Texture analysis of binary mixtures of MgGeO$_3$ and (Mg,Fe)O at in situ high pressure and temperature in the resistive-heated radial diamond anvil cell

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Plastic deformation of mantle minerals is the mechanism through which large scale convection takes place in the Earth interior. Different experimental methods furnish information about the deformation behavior of minerals in a wide range of pressure and temperature conditions. However, the conventional deformation devices cannot reproduce conditions of the deep lower mantle [1], whose pressure regimes are easily accessible with the diamond-anvil cell (DAC) [2].

X-ray diffraction in radial geometry in the DAC (RXDAC) is a method that allows one to measure stress levels and lattice preferred orientation in polycrystalline samples compressed to pressures of the very deep mantle [3]. Modelling the measured preferred orientation with self-consistent viscoplastic simulations, allows one to infer the predominant slip systems active in deep mantle environments at elevated stress levels, such as in ultra-deep subduction, where the main deformation mechanism is dislocation creep [4,5]. Different studies based on RXDAC technique have produced new important information about the deformation behaviour of individual minerals of the lowermost mantle at pressures of the D” layer and of ε-Fe at inner core pressures [3,6]. However, these studies were conducted at room temperature. A recent development of the technique is the combination of the RXDAC with resistive- or laser-heating techniques [7,8]. The few available results show that this combination can potentially access conditions relevant to the lowermost mantle.

At the Extreme Condition Beamline ECB (P02.2) of PETRA III we have developed a new resistive-heated RXDAC setup for high pressures – high temperatures (HP/HT) experiments. We have tested it for the first time on a binary mixture of (Mg$_{0.8}$Fe$_{0.2}$)O and MgGeO$_3$. MgGeO$_3$ has a sequence of high-pressure polymorphs, which are isostructural to those of MgSiO$_3$ and are stable at lower pressures than the corresponding silicates. Our plan is that of studying the deformation behaviour at high pressures and temperatures of binary mixture of germanate and oxide as a proxy of the lower-mantle perovskite – ferropericlase mineral assemblage.

Two different experiments were performed on mixtures of MgGeO$_3$ (initially with pyroxene structure) and (Mg$_{0.8}$Fe$_{0.2}$)O ferropericlase in 70:30 mass ratio ground to a particle-size below 5 µm. The samples were loaded in sample chambers obtained by drilling a 80 µm diameter holes in 400 µm diameter B-epoxy gasket of 50 µm thick surrounded by a kapton ring. A Pt flake, few microns wide, was placed on top of the sample at the center, which served as a reference for sample positioning during the experiment and as a pressure (and stress) calibrant. The powder samples were compressed between two 300 µm diameter culets in a Mao-Bell DAC with wide lateral windows (for radial XRD) modified for remote compression with a gas-membrane system available as part of the standard setup of the ECB.

Angle-dispersive x-ray diffraction measurements were performed at high pressures and high temperature at the ECB (P02.2). The energy of the monochromatic X-ray beam was 42.4 keV (corresponding to a wavelength of 0.2922 Å). The measurements were performed at the general purpose experiment where the beam was focused to 8 µm (H) × 2 µm (V) size by means of Be Compound Refractive Lenses (CRL). In this series of experiments the DAC was placed in a new-designed vacuum chamber that is an improved version of that described by Liermann et al. [7]. With the new setup we were able to reach a vacuum of 10$^{-5}$ mbar, which substantially improves the thermal insulation and better preserves diamond and electrical leads from severe oxidation at high temperatures.
We successfully performed measurements of textures up to a maximum of 40 GPa and 600 °C. At these conditions MgGeO$_3$ was not yet converted into its perovskite polymorph. Ferropericlase (Mg$_{0.8}$Fe$_{0.2}$)O presents a typical 001 compression texture consistent with all the available high pressure results at lower mantle pressures (Fig. 1). We collected x-ray diffraction up to peak pressure-temperatures above 45 GPa and 1000 °C. However, at temperatures above 600 °C the Al$_2$O$_3$ insulation layers surrounding the graphite heater failed and obstructed the path for the diffracted X-rays resulting in loss of useful signal from the sample assemblage. We have evaluated the sample assemblage after decompression. The gasket and sample were preserved up to the highest pressure and temperature conditions. These results are promising showing that, after solving few issues with the design of the insulation layers, we will be able to investigate texture development of a binary assemblage of perovskite and ferropericlase at \textit{in situ} lowermost mantle conditions also across the perovskite-postperovskite transition.

![Figure 1: Radial X-ray diffraction of MgGeO$_3$ plus (Mg$_{0.8}$Fe$_{0.2}$)O at 30 GPa and 500 °C. (a) X-ray diffraction image, (b) unfolded image sliced in sections of 5 degrees. The lower panel shows the experimental intensities while the top shows the intensities calculated using a LeBail full spectrum approach combined with stress and texture modeling, (c) inverse pole figures of the compression direction for (Mg$_{0.8}$Fe$_{0.2}$)O.](image)

References