In situ energy-dispersive XRD & imaging in the Large Volume Press at P61B

Satellite workshop P61B
20 January 2025

Robert Farla

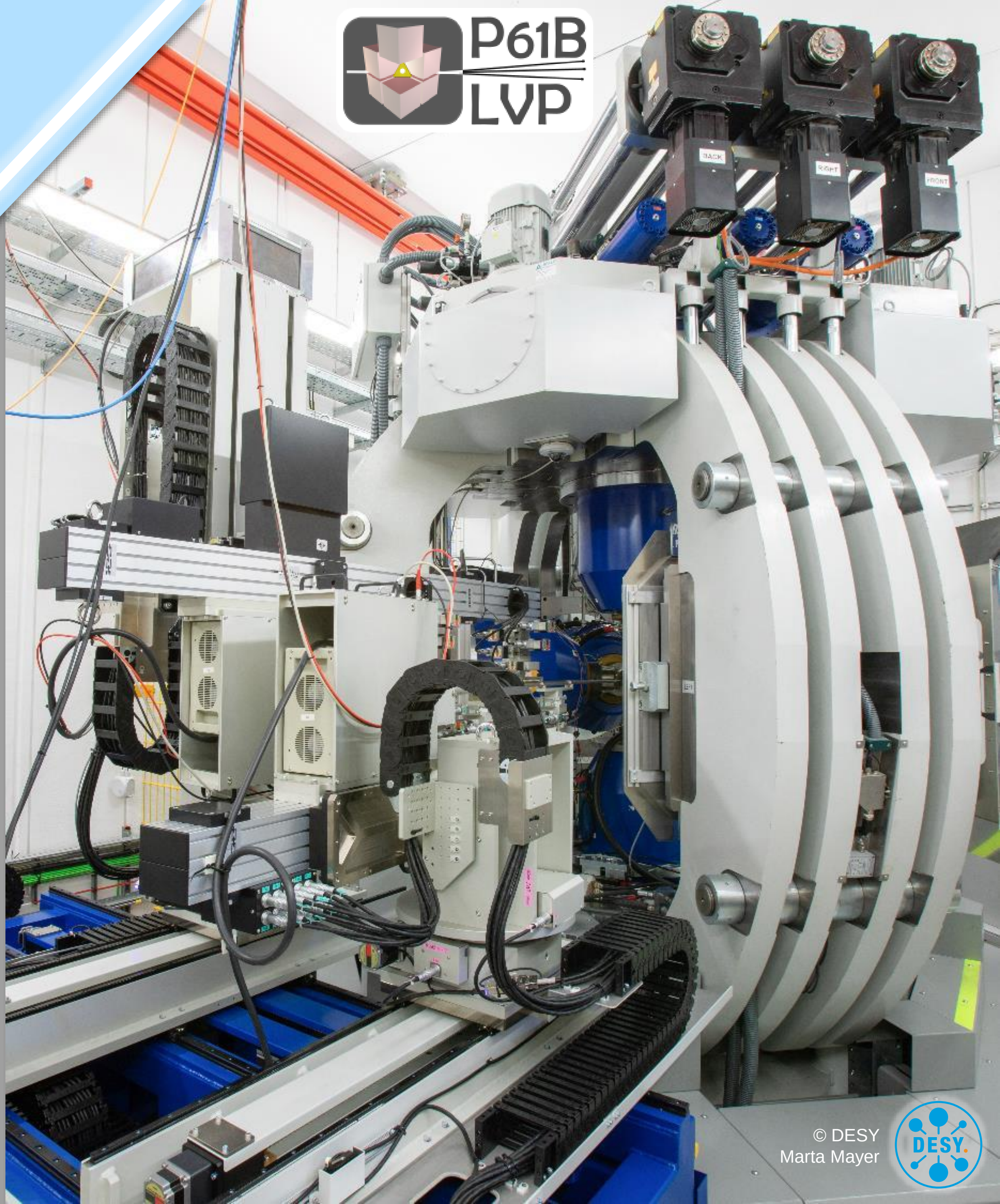
Beamline staff:
Shrikant Bhat (BL Scientist), Stefan Sonntag (Engineer),
Kristina Spektor (Guest), Xiaokang Feng (Guest)

Key external collaborators:
Tomoo Katsura (BGI), Ulrich Häussermann (KTH), Artem Chanyshev (BGI),
Shuailing Ma (Ningbo), Adrien Néri (Lille), Lianjie Man (BGI), Julien Gasc (ENS Paris)
Christian Lathe (GFZ, Potsdam)

Support: **DESY Support Groups (FS-TI, -EC, -BT, Machine…)**

HELMHOLTZ RESEARCH FOR GRAND CHALLENGES · BGI Bayerisches Geoinstitut · UNIVERSITÄT LEIPZIG · KTH · GFZ Helmholtz-Zentrum Potsdam

DESY.



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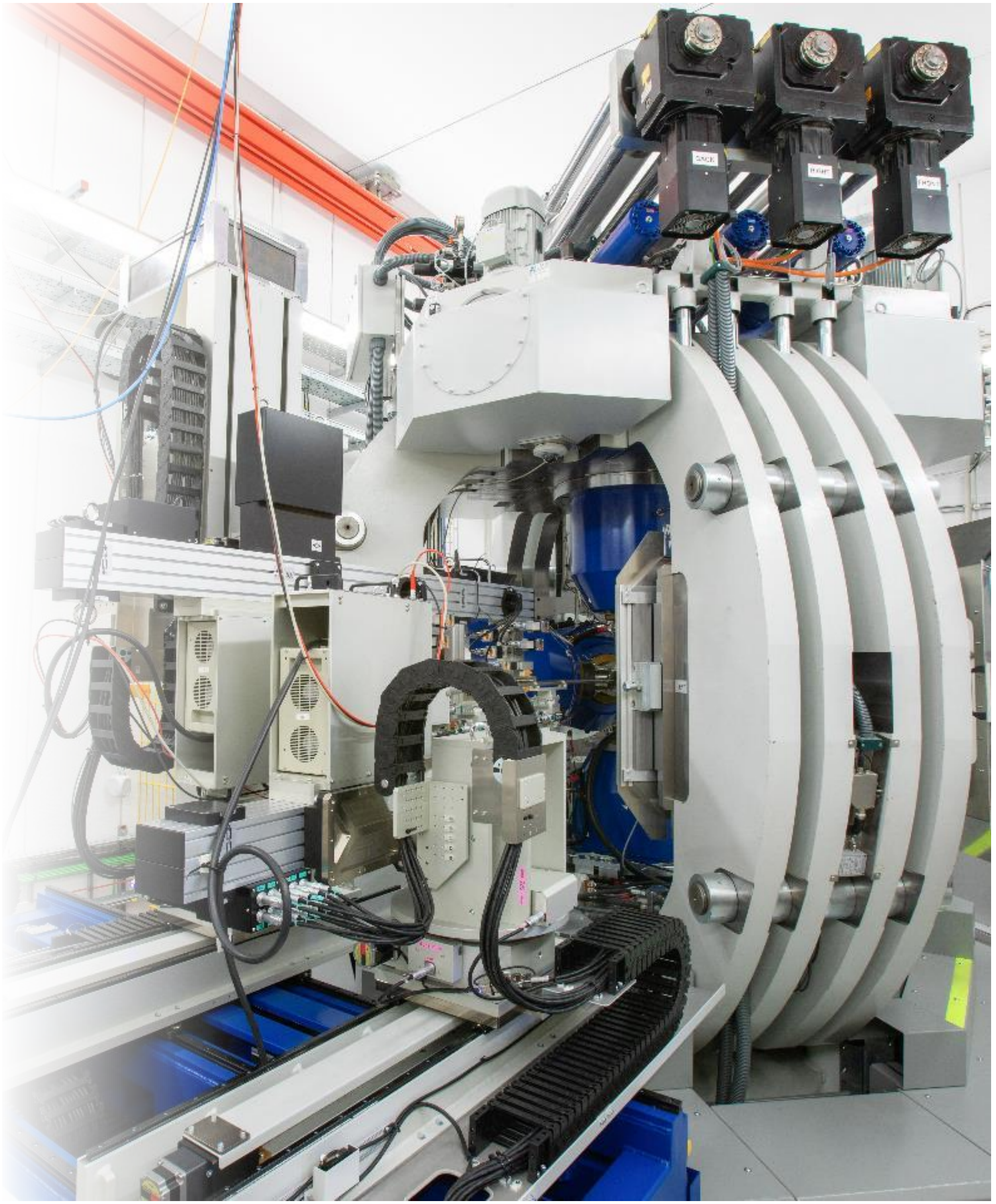
Marta Mayer

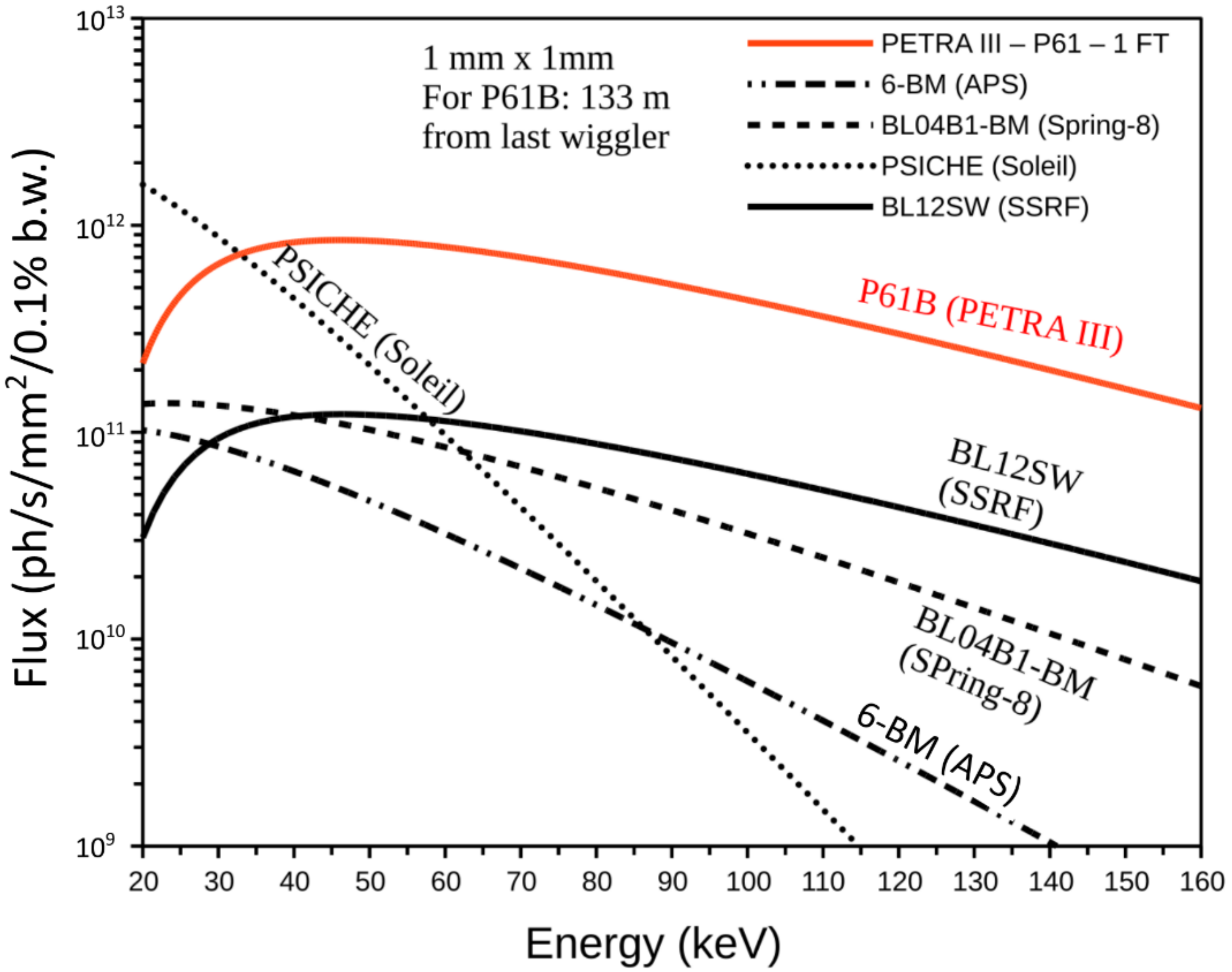
Outline

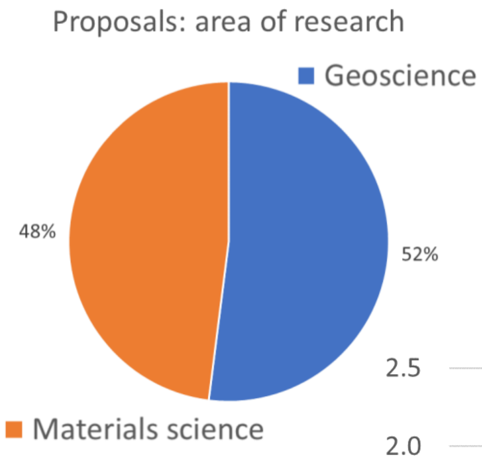
1. Introduction of the beamline
2. High Pressure & X-ray techniques at P61B
3. Geosciences research highlights
5. Materials sciences research highlights
4. LVP-XPRESS @ PETRA IV (2032+)
6. Summary and outlook

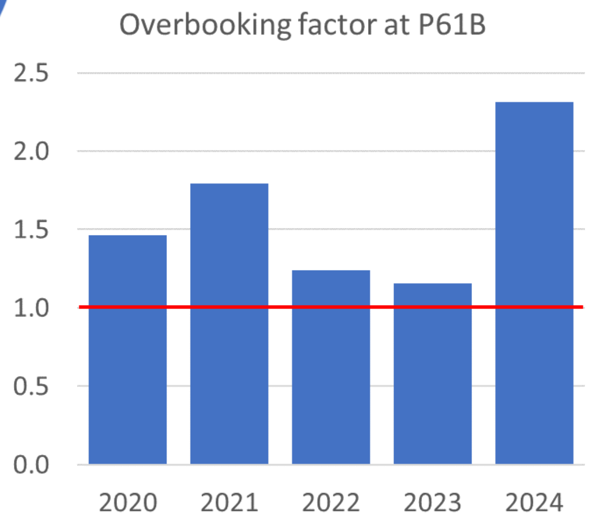


Comparison to other white-beam LVP stations

A competitive edge







Since 2020, ~24 proposals accepted for beam time yearly

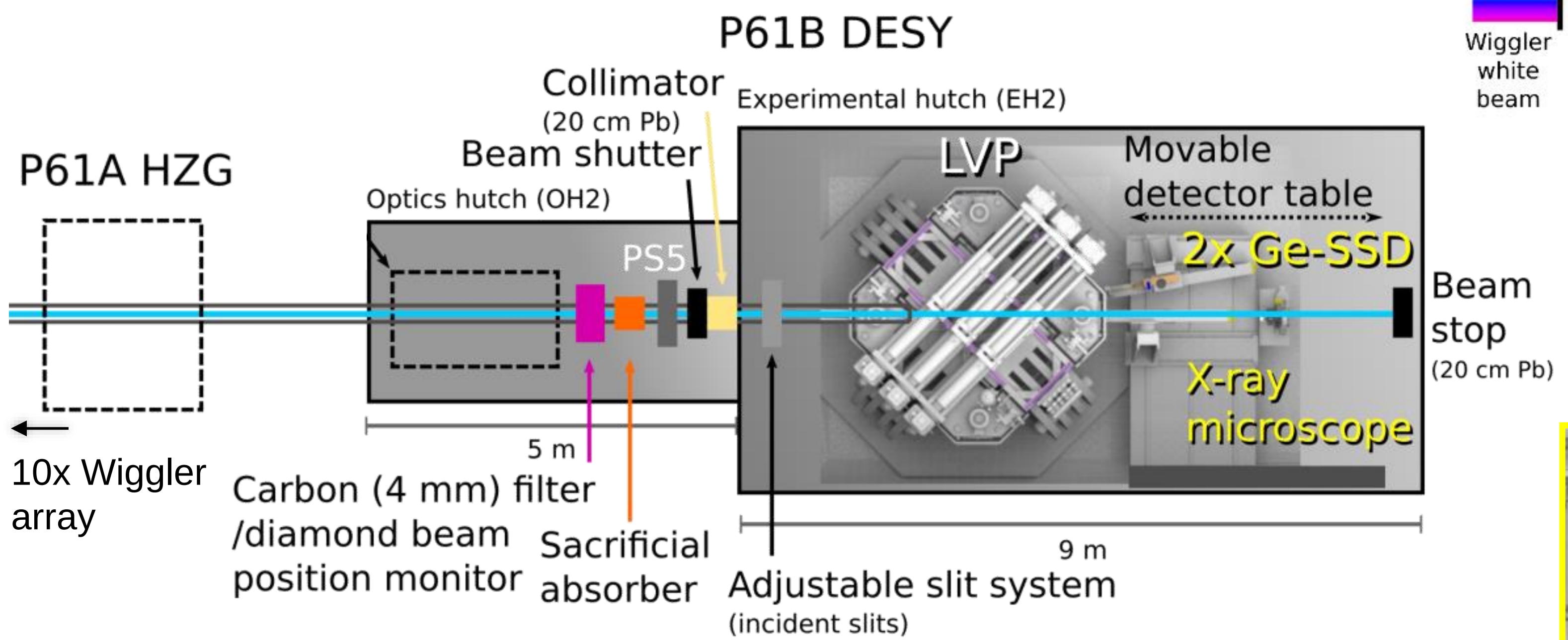
Ideal place for high-energy (> 30 keV) energy-dispersive x-ray diffraction (ED-XRD)

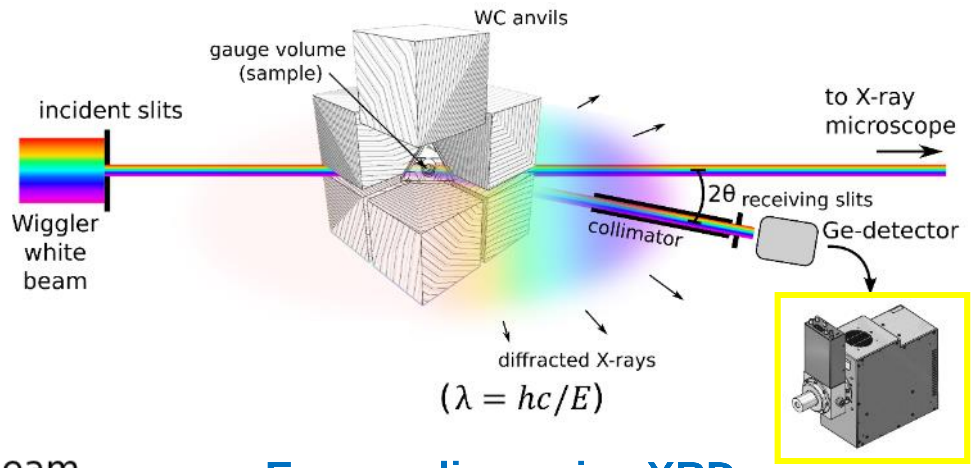
Farla *et al. J. Sync. Rad.* 2023

P61B LVP

Beamline station layout and experiment types

The study of properties and structure of solid & liquid materials under (ultra-)high pressure, temperature and stress

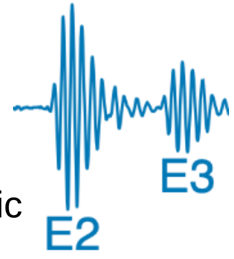




Energy-dispersive XRD

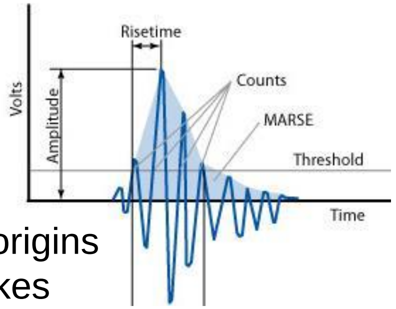
→ HP-HT phase relations studies

Ultrasonic velocity measurements



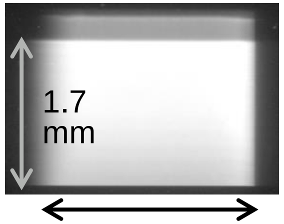
→ Investigate seismic structure of Earth

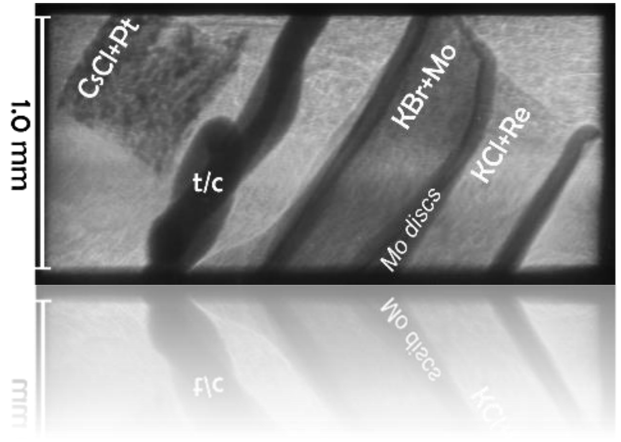
Acoustic Emission detection



→ Investigate origins of earthquakes

Full beam size





High resolution imaging

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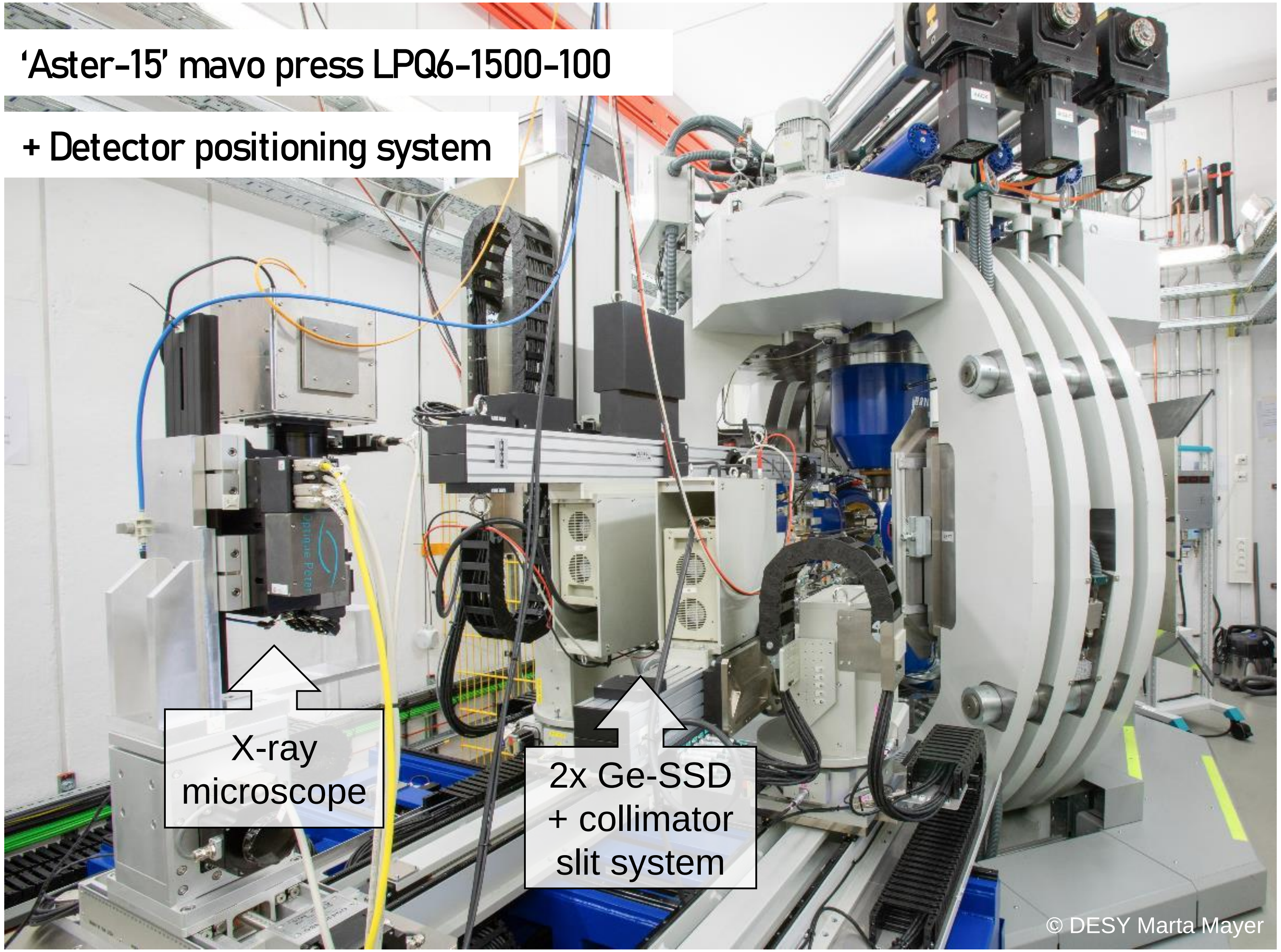
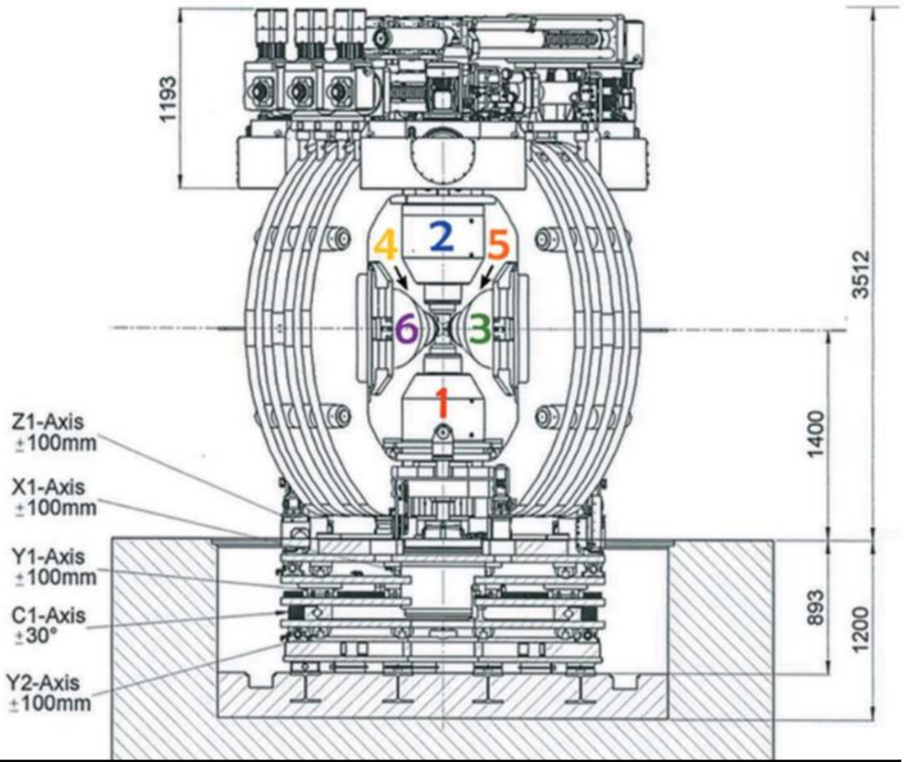
P61B LVP

The Aster-15 LVP, financed by BMBF grants 05K16WC2 & 05K13WC2





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 compr.	Axial symmetric, triaxial
5-axis stage	x,y1,y2,z (± 100 mm), rotation: ± 11.5°
Combined weight	ca. 45 ton

P61B LVP

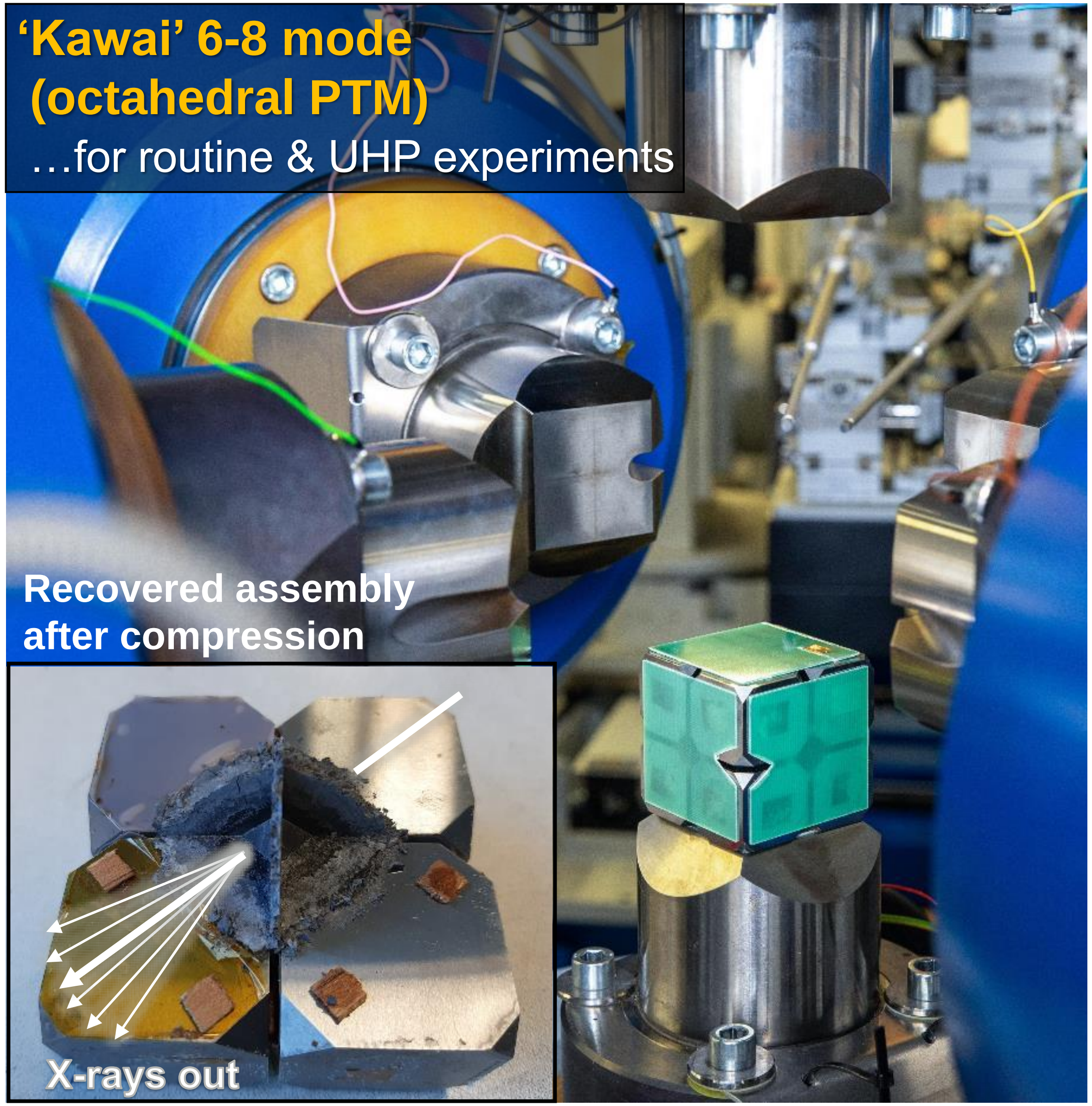
Other investments

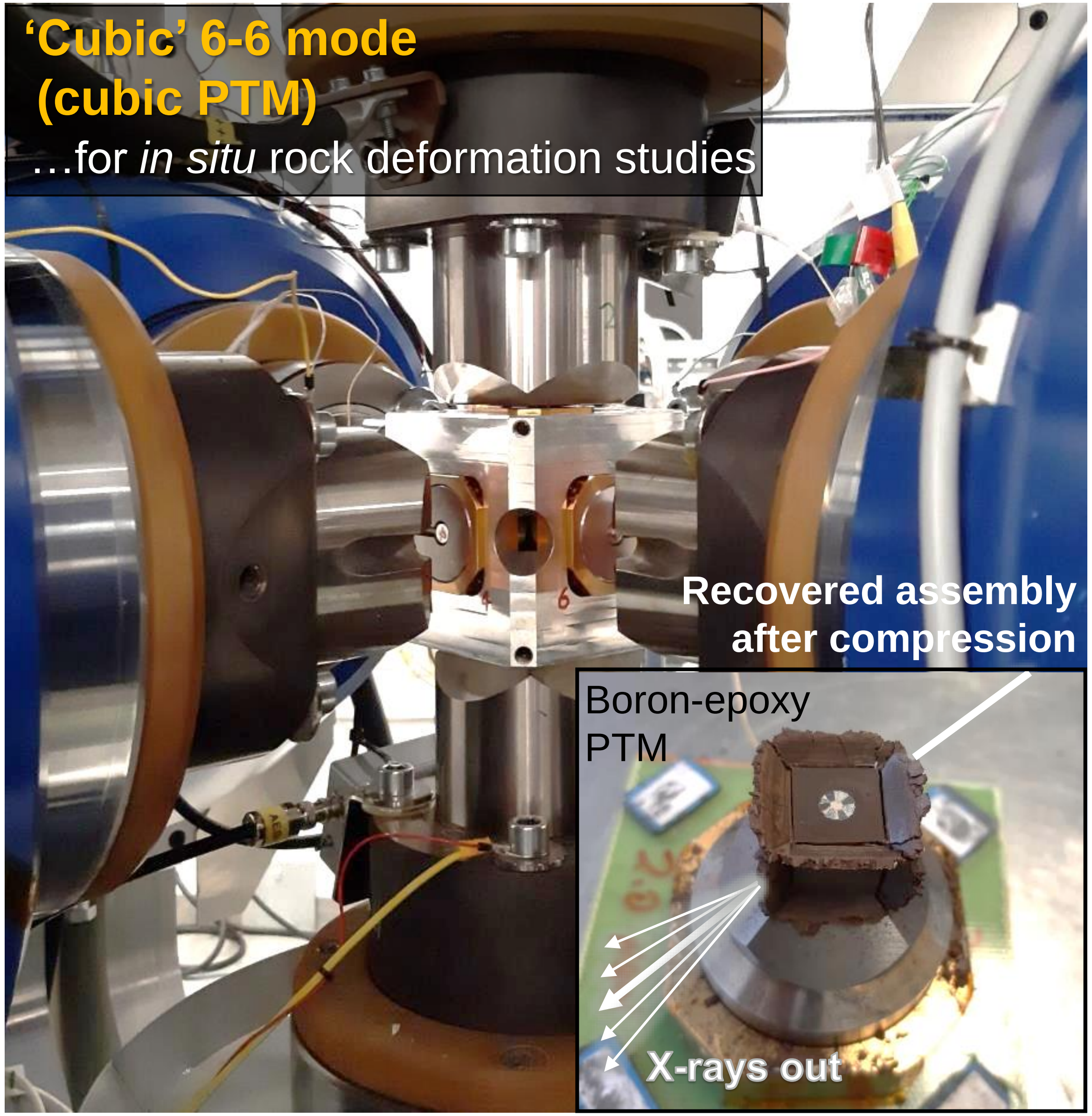
AC heating system, 3.3 kW (BGI)

DC heating system, 10 kW (in house)

Glovebox (RÅC/BMBF # 05K20OLA)

High-pressure techniques

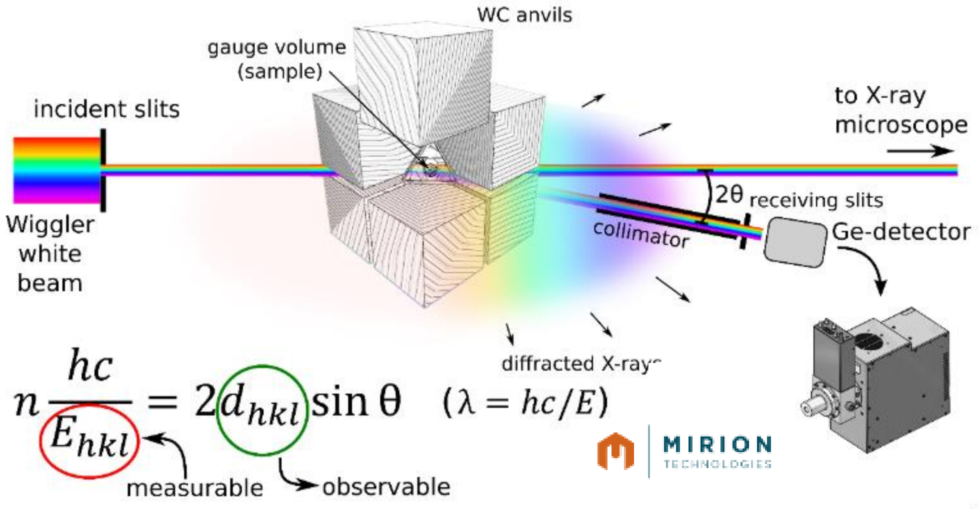




X-ray techniques using white beam

ED-XRD and Absorption Contrast Imaging in the Large Volun

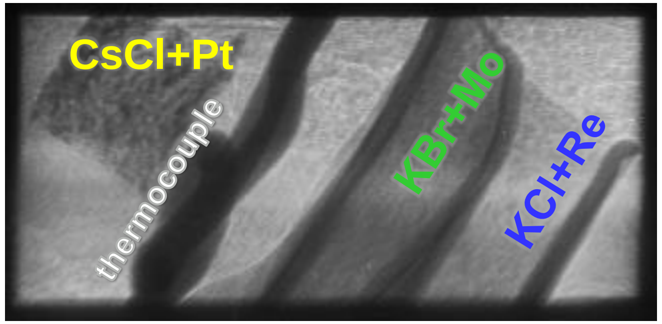
1. High spatial resolution (defines a gauge volume)
   → avoid high temperature & pressure gradients
   → no diffracted X-rays from sample environment
2. Fast acquisition (can be < 10 s), large Q-range (12 Å^{-1})



Experimental procedure:

Radiography image

1 mm (anvil gap)



X-ray microscope

pco. **edge 5.5MP**
100 Hz (full res)
1 kHz (ROI)

Optique Peter
OPTICAL & MECHANICAL ENGINEERING

Two objectives (5x, 10x)

Scintillators
- GGG:Eu (32 ph/keV)
- GaGG:Ce-HL (54 ph/keV)

X-ray techniques using white beam

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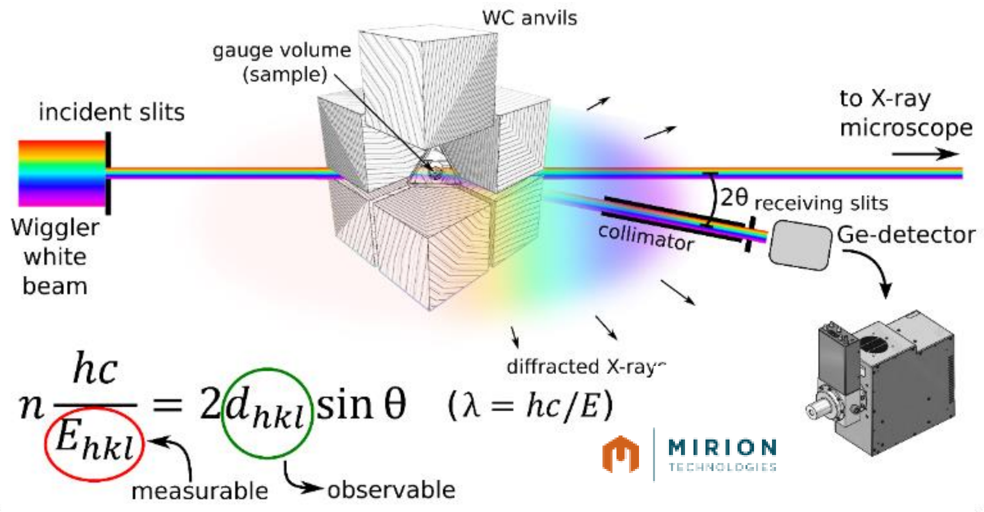
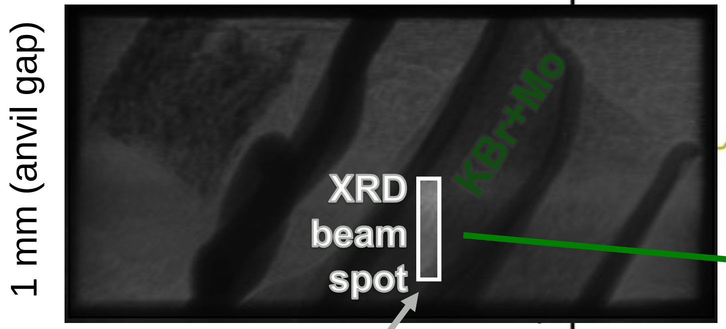
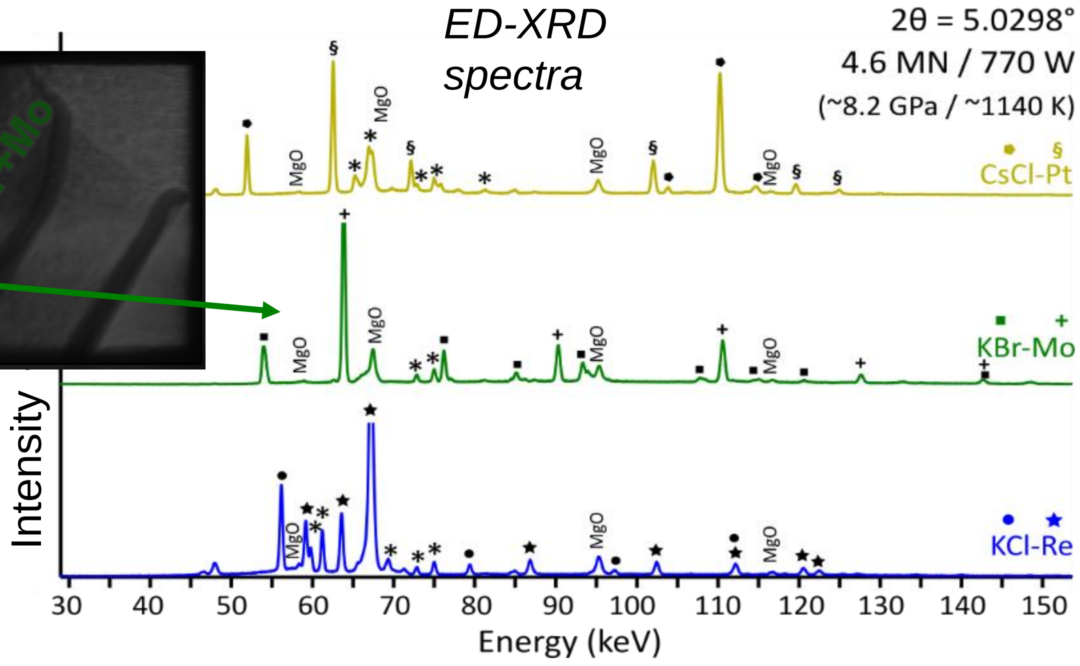


Experimental procedure:

Radiography image



Set incident slits to 50 x 300 µm^2



X-ray data analysis

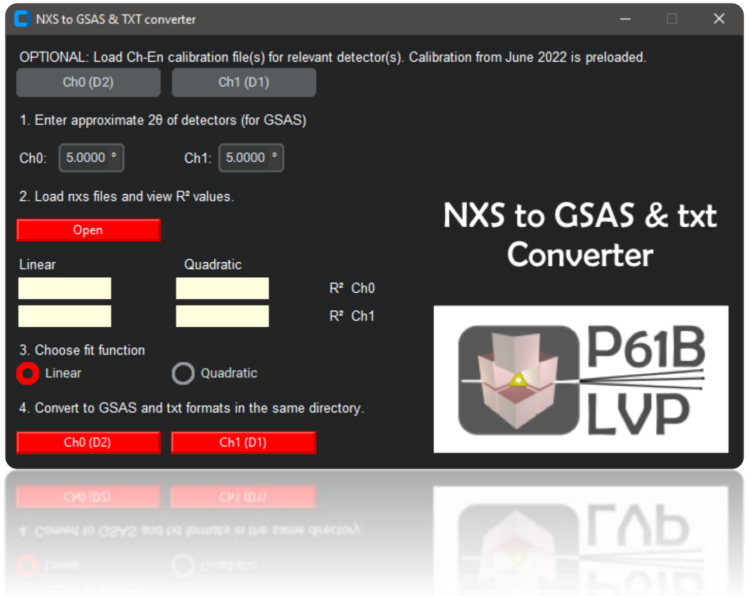
Beamline software development

- Ch2En calib. Detector energy calibration
- LaB6 2theta 2θ detector angle calibration
- NXS2 GSAS File conversion to GSAS-II / csv
- GSAS-II lst Extract GSAS data extraction tool
- NXS Frame viewer Frameviewer & exporter
- Eos Cross EosCross fit PT EoS estimation
- NXS 2 Qhkl Stress (lattice strain) calculator
- Aster-15 Aster-15 Log viewer and profile maker
- Acoustic Emissions Detection tools

X-ray data analysis

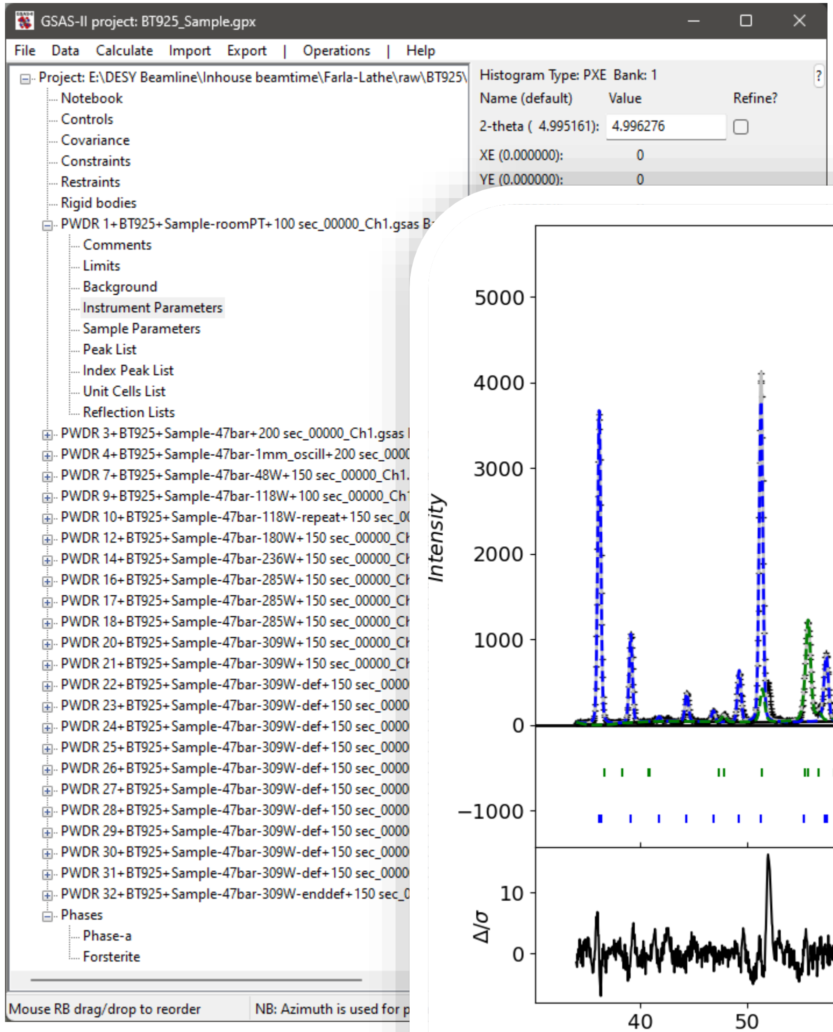
Beamline software development

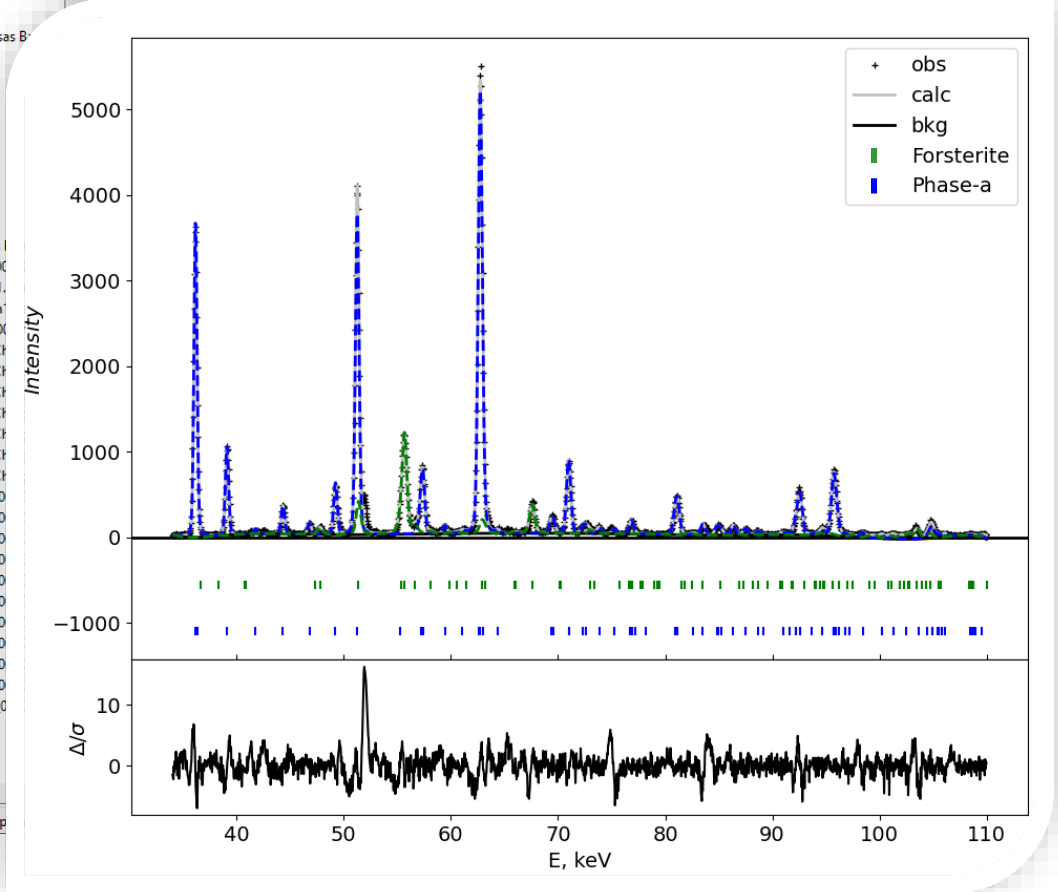
- File conversion to GSAS-II / csv
- GSAS data extraction tool



I prepared a guide for Le Bail full profile refinement of ED-XRD data (on the website)

Please consider using these tools and GSAS-II to process & analyse data:





- Unit cell volume of phases
- Density of phases
- Phase proportions*

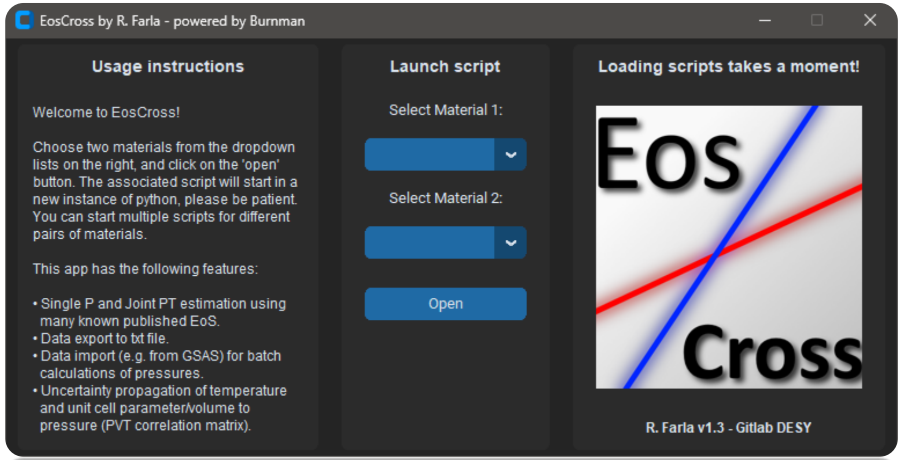
* by relative intensity changes

X-ray data analysis

Beamline software development

BurnMan
A thermodynamic and geophysics toolkit for the Earth and planetary sciences

- EosCross fit PT EoS estimation

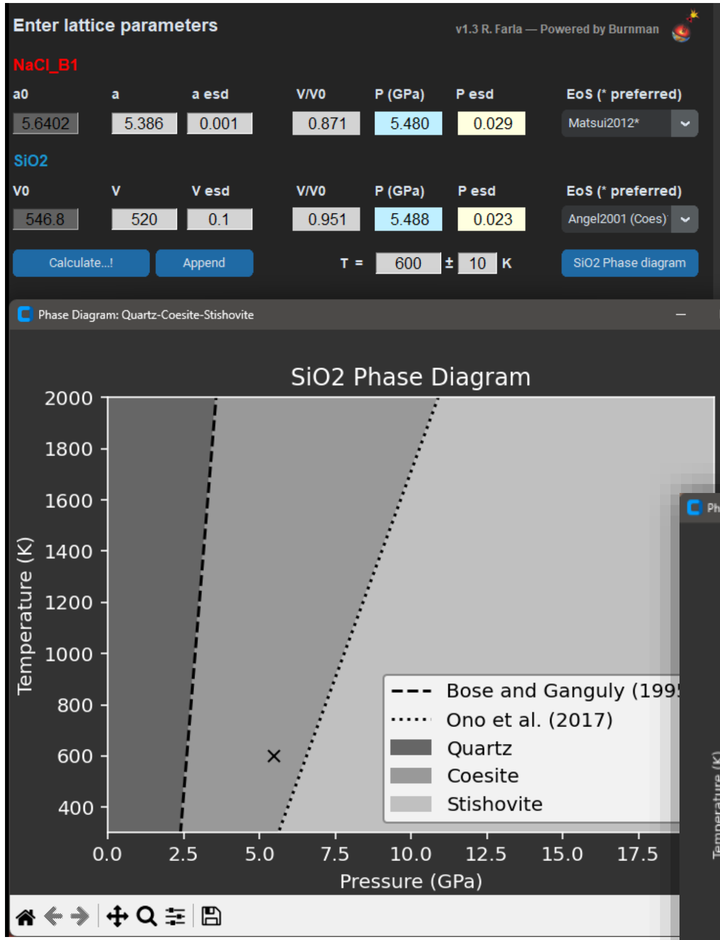


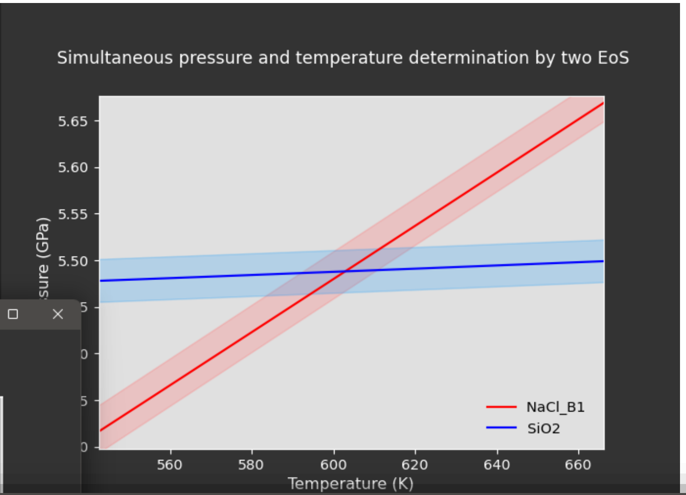
Materials 1 list:

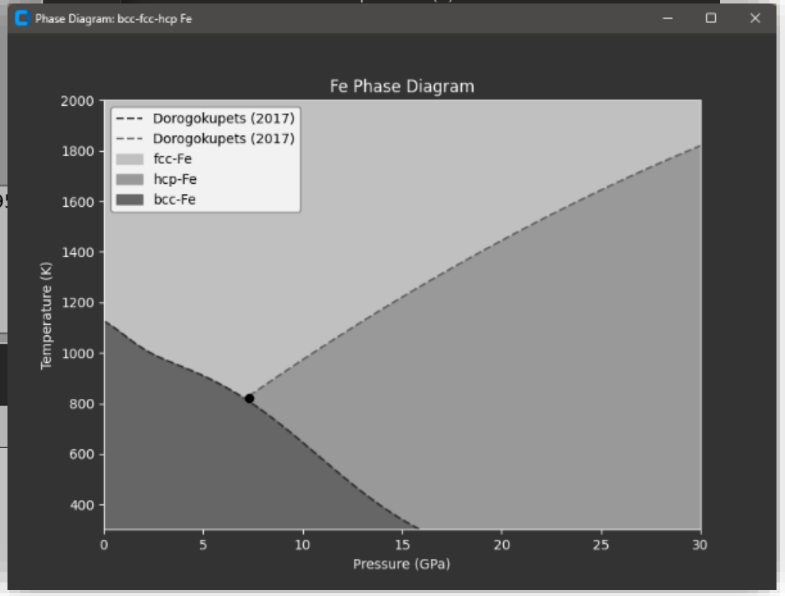
- NaCl
- KCl
- CsCl
- KBr
- hBN
- MgO
- Al2O3

Materials 2 list:

- Au
- fccCo
- Fe
- Mo
- Ni
- Pt
- Re
- W
- MgO
- SiO2





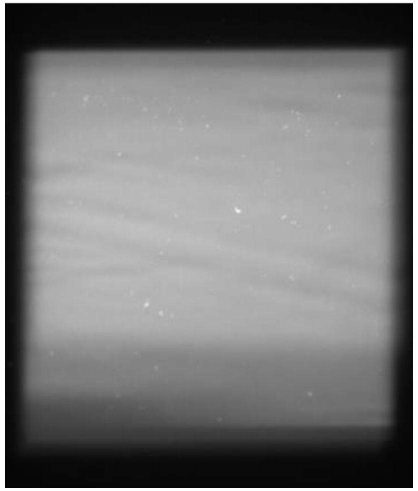


EosCross: Joint P & T estimation
(Farla *J. Syn. Rad.* 2023)
https://gitlab.desy.de/robert.farla/eoscross

Monochromator development

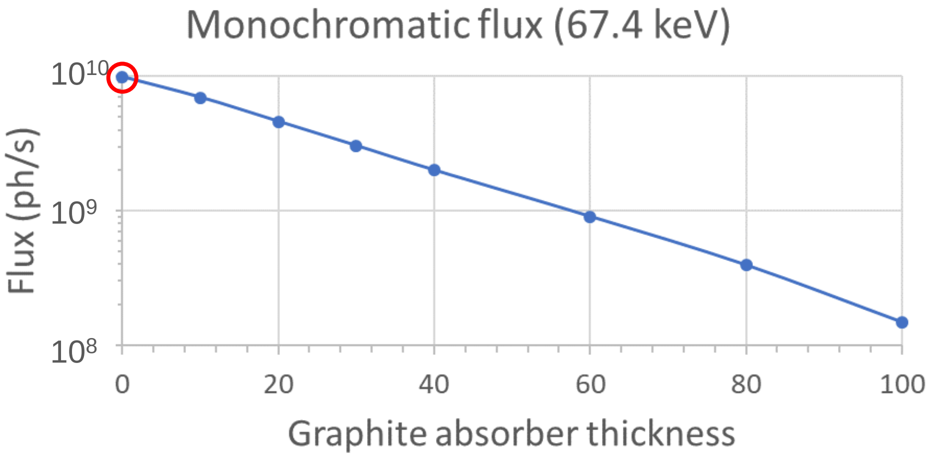
Imaging results (P61B)

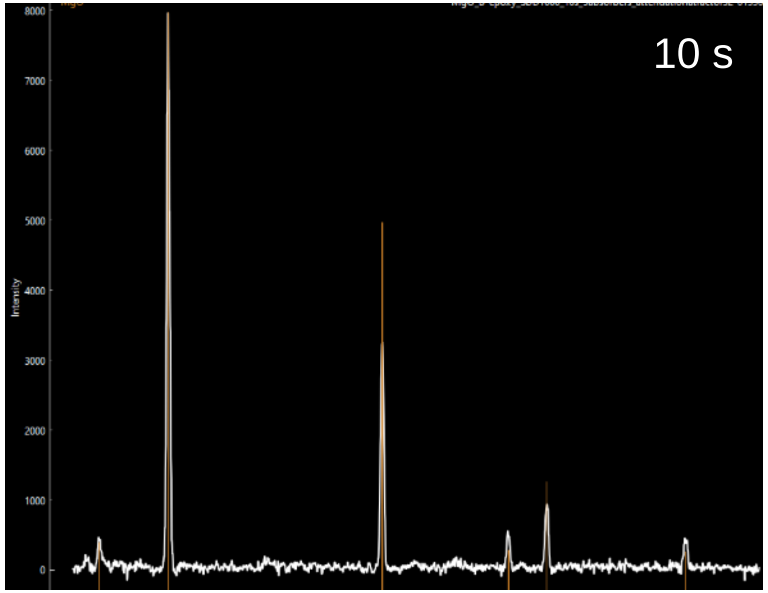
Mono beam (1 mm^2) – 10 sec



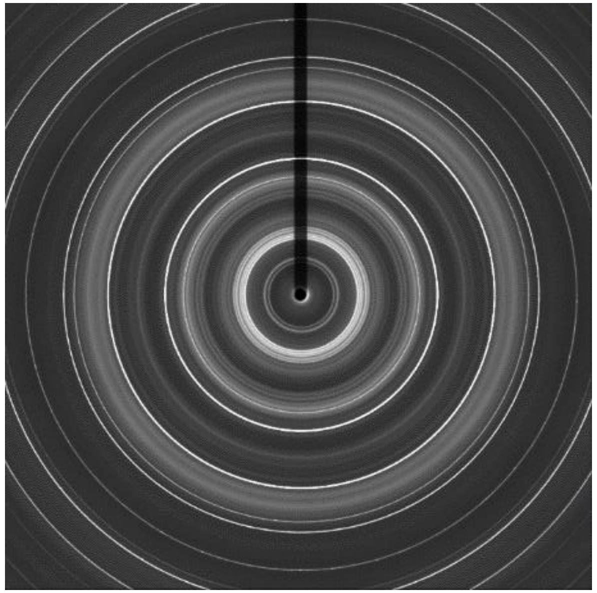
White beam (1 mm^2) – 0.03 sec

Si diode measurement (P61B)



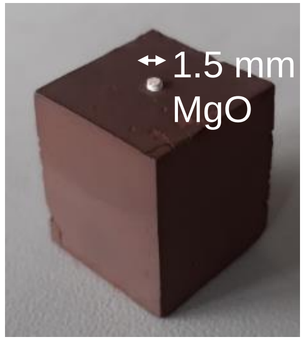


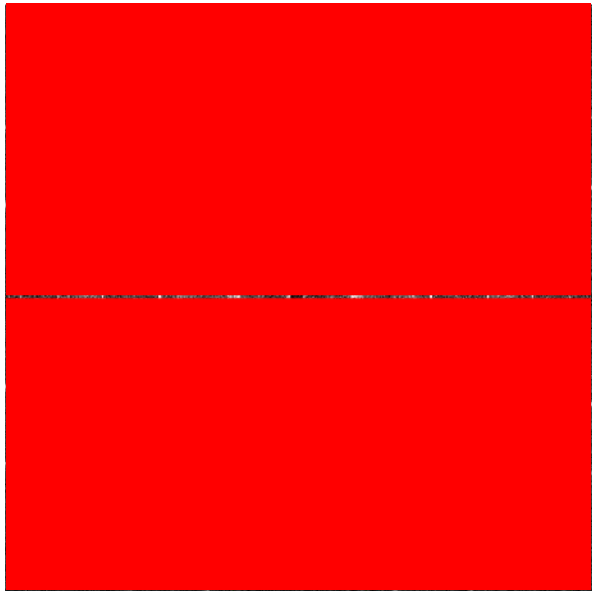
P02.1 test (60 keV, 1 m distance)



No filters
10 s acquisition

~4x10^{10} ph/s/mm^2





32x absorption
10 s acquisition

~1.2x10^9 ph/s/mm^2

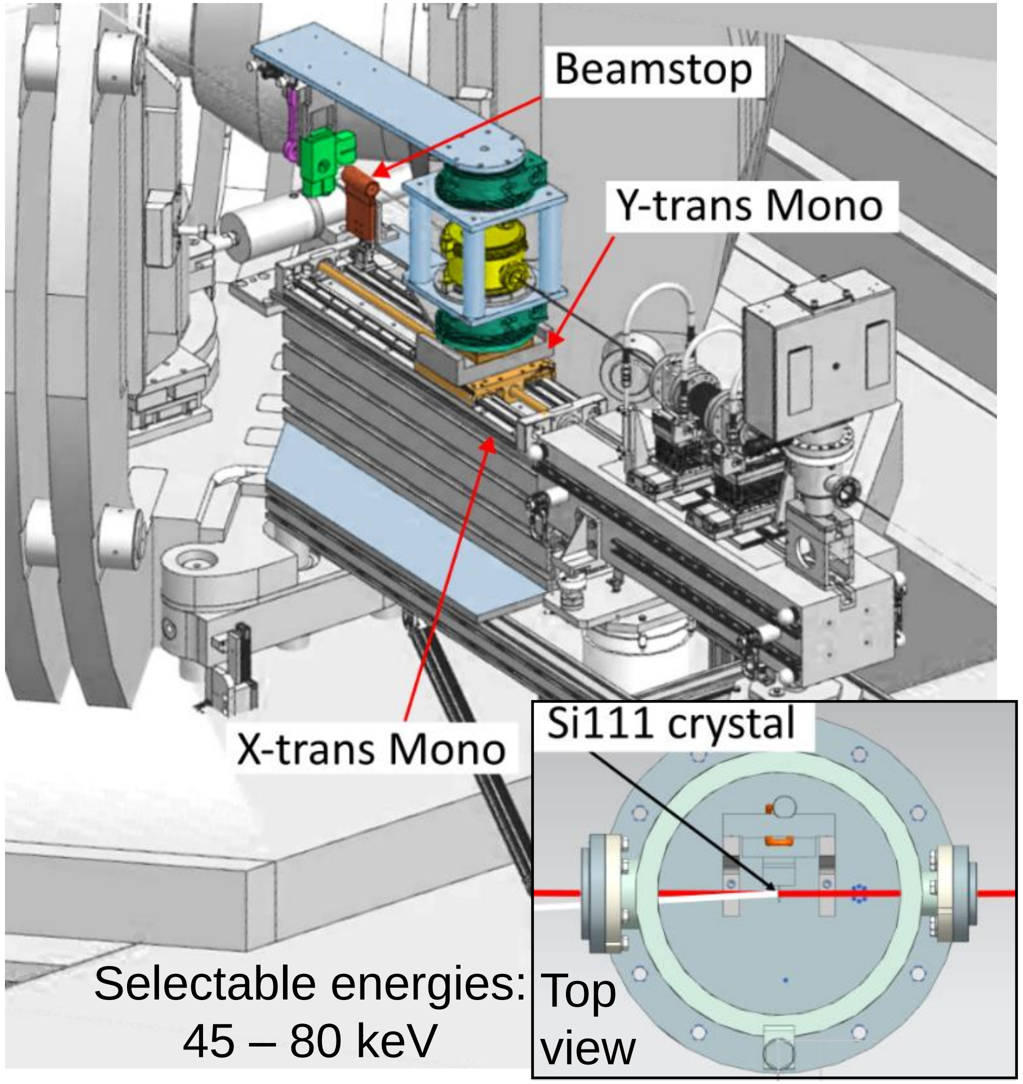
‘Anvil gap’

(‘worst’ case scenario)

Monochromator development at P61B (PETRA III)

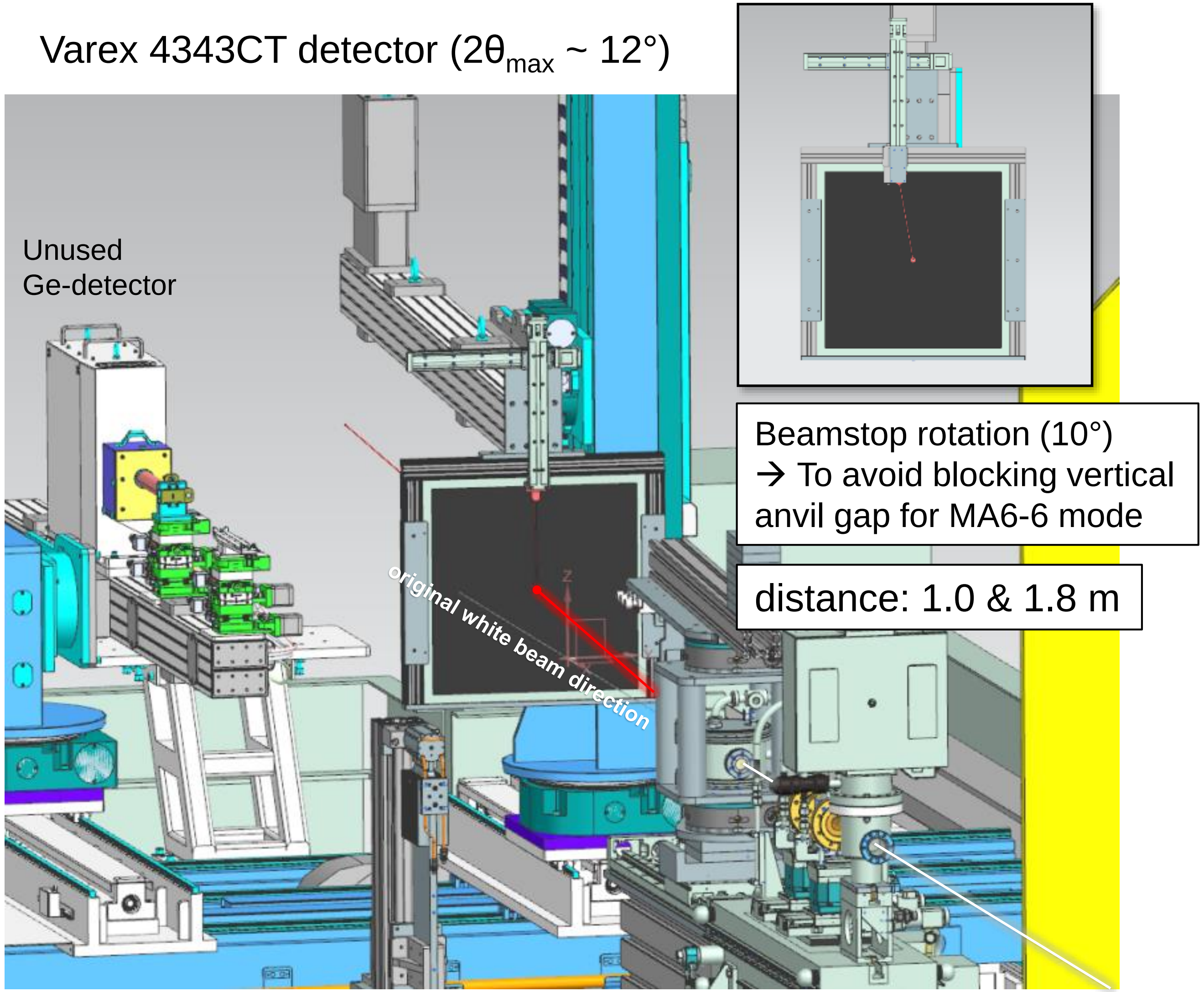
Monochromator design

Design: Stefan Sonntag



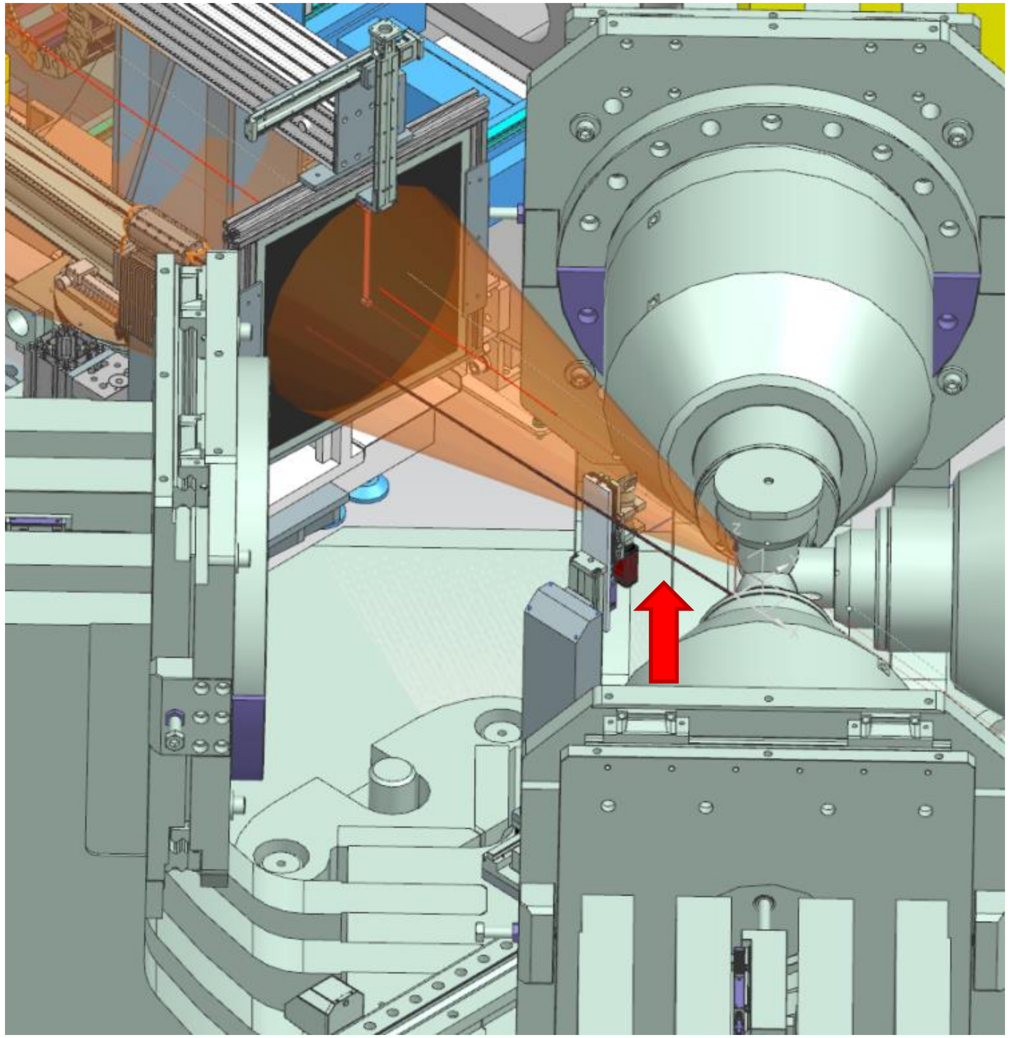
Diffraction operation (AD-XRD)

Varex 4343CT detector ($2\theta_{max}$ ~ 12°)

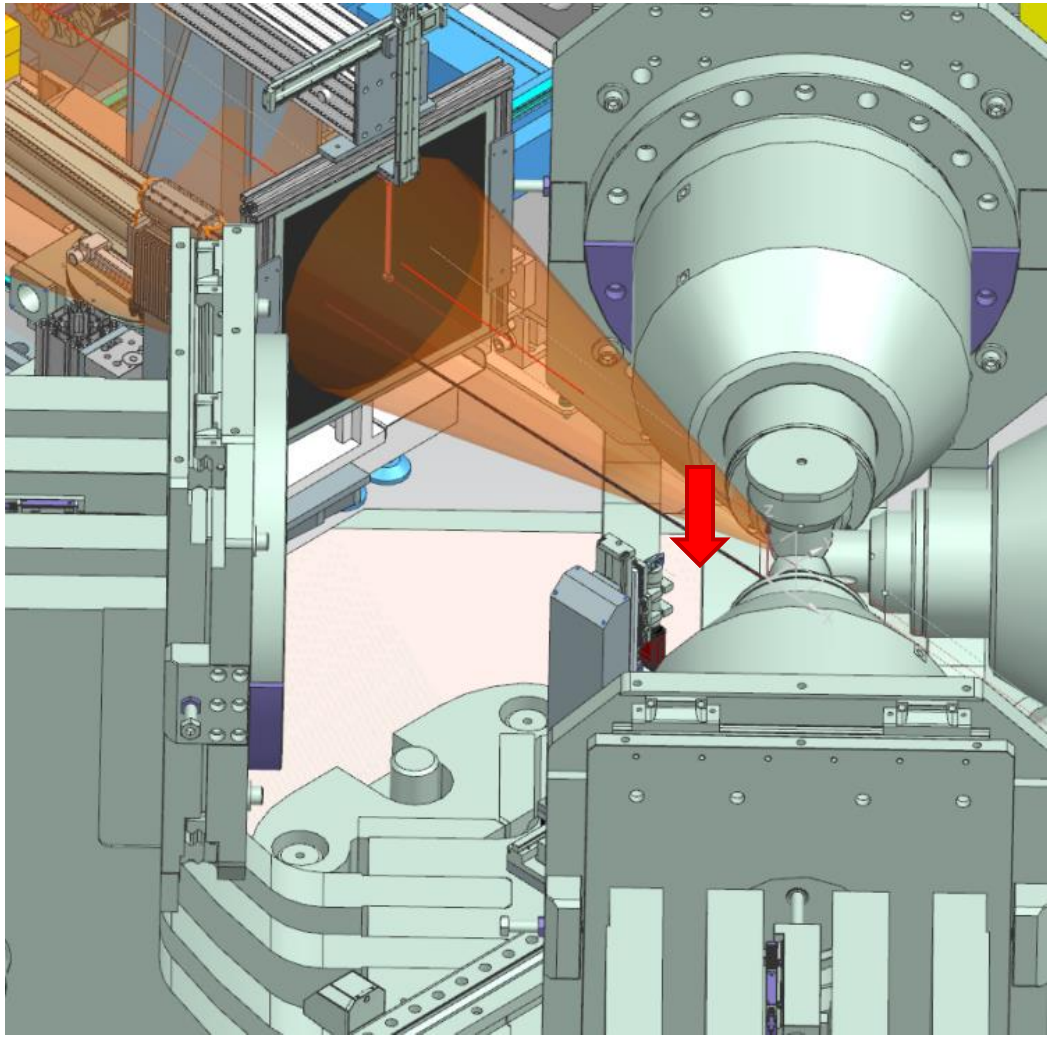


Monochromator development at P61B (PETRA III)

Imaging operation – using an X-ray eye before the detector for fast mode switching



Imaging position



Diffraction position

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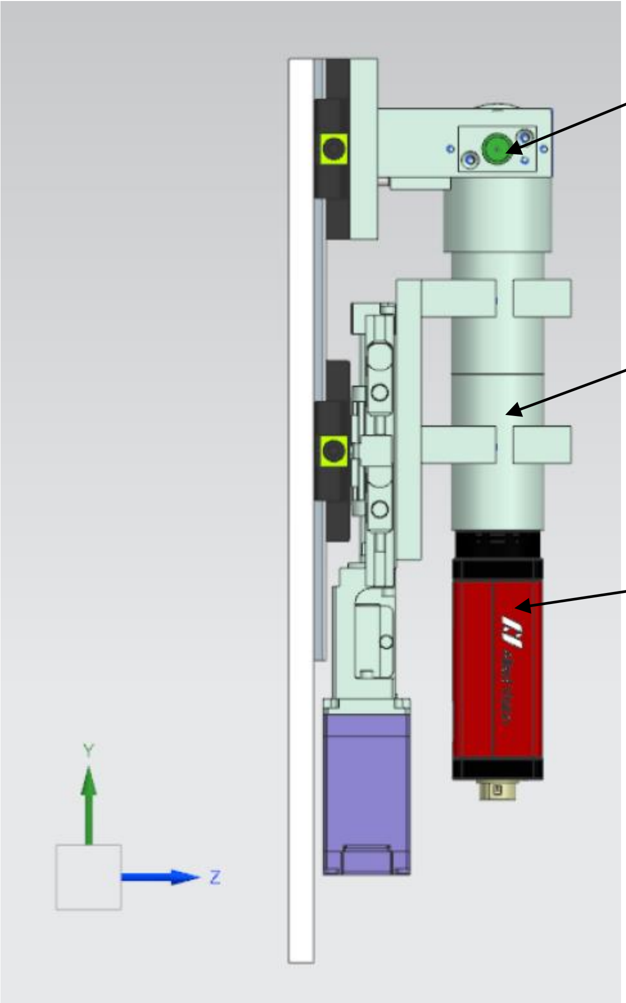

Scintillator (GAGG:Ce) & mirror

Objective
- 5x
- Resolution ~3 µm

Camera: Manta G507
- **Pixel size:** 3.45 µm × 3.45 µm
- **Resolution:** 5.1 MP: 2464 (H) × 2056 (V)
- **Sensor area:** 8.5x7.1 mm^2
→ Good for ~1 mm^2 beam (5x mag)
- **Global Shutter**
- **Frame rate:** 23 fps (full resolution)
→ Good enough for deformation exps

Commissioning of the monochromator, AD-XRD detector and imaging system planned for July 2025

If successful,
→ looking for friendly users,
→ long-term proposal (LTP) submission

Research directions

Geoscience research at P61B

PVT equation of state of (Earth) materials

- Farla. *J. Sci. Rad.* 2023
- Chanyshev *et al. Chem. Phys. Chem.* 2024

Stability of high-P hydrous minerals

- Lathe *et al. Eur. J. Min.* 2022 & 2023
- Sieber *et al. Am. Min*. 2023

P61B LVP

Deep-Earth phase relations (solid-state transformations)

- Chanyshev *et al. Contrib Mineral. Petrol.* 2021
- Xie *et al. Rev. Sci. Instrum*. 2021
- Chanyshev *et al. Nature*. 2022
- Chanyshev *et al. Earth Space Chem*. 2023

Research directions

Geoscience research at P61B

Ultrasonic wave speed in materials (seismology)

- Néri *et al. Rev. Sci. Instrum.* 2024

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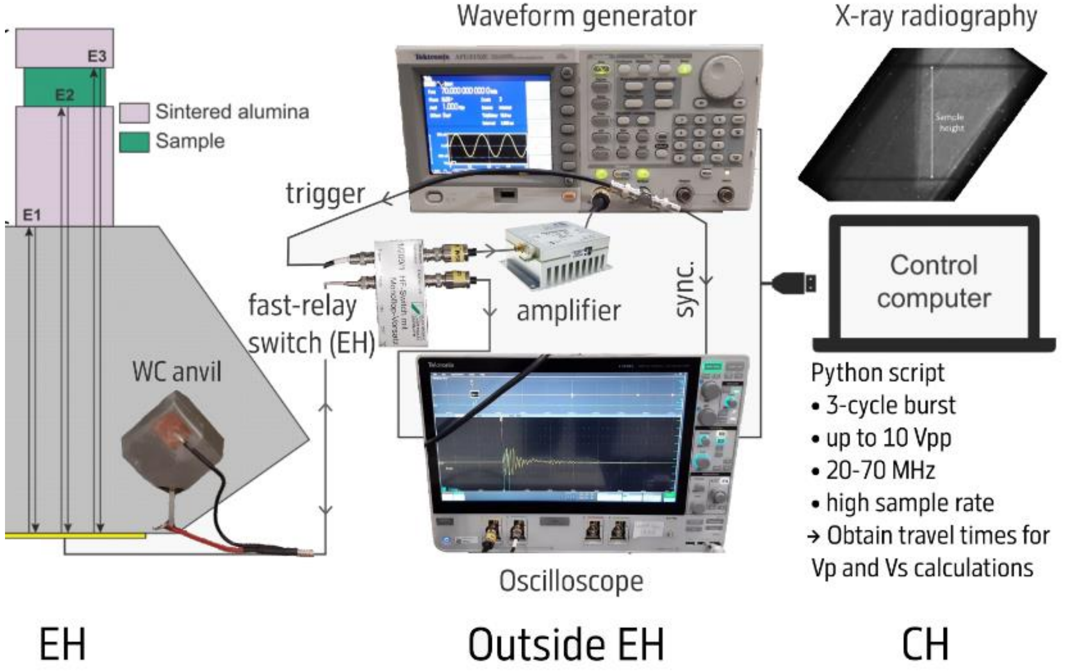

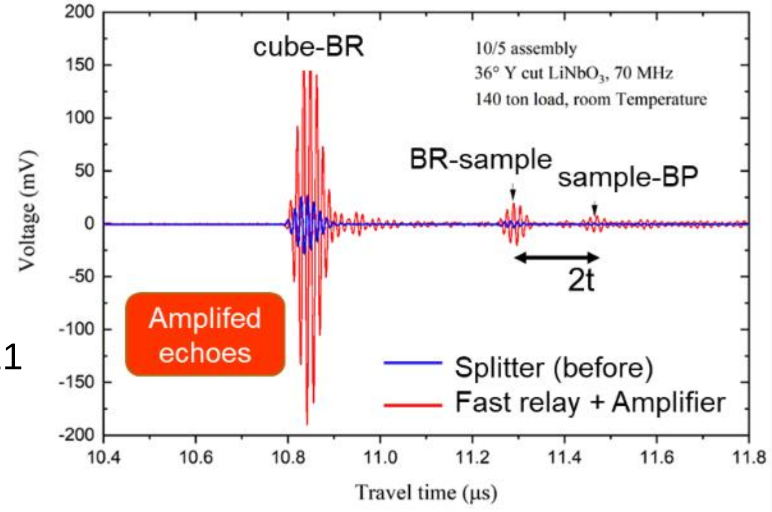

Research directions

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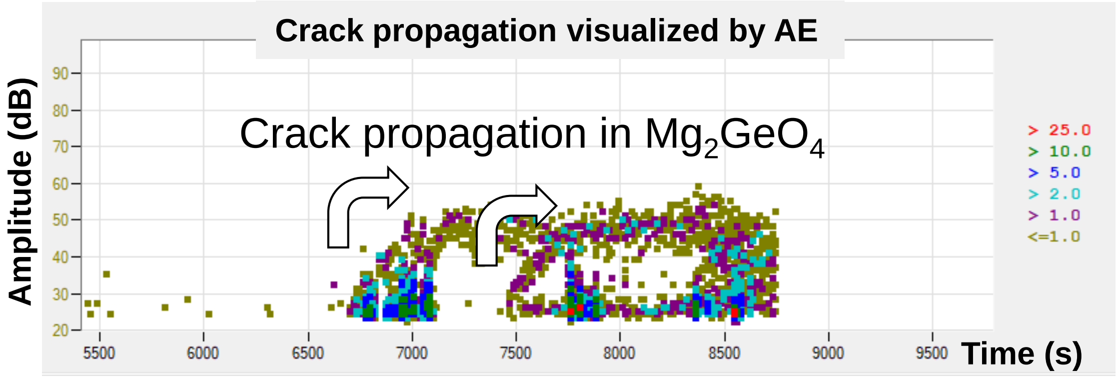

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- Chanyshev *et al. Nature.*
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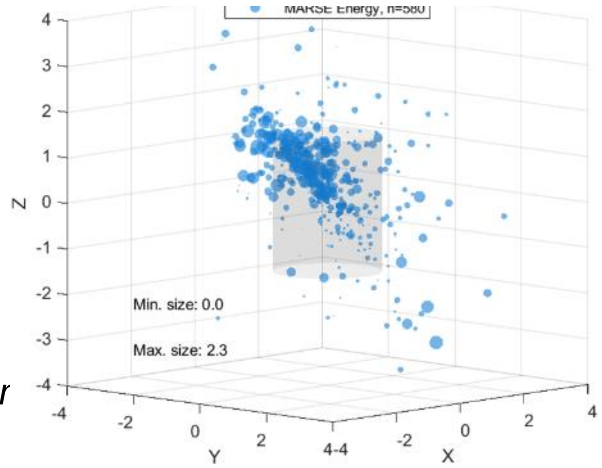
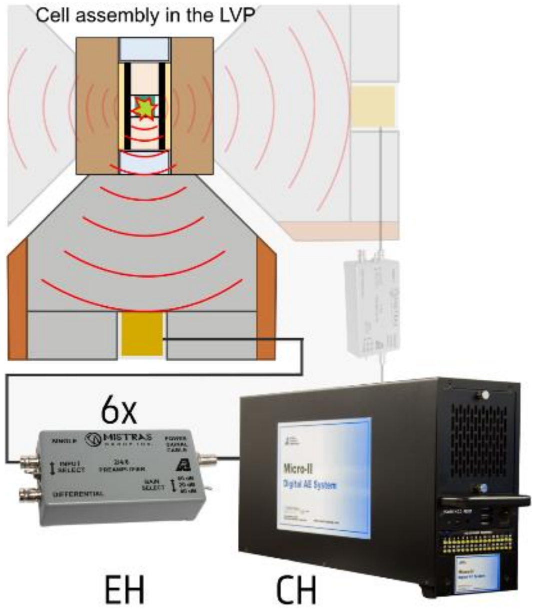
3d event relocation



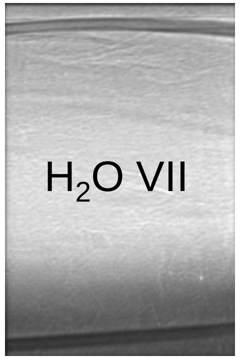


Possible transformation faulting in Ge-olivine to spinel transition

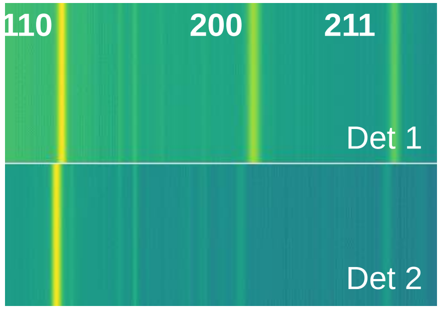
Research directions

Geoscience research at P61B

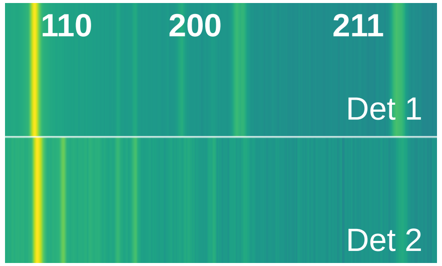
Macro-strain



Peak compressive stress



Peak tensile stress



Energy (KeV)

Ultrasonic wave speed in materials (seismology)

- Néri *et al. Rev. Sci. Instrum.* 2024

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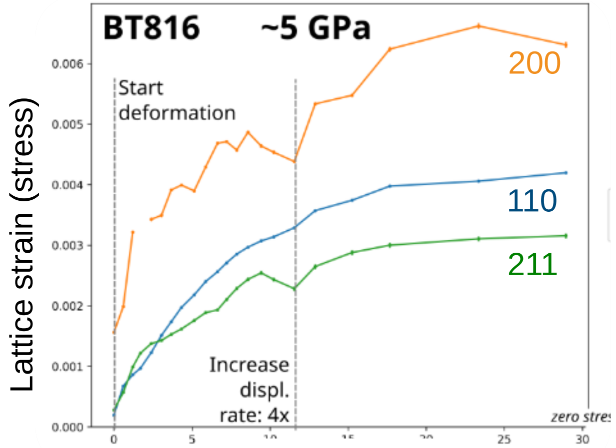
- Chanyshev *et al. Nature*. 2022
- Chanyshev *et al. Earth Space Chem*. 2023

Rheology of mantle minerals and H_2O ices

- Howard *et al.* *In progress*



Research directions

Materials research at P61B

Nanomaterials

- Gaida *et al. J. Am. Cer. Soc.* 2018

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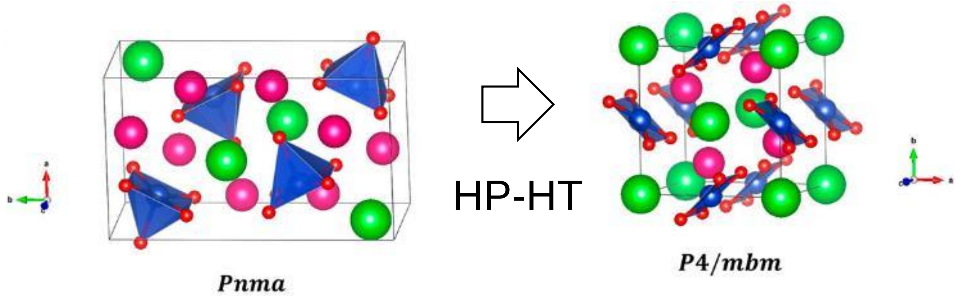
Magnetic materials

- Shanbhag *et al. Physical Review B* 2024
- Mishra *et al. Frontiers Chemistry* 2023
- Ma *et al. Nanoscale* 2021



Phase transitions of R_2BaCuO_5 (R = Sm / Eu)

Mishra *et al. Front. Chem.*, 11, 1166475, 2023

Research directions

Materials research at P61B

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Nanomaterials

- Gaida *et al. J. Am. Cer. Soc.* 2018

Nitride materials

- Gollé-Leidreiter *et al. Acta Cryst.* 2024
- Ambach *et al. Inorganic Chemistry* 2024
- Li *et al. J. Adv. Ceramics* 2023
- Bhat *et al. Scientific Reports* 2020
- Bhat et al. *Chemistry – Eur. J.* 2019

Magnetic materials

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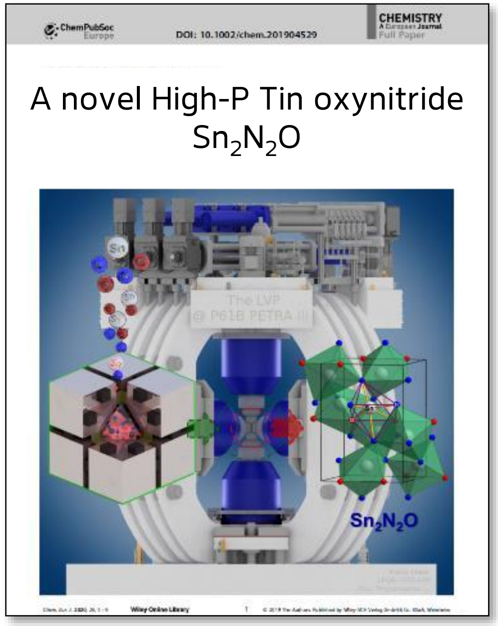
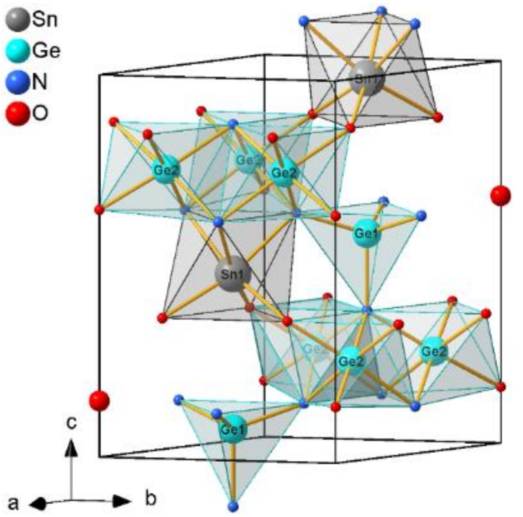
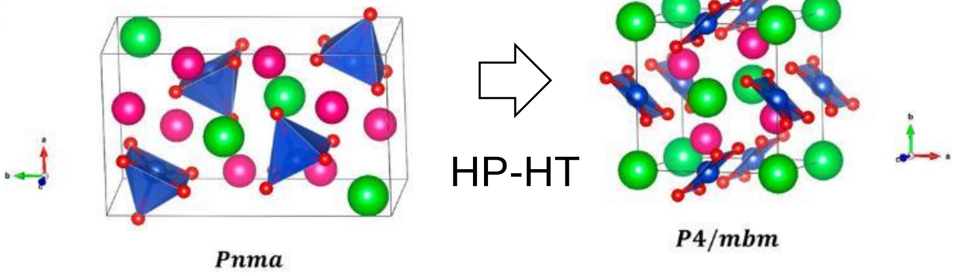
- Mishra *et al. Frontiers Chemistry* 2023
- Ma *et al. Nanoscale* 2021





Nolanite-type $SnGe_4N_4O_4$ ($P6_3mc$)
Golle´-Leidreiter *et al.* . *Acta Cryst.* (2024)

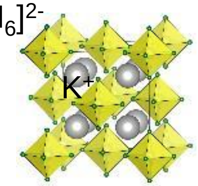


Phase transitions of R_2BaCuO_5 (R = Sm / Eu)

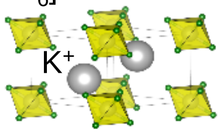
Mishra *et al. Front. Chem.*, 11, 1166475, 2023

Research directions

Materials research at P61B



cubic K_2SiH_6
< 7 GPa



270-725 °C:
Trigonal K_2SiH_6
8 - 13 GPa

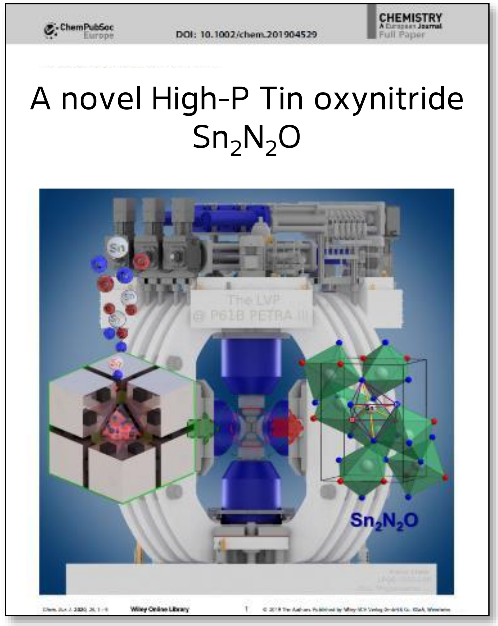
P61B LVP

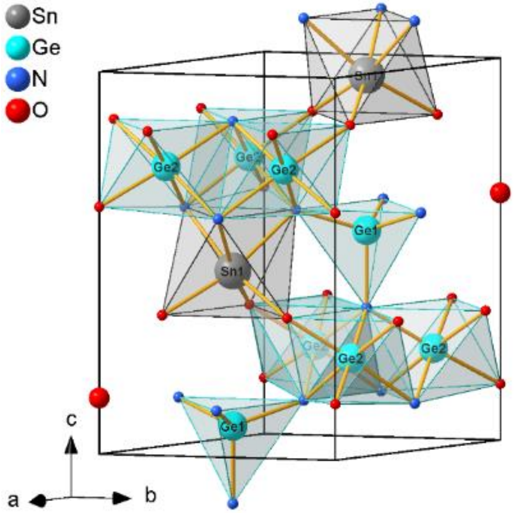
Hydrogenation synthesis

- Vekilova *et al. Inorganic Chemistry* 2023
- Spektor *et al. Frontiers Chem.* 2023

Nanomaterials

- Gaida *et al. J. Am. Cer. Soc.* 2018





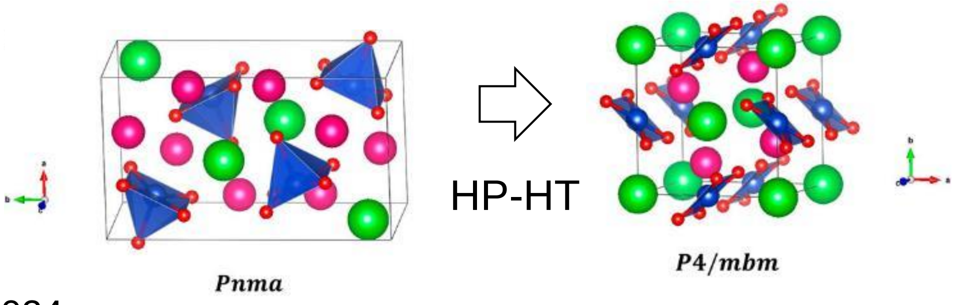
Nolanite-type $SnGe_4N_4O_4$ ($P6_3mc$)
Gollé-Leidreiter *et al.* . *Acta Cryst.* (2024)

Nitride materials

- Gollé-Leidreiter *et al. Acta Cryst.* 2024
- Ambach *et al. Inorganic Chemistry* 2024
- Li *et al. J. Adv. Ceramics* 2023
- Bhat *et al. Scientific Reports* 2020
- Bhat et al. *Chemistry – Eur. J.* 2019

Magnetic materials

- Shanbhag *et al. Physical Review B* 2024
- Mishra *et al. Frontiers Chemistry* 2023
- Ma *et al. Nanoscale* 2021



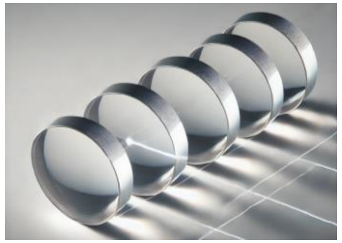
Phase transitions of R_2BaCuO_5 (R = Sm / Eu)

Mishra *et al. Front. Chem.*, 11, 1166475, 2023

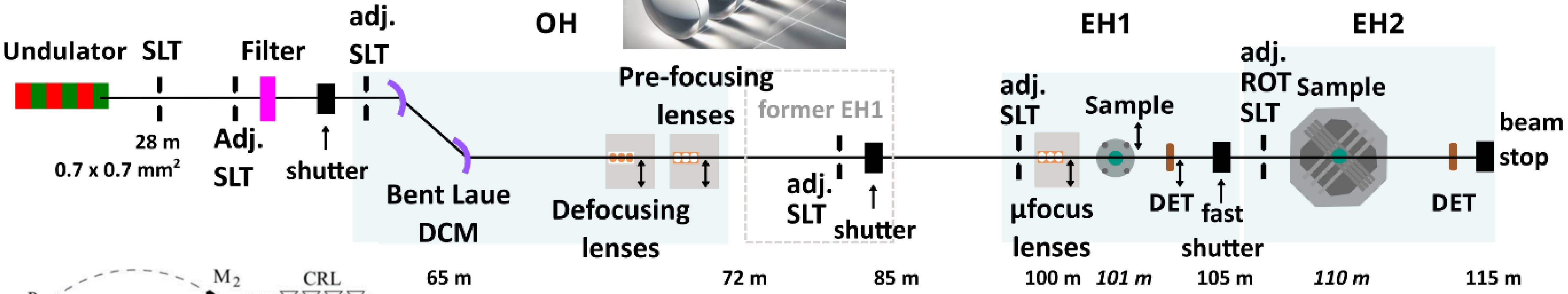
The *in situ* Large Volume Press Beamline at PETRA IV

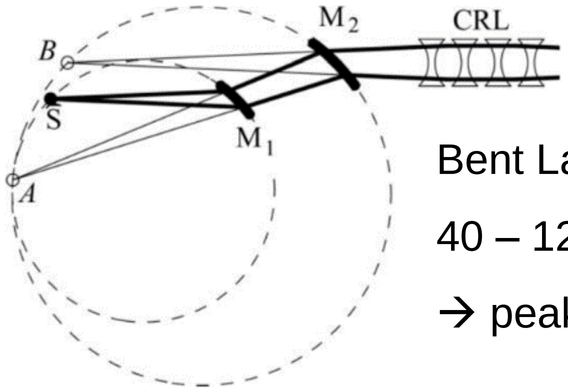
LVP-XPRESS: X-ray Probe for Research in Extreme Synthesis and planetary Studies

The new beamline (still at P61, PXN) will use monochromatic high-energy beams and new optics



Diamond Compound Refractive Lenses





Bent Laue Double Crystal Monochromator

40 – 120 keV

→ peak flux at 6 (odd) harmonics

PETRA IV. NEW DIMENSIONS 2032+

The *in situ* Large Volume Press Beamline at PETRA IV

LVP-XPRESS: X-ray Probe for Research in Extreme Synthesis and planetary Studies

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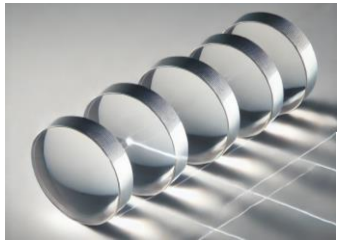
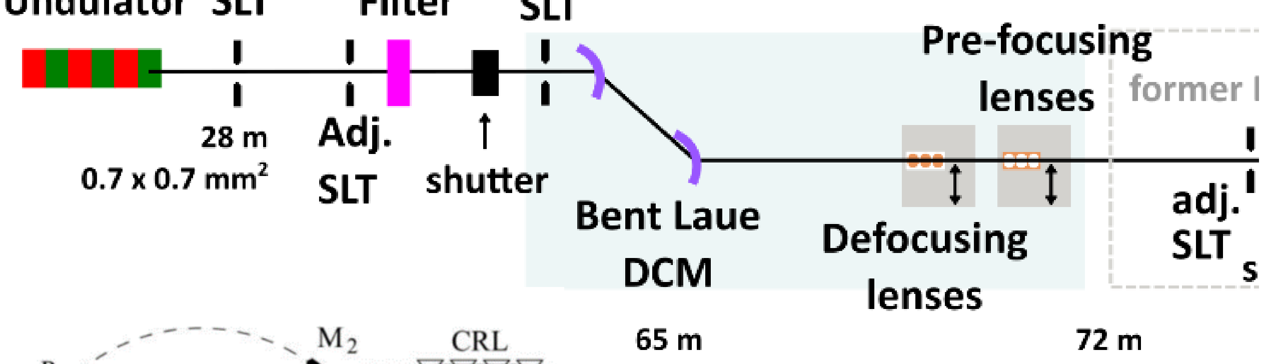
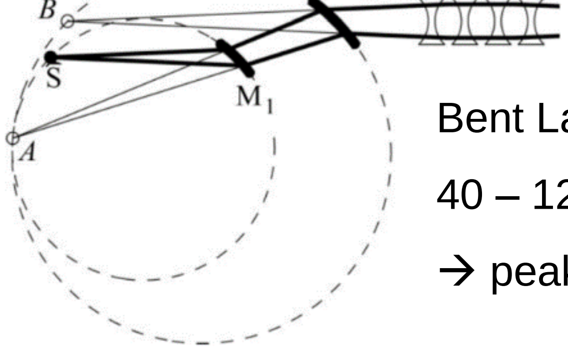


Diamond Compound Refractive Lenses





Bent Laue Double Crystal Monochromator

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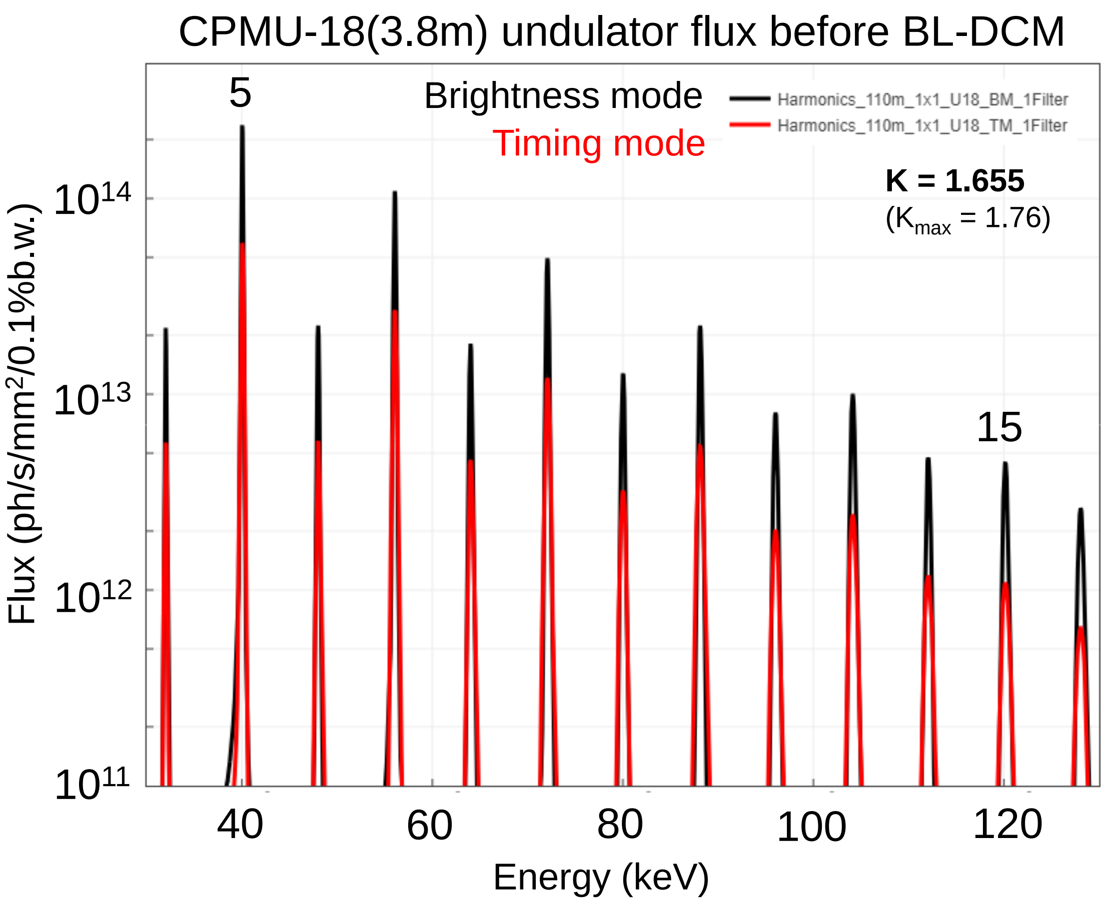


The *in situ* Large Volume Press Beamline at PETRA IV

LVP-XPRESS: X-ray Probe for Research in Extreme Synthesis and planetary Studies

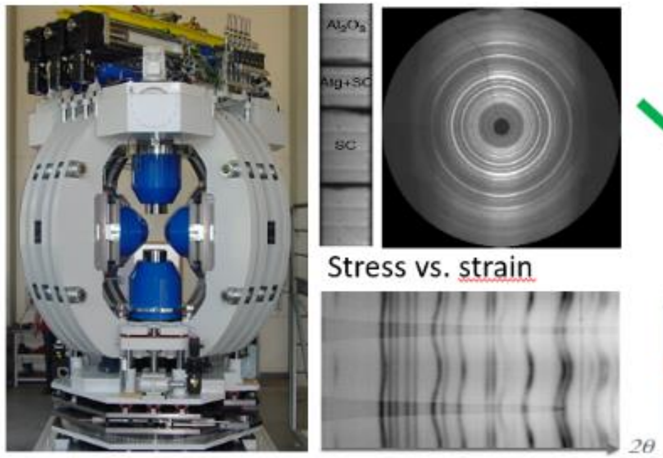
The new beamline will accommodate **multiple LVPs** and a larger portfolio of *in situ* HPHT X-ray techniques

- **AD-XRD (Powder/Single-crystal/Multi-grain/PDF/Rietfeld), ED-XRD (?)**
- **Absorption & Phase Contrast radiography, high-frame rate**
- **Absorption contrast µ-tomography, diffraction-scattering computed tomography (DSCT), time-resolved, ...**

Multianvil (6-rams)
Aster 15

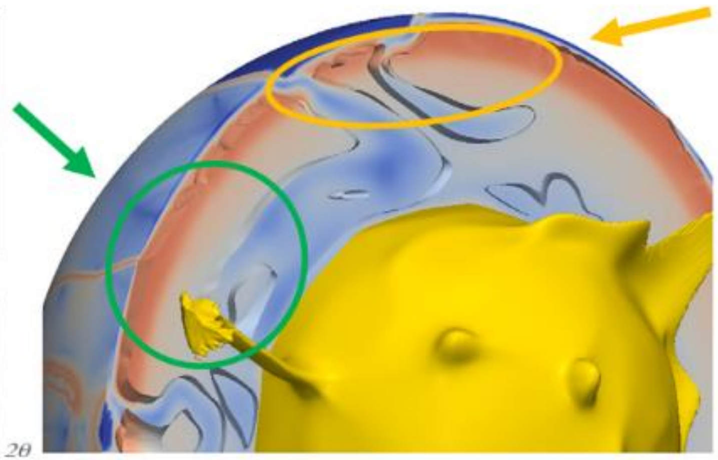
EH2

Angle-dispersive XRD - Structure refinement



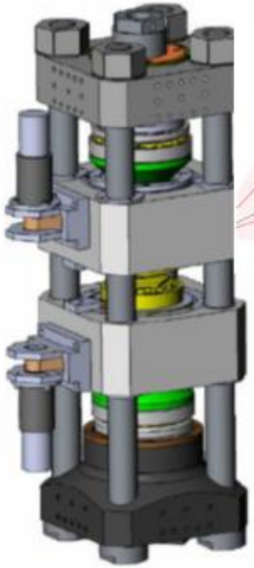
Earth's convection & plate tectonics

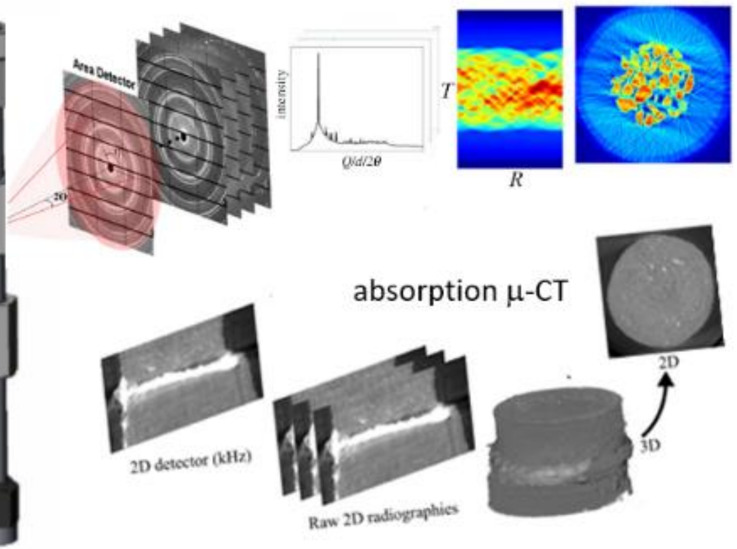
@ LVP-XPRESS



Paris-Edinburgh LVP

EH1





PETRA IV. NEW DIMENSIONS 2032+

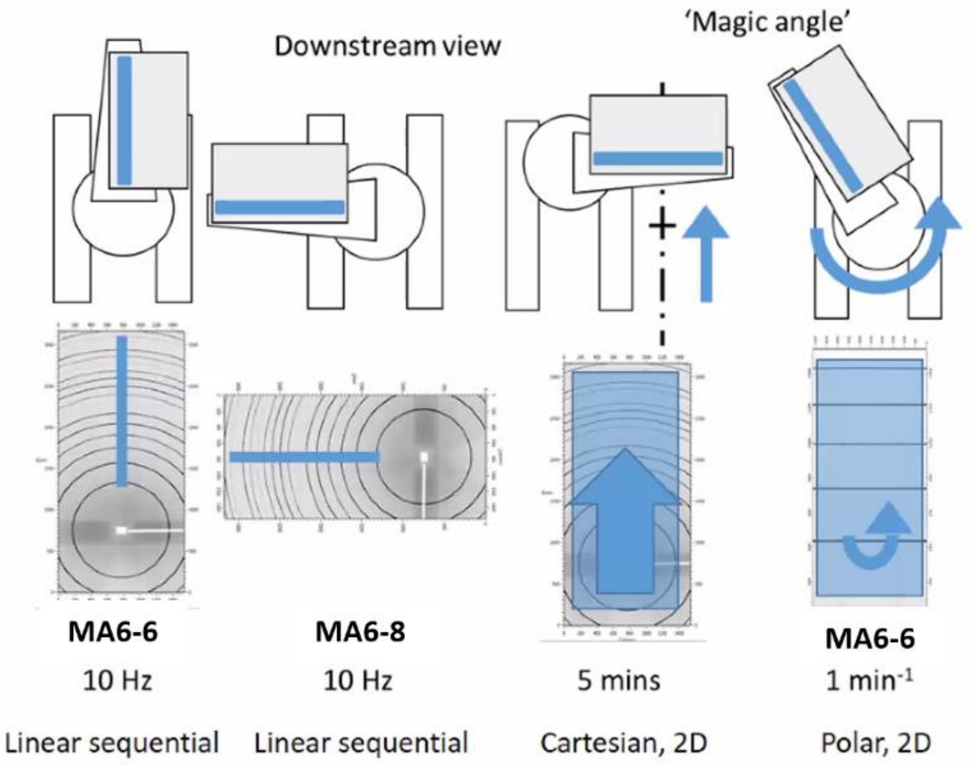
The *in situ* Large Volume Press Beamline at PETRA IV

LVP-XPRESS: X-ray Probe for Research in Extreme Synthesis and planetary Studies

The new beamline will operate using the latest detector technologies and systems

- **Photon-counting efficiency at high energies** – CdZnTe
- **Small pixel size for high spatial resolution** – e.g. 55 µm
- **Large radial coverage, access to high Q-range** – 700 mm
- **High acquisition rates** – typical 10 Hz, up to 200 Hz
- **Gapless technology between modules**
- **High bit rate (counting depth)** – good for weak scattering
- **Versatile detector positioning** – 1.5 m to 4 m from sample
- **Goniometer rotation** akin to ID06 LVP (ESRF-EBS)

- High-speed (1 – 10 kHz), sCMOS cameras for absorption/phase contrast imaging



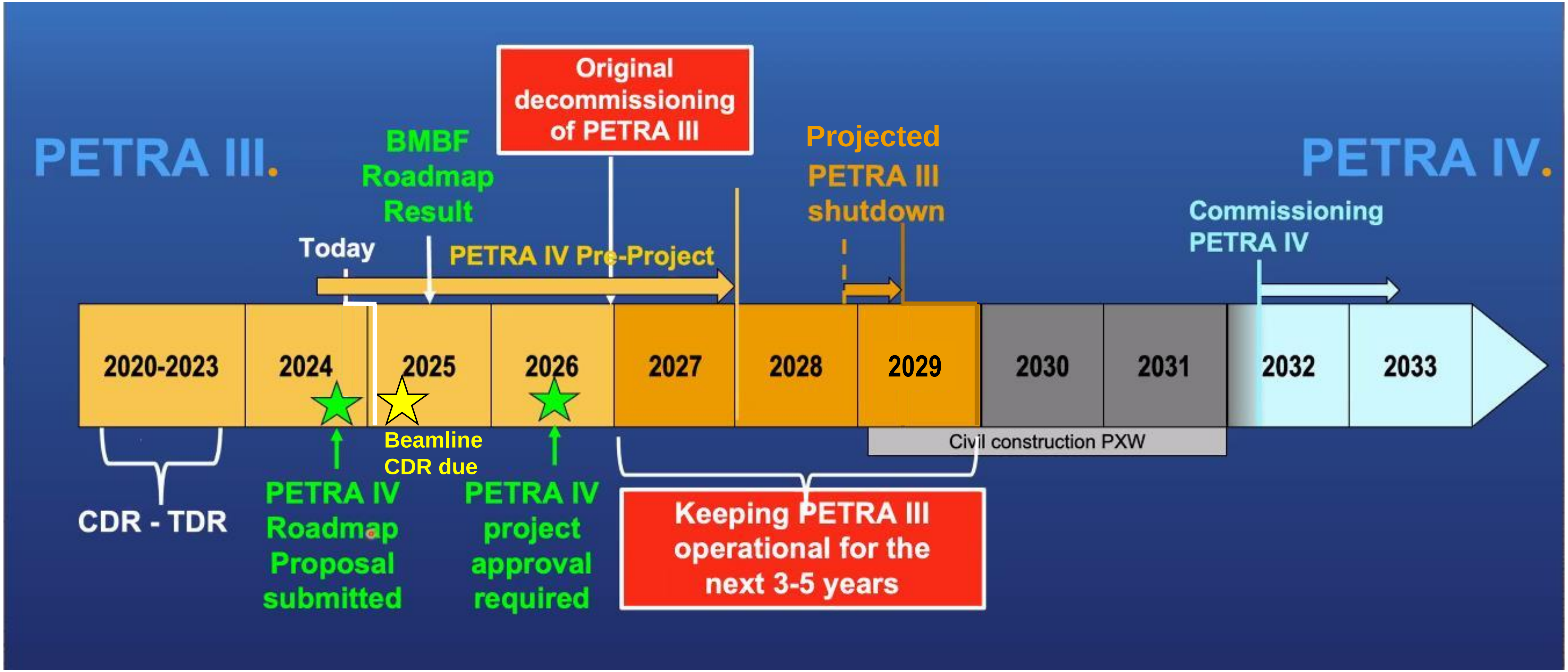
Diffraction detector geometries for different types of LVP experiments (courtesy of W. Crichton)

PETRA IV. NEW DIMENSIONS 2032+

The *in situ* Large Volume Press Beamline at PETRA IV

LVP-XPRESS: X-ray Probe for Research in Extreme Synthesis and planetary Studies

The new beamline is a **Phase-I beamline** at PETRA IV:



Summary

Dedicated user operation at P61B

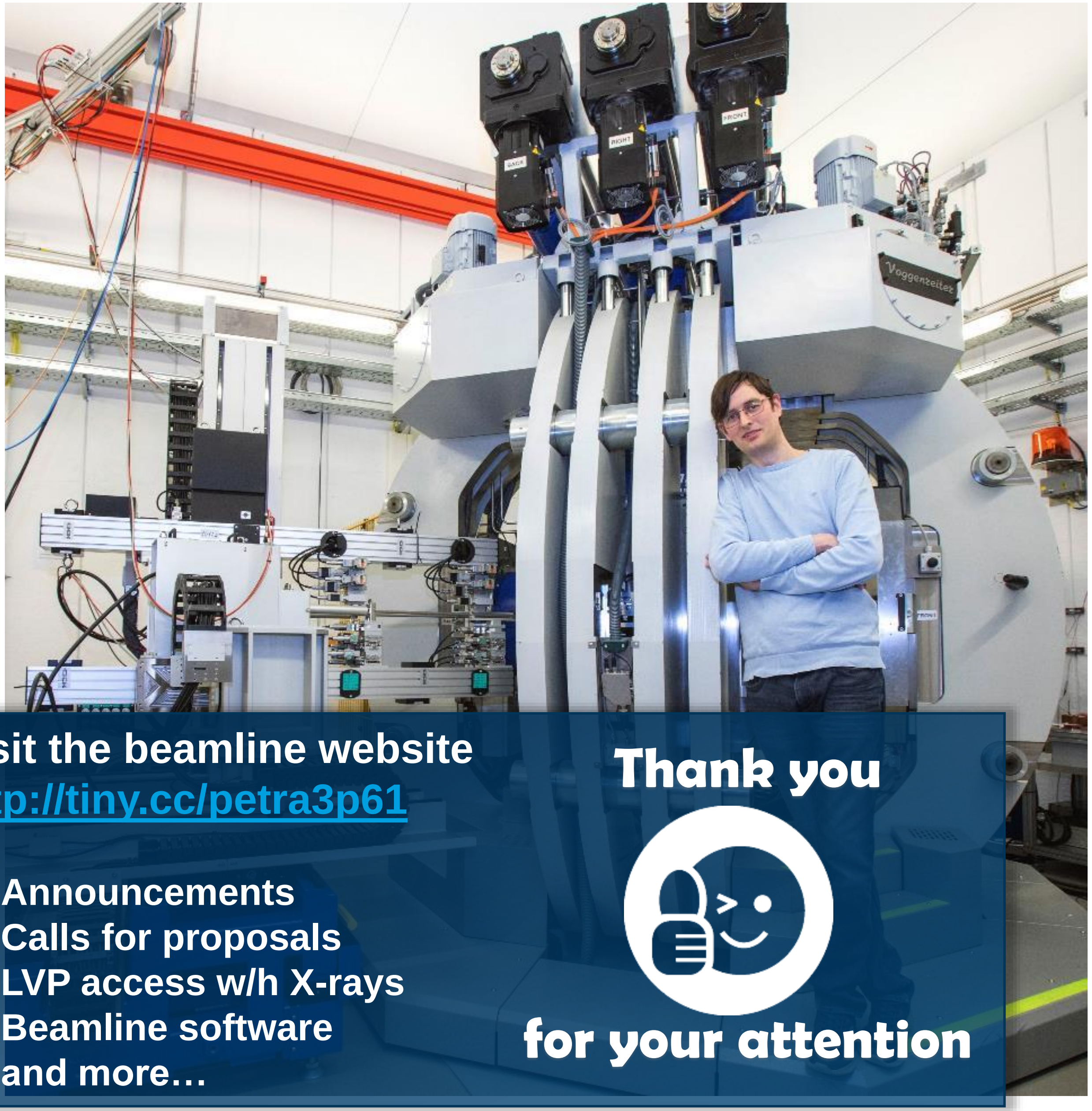
- LVP ready for *in situ* and *ex situ* experiments over wide range of HPHT conditions.
- Ge-detectors provide excellent XRD data quality, high count rate (> 1 Mcps), low acquisition time.
- Operation with user-friendly GUIs and tools.

Support for special *in situ* experiments

1. Controlled rock deformation (2 Ge-SSD)
2. Acoustic Emissions (AE) w/ deformation
3. Ultrasonic wave speed measurements (using 26 mm or 32 mm WC anvils)
4. Falling sphere viscosimetry (w/ GaGG:Ce scintillator)

Commissioning in 2025

1. Monochromator / ADXRD system
2. Impedance analyser (electrical cond.)



Visit the beamline website
http://tiny.cc/petra3p61

- **Announcements**
- **Calls for proposals**
- **LVP access w/h X-rays**
- **Beamline software**
- **and more…**

Thank you for your attention

Acknowledgments

Bayerisches Geoinstitut (BGI)
Prof. Katsura
Prof. Frost
Dr. Boffa Ballaran
Dr. Chanyshev
Dr. Néri (now @ Lille, France)
Dr. Withers
Mr. Lianjie
Mr. Dolinschi
…and others.

Stockholm University
Prof. Haeussermann

Leipzig University
Prof. Kohlmann
Dr. Beyer

Potsdam University
Dr. Sieber

GFZ Potsdam
Prof. Koch-Mueller
Prof. Sergio Speziale
Dr. Lathe

Ningbo University, China
Dr. Ma

At the beamline:
Dr. Bhat
Dr. Spektor
Dr. Feng

GSECARS, APS, ANL
Prof. Wang
Dr. Officer
Dr. Yu

Ecole Normale Supérieure PSL University (Paris)
Prof. Schubnel
Dr. Gasc
Ms. Mingardi

TU Darmstadt
Prof. Riedel
Dr. Wiehl

IMEM (CNR), Italy
Dr. Coppi
Dr. Delmonte
Dr. Gilioli

Contact

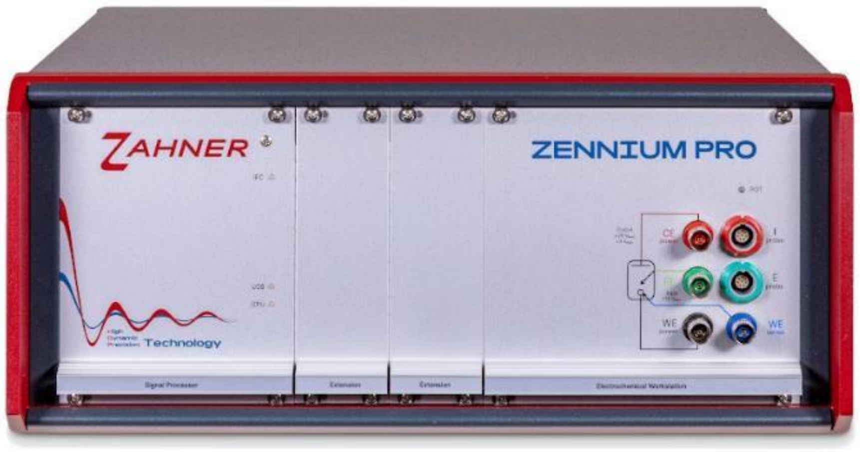
DESY. Deutsches Elektronen-Synchrotron
www.desy.de

Robert Farla
FS-PETRA-D
robert.farla@desy.de
Tel: 4470

Electrical conductivity experiments

To be commissioned (2025-I)

https://tiny.cc/petra3p61



The **ZENNIUM PRO** is a modular high-end potentiostat / galvanostat including a frequency response analyzer (FRA).

- DC voltage ranges: ±5 V and ±15 V
- Current up to ±3 A over 12 current ranges
- **EIS frequency range from 10 µHz to 8 MHz**
- AC amplitude of 0 - 6 V or 0 - 2 A for EIS
- Up to 5 parallel channels for EIS (1 PAD4 card)
- Switchable floating / grounded mode
- HiZ-probe addon included

Electrical conductivity on melt-bearing olivine at HPHT

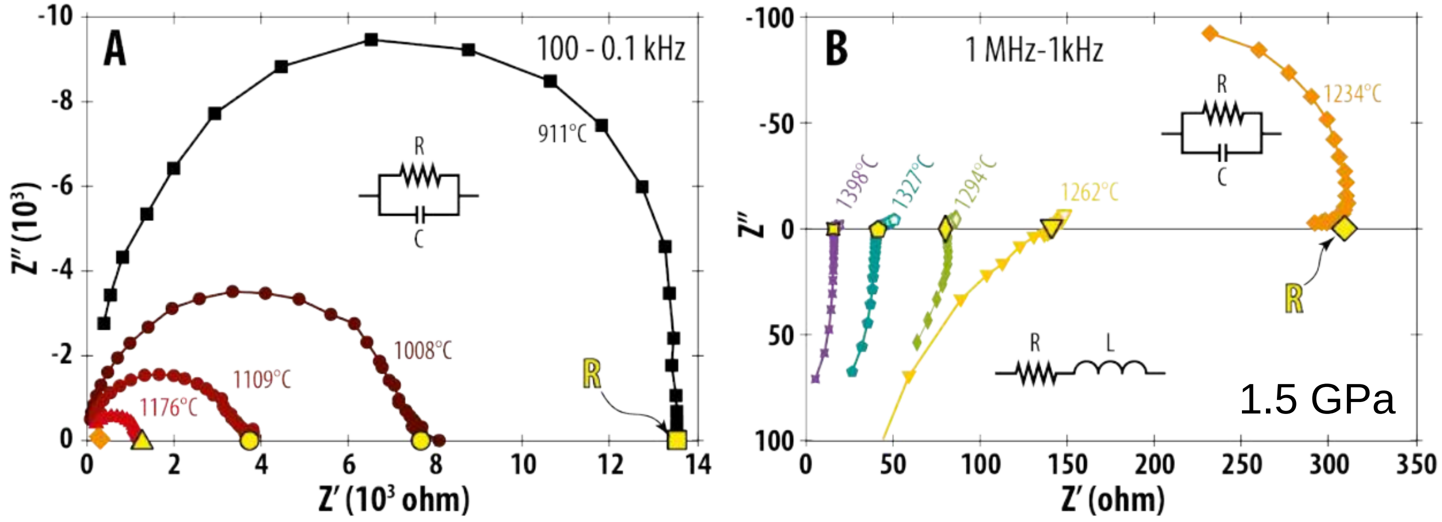


Figure 2: Impedance spectra in the Nyquist plane and equivalent electrical circuits (Huebner & Dillenburg, 1995) obtained at low (A: T<1200°C) and high (B: T>1200°C) temperatures on the pure basalt sample. R, C and L in electrical circuits stand for resistance, capacitance and inductance respectively. The real resistance (yellow R) is shown by a yellow symbol.

Laumonier, Farla, *et al.* 2017

Thales XT software for acquisition and processing

&

Python – scriptable communication