Status & development of P61B LVP at PETRA III, DESY

Ultra-LVP workshop

Robert Farla
23-09-2021

Acknowledgments:
Shrikant Bhat, Stefan Sonntag, Artem Chanyshev, Shuailing Ma, Christian Lathe, Kristina Spektor, Tomoo Katsura (BGI), Ulrich Häussermann (Stockholm Uni), Holger Kohlmann (Leipzig Uni)
DESY Support Groups: FS-BT, FS-EC, FS-TI, Machine group

HELMHOLTZ RESEARCH FOR GRAND CHALLENGES

BGI Bayerisches Geoinstitut

UltraLVP RESEARCH PROJECT

erc European Research Council

P61B LVP Mission

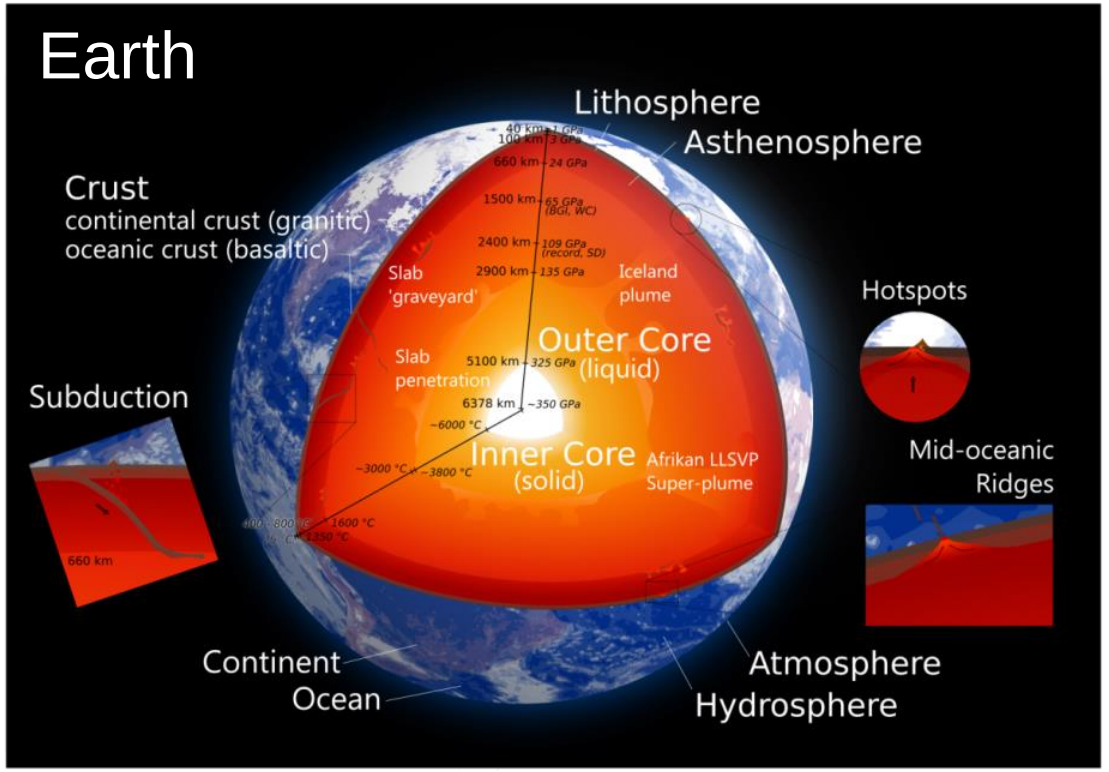
Applications in geo- and material sciences:

For 50% beam time:

- Phase relations:
  - Transformation/nucleation
  - Melting curves (solidus/liquidus)
  - Equations of state
- Crystallography (w/ CAESAR or monochromator)
- Controlled rock deformation
- Melt viscosimetry measurements
- Structure of amorphous materials

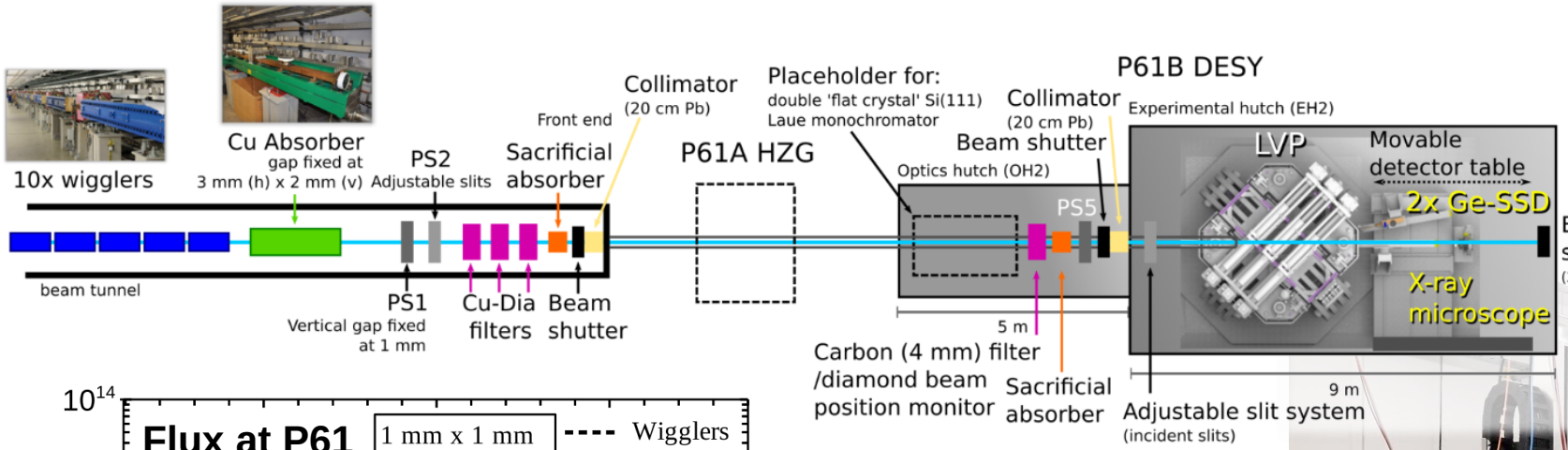
For 50% beam time / experiment time:

- MA experiments with complementary *in situ* techniques:
  - Ultrasonic interferometry
  - Acoustic Emissions testing
  - Electrical conductivity (upcoming)
- Synthesis of novel (recoverable) materials

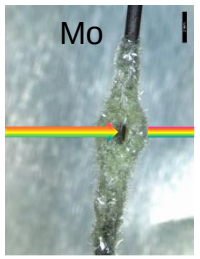


Beamline layout

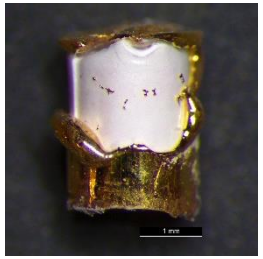
The Large Volume Press (LVP) extreme conditions beamline (50% in situ / 50% ex situ)



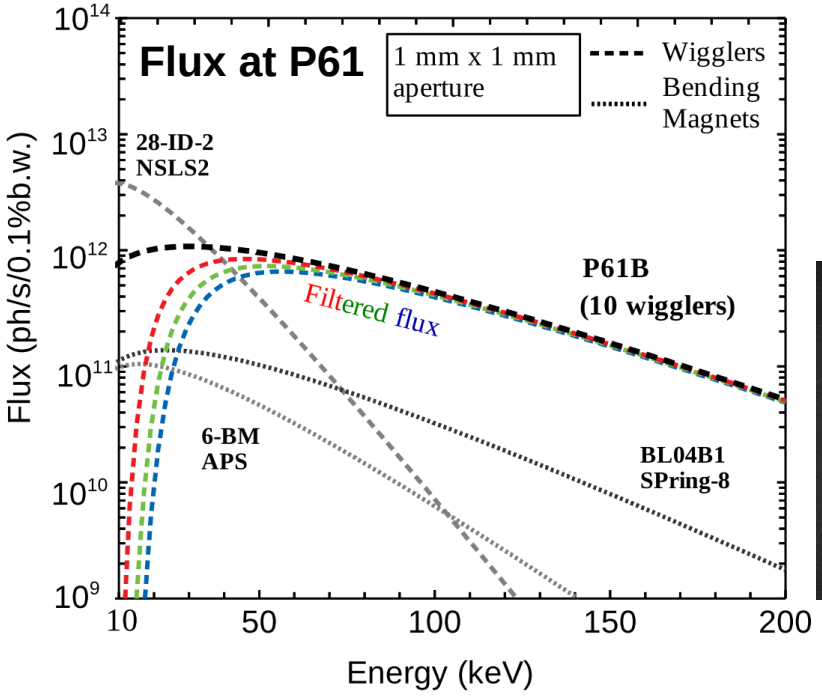
CAUTION: HOT BEAM!

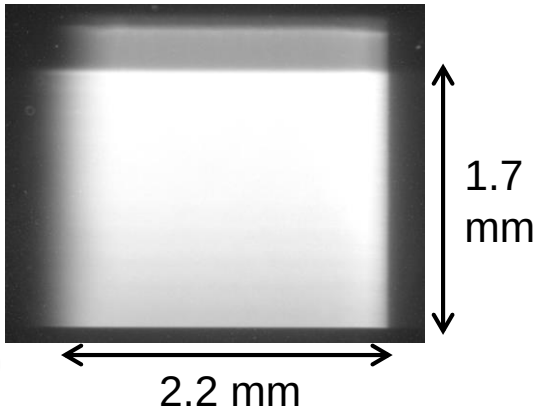


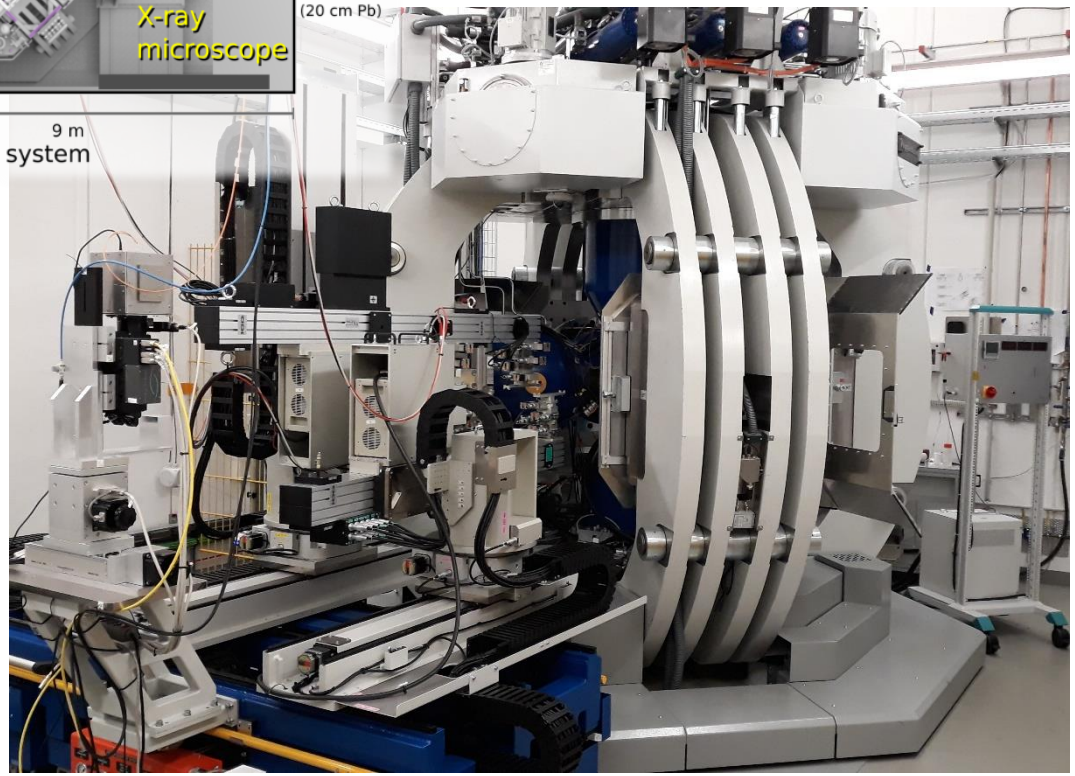
Crystal growth of MoO_3



Molten Au capsule



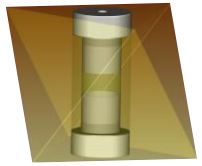




New detector positioning system (since March 2021)

High-pressure techniques

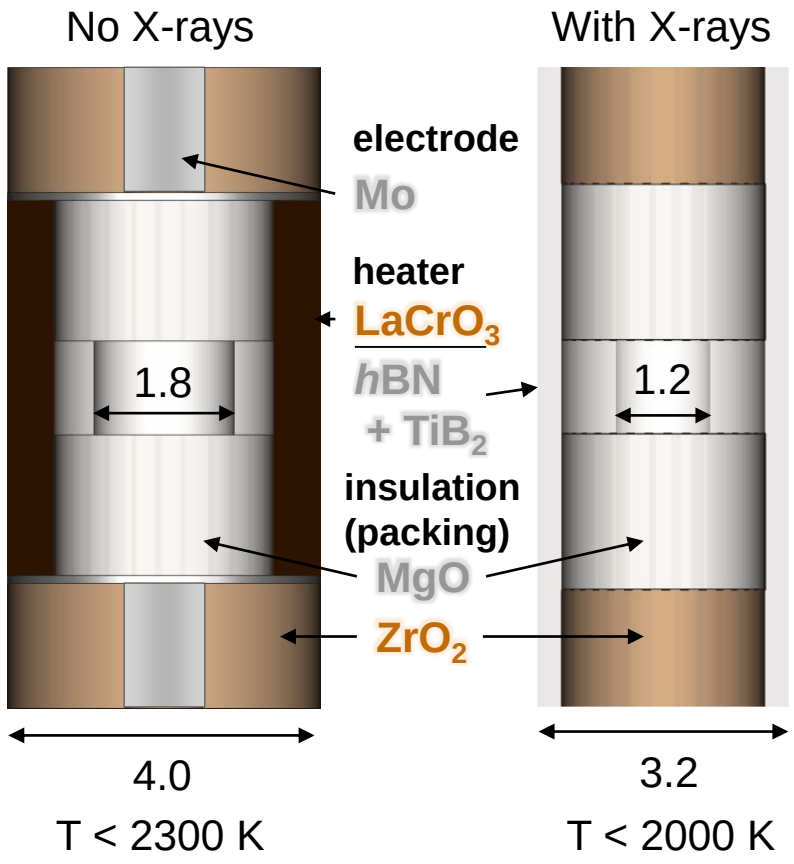
Standard assemblies for isotropic compression

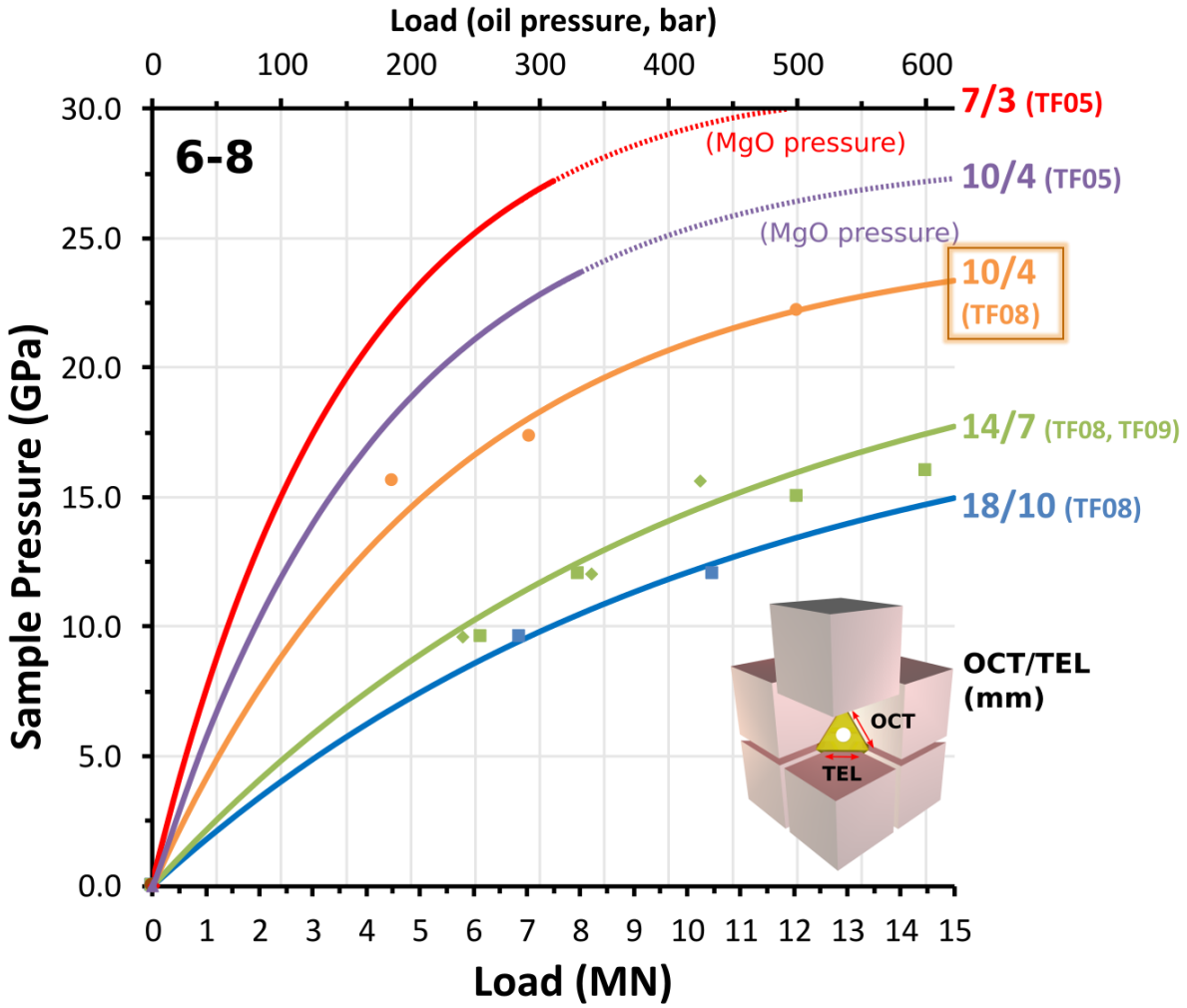


Simultaneous generation of ultrahigh pressure and temperature to 50 GPa and 3300 K in multi-anvil apparatus [also at P61B]
Longjian Xie *et al. Rev. Sci. Instrum.* 2021 *(in press)*

‘Kawai’ 6-8 mode

Example assembly “10/4” for P ≥ 15 GPa

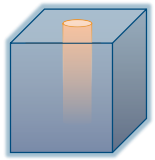




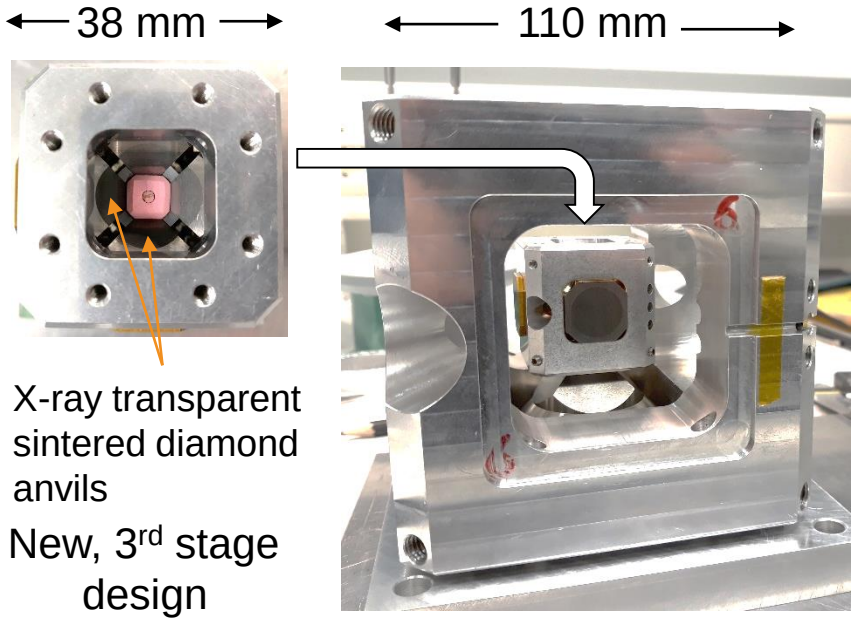
Original assembly design by Dr. Nishiyama

High-pressure techniques

Standard assemblies for *in situ* studies of rock deformation

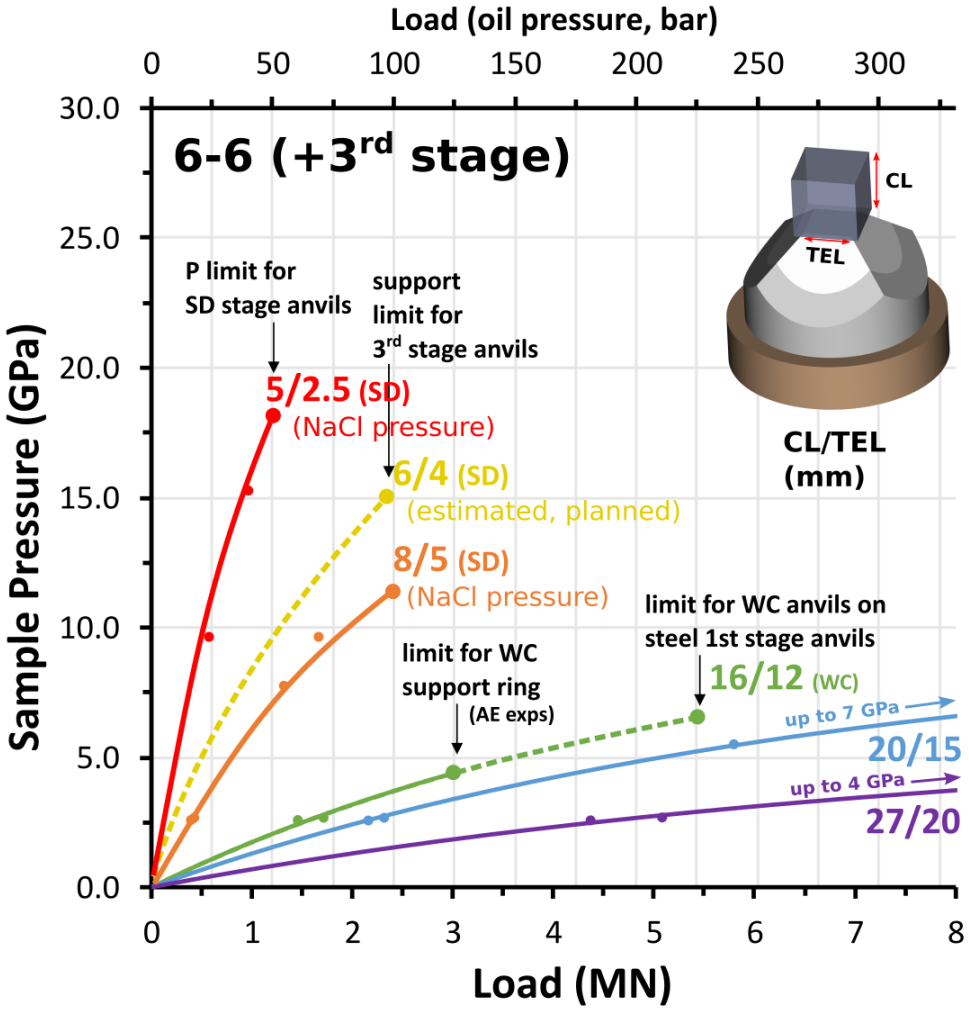


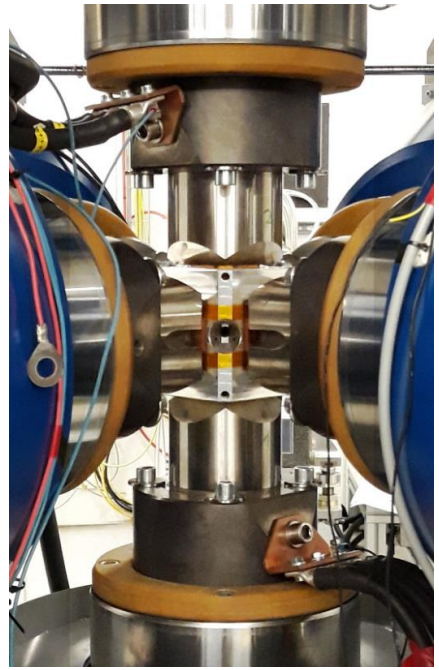
‘Cubic’ 6-6 mode



X-ray transparent sintered diamond anvils

New, 3rd stage design





Inside the LVP ‘Aster-15’

Generally for *ex-situ* synthesis

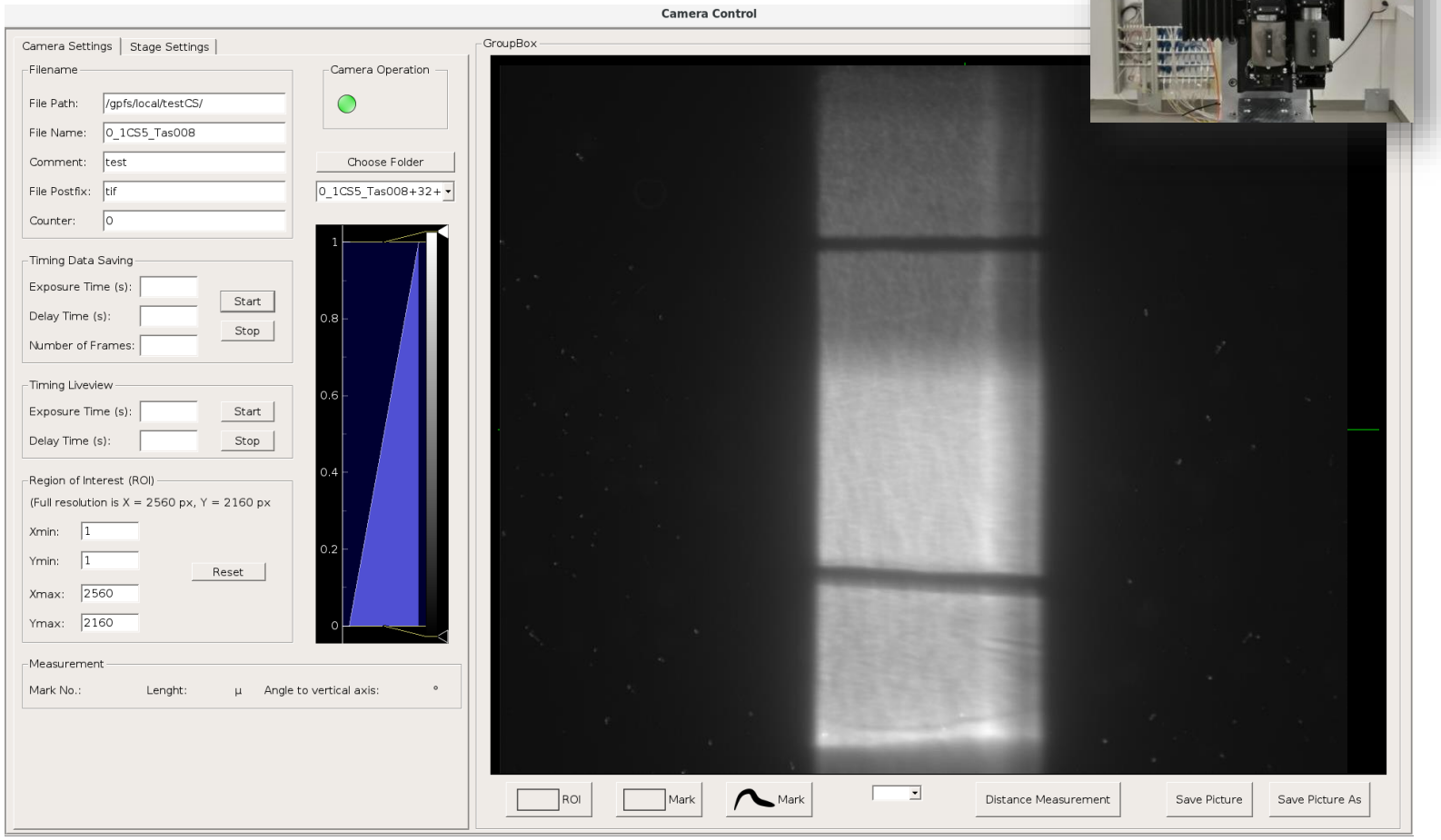
The whitebeam X-ray microscope

Optique Peter
OPTICAL & MECHANICAL ENGINEERING

pco.

X-ray radiography

- **Double objectives (5x, 10x)** for high-resolution, full beam imaging
- **GGG: Eu / LuAG:Ce scintillators:** 20, 40 µm
- **PCO.edge 5.5 MP sCMOS camera**
  - True global & rolling shutter
  - 100 fps @ full-resolution (up to 1000 fps for ROI)
  - Live view & frame capture
  - LVP Z-stage imaging scan

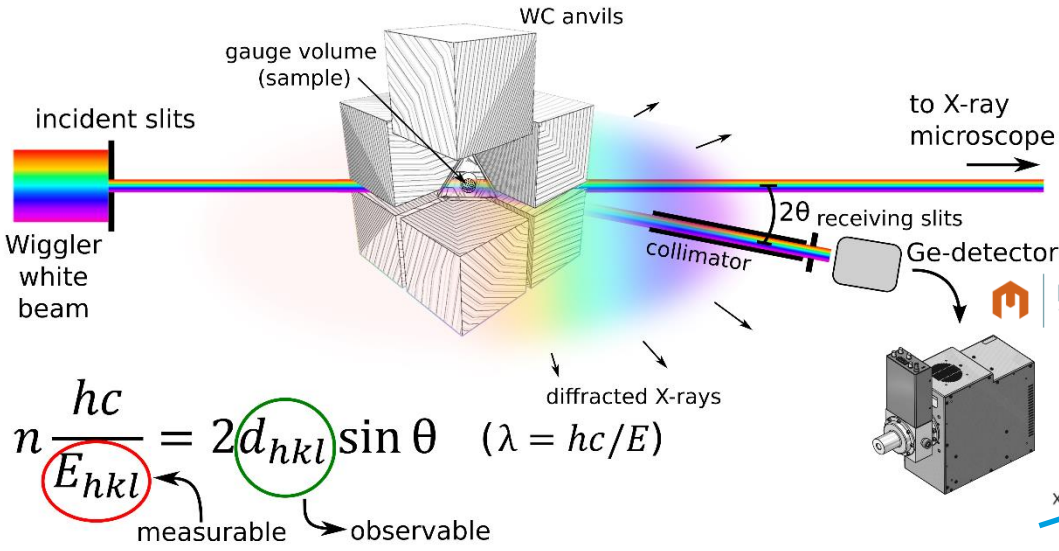


X-ray powder diffraction using white beam

Energy-dispersive X-ray diffraction (ED-XRD) in the Large Volume Press

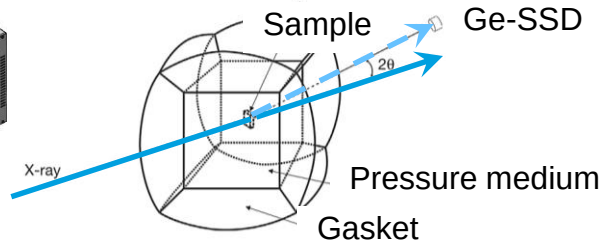
ED-XRD in the LVP

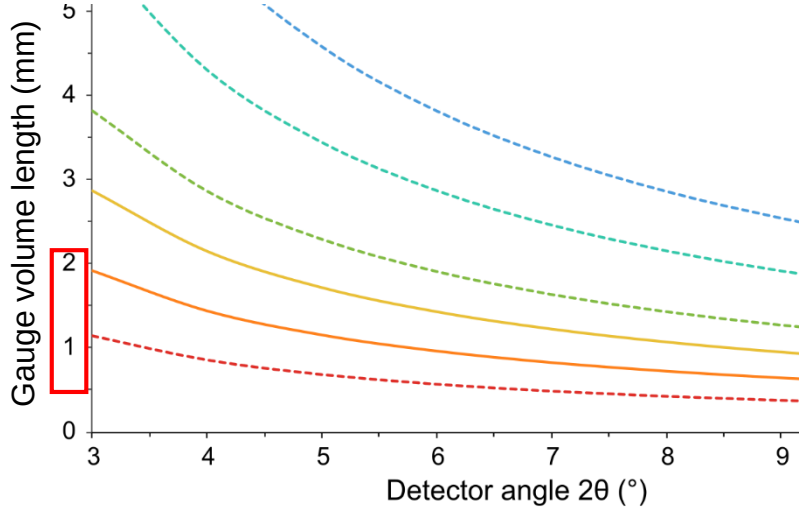
1. 2x Ge-SSD, electronic cryostat, CMOS pre-amp & 4k digital analyser (no saturation at millions of cps)
2. High spatial resolution (small gauge volume)
   → avoid high temperature & pressure gradients
   → multiple samples in one experiment
3. Useful for low-Z (X-ray transparent) materials
4. Fast acquisition (10-100 s) covering large Q-range
   → great for reaction kinetics experiments / melts & glasses



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

measurable — observable

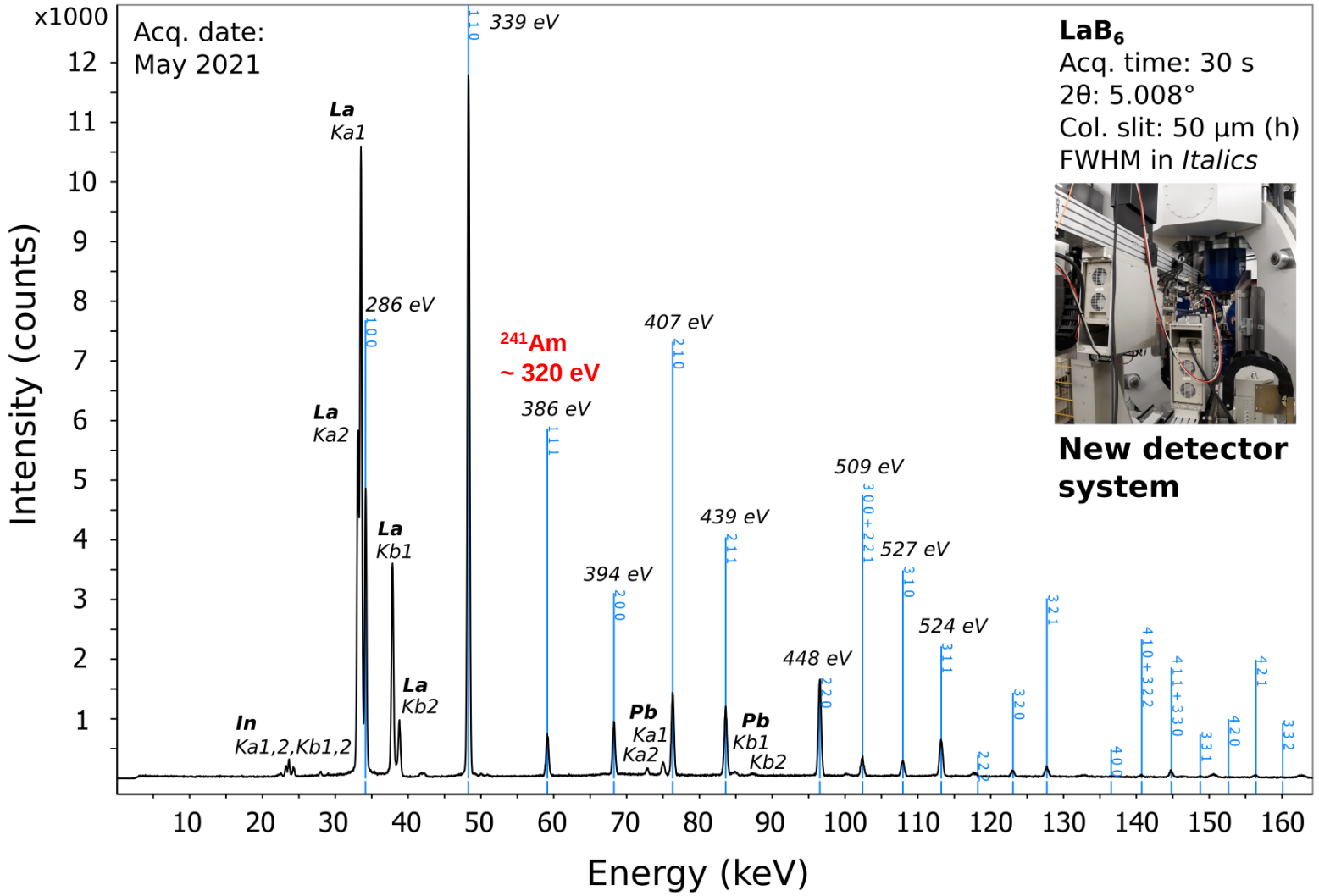




Ge-SSD (2x)	Mirion (Canberra)
Horz. detector positions	1xGe : min 3° - max 20° 2xGe : min 5° - max 10°
Horz. & vert. positions	Ge_{vert} : min 7.5° - max 23° Ge_{horz} : min 6.5° - max 10°

X-ray powder diffraction using white beam

Energy-dispersive X-ray diffraction (ED-XRD) in the Large Volume Press

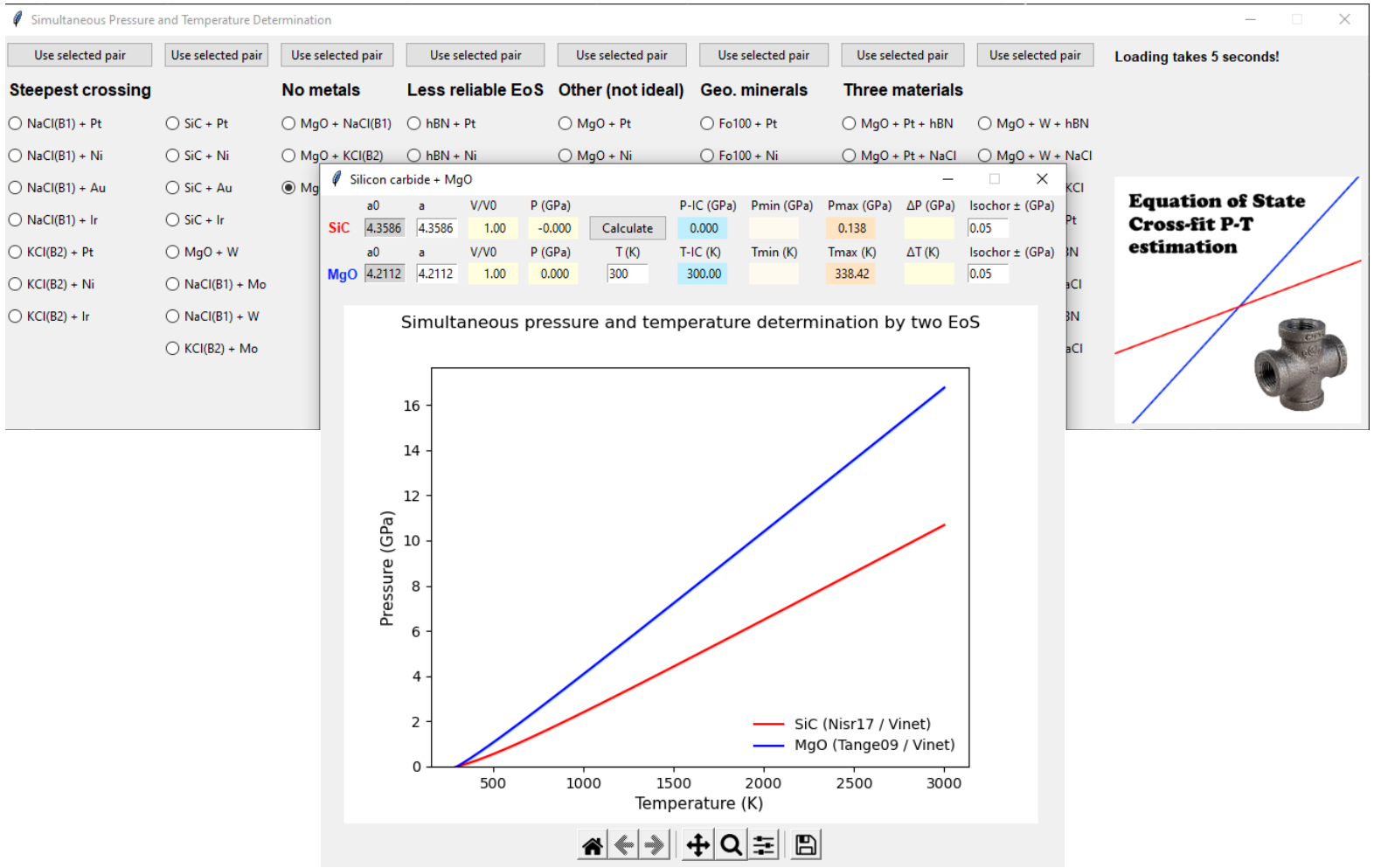


ED-XRD resolution test on NIST powder(s) e.g. LaB_6 in a 0.8 mm diameter capillary tube.

- Good resolution!
- Low dead time / high count rates with CMOS pre-amplifier on Ge-SSD.

X-ray powder diffraction using white beam

Energy-dispersive X-ray diffraction (ED-XRD) in the Large Volume Press

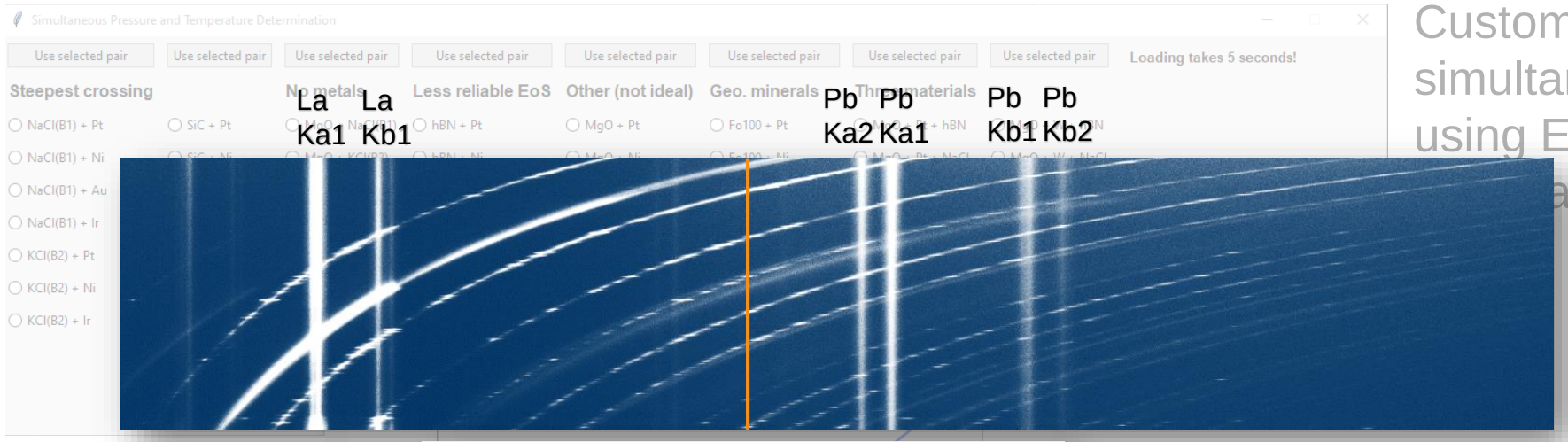


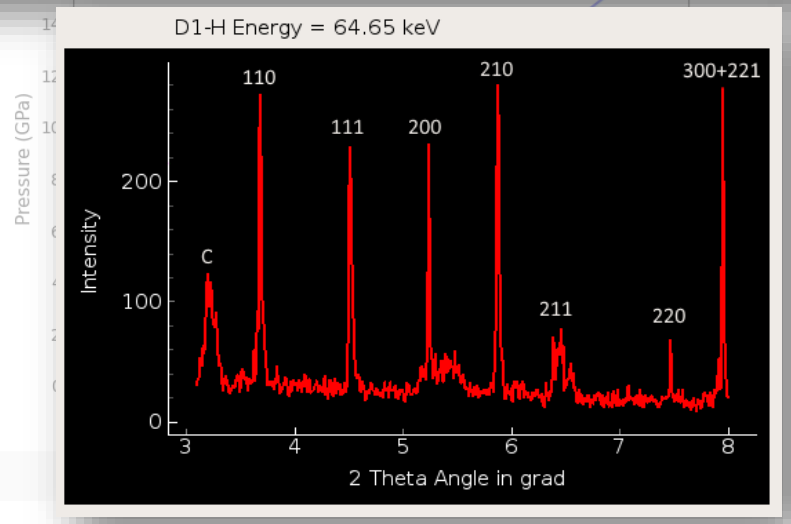
Custom software for simultaneous PT estimation using EoS of pairs of materials (e.g. MgO+SiC)

…And other ‘user-friendly’ beamline tools.

X-ray powder diffraction using white beam

Energy-dispersive X-ray diffraction (ED-XRD) in the Large Volume Press





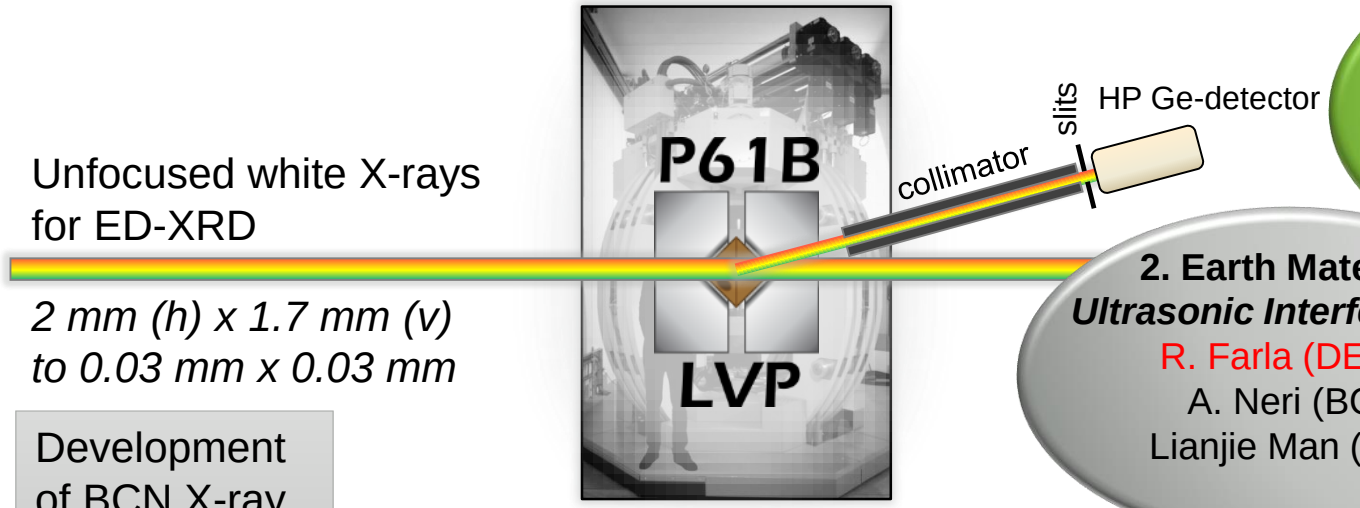
Custom software for simultaneous PT estimation using EoS of pairs of materials (e.g. MgO+SiC)

Combined angle and energy-dispersive structural analysis refinement (CAESAR) (Wang et al. 2004).

First result on LaB_6.

- Spotty pattern can be improved by simultaneous press rotation.
- Gauge volume to be calibrated better.

P61B LVP Research & Development



Unfocused white X-rays for ED-XRD

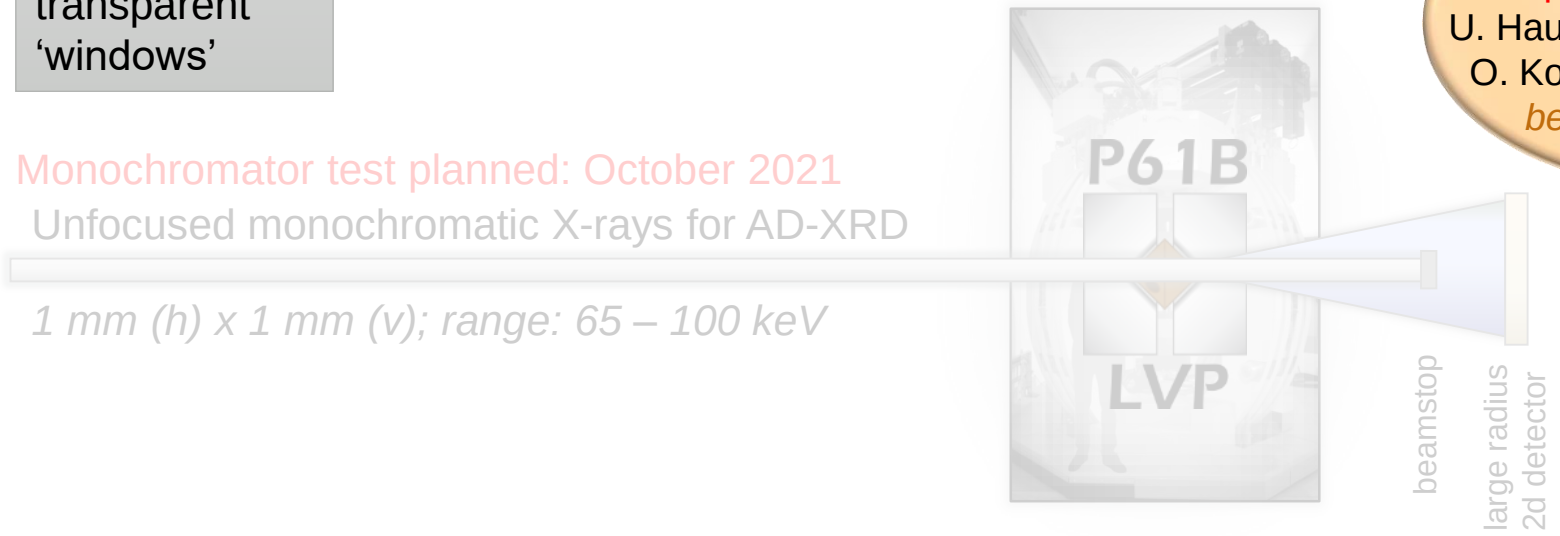
2 mm (h) x 1.7 mm (v) to 0.03 mm x 0.03 mm

Development of BCN X-ray transparent 'windows'

Monochromator test planned: October 2021

Unfocused monochromatic X-rays for AD-XRD

1 mm (h) x 1 mm (v); range: 65 – 100 keV



1. Earth Materials
HP Silicates
A. Chanyshev (BGI/DESY)
T. Katsura (BGI),…
(Talk @ 24.09, 15:00)

Materials Science
Novel nitrides research
S. Bhat (DESY)
R. Riedel (Uni Darmstadt)

2. Earth Materials
Ultrasonic Interferometry
R. Farla (DESY)
A. Neri (BGI)
Lianjie Man (BGI)

CMWS
Hydrous phases
C. Lathe (DESY/GFZ)
M. Koch Müller (GFZ)
M. Sieber (Potsdam/GFZ)

RAC
Ternary hydrides
K. Spektor (Leipzig/DESY)
U. Haussermann (Stockholm)
O. Kohlmann (Leipzig Uni.)
beam time upcoming

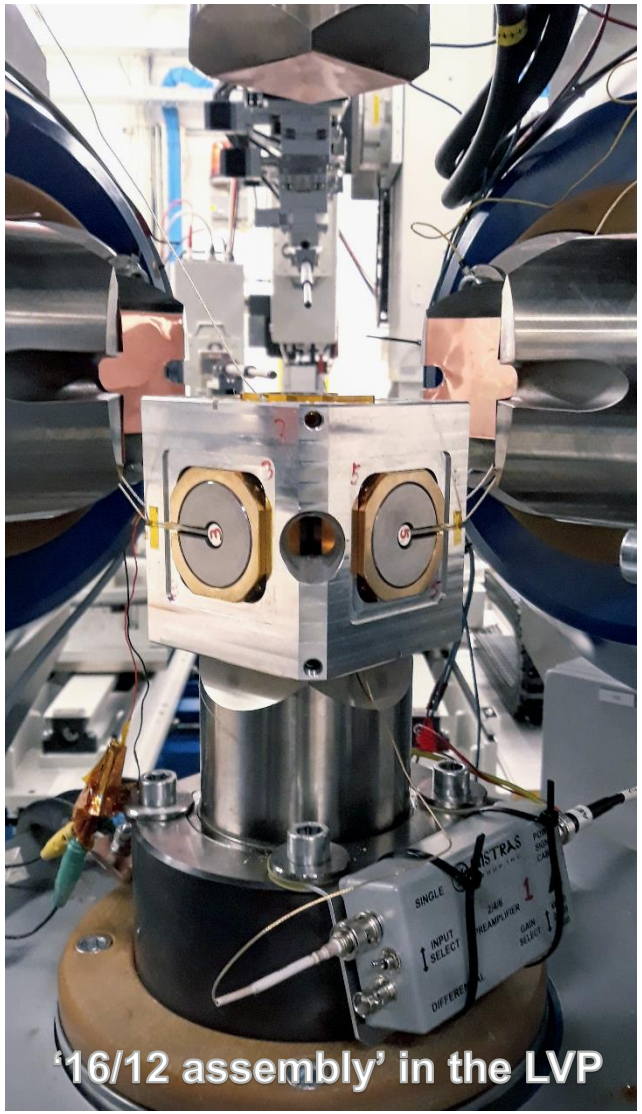
3. Earth Materials
Acoustic Emissions
S. Ma (Jilin Uni, China/DESY)
J. Gasc (Uni Montpellier)
S. Incel (Bochum)

4. Earth Materials
Rock Mechanics
R. Farla (DESY)
Contact me

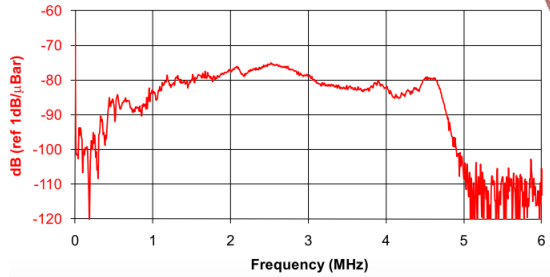
Acoustic Emissions testing (*in situ*)

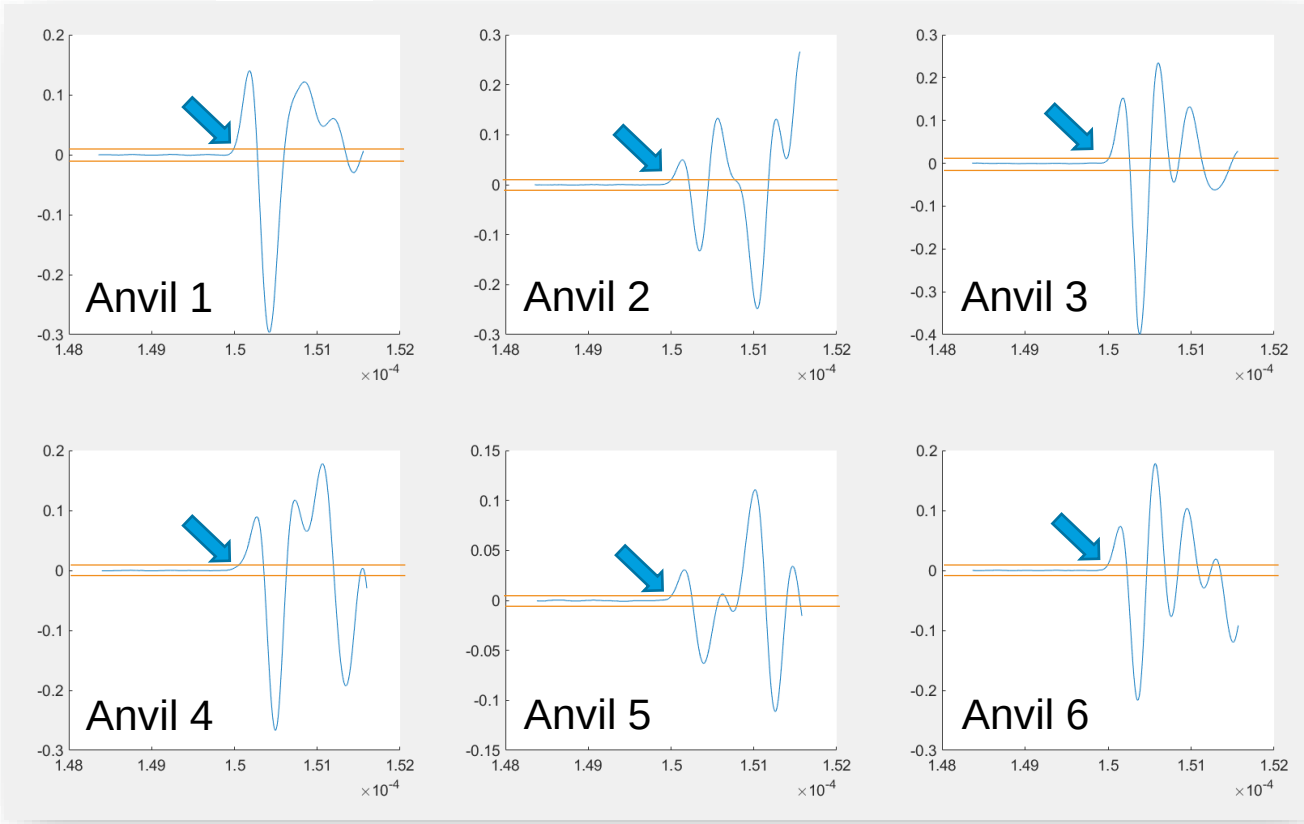
Methodology (MA6-6 compression)

Acoustic Emissions
S. Ma (Jilin Uni, China/DESY)
J. Gasc (Uni Montpellier)
S. Incel (Bochum)



Magnified portion of an 'event': nearly-simultaneous hits on the sensors of each of the 6 anvils. →



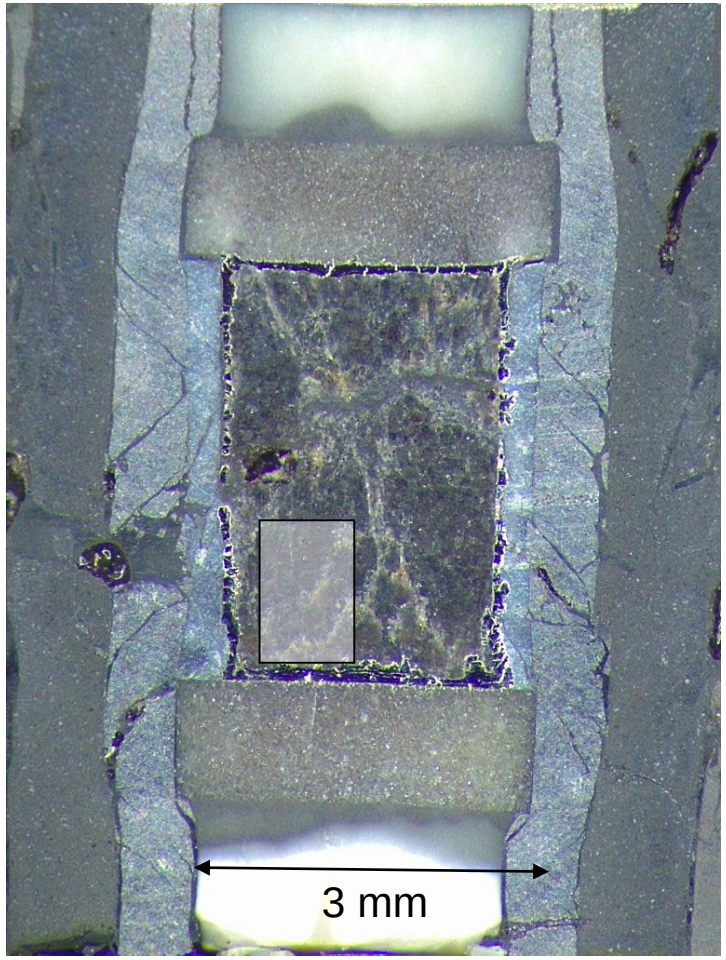


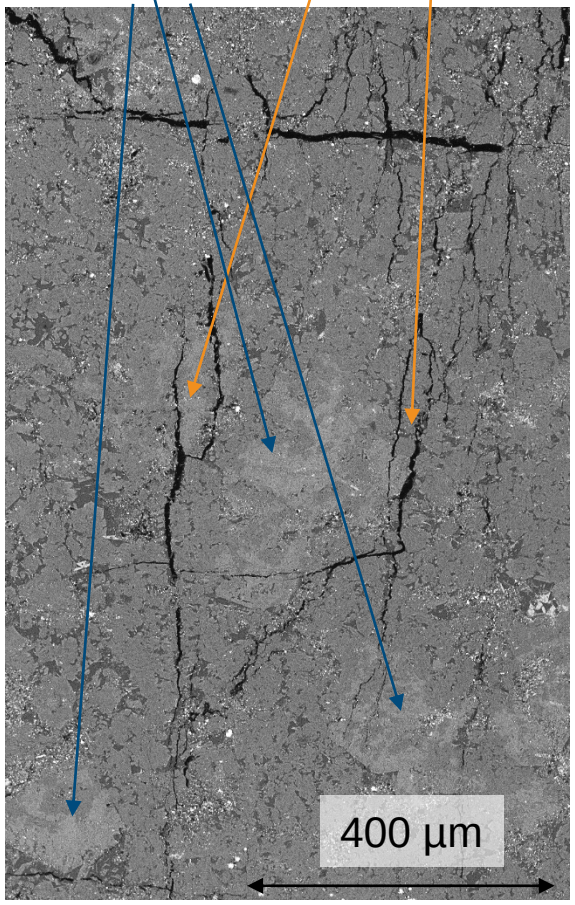
Aim: to investigate focal mechanisms & radiated energy of crack propagation.

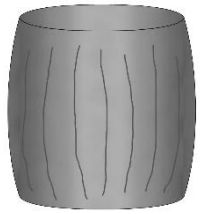
Acoustic Emissions testing (*in situ*)

Dehydration of amphibolite: Natural amphibolite deformed in the eclogite stability field

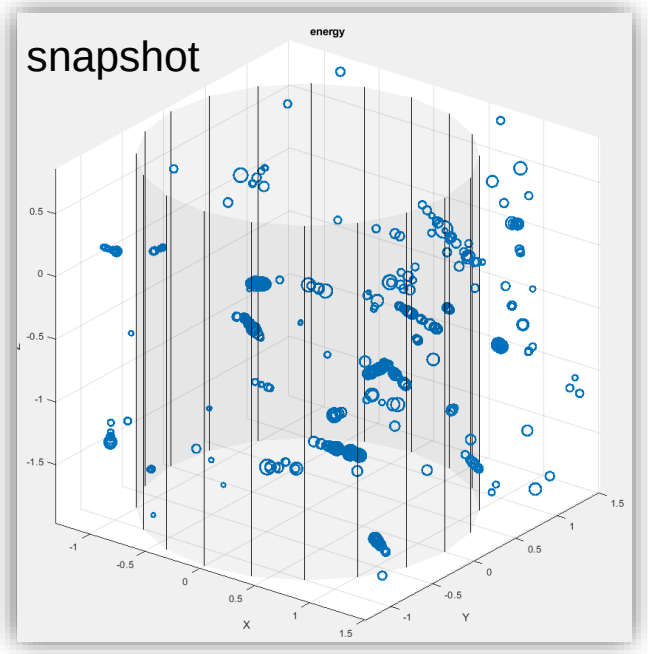
Recovered deformed sample
(700 °C / 55 bar ~ 2 GPa / 4 x 10^{-5} /s)



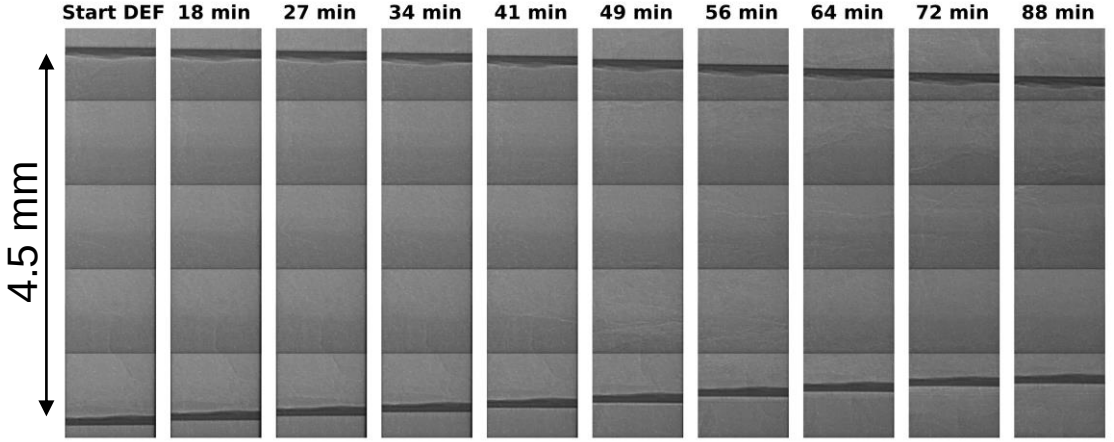



cracked specimen

Acoustic Emissions:
3d event location
(circle size ∝ energy release)



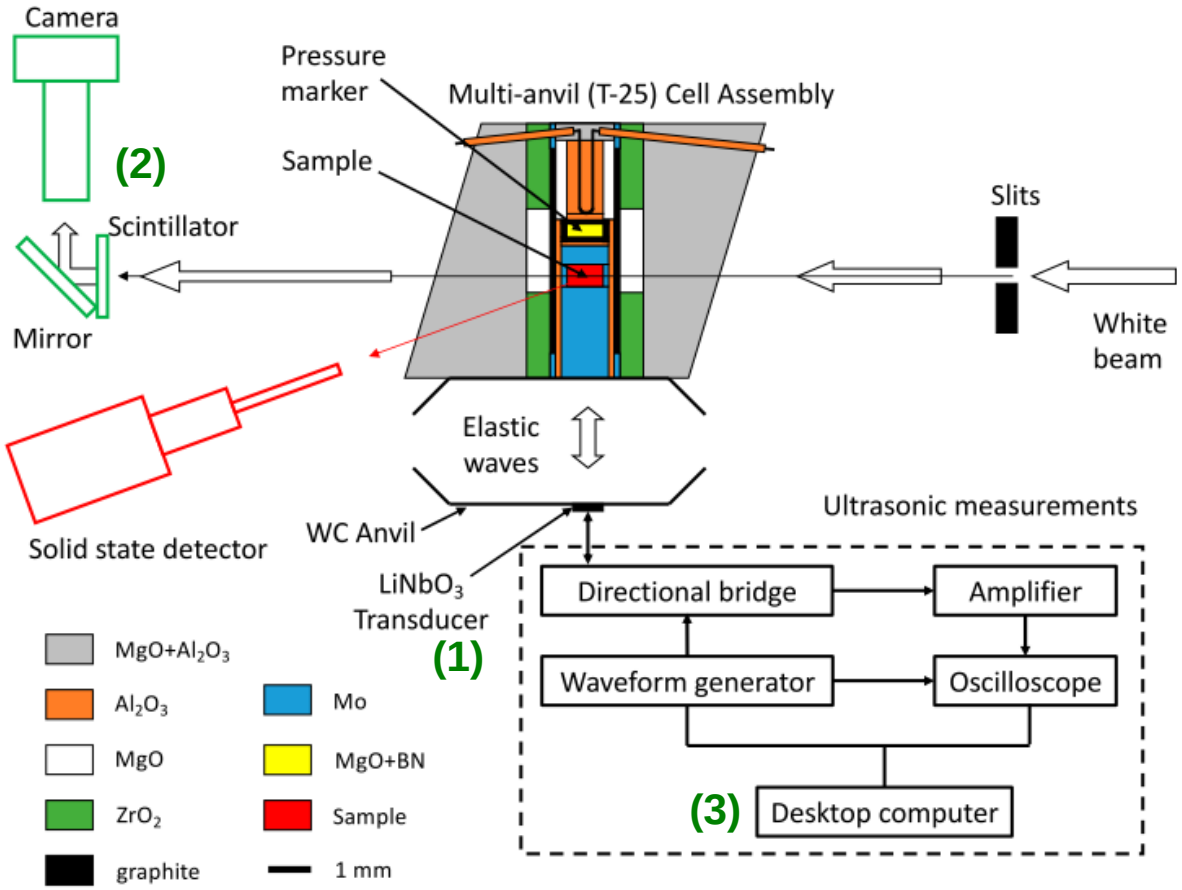
Strain history



Wave speed measurements (*in situ*)

Ultrasonic Interferometry: Now available at P61B

Ultrasonic Interferometry
R. Farla (DESY)
A. Neri (BGI)
Lianjie Man (BGI)



Setup at GSECARS, APS (USA) – Jing *et al.* 2020

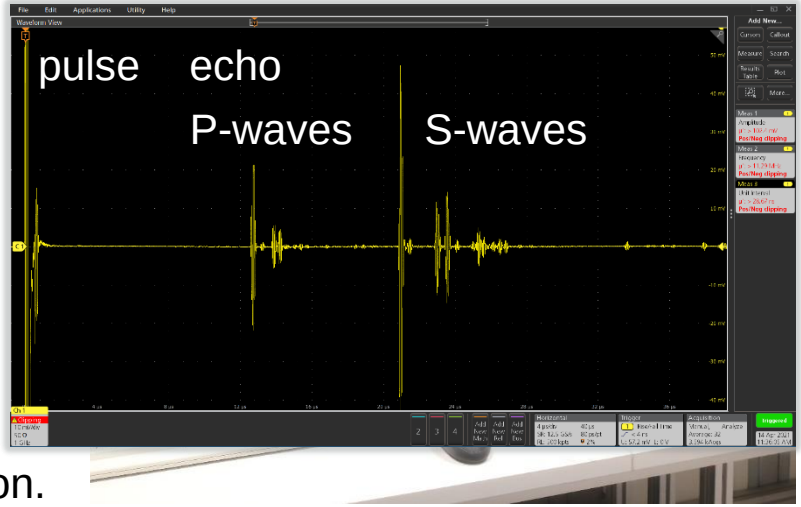
General method

(1) A LiNbO3 sensor of choice on the back of a mirror polished anvil, transmits a pulse and receives an echo.

(2) Simultaneous imaging (**radiography**) provides sample length with sub-pixel resolution (< 1 µm).

(3) Wave speed at given P,T is calculated to determine elastic moduli (with density information) and/or pressure.

→ Simultaneous measurement of P and S wave travel time, density, and sample length.



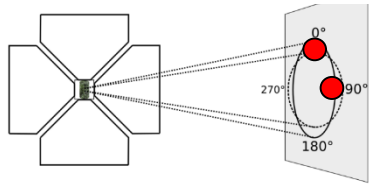
→ Acquisition routine can be scripted using python.

Rock deformation (*in situ*)

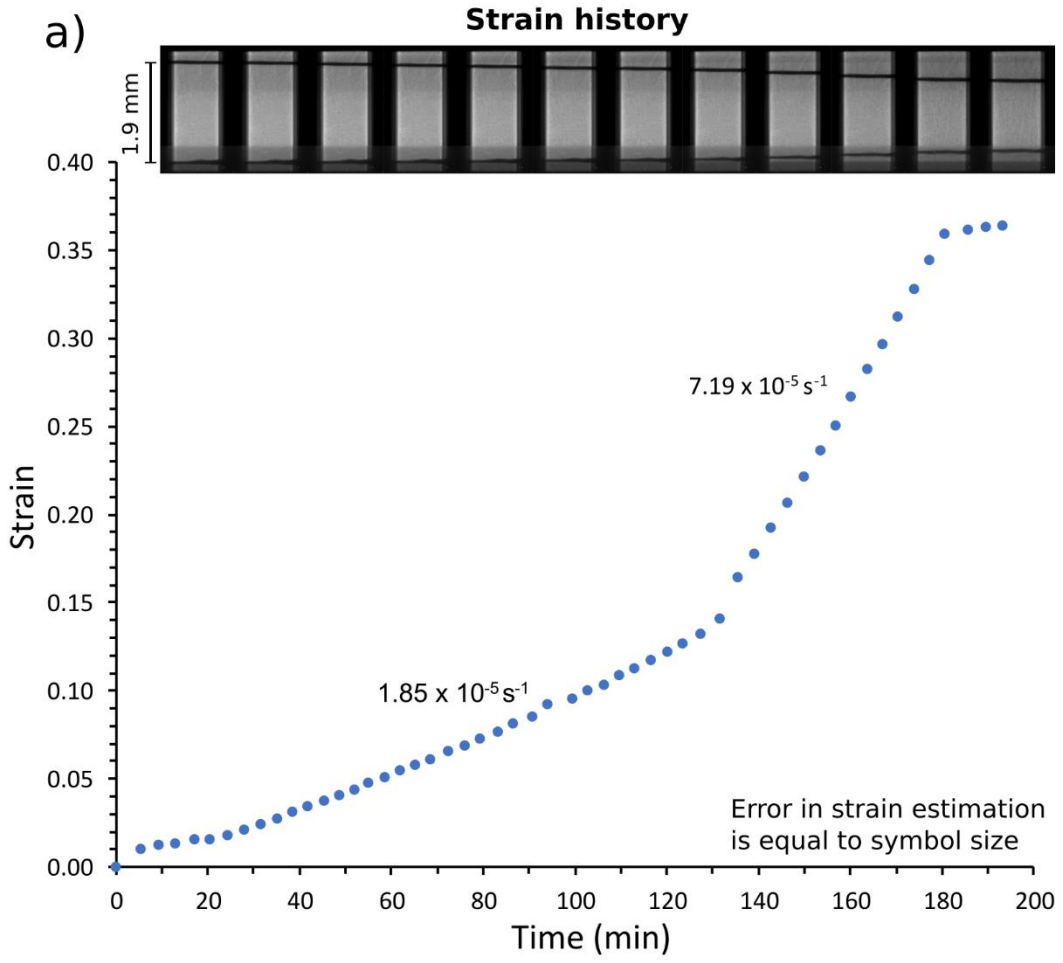
Concept experiment: deformation of NaCl

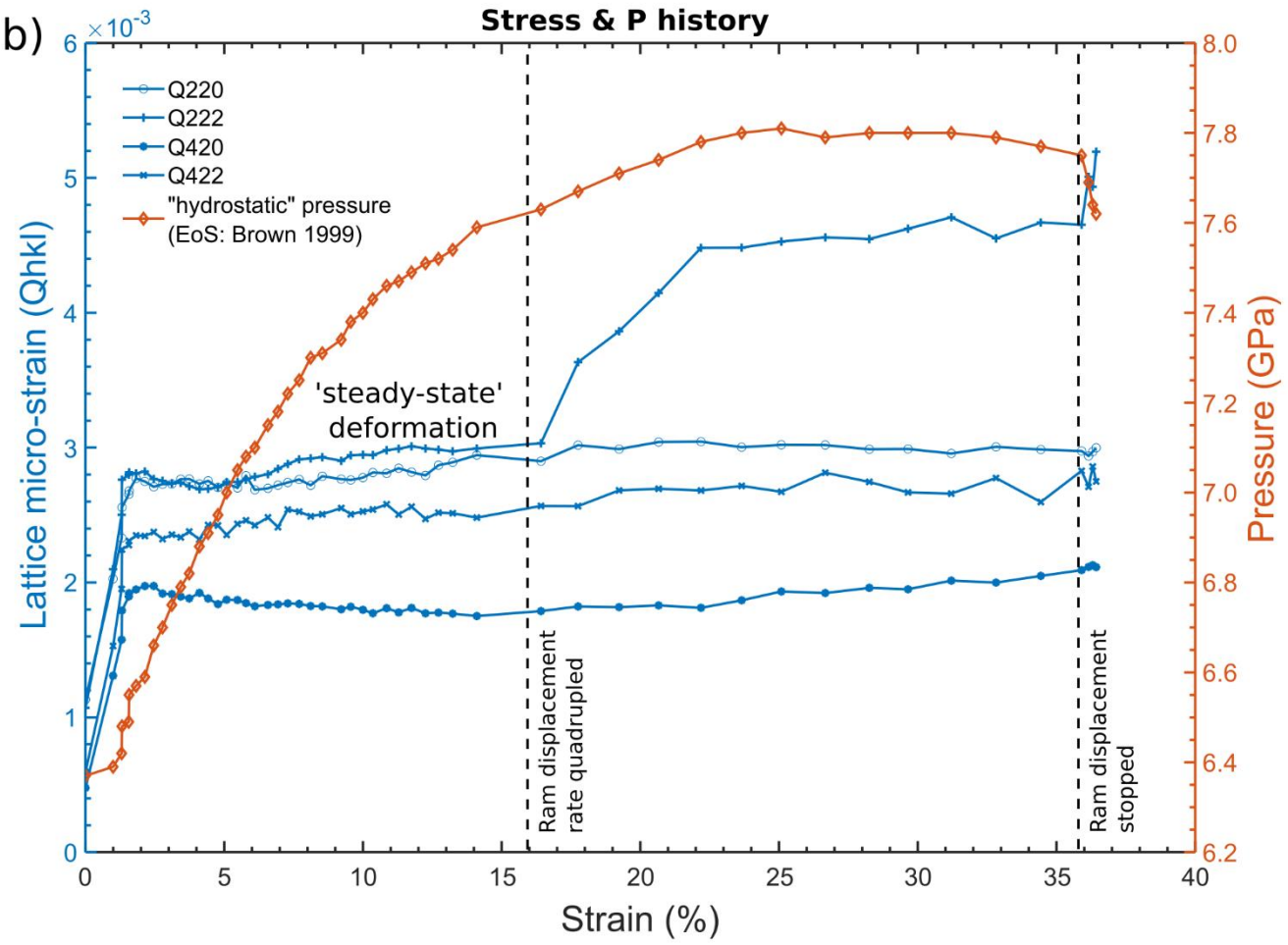
- 1.9 mm sample deformed at room T and high P.
  Diffraction acq. using 2 Ge-SSD (at 0°, 90° azimuth, 2θ = 7.5°)

2 Ge-SSD



Rock Mechanics
R. Farla (DESY)



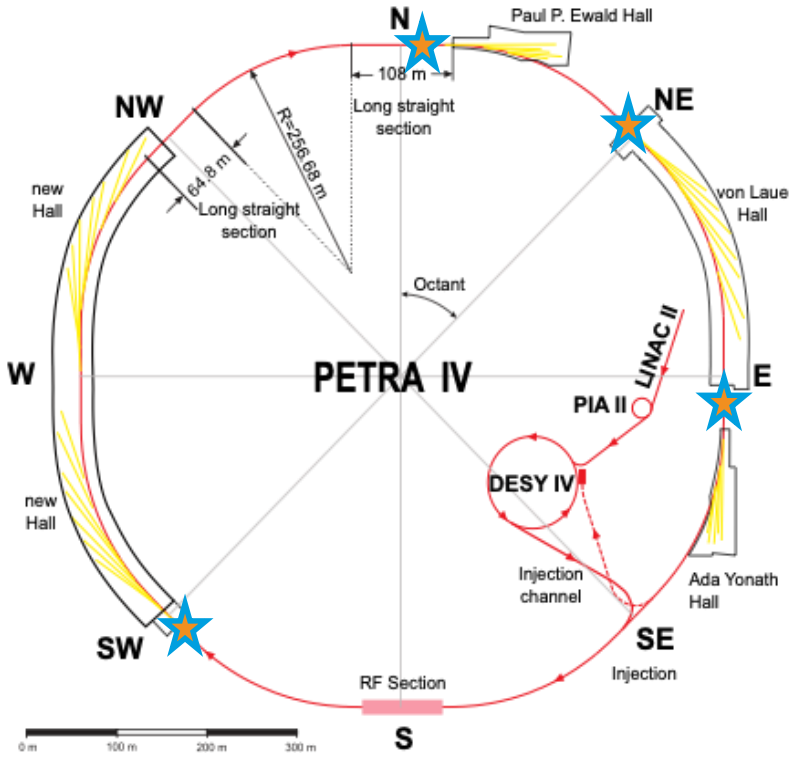


PETRA IV

Why? See CDR report at DESY.de

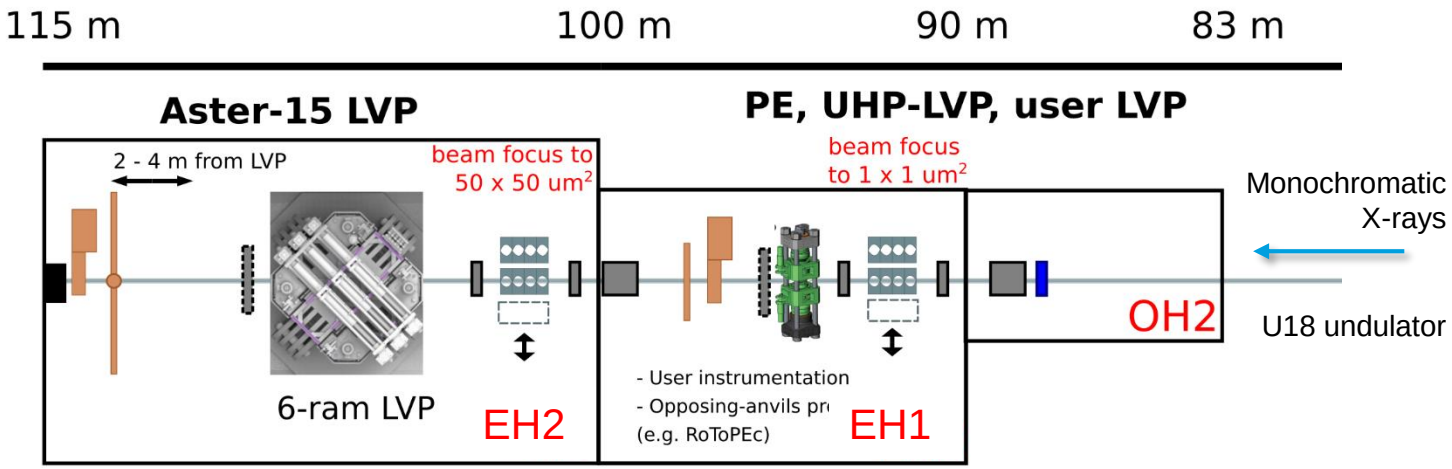
What? New *in situ* LVP beamline at PETRA IV, supported by proposals from the user community (Thank you!)

Where? LVP position to be decided.



When? PETRA III end operation: 2026.
PETRA IV BL operation: 2028 (existing halls), 2028-30 (west hall).

Possible BL concept:



- High energies (40 – 120 keV harmonics)
- High-res PXRD / Imaging / µ-tomography
- CRLs for expanding/focusing beam

- EH1: X-rays ~30-50% for Paris-Edinburgh-Press / new ultra-high P LVP / user-press (e.g. RoToPEc).
- EH2: X-rays ~50-70% for Aster 6-ram LVP (plus 50-30% offline operation)

Summary:

Beamline collaboration partners

- UHP geo-research (BGI, Bayreuth)
- Ternary hydrides (Stockholm/Leipzig Uni)
- Water-related HP research in CMWS (GFZ)
- Targeting in-house research goals for project oriented funding (Helmholtz)

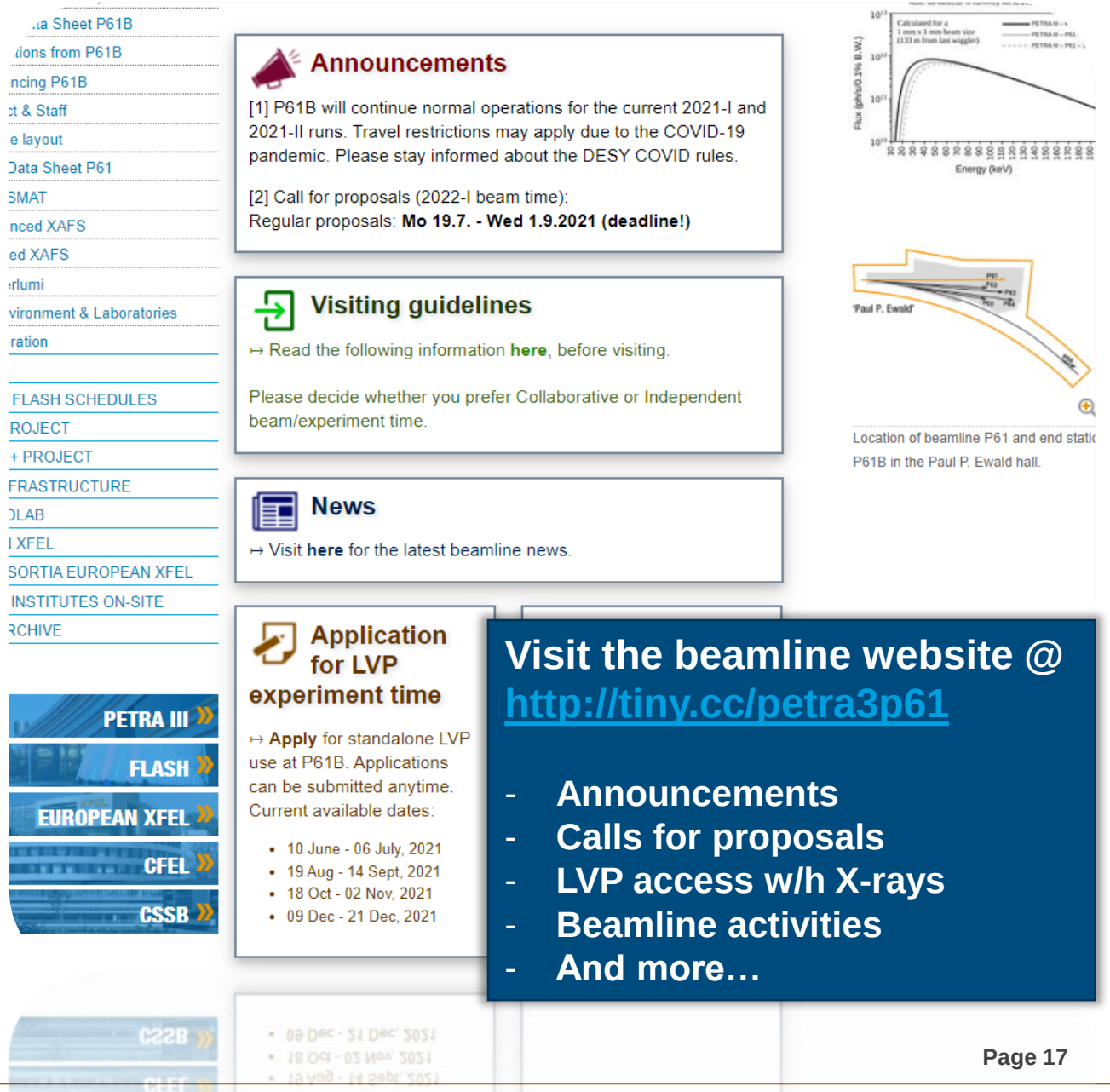
User operation started at P61B

- LVP upgraded for wide range of *in situ* and *ex situ* experiments for wide P and T range.
- Ge-detectors provide excellent XRD data quality, high count rate (200+ kcps), low acquisition time.
- Development of user-friendly GUIs.

Upcoming and newly available

- CAESAR operation using Ge-detector positioning system (2022-II).
- New *in situ* experiments! Rock deformation, Acoustic Emissions (AE), and ultrasonic interferometry (wave speed measurements)

Thank you for your attention!



Contact

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Elektronen-Synchrotron

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