ESRF	**Experiment title:** **Phonon spectroscopy at oriented hcp iron at pressures up to 150 GPa**	**Experiment number**: HS1765
Beamline: ID18	**Date of experiment**: from: 10.07.02 to: 16.07.02	**Date of report**: 29.08.2002
Shifts: 18	**Local contact(s)**: Dr. A.I. CHUMAKOV	*Received at ESRF:*

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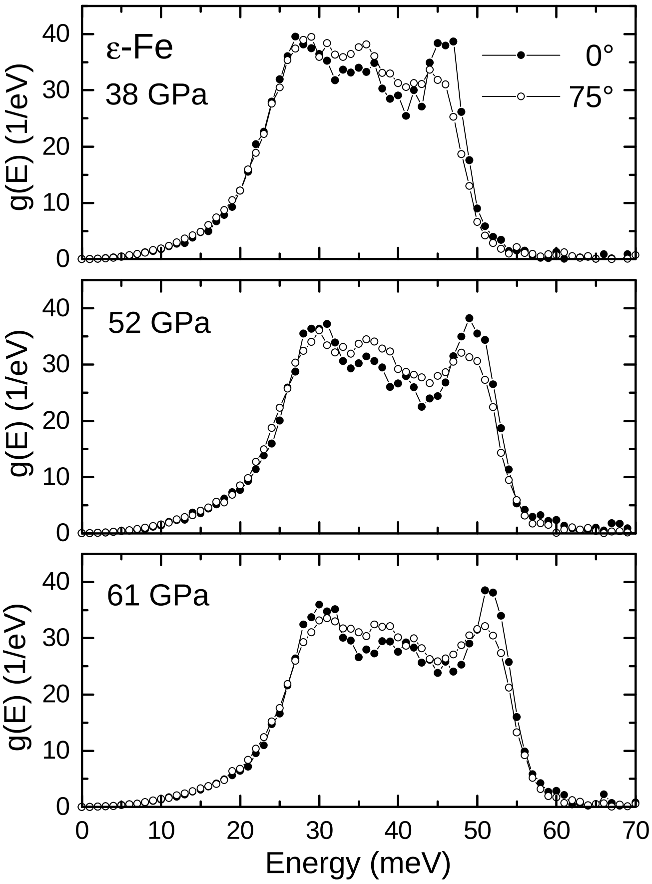
Report:

The goal of this beamtime (18 shifts) was a continuation of our high-pressure phonon spectroscopy at the hexagonal high-pressure phase of iron (ε-Fe), where we reached 40 GPa and observed, for the first time, texture induced changes in the phonon density-of-states (DOS) [Ref.1, 2]. These experiments were performed at pressures of 38 GPa, 52 GPa and 61 GPa using a diamond-anvil cell (DAC): Unfortunately, a diamond anvil broke when we were going to higher pressures. Since there was no possibility for a replacement of the broken diamond during this beamtime, no further high-pressure studies of ε-Fe were possible. So we used the remaining 6 shifts of beamtime for the commissioning of a new oven designed for NIS experiments at high temperature and measured $Fe_{65}Ni_{35}$ in the temperature range 300 K to 660 K.

The NIS experiments on ε-Fe were performed at the beamline ID18 with the use of a LN_2-cooled Si(1,1,1) double-crystal premonochromator and a high-resolution monochromator (HRM) consisting of two pairs of ("nested") Si(4,2,2,) and Si(9,7,5) crystals. Due to the focusing optics (compound refractive lenses at the beam entrance and, in particular, two Kirckpatrick-Baez mirrors behind the HRM, the monochromatized beam was focused on a spot of 15 × 10 μm (h × v). We used our special high-pressure cells with Be gaskets [1], which allowed a transmission of the pressurized sample through the diamond anvils, but also at an angle near 90° with respect to the axis of the diamonds (see Fig. 2). For the detection of the inelastic Fe $K_{\alpha,\beta}$ x-ray fluorescence radiation two avalanche photo diodes were used.

Fig. 1 shows typical spectra of ε-Fe at 38, 52 and 61 GPa; the 38 GPa spectrum closely resemble that of 40 GPa measured in the previous beamtime on ε-Fe. Texture effects are clearly evident and more pronounced than in our last study. This was expected from the use of mineral oil instead of methanol-ethanol-water mixture as pressure transmitting medium. A preliminary evaluation of the data yield slightly larger Debye temperatures and also sound velocities in the spectra taken with the beam parallel (labelled with 0°) to the load axis of the diamonds (which is also the preferred orientation of the hexagonal c-axis) than in the spectra taken at an angle of 75°. These texture effects in the phonon DOS spectra have been observed already in our previous study [1,2] and also derived in theoretical *ab-initio* calculations of ε-Fe [2]. They support suggestion that the sound velocities in ε-Fe are anisotropic with respect to the crystallographic c-axis. This information has direct impact for a geophysical interpretation of the anisotropy in the sound velocities observed for the Earth´s core. The final evaluation of the present data is underway. We will apply for a new beamtime to continue these studies of ε-Fe with a slightly modified DAC to pressures well above 100 GPa (1 Mbar).



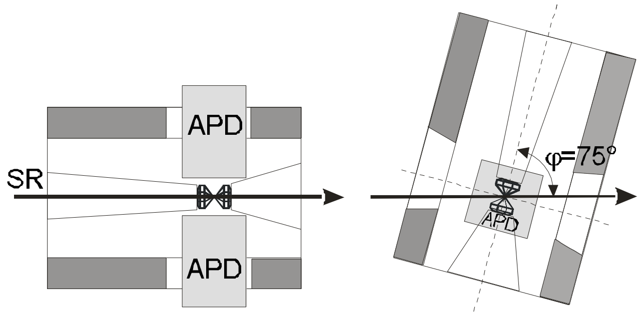


Fig. 2: The setup of our HP cell to study the texture effects in oriented samples.

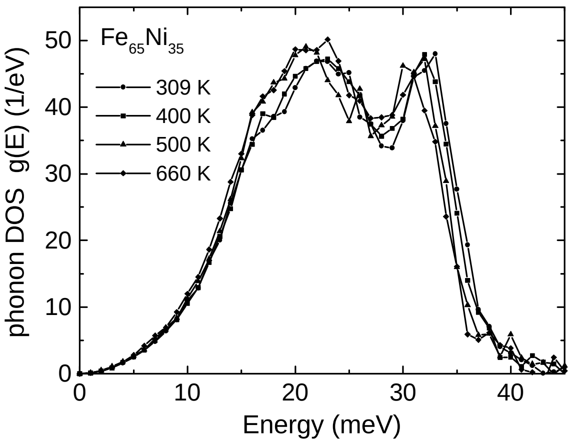


Fig. 1: Phonon DOS derived from NIS spectra at various pressures, measuerd with different directions (0° and 75°) with respect to the load axis of the DAC (see Fig. 2).

Fig. 3: Typical NIS spectra of spectra of $Fe_{65}Ni_{35}$.at various temperatures.

For the commissioning of the new oven we used a $Fe_{65}Ni_{35}$ Invar sample already studied in the temperature range 5 K - 300 K and at room temperature, at pressures up to 17 GPa [3, 4]. Due to the special Invar properties, the phonon DOS of $Fe_{65}Ni_{35}$ exhibited almost no changes between 5 K and 300 K [3, 4]; this behaviour, however, changed markedly in the present study, already at 400 K, well below the Curie temperature at 520 K, NIS spectra exhibit characteristic shifts of the spectral features to lower energies (Fig. 3). A preliminary evaluation of the phonon DOS data yielded a decrease of the high-temperature Debye temperature and other elastic parameters (e.g. force constant, f-factor) with a discontinuity around 450 K, indicating a continuous loss of the Invar properties in the temperature range from 400 K to 520 K. This behaviour is also sensitively monitored by the low-temperature Debye temperature (derived from the sound velocity). The final evaluation of the data of $Fe_{65}Ni_{35}$is underway.

The NIS oven was constructed by Matthias Brandstätter; we appreciate the continuous help of Ernst Schreier during the measurements.

References:

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