Report on proposal ES27 "Effect of pressure on Orthopyroxenes Rheology"

The P.I. (Paul Raterron) has been awarded 18 shifts at beamline ID06-LVP (C05), which were allocated in February 2014. Although proposal ES27 was focused on Earth's upper-mantle materials rheology, beamtime was awarded "*to help commissioning [deformation] experiments*" in the large volume press (LVP) that equipped the beamline. The P.I. is also an associate investigator of proposal ES82 (serpentine deformation, P.I. Nadège Hilairet), which was allocated beamtime in November 2013. Since this past November, our team has thus been working, with the help of Wilson Crichton and Jeremy Guignard, on designing a proper deformation cell assembly for the LVP at ID06, developing the corresponding analytical tools (diffraction and imaging protocols) to measure the applied stress and materials strain rate during deformation, and using it to investigating mantle materials rheology.

The goal was achieved: high-pressure (*P*) high-temperature (*T*) deformation experiments were successfully carried out in the LVP at ID06. I summarize below our technical accomplishments as well as preliminary rheological data obtained on two critical mantle materials: olivine, Earth's upper mantle main constituent, and its high-*P* polymorph ringwoodite, a constituent of the Earth's transition zone.

Technical and analytical developments – Figure 1 shows a schematic cross section of the deformation cell assembly we designed. The particularity of the D-DIA module at ID06 is that the furnace, which needs to be vertical for deformation (vertical compression), is powered by lateral (horizontal) anvils. We tried several designs with horizontal connectors, which were not reliable: at high *P* and/or high *T* the metallic contacts were squeezed within the forming gaskets, resulting in a power failure. The solution is to place the contact in the centre of the cubic pressure-medium faces, with connectors at 45° angle (Fig. 1). The new cell assembly was tested and is reliable to temperature in excess to 1400°C and, at high temperature, to pressure in excess of 5 GPa. Improvements are planned, with Pt connectors (instead of the present Cu connector) to achieved higher temperature. As is, a couple of *T* calibrations were obtained - one with a thermocouple to 300°C, the other with standard materials to *T* in excess of 1400°C (proposal ES82). The vertical *T* gradient within the cell will be estimated from the diffraction data collected in alumina, when analysed (e.g., Raterron et al., 2013). This D-DIA cell assembly offers several unique advantages, among which: i) its size, with a specimen chamber 3-mm high and 2-mm wide, the biggest available worldwide, which will allow better controlling deformation conditions (e.g., double capsule for gas fugacity control, etc.); ii) its thermal efficiency, likely due to the zirconia end plugs - no direct contact between furnace and anvils - which will likely correspond to a relatively homogeneous *T* (to be analysed).

For imaging of the compression column (piston and specimens, about 5-mm at *P*) with the available 1x1 mm^2 beam cross section, a vertical scan command was implemented by the beamline scientists which, coupled with a macro in Fit2D, allows recomposing the image in less than 1 min (Fig.2, top). This time frame is appropriate for the characteristic strain rate of the D-DIA (typically ~10^{-5} s^{-1}). The obtained images can be accurately quantified leading to accurate strain-rate measurements (Figure 2, bottom). To measure the applied stress during deformation, diffraction is collected using a line detector rotating 360° in less than a min, again an appropriate time frame. Examples of typical diffraction rough patterns are showed in Figure 3.

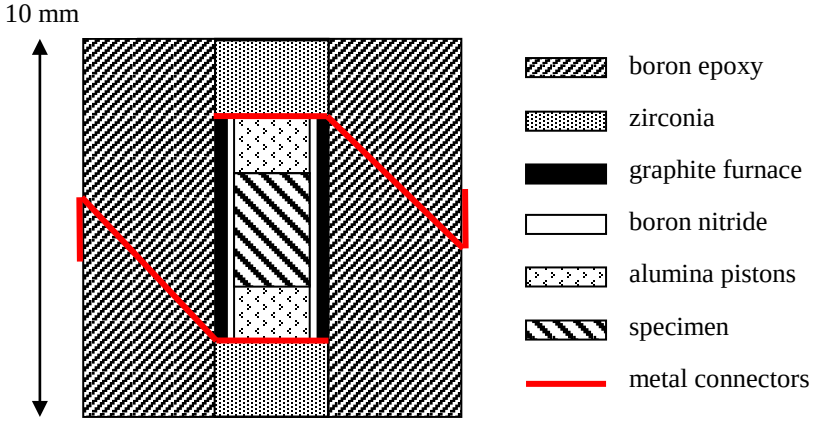

Figure 1: D-DIA Deformation cell assembly, as developed and tested during this cycle. The furnace is powered by lateral (horizontal) anvils. Note that the contacts are in the centre of the cube faces to prevent squeezing them within gaskets (and braking them) during compression, heating and/or deformation.

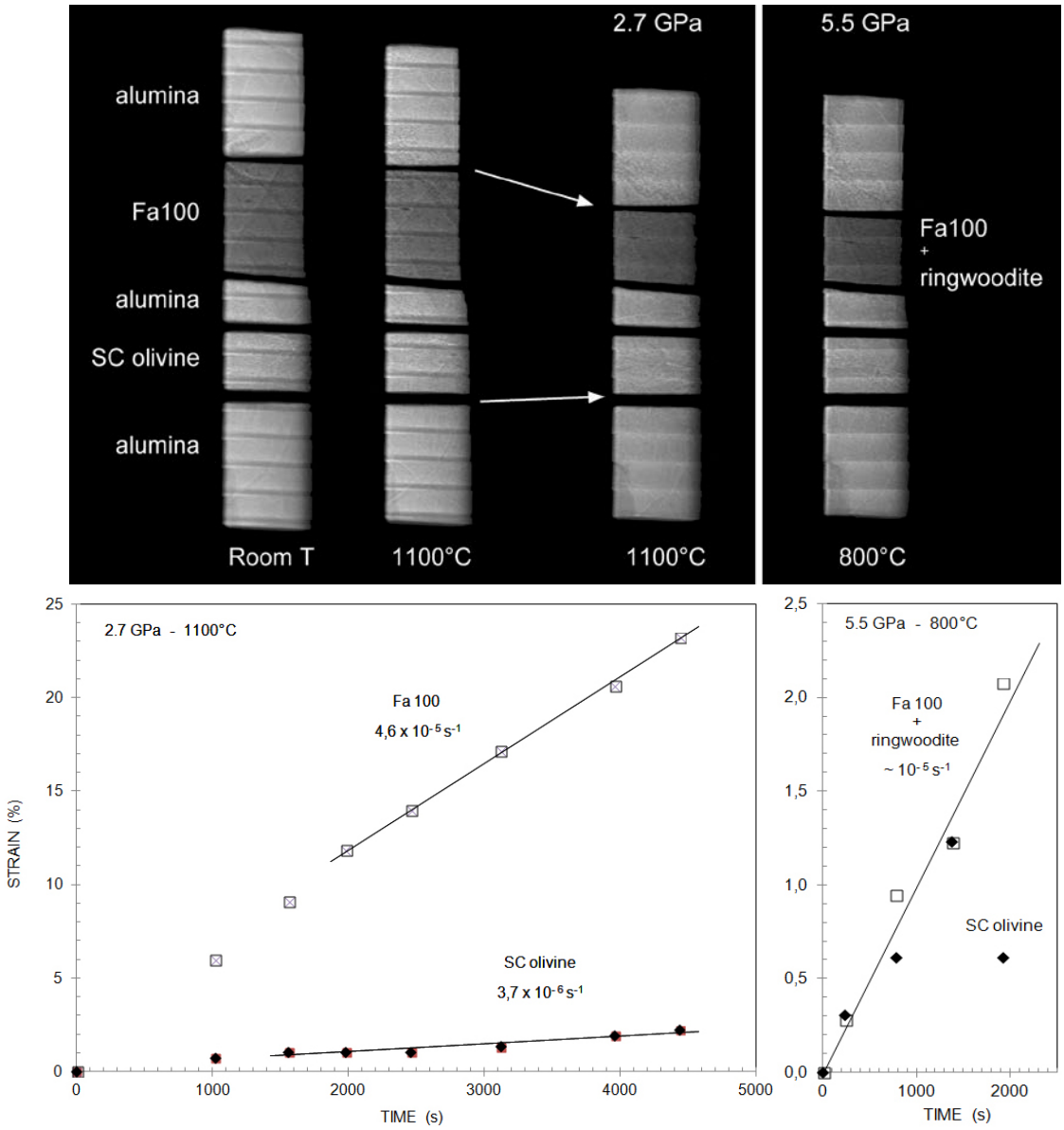

Figure 2: Top, x-ray radiographs of D-DIA compression column as seen through the front-anvils gap (~1 mm wide) after compression and during deformation at the indicated *P* and *T*. The image is composed of a series of 24 images (1x1 mm^2) recombined together. The acquisition time is < 1 min. Note the (dark) Re foils indicating specimen ends.

Bottom: specimen strain vs. time curves during the deformation episodes showed above; the slopes (values indicated) are specimen strain rates (indicated). Note that, at 2.7 GPa and 1100°C, steady-state conditions of deformation where achieved. See text for further explanation.

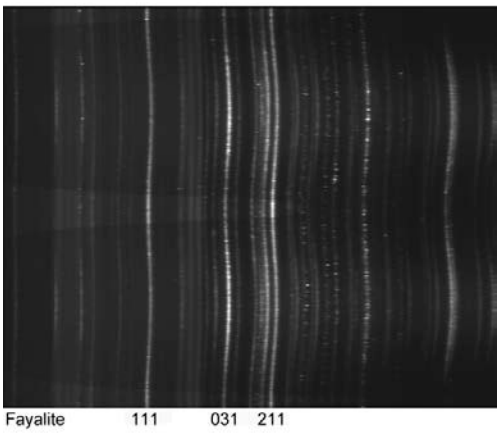

Figure 3: Fayalite (α) raw diffraction spectrum (55 keV) obtained *in situ* after deformation at 2.7 GPa and 1100°C, and quench at room *T*. The x axis is the diffraction angle θ (3 hkl lines indicated). The y axis is the azimuthal angle (ψ) with the horizontal directions at ψ = 0° in the middle of the image, and at ψ = ±180° (top and bottom). The high stress in the specimen results in wavy lines, with smaller *d*-spacing (higher θ) along the vertical compression direction (ψ = ± 90°). Stress is quantified from *d*-spacing measured at different azimuthal directions, knowing the elastic constants of the material (e.g., Raterron and Merkel, 2013).