Status & development

P61B LVP

PETRA III, DESY

Beamline satellite workshop
23 January 2024

Robert Farla

Collaborators:
Shrikant Bhat (P61B), Kristina Spektor (P61B), Stefan Sonntag (P61B), Christian Lathe (GFZ), Shuailing Ma (Ningbo), Adrien Néri (Lille), Lianjie Man (BGI), Artem Chanyshev (BGI), Julien Gasc (Montpellier), Ulrich Häussermann (Stockholm), Holger Kohlmann (Leipzig), Tomoo Katsura (BGI)

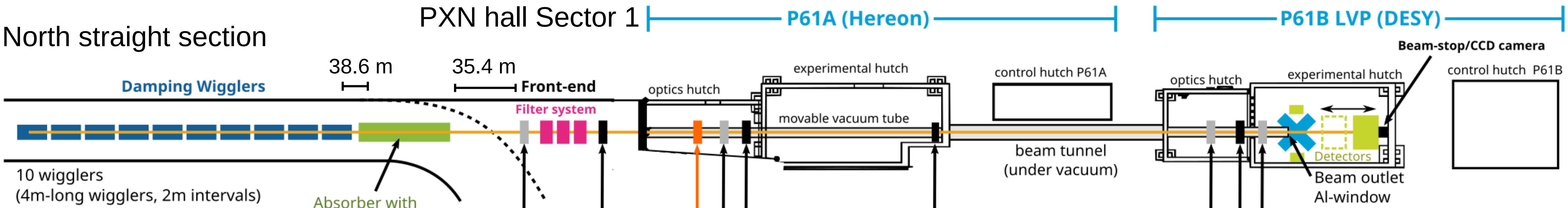
Support: **DESY Support Groups (FS-TI, -EC, -BT, Machine…)**

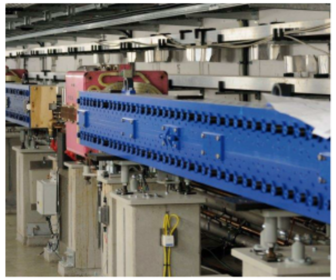
HELMHOLTZ RESEARCH FOR GRAND CHALLENGES   BGI Bayerisches Geoinstitut   UNIVERSITÄT LEIPZIG

P. P. Ewald hall, sector 1, P61

Description of the beamline

High energy wiggler beamline P61 – user operation since 2020



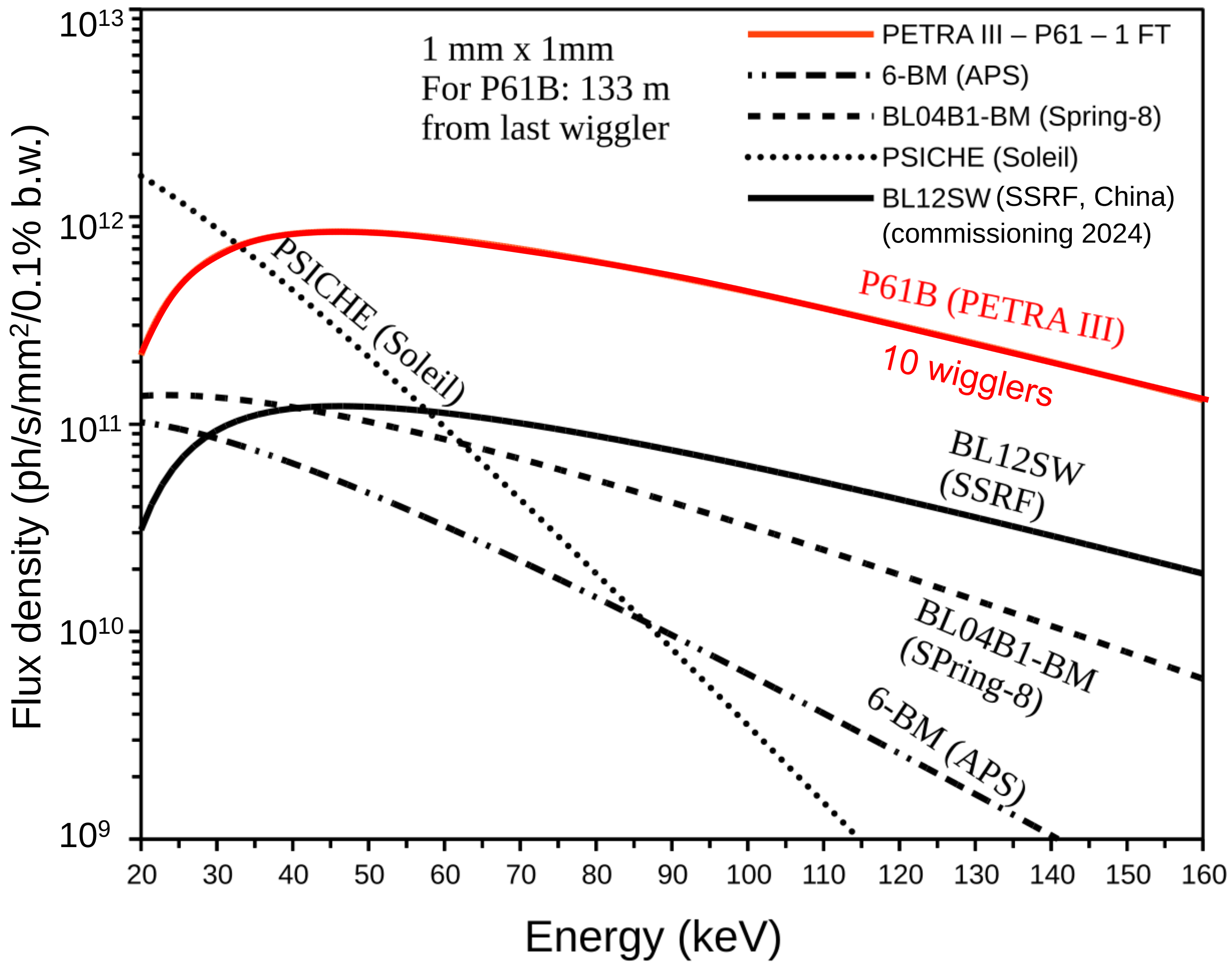


Incident slits
before LVP
in EH2 (P61B)

10 mm slit blades [Densimet, $(Ni,Fe)_{0.02}W_{0.98}$]

Beam characteristics

Photon flux and comparisons



Calculated flux at P61 (upper limit)

Peak at @ 50 keV

~10^{12} ph/s/mm^2

Integrated flux (1FT)

~10^{14} ph/s/mm^2

P61B is ideal for
energy-dispersive x-ray diffraction (ED-XRD)
at high-energies (> 30 keV) in the LVP

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Extreme conditions research using the large-volume press at the P61B endstation, PETRA III

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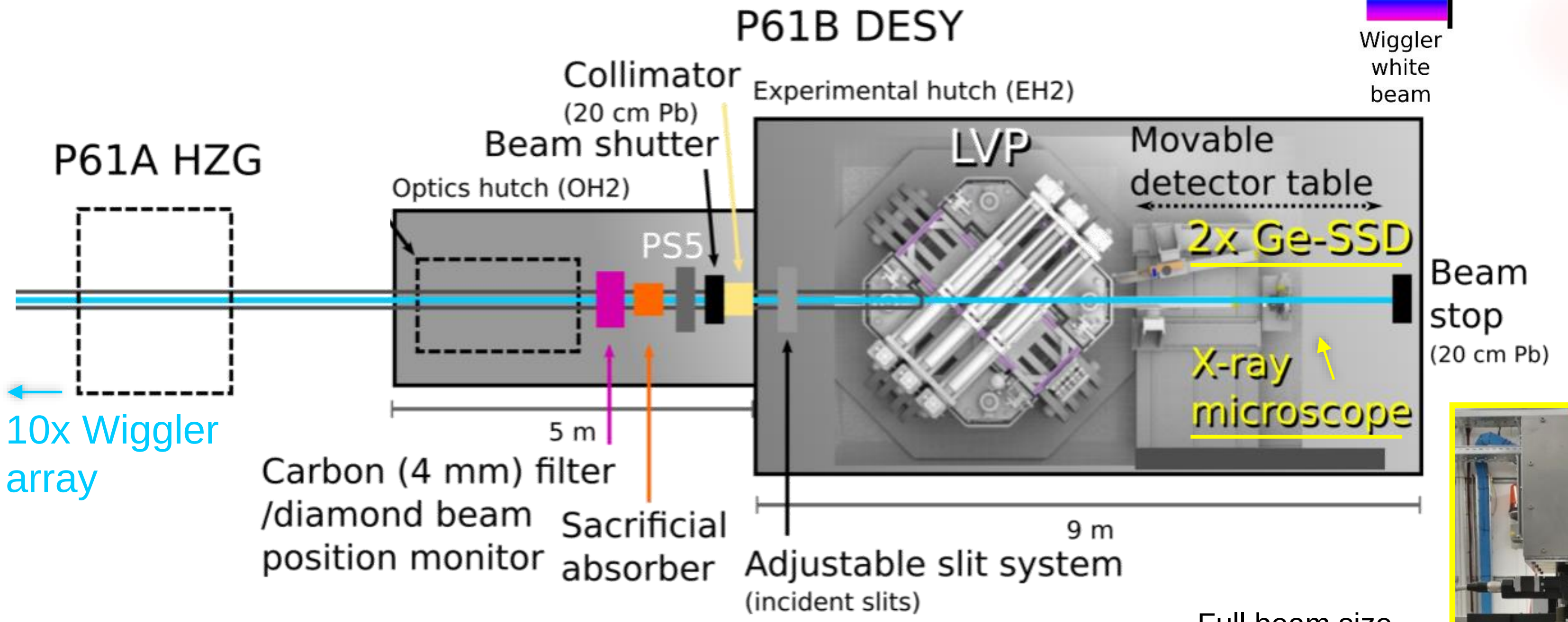
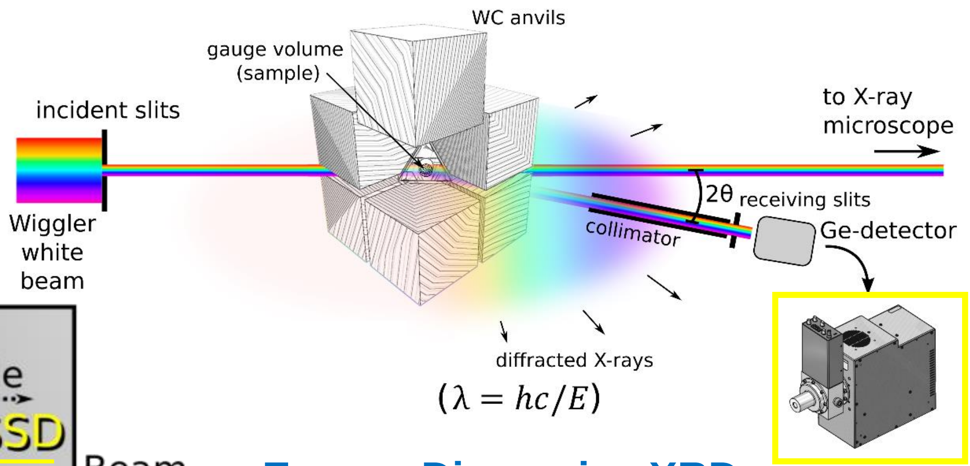
The LVP end-station P61B

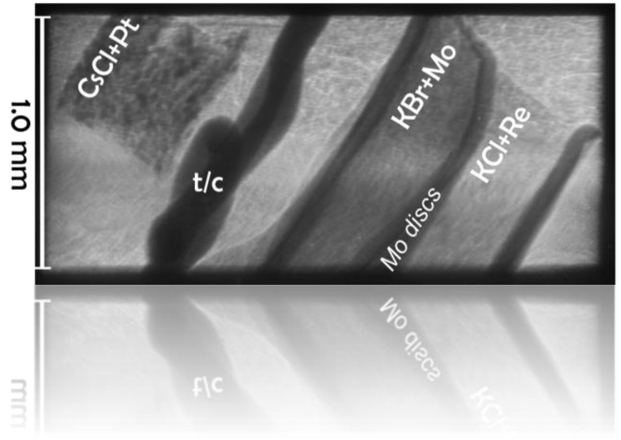
ca. 90 days user operation per year with x-rays

Multi-purpose white beam station optimized for the study of crystalline materials (& liquids) under (ultra-)high pressure, temperature and stress

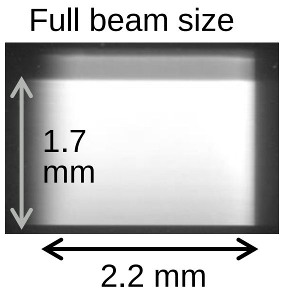




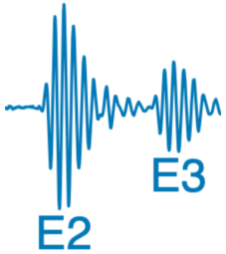
Energy-Dispersive XRD



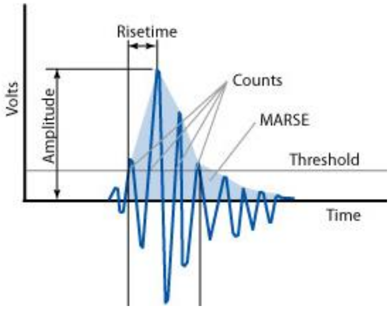
High resolution imaging



Ultrasonic velocity measurements



Acoustic Emission detection



The LVP end-station P61B

Rapid-access user operation without x-rays

- Synthesis of novel (recoverable) materials

  Band-gap tuning/semiconductors, optical windows, super-hard/conductive, catalyzers, hydrogen storage
- Trial experiments before scheduled beamtime
- Offline Acoustic Emissions experiments
- Offline Electrical Conductivity experiments

  (from 2nd half of 2024)
- Other experiments producing publishable data…

Access to the DESY NanoLab (SEM/EBSD) and Chemistry Lab can be applied for.
(at least 4 weeks in advance)

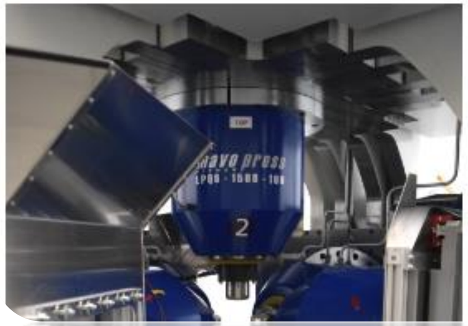
Aster-15

LVP-P61B

Standalone use of the LVP (no X-rays)

APPLICATION PROCEDURE

Visit the beamline website



To apply for offline use of the LVP please contact the Beamline Manager (Robert Farla).

Applications must include:

1) a Short Proposal of the project

2) a completed Proposal Form.

Available dates in 2024:

Jan - Feb (discuss with Beamline Manager)

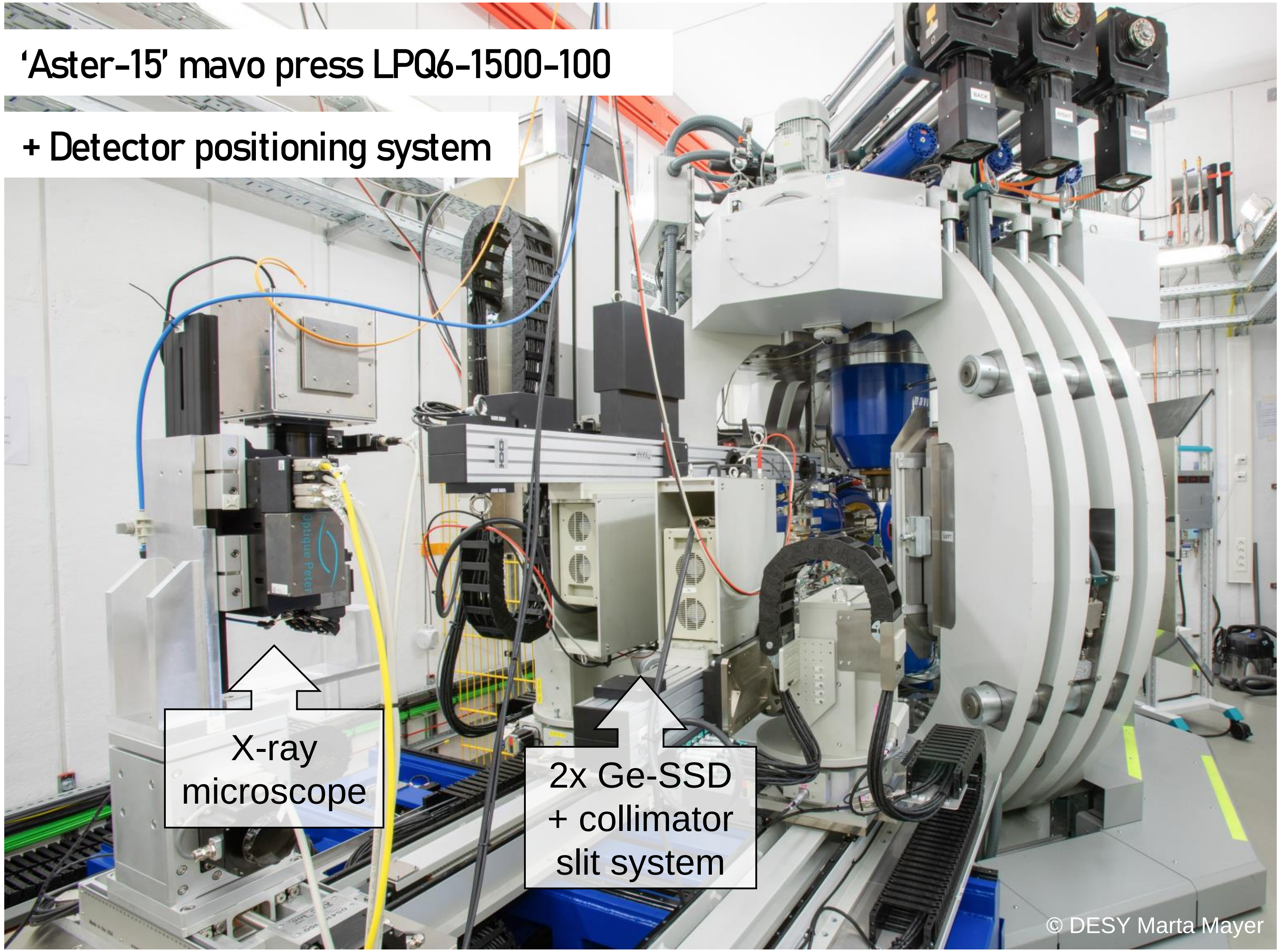
08 - 19 April

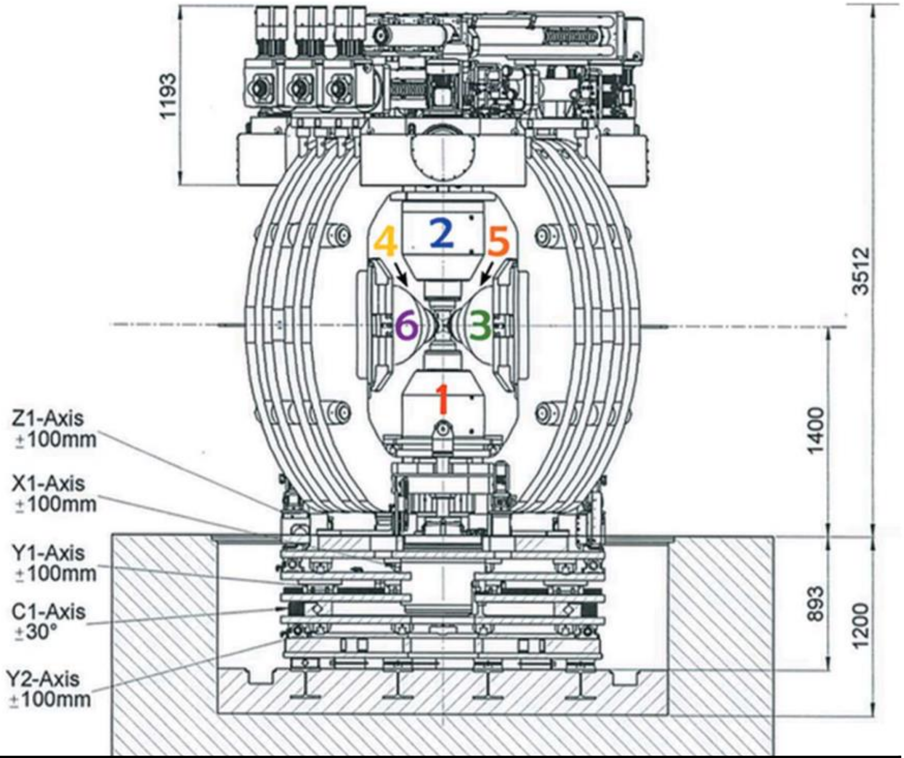
27 - 31 May

24 - 28 June

The LVP 'Aster-15'

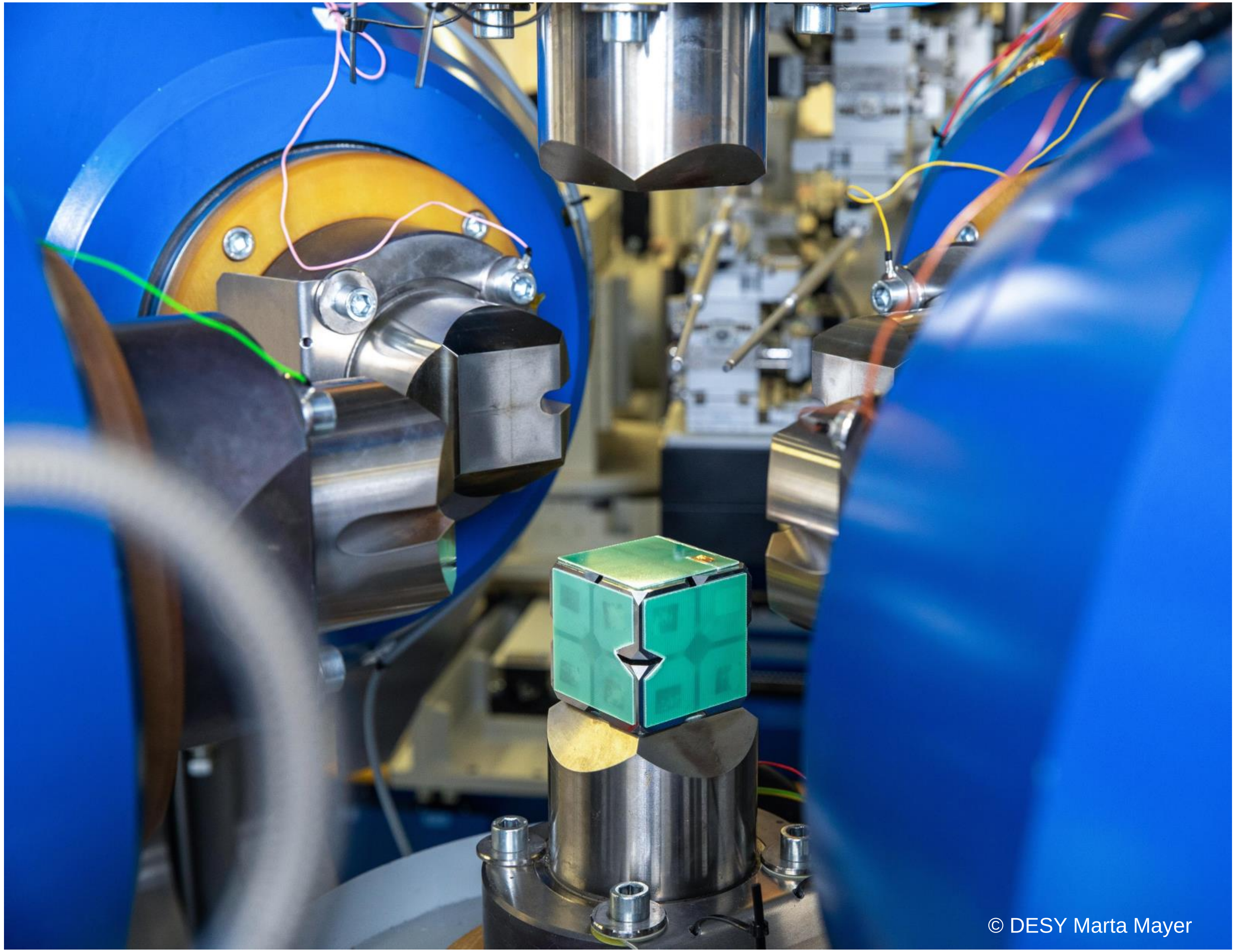
Key specifications





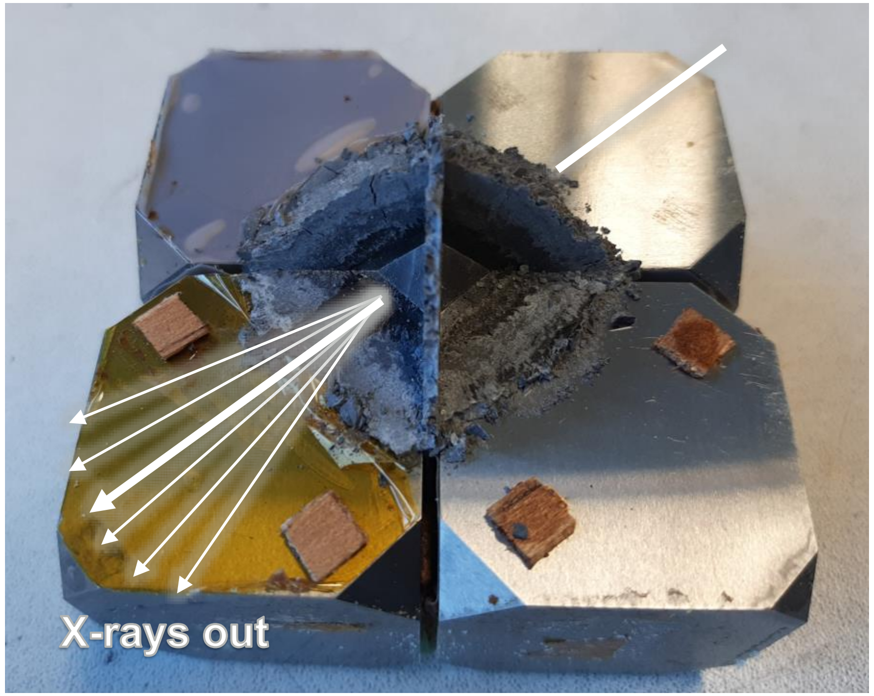
Maximum load	15 MN – 5 MN/axis
Ram position control	1 µm step – 100 mm
Oil pressure control	0.5 bar – 620 bar/ram
Anisotropic compr.	Axial symmetric, triaxial
5-axis stage	x,y1,y2,z (± 100 mm), rotation: ± 11.5°
Combined weight	ca. 45 ton

High-pressure techniques



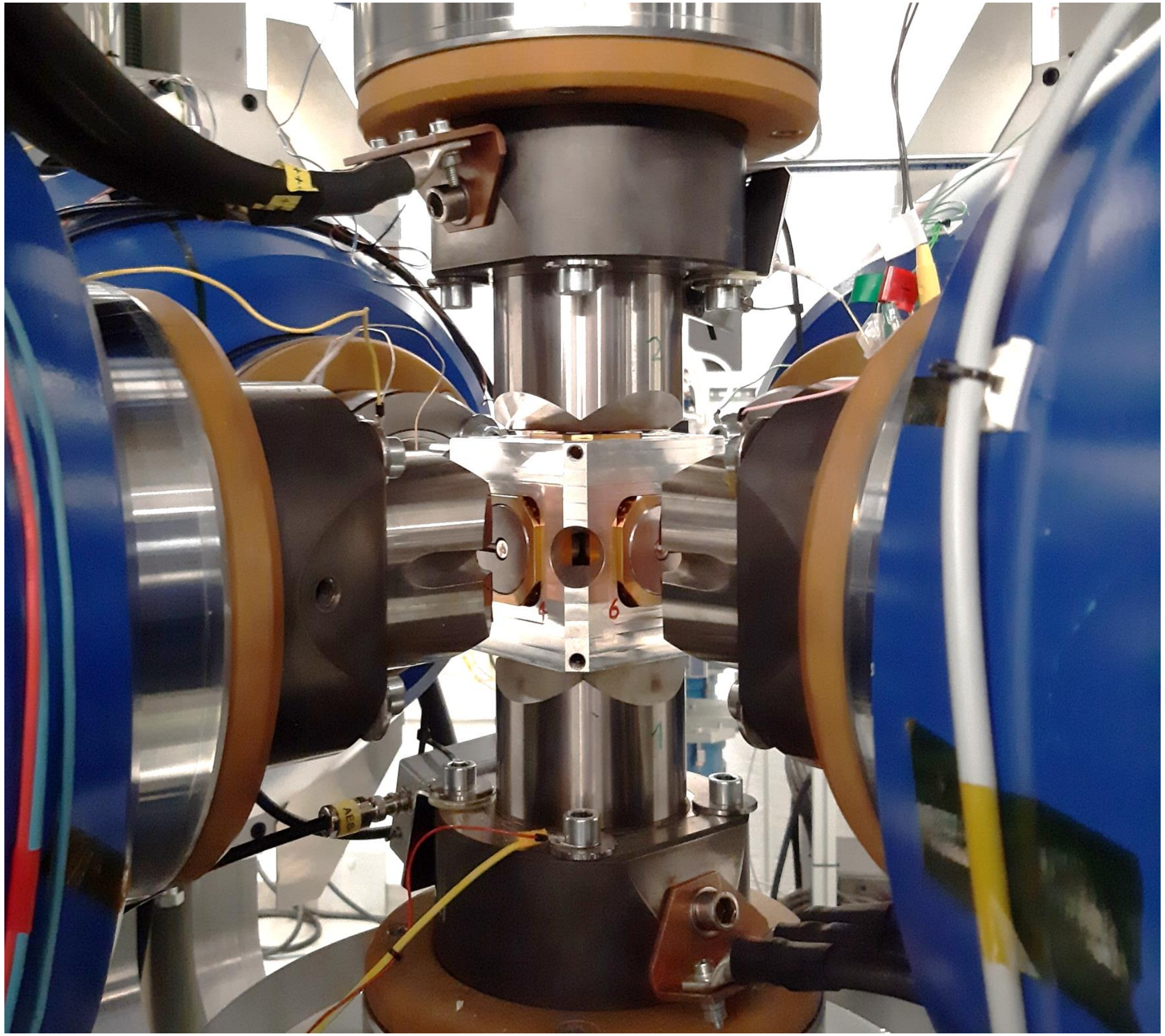
'Kawai' 6-8 mode (octahedral PTM)

Recovered assembly after compression



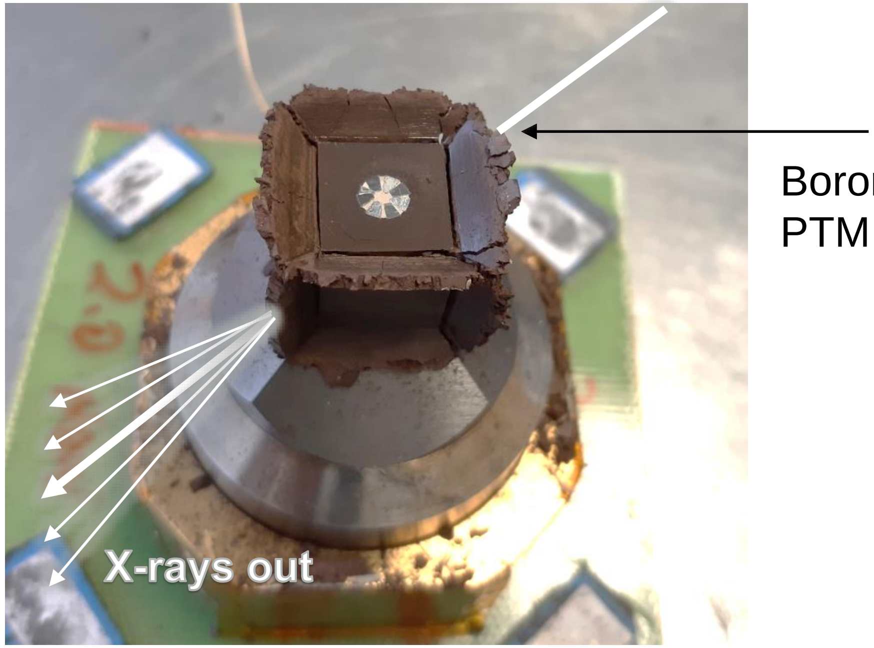
Useful for standard isotropic compression and UHP studies.

High-pressure techniques

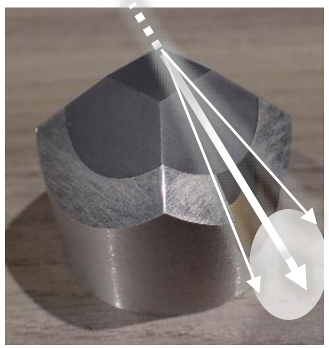


‘Hall’ 6-6 mode (cubic PTM)

Recovered assembly after compression



Boron-epoxy PTM



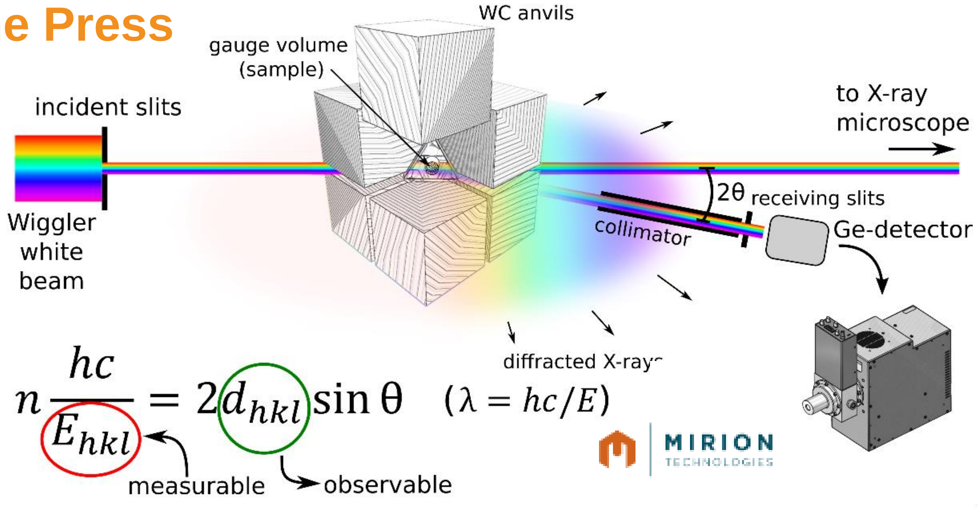
Large x-ray transparent cBN anvil.

Useful for high-P/T *in situ* rock deformation studies.

X-ray techniques using white beam

ED-XRD and Absorption Contrast Imaging in the Large Volume Press

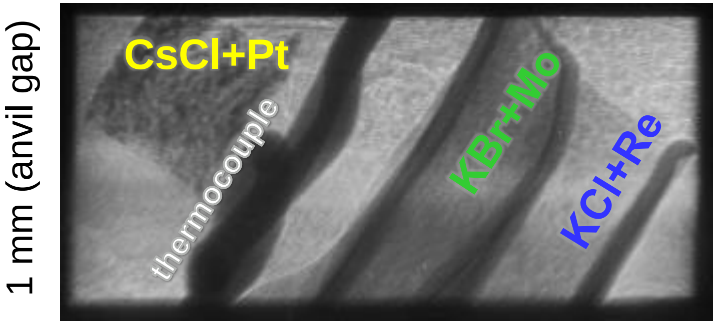
1. High spatial resolution (defines a gauge volume)
   → avoid high temperature & pressure gradients
   → no diffracted X-rays from sample environment
2. Fast acquisition (can be < 10 s), large Q-range (12 Å^{-1})



$$n\frac{hc}{E_{hkl}} = 2d_{hkl}\sin\theta \quad (\lambda = hc/E)$$

Experimental procedure:

Radiography image



X-ray microscope

pco. edge 5.5MP

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

ED-XRD and Absorption Contrast Imaging in the Large Volume Press

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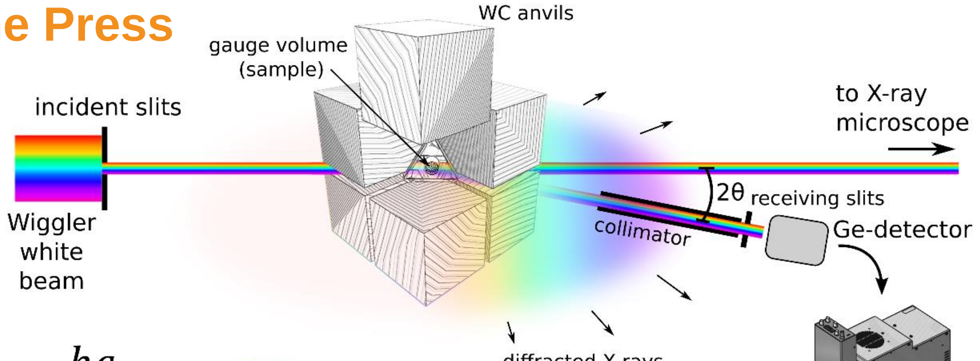
2. Fast acquisition (can be < 10 s), large Q-range (12 Å^{-1})



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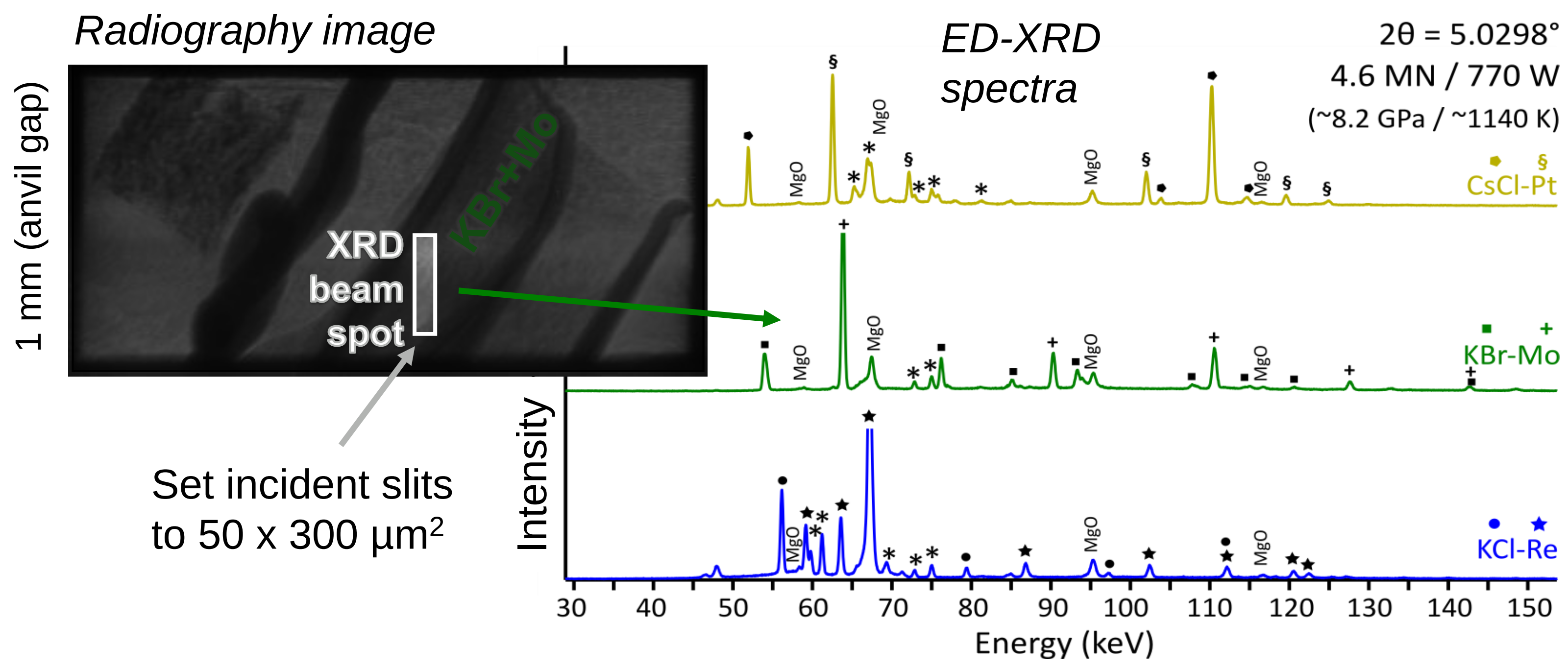
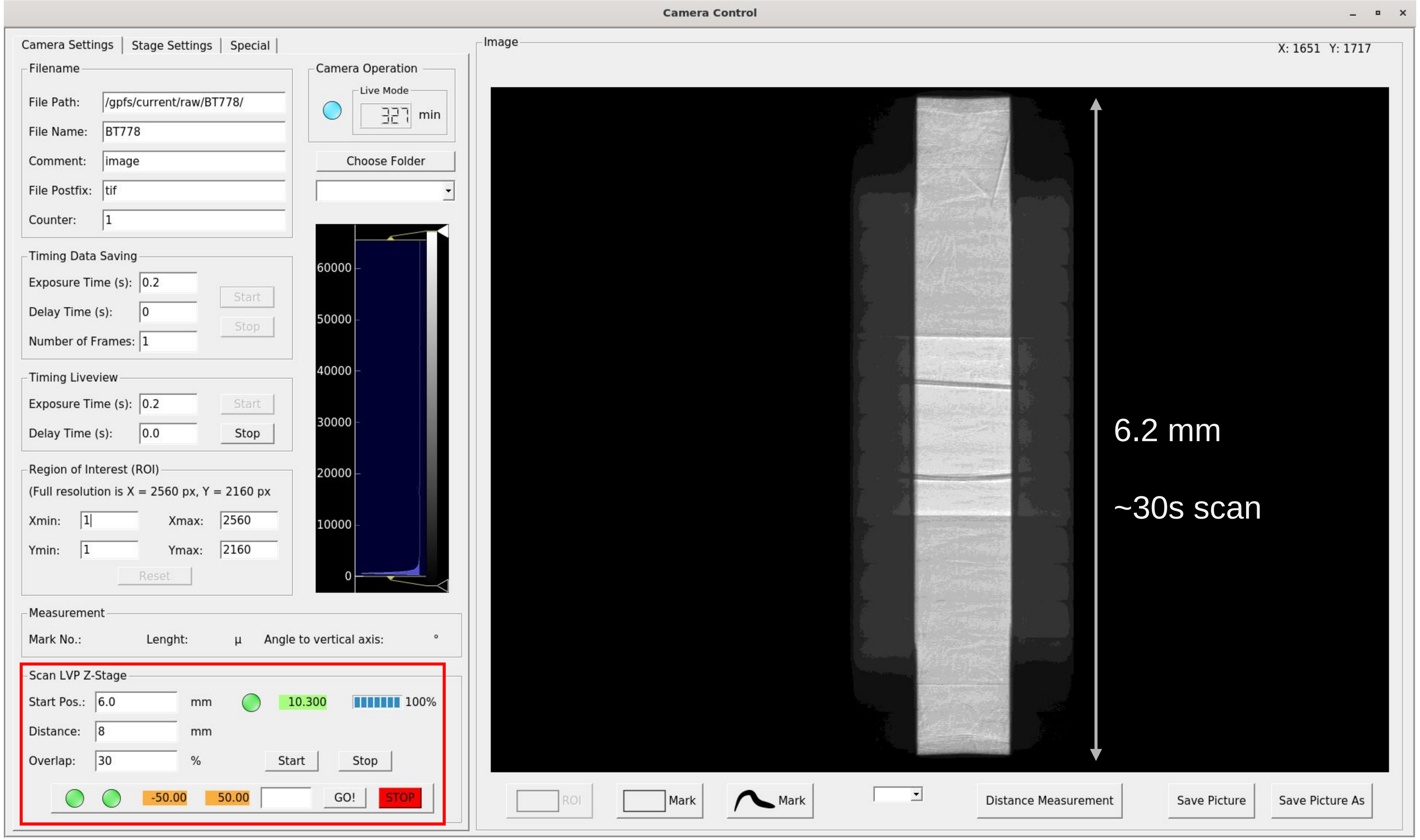


X-ray techniques using white beam

Beamline control software: imaging, image scans

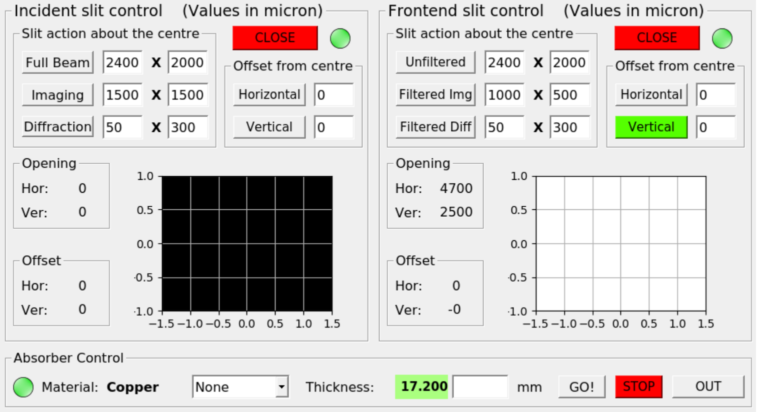
Live view and acquisition

- Set experiment run #, comment, file path
- Set exposure time (live/saving), number of frames (saving)
- Optionally, set ROI
- **Scan sample / assembly: → create a montage from acquired frames**



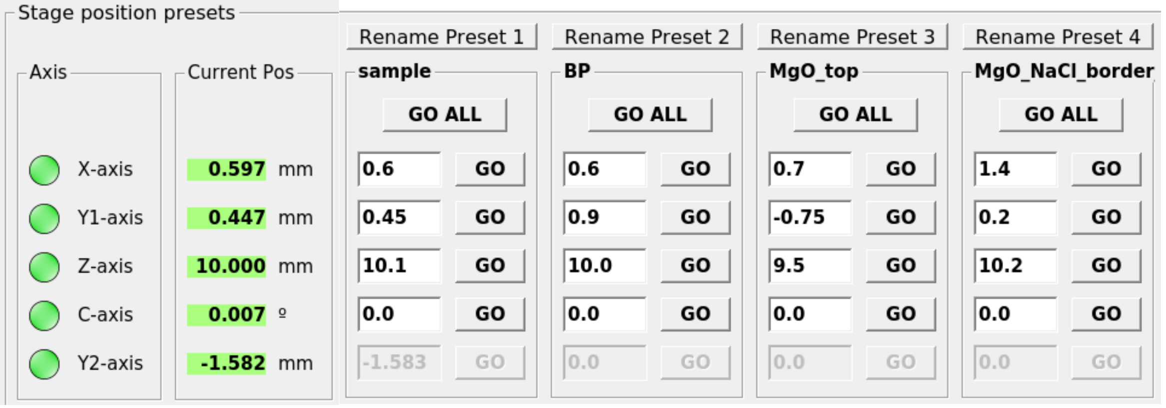
Beamline development

Beamline control software: slits, motors, stages, XRD acquisition (python - using TaurusGUI)

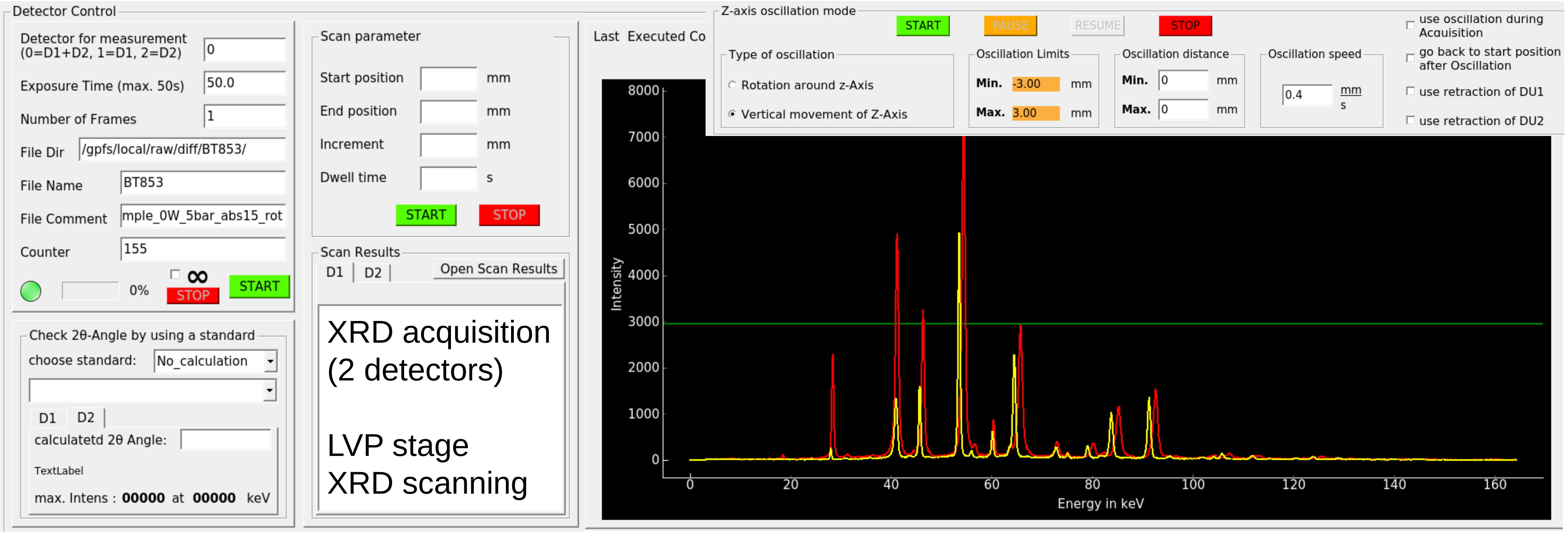


Incident slits control

Variable absorber



Preset positions for multiple samples



XRD acquisition (2 detectors)

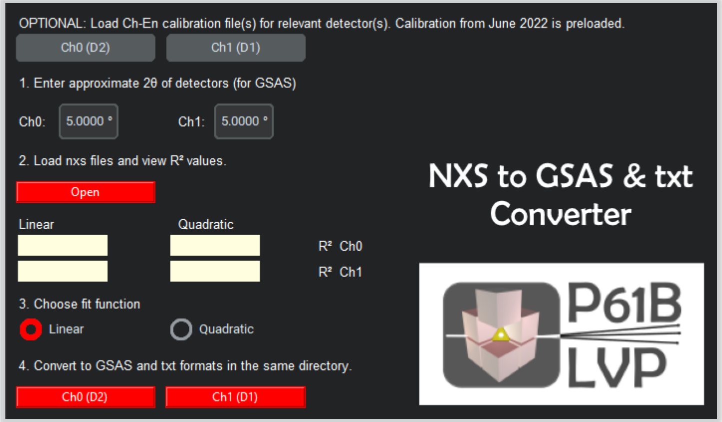
LVP stage XRD scanning

LVP oscillation modes

Beamline development

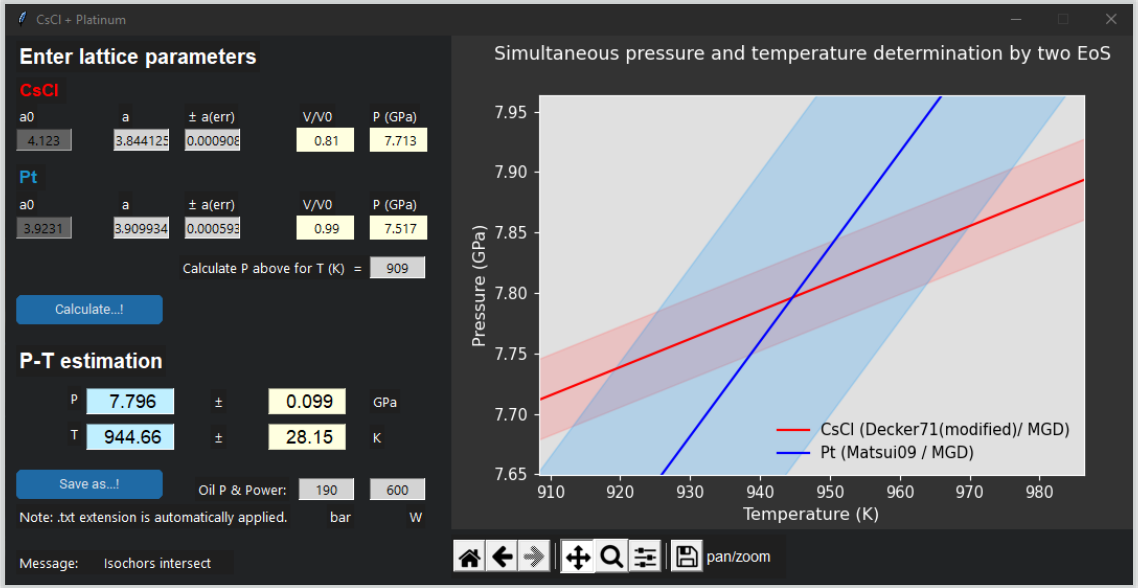
Custom software tools (python - using tkinter GUI)

2-theta calculator using NIST standard: LaB6 660c



HDF5 (nxs) to GSAS-II conversion

AE tools

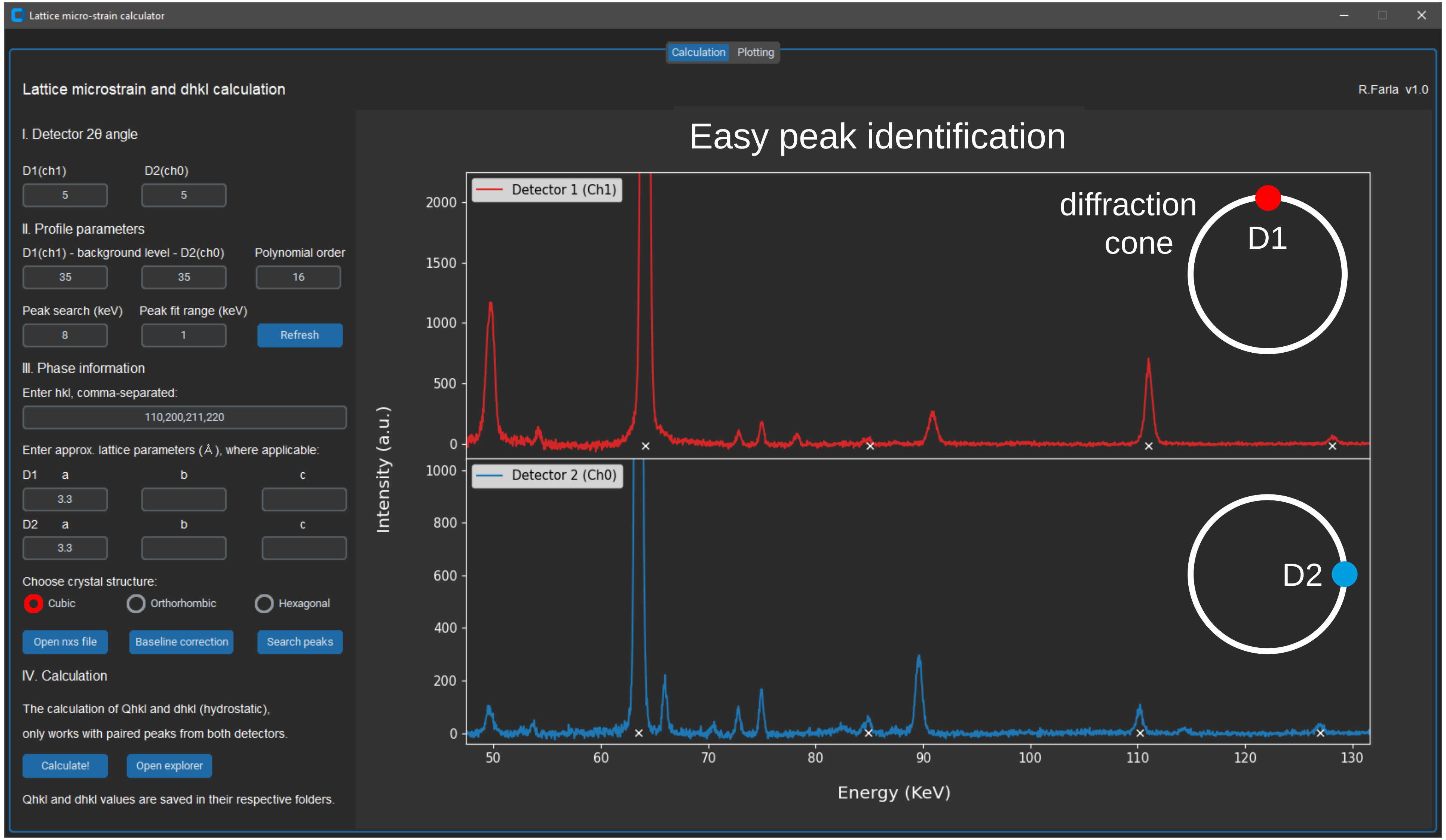


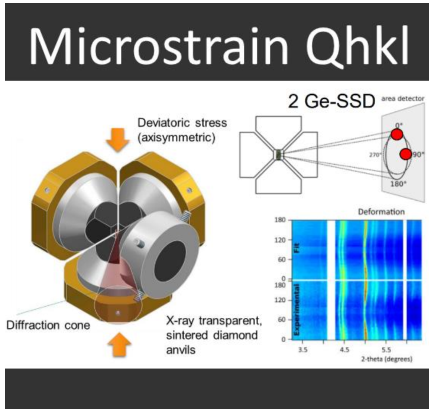
EosCross: Joint P & T estimation (Farla *J. Syn. Rad.* 2023)

https://gitlab.desy.de/robert.farla/eoscross

Beamline development

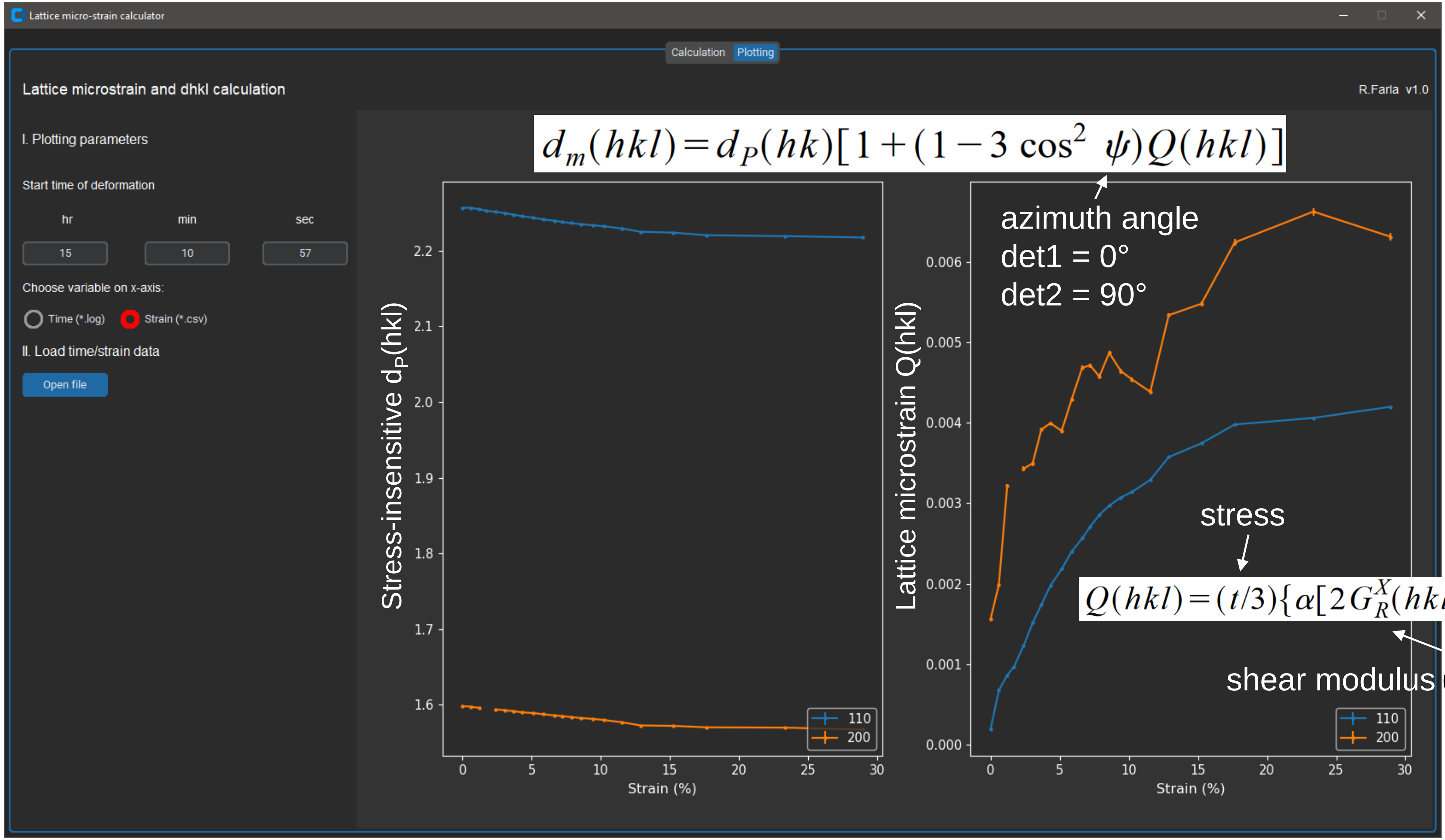
Latest software tool: microstrain (i.e. stress) analysis for *in situ* deformation experiments





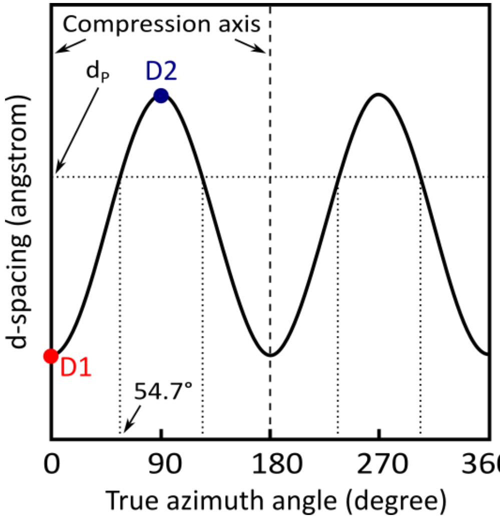
Beamline development

Latest software tool: microstrain (i.e. stress) analysis for *in situ* deformation experiments



$$d_m(hkl) = d_P(hk)[1 + (1 - 3\cos^2\psi)Q(hkl)]$$

$$Q(hkl) = (t/3)\{\alpha[2G_R^X(hkl)]^{-1} + (1-\alpha)(2G_V)^{-1}\}$$



Warning! Only true for uni-axial compression!

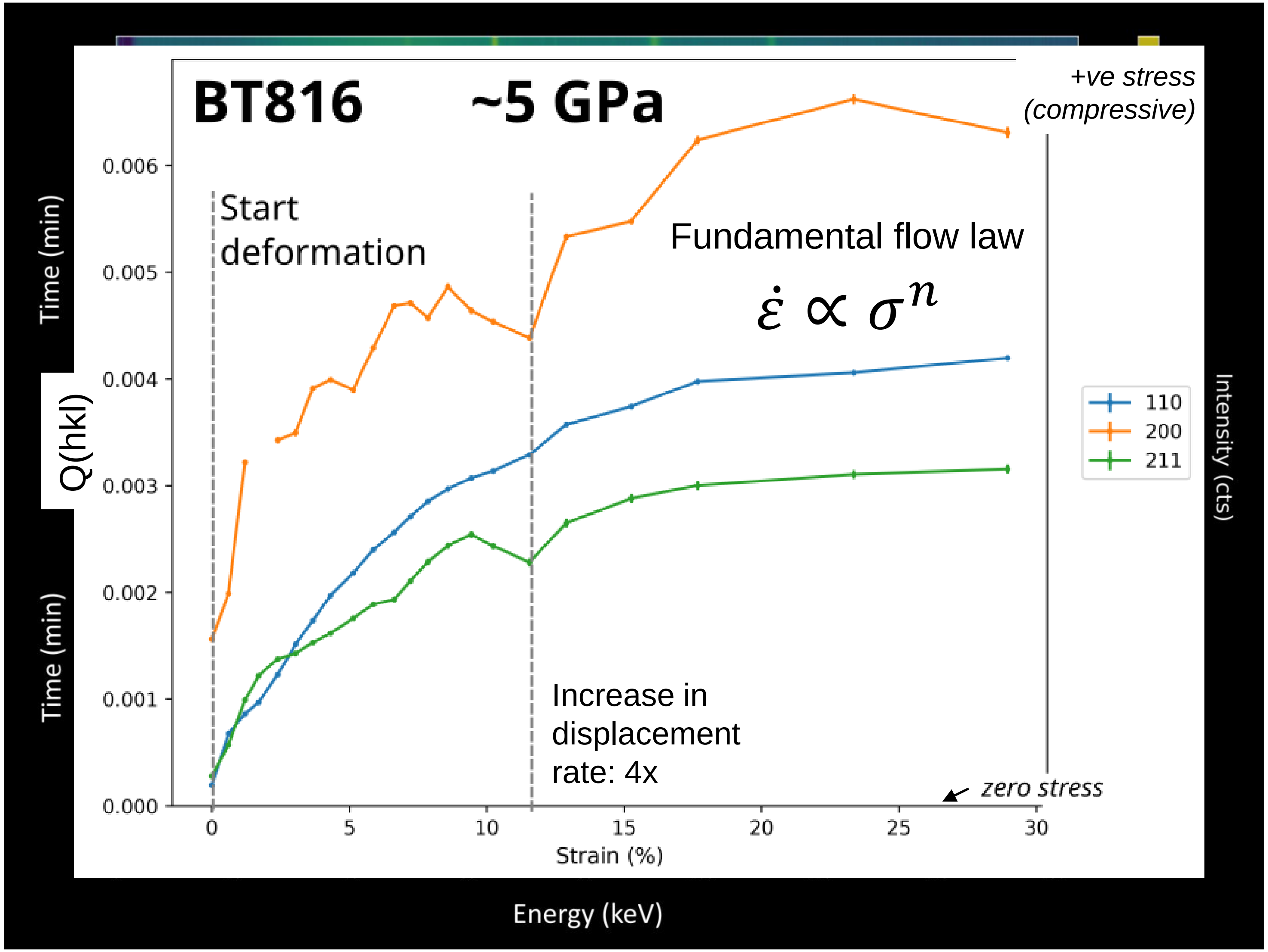
Singh *et al. J. App. Phys.* 1998

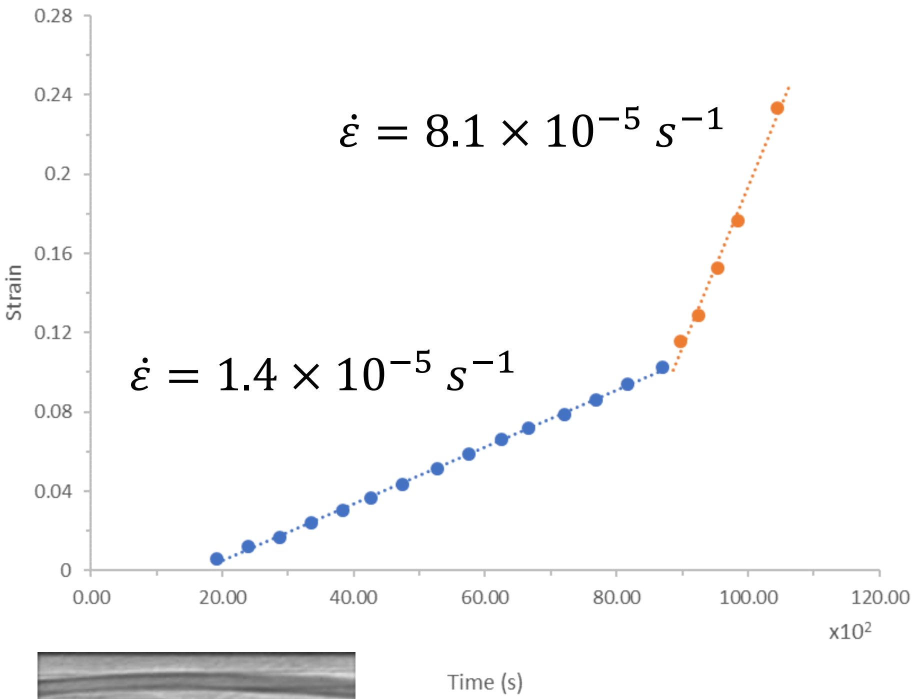
Research highlights

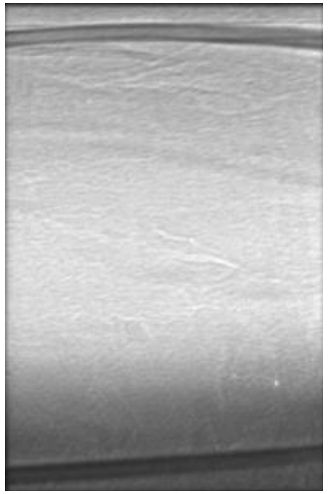
Case study 1: rheology of H_2O ice VII

EoS

R. Farla (DESY), C. Howard (DIAMOND, UK),
N. Walte (FRM II, Garching)







Macro-strain history

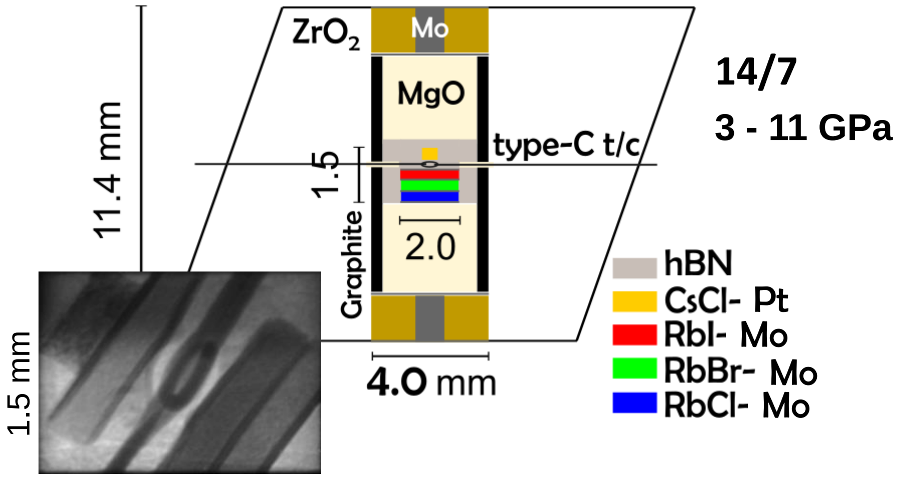
Preliminary results

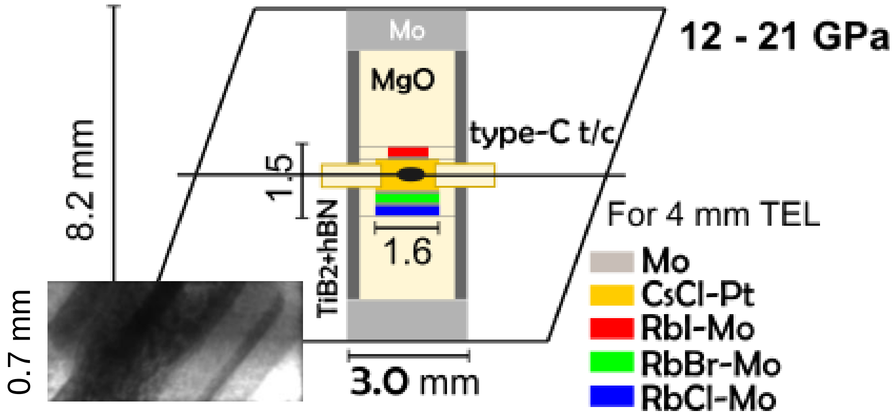
Research highlights

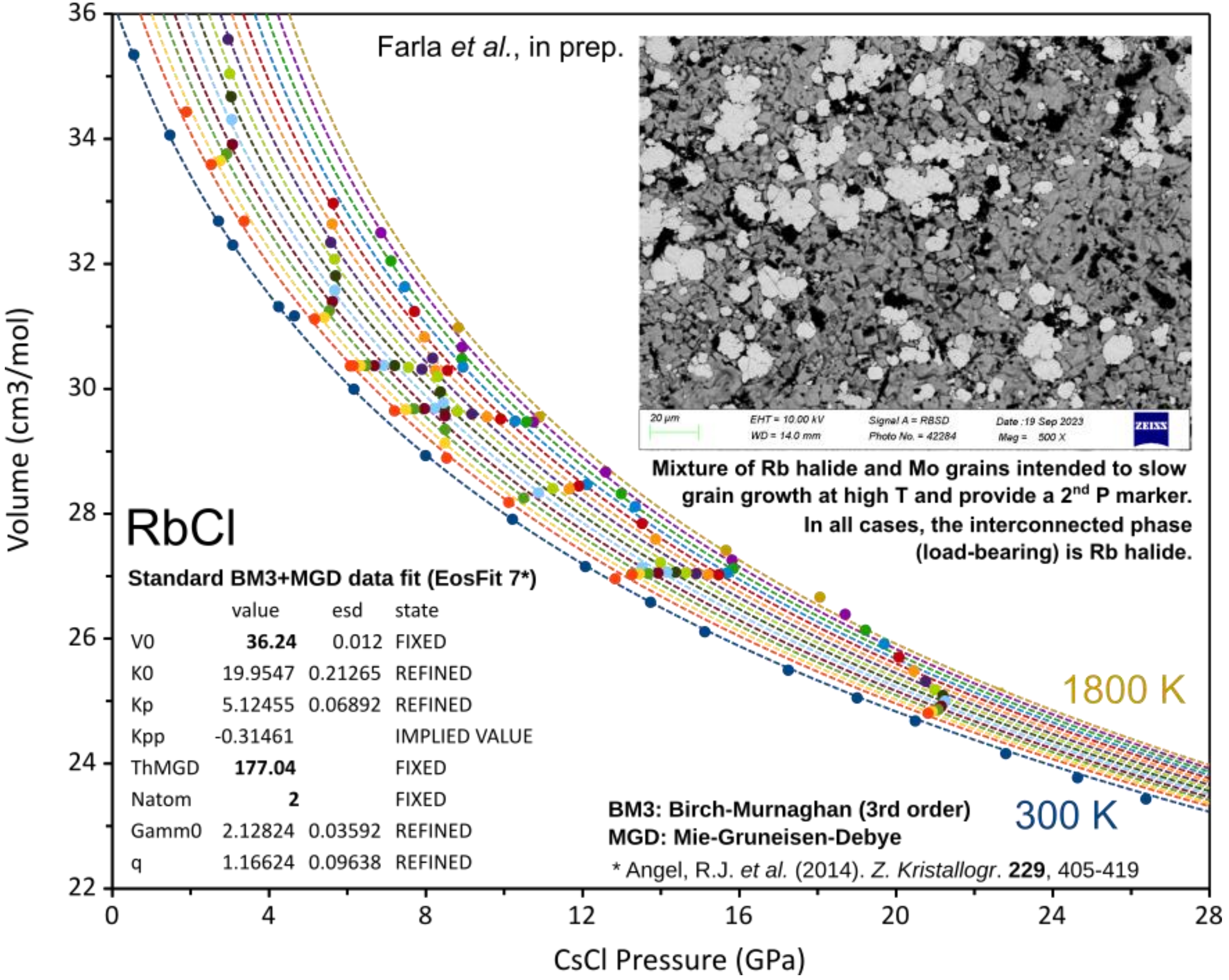
Case study 2: thermal EoS of Rubidium halides

EoS
R. Farla, K. Glazyrin (DESY)
A. Neri, T. Boffa-Ballaran,
M. Pöppelbaum (BGI)

Data fitted using EosFit 7
(Angel et al. 2014,
Gonzalez-Platas et al. 2016)





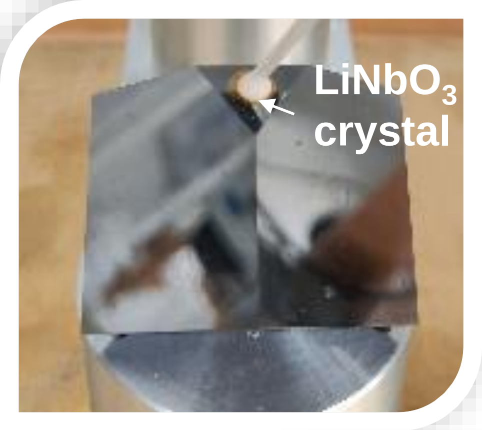


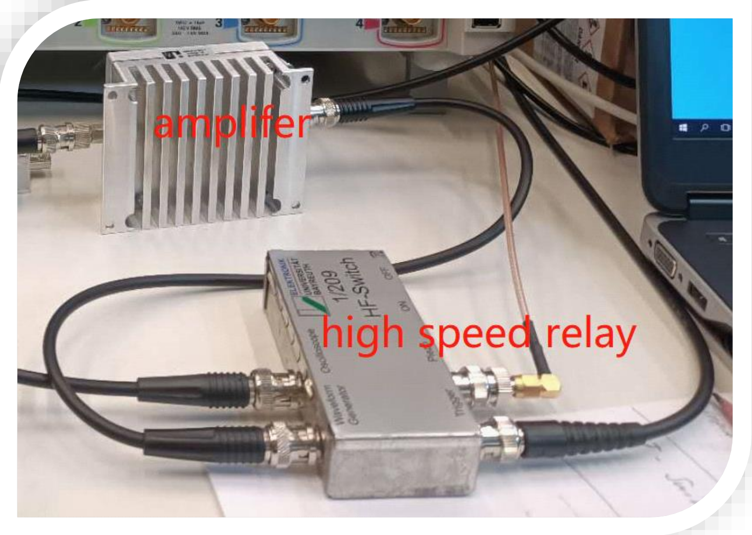
→ **Full-profile XRD refinement (Le Bail) in GSAS-II**

Research highlights

Ultrasonic Interferometry
A. Néri, L. Man & R. Farla (BGI / DESY)

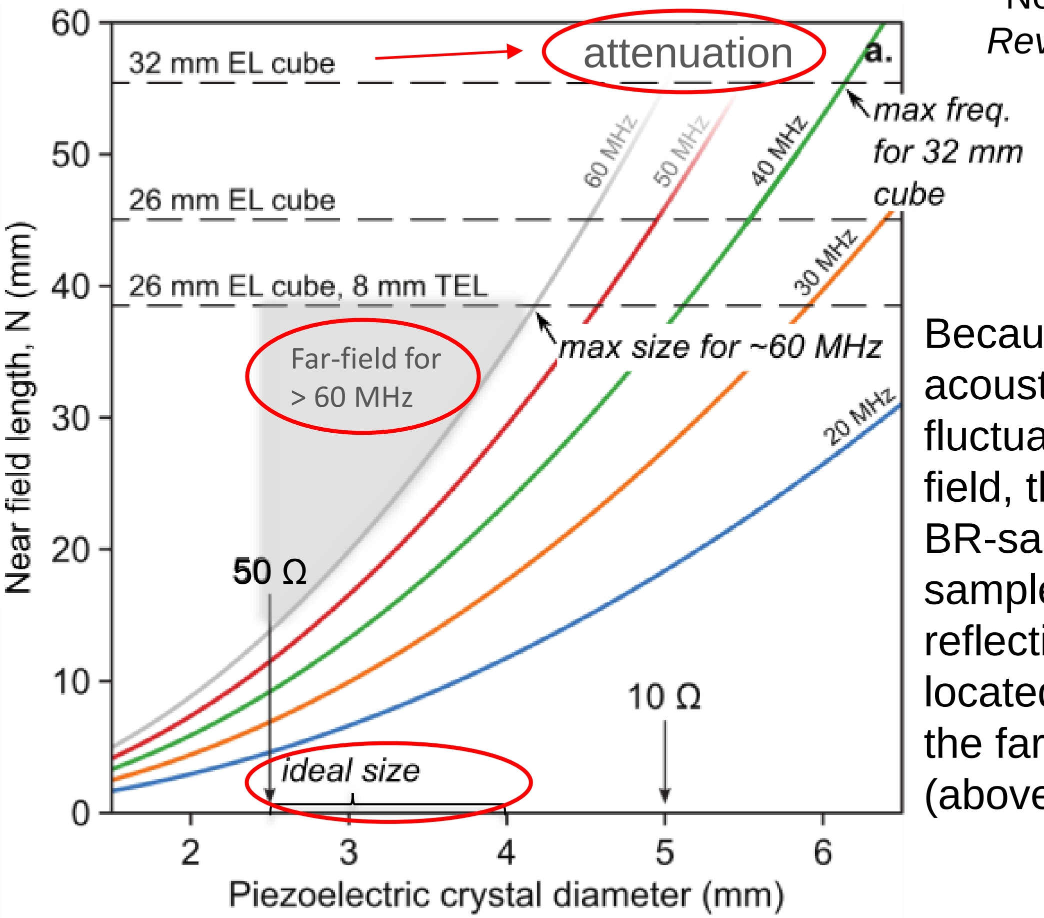
Ultrasonic Interferometry: the Pulse – Echo technique





Choice of $LiNbO_3$ transducer size / anvil size

Néri *et al*. 2023, *Rev. Sci. Instrum*. *accepted.*



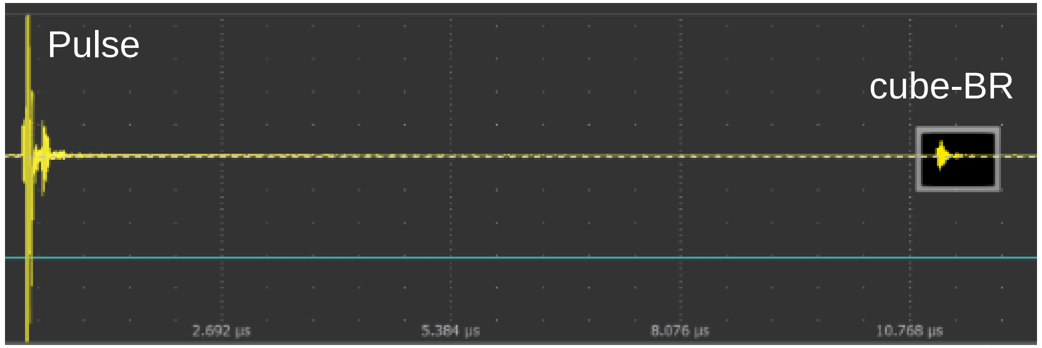
Because the acoustic pressure fluctuates in the near field, the anvil-BR, BR-sample, and sample-BP reflections should be located well within the far field (above each curve)

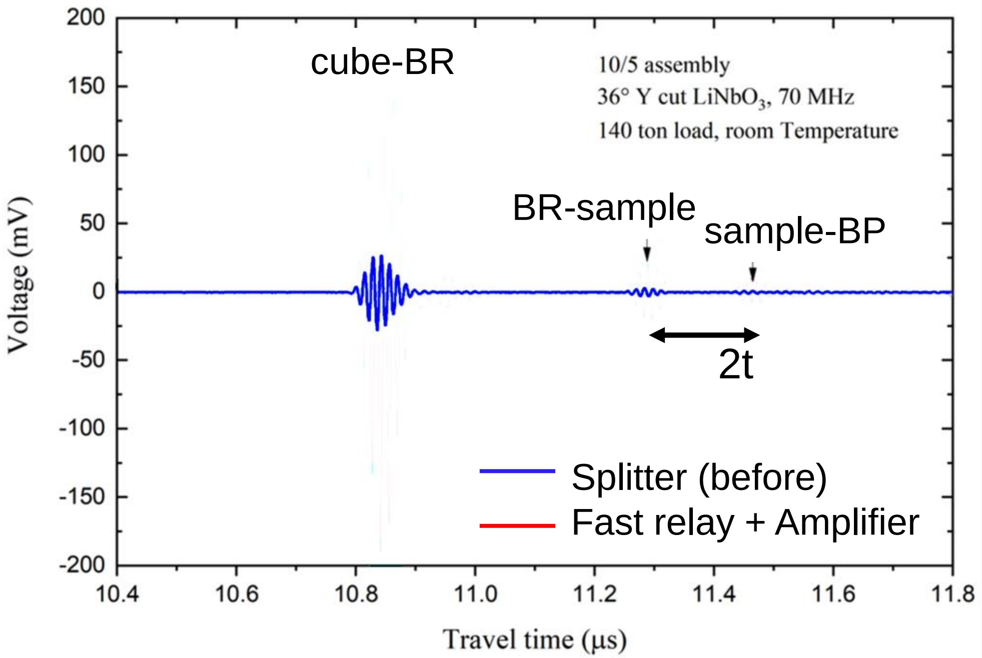
Research highlights

Ultrasonic Interferometry
A. Néri, L. Man & R. Farla (BGI / DESY)

Ultrasonic Interferometry: the Pulse – Echo technique

Fast relay OFF



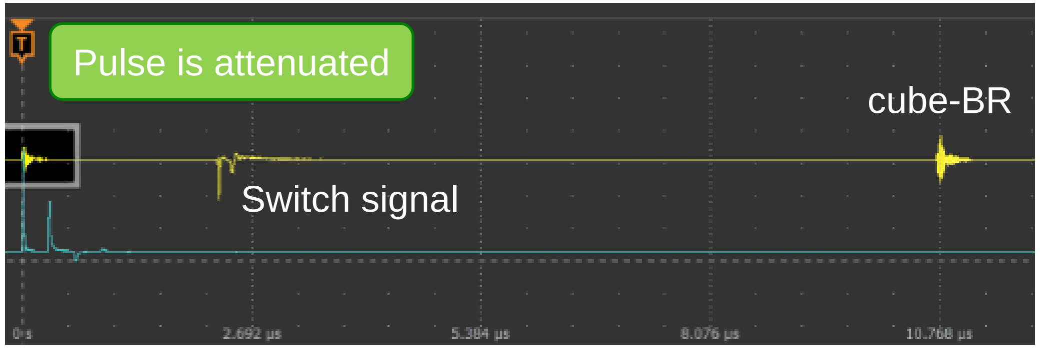


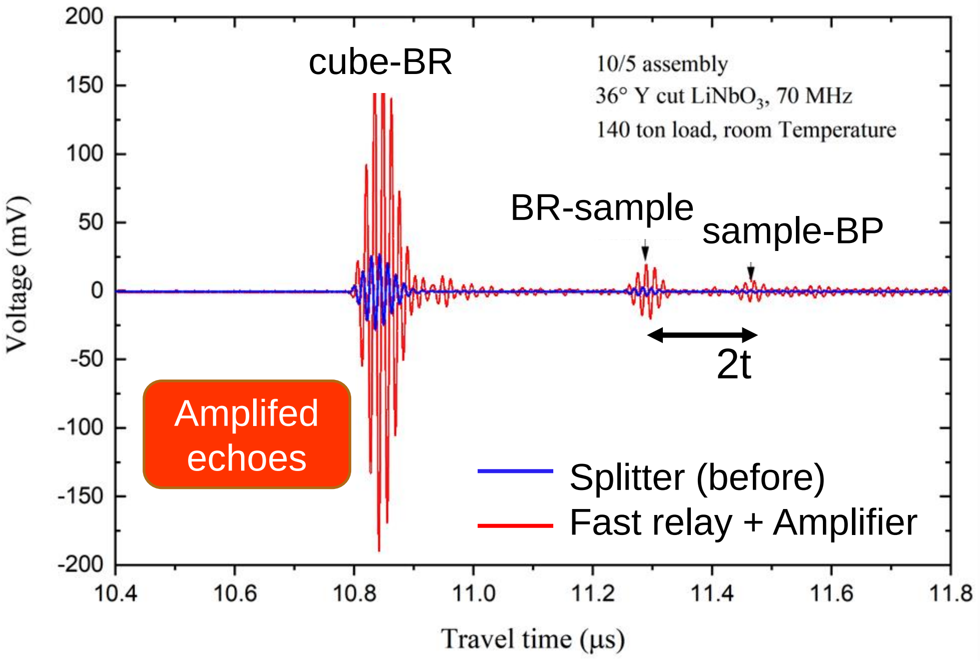
Research highlights

Ultrasonic Interferometry: the Pulse – Echo technique

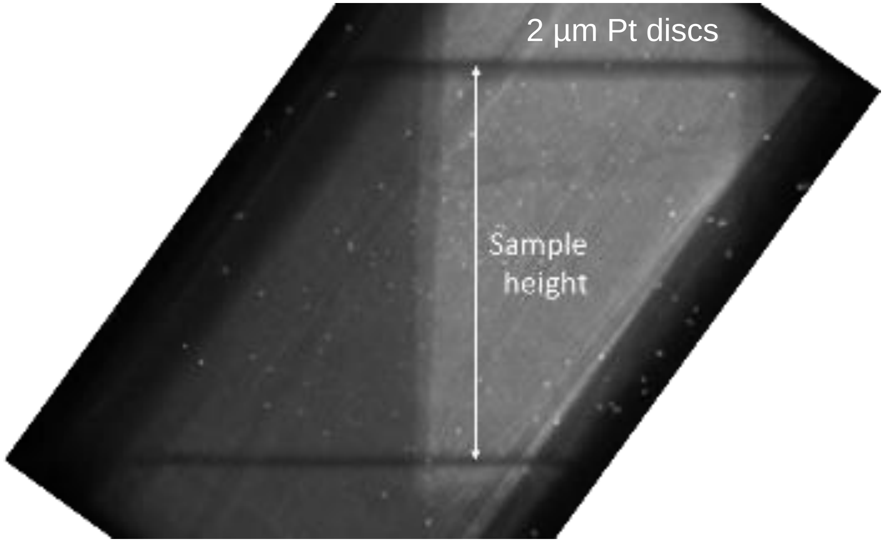
Ultrasonic Interferometry
A. Néri, L. Man & R. Farla (BGI / DESY)

Fast relay ON + amplifier





Two-way travel time (2t) and sample length (by radiography) are used to calculate: **P and S wave velocities**



Research highlights

Ultrasonic Interferometry
A. Néri, L. Man & R. Farla (BGI / DESY)

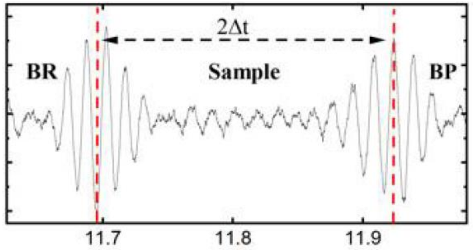
Ultrasonic Interferometry: the Pulse – Echo technique

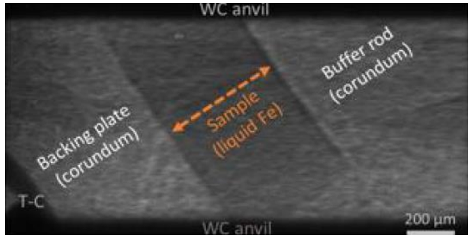
Ultrasonic measurements on liquid Fe at 9.5 GPa / 2023 K to study the Martian core

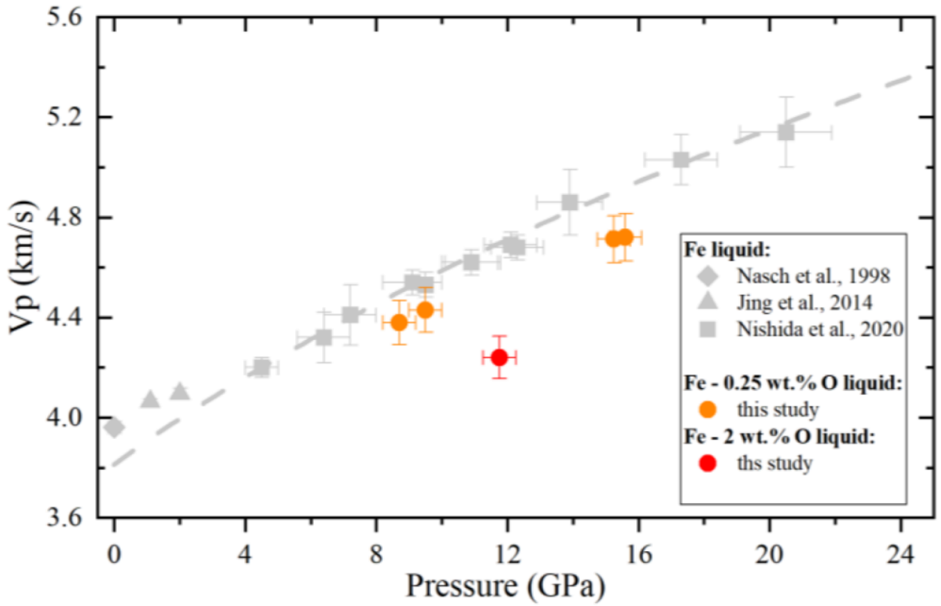
Man *et al*. 2023, *in prep.*

Development of internal pressure standards for in-house elastic wave velocity measurements in multi-anvil presses

Néri *et al*. *Rev. Sci. Instrum*. 2024, *accepted.*

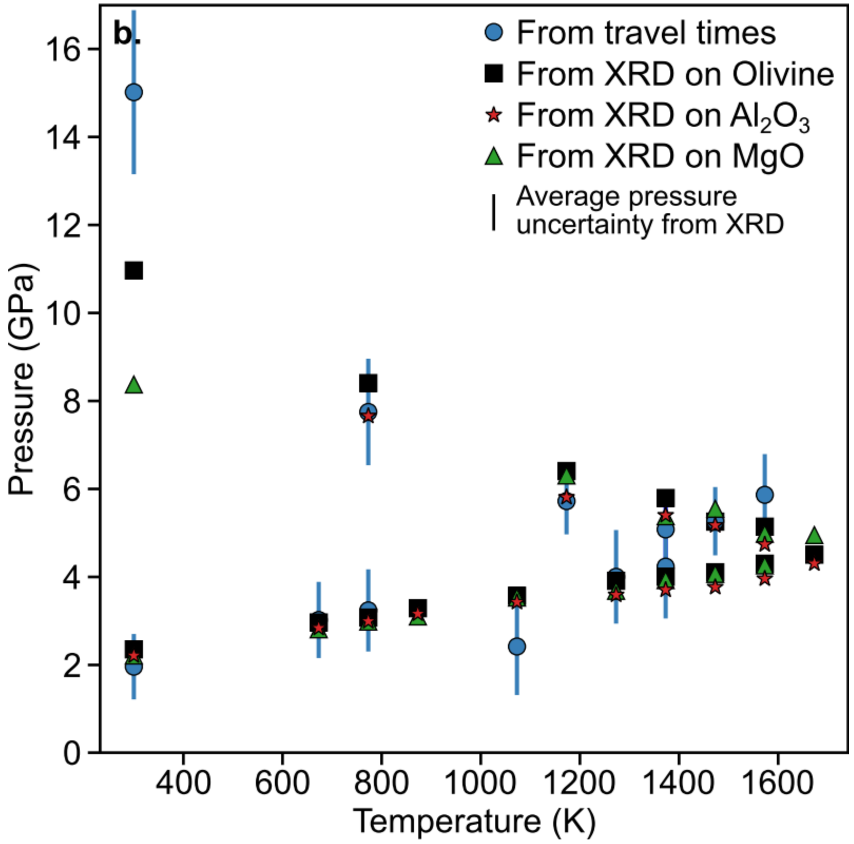






Right. Comparison of pressures by XRD standards and our novel dual travel time method, as a function of temperature.
→ in-house pressure measurements possible without x-rays.

Left. Sound velocity reduction in an iron-oxygen rich liquid.

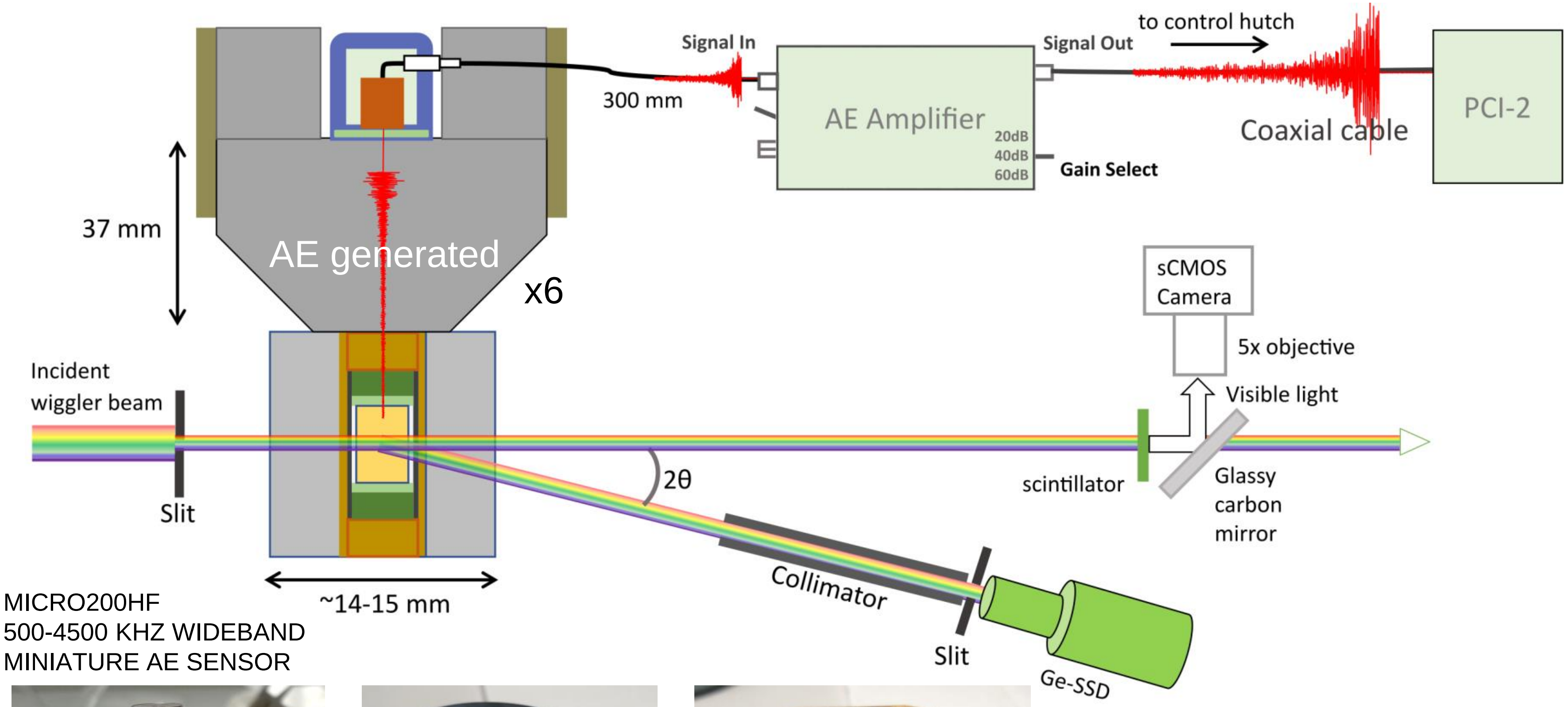


Research highlights

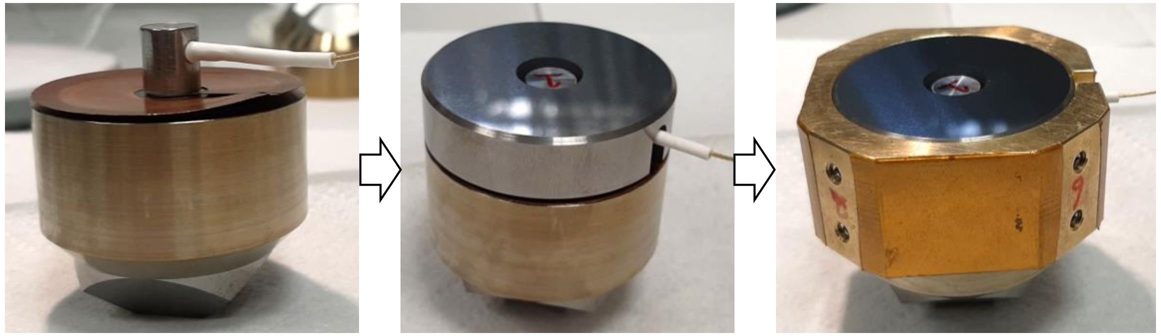
In situ Acoustic Emissions (AE) testing

Acoustic Emissions detection

J. Gasc, S. Ma & R. Farla
(Uni Montpellier / Uni Ningbo / DESY)



MICRO200HF
500-4500 KHZ WIDEBAND
MINIATURE AE SENSOR



S. Ma, J. Gasc & R. Farla, *rev. sci. inst.* 2023

→ **Investigations on the possible origins of earthquakes.**

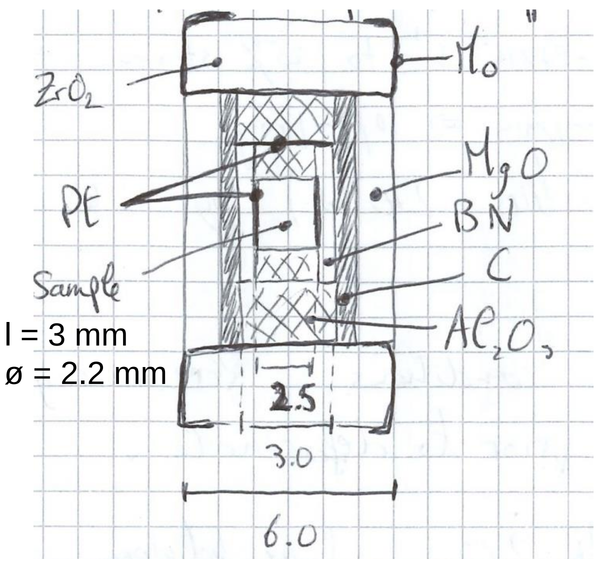
Research highlights

AE generation from deformation of Ge-olivine (Mg_2GeO_4)

Acoustic Emissions detection

J. Gasc, S. Ma & R. Farla
(Uni Montpellier / Uni Ningbo / DESY)

Seismogenic faulting at high pressure is considered responsible for the 'so-called' deep-focus earthquakes in transition zone of the Earth's mantle.



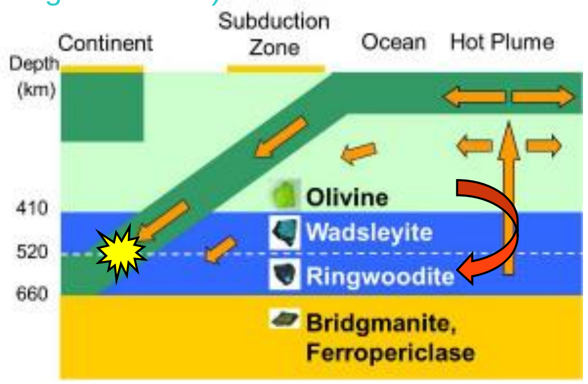
l = 3 mm
ø = 2.2 mm

Assembly (B-epoxy cube)

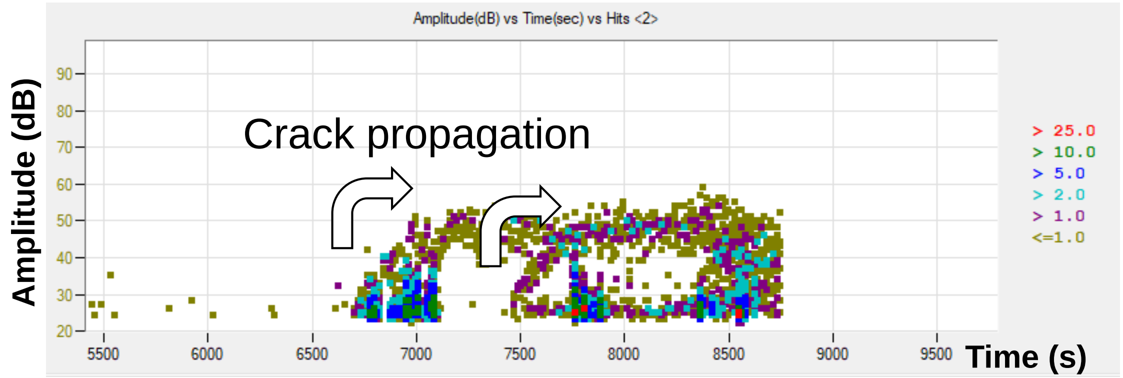
Sample environment:

P =1.5 GPa
T = 700 °C
Ram displacement rate: -8 µm/min
Equivalent strain rate: ~ 3 x 10^{-5} /s

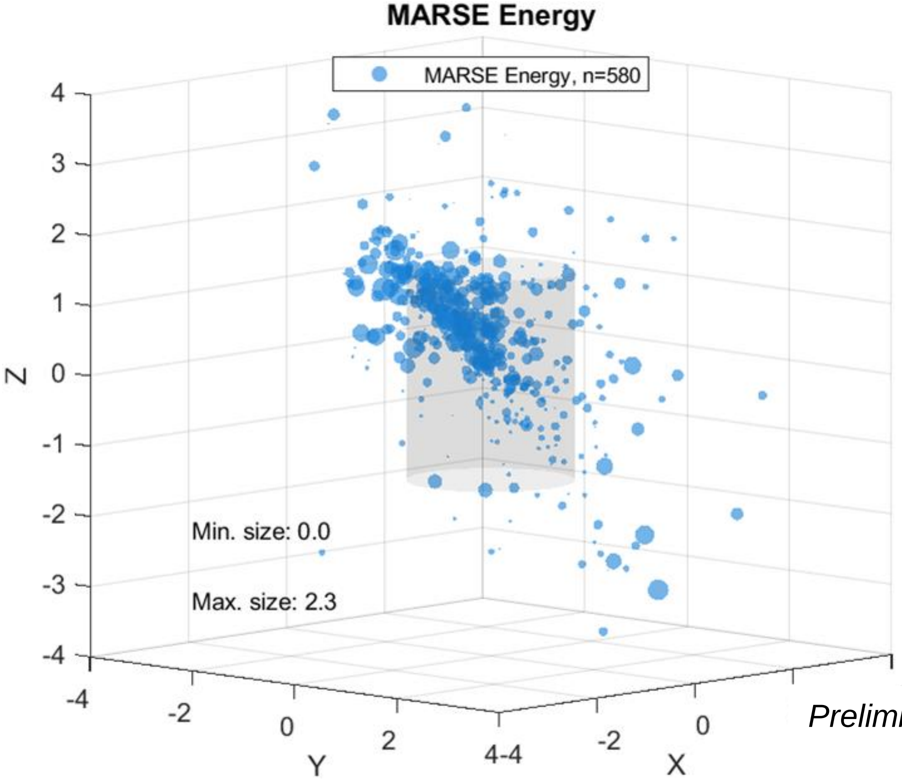
Simulate behavior of $(Mg,Fe)_2SiO_4$



Crack propagation visualized by AE



3d relocation of events



Preliminary results

Research highlights

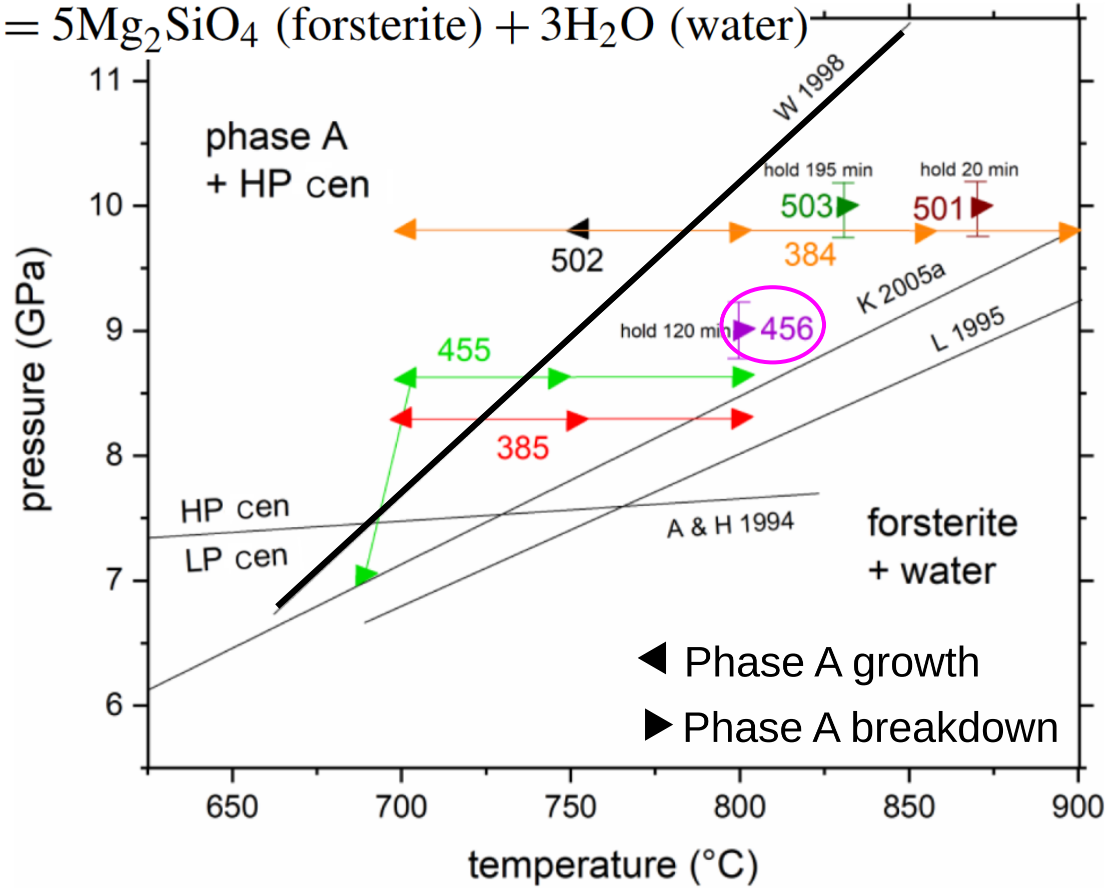
Phase Relations

C. Lathe *et al.* (GFZ / DESY / CMWS)

Determination of the phase boundary for dehydration of phase A, $Mg_7Si_2O_8(OH)_6$ (H_2O-saturated conditions)

The dehydration reaction of phase A:

$$Mg_7Si_2O_8(OH)_6 \text{ (phase A)}$$
$$+ 3MgSiO_3 \text{ (high-}P\text{ clinoenstatite)}$$
$$= 5Mg_2SiO_4 \text{ (forsterite)} + 3H_2O \text{ (water)}$$

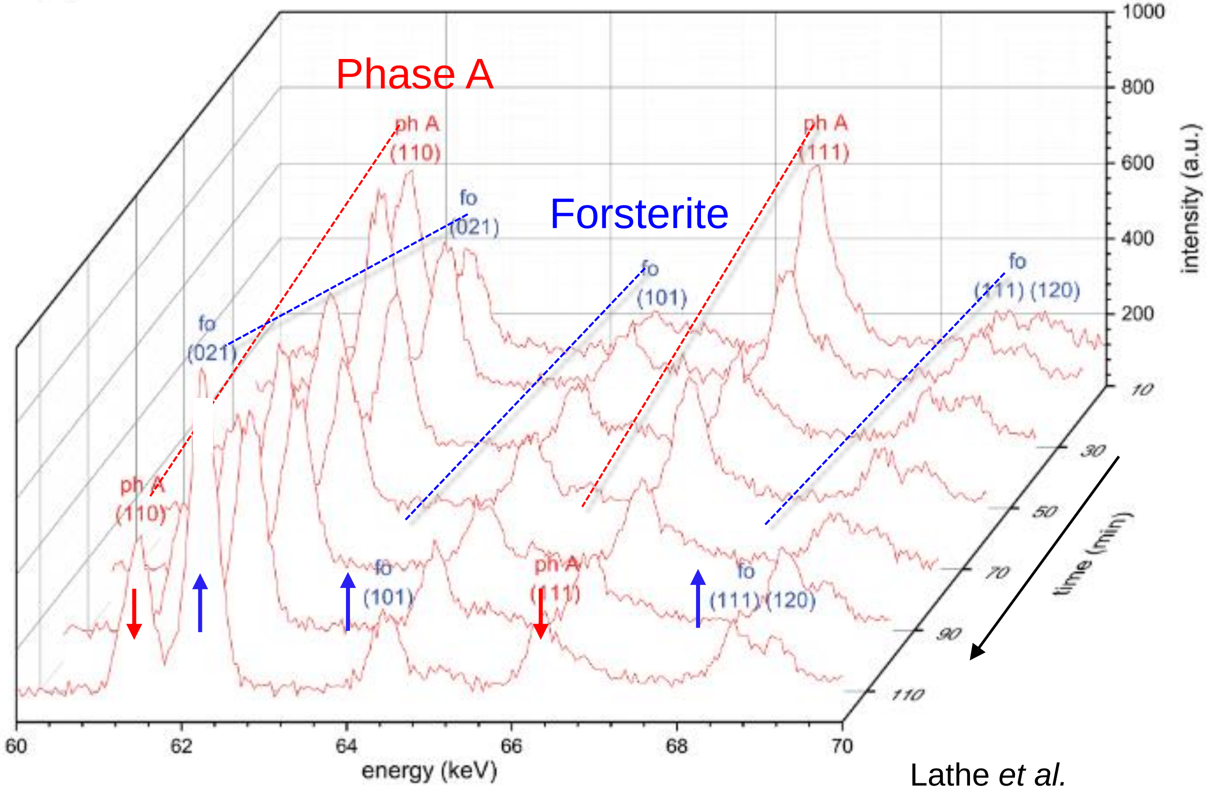


The phase boundary after Wunder *et al.* 1998 is re-established for hydrated phase A.

Run 456

P = 9 GPa

T = 800 °C

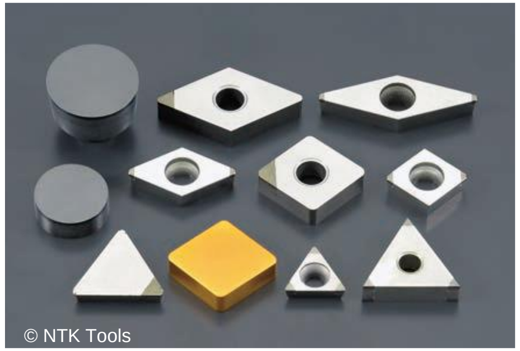


Lathe *et al.*
Eur. J. Min. 2022,2024

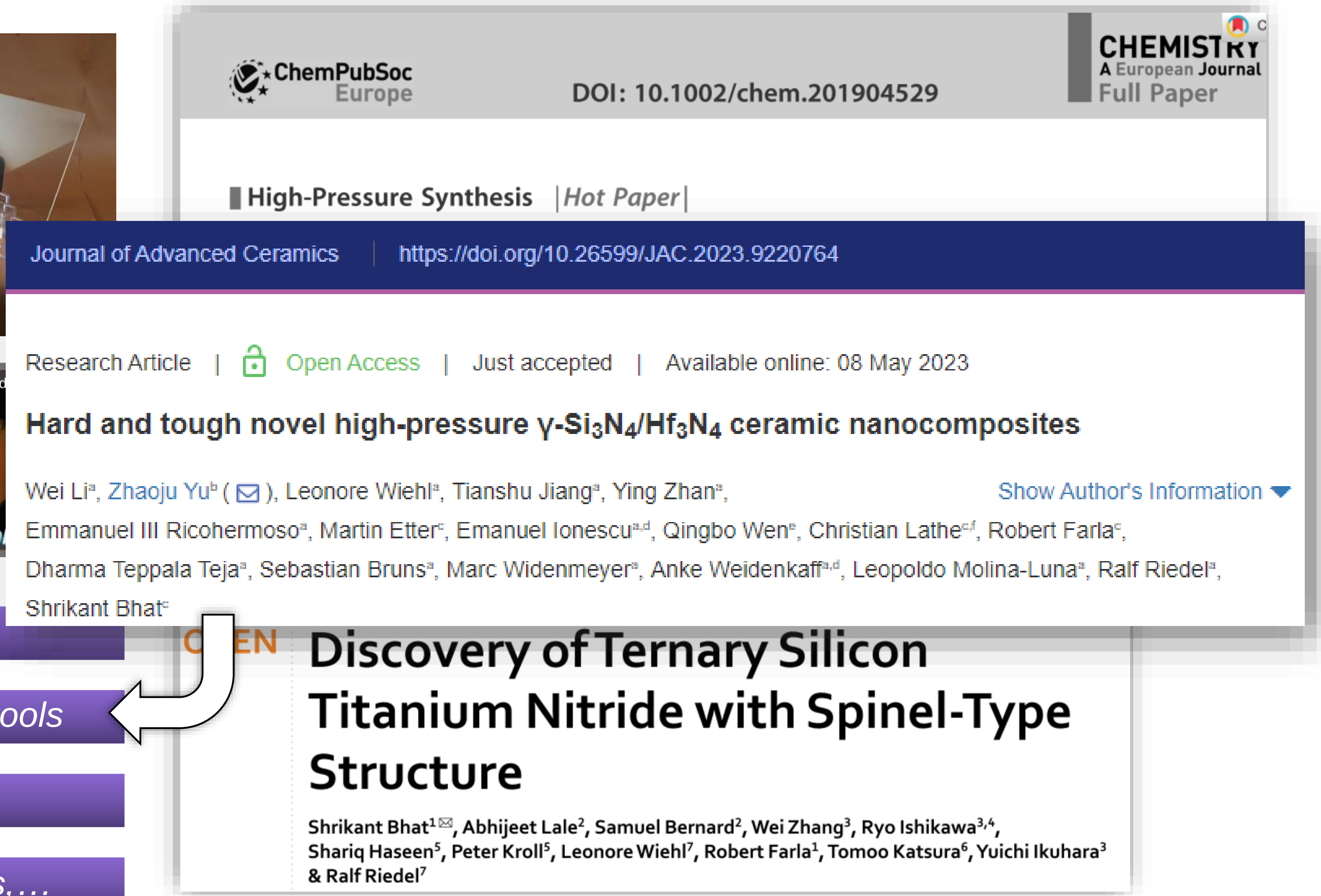
Research highlights

Nitrides
S. Bhat, *et al.* (DESY)

World of Nitrides



- Superconductivity (NbN, HfN)
- Hard Materials (c-BN, γ-Si3N4) – *Cutting tools*
- Magnetic (FeN), Catalysis (Ta_3N_5, TaON)
- Luminescence (SiAlON) – *LEDs, crucibles,…*
- Semiconductors (GaN, AlN) – *LEDs*


ChemPubSoc Europe
DOI: 10.1002/chem.201904529
CHEMISTRY A European Journal
Full Paper

High-Pressure Synthesis | *Hot Paper* |

OPEN

Discovery of Ternary Silicon Titanium Nitride with Spinel-Type Structure

Shrikant Bhat[1], Abhijeet Lale[2], Samuel Bernard[2], Wei Zhang[3], Ryo Ishikawa[3,4], Shariq Haseen[5], Peter Kroll[5], Leonore Wiehl[7], Robert Farla[1], Tomoo Katsura[6], Yuichi Ikuhara[3] & Ralf Riedel[7]

Journal of Advanced Ceramics | https://doi.org/10.26599/JAC.2023.9220764

Research Article | Open Access | Just accepted | Available online: 08 May 2023

Hard and tough novel high-pressure γ-Si_3N_4/Hf_3N_4 ceramic nanocomposites

Wei Li[a], Zhaoju Yu[b], Leonore Wiehl[a], Tianshu Jiang[a], Ying Zhan[a], Emmanuel III Ricohermoso[a], Martin Etter[c], Emanuel Ionescu[a,d], Qingbo Wen[e], Christian Lathe[c,f], Robert Farla[c], Dharma Teppala Teja[a], Sebastian Bruns[a], Marc Widenmeyer[a], Anke Weidenkaff[a,d], Leopoldo Molina-Luna[a], Ralf Riedel[a], Shrikant Bhat[c]

Show Author's Information

Information provided by Dr. S. Bhat

Research highlights

Hydrides & hydrogenations

Hydrides
K. Spektor, D. Beyer, *et al.*
(DESY/Stockholm/Leipzig)

Target-driven projects

Exploration of ternary hydrides

Using BH_3NH_3 as an internal hydrogen source

- **Ba – Si – H**
- **K – Si – H**
- Li – Si(Ge) – H
- Ae – Si(Ge) – H
- RE – Si/Ge/Al/Ga – H
- K – Au – H

LTP project

Investigation of O_2 and F_2 sources (via breakdown) for high-pressure chemistry of novel materials in sealed halide capsules.

E.g.

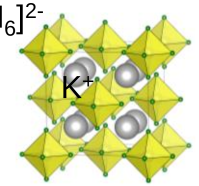
$NaClO_3 \rightarrow NaCl + 3/2\ O_2$ … in progress

$XeF_2 \rightarrow Xe + F_2$ … proposed

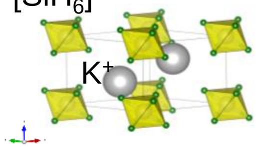
Inorganic Chemistry 2023
pubs.acs.org/IC Article

Formation and Polymorphism of Semiconducting K_2SiH_6 and Strategy for Metallization

Olga Yu. Vekilova, Doreen C. Beyer, Shrikant Bhat, Robert Farla, Volodymyr Baran, Sergei I. Simak, Holger Kohlmann,* Ulrich Häussermann, and Kristina Spektor*

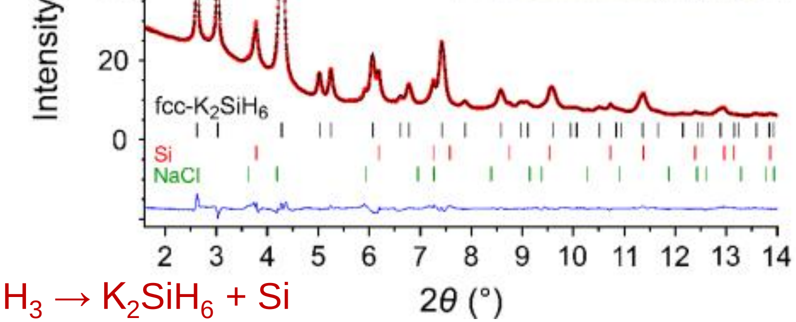


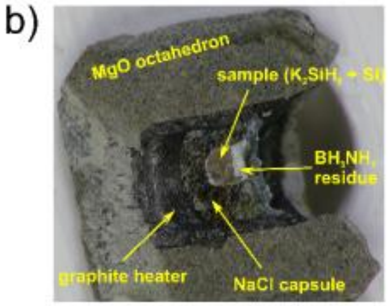
cubic K_2SiH_6
(K_2PtCl_6 structure type)
$a \approx 7.82$ Å at ambient



Above 270°C:
Trigonal K_2SiH_6

$(NH_4)_2SiF_6$ type
$a \approx 5.05$ Å, $c \approx 4.06$ Å





$2\ KSiH_3 \rightarrow K_2SiH_6 + Si$

Information provided by Dr. K. Spektor

Summary

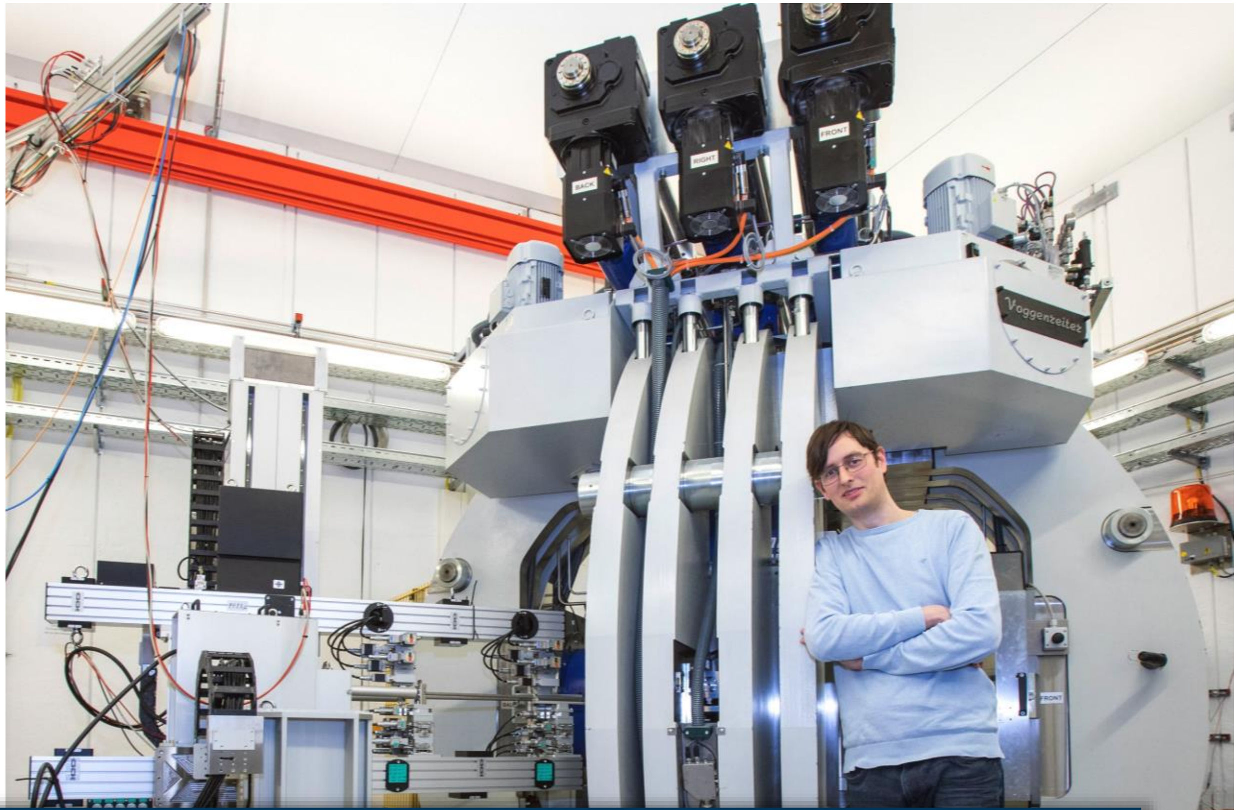
Dedicated user operation at P61B

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

Support for new *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)

- PDF measurements? Tests needed…!
- Electrical conductivity? – possibly from 2024-II



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

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Dr. Spektor
Dr. Lathe (returned to GFZ)

GSECARS, APS, ANL

Prof. Wang
Dr. Officer
Dr. Yu

University Montpellier

Dr. Gasc

TU Darmstadt

Prof. Riedel
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