ESRF	Experiment title: Transformation Microstructures in (Mg,Fe) ₂ SiO ₄ : Study Using In-Situ X-Ray Diffraction	Experiment number : ES-656
Beamline: ID 27	Date of experiment:from:12-dec-2017to:16-dec-2017	Date of report : 5-mar-2018
Shifts:	Local contact(s): Volodymyr SVITLYK	Received at ESRF:
Names and affiliations of applicants (* indicates experimentalists):		

Marine COLLEIT (Unité Matériaux et Transformations, CNRS, Université Lille 1, France) Sébastien MERKEL (Unité Matériaux et Transformations, CNRS, Université Lille 1, France) Julien CHANTEL (Unité Matériaux et Transformations, CNRS, Université Lille 1, France) Nadège HILAIRET (Unité Matériaux et Transformations, CNRS, Université Lille 1, France)

Report:

In this proposal, we wanted to follow a sample microstructures as (Mg,Fe)₂ SiO₄ transforms from olivine to wadsleyite or ringwoodite. Such phase transitions occur at temperatures and pressures in the 1500-2000 K and in the 10-20 GPa ranges respectively.

We prepared samples of $(Mg,Fe)_2SiO_4$ polycrystalline olivine, shaped into disks of 30 µm diameter and 15 micron thickness, with a 100 nm coating of platinum on both sides. They were loaded in a rhenium gasket with MgO as a pressure-transmitting medium. The goal of the experiment was to bring the sample to a fixed temperature using laser-heating, increase pressure to 10-20 GPa, and convert olivine to its high pressure polymorph at constant temperature We also wanted to collect insitu multigrain crystallography data during the transformation, as pressure was increased at constant temperature, with the aim of following the phase proportions and microstructure evolution during the transformation.



Typical sample preparation. Polycrystalline olivine, shaped into disks of 30 μ m diameter and 15 μ m thickness, with a 100 nm coating of platinum on both sides, loaded in MgO pressure medium inside a 100 μ m diameter hole in a rhenium gasket.

Based on previous experience with such system, we anticipated that maintaining a constant temperature with laser heating during transformation was going to be difficult. The 100 nm platinum coating on both sample sides, which served as a laser absorber, was critical and allowed for successful data collection.

During our beamtime, we worked on 5 samples

- Sample 1 was compressed at temperatures between 1700 and 2000 K, between 9 and 13 GPa with a conversion from olivine to wadsleyite.
- Sample 2 was compressed up to 5 GPa at ambient T, further compressed up to 11 GPa at 1700 K with a clear transformation of olivine to wadsleyite.
- Sample 3 was compressed to 5 GPa at ambient T, compressed up to 20 GPa at 1400 K and later 1800 K. Transformation to either wadsleyite or ringwwodite.
- Sample 4 was compressed to 4.25 GPa and heated at 1700 ± 200 K. Unfortunately, this resulted in a sample breaking in several parts. The experiment was stopped.
- Sample 5: time was running out. Instead of attempting a transformation to a high pressure phase, we collected a reference dataset on an olivine sample, inside a diamond anvil cell at ambient pressure and temperature. This dataset will be used to test and develop new multigrain crystallography procedures for diamond anvil cell experiments.

Overall, we could clearly observe the transformation of olivine to wadsleyite in two samples. We observed a potential phase transformation in a third sample. Finally, we collected a reference dataset.

We will now processed the multigrain XRD data in order to analyse the sample microstructure before, during, and after conversion. We will also study the recovered samples using electron microscopy. A reference dataset was also collected. This dataset was collected in the typical conditions of a DAC experiment but not pressure was applied in ordre to fully preserve the sample and allow a 100% comparison between XRD and electron microscopy.

These results will be important for understanding dynamical processes at the discontinuity at 410 km in the Earth's mantle. Sample microstructure affect the mechanical properties of the rocks. They also have an effect on the seismic waveforms and can hence be probed using seismic observations in order to validate predictions of microstructures based on experiments and our understanding of Earth's processes.