ESRF	**Experiment title:** Elasticity of (Mg,Fe)O across the spin-pairing transition: spin-phonon coupling?	**Experiment number**: HS-3864
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Report:

We carried out inelastic x-ray scattering (IXS) measurements on $Mg_{0.83}Fe_{0.17}O$-ferropericlase single crystals compressed in diamond anvil cells (DAC) up to 62 GPa.

Oriented single crystals of ferropericlase were synthesized starting from pre-aligned crystal of MgO (normal parallel to [110] direction) by high-temperature Fe-Mg interdiffusion in a piston cylinder press (1400 C at 1 GPa for 200 hours). The oxygen fugacity condition of the experiment was buffered close to IW (Iron-Wüstite) buffer by loading the sample in an iron capsule. A 40 nm thick focused ion beam section was cut out of the recovered sample for electron energy loss spectroscopy measurement. No detectable amount of ferric iron was measured in the single crystal of ferropericlase.

The samples were polished to ~15-20 μm thickness and cut to approximately 40 μm diameter. We produced samples with different iron content x, varying in the 0.13 to 0.21 range; the exact composition was determined individually for each disk by electron microprobe analysis. For the IXS experiment we employed a sample with x=0.17, loaded in a DAC equipped with 300 μm culet diamonds and rhenium gasket. We used neon as pressure transmitting medium to ensure quasi-hydrostatic compression.

We performed IXS measurements on beamline ID28, operating the instrument in the Si(9,9,9) configuration, which provides 3 meV total energy resolution full-width-half-maximum (FWHM). The direction and size of the momentum transfer were selected by an appropriate choice of the scattering angle and the sample orientation in the horizontal scattering plane. The momentum resolution was set to 0.28 nm^{-1} and 0.84 nm^{-1} in the horizontal and vertical planes, respectively. The focused x-ray beam of 30 x 90 μm^2 FWHM was further reduced in the vertical by slits to match sample dimensions. Measurements have been performed in transmission geometry, with the incoming x-ray impinging along the cell axis, across the diamonds.

We carried out IXS and diffraction measurements at 1.8, 26, 47, 54 and 62 GPa. At each investigated pressure point we collected 2-3 spectra in the linear part of the dispersion of four independent acoustic phonons, LA (100), LA(110), $TA(110)_{<001>}$ and $TA(110)_{<-110>}$, deriving the sound velocity from the initial slope of the dispersion. We then obtained the three independent element of the elastic tensor (C_{11}, C_{12} and C_{44}) solving the Christoffel equation.

In the allocated beam time we have been able to investigate the pressure stability range of the high spin state [1] and just across the spin-pairing transition (around 58 GPa for this specific composition [2]). While C_{44} and C_{12} exhibit a softening approaching the transition, C_{11} retained a normal behavior, monotonically increasing with increasing pressure, so that the overall effect on the aggregate velocities is rather small. Data

at 62 GPa, in the low spin state, show a hardening of C_{44} and C_{12}, but the measured values are still lower than what expected on the basis of a linear extrapolation of low-pressure data.

The pressure evolution we observed for C_{44} and C_{12} compares overall favourably with previous experimental [3-5] and theoretical studies [6], while our IXS measurements do not evidence the large softening of C_{11} reported in [3,5-6]. In support of our observations, we note that we have been able to directly determine all the elastic moduli without any independent input, while all previous measurements necessitated extra constraints (equation of state or bulk modulus). This might be particularly critical in the pressure range where the two spin-state coexist, and specifically across the transition, where the compression curve exhibit a discontinuity (see for instance [7]).

References

[1] *"Iron partitioning in the Earth's mantle: toward a deep lower mantle discontinuity*", J. Badro *et al.,* Science 300, 789 (2003).

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[4] *"Elastic shear anisotropy of ferropericlase in Earth's lower mantle",* H. Marquardt *et al.,* Science 324, 224 (2009).

[5] *"Single-crystal elasticity of ($Mg_{0.9}Fe_{0.1}$)O to 81 GPa"*, H. Marquardt *et al.,* Earth Planet. Sci. Lett., in press (2009).

[6] *"Anomalous compressibility of ferropericlase througout the iron spin cross-over"*, R. Wentzcovitch *et al.,* PNAS 106, 8447 (2009).

[7] *"Spin transition and equations of state of (Mg, Fe)O solid solutions"*, Y. Fei *et al.,* Geophys. Res. Lett. 34, L17307 (2007).