesy of M. Mezouar).

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, while the composite gasket ensured relatively thick samples (8 to 12 µm at the highest pressure), and hence proper IXS signal. Pressure was measured off line by collecting Raman spectra at the tip of the diamonds, and crosscheck after collection of iron diffraction by using literature equation of state.

At each investigated pressure point, we mapped the aggregate longitudinal acoustic phonon dispersion throughout the entire first Brillouin zone collecting 6 to 8 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%.

Unfortunately, detector problems prevented collection of data on five of the nine analyzers, so that we were obliged to use two different q-settings to cover the entire first Brillouin zone, basically doubling the collection time. Furthermore, the last 3 shifts out of the 18 allocated have been used by beamline staff to replace the detector to be back to full operation for before the following experiment.

The measured sound velocities are plotted as a function of the measured density in Figure 1, together with IXS determination from previous experiments carried out on ID28.

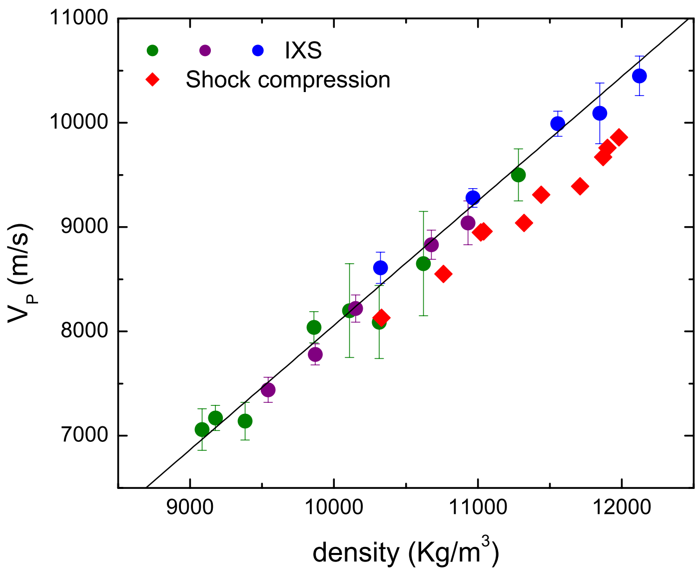

Figure 1: Compressional sound velocity V_P as a function of density. Circles are IXS data at ambient temperature: green circles data from [2], purple circles data from [3], and blue circles present data. The solid line is a linear regression to the entire data set, as guide for the eye. Red diamonds are sound velocities determined by shock experiments not reduced to 300 K.

The collected data compare favourable with previous IXS determination on ID28, and appreciably enlarge the covered density range. The pressure vs. density evolution is still well described by a linear relation. Interestingly, sound velocity measured by IXS at ambient temperature plots higher then sound velocities determined on solid Fe by shock experiments not reduced to 300 K, suggesting high-temperature anharmonic effects at constant density.

References

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