Discussion Summary

"Workshop for Extreme Conditions Research in a Large Volume Press (LVP) at PETRA III"

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moderated by E. Weckert
and summarized by H.-P. Liermann

Introduction by Edgar Weckert

The presentations during the workshop have provided enough information for a good scientific case for a LVP instrument at the extension of PETRA III. Aim of this discussion is to collect some more information and ideas from the user's community. Detailed discussion of technical issues including potential constraints imposed by other instrumental options in this beamline will follow at a later stage in smaller expert groups.

Science Case

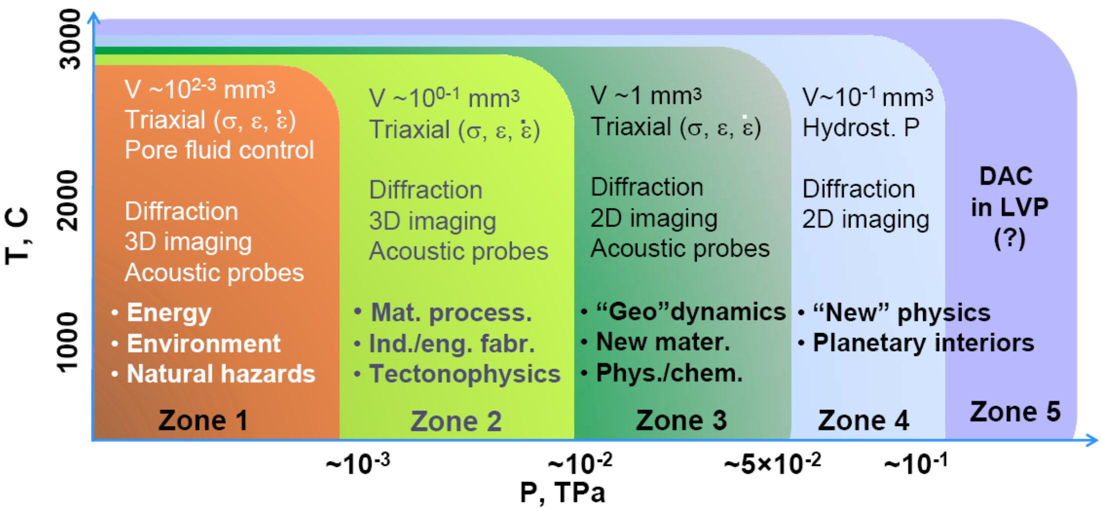

Figure 1: Pressure Temperature Diagram that indicates the different techniques that can be explored with in the different LVP devices. Curtsey of Y. Wang from GSECARS, The University Of Chicago, USA.

The techniques and pressure temperature regimes described within zones 1 - 4 can be accommodated with a double stage multi-anvil device. Zone 5 could be achieved in the future and needs to be explored. While all zones can be accommodated with a flexible double stage multi-anvil device by using different modules (see example from GSECARS, APS), the community felt that one should focus on zone 2-3 because there is a large material science community in Germany that would make use of such an instrument and explore zone 4. In addition smaller devices such as the Paris Edinburgh Press (20-30 GPa, green zone) could be accommodated as additional instruments within the hutch.

Pink vs. Monochromatic Beam

- ***Pink beam*** for energy dispersive diffraction is an important tool because one can separate the sample well from the surrounding pressure medium. Even with a small bandwidth one could achieve sufficient coverage of reciprocal space if one uses multiple energy dispersive detectors that are positioned at different 2theta angles (T-Cup design from NSLS). A pink beam spectrum with 45 – 120 keV is possible when using a normal Pt coated mirror. The spectrum will have a slice (~1e^{-3}) cut out of the energy spectrum where the pink beam monochromator for the side station will be operating.
- ***Monochromatic beam*** for angle dispersive diffraction is very attractive because it does offer much higher resolution in comparison to the energy dispersive techniques. Thus, one would be able to resolve phase transitions (both in geophysical and materials science applications) more rigorously. Nevertheless, the resolution should match the divergence of the beam, which could be the limiting factor for the resolution when the beam is focused (match energy resolution, divergence and sample detector distance). The new instrument should offer both:

> a) Monochromatic beam for angle dispersive diffraction with a low resolution but high flux, by using an imperfect Laue crystal and
> b) Monochromatic beam for angle dispersive diffraction with a high resolution with lower flux by implementing a high resolution Laue diffraction crystal.

Option a) would work for most of the experiments whereas b) would be for special applications. The energy of the monochromatic beam should range from 45 – 150 keV (above that energy little is gained due to almost constant inelastic Compton scattering) with a core energy range of 45 – 100 keV. One should be able to switch between different energies rather quickly, i.e. use a fixed exit monochromator. The power and heat of the monochromatic beam should be adjustable so that sensitive samples (e.g. mercury bearing samples) are not altered during the experiment. Energies below 45 keV cannot be provided because the power and heat of the damping wiggler beam will be too high for the optical components such as monochromator and mirrors to survive.

X-ray Techniques

a) ***Powder diffraction*** should be the primary focus of a LVP instrument. The incident beam size will depend on the x-ray technique. For energy dispersive diffraction a beam of 0.2 x 0.2 to 0.01 x 0.01 mm^2 (for very high pressure experiments) will be needed. In the case of angle dispersive diffraction the standard beam size could be somewhat larger than in the energy dispersive mode, i.e. closer to 0.2 x 0.2 mm^2. Nevertheless, for very small samples at very high pressures we will need also a 0.01 x 0.01 mm^2 beam. There is one more problem that needs to be considered. For angle dispersive diffraction one will need a Soller Slit system made of tungsten especially if one likes to discriminate between low Z elements such as in silicates and the pressure cell materials. At high energies this slit system will have to be rather thick and consequently costs will be significant.

b) ***Single crystal diffraction*** is a developing monochromatic technique that does require the rotation of the press to measure a sufficient amount of reflections. Nevertheless, this should not be the focus of the LVP instrument but rather a potential development project.

c) ***Radiography for imaging*** of the sample e.g. during viscosity measurement, should be a standard technique that complements the x-ray diffraction techniques. The incident beam size for the monochromatic and pink beam will be very similar, i.e. 2 x 2 mm^2. This is also a size that can be provided according to current simulations. The flux of such a beam does not need to be very high. The energy in the monochromatic case should be as low as possible, i.e. 45 keV in order to maximize the contrast.

d) ***Tomography*** is a technique that requires a large volume and hence the achievable pressure will be rather low (zone 1). It will also require a separate much smaller instrument (press) that could be easily moved to different beamlines and thus should not necessary be part of the new LVP instrument, at least at first. The incident beam size (monochromatic and pink beam) for this instrument should be much larger then for imaging the sample via radiography, i.e. more like 3 x 3 mm^2. Nevertheless, at energies above 45 keV the tomographical images will show very little contrast and thus it might be wise to possibly implement such technique (since it is portable) on a dedicated tomography beamline.

e) ***Spectroscopy (Fluorescence, XANES and EXAFS)***

   a. Fluorescence edges that can be effectively activated must have a rather high energy because the incident beam spectrum starts at 45 keV (e.g. Fe can not be excited). Nevertheless, the K-edges of rare earth and heavy elements can be excited and this would make fluorescence a nice additional tool for looking at rare earth and heavy oxides that are of significant interest for the material science community.

   b. XANES and EXAFS could be used in a similar way, e.g. for the determination of the oxidation state and the chemical neighborhood of a rare earth or heavy element oxide.

   In any case the application of such techniques will require more thorough evaluation, i.e. a selection of the elements of interest and the science case behind it. Only then should one start thinking about the incident spectrum and/or resolution of the beam. A beam size of 1 x 1 mm or smaller should be sufficient for these types of experiments.

f) ***Rheology*** experiments are important and the community has been growing (e.g. 50% of the users at the NSLS LVP instrument conduct Rheology experiments and half of them are coming from Europe). The technique will require a D-DIA press that is operated in either energy dispersive (using multiple detectors in a T-CUP configuration with multiple detectors) or angle dispersive mode.

Focusing

Angle dispersive as well as energy dispersive powder diffraction experiments will require a small beam that can probe small portions of the sample in the pressure cell assembly. This will reduce the background and potential temperature/pressure gradients in the illuminated part of the sample. Slitting down the beam will probably be insufficient because there will be too little flux on the sample to get a good diffraction image and thus, focusing might be required. Latter is difficult to accomplish at a damping wiggler section that consists of multiple sources. However, one can create a secondary source by only using the last wiggler (contributes to 15 % to the overall spectrum) and focus it. In the ideal case one would be switching from unfocused beam for radiography to a focused beam by moving Compound Refractive Lenses (CRLs) in and out of the beam. However, this of course only works for monochromatic beam at a discreet energy. For pink beam one needs to implement mirrors such as a Kirkpatrick-Baez system. Nevertheless, before thinking further about this subject one should evaluate if the focused beam size of 0.2 x 0.2 (0.01 x 0.01) mm from the last wiggler will offer more flux than the unfocused beam of the entire wiggler section slit down to the same size.

Large Volume Press and location of the LVP instrument

The press that is located at DORIS III could/should be modified to be used at the extension of PETRA III. The details of such an undertaking should be worked out by an expert group and the results presented at a later workshop. The location of the press should be at the end of the wiggler beamline to allow offline operation and in order to prepare an experiment prior to receiving beam. The size of the experimental hutch should be sufficient to accommodate the press and the detector support. Switching of the beam from the front stations to the end stations should be rather quickly to make maximum use of the beamtime.