ESRF	**Experiment title:** **Phonon spectroscopy under high pressure using inelastic nuclear scattering II**	**Experiment number:** HS-583
Beamline: BL11/ID18	**Date of experiment:** from: 04.06.98 to: 07.06.98	**Date of report:** **30.08.98**
Shifts: **9**	**Local contact(s):** H.F. Grünsteudel	*Received at ESRF:*

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Report:

This experiment is a continuation of experiment HS-271, where we performed the first high-pressure study of inelastic nuclear scattering (INS) in a-iron up to 10 GPa (see corresponding report). The INS technique uses the same setup as for elastic nuclear forward scattering (NFS), but is scanning the high-resolution monochromator (HRM), here with a bandwidth of about 4.4 meV, instead of 6.6 meV, over the nuclear resonance [1,2] and monitoring the inelastically in 4π geometry scattered photons, originating from the creation or annihilation of phonons in the (inelastic) absorption process by the Mössbauer nuclei. In the case of Fe-57, one observes the reemitted 14.4 keV gammas and the Fe $K_{\alpha,\beta}$ x-rays of 6.4 and 7.1 keV (from the internal conversion process) in an intensity ratio of 1 : 9. The Fe $K_{\alpha,\beta}$ x-rays are, due to their low energy, completely absorbed in a normal high pressure cell. In our first experiment we used a specially developped diamond anvil cell (DAC) with epoxy as gasket material, allowing an easier transmission of the reemitted photons perpendicular to the SR beam. This gasket material limited the maximum pressure to 10 GPa, where iron is still in the a-phase.

For the present experiments we improved the performance of our DAC by using beryllium as gasket material and by increasing the opening angle for the inelastic photon detection. We reached 24 GPa, where iron is completely transformed to the E-phase (as evidenced also by the NFS spectrum) and measured for the first time the INS spectrum of this modification. The spectra for 0 and 24 GPa are shown in Fig. la. Due to the small sample size (150 μm) and the strong absorption in the DAC, the inelastic count rate is strongly reduced and the measuring time for the spectrum at 24 GPa was ca. 40h. After fitting and removing the elastic peak at zero energy, the measured spectra can be normalized according to ref. [1], using a sum rule for the probability of recoil. The Lamb-Mössbauer-factor f_{LM} can be calculated from the integrated intensity of the

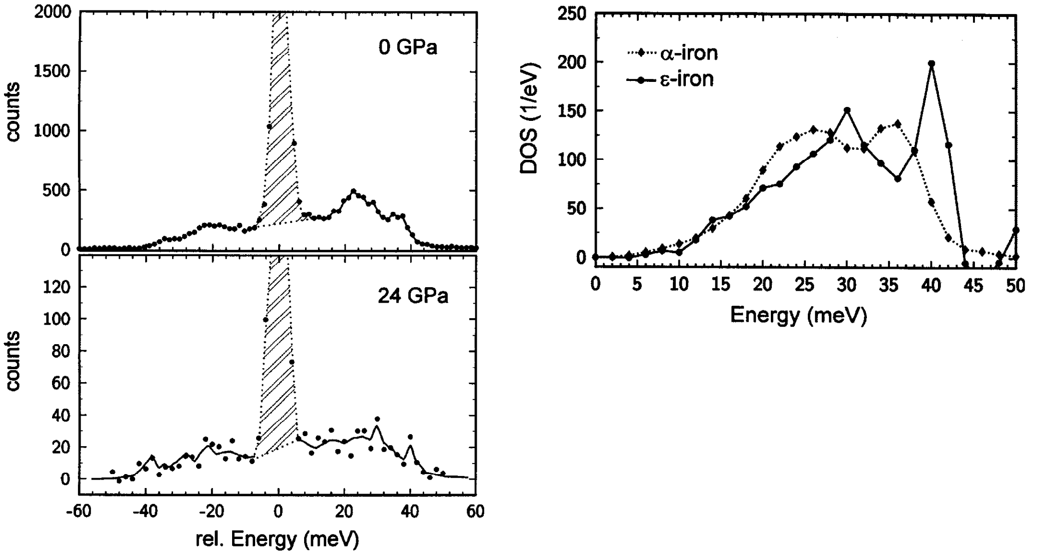

Fig. 1: a)(left panel) Inelastic nuclear scattering of iron at ambient pressure and at 24 GPa, corrected for background. b) (right panel) Derived Phonon DOS for both iron modifications.

normalized spectrum [see 1,2]. For &-iron we get f_{LM} = 0.84(10), which compares to 0.79(1) for a-iron at ambient pressure. The basic goal of the present NFS study is the extraction of the phonon density of states (DOS) for ε-iron from of the one-phonon contributions to the normalized INS-spectra (assuming a harmonic behaviour of the lattice [see 1,2]). The corresponding spectra for both iron modifications are shown in Fig. lb. Although the statistical accuracy of the high-pressure data is still low, a significant increase of the phonon frequencies is already visible.

This experiment was carried out without any kind of focusing optics (attempts to install a focusing crystal failed because of the limited beamtime). From the allocated 9 shifts we had to use 4 shifts for the installation of the 4.4 meV monochromator and for reference measurements. With the sample diameter of 150 µm we are presently using less than 2% of the beam size. After the installation of the new pre-monochromator and the use of focusing elements at ID 18, we should gain a factor of at least 10 in the beam intensity on the pressurized sample; spectra with the same statistical accuracy as that at ambient pressure should be measured within a day.

Having in mind the importance of ε-iron for geophysics [3], a detailed knowledge of the phonon DOS of ε-iron at different pressures and the derived parameters (like mean force constant, specific heat, kinetic energy per atom, . . .) is of actual scientific interest.

References:

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[2] A. Chumakov et al. , Phys. Rev. B 54, R9596 (1996).
[3] S. Gilder et al., Science 279,72 (1998) and 0. Anderson, Science 278, 821 (1997)