ESRF	**Experiment title:** **Phonon spectroscopy with nuclear scattering at high pressure: Lattice dynamics of** a**- and** e**-iron**	**Experiment number**: HS-1175
Beamline: ID22 N	**Date of experiment**: from: 08/07/2000 to: 16/07/2000	**Date of report**: 10/08/2000
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In the preceding experiment HS-868, we performed the first successful nuclear inelastic scattering (NIS) study of the phonon density of states (DOS) in the ε-phase of iron at pressures up to 42 GPa [1]. In the present experiment three major components of the experimental set-up (see [1] and report HS-868) were improved:

- most importantly, the spectral resolution of the high-resolution monochromator (HRM) was reduced from about 6 meV to 3.5 meV for the 14.413 keV transition of Fe-57;
- a second compound refractive lens (CRL; diameter of holes: 1.0 mm, N = 28) was used for vertical focusing of the monochromatized beam, providing together with a CRL behind the undulator and a bent crystal behind the HRM, a beam size of 90 μm x 60 μm (h x v) with 3×10^8 photons/s @ 90 mA storage ring current;
- the diamond-anvil cell (DAC) was modified by two additional openings allowing now also NIS studies of the pressurized sample with the x-ray beam at an angle of 70° – 90° (instead of the usual 0°), relative to the DAC axis. The NIS signal (Fe $K_{\alpha,\beta}$ x-rays) is recorded in both cases perpendicular to the x-ray beam and the DAC axis. Fig. 1 shows the two arrangements, which allow to study the impact of texture, a common feature in DACs with non-hydrostatic pressure conditions, by comparing the spectra measured parallel and (almost) perpendicular to texture axis (which is parallel to the load axis of the diamonds).

According to recent publications, ε-Fe exhibits significant texture effects in a DAC [2,3]. Due to small uniaxial stress components, the c-axis of hcp structure exhibits a preferred orientation parallel to the load axis of the DAC. In a NIS experiment the phonons are probed in the direction of the exciting SR beam. Hence, the effect of texture could be studied by comparison of the NIS spectra measured at different orientations of SR and DAC axis.

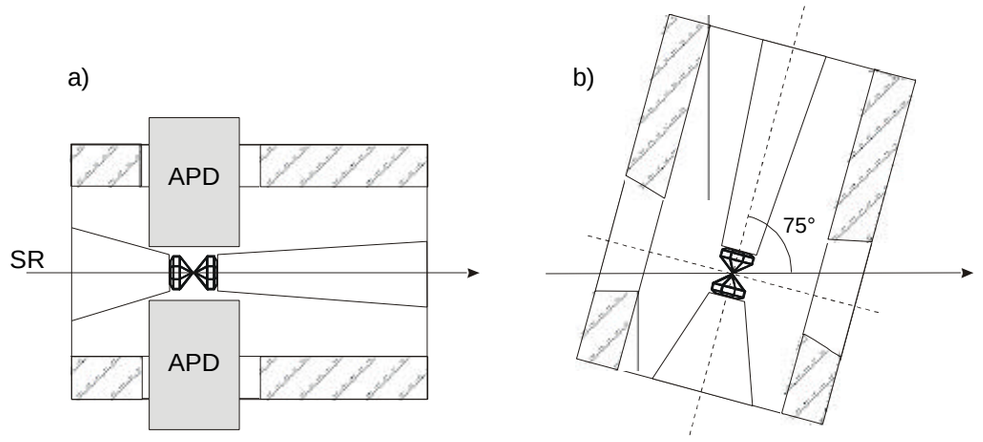

Fig. 1: Sketch of the diamond-anvil cell (DAC) and detectors (APD) indicating two measuring geometries for NIS with different orientations between incident SR beam and DAC axis (dashed line): (a) parallel to cell axis and (b) 75° to cell axis.

We recorded NIS spectra of α-Fe (bcc) at ambient pressure and 7 GPa as well as of ε-Fe (hcp) at 28 GPa and 40 GPa. The measuring time for each pressure and orientation was about 1 day. The phonon DOS, g(E), extracted from the raw data according to Ref. [4], exhibit pronounced differences for α- and ε-phase as well as differences for the different orientations, less pronounced in α-Fe and more pronounced in ε-Fe, indicating the existence of preferred orientations under pressure (see Fig. 2). A preliminary evaluation of the thermodynamic and elastic parameters from g(E) at 40 GPa indicated, within the error bars, slightly higher values for the Debye temperatures (by 5%) and sound velocities (by 4%) parallel than perpendicular to the c-axis. This indicates a rather low anisotropy of the elastic parameters in ε-Fe, similar to the findings of a recent Raman study [5], where the E_{2g} Raman mode was observed; the energy compares well, at 40 GPa, with the low-energy maximum in the phonon-DOS at 27 meV, enhanced in the spectrum recorded more parallel to the diamond axis, where the optical modes are best observable.

– An XRD analysis of the texture and further data analysis are in progress.

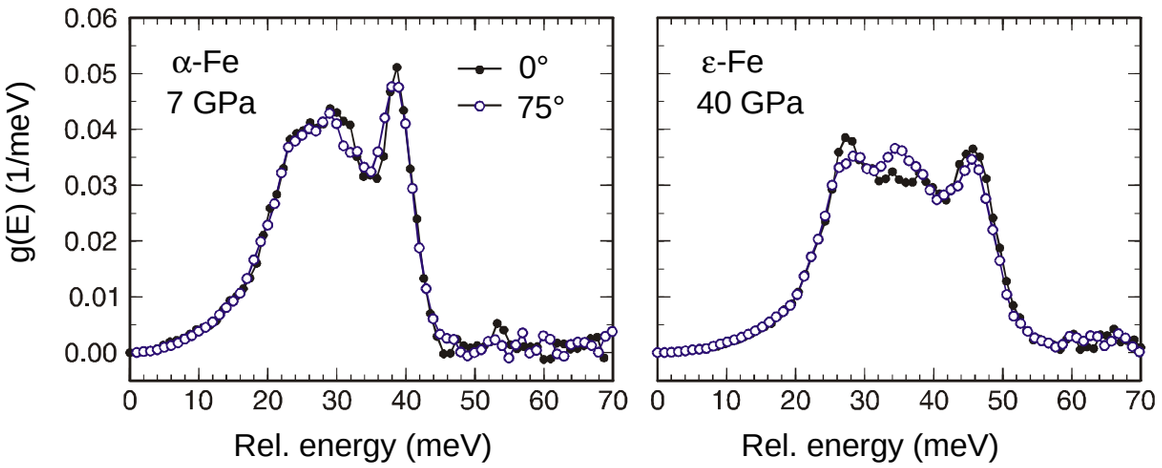

Fig. 2: Phonon density of states g(E) or iron. Full dots (open dots) represent data measured with an angle of 0° (75°) between incident SR and DAC axis; see also fig. 1.

References:

[1] R. Lübbers, H.F. Grünsteudel, A.I. Chumakov, G. Wortmann, Science **287** (2000) 1250.
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[4] A.I. Chumakov et al., PRB **54** (1996) 9596. [5] S. Merkel et al., Science **288** (2000) 1626.