![ESRF logo] **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-4109
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Report:

We performed sound velocity and density measurements on polycrystalline iron and iron alloys in the hcp phase at high pressure and high temperatures.

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 transverse dimensions of the focused x-ray beam of 30x90 μm^2 (horizontal x vertical, FWHM) were further reduced by slits on the vertical direction. 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.

During this first allocated beamtime, we tested two different resistively-heated diamond anvil cell (DAC) setups.

i) 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. The temperature was determined according to the power-T calibration obtained for each individual by spectroradiometric measurements carried out on ID27.

ii) Externally heated DAC, with micro-machined furnaces placed around the diamonds and thermally insulated from the body of the cell by means of ceramic seats. In this case the temperature was directly measured by means of an S-type thermocouple placed on the gasket.

In both cases measurements have been conducted in a specifically designed vacuum chamber to minimize scattering from air and to reduce oxidation of the furnaces.

The first setup granted higher temperatures (up to 1500 K) at the price of reduced sample dimension. This resulted into a critical problem for the collection of IXS data. Indeed, samples about 15 μm thick allowed collection of good-quality IXS spectra in few hours, but not stable and uniform temperature generation, while reducing the sample dimensions to 10 μm permitted to maintain 1200 K over 24 hours, but the obtain IXS spectra were of poor quality (irrespectively of the collection time). Alloying few wt.% of

silicon to the iron samples improved the heating characteristics in terms of necessary power and dimension of hot spot, and preliminary tests on iron-silicon alloy with Si ~ 4wt.% provided encouraging results.

With the second setup we have been able to use larger samples (70-90 μm diameter and ~30 μm thick), resulting in stronger IXS signals. We collected data at increasing compression (pressures measured as reference only at ambient temperature by ruby luminescence) along two isotherms, one at 300 K and the second at 700 K. Technical problems with vacuum pump and the relatively low vacuum (of the order of 10^{-4}) caused the quick oxidation of the furnaces when operating at 1000 K, preventing collection of IXS data, so that only diffraction have been obtained at this temperature.

At each investigated pressure/temperature point, we mapped the longitudinal acoustic phonon dispersion throughout the entire first Brillouin zone collecting 4-5 spectra in the 3.5-12.5 nm^{-1} range (unfortunately, only 5 of the 9 analyzer were operational). 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 ±2 and ±3%.

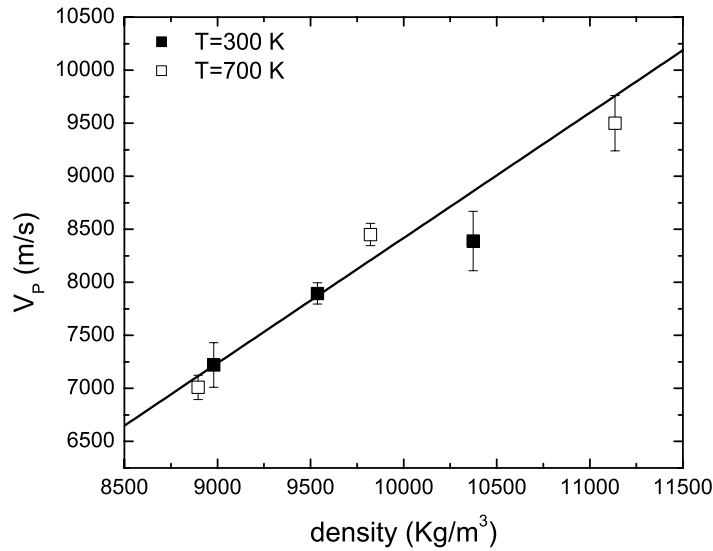The measured compressional sound velocity is plotted for two temperatures as a function of density in Figure 1.

Figure 1: Compressional sound velocity V_P as a function of density for T=300 K (solid symbols) and T=700 K (open symbols). The solid line is a linear regression to the entire experimental dataset, as guide for the eye.

Over the investigate range, and within the experimental error bars, we do not observe any significant difference between the density evolution at 300 K and 700 K.