ESRF	**Experiment title:** High-pressure phonon spectroscopy of Fe-Ni alloys	**Experiment number**: HS-1614
Beamline: ID22N	**Date of experiment**: from: 05.12.2001 to: 11.12.2001	**Date of report**: 28.02.2002
Shifts: 18	**Local contact(s)**: Dr. Olaf Leupold, Dr. Bryan Doyle	*Received at ESRF:*

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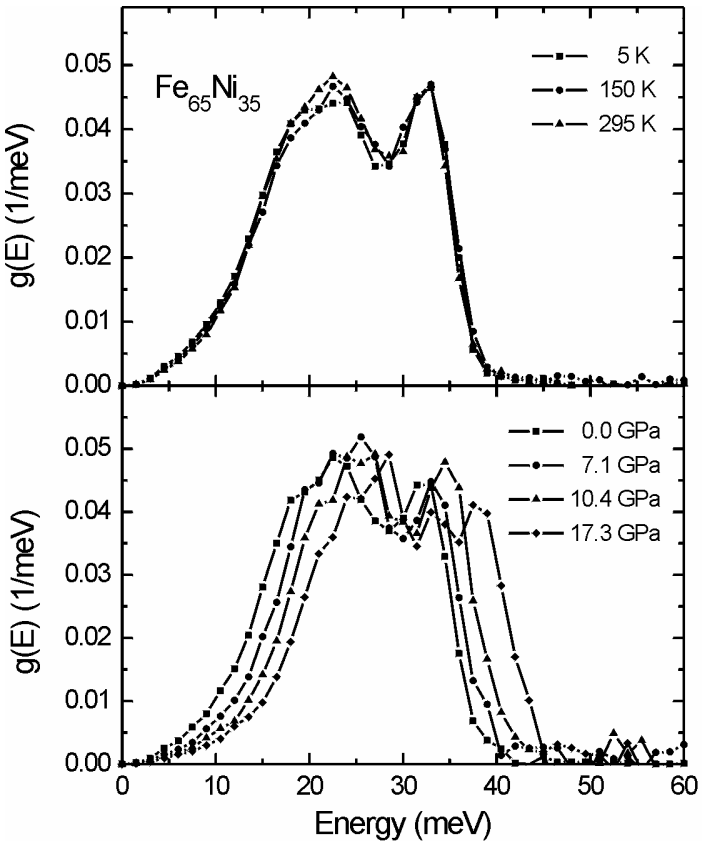
Report:

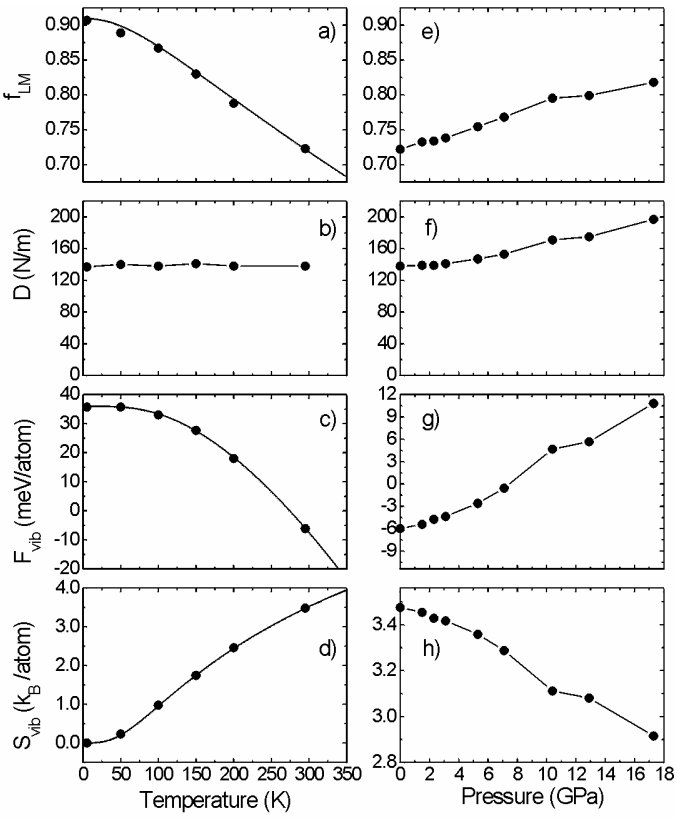
This beam time was a continuation of our proposal HS-1488: "High-pressure phonon spectroscopy of Fe-Ni alloys". In that previous beam time we have studied the classical Invar system $Fe_{65}Ni_{35}$, at 300 K and pressures up to 10.4 GPa as well as at ambient pressure and temperatures between 5 K and 300 K (see experimental report HS-1488). We did observe only subtle temperature effects on the phonon density-of-states (DOS), reflecting the special Invar property, namely the anomalous small thermal expansion of $Fe_{65}Ni_{35}$. The application of pressure, however, lead to strong and characteristic changes in the phonon-DOS, in particular a strong increase of the low-energy modes to higher energies and only minor changes of the high-energy modes (see Fig. 1). Even between 7.1 and 10.4 GPa, the highest pressures, a non-proportional increase of the low- and high-energy modes was observed, pointing to remains of the Invar properties. Since an inelastic IXS study [1] has reported that the Fe moment vanishes around 12 GPa, and since the Invar properties are inherently connected with the Fe magnetism, we continued our studies to higher pressures, here 12.9 and 17.3 GPa, where we expected to observe a "normal" Debye behaviour, here an about proportional variation of low-energy and high-energy modes in the phonon-DOS. The same sample (15 μm thick, 95% enriched in Fe-57), resting half a year in the diamond-anvil cell at 10.4 GPa, was used. The experimental conditions were similar to the previous study, the monochromatized beam had the same width of 3.5 meV. Again we used the two techniques connected with nuclear scattering of synchrotron radiation, the local phonon-DOS was obtained from nuclear inelastic scattering (NIS) and the magnetic properties and absorber thickness (proportional to f_{LM}, the Mössbauer-Lamb factor) were measured with (elastic) nuclear forward scattering (NFS).

Fig. 1 shows on the left panel the phonon DOS extracted from NIS spectra of $Fe_{65}Ni_{35}$ measured at various pressures and temperatures in both beam-times. Details of the evaluation of the DOS and the derived elastic and thermodynamic properties, shown in Fig. 2, are described in Ref. 2. Comparison of the phonon-DOS at 10.4 and 17.3 GPa reveal now a proportional variation of the low- and high-energy region of the phonon-DOS as expected for the suppression of the Invar properties. This is also clearly reflected by a kink around 12 GPa in the derived parameters shown in Fig. 2. These results have just been submitted for publication [2].

The NFS spectra shown in Fig. 3 were measured with decreasing pressure starting from 16 GPa. The sharp Bessel beats arise from the effective sample thickness, which is proportional to f_{LM}. At 5 GPa, these Bessel beat structures start to broaden, and already at 4 GPa magnetic interaction have completely changed the spectral features, demonstrating that the onset or suppression of magnetic order can be sensitively monitored by NFS spectra.

[1] J.P. Rueff et al., PRB 63, 132409 (2001).
[2] H. Giefers, K. Rupprecht, O. Leupold, G. Wortmann, submitted.





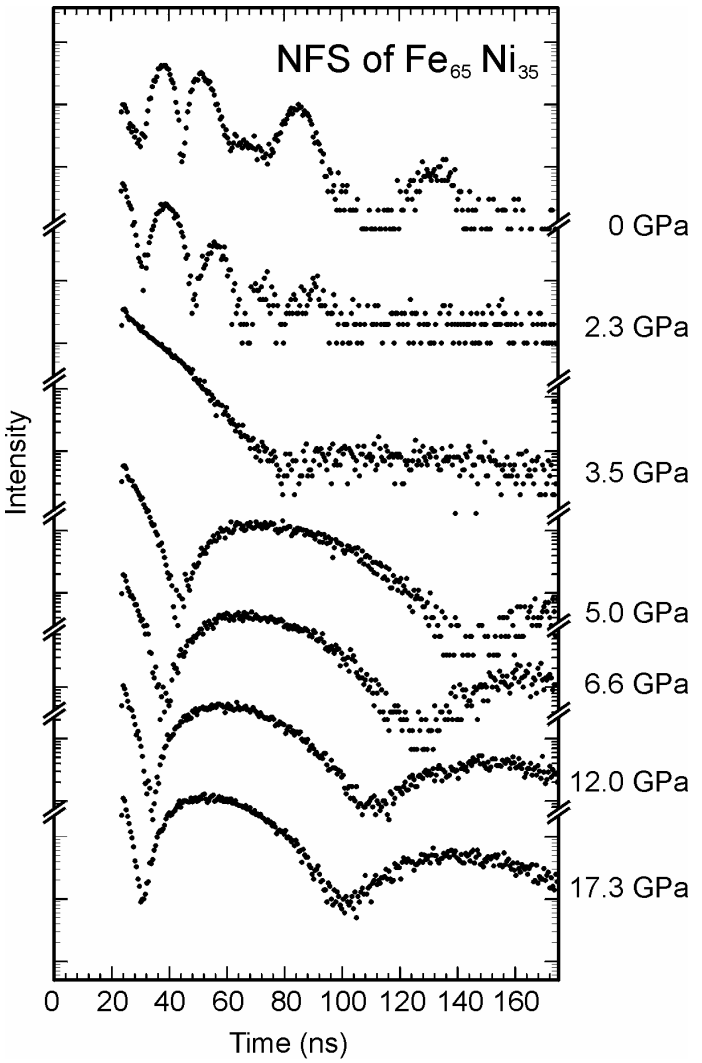


Fig. 1: (up left): Phonon-DOS of $Fe_{65}Ni_{35}$ at selected temperatures and pressures. Note the different variation of the low-energy and high-energy side of the DOS with pressure.

Fig. 2: (up right) Elastic and thermodynamic parameters of $Fe_{65}Ni_{35}$, derived from the phonon-DOS. Note a characteristic kink in the pressure dependence around 12 GPa, indicating the suppression of the Invar properties.

Fig. 3: (left) NFS spectra of $Fe_{65}Ni_{35}$ at different pressures, measured from the highest pressure downwards, exhibiting between 5 and 16 GPa Bessel beats, whose minima position depend on the effective absorber thickness (and here directly to f_{LM}, since the geometrical thickness of the absorber foil does not change with decreasing pressure).