ESRF	**Experiment title:** Sound velocity measurements of hcp-Fe at high pressure and temperature by inelastic x-ray scattering: assessing the limit of the Birch's law	**Experiment number**: HS-4383
Beamline: ID28	**Date of experiment**: from: 06.04.2011 to: 12.04.2011	**Date of report**: 26 August 2011
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Report:

We performed sound velocity and density measurements on polycrystalline iron in the hcp phase at simultaneous high pressure and high temperature in the 30-93 GPa range and 300-1100 K.

Inelastic x-ray scattering measurements have been performed on the ID28 beamline, using the Si(8,8,8) instrument configuration, which provides the best compromise between flux and energy resolution (5.5 meV full width half maximum, FWHM) for polycrystalline samples compressed in DAC. 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 30x20 μm^2 (horizontal x vertical, FWHM) by optics in Kirkpatrick-Baez configuration. Momentum resolution was set to 0.25 nm^{-1}.

By scanning the scattering angle at the elastic energy (*i.e.* q-scan at $\Delta E=0$) we also collected diffraction pattern to directly derive the density of the investigated samples.

Pressure and temperature were generated by means of externally heated diamond anvil cells (DAC), with resistive micro-machined furnaces placed around the diamonds and thermally insulated from the body of the cell by means of ceramic seats. Pressure was measured at ambient temperature by ruby fluorescence, and the temperature was measured by means of an S-type thermocouple placed on the gasket. We also successfully tested an internally heated DAC, where the properly shaped iron sample is thermally insulated from the diamond and the gasket and heated by directly applying a DC voltage. In this last case, the temperature was determined according to the power-T calibration obtained by spectroradiometric measurements carried out on ID27. Both type of cells were water cooled and placed in a specifically designed vacuum chamber to reduce oxidation and to minimize the quasi-elastic scattering contribution from air.

At each investigated pressure/temperature point, we mapped the aggregate longitudinal acoustic phonon dispersion throughout the entire first Brillouin zone collecting 8-9 spectra in the 3.5-12.5 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%.

The sound velocities measured at 300 K are in overall agreement with results from previous studies (Figure 1).

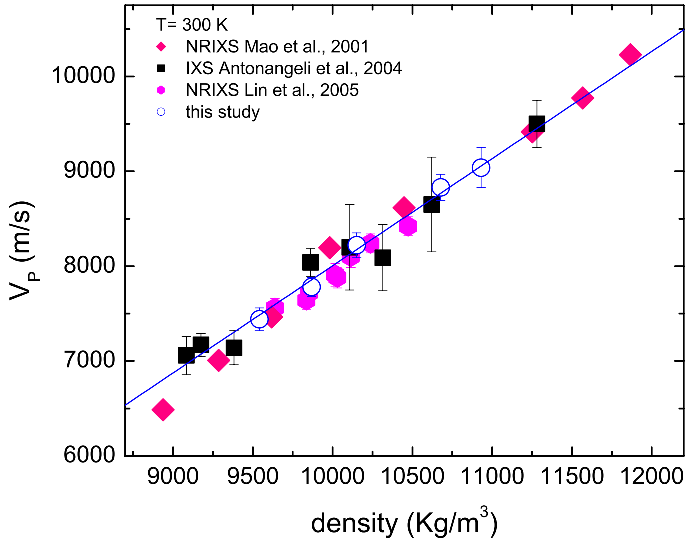

Figure 1: Compressional sound velocity V_P as a function of density at ambient temperature. The solid line is a linear regression to the experimental data, as guide for the eye. We can observe an overall agreement among the various studies.

On the contrary, the sound velocities measured at high temperature do not show the softening expected according previous nuclear resonant inelastic x-ray scattering investigations (Figure 2).

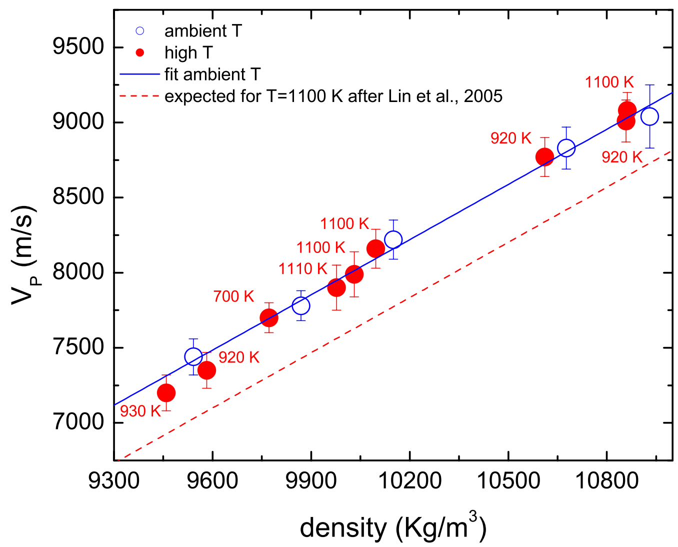

Figure 2: Compressional sound velocity V_P as a function of density at ambient and high temperature.

Our results indicate that high-temperature anharmonic correction are negligible at least up to 1100 K and that V_P scales linearly with density irrespectively of specific pressure and temperature conditions over the investigated range.