Summary Radial Diffraction Experiments ESRF ID9a

Proposal ES-208

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In ES-208 we proposed to measure the differential strains and preferred orientation of $(Mg_{0.8\text{-}9}Fe_{0.1\text{-}2})O$ by means of radial x-ray diffraction in the diamond-anvil cell to pressures as high as 100 GPa and temperatures of above 1000 K at Beamline ID9b. Ferropericlase (Mg,Fe)O is thought to be the second most abundant mineral phase in Earth's lower mantle and thus may influence the rheology of the lower mantle significantly. In particular the effect of iron spin crossover in ferropericlase between 40-70 GPa has not been investigated at simultaneous high-pressure and –temperature. Such data may profoundly influence our understanding of the rheology of the lower mantle.

For the experiments we designed a new vacuum chamber for graphite resistive heated radial diffraction experiments with a modified Mao Bell type DAC that fits on beamline ID9a. During the beamtime we successfully commissioned the vacuum chamber by conducting a series of 4 experiments with the following specifications.

a) Run 1
   T = 1150 K, Pmax = 34 GPa, 0.2 mm culet, cBN gasket => diamonds broke at the highest pressure
b) Run 2
   T = 1120 K, Pmax = 20 GPa, 0.2 mm culet, cBN gasket => diamonds broke at the highest pressure
c) Run 3
   T = 1330 K, Pmax = 1 bar, 0.1 mm bevel culet, cBN gasket => diamonds broke before we could increase pressure because we had to exchange the membrane multiple times
d) Run 4
   T = 1400 K, Pmax = 20 GPa, 0.3 mm culet, amph. B gasket => diamonds broke at 20 GPa

Summary of performance:

Overall the experiments went well considering that this was a brand new setup that we had never tested at the ID9a beamline. In the first two runs temperature stability was reasonable but we encountered large fluctuations when we started to increase pressure. Runs 3 and 4 temperature was higher and in run 4 also stable during P increase. In Run 1, 2 and 4 the diamonds broke at the highest pressure. We have been discussing the reason for the breakeage of the diamond at these relative low pressures. We wanted to go to 70-80 GPa that should be possible with the 0.2 and 0.1 mm culets. The major reason for the failure seems to have been the cBN gasket that did not perform as earlier batches synthesized by L. Miyagi that did not break until very pressures of 70-100 GPa. In further experiments we will try to improve the gaskets and also improve some minor issues with the vacuum vessel such as leaking membranes.