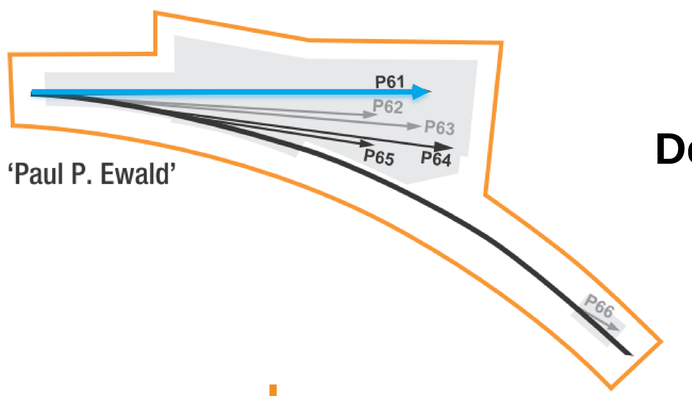
# Beamline P61B: Large Volume Press.





Deutsches Elektronen-Synchrotron DESY
A Research Centre of the
Helmholtz Association


## Research at High Pressure and Temperature


Robert Farla[1], Shrikant Bhat[1], Artem Chanyshev[1,2], Shuailing Ma[1,3], Stefan Sonntag[1], Takayuki Ishii[2], Tomoo Katsura[2]

[1]Deutsches Elektronen-Synchrotron DESY, Hamburg, Germany [2]Bayerisches Geoinstitut, University of Bayreuth, Germany [3]State Key Laboratory of Superhard Materials, Jilin University, Changchun, China


## Mission statement

**To probe the structure and properties of materials *in situ* at high pressures and temperatures in a Large Volume Press (LVP) using X-ray diffraction and imaging techniques, as well as ultrasonic and electrical resistivity methods.**

**Applications in geo- and materials sciences:**

- Phase relations:
  - Transformation/nucleation
  - Melting curves (solidus/liquidus)
  - Equations of state
- Crystallography
- Controlled rock deformation
- Melt viscosity measurements
- Structure of amorphous materials
- Ultra-high pressure (60+ GPa) & temperature (3000 K)

**Additional methods:**

- Ultrasonic wave speed measurements
- Acoustic Emissions testing
- Electrical conductivity (coming up)

## LVP & detector specifications

| | |
|---|---|
| **mavo press LPQ6-1500-100** | 6 indep. controlled rams |
| **Maximum load** | 15 MN – 5 MN/axis |
| **Ram position control** | 1 µm step – 100 mm |
| **Oil pressure control** | 0.5 bar – 620 bar/ram |
| **Anisotropic compression** | Axial symmetric, triaxial |
| **5-axis stage** | x,y1,y2,z (± 100 mm), rotation: ± 11.5° |
| **Combined weight** | ca. 45 ton |
| **Ge-detector (2x) – ED XRD** | Mirion (Canberra) |
| **Collimator slit (mm)** | 0.03, 0.05, 0.1, 0.2 |
| **Receiving slits (mm)** | 0.05, 0.1, 0.2, 0.5, 1.0, 2.0 |
| **Horz. detector positions** | 1xGe: min 4° - max 20°<br>2xGe: min 5° - max 10° |
| **Horz. & vert. positions** | $Ge_{vert}$: min 7.5° - max 23°<br>$Ge_{horz}$: min 6.5° - max 10° |

## Beamline P61 specifications

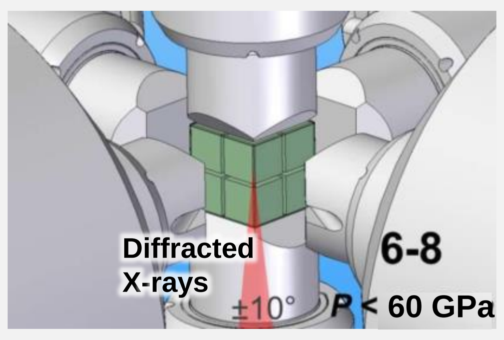
| | |
|---|---|
| **Source** | 10x wigglers |
| **Length (m)** | 10 x 4 |
| **Period length (mm)** | 200 |
| **# periods** | 10 x 19 |
| **Peak field B0(T)** | 1.52 |
| **Def. parameter K** | 28.4 |
| **Max. power (kW)** | 10 x 21 |
| **Usable energy range P61B** | 30 – 160 keV (Ge-SSD) |
| **Power density P61B** | 16 W/mm$^2$ |
| **Filtered power P61B** | 12 – 10 W/mm$^2$ |
| **Peak flux density P61B @ 50 keV** | $10^{12}$ ph/s/mm$^2$/0.1% b.w. |
| **Max. beam size P61B** | 2.2 mm (h) x 1.7 mm (v) |
| **Min. beam size P61B** | 0.03 mm x 0.03 mm |

## High-pressure *in situ* measurement techniques

### Versatile compression modes

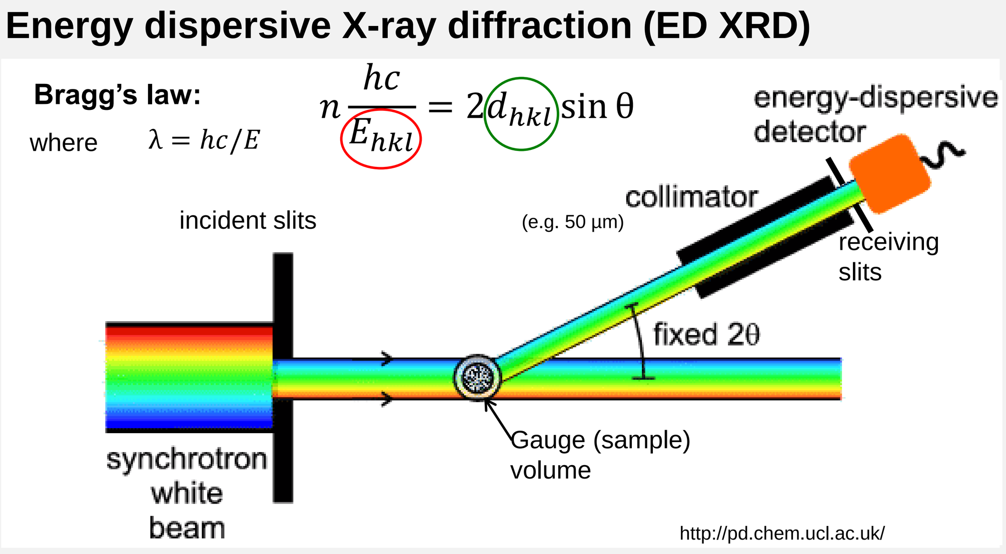
**Kawai-type (8-6):**
Extreme pressures, reliable geometry

**Cubic (6-6):**
Large X-ray window, anisotropic compression

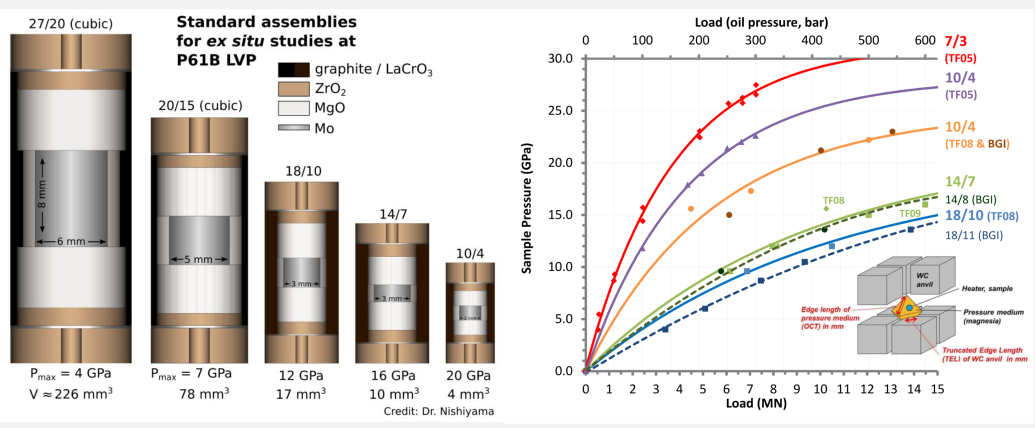




### Energy dispersive X-ray diffraction (ED XRD)



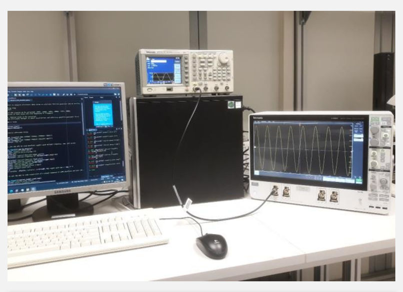

### Assemblies and pressure generation




### Complementary *in situ* techniques

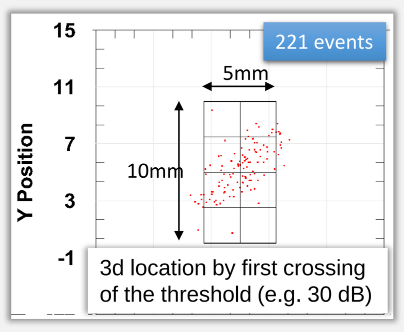
**Ultrasonic interferometry**

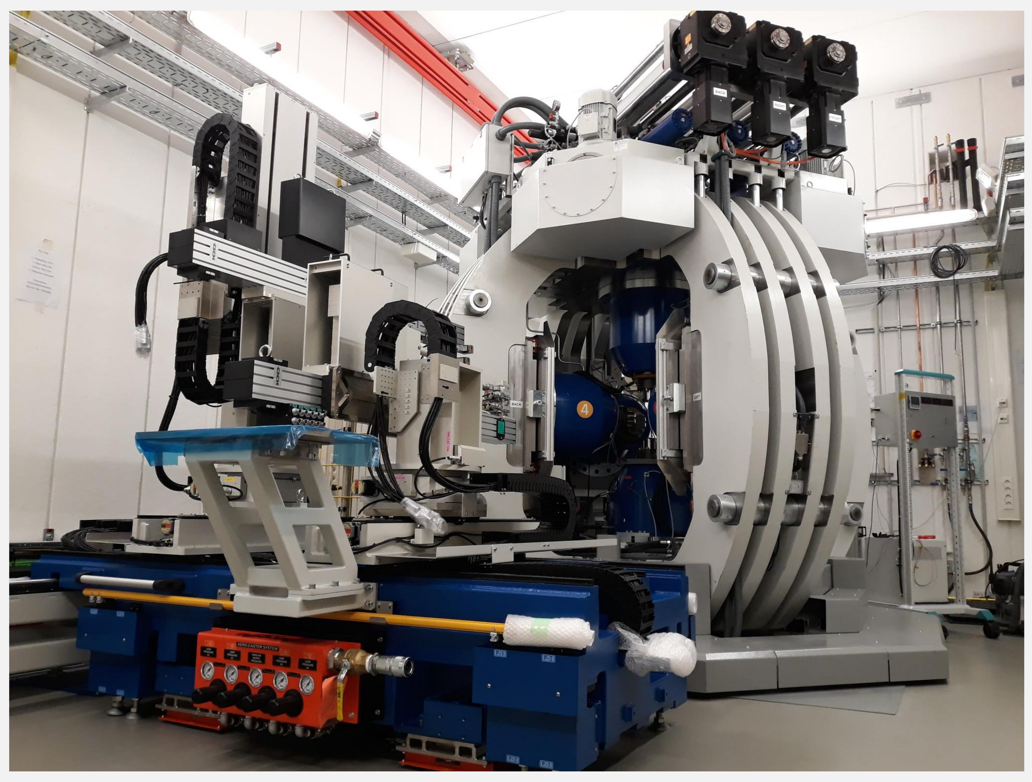
Measurement of two-way travel time of ultrasonic waves in a sample at high P and T using a $LiNbO_3$ sensor on the back of an anvil, which transmits a pulse and receives an echo. Simultaneous imaging (**radiography**) provides sample length. Wave speed at given P,T is calculated for determination of elastic moduli.

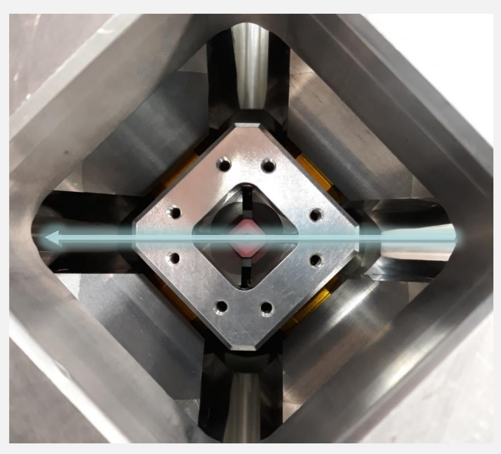


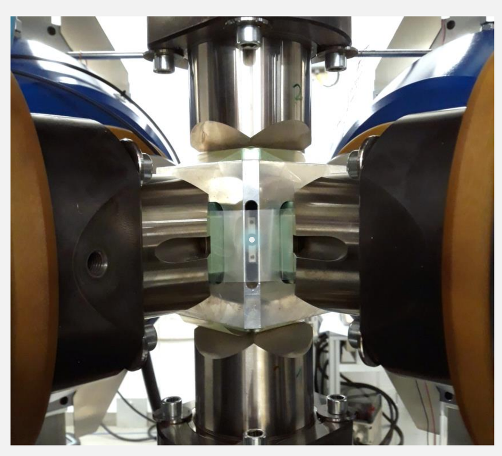
**Acoustic Emissions testing**

Measurement of *in situ* cracking of specimens under deviatoric stress at high P and T. Useful for the understanding of intermediate- and deep-focus earth quake generation and host-rock failure e.g. in empty reservoirs for $CO_2$ sequestration. In 6-6 mode, each anvil is fitted with an acoustic sensor for AE detection for 3d location & moment tensor analysis).



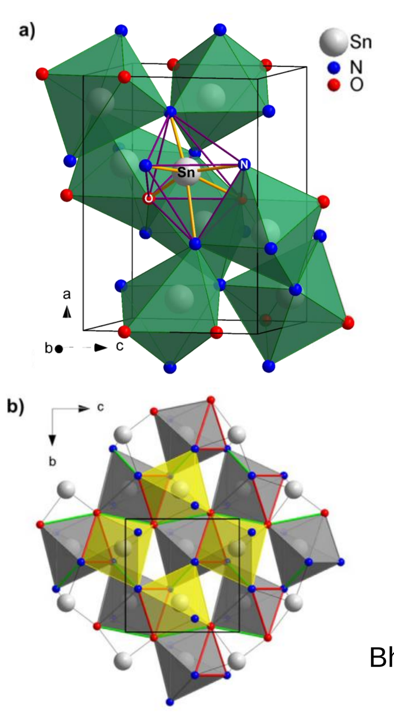


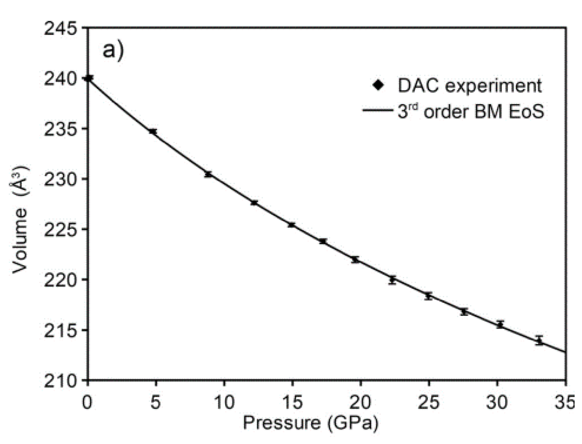


## Beamline research highlights

### I. Discovery of the first tin oxynitride, $Sn_2N_2O$ (SNO)

SNO was synthesized at 20 GPa and 1200 – 1500°C in the large volume press at P61B. It has a $Rh_2S_3$-type crystal structure with space group Pbcn. All Sn atoms are in six-fold coordination, in contrast to Si in silicon oxynitride ($Si_2N_2O$) and Ge in the isostructural germanium oxynitride ($Ge_2N_2O$), which appear in four-fold coordination. The isothermal bulk modulus was determined as $B_o$ =193(5) GPa using *in-situ* synchrotron X-ray diffraction in a diamond anvil cell. The structure model is supported by DFT calculations.



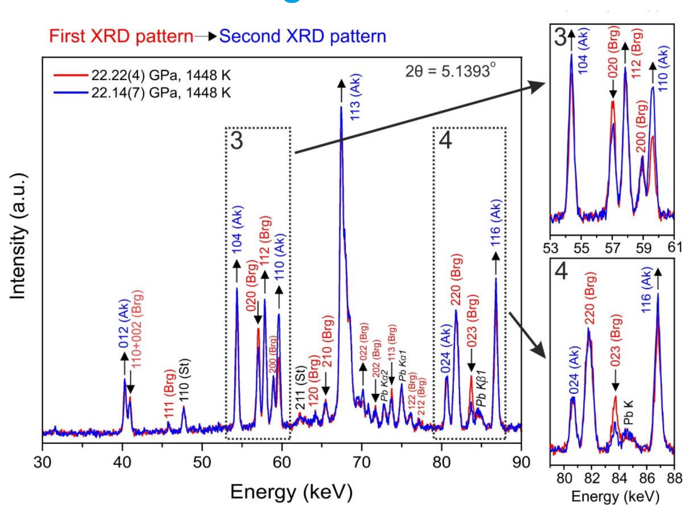




**Pressure dependence of the unit-cell volume of SNO and the 3rd order Birch–Murnaghan EoS fitted to the data.**

Bhat, S. *et al. Chem. Eur. J.* **2020**, 26, 2187 – 2194

**Crystal structure of SNO. a) Unit cell, showing the distorted octahedral coordination of Sn. b) View along the a-axis, showing grey and yellow coloured octahedra sharing faces (indicated as red triangles).**

### II. Depressed 660-km discontinuity caused by akimotoite-bridgmanite transition

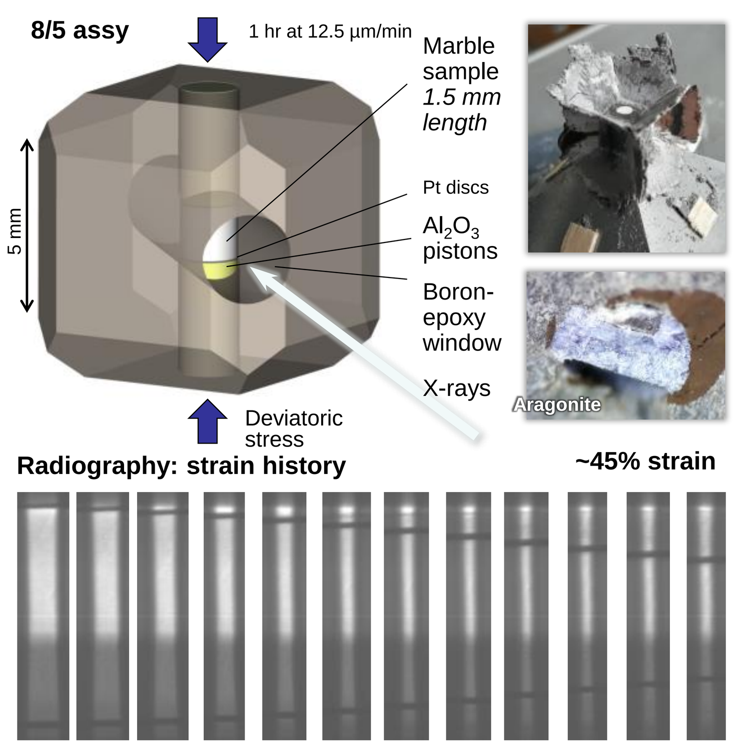



**Accurate phase identification by means of *in situ* X-ray diffraction in a multi-anvil press. An example of the change of intensity ratio between akimotoite and bridgmanite at 1448 K and 22.14(7) – 22.22(4) GPa.**

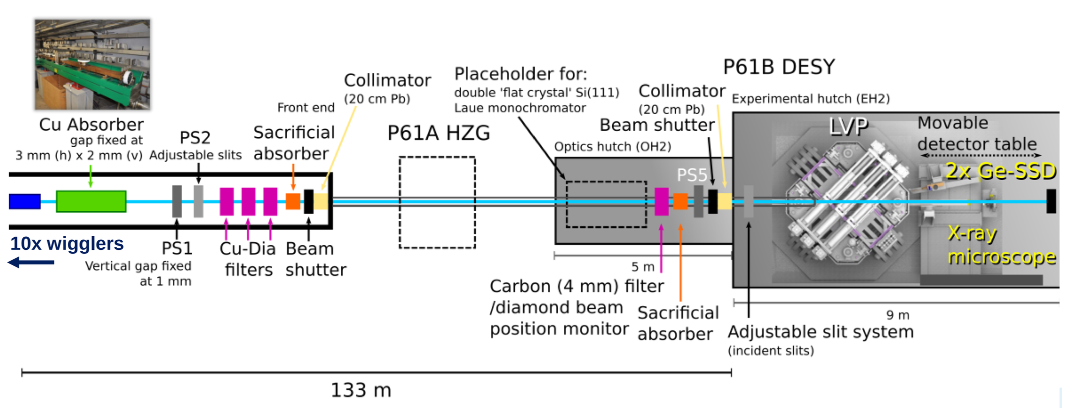
Chanyshev, A. *et al. In review*

### III. Controlled *in situ* rock deformation

**Absorption contrast imaging using a double objective microscope (5x, 10x magnification)**




### Beam line and hutch layout




## Future perspectives

1. Enhance capabilities at the beamline for crystallography and rock deformation studies using angle-dispersive XRD. This requires a monochromatic beam at selectable very high energies (> 60 to 100 keV).

**Required Instrumentation:**

- Double-xl Laue monochromator
- Large radius flat panel detector.

2. Studies of stability and dehydration reactions of hydrous minerals at high pressure, simulating onset of melting and/or seismicity for better understanding of the origin of volcanism and earthquakes, respectively.

**Required Instrumentation:**

- Impedance analyzer for electrical conductivity.

3. Expansion of the beamline with a second LVP (Paris Edinburgh-type) for e.g. tomography.


HELMHOLTZ
RESEARCH FOR GRAND CHALLENGES

BGI Bayerisches Geoinstitut

Bundesministerium für Bildung und Forschung