<table>
<tr><td>ESRF</td><td>Experiment title:
Grain by grain study of the perovskite to post-perovskite transformation in (Mg,Fe)SiO3</td><td>Experiment number:
es-399</td></tr>
<tr><td>Beamline:
ID27</td><td>Date of experiment:
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18</td><td>Local contact(s):
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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 new rotating laser heating system at ID27, we were able to perform multi-grain crystallography at pressures up to 170 GPa and temperatures above 3000 K and work on $(Mg,Fe)SiO_3$ itself. Multigrain crystallography under such conditions is a world's first. Multigrain crystallography will be used for extracting individual grain orientations within the sample and decipher the transition mechanism in $(Mg,Fe)SiO_3$.

During this experiment, we performed 11 runs on 11 different samples under different P/T conditions. For each run, the starting composition was $(Mg,Fe)SiO_3$ along with KCl or NaCl as pressure transmitting media and pressure markers. The samples were loaded in Diamond Anvil Cells (DAC) equipped bevelled diamonds with inner culet diameter of 100 and 150 µm and a rhenium gasket. The ID27 beamline with tuned to a 3.0 (H) x 2.4 (V) μm^2 monochromatic X-ray beam of 0.3738 Å. X-ray diffraction pattern were collected using MAR 265 CCD at a distance of 241 mm from the sample. Temperature measurements were performed using spectral radiometry.

Run 1 was a test of multigrain crystallography with laser heating. We successfully transformed enstatite into perovskite in $(Mg,Fe)SiO_3$ and followed the growth of individual perovskite grains over several hours up to 50 GPa and 2000 K. Other runs were dedicated to the study of the perovskite to post-perovskite transformation. Among them, runs 5, 9,10, and 11 provided valuable information.

In run 11, we successfully transformed from $MgSiO_3$-enstatite to perovskite at 50 GPa and 3000 K. The sample microstructure was preserved thanks to a slow compression to 120 GPa at ambient temperature. We then heated the sample to 2500 K to reach the Earth's D'' conditions with no apparent change in the diffraction patterns. Pressure was then slowly increased to 170 GPa while maintaining the temperature between 2500 K and 3000 K. In this run, we suspect the appearance of some grains of $MgSiO_3$-post-perovskite but this observation will need further confirmation and data evaluation. In any case, the full transformation to post-perovskite was not observed. Experiment ended with diamond failure at 170 GPa.

In runs 5 and 9, we transformed from $MgSiO_3$-enstatite to $MgSiO_3$-perovskite at 82 GPa and 2200 K and 43 GPa and 1500 K, respectively. We then increased pressure up to 120 GPa, heated the sample to 2500 K, and further increased pressure. We observed the start of a transformation from the post-perovskite structure. Near 150 GPa, for both runs, experiment ended due to diamond failure. The details of the phase transformation are now being analysed. Run 10 was similar except that $(Mg,Fe)SiO_3$-perovskite was synthesized from enstatite at higher pressure and temperature (88 GPa and 5500K).

Multigrain crystallography data were collected while rotating the sample in ω over $\Delta\omega = 50°$ in 0.5° δω increments, resulting in 100 diffraction images per P/T points. Preliminary analysis demonstrates that *i)* the rotating laser heating stage is stable and, *ii)* growth of invidual post-perovskite grains can be monitored over time (Fig 1). Data analysis is now in progress using the software package FABLE. These data will allow extracting information from single grains inside the polycrystalline sample and will help us in understanding the mechanism of transformation between perovskite and post-perovskite.

Collecting stable multigrain crystallography data up to 170 GPa and 3000K is a great success. We were, on the hand, disappointed with the fact that our sample did not fully convert to post-perovskite. Hence, in a following proposal, we will aim for a complete transformation from perovskite to post-perovskite by varrying sample composition (Fe-content) and loading (Pt absorber or no absorber, pressure media, etc). These data will allow us understand the observed mechanism for the perovskite/post-perovskite transformation in $(Mg,Fe)SiO_3$ at Earth's D'' conditions.

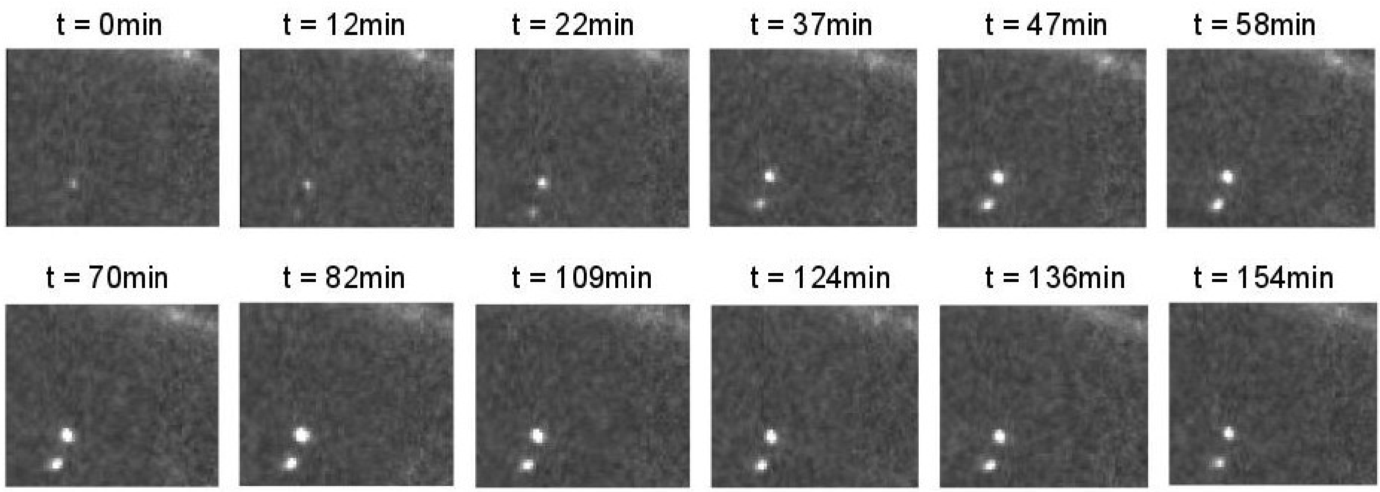

Figure 1: Sample data collected at a single ω angle (12°) and in the same portion of the detector at P and T in the range of ~150 GPa and ~3000 K. Over 150 min, we observe the growth of a single post-perovskite grain. Multigrain analysis will allow characterizing its orientation, crystal structure, and grain size evolution. Possibly, we will also determine its relative orientation to the parent perovskite grain.