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Iron partitioning between ferropericlase and silicate perovskite: 2D micro-XAS mapping in diamond anvil cell

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Energy Dispersive X-ray Absorption Spectroscopy (EDXAS) is a now a well-established method which has been applied to a broad range of applications. The advantages of an energy dispersive spectrometer, that features no movement of optics during acquisition leading to an enhanced stability of energy scale, spot size and position, combined with a micron sized spot and the option of fluorescence detection, has made it possible to address 2-dimensional mapping with micron resolution on heterogeneous samples, providing full XAS information on each pixel. It is worth noting that due to the absence of mechanical scanning of the monochromator, the spatial resolution is not affected by the energy scan and remains fixed to the dimensions of the probe. In addition, the energy scale is preserved. Moreover, the dwell time per pixel is short enough to make it practically possible to acquire 100 x 100 pixel images in a few hours.

We tested 2D mapping in transmission mode to perform “in-situ” investigations in the diamond anvil cell. Maps of redox and speciation at extreme conditions of pressure and temperature yield information on possible phase transitions and/or chemical reactions that occur at P and T conditions in the Earth interiors. As test sample, we chose a major component of Earth’s transition zone, ringwoodite [γ-(Mg,Fe)$_2$SiO$_4$]. Sample synthesized in large-volume press at 19 GPa and 1700 C from natural olivine ($Mg_{0.88}$,$Fe_{0.12}$)$_2$SiO$_4$ was polished, loaded into the DAC, compressed to desire pressure, and laser-heated. We aquired Fe K-edge XANES maps at different pressures, up

to $\sim$ 40 GPa, before and after laser heating, covering for each map an area of 200 x 200 μm^{2}at 5 μm resolution.

We found that laser heating does not result in re-distribution of iron between heated and non-heated areas. Within precision of measurements there are no detectable changes in iron oxidation state upon decomposition of ringwoodite in to silicate perovskite and magnesowüstite. We also observe that iron preferably partitioning in to ferropericlase.