ESRF	**Experiment title:** Grain by grain study of the preovskite to post-perovskite transformation in $NaNiF_3$	**Experiment number:** ES-334
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Report:

The aim of this project is the study of microstructures induced by 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. As such transformation microstructures induced by the perovskite to post-perovskite transformation in ABX_3 compounds are relevant for deep Earth geophysics. During this experiment, the phase transformation was studied in a structural analogue $NaNiF_3$.

Using the external heating diamond anvil cell (EH-DAC) and internal heating diamond anvil cell (IH-DAC) developed at the beamline ID27, we conducted simultaneous high pressure and high temperature multigrain crystallography experiment up to 25 GPa and 900 K. Multigrain crystallography will be used for extracting individual grain orientations within the sample and understand the mechanism for the perovskite to post-perovskite transformation in this material.

During the experiment, we studied four different samples, under different P/T conditions. For each run the sample was $NaNiF_3$ with gold or tungsten as pressure marker. Thermocouple were used for temperature calibration. Two samples were loaded in EH-DACs equipped with 350 µm x 400 µm culet diameter anvils and a rhenium gasket. The two other samples were loaded in IH-DACs equipped with 300 µm x 350 µm culet diameter anvils and a rhenium-boron/epoxy gasket. The ID27 ESRF beamline experimental setup consisted of a 6.8 (horizontally) x 6.3 (vertically) μm^2 focused monochromatic X-ray beam tuned to 0.3738 Å. X-ray diffraction pattern were collected using Perkin Elmer Detector with an active area of 2048 x 2048 pixels at a distance of 497 mm from the sample. Run 1 and 2 failed due to experimental difficulties (loss of vaccum around the DAC, chemical reaction between sample and gasket, electrical connections). Runs 3 and 4 were sucessful.

In run 3, we used an IH-DAC. We first first increased the pressure to ensure the closure of the sample chamber. Then we heated the sample to 630°C (900 K) at low pressure. Pressure was then increased while maintaining a constant temperature of 900 K.. The sample were fine textured powder and grains. At about

16.7 GPa, we observed signs of post–perovskite in the diffraction signal. Pressure was then increased to 21.4 GPa, increasing the amount of post-perovskite grains in the signal but never achieving a full transformation. Pressure was then decreased to study the back transformation. We observed a back transformation into prerovskite grains at around 11 GPa. Near 9 GPa, the transformation from post-perovskite to perovskite was fully achieved.

In run 4, we used an EH-DA. Firstly, we heated the sample up to 475°C (700 K) at ambiant pressure, compressed it and saw a transformation from large perovskite grains to small post-perovskite grains near to 13 GPa. Here again, we could not have a complete transformation. We later decreased the pressure and saw a back transformation at around 10 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 -21° to 24 ° in 0.5° steps, resulting in 90 images per pressure point.

Data analysis is in progress using single grain analysis technique and the sofware FABLE.These data will allow extracting various information from single grain inside the polycristalline sample and will help us to understand the mechanism of transformation between perovskite and post-perovskite. Nevertheless the data collected in run 3 and 4 include both big grains and a fine grain matrix. For that reason the multigrain crystallography data analysis will be different from a "classical treatment" in order to accormodate both larger grains data, using fable, and that of the fine matrix.

Thanks to to experimental improvement on ID27, we propose to collect data on the $(Mg,Fe)SiO_3$ perovskite to post-perovskite phase transformation itselt in the following proposal ES-399. These data will allow us to observe mechanism of this transformation in the Earth's D" layer and confirm the behaviour of analogues.