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Report:

Both hcp iron [1] and hcp cobalt (see experimental report HS-1905) develop preferential alignment, when non-hydrostatically compressed into a diamond anvil cell (DAC). Such texture is characterized by a cylindrical symmetry and the alignment of the c-axis along the main compression axis. Consequently the single-crystalline anisotropy of longitudinal acoustic wave propagation in the meriadian plane, from c- to a-axis, can be estimated probing the aggregate compressional sound velocity V_P as a function of the direction of propagation with respect to the compression axis of the cell. This can be achieved in a geometry where the DAC, equipped with a beryllium gasket, is rotated around the incident x-ray beam axis.

An Inelastic X-Ray Scattering (IXS) test experiment has been performed on polycrystalline hcp-Fe non-hydrostatically compressed at 80 GPa. The employed scattering geometry, allowed us to get the sound dispersion along the compression axis of the cell, ideally complementing the results previously obtained [2]. The exposed sample section was 20x75 μm^2 (horizontal x vertical): this corresponds to the best sample-beam size match, recalling that the beam size was 25x60 μm^2 (FWHM, horizontal x vertical), reduced by slits to 25x50 μm^2 in order to enhance the contrast between the iron and the beryllium signal.

The phonon dispersion was determined for six Q-values in the range between 5 and 10.5 nm^{-1}. Typical examples of the recorded spectra are shown in *Figure 1*. The quality of the spectra are much worse than the ones obtained in the standard geometry through the diamonds [2], due to the more intense and broader elastic line and the large Be phonon, features attributable to the polycrystalline structure of the Be gasket. As a consequence the iron phonon appears, even on a logarithmic scale, just as a low energy shoulder of the intense Be phonon peak. The energy determination from the fit to the experimental data therefore suffers from an important correlation with the other parameters of the fit, especially the phonon width and the background, leading to a quite large energy indetermination. As a consequence, the sound velocity can be derived only with an accuracy of about 8%, while typical errors are around 2% in the standard geometry.

The transmission through the Be gasket makes the sample phonon detection critical and at the limit of the actual beamline capabilities. Possible solutions to enhance the contrast between the iron and the beryllium signal are to drill a hole in the gasket, perpendicular to the diamond axis, through which the x-ray beam could impinge on the sample, or to use a slit system, placed very close to the Be gasket.

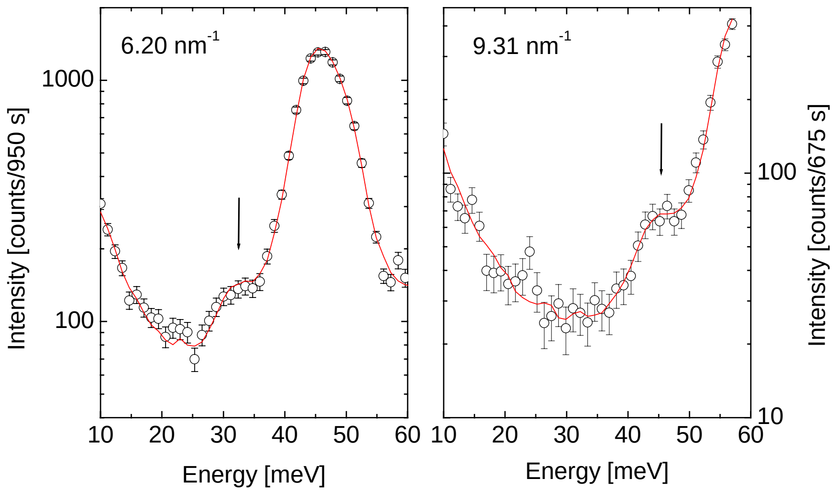

Figure 1: IXS spectra of hcp Fe at room temperature and 80 GPa. The experimental data and their statistical error bars are shown together with their best fits. The momentum transfer values, at which the spectra were recorded, are indicated in the figures. The spectra are shown on a logarithmic scale in order to visualize the Fe phonon (arrows).

In the case of cobalt the standard geometry through the diamonds has been therefore employed (mainly probing the basal plane, according to the experimentally determined texture). The longitudinal acoustic phonon dispersion of polycrystalline cobalt was determined by IXS up to 99 GPa, over the whole hcp phase stability range. The derived density evolution of the aggregate compressional and shear sound velocities was compared with ambient pressure ultrasonic (US) results [3], impulsive stimulated light scattering (ISLS) measurements [4] and first principle calculations [5]. This comparison is shown in *Figure 2* for the case of V_P.

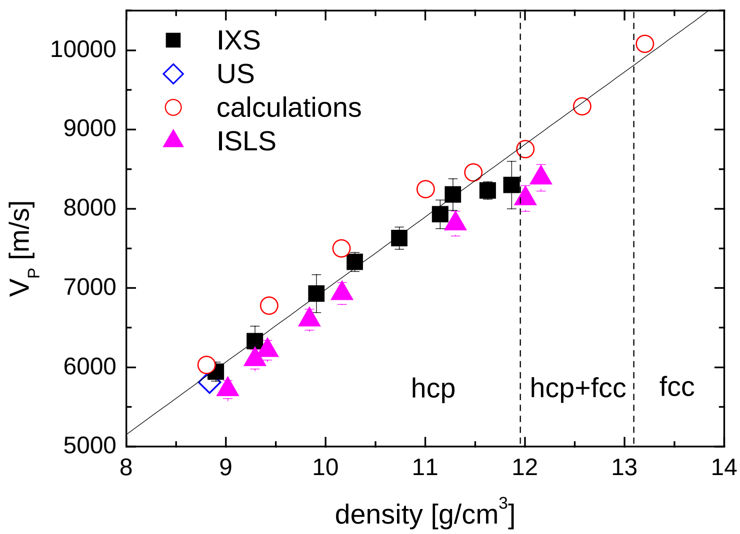

Figure 2: Aggregate compressional sound velocity V_P of polycrystalline hcp cobalt as a function of density. The linear fit takes into account IXS data up to 75 GPa (11.28 g/cm^3) The limits of the phase stability are indicated by dashed lines.

The sound velocity scales linearly with density, as expected within the quasi-harmonic approximation, up to 75 GPa. The results from IXS measurements compare well with the ambient pressure ultrasonic measurements and lie in-between the calculations and the ISLS values. Above 75 GPa, approaching the martensitic hcp-to-fcc transition, a deviation from the linear behavior for $V_P(\rho)$ can be observed. This softening is also predicted in the same density region by the calculations, although theoretical values are systematically higher than the IXS ones. ISLS results show qualitatively the same trend as well, but the derived sound velocities are systematically lower than the IXS values, and, when back-extrapolated to ambient pressure, significantly lower than the ultrasonic results.

These observations highlight a common scenario, characterized by a regular evolution of the sound velocities up to a key pressure point (P_{max}) above which both the compressional and the shear aggregate sound velocities exhibit a softening. The departure from the linear behavior was suggested in [4] to start at

lower pressure ($P_{max} \approx 60$ GPa $\leftrightarrow$ 10.88 g/cm^3) in contrast to the present IXS measurements, which clearly places P_{max} above 75 GPa. Causes for the high pressure softening are still unclear and different possibilities are under consideration.

Aggregate elastic properties can be as well derived from the single crystal elastic tensor, when an appropriate averaging scheme is employed. The bulk modulus, the shear modulus and the aggregate compressional and shear sound velocities were computed according to the Voigt, Reuss and Voigt-Reuss-Hill average, starting from the single-crystalline elastic moduli previously determined by IXS [6]. The three calculations give results that differ by less than 1% even at 99 GPa: thus the observed single crystal anisotropy [6] is very weakly reflected by the effective elastic anisotropy of the aggregate, when a completely random distribution of crystallites is considered [7].

The density evolution of the aggregate compressional sound velocity is illustrated in *Figure 3*, where the experimental results obtained on powders are reported together with the ambient pressure ultrasonic determination [3] and the Voigt-Reuss-Hill average of the single crystal elastic moduli [6].

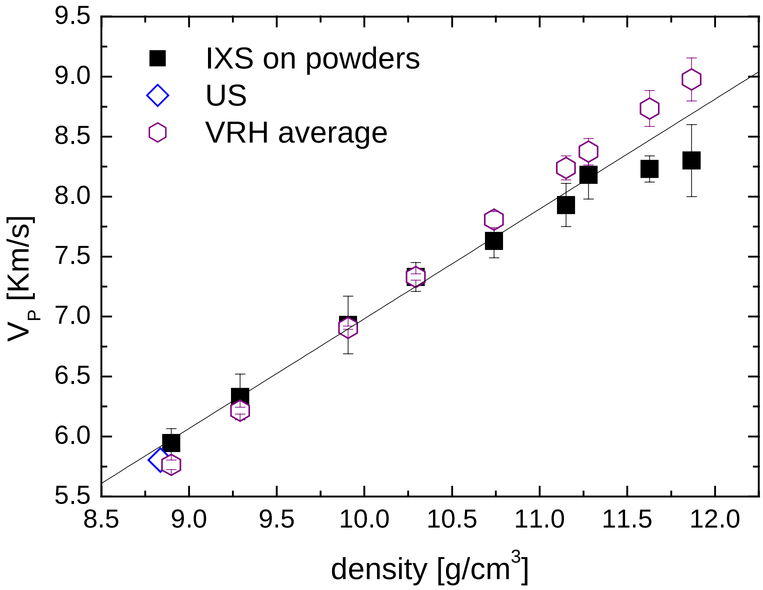

Figure 3: Aggregate compressional sound velocity V_P of hcp cobalt as a function of density.

Despite a different slope, the two IXS data sets differ by less than 3% up to 75 GPa, thus testifying that an over-simplified randomly oriented distribution describes quite well the elastic properties of the polycrystal under compression. The effect of the developed texture could potentially be important and explain the remaining discrepancies. A more quantitative analysis, keeping into account the evolution of the texture with pressure, is still in progress.

References:

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