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Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** Deformation mechanisms in Phase D at HP-HT by X-ray radial diffraction: Implications for seismic anisotropy in deep subducted slabs	**Experiment number**: **HS-4330**
Beamline:	**Date of experiment**: from: 06 July 2011 to: 11 July 2011	**Date of report**: 05.04.2012
Shifts:	**Local contact(s)**: Wilson Crichton (crichton@esrf.fr)	*Received at ESRF:*

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Report:

Phase D is considered to be the ultimate water carrier in cold slabs descending into lower mantle regions. Knowledge of its deformation mechanisms at relevant high PT conditions is thus crucial to understand global water recycling *via* subduction and the generation of seismic anomalies. Previous studies on the elasticity of Phase D have shown that 16 vol.% of AlFe- Phase D in a hydrous peridotite (containing 1.2 wt.% of water) could provide a plausible explanation for the negative velocity anomaly of 3% in the detached Tonga slab fragment [1]. Due to the high elastic anisotropy of phase D and the favoured alignment of its layered structure it is also considered to be a plausible explanation to the observed shear wave splitting in subducted slabs [2, 3]. The combination of elastic properties obtained from Brillouin scattering experiments and rheological properties obtained from synchrotron radial X-ray diffraction deformation experiments at the ESRF (see experimental report HS-4059) allow to infer the contribution of phase D to the seismic shear wave splitting in subducted slabs [4, 5]. However, previous deformation experiments on phase D have been conducted at room temperatures, because high-pressure and high-temperature experiments are very challenging. Deformation experiments at realistic conditions are however necessary to constrain better the contribution of phase D to seismic anomalies, including seismic shear anisotropy and deep earthquake mechanisms.

During the allocated beam time at ESRF ID09A, high-pressure and high-temperature radial X-ray diffraction experiments on phase D have been envisaged. The setup consisted of a 30x30μm^2 focused monochromatic X-ray beam tuned to λ = 0.416069 Å (29.9 keV). X-ray diffraction (XRD) patterns were collected using a Mar555 large area image-plate detector with a 430 x 350 mm active area located at a distance of 400 mm from the sample. A silicon standard was measured before the experiment in order to obtain precise calibration parameters of the sample to detector distance, detector tilt, instrument broadening and pixel size ratios.

Unfortunately, the commissioned resistive heating diamond anvil cell (DAC) and vacuum vessel for these experiments were not available for the beamtime due to delay in delivery by an external company. We thus attempted to perform the experiments using a standard panoramic cell [6] in which a 5mm thin graphite foil was used as resistive element. Ceramic rings were used as insulating material around the diamonds. Channels for the incoming and scattered X-ray beams were cut into the graphite foil to reduce the background signal. Amorphous boron gaskets of 50 μm thickness drilled with a hole of 80 μm and mounted in a kapton sheet were used. A thermocouple was glued close to one of the diamond tips for temperature determination during the experiment. The cell was continuously flushed with a reducing gas (Ar-2% H_2) to o avoid oxidation of the diamonds, CW seat and other parts of the cell at high temperature. Three high PT assemblies were prepared during the beamtime in this way but the instability under pressure of the assembly resulted in failure most probably due to shortcuts. Problems with the setup could not be solved during the allocated beamtime.

Due to the long preparation time for the high PT setup, we meanwhile successfully conducted high P uniaxial deformation experiments on carbonate samples at room temperature conditions up to 50 GPa. Carbonates play an important role in the recycling of CO_3 into mantle *via* subduction of the sedimentary layer of the oceanic crust. The release of carbon during the breakdown of these phases in the shallow upper mantle (below 150 km) influence essential geochemical processes including melt generation and arc volcanism. The subduction of carbonates to greater depth is discussed as a possible scenario based on mass balance calculation of subducted and out gassed carbonatitic masses [7] and thermodynamic calculations on the high PT stability of carbonates [8]. These calculations have shown that ferromagnesian [$(Mg,Fe)CO_3$] down to lower mantle conditions along geotherms of cold subducted slabs. However, the seismic detection of carbonates in those depths remains difficult due to a lack of data on seismic properties of carbonated lithologies. Knowledge of the mechanical properties of these minerals is thus necessary to constrain the amount of carbon recycled to great depth and to understand seismic observations in deep slabs, including seismic shear anisotropy. Recent single-crystal measurement of the single-crystal elastic properties of carbonates along the $(Mg_{1-x}Fe_x)CO_3$ join (with x = 0,

0.65, 0.95 and 0.99) [9] have shown that carbonates display large shear elastic anisotropy. In order to understand their contribution to observed seismic shear anisotropy in deep labs knowledge on their deformation mechanisms at relevant high PT conditions are needed.

We perfomed X-ray radial diffraction measurments on carbonate samples with composition $MgCO_3$, $(Mg_{0.35}Fe_{0.65})CO_3$ and ankerite in a panoramic DACs using gaskets made of a mixture of cubic boronnitride and epoxy. Sample preparation procedure and experimental setup for angular-dispersive radial XRD measurements has been chosen similar too the experiment HS- 4059. No transmitting pressure medium was loaded with the sample to maximize the uniaxiall stress. The evolution of plastic properties of the sample with pressure have been monitor *in situ* using radial XRD diffraction (exposure time of 10 sec) and increasing the pressure with steps of 3-5 GPa from ambient conditions up to 50 GPa. The pressure standard Au was exposed seperatly for each pressure point and the pressure was determind from the lattice parameter obtained at the azimuthal angle corresponding to hydrostatic conditions for this stetup. The pressure evolution was simutaniously monitored and crosschecked by ruby florescence method after and before each pressure point using a portable laser and spectrometer installed at ID09A.

Upon compression, the developments of strong textures and significant lattice strains in the sample were observed from the intensity variations for each lattice plane (hkl) and the variation of diffraction peak position d(hkl) relative to the load direction (see. Fig.1a-c).

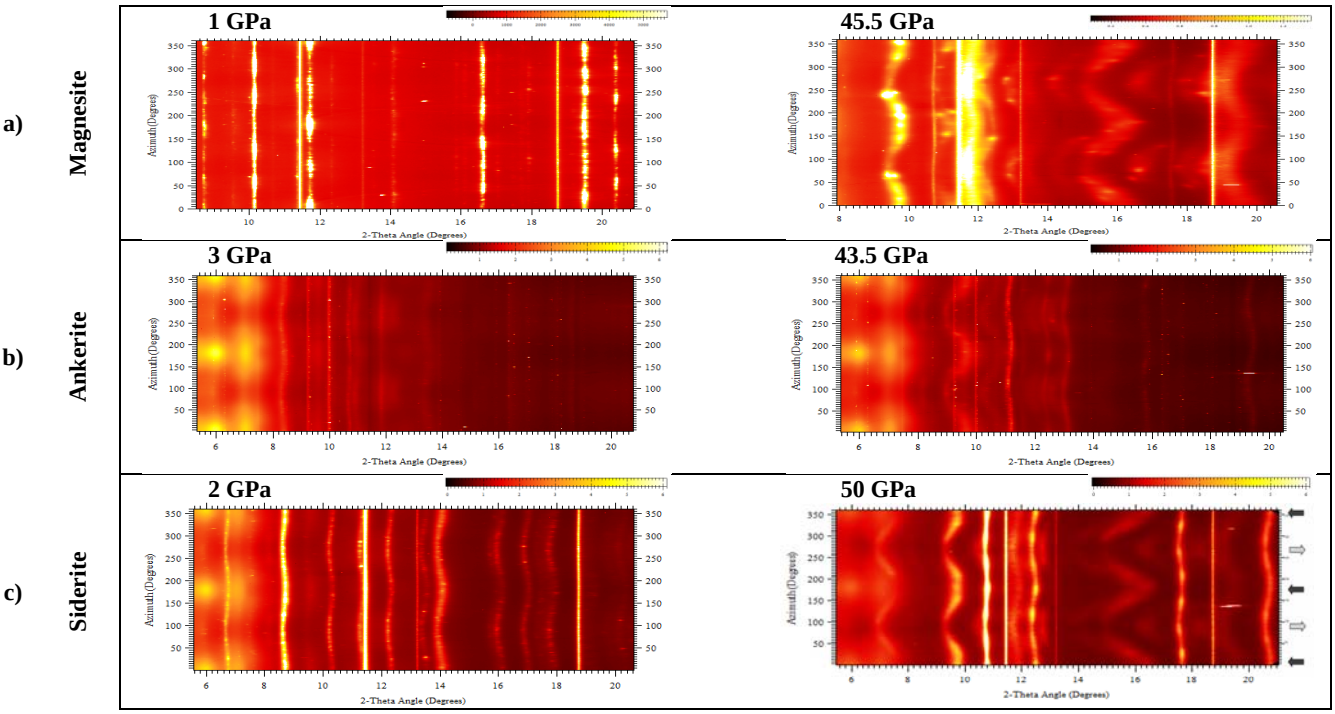

Figure 1a-c. Unrolled radial diffraction images of carbonates at selected pressures (see above each image) collected at room temperature with an exposure time of 10 seconds. The azimuth angle is plotted against 2Theta angle, intensity variations are given by red to yellow shading (scale above the image multiplied by 10*E4). At the lowest pressure (left column) little texturing and almost no strain is observable for each composition from the equal lattice planes (hkl) (Debye rings are plotted as almost straight lines). At high pressures and high degree of deformation (right column) the sample is clearly textured (intensity variation for the equal lattice planes depending on orientation to maximum and minimum stress direction, indicated by black and grey arrows) and becomes highly stressed (variation of 2Theta position for equal lattice planes depending on orientation).

Data processing is currently in progress. The collected diffraction patterns are of high-quality and will allow the extraction of the texture, texture strength, lattice and lattice strain parameters by Rietveld refinement method using MAUD [10]. The strength evolution and the seismic properties of the deformed carbonate aggregates will be calculated using the known single-crystal elastic properties [9] and the observed textures. The results will provide important information on the plastic deformation mechanism and strength properties of these carbonates at pressures relevant for the Earth mantle. Ultimately, these results will allow inferring the plausible contribution of carbonated lithologies to observed seismic anisotropy in subducted slabs and thus will put tighter constrain on the chemical composition on deep subducted slabs.

Given the interesting results already obtained on phase D in experiment HS-4059 [4, 5] and the recent development of a high temperature resistively heated panoramic cell that has been successfully tested at ID 27 [Dr. Sylvain Petitgirad, personal communication], we will apply for an extension of HS-4330 to study the deformation mechanisms of phase D at simultaneous high PT.

References:

[1] Rosa et al., (2012) Geophy. Reas. Lett. 39, L06304; [2] Chen & Brudzinski, (2003) Geophys. Res. Lett. 30, 1682; [3] Di Leo et al., (2011) Phys. Earth Planet Int. 194–195, 38; [4] Rosa et al., in prep. for Earth Planet Sc.Lett.;[5] Rosa et al., (2011) AGU Fall Meeting San Francisco; [6] Liermann et al., (2009) Rev. Sci. Instrum., 80, 104501 ; [7] Dasgupta and Hirschmann, (2010) Earth Planet. Sci. Lett., 298, 1–13; [8] Kerrick & Connolly, (2001) Nature, 411, 293; [9] Sanchez-Valle et al., (2011) Geophys. res. Lett. 38, 24; [10] Lutterotti et al., (1997) J. Appl. Phys., 81, 594.