ESRF	**Experiment title:** Multigrain crystallography study of the perovskite to post-perovskite transformation in $(Mg,Fe)SiO_3$	**Experiment number**: ES-570
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Report:

The goal of this experiment is the study of microstructures induced by the perovskite to post-perovskite phase transition using multigrain crystallography. This transition occurs in the D" layer, 2900 km below the Earth's surface in $(Mg,Fe)SiO_3$ and is important for understanding the dynamics of the lower mantle. Thanks to the installation of the new laser heating system at ID27, this transformation can now be studied in situ at relevant Earth P/T conditions using multigrain crystallography, which is what we did during our experiment.

Using the Laser Heated (LH) system coupled with the rotational stage developed at ID27, we conducted simultaneous high pressure high temperature multigrain crystallography experiments up to 160 GPa and 5000 K. Multigrain crystallography will be used for extracting individual grain positions and orientations within the sample and decipher the transition mechanism in this material.

During this experimental run, we studied 6 different samples under different P/T conditions. For runs 1, 2 and 4, the sample was $NaCoF_3$ along with Pt and MgO or Argon pressure marker. For runs 3 and 5, the sample was $(Mg,Fe)SiO_3$ along with Pt pressure marker and Aron as pressure medium. For the last run (run 6), the sample was Olivine along with KCl pressure medium and marker. Double-sided Planck and Wien fits, were used for temperature calibration. The samples were loaded in diamond anvil cells equipped with 100 or 150 or 300 or 350 µm culet diameter anvils and a rhenium gasket. The ID27 beamline experimental setup consisted of a 3.0 (Horizontally) x 3.0 (Vertically) $µm^2$ focused monochromatic X-ray beam tuned to 0.3738 Å. X-ray diffraction pattern were collected using MAR 265 CCD at a distance of 254 mm from the sample.

For the sample $NaCoF_3$, Run 1 was successful to collect diffraction image in order to build a P/T phase diagram between to 0 and 25 GPa and 1000 to 1500 K. However, we could not collect multigrain cristallography data because of the argon melting inside the sample chamber and, also, because we realized that inside out target P/T region, the phase of interest (post-perovskite) was decomposing into other phases, with no relevance to our scientific objectives. Morevover, we realized that the argon pressure medium was melting inside the sample chamber, preventing any multigrains crystallography data collection. Run 2 and 4 failed because of experimental difficulties.

For the run 3, we successfully transformed from $MgSiO_3$-enstatite to $MgSiO_3$-perovskite at 84 GPa and 2500K. We then tried to improve the microstructure of our sample (i.e. inducing grain growth) by heating it at higher temperature and highe pressure. At 3600 K, the pressure was 98 GPa and we converted the sample into a mixture of $MgSiO_3$-perovskite, argon, MgO and an unknown phase that could be either stishovite or the H-phase. This unknown phase disappeared upon further heatig at constant pressure. We then increased pressure and decreased the temperature to 2600 K to reach the D" conditions and tried to pass the phase transformation from $MgSiO_3$-perovskite to $MgSiO_3$-post-perovskite. Pressure was then increased to 160 GPa while maintaining constant temperature. We observed the start of transformation from $MgSiO_3$-perovskite to $MgSiO_3$-post-perovskite. This will have to be confirmed with further data processing. We then attempted a reverse transformation to perovskite but did not succeed because of the failure of a diamond anvil.

For run 5, we successfully transformed from $MgSiO_3$-enstatite to $MgSiO_3$-perovskite at 75 GPa and 1700 K. We then compressed $MgSiO_3$-perovskite to 115 GPa and heated the sample to 2500 K to reach the Earth's D" conditions. We observed a beginning of transformation from $MgSiO_3$-perovskite to $MgSiO_3$-post-perovskite. Pressure was then increase to 153 GPa with no apparent change in the diffraction patterns. After checking the alignement of the heating spot, we realized the laser had drifted by 7 microns from the X-ray beam. At the heating position, the sample was completely and successfully transformed to $MgSiO_3$-post-perovskite. Pressure was then decrease to 122 GPa until we saw a back-transformation from $MgSiO_3$-post-perovskite to $MgSiO_3$-perovskite at 2500 K.

In run 3 and 5, multigrain crystallography data were collected while the sample was heated and compressed. At pressures (up to 170 GPa) and temperatures (up to 3000K), multigrain crystallography relies on diffraction images collected while rotating ω from -22° to 22° in 0.5° increments, resulting in 88 diffraction images per {pressure, temperature} points. Data analysis is in progress using single grain analysis technique and the software FABLE. These data will allow extracting various information from single grains inside the polycrystalline sample and will help us to understand the mechanism of transformation between perovskite and post-perovskite.

In run 6, Beamtine was running out after the study of these 5 sample. The last hours of beamtime were used to check the feasability of a multigrain cristallography experiment on phase transformation in olivine.

Overall, we are satisfied from this experiment. We used the allocated beamtime to successfully study the phase transformation from perovskite to post-perovskite in $NaCoF_3$ and in $(Mg,Fe)SiO_3$ using multigrain crystallography method. These data will definitely allow us understand the observed mechanism in the $(Mg,Fe)SiO_3$-perovskite to $(Mg,Fe)SiO_3$-post-perovskite transformation and the reversed transformation in the Earth's D'' layer.