Synchrotron X-ray diffraction of nano-crystalline MgO Powder to 65 GPa

HAUKE MARQUARDT[1], SERGIO SPEZIALE[1], HANS JOSEF REICHMANN[1] AND HANNS-PETER LIERMANN[2]

[1]German Research Center for Geosciences (GFZ), Telegrafenberg, 14473 Potsdam (hama@gfz-potsdam.de)
[2]DESY, PETRA III, Notkestr. 85, 22607 Hamburg

The compression behavior of nano-crystalline materials (crystallite sizes smaller than 100 nm) might significantly differ from the behavior of larger grained materials.

We compressed MgO powder with an average crystallite size of about 20 nm in a diamond-anvil cell. The sample material was loaded either with or without a pressure-transmitting medium. Our results indicate that the crystallite size is preserved in the quasi-hydrostatic experimental run, whereas it reduced to below 10 nm in the non-hydrostatic experiment.

High-pressure synchrotron x-ray diffraction was performed at DESY/PETRA III beamline P02.2 using an energy of about 42.8 keV, a focusing spot of ~2 x 2 μm^2, and a collection time of 30-60 seconds. Up to nine diffraction lines could be detected at high-pressures.

Our results that were collected in hydrostatic conditions indicate that the zero-pressure bulk modulus is slightly decreased as compared to large-grained MgO [1], but both zero-pressure volume and zero-pressure derivative of the bulk modulus are unaffected within the experimental uncertainties. The material that was non-hydrostatically compressed appears to be less compressible compared to the same (starting) material that was quasi-hydrostatically pressurized. The compression behaviour in this run is, however, likely also influenced by a continuous crystallite size reduction and the consequent increase of the volume fraction of intercrystalline material in the sample, that occurs upon compression in non-hydrostatic conditions.

[1] Speziale *et al*. (2001) *JGR* **106**, 515–528.

Focused ion beam cutting of large samples for Brillouin spectroscopy

KATHARINA MARQUARDT AND HAUKE MARQUARDT

German Research Center for Geosciences (GFZ), Telegrafenberg, 14473 Potsdam (hama@gfz-potsdam.de)

Brillouin spectroscopy is a major technique for the determination of elastic properties of single-crystals at high-pressures and high-temperatures. Brillouin scattering at extreme conditions is usually carried out in symmetric platelet forward scattering geometry, which allows for a straightforward evaluation of shear and compressional velocities. However, well-polished platelet samples with parallel faces are required. Mechanical polishing is restricted to materials of sufficient size and mechanical stability. This precludes the preparation of a number of compounds with significant geophysical relevance, including both natural samples and candidate Earth materials that are synthesised at high-pressure/high-temperature conditions (for instance ferropericlase, perovskite).

Here, we show that ion cutting and polishing is a very elegant approach to prepare μm-sized samples of well-defined thickness with high surface quality. It does not expose the samples to mechanical forces, thus allows for preparing materials that are brittle, meta-stable, or show a strong cleavage. In addition, it offers the chance to cut more than one sample from a piece of material, for instance two platelets with different orientation from one single-crystal (fig. 1). A transmission electron microscopy (TEM) foil can be produced simultaneously. This allows for a detailed characterization, including chemical composition, crystallographic orientation, defect structure and secondary phases, of the same sample material, i.e. the same single-crystal, that is later used for optical spectroscopy.

We successfully tested this method on different geomaterials, including perovskite, ferropericlase, spinel, and antigorite.

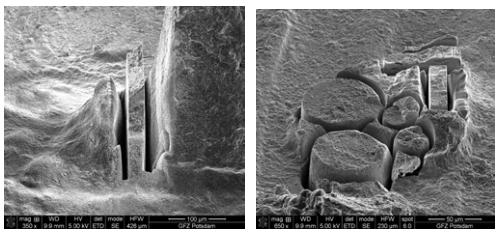

Figure 1: Secondary electron images of a perovskite single-crystal platelet that was cut from an aggregate made up of several single-crystals. The platelet, with approximate dimensions of 180 x 180 x 30 μm^3, was double-side polished (left). The platelet was manually flipped by 90° and again inserted into the dual beam machine (right). Several samples were cut for Brillouin spectroscopy, single-crystal x-ray diffraction and TEM analysis.