	Experiment title: High-pressure deformations mechanisms and rheology of serpentine+olivine aggregates using synchrotron X-Ray diffraction	**Experiment number**: ES-82
Beamline: ID06	**Date of experiment**: From: 5 nov. 2013 to: 7 nov. 2013 (preparation) from: 13 nov to: 15 nov 2013 (9 shifts) from: 29 jan 2014 to: 1st feb. 2014 8:00 am (9 shifts)	**Date of report**: 27/08/2015
Shifts: 18 (9+9)	**Local contact(s)**: Jérémy Guignard, Wilson Crichton	*Received at ESRF:*

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Report:

ES-82 was aimed at understanding and measuring the rheology at high pressure of serpentinized peridotite, a rock relevant to subduction zones dynamics.

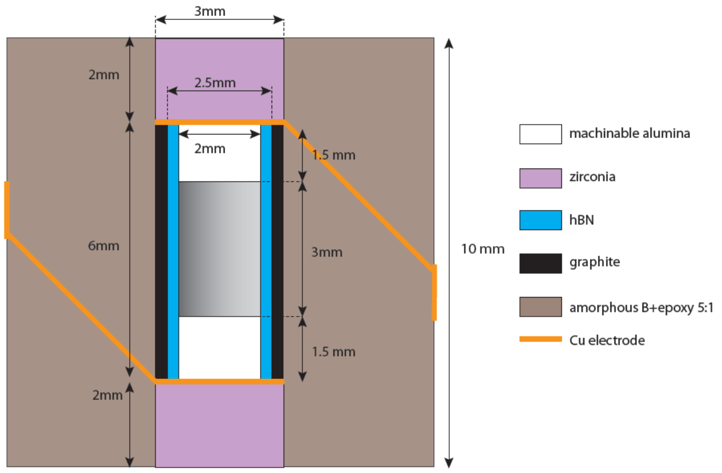
Context: ES-82 and ES27 were the first deformation runs on the large volume press on ID06. Our team shared two beamtimes in nov. 2013 and feb. 2014, dedicated to deformation at high pressure in the large volume press (ES-82 and ES-27). Some technical developments have been realized during these two beamtimes.

Several days prior to the beamtime were necessary to machine and load the high pressure cells (5th to 8th november).

Technical progresses :

- ❑ Our optimal design at the end of the beamtime is presented in fig 1.

Fig.1 Optimized cell design tested up to 30 tons and 1300°C (170W) then 80 tons 150W. The main difference with the usual multi-anvil assemblies is the furnace connection : Cu electrodes, at 45 degrees of the deformation/main compression axis, reach the lateral anvils that are connected to the power supply. This design was adpoted after multiple failures, where "straight" Cu electrodes were



pinched between anvils during compression or at the beginning of deformation.

- ❑ Several shifts (3 or more) were used to set up running conditions (e.g. alignement and optimization of the diffraction conditions: e.g. press rotation to maximize anvil gap, linear detector rotation speed).
- ❑ A calibration has been realized. The temperature vs. furnace power during heating, and pressure vs. load at ambient T during compression, have been determined for the cell design in fig. 1 by using a Au+NaCl+hBN calibrant. The results for the best cell design are presented fig. 2. Deformation has been tested on a range of velocities (please see experimental result below).
- ❑ S. Merkel integrated specificities of ID06 LVP setup into his analysis software for lattice strains and stresses, available freely at http://merkel.zoneo.net/Multifit-Polydefix/
- ❑ At the end of ES82 and ES27, we were operating smoothly on the beamline, had a functional high pressure cell and a software ready for analysis.
- ❑ At that time, improvements to be made in 2014, that required more beamtime or offline work on the LVP were :

- automatic data acquisition for diffraction
- automatic switching between image and diffraction
- on the software for controlling the large volume press (interface not user-friendly even for experienced users)
- cell design still to be optimized because complex (prone to failures) and time consuming
- consumables: a reasonable number of anvil sets needed to be available.

Serpentinites deformation experiments:

The runs carried out during this project are listed in the following table.

run	main load (bars)	max power (W)	deformation	comments
Run1	20	70	y	Test deformation speeds and T profile
Serp_01	30	20	y	cored peridotite 1 (serp 1%)
Serp_02	30	37	y	cored peridotite 2 (serp 15%)
Serp_03	30	50	y	cored peridotite 2 (serp 15%)
Serp_04	28	n	n	oven contact failure during compression
Serp_05	30	70	y	cored peridotite 1 (sep 1%)
Serp_05	30	70	y	cored peridotite 1 (sep 1%)
calibration	30	190	n	T calibration with NaCl + Au flakes

The first run was aimed at testing the cell efficiency, that is the deformation speed achievable with the differential rams, using radiography, and the temperature profile in the column using cross calibration. The whole column was made of crushable alumina pistons and gold.

Five over six runs (Serp_01 to Serp_05) with cored serpentinites were carried out successfully. The experimental stacks contained San Carlos olivine as a reference for strain rate and the sample to be investigated. The samples were deformed under pressure and temperature in pure shear.

The strain was measured by taking a series of pictures (about 0.5 mm height) scanning the whole column length (3mm), which were put together using a macro in Fit2D. After optimizing the press rotation in order to maximize the anvil gap on the diffraction side and therefore the 2-theta coverage, the diffractions were taken by rotating a linear detector centered over the direct beam. This typically required several minutes during which the images could not be collected (see gaps in strain measures fig. 2).

Fig. 2. Typical deformation (strain) mesured over time with correponding diffractions. The peridotite containing 1% serpentine deforms four times faster than the San Carlos olivine polycrystal and this is not strain rate dependent in these experiments. Stresses and preferreod orientation acquired during deformation still have to be analyzed.