PETRA IV Workshop: Earth, Environment, and Materials for Nanoscience and Information Technology

Session: Extreme Pressure & Temperature Research

In this session there were 6 invited talks covering the core Scientific Instrument Proposals (SIPs) for extreme high-pressure and temperature research at PETRA IV. The maximum number of participants was around 80. In chronological order, the speakers were Dr. Melanie Sieber (University of Potsdam/GFZ), Dr. Clemens Prescher (University of Freiburg/DESY), Dr. Ingrid Blanchard (University of Potsdam), Prof. Christian Sternemann (TU Dortmund), Dr. Ilya Kupenko (University of Münster), and Prof. Dominik Kraus (University of Rostock).

The first speaker (Dr. Sieber), discussed the SIPs for research and X-ray techniques using Large Volume Presses (LVP) at PETRA IV. The five other speakers discussed SIP concerning research and X-ray techniques using Diamond Anvil Cells (DAC) to create extreme conditions of P and T. Each contribution from the speakers is described in more detail below.

I. In situ rock deformation and liquids under extreme conditions - the value of specialized LVPs at PETRA IV (M. Sieber)

a. *A general overview of the talk*
   - In situ rock deformation requires a (6-ram) LVP with differential actuators. Thus, using AD-XRD and imaging are required to record lattice micro-strain (stress) and macroscopic strain for studies of mantle dynamics and mechanical behaviour of materials.
   - Liquids under HPT conditions. Melts and aqueous fluids in the Earth. Requires in situ measurement of viscosity, structure, melting point/melt propagation. Some liquids are unquenchable. Precise PT control needed for long duration experiments.
     - Example: In situ determination of melting of carbonates at P61B.
     - Example: X-ray absorption imaging/tomography in opposing-anvil-type presses (Paris-Edinburgh, PE-LVP) to study rhyolite melt percolation through the mantle. Compressibility of basaltic glass with tomography to obtain volume information and calculate density and bulk modulus.
     - Liquid phase separation without absorption contrast, resolved with phase contrast imaging at high PT (needs coherence!).
   - 6-ram LVP (Hall-type LVP)
     - Structural characterisation & phase transitions
     - Axial deformation
   - PE-type LVP (opposing anvil type LVP)
     - Structure, density & viscosity of liquids
     - Torsional deformation / attenuation
     - In situ imaging of evolution of microstructures and phase distribution
   - Uni-axial DIA-type press
     - Expand studies to UHP (> 60 GPa) and at high T (>3000 K) exclusively using ED-XRD (if possible).

b. *Scientific and technical requirements for PETRA IV*
   - Improvement in stress and strain determination in situ.

- Opposing-anvil press (e.g. PE-type) for radiography/phase-contrast micro-tomography to 180 or360 deg. High flux, low exposure times with large beam imaging).
- Opposing-anvil press (e.g. PE-type) for computed X-ray diffraction/scattering tomography (CT) using focused micro-beams (< 1 um^2). PETRA IV is expected to improve acquisition times from 8 hours to 1 hour. Rapid sub-second XRD.
- Highly coherent/low divergence monochromatic beam, high flux, good energy resolution down to $\Delta E/E = 10^{-4}$, sensitive large-radius 2d detector, small (focused) beams (1-200 um) for diffraction and large beam for imaging (~2x2 mm, or more). High energies > 40 keV – 100 keV.
- Fast data transfer and high storage capacity. On the fly data pre-processing e.g. for computed XRD tomography.

c. *Special needs for on-site infrastructure (Preparation Laboratory, NanoLab, others)*
   - As mentioned in first workshop. Carry over current resources to the new BL.
   - Possibility for installation of an offline synthesis press with rotation capability (?)

d. *Important scientific or technical discussions*
   - Out of time/no urgent questions

II. Modeling and understanding of planetary interiors by in-situ high pressure and high temperature XRD and X-ray imaging (C. Prescher)

a. *A general overview of the talk*
   - SIP intended to be a 'spiritual' successor of the current ECB (P02.2).
   - Study of Earth materials – main driver of HP research
     - Obtaining accurate Equation of State of materials to high PT.
       a. Determine composition and seismic wave propagation in Earth
     - Study of the Fe-O system – new structures, polymorphs at UHP
     - Carbonates in deep Earth: FexCxOx, many new compositions at high P
     - Dehydration of hydrous materials: glaucophane decomposition –>> deep-focus earthquakes/ water transport.
     - Formation of Earth: Liquid Fe and Fe alloys.
   - Achievement of Ultra-high pressures > 200 GPa requires sub-micron/nano-size X-ray beams.
   - Imaging in DAC using Phase Contrast Imaging to observe melting and freezing of melts. Addition of other new imaging techniques.
   - Deformation in the DAC: measurement of lattice micro-strain e.g. in (Mg,Fe)O. Add imaging to obtain macrostrain information.
   - Phase transformation kinetics: Bridgmanite to post-perovskite transition (at core-mantle boundary). Add tomographic imaging techniques to observe phase nucleation and grain growth. Addition of laser heating. Easier sample alignment with imaging.

b. *Scientific and technical requirements for PETRA IV*
   - Improved focusing to nano-beams (for UHP studies).
   - Single crystal XRD (low-Z) to 200 GPa / (high-Z) to 500 GPa
   - High-energy XRD on low-Z / amorphous materials, larger Q-range, more flux
   - Improved (transverse) coherence to enable new imaging techniques (at high E).

c. *Special needs for on-site infrastructure (Preparation Laboratory, NanoLab, others)*

- See Kai's question below. Of course, FIB access is paramount. A 2nd FIB on an SEM is seriously needed to meet all the BL demands.

d. *Important scientific or technical discussions*
   - Kai: How does it work to use many techniques simultaneously? Clemens: Combination of XRD and imaging (fast and at same E) is possible. HP: all you need is to change the focus / sample position change.
   - Kai: Labs and infrastructure needs? Clemens: Yes, we need DAC preparation lab, drilling, Raman lab,... Transfer and upgrade of current facilities.

III. Shaping the future of extreme condition science for the Earth and Environment: a unique synergy of diffraction and spectroscopy at PETRA IV (I. Blanchard)

a. *A general overview of the talk*
   - Core & mantle boundaries phase transitions and compositions to explain ultra-low velocity zones. Studies using XRD in DAC (with deformation).
   - There is still a lot of work to be done in order to untangle multiple inconsistencies, application of multiple techniques to the same sample at the same conditions – is important.
   - XRD to determine structure, plus in situ spectroscopy & imaging for full characterisation:
     - XAS (EXAFS & XANES)
       a. Element-specific short-order investigations
     - SAXS/WAXS
       a. Topological heterogeneities of crystalising amorphous systems
       b. Element-dependent particle growth, average interatomic distances
     - XES/rXES+XRD
       a. Determination of valence states
       b. Determination of spin states (magnetic moment) of trans. elements
       c. Local coordination (valence to core XES)
     - XRF (in situ and ex situ)
       a. Trace level concentration
       b. Fluorescence tomography
     - X-ray imaging with XRD, XES and XANES
       a. E.g. low/high spin mapping
       b. Full field mode (phase/absorption contrast) and transmission X-ray microscopy across absorption edge (XANES)
   - Additional lab techniques: Nuclear Magnetic Resonance (NMR) / resistivity measurements /visible light spectroscopy (Raman, Brillouin, UV)

b. *Scientific and technical requirements for PETRA IV*
   - State-of-the-art X-ray spectroscopy, diffraction and imaging at same PT conditions possible for 1st time
   - Most X-ray beam characteristics can be adopted for many of these techniques simultaneously (calcs).
   - State of the art detectors (Varex, Dectris)…

c. *Special needs for on-site infrastructure (Preparation Laboratory, NanoLab, others)*

- High-T generation for resistive and laser heating
- Conventional offline IR, IV, visible light spectroscopy
- DAC preparation lab space: gas loading, NMR prep, CVD dep, FIB machining, etc.
- Magnets (1 T and 7 T)

d. *Important scientific or technical discussions*
   - Andreas Zerr: Some confusion about 3D phase mapping for energy and rotation angle, and what the resolution of each slice is. Max Wilke: Full field imaging with energy scanning. The exact example – spatial through full field with energy and sample rotation slices. Glazyrin: The specific example was not in DAC (2011), but with new development we should be able to do some rotation in DAC at high P (e.g. 100deg aperture) and extract some useful information.

IV. Electronic structure and bonding properties of light-element compounds at conditions of planetary interiors (C. Sternemann)

a. *A general overview of the talk*
   - XRS methods
     - Structure of SiO_2 glass by XRS (e.g. up to 100 GPa in a-SiO_2 at ESRF ID20).
     - Oxygen K-edge of hydrothermal fluids (NaCl/ H_2O fluids)
     - Electronic structure via XRS /determination of partial unoccupied DOS
       a. P and T induced electronic transitions of light elements
   - X-ray Raman scattering at Mbar with laser heating - promising
   - Spin-state imaging
     - K_β emission of siderite at P01 beamline, 8 µm spatial res. However, optical Raman offers 1-2 um spotsize. In situ XES at 70 GPa/2300 K.
   - Resonant X-ray emission (RIXS) possible.
   - P01 set up: XRS spectrometer with 12 spherically bend analysers, von Hamos spectrometer (XES), laser heating.

b. *Scientific and technical requirements for PETRA IV*
   - (sub)µm-focus and transparent DAC gaskets
   - 2D spatial imaging (XES) promising with small sub-µm focus and high intensity / combine with XRD. Add 2 more XRS spectrometers & XRD detector.
   - Complete solution: XRS/XES/XRD for mapping distributions in DAC with laser heating.

c. *Special needs for on-site infrastructure (Preparation Laboratory, NanoLab, others)*
   - Not discussed here (see previous talk).

d. *Important scientific or technical discussions*
   - Peter: Can XRD be combined with other techniques at low energies? Sternemann: not state-of-the-art XRD but a few peaks will offer valuable additional information.

V. Scientific Instrument Proposal for Nuclear Resonance Scattering instrument at extreme conditions in the field of Earth Sciences (I. Kupenko)

a. *A general overview of the talk*
   - P01: 15% research for DAC (extreme conditions research). Useful to study magnetic and electronic properties, magnetism, and spin and valence states.
   - Smaller samples at higher P, requires smaller intense beams.
   - Distribution of carbon in Earth (carbonates)

    - FeCO3 (siderite) compression & heating – high-spin to low-spin state-carbonates with new UHP structures.
- Magnetic carriers (Fe2O3) and (Fe3O4), locking of hematite magnetism (Neel temp at different P and T). Paleo-tectonic reconstruction.
    - Subducted banded-iron formations as source of ultra-low velocity zone at core-mantle boundary – discovery of new FexOx structures at UHP (>200 GPa), multiple coexisting phases. Need smaller beam to probe single phases.
- Electronic structure of Fe-bearing amorphous silicates (magma Earth). Magmas were denser or buoyant in early molten Earth? Study of amorphous glasses in DAC – density measurements, spin state measurements.
- Density and velocity deficit in Earth's core.
    - Earth's core requires light elements to explain its density. Carbon a possible candidate, estimate sound velocities and density of iron and Fe7C3 under UHP and UHT. Currently only possible up to 150 GPa, must go higher P.
- Development of 2-stage DAC for UHP, explore other, larger superEarths and gas giants.

b. *Scientific and technical requirements for PETRA IV*
- Highly focused (0.1x0.1 um^2), intense beams allow studies of magnetic transitions in Fe-oxides to UHP and UHT and study heterogeneous samples.
- P IV allows study of more dilute systems (melts, amorphous glasses) expected in natural environment to study density, and composition, spin-state, etc.
- Add synchrotron Mossbauer source, optics with focusing mirrors.
- Easier data processing with complex systems.
- 7-fold higher flux in timing mode, 16x more in brightness mode
- Time-resolved measurements, explore time resolution of detection and systems (e.g. 14.4 keV 57Fe decay with half-life of 98 ns).
- Timing with pulse laser heating. Several angstroms displacements in sample. Vibration spectroscopy with sub-micron beams.

c. *Special needs for on-site infrastructure (Preparation Laboratory, NanoLab, others)*
- Not further discussed.

d. *Important scientific or technical discussions*
- Glazyrin: Incorporate imaging with these techniques (SMS)? Kupenko, did not consider until now. Could be possible at 14 keV? Flux might not be sufficient for this SMS imaging even at PETRA IV.
- Zerr: Time-resolved 'vibrational' spectroscopy was already done at high P, except not at synchrotron source.

VI. Mimicking planetary interiors and violent impacts by dynamic compression experiments (D. Kraus)

a. *A general overview of the talk*
- Dynamic compression can generate extreme pressures not possible by ordinary techniques to study superEarths and gas giants' interiors. – TPa and 10,000 K
- Impacts (meteor), high velocities and UHP. Shock wave propagation. Moon formation by giant impact of Mars-sized body on proto-Earth.

- Two drivers: dDAC (strainrates: $10^{-3} - 10^{3}$ /s) and shock compression (strain rates: > 10^{4} /s) using e.g. high-energy laser and gas gun. Fast pulses.
- Combining XRD, phase contrast imaging and possibly (if it can be done) XANES/EXAFS, as in situ techniques
- Study of Fe/Fe-alloys under dynamic compression. Expand phase diagrams to EHP (extreme high pressure) and EHT. Match results with static compression at lower P to correct for any differences.
- Mimicking seismic waves using dDAC.
    - Oscillate dDAC at seismic frequencies to study young's modulus/p-wave dispersion and attenuation.
- Study of icy giant planets: phase transitions and chemistry
    - Interiors of gas giants are still unknown in composition/chemistry and phase transitions. Diamond precipitation? Superionic water?
- Impact studies: laser compression (ns), high explosives and gas guns (us). Natural times scales match best with dDAC, but limited in P (100 GPa).
    - SiO_2 compression in dDAC. Can achieve amorphization during fast compression. Stishovite at 25 GPa, no coesite formation. Similar natural result
    - Lonsdaleite (hexagonal diamond) synthesis using dDAC. Need better resolution of XRD to confirm!

b. *Scientific and technical requirements for PETRA IV*
- Unique spectral brightness at high energies (> 25 keV) for identification of new materials / structures
- Greater imaging capabilities and probing of smaller volumes (with coherence and focusing). E.g. shock wave compression in thick container penetrated with high energy X-rays for imaging.
- Requirement of high photon flux per pulse (with 10 m long undulator). Better angular resolution. Study paid correlation functions of liquids…
- SAXS application for nanoparticles formation studies with multiple laser shock waves. Metallic hydrogen may be generated in a mixture.

c. *Special needs for on-site infrastructure (Preparation Laboratory, NanoLab, others)*
- dDACs and support lab
- Shock-compression laser (expensive? How much power?)
- Gas gun (only if it does not generate vibrations in the Hall)

d. *Important scientific or technical discussions*
- No other questions.