ESRF	Experiment title: Phonon lifetime and lattice thermal conductivity of MgO at simultaneous high pressure and high temperature	Experiment number: ES-402
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Names and affiliations of applicants (* indicates experimentalists):
HE* Fei, GIURA* Paola and ANTONANGELI* Daniele
IMPMC, CNRS, Sorbonne Universités, Paris, France

Report:

The goal of this research project is the study the phonon energies and phonon widths of selected longitudinal acoustic (LA) and transvers acoustic (TA) modes in single crystalline magnesium oxide (MgO) at high pressure and high temperature, extending upon previous measurements at high temperature and ambient pressure (see experimental report ES-148).

Commercially available MgO crystals from Sigma-Aldrich with dimensions of 10x10x0.5 mm and pre-oriented in [100] direction were sized for diamond anvil cell experiments by mechanical polishing and fs laser cutting. So-prepared samples were ~ 40 μm in diameter ~20 μm in thickness.

In order to simultaneously generate high pressure and high temperature we used external resistive cells from the ESRF pool, equipped with Re gaskets and 350 microns culets diamonds. Samples have been loaded together with a ruby chip and some $SrB_4O_7:Sm^{2+}$ powder as pressure calibrants using neon as pressure transmitting medium. Pressure was increased up to ~11 GPa prior beginning of the experiment, and samples checked by diffraction to define the orientation matrix and to select those with the most favorable orientation.

Inelastic x-ray scattering measurements have been initially carried out using the Si(11,11,11) configuration. However, with a sample of a thickness of only ~20 μm and the limited flux provided by Si (11,11,11), integration time resulted to be prohibitively too long (we remind that measurements on bigger samples at ambient conditions have been carried out using Si(12,12,12) reflection). To optimize the use of the allocated beamtime, we opted for the Si(9,9,9) configuration, with incident photon energy of 17.794 keV and a total instrumental energy resolution of 3 meV full-width-half-maximum (FWHM). Flux was enough to collect good-quality data in reasonable amount of time (~80-120s per energy point at low q, ~180-240s per energy point at high q). The dimensions of the focused x-ray beam were 25×60 μm^2 (horizontal×vertical, FWHM), further reduced by slits to 25×40 μm^2 to better match sample's dimensions. The momentum resolution was set to 0.3 nm^{-1}. The inelastic excitations have been fit by a Lorentzian function convoluted with the experimental determined resolution function, utilizing a standard $\chi 2$ minimization routine.

-Experiments at P = 13.3GPa T = 293K:

The pressure of the cell was increased from 11 GPa to 13.3GPa.

Sample was of very good crystalline quality (rocking width <0.1°) and still oriented with normal to the surface along the (1,0,0) direction after the loading. We probed the dispersion of LA(ξ,0,0) phonon mode. An example of collected data is shown in Figure 1.

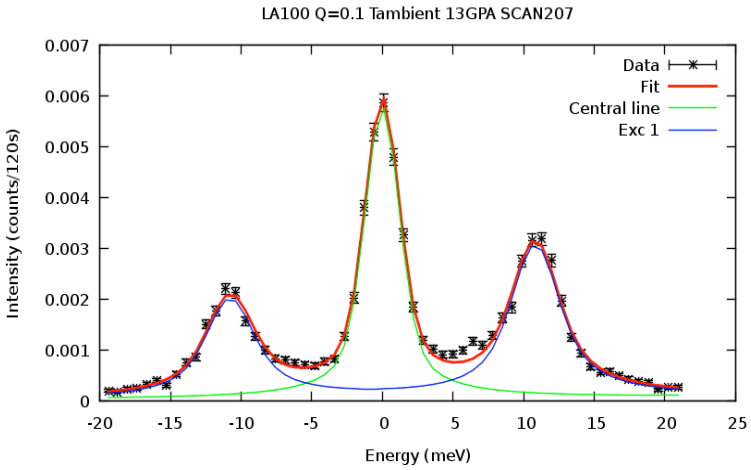

Figure 1: Example of collected IXS spectra. LA(ξ,0,0) at reduced q=0.1 measured at 13.3 GPa. The red line is the fit to the experimental points. Blue and green lines are individual contributions.

-Experiments at P = 15.4GPa T = 673K:

We increased temperature to 673 K, which resulted in an increase in the pressure to 15.4 GPa. Temperature's fluctuations were within 50K over the entire collection time. We collected the dispersion of the TA(ξ,0,0)$_{<00ξ>}$, LA(ξ,0,0) and LA(ξ, ξ,0) phonons.

-Experiments at P = 19GPa T=873K:

Both pressure and temperature of the cell were further increased to 19 GPa and 873 K to probe temperature effects at constant volume (anharmonic effects). Temperature's fluctuations were within 10K over the entire collection time. We collected the dispersion of the TA(ξ,0,0)$_{<00ξ>}$ and LA(ξ,0,0) phonons. An example of collected data is shown in Figure 2.

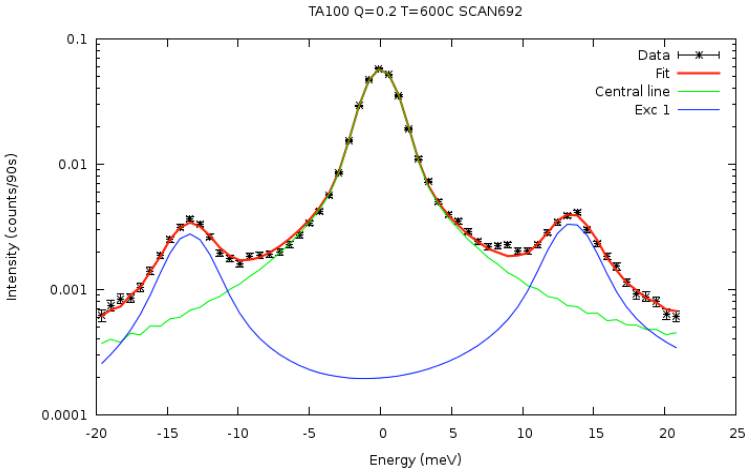

Figure 2: Example of collected IXS spectra. TA(ξ,0,0) at reduced q=0.2 measured at 19 GPa and 873K. The red line is the fit to the experimental points. Blue and green lines are individual contributions.

-Experiments at P = 15GPa T=293K:

The heating is stopped and the temperature of the cell is slowly lowered to room temperature. The pressure of the membrane is then released to have a sample volume as close as possible to that probed in the high-temperature experiments. We collected the dispersion of the TA(ξ,0,0)$_{<00ξ>}$ and LA(ξ,0,0) phonons.

In order to perform experiments at higher temperature we also tried twice to use a resistive heated DAC equipped with internal heating (around the diamonds). Unfortunately no one of the attempts were successful: the first failed because of the poor quality of the sample after the gas loading, while in the second diamonds cracked before temperature increase. Further attempts will be carried out in next beamtime.

In summary, we performed IXS measurements of MgO at high temperature and high pressure up to 19GPa and 873K. Present data allow us to track the evolution with pressure and temperature of the phonon energies. Concerning the phonon widths, preliminary results are promising, but detailed analysis is still in progress as the energy resolution of the Si(9,9,9) make challenging to derive conclusions on the temperature effects.