ESRF	**Experiment title:** Grain by grain study of the perovskite to post-perovskite transformation	**Experiment number**: ES-219
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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 and is important for understanding the dynamics of the lower mantle. Transformation in the real Earth composition, $(Mg,Fe)SiO_3$, can not be studied using multigrain crystallography at the moment because of the required conditions of pressure and temperature. Hence, during this experiment, the phase transformation was studied in a structural analogue $NaCoF_3$. $NaCoF_3$ is in the perovskite structure at ambient pressure and transforms to post-perovskite at high pressure.

Using the external heating diamond anvil cell (DAC) developed at the beamline ID27, we conducted simultaneous high pressure and high temperature multigrain crystallography experiments up to 30 GPa and 900 K. Multigrain crystallography will be used for extracting individual grain positions and orientations within the sample and decipher the transformation mechanism in this material.

During the experiment, we studied five different samples, under different P/T conditions. For each run, the sample was $NaCoF_3$ along with gold pressure marker. Thermocouple were used for temperature calibration. The samples were loaded in DAC equipped with 350 µm culet diameter anvils and a rhenium gasket. The ID27 ESRF beamline experimental setup consisted of a 7.5 (Horizontally) x 5 (Vertically) μm^2 focused monochromatic X-ray beam tuned to 0.3738 Å. Moreover X-ray diffraction pattern were collected using Perkin Elmer Detector with an active area of 2048 x 2048 pixels at a distance of 478 mm from the sample. Runs 1, 2, and 4 failed because of experimental difficulties (loss of vacuum around the DAC, problems with sample characterization, electrical connections). Runs 3 and 5 were successful.

In run 3, we heated the sample to 270°C at low pressure and compressed it. At about 17 GPa, grains broke apart into a very fine powder, with signs of post-perovskite in the diffraction signal. However, the amount of post-perovskite in the signal was small and did not increase with a pressure increase to 35 GPa. We then heated the sample to 570°C with no apparent change in the diffraction patterns. Pressure was then decreased while maintaining the temperature at 570°C. We observed a back-transformation into large perovskite grains at around 12 GPa with no further evolution upon pressure decrease to 0 GPa and temperature decrease to ambient conditions.

In run 5, we heated the sample up to 620°C at ambient pressure, compressed it, and saw a transformation from large perovskite grains to large post-perovskite grains near 17 GPa. We later decreased pressure, and saw back-transformation into large perovskite grains at around 11-12 GPa.

In all cases, multigrain crystallography data were collected, before, after, and during the phase transition. At each pressure, multigrain crystallography relies on diffraction images collected while rotating ω from -30° to +30° in 0.25° or 0.5° increments, resulting in 240 or 120 diffraction images per pressure point.

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. The data collected in run 5 where large grains of a pure post-perovskite phase were observed above 17 GPa will be particularly helpful for this task.

In a following proposal (ES 334), we propose to collect data on another low pressure analogue, $NaNiF_3$, to confirm the results obtained here in $NaCoF_3$. These data will allow us extrapolate the observed mechanism for the $(Mg,Fe)SiO_3$-perovskite/post-perovskite transformation in the Earth's D'' layer.