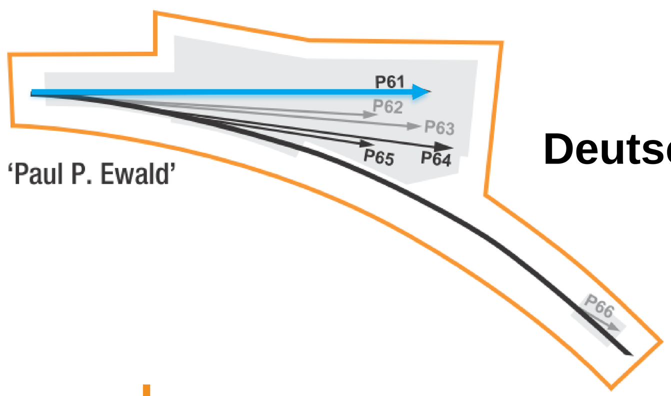
Current and future role of P61B in Nano-Mat



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

Research at High Pressure and Temperature

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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 using complementary 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° 2xGe: min 5° - max 10°
Horz. & vert. positions	Ge_{vert}: min 7.5° - max 23° 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

A high-pressure beamline for synthesis and *in situ* characterisation of novel materials

Overview

Beamline P61B has **experience producing novel materials** at high pressures and temperatures.

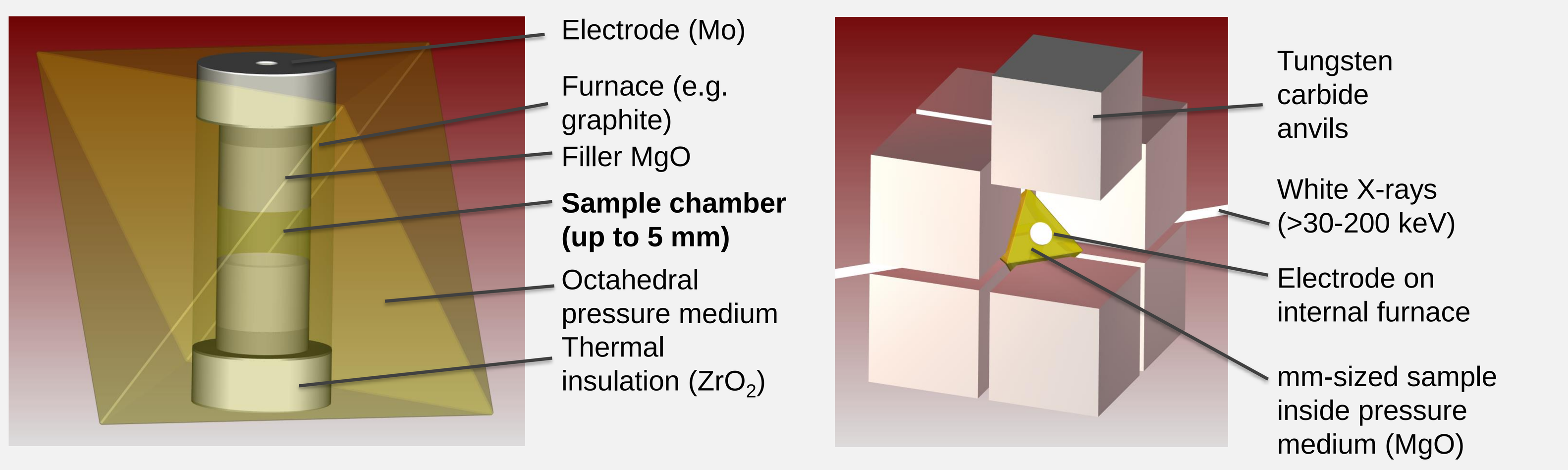
High-pressure solid-state synthesis opens up new possibilities to explore material reactions, which would otherwise be energetically (and dynamically) unfavourable to occur. The LVP offers mm- to cm-sized samples, which can be synthesized and simultaneously characterized *in situ* by ED XRD, and recovered **meta-stably** for further *ex situ* analysis.

Particularly, using *in situ* synchrotron X-ray diffraction techniques, hitherto unknown thermodynamic and kinetic pathways for optimal synthesis at high pressures and temperatures can be explored and controlled. A range of different end products can be recovered with **interesting properties** such as **electronic**, **magnetic**, **structural** and **vibrational** for a wide range of applications. The discovery of unexpected high-pressure physical and chemical processes, which lead to the formation of novel materials, becomes the **rule rather than the exception**.

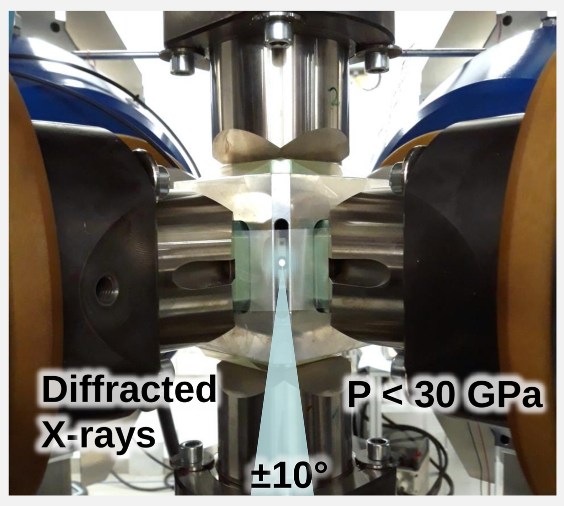
Common classes of materials

- **Carbides**
  Examples: Diamond, SiC, WC,…
  Applications: cutting tools, machine components, quantum computers,…
- **Borides**
  Examples: Metal-B, Metal$_2$B,…
  Applications: Fiberglass, cutting tools, electromotors,…
- **Nitrides**
  Examples: h/cBN, Si_3N_4, TiN, AlN,…
  Applications: cutting tools, solar panels, LEDs, circuits,…
- **Hydrides**
  Examples: MgH_2, $NaAlH_4$, $LiAlH_4$,…
  Applications: H_2 storage, batteries, superconductors,…
- **Oxides**
  Examples: TiO_2, ZnO, Fe-oxide,…
  Applications: magnets, fuel cells, electronics, catalysts,…
- **Silicates**
  Examples: SiO_2, $(Mg,Fe)_2SiO_4$,…
  Applications: Glass production, load sensors, microchips, biomedical,…

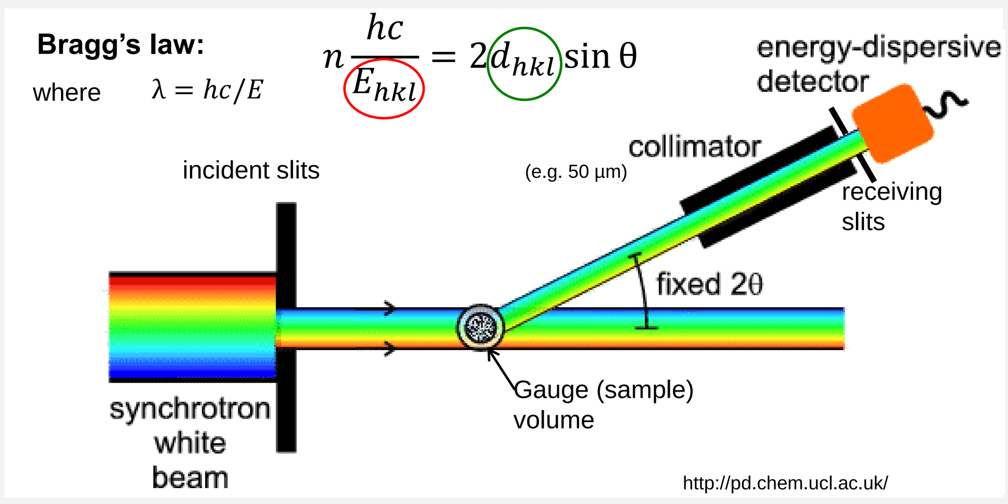
Assemblies and pressure generation



Compression in the LVP



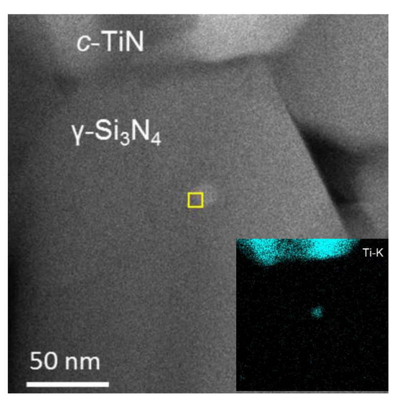
Energy dispersive X-ray diffraction (ED XRD)



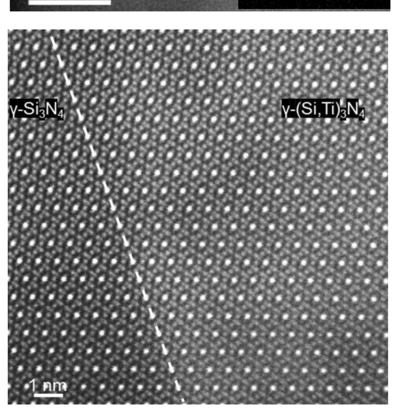
Beamline research highlights

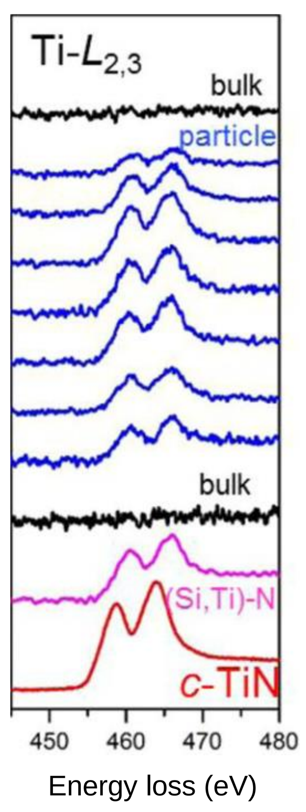
I. Discovery a new ternary silicon titanium nitride

The novel nitride is formed from an amorphous silicon titanium nitride (SiTiN) precursor under high-PT conditions in the P61B Large Volume Press. Under conditions of 15-20 GPa and 1800–2000°C, spinel-type γ-Si_3N_4 and rock salt-type c-TiN are formed. In addition, crystals of the discovered nano-sized ternary phase $(Si_{1-x}Ti_x)_3N_4$ embedded in γ-Si_3N_4 are identified. The ternary compound is analyzed to the spinel-type structure with ca. 8 atom% of Ti. The Ti atoms occur in both Ti^{3+} and Ti^{4+} oxidation states and are located on the Si sites.



Left: **ADF-STEM image obtained from a single grain, with Ti-K edge STEM-EDX, and atomic resolution ADF-STEM image on the boundary of the ternary $(Si,Ti)_3N_4$ particle.**





Right: **EELS profile of the Ti-$L_{2,3}$ edge across the nano-particle.**

Bhat, S. *et al. Sci Reports* **2020**, 10, 7372.
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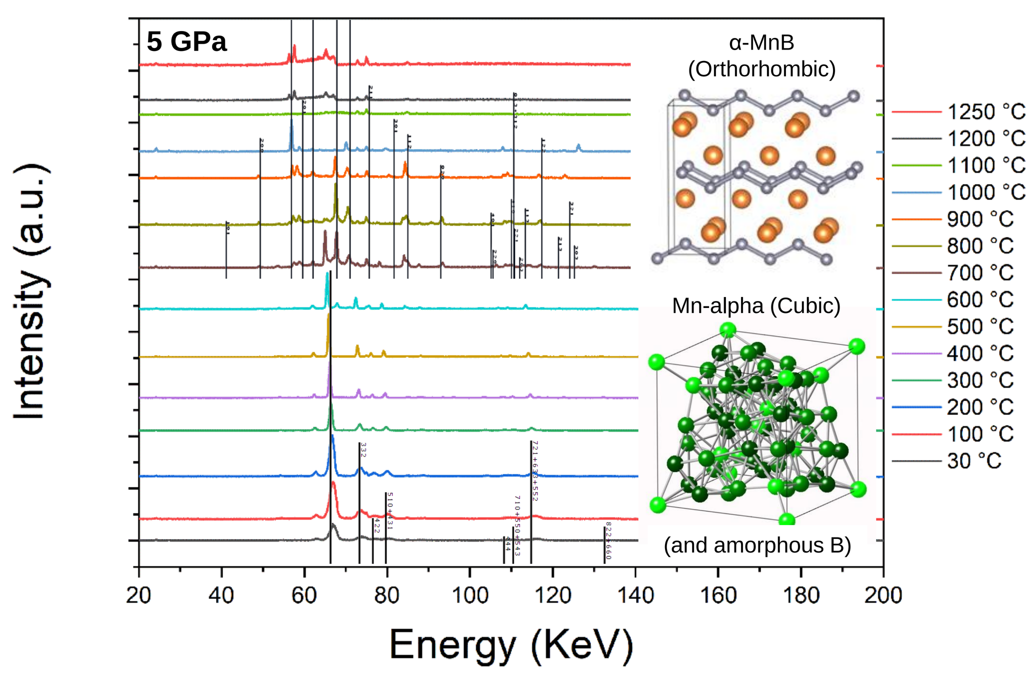
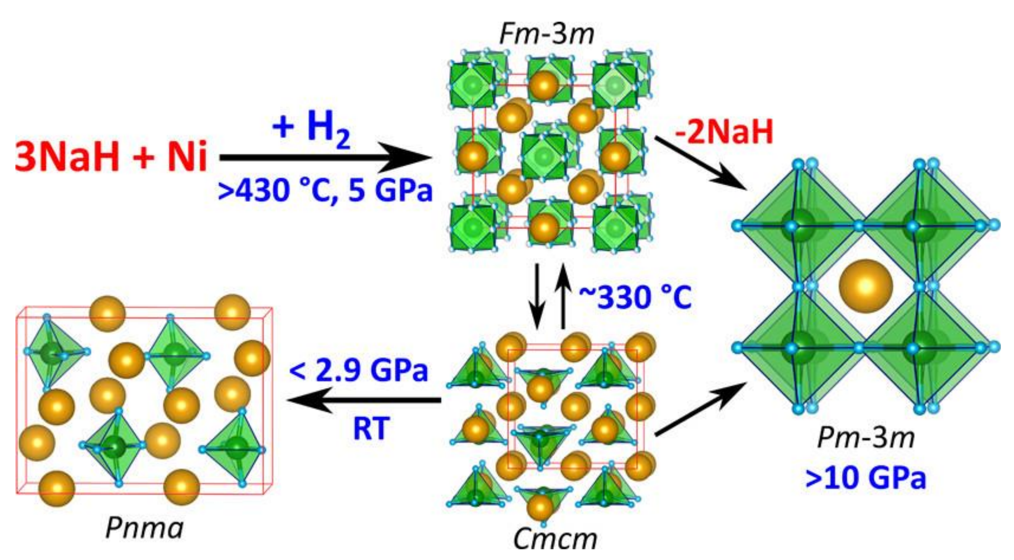
II. High-pressure synthesis of manganese boride (α-MnB)



- Due to large numbers of unpaired electrons of manganese ions and strong covalent boron zigzag chains, MnB exhibits **strong ferromagnetic**, **magnetocaloric** behavior, and possesses **high Vickers hardness**.
- Zigzag boron chains in the α-MnB structure not only play a pivotal role in **strengthening mechanical properties**, but also **tune the exchange correlations** between manganese atoms.
- The unique combination of **high hardness, magnetism, and electrical conductivity** properties makes it a particularly promising candidate for a wide range of applications, such as **high-speed motors** and **magnetic sensors** in micro-mechanical systems.

→ See poster by Shuailing Ma →

III. New synthesis route for quenchable ternary hydrides targeting room T (high-P) superconductors



Previous results

Investigation of a reaction mixture NaH/Ni/H_2 at ~5.5 GPa. Initially, after BH_3NH_3 decomposition & H_2 fluid formation, NiH forms which then reacts to previously unknown polymorphic Na_3NiH_5 (cmcm), which transforms into a quenchable polymorph (Pnma) below 3 GPa. The cubic high-temperature form is dynamically disordered, whereas the orthorhombic low-temperature phase is ordered. Above 10 GPa, metallic perovskite $NaNiH_3$ forms.

Current objectives at P61B

Exploring potentially superconducting hydrogen-rich hydrides with ternary compositions: A-X-H A = Li, Mg, Sc, Y, La; X = Si, Ge, P, S in the LVP at pressures up to 20 GPa, elucidating P,T formation conditions and reaction pathways.

Spektor, K. *et al. ACS Omega.* **2020**, *5*, 8730–8743.
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