ESRF	**Experiment title:** Phase transition(s) in MgO and (Mg,Fe)O at pressures over 500 GPa	**Experiment number**: HC-2829
Beamline: ID16b	**Date of experiment**: from: 02/11/2016 to: 07/11/2016	**Date of report**:
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Report:

Scientific Background

Ferropericlase (Mg,Fe)O is the second most abundant component of the Earth's lower mantle and believed to be a very important phase in the interior of super-Earth planets. The end-member of this solid solution, magnesium oxide MgO, is one of the most studied compounds at high pressures. This simple ionic oxide crystallizes in a cubic B1 (NaCl-type) structure and is stable to at least 300 GPa at ambient and high temperatures. Due to high symmetry of its structure and a lack of phase transitions, MgO is widely used as a pressure calibration standard. A phase transition into a B2 (CsCl-type) phase has been theoretically predicted at pressures exceeding ~500 GPa (see, for example, Cebulla & Redmer 2014). The predicted phase transition Clapeyron slope is unusually steep, i.e. temperature has a relatively small effect on the transition pressure. MgO is a wide-gap ionic insulator, which is expected to be extremely difficult to turn into a metal, as required pressures exceed 20 TPa. Currently, there are substantial discrepancies regarding the position of the B1-B2 phase transition as measured in dynamic experiments (McWilliams et al. 2012; Coppari et al. 2013) and predicted by *ab initio* calculations (Oganov et al. 2003). The phase transition determined from decaying shock experiments of McWilliams et al. (McWilliams et al. 2012) and ramp compression experiments of Coppari et al. (Coppari et al. 2013) suggests that actual transformation pressures are of about 100 GPa higher than those obtained by theoretical simulations. It is worth noting that experimental results were acquired by dynamic methods, such as decaying shock compression and ramp compression, and only the latter provides some structural information based on the appearance of a single additional diffraction peak.

Experimental procedure

The ds-DACs (Dubrovinsky et al. 2012) were prepared in Bayreuth using a small version of the BX90 (weight about 180g that can fit on ID16B). A gasketed version of ds-DACs was employed in order to confine a powder sample in between the two NCD hemispheres (of about 10 microns in diameter) acting as secondary anvils. Two cells were used in the experiment: i) one with three NCD balls pressing on a 2 micrometre rhenium foil loaded in argon gas was used for preliminary tests, and ii) the second one for the compression experiment with a mixture of MgO and tungsten, used as a pressure standard, placed in between the two NCD half-spheres. The size of the 'MgO+W' sample was about 3 μm in diameter and about 1 μm in initial thickness. We used a very small beam at ID16B of about 50nm to obtain X-ray diffraction as well as absorption maps covering the entire sample chamber (10x10 μm^2) at multimegabar pressures. Although nominal size of the beam was 50 nm (FWHM), tails were much larger and we observed quite strong Re diffraction even in the middle of the sample (Fig. 1). A profile across the Re foil in the first cell showed that diffraction rings form the Re disappear about 1 μm away from the rim of such foil. Still, in comparison with any other facilities (different beamlines at APS, PETRAIII, etc.), at which we tried experiments with gasketed dsDACs, the conditions and quality of diffraction patterns collected at ID16b are the best.

Preliminary results

We were able to compress the sample to over 530 GPa in a few steps. As ID16B is not dedicated to high pressure, we spend some time to evaluate the capabilities of the beamline and implement and test procedures to put the sample at the focal point. All our tests and procedure worked, but still the search for an optimum position requires mapping of the whole pressure chamber, and thus the data acquisition for one pressure point takes 6 to 8 hours. We did not notice any significant stresses on MgO or W. At pressures between 400 GPa and 530 GPa B1-MgO transforms into the B2-MgO phase. A manuscript is in preparation describing the results on MgO transition above 400 GPa.

We consider the experiment as highly successful, and several technical advances can be suggested for the future to make experiments with dsDACs at ID16b easier and improve the quality of the results: (a) use of "standard" (50 mm diameter, about 300 g weight) BX90 cells; (b) implementation of clean-up pinhole to reduce the size of the tales of the beam; (c) introducing a procedure which would allow to bring/keep the sample on the vertical rotation axis of the goniometer; (d) installation of high-magnification optics which would allow to visualize sample. The first point is already feasible and will allow us a better control on the pressure steps as well as in he loading procedure. Custom-made pinhole of few micrometres can be manufactured at the BGI using the FIB. The two last points would clearly be assets to perform high-pressure experiments at ID16B.

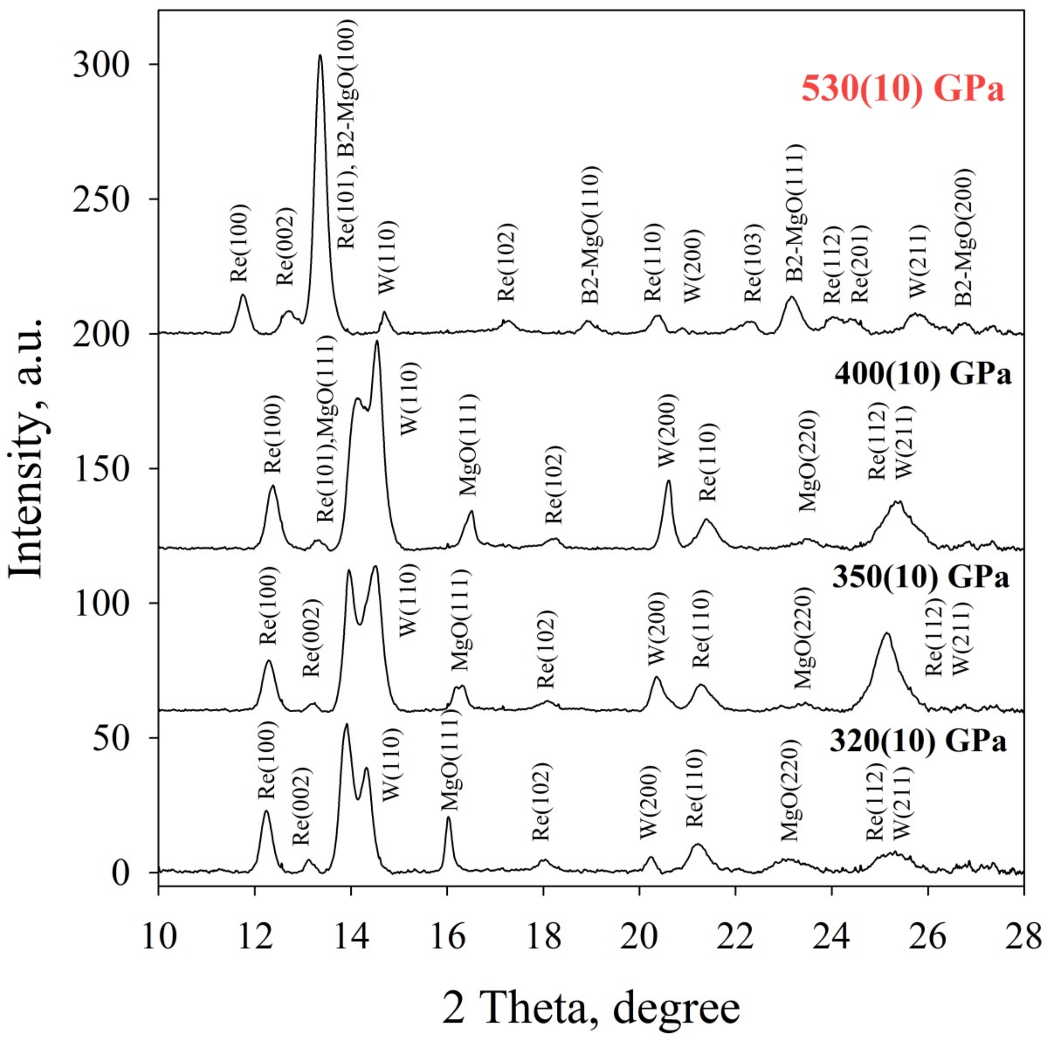

Fig. 1. Integrated diffraction patterns collected on a mixture of MgO and W compressed in ds-DAC at the ID16b beamline at the ESRF.

References

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