# PETRA IV Workshop: Materials and Processes for Energy and Transport Technology

*Session: Extreme Pressure & Temperature Research*

In this session there were 4 invited talks covering many of the Scientific Instrument Proposals (SIPs) for Extreme Conditions Research at PETRA IV. The number of participants was about 48. In chronological order, the speakers were Dr. Melanie Sieber (GFZ, Potsdam), Dr. Clemens Prescher (University of Freiburg/DESY), Dr. Dominique Laniel (Bayerisches Geoinstitut, University of Bayreuth), and Prof. Dominik Kraus (University of Rostock).

The first speaker (Dr. Sieber), discussed the 2 SIPs for research and X-ray techniques using the Large Volume Press (LVP) at PETRA IV. The three other speakers discussed each an SIP for research and X-ray techniques combined with the Diamond Anvil Cell (DAC). Each contribution from the speakers is described in more detail below.

## I. Benefits at PETRA IV for high-pressure research using the LVP: synthesis of novel nitrides and fullerenes (M. Sieber)

a. *A general overview of the talk*
    - Nitrides and oxynitrides are relatively unexplored class of inorganic compounds with potential useful properties (super hardness/optoelectronic) for functional materials.
    - High-pressure/high-temperature synthesis enables the stabilisation of many binary and ternary compositions for nitrides in large volumes (1-100 mm$^3$), not possible at ambient pressure.
    - In situ X-ray diffraction techniques enables the direct observations of reaction pathways and new structures at precisely determined high pressure/temperature (in LVP) and determination of meta-stability of the quench products.
    - Detailed characterisation of hardness, toughness and other properties can only be done on large quenched samples from the LVP (e.g. Rhenium nitride pernitride synthesis at 33 GPa/2000C)
    - Tomographic study of materials under pressure in PE-type presses. E.g. C60 polymerization process of fullerenes. Phase diagram (PT range) study. Useful for photovoltaic applications, hydrogen storage, energy materials.

b. *Scientific and technical requirements for PETRA IV*
    - The 6-ram and Paris-Edinburgh-type presses to create high pressure/temperature sample environments for studies
    - Very high energies required (30 – 120 keV) for transmission through pressure medium, cell assembly materials.
    - Improved energy resolution (better than 10$^{-3}$) over Ge-detectors required (e.g. using monochromatic beam and high-throughput & high-resolution monochromators). Enabling crystallography, particularly on low-symmetry phases! Large radius CdTe detectors.
    - Ample space to install complementary in situ techniques such as ultrasound wave speed measurements and electrical conductivity measurements)
    - Fast, sub-second, XRD and imaging acquisition for in situ kinetics studies

- Tuneable beam size for imaging (2 x 2 $mm^2$ or larger) and for XRD ('focussed' pencil beam of 50 to 200 um in vertical and horizontal) for 6-ram LVP.
- Tuneable beam size for tomographic radiography/phase contrast imaging (2 x 2 $mm^2$ or larger) and for computed tomographic XRD ('microfocussed' beam of ~1x1 $um^2$ in vertical and horizontal) for PE type presses (e.g. RoToPEc).
- Fast microtomography scanning with focused intense PETRA IV beams to shorten acquisition times e.g. by an order of magnitude compared to times at the former ID27 ESRF beamline (where 2350 XRD patterns = 8 hrs).
- Computed tomography requires fast data transfer rates (10 Ge) and large data storage capacities (1 TB/day)! More storage requirements with more pixels (from 2K x 2K to 8K x 8K = 1TB single scan!) Option for pre-procession of data via algorithms for reconstructing images from XRD patterns.

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

- Dedicated sample preparation lab with all the necessary tools for machining cell parts, offline LVP for synthesis, furnaces and ovens, etc. Access to SEM with EDX, EBSD, etc. See SIP.

d. *Important scientific or technical discussions*

- Kai asked the question about scan rates in the PE-type press for XRD tomography at PETRA IV. Answered above.
- No time for further discussions.

## II. Materials Discovery and Characterization under extreme static high pressure and temperature conditions using XRD and imaging (C. Prescher)

a. *A general overview of the talk*

- SIP dedicated to micron to sub-micron focus high-energy XRD in DAC using laser heating, an upgraded P02.2 beamline.
- DACs to study/synthesise materials under ultra-high pressures.
  1. Ultrahard materials (e.g. $ReN_2$)
  2. High-entropy materials (for CO oxidation catalysis, low thermal conductivity, Invar compounds, electrochemical energy storage, membrane batteries…). What are their stability limits? Synthesis to obtain better properties (electronic, magnetic, elastic).
  3. Superconducting materials; room-T, ultra-high-pressure materials discoveries (C-S-H, unknown structure, need more scattering power of PETRA IV).
  4. Double-stage + toroidal DAC for extremely high-pressure (1 TPa) generation.
  5. Polymeric Nitrogen; molecular nitrogen compressed to 140 GPa, laser heater to 2500 K. New structure for phase diagram of N2.
  6. Other nitrogen compounds: $Os_5N_{34}$ at 100 GPa.
  7. Metallic glasses/silicate glasses (long-range structures, pdfs), PETRA IV can offer more flux and higher Q range, better signal/noise ratio/real space resolution.

b. *Scientific and technical requirements for PETRA IV*

- Strong increase in brilliance (factor of 10)

- Improved focusing (nano-beam) for e.g. toroidal DAC
- Improved coherence – new imaging techniques
    1. Better transverse coherence at 25 keV!
    2. Study macro-strain in DAC/micro-strain (stress). Single grain CXDI imaging in DAC!
    3. Phase contrast imaging using coherence of melting/crystallisation reactions of materials (by laser heating). i.e. Image kinetics of phase transformations/melting behaviour
    4. 3d tomography techniques in DAC to study more complex materials
- Chance to study grain microstructure (grain distributions and boundaries), formation mechanisms…
- Single Xl: Benefits for study of low-Z materials up to 200 GPa/ higher Z materials up to 500 GPa/ realtime in situ structural studies.
- High-energy XRD for low-Z materials, larger Q-range = better real space resolution.

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

- Not mentioned in detail, see SIP

d. *Important scientific or technical discussions*

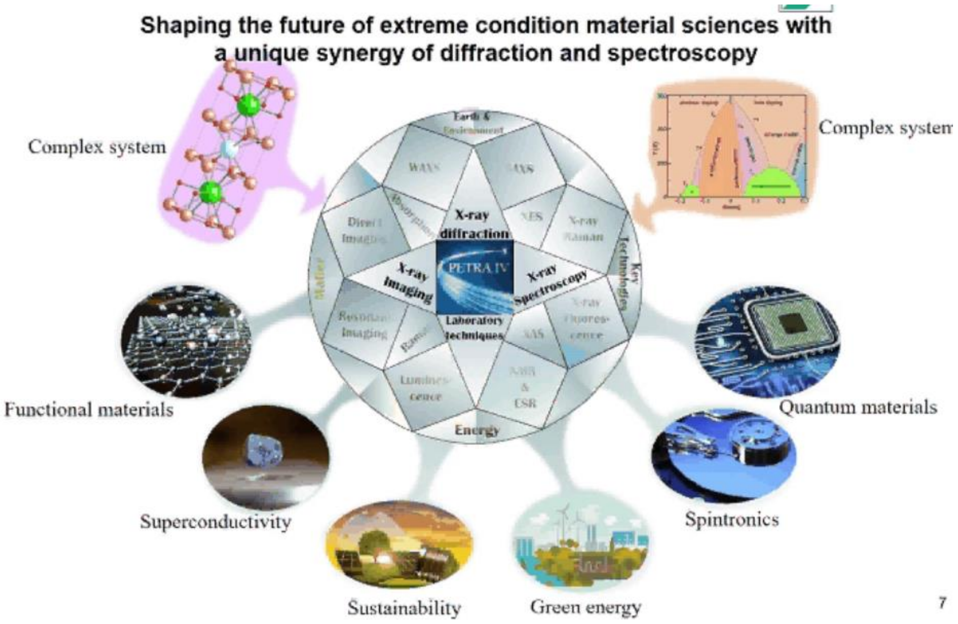
- Robert Farla asked if XRD and new imaging techniques can be used in the DAC at the same PETRA IV beamline, no requirement to increase energy (same values as at PETRA III)

## III. Shaping the future of extreme condition materials science: a unique synergy of diffraction and spectroscopy at Petra IV (D. Laniel)

a. *A general overview of the talk*

- Spectroscopy often an overlooked method at high-pressure beamlines (secondary to XRD) - Raman, resistivity, magnetic susceptibility, Xanes, XAS…
    1. No beamline combines all techniques and samples are unique. This can be changed at PETRA IV.
- Highlights of high-pressure research using DACs, which could benefit from spectroscopy techniques
    1. High super conducting temperatures in new materials (LaH10, S-C-H compound).
    2. Strongly correlated materials (quantum/functional)
        a. Metallic glasses (electronic,magnetic,mechanical properties)
        b. High-entropy materials
        c. Quantum spin liquid materials under high pressures

- Diamond of combining all XRD/Imaging/Spectroscopy techniques (see below)



- **Use of XAS/WAXS/SAXS/ASAXS**: XAS of nucleation centres will give element specific information on their coordination, SAXS is sensitive to topological heterogeneities of crystallising amorphous systems (mid- to long-range structure), SAXS/WAXS is used for element-independent particle growth, determination of average interatomic distances. **ALL at high P and T in DAC**!
- **Use of XES (X-ray emission spectroscopy) and XRD**: Determination of valence states (XES) and local spin states of trans. elements. 3d-orbital population of compounds cont. trans. metals. **ALL at high P and T in DAC!**
- **Studies on**: phase stability, transformations, diffusion, recrystallization, kinetics, modification of spin state/valence state/magnetism/local structure…
- **X-ray Imaging**: combined with the above. E.g. visualise where low spin and high spin states occur, or image scanning over an absorption edge (XANES).

b. *Scientific and technical requirements for PETRA IV*

- Assumption of U29 undulator at PETRA IV (10 – 50 keV) – sufficient flux ($10^{12}$ ph/s/0.1% bw.) and beam size for all the above techniques. Possibility to add micron to sub-micron focusing.
- Achieve quest to determine sample properties using combined techniques on a unique sample at high P,T in DAC in one beam time.
- State of the art detectors – suitable for various spectroscopy/XRD/imaging techniques (flat panel and photon counting detectors, for fast kinetics). And von Hamos spectrometer.

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

- High-temperature (resistive heating and laser heating) in DAC
- Cryogenic temperature environments
- Conventional offline IR, UV, visible light spectroscopy lab
- Experiment preparation lab
- NMR preparation station
    1. CVD deposition
    2. FIB machining SEM

        3. Offline magnets
            a. 1 T and 7 T

*d. Important scientific or technical discussions*

- Kai: Crazy many techniques at one beamline. Very ambitious. Can it work? – Yes, apparently. Even all constrains for detector requirements, geometry requirements, beam size requirements, sample size requirements (asked by Caliebe), can be taken care of. Glazyrin: Of course, not all techniques are applied at the same time on same sample. Depends on case-by-case scientific study. R. Farla privately worries about long times required to switch set ups/optics and perform required calibrations! Some combinations of techniques should be emphasised with higher priority/likelihood of realisation.

## IV. Opportunities for dynamic compression science at synchrotrons (D. Kraus)

*a. A general overview of the talk*

- Early stage for dynamic compression studies in DAC using synchrotron radiation (<10 years of development).
- Applications to physics at extreme PT/strain rate conditions, generation of new materials only forming under dynamic conditions, performance of materials under transient high P/T conditions (e.g. space craft impact in Earth's atmosphere/surface).
- Extreme PT conditions + fast compression/deformation rates: changing phase diagram boundaries of materials.
- Tools: **Dynamic DAC (dDAC)** and **shock compression (laser/gas gun)** at a PETRA IV beamline to cover various ranges in strain rates.
- Examples
    1. **Hexagonal diamond** – does it exist?? Maybe! Can be created under dynamic conditions, not readily under static conditions. Better quality XRD required!!
    2. Shift of phase transition boundaries in **bismuth** under dynamic compression compared to slow 'static' compression.
    3. High-precision pair correlation function of high P/T liquids
    4. XRD+Imaging (phase contrast) during dynamic DAC compression on high-Z materials and in thick X-ray absorbing containers. High spectral brightness at PETRA IV will help!
    5. Strength and plasticity studies under extreme strain rates using radial XRD in dDAC.

*b. Scientific and technical requirements for PETRA IV*

- Unique brightness at high energies (> 25 keV), new imaging capabilities and small sample probing with coherence and focussing. Unprecedented flux per bunch for fast timing experiments, sharp energy resolution, great for e.g. laser shock compression. Option to study higher-Z (absorbing) materials, thick samples, and liquids than possible elsewhere.
- Combining: XRD, SAXS, X-ray imaging over various length and time scales.
- Instruments: dDAC, shock-compression laser, gas gun (maybe).
- Techniques: XRD, X-ray phase contrast imaging, small angle X-ray scattering

c. *Special needs for on-site infrastructure (Laboratory, NanoLab, others)*
    - Not discussed/mentioned in particular

d. *Important scientific or technical discussions*
    - D. Pennicard asks about detector requirements. AGIPD detectors work very well, we take what is available (HP Liermann). Collection of 325 images are a good start. 1 kHz to 100 kHz range is perfect!
    - dDAC diamonds can survive experiments. But shock laser and detectors can be very costly.
    - Beamline requires 10m long undulator for sufficient flux (HP Liermann).