ESRF	**Experiment title:** **High-pressure phonon spectroscopy of Fe-Ni alloys**	**Experiment number**: HS-1488
Beamline: ID22N	**Date of experiment**: from: 09.07.2001 to: 17.07.2001	**Date of report**: 31.08.2001
Shifts: 18	**Local contact(s)**: Dr. O. Leupold, Dr. B. Doyle	*Received at ESRF:*

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Report:

This beam-time was initially devoted to our proposal **HS-1488: "Phonon spectroscopy at oriented hcp iron at pressures up to 150 GPa"**. For this purpose we needed at least the same experimental conditions as in our last successful study of α-Fe and ε-Fe at pressures up to 40 GPa (see ESRF Highlights 2000, p. 48). It turned out, however, that the monochromatized beam could not been focused on a spot of 90 x 60 μm^2 as in experiment HS-1175, but only on about 130 x 130 μm^2, resulting in almost 4-times less inelastic counting rate with the same ε-Fe sample in the diamond-anvil cell and making, in addition, a tilting of the cell/sample with respect to the beam impossible. After using 3 shifts for taking one inelastic spectrum of ε-Fe at 60 GPa, and discussion with the beamline contacts, we changed the experiment to our approved proposal **HS-1614: "High-pressure phonon spectroscopy of Fe-Ni alloys"**, which was scheduled for Nov./Dec. 2001. At this time there is good chance that we can successfully continue with proposal HS-1488 when the presently installed Kirckpatrick-Baez mirrors deliver a better spot size (envisaged ca. 30 x 30 μm^2), which allows for pressures above 100 GPa (1 Mbar).

The current interest in Fe-Ni Invar alloys has been revitalised by actual theoretical calculations and experimental work [1-3]. Here we investigate $Fe_{65}Fe_{35}$, which can be considered as the classical Invar phase, by the two techniques connected with nuclear scattering of synchrotron radiation by the Fe-57 nuclei: the local phonon-DOS were obtained from nuclear inelastic scattering (NIS) and the magnetic hyperfine fields were measured with (elastic) nuclear forward scattering (NFS). The sample was 95% enriched in Fe-57 and previously studied with normal Mössbauer effect and NFS [4]. The sample size in the h.p. cell was 300 x 200 x 15 μm^3, collecting the whole monochromatized radiation with a bandwidth of 3.5 meV (see report on HS-1175). We used the same high-pressure cell with a Be gasket as in the previous studies of α-Fe and ε-Fe [5, 6]. For the low temperature experiments a He flow cryostat was used, the $Fe_{65}Ni_{35}$ foil was clamped between two Be windows for thermal contact and tilted with respect to the beam.

Two series of experiments were performed with the $Fe_{65}Ni_{35}$ sample as function of pressure and temperature. Fig. 1 shows on the left panel some NIS spectra measured at various pressures and at various temperatures. The phonon DOS were extracted from NIS spectra as described in [7] and are shown in the right panel of Fig. 1. The relatively strong changes induced to the phonon DOS by rather modest pressures from 0 to

10.4 GPa reflect the special Invar properties. Up to 5 GPa, where we know from the simultaneously recorded NFS spectra as well as from previous studies that the (ferro)-magnetic ordering temperature is reduced from 520 K to well below 300 K [3, 8], the low-energy slope in the DOS is changed more pronounced than the high-energy structures. Only between 7.1 GPa and 10.4 GPa there is a "proportional" change of the spectral features at low and high energy, as observed, for instance, in a-Fe [5, 8].

The DOS spectra observed as function of temperature between 5 and 300 K are, in contrast to the above observations, almost unchanged. This again reflects the special properties of Invar with an anomalous small thermal expansion coefficient for $Fe_{65}Ni_{35}$ around 300 K: a_V @ $6 \cdot 10^{-6}$ K^{-1} in comparison to $a_V = 3.5 \cdot 10^{-5}$ K^{-1} for a-Fe, where a previous NIS study at the ESRF [7] delivered a well observable decrease of the phonon frequencies of 3% in the temperature range 5 K - 300 K.

The evaluation of the thermodynamic and elastic properties is still in progress.

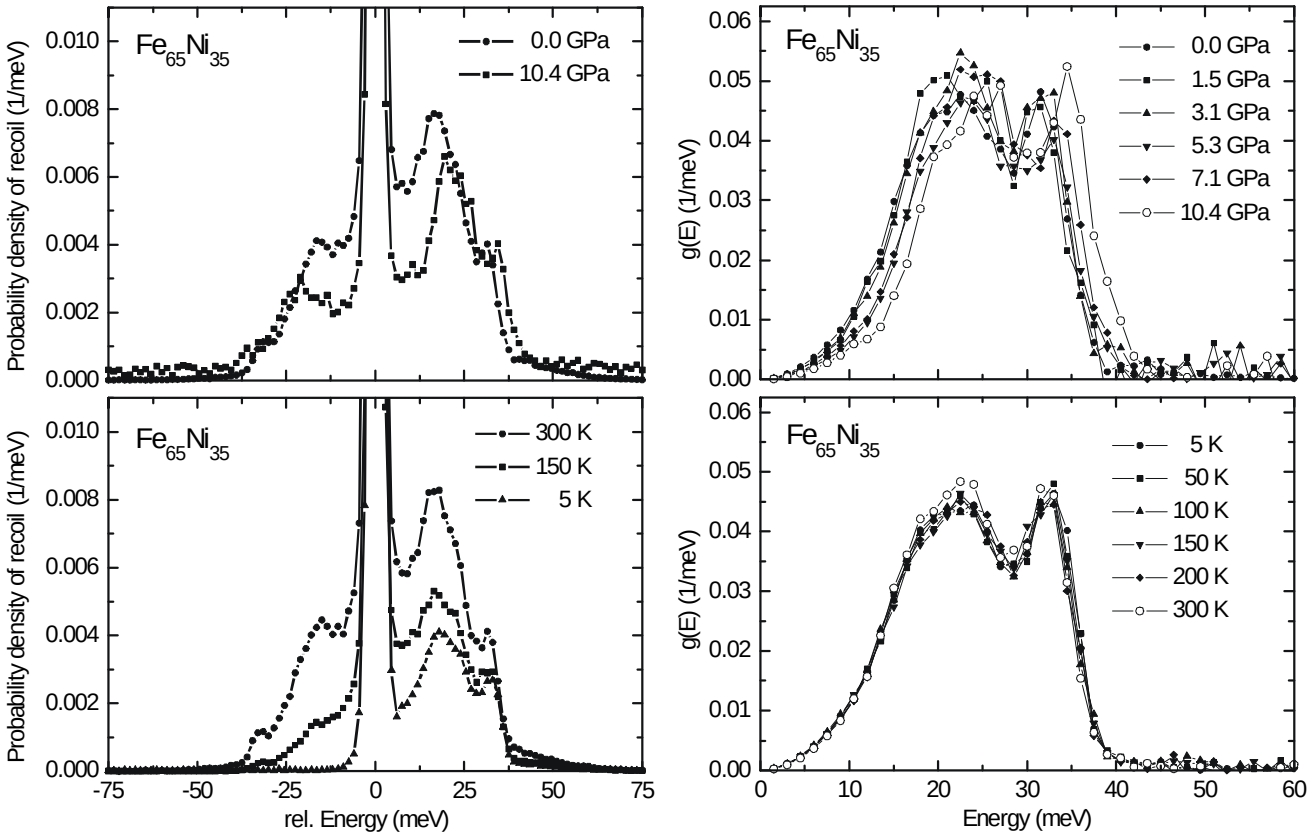

Fig. 1: (left): Typical NIS spectra of $Fe_{65}Ni_{35}$ at various pressures and temperatures. Collection time for a pressure spectrum in a diamond anvil cell was about 20 h, for a temperature spectrum in the cryostat about 4 h. (right): Phonon DOS g(E) for $Fe_{65}Ni_{35}$ for various pressures and temperatures.

References:

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