Status & development of P61B LVP at PETRA III, DESY

Beamline satellite workshop

Robert Farla
25-01-2022

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

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

- Complementary *in situ* techniques:
  - Ultrasonic interferometry
  - Acoustic Emissions testing
  - Electrical conductivity (pending upon request)

For 50% experiment time:

- Synthesis of novel (recoverable) materials
- Other

P61B LVP Mission

First publications using X-rays

Article

Nature | Vol 601 | 6 January 2022 | 69

Depressed 660-km discontinuity caused by akimotoite–bridgmanite transition

https://doi.org/10.1038/s41586-021-04157-z
Received: 26 November 2020
Accepted: 18 October 2021
Published online: 5 January 2022
Open access

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Robert Farla[1], Keisuke Nishida[2], Zhaodong Liu[2,4], Lin Wang[2,5], Ayano
Hu Tang[3], Zhen Chen[3], Yuji Higo[7], Yoshinori Tange[7] & Tomoo Katsura

The 660-kilometre seismic discontinuity is the boundary betw
mantle and transition zone and is commonly interpreted as bei

Review of Scientific Instruments | ARTICLE | scitation.org/journal/rsi

Simultaneous generation of ultrahigh pressure and temperature to 50 GPa and 3300 K in multi-anvil apparatus

Cite as: Rev. Sci. Instrum. 92, 103902 (2021); doi: 10.1063/5.0059279
Submitted: 8 June 2021 • Accepted: 10 September 2021 •
Published Online: 5 October 2021

View Online | Export Citation | CrossMark

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Contributions to Mineralogy and Petrology (2021) 176:77
https://doi.org/10.1007/s00410-021-01829-x

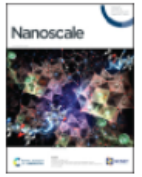
ORIGINAL PAPER

Determination of phase relations of the olivine–ahrensite transition in the Mg_2SiO_4–Fe_2SiO_4 system at 1740 K using modern multi-anvil techniques

Artem Chanyshev[1,2] · Dmitry Bondar[2] · Hongzhan Fei[2] · Narangoo Purevjav[2] · Takayuki Ishii[2,3] · Keisuke Nishida[2] · Shrikant Bhat[1] · Robert Farla[1] · Tomoo Katsura[2,3]

Received: 31 March 2021 / Accepted: 11 August 2021

An electrically conductive and ferromagnetic nano-structure manganese mono-boride with high Vickers hardness†

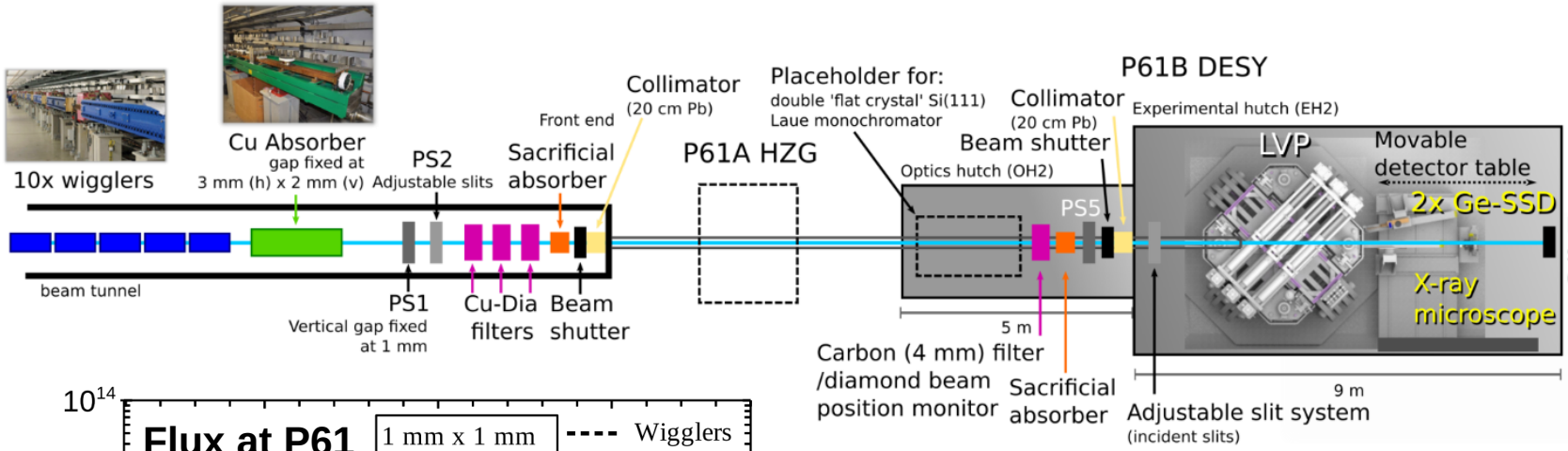


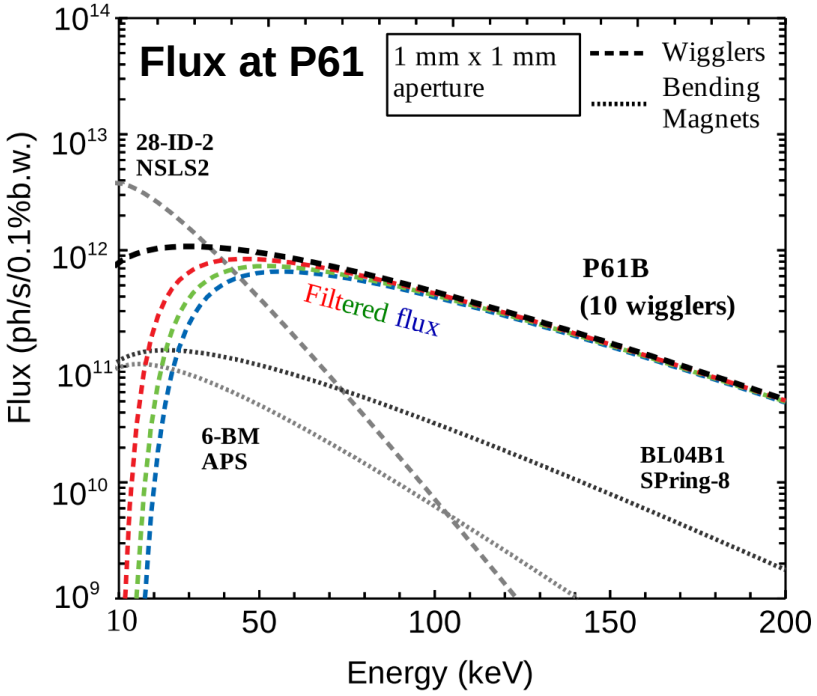
From the journal: Nanoscale

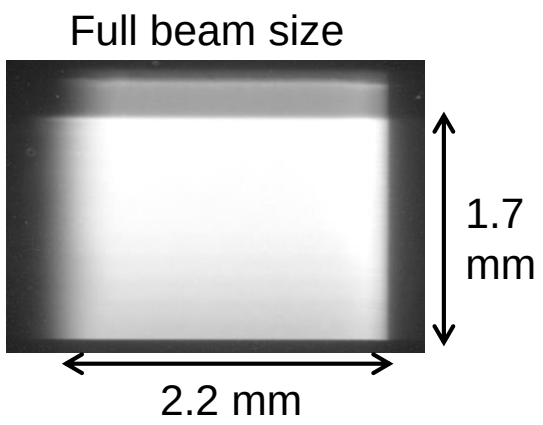
Shuailing Ma,[ab] Robert Farla,[b] Kuo Bao,*[a] Akhil Tayal,[b] Yongsheng Zhao,[ab] Qiang Tao,[a] Xigui Yang,[c] Teng Ma,[a] Pinwen Zhu[a] and Tian Cui*[ad]

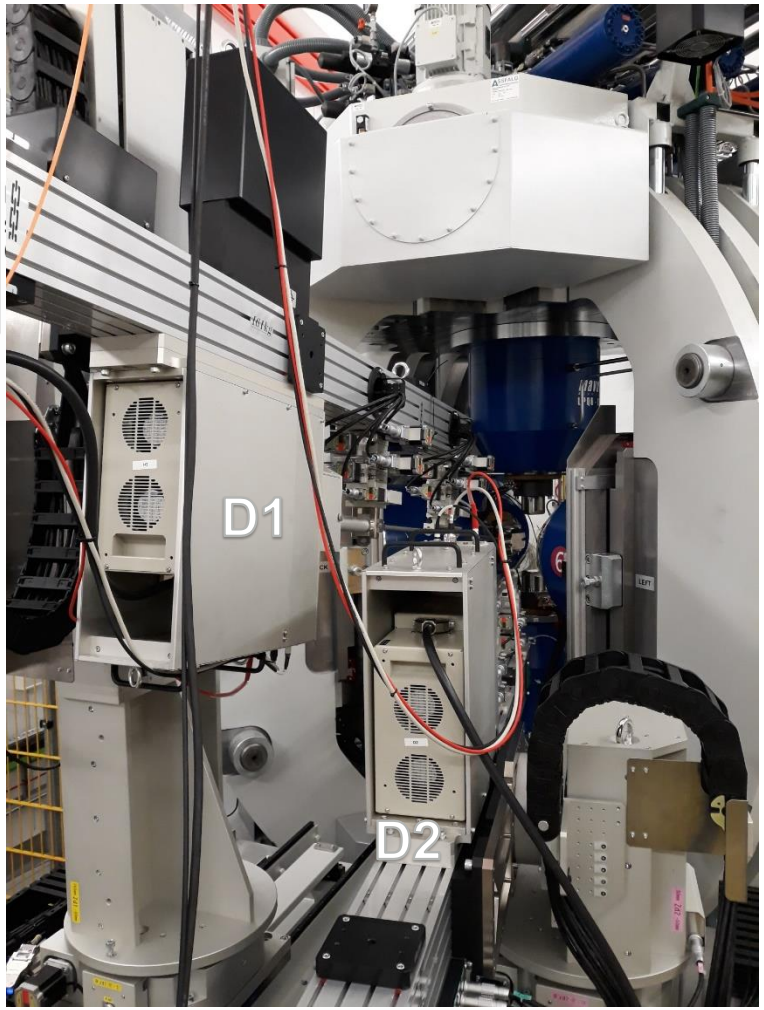
Beamline layout

The Large Volume Press (LVP) extreme conditions beamline (*50% X-rays, 50% stand alone*)





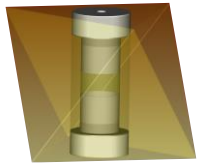




Detector positioning system

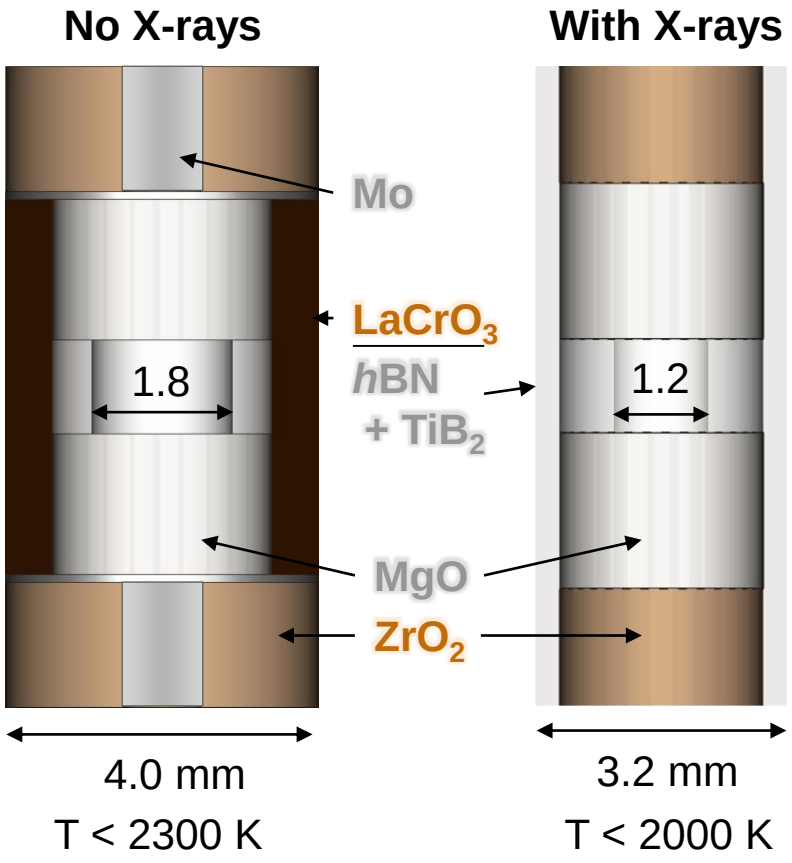
High-pressure techniques

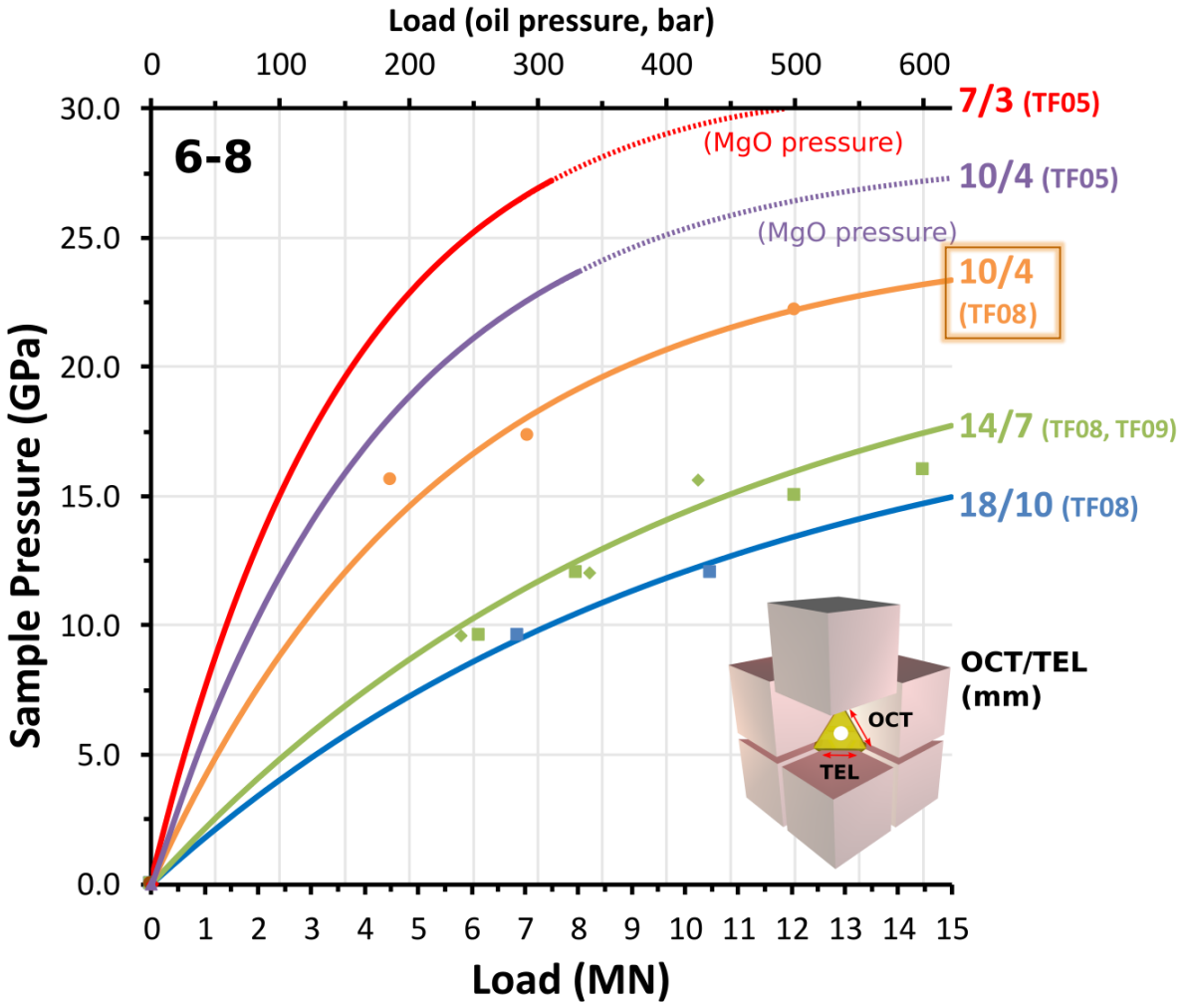
Standard assemblies for *in situ* hydrostatic high pressure experiments



‘Kawai’ 6-8 mode

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

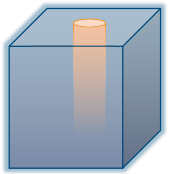




Original assembly design by Dr. Nishiyama

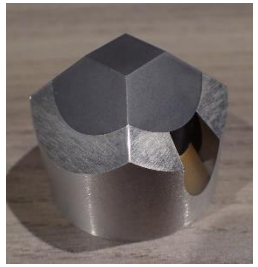
High-pressure techniques

Standard assemblies for *in situ* studies of rock deformation



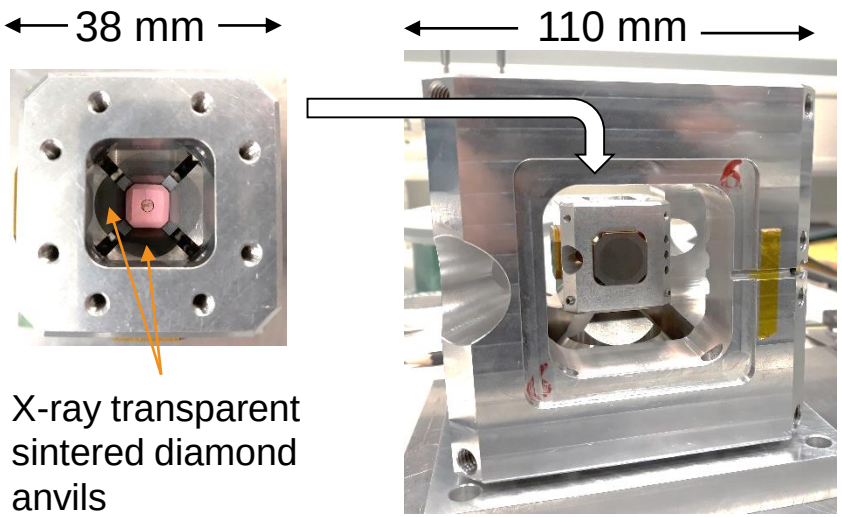
‘Cubic’ 6-6 mode (p = 0.5 - 4 GPa)

New! Large cBN anvil (X-ray transparent)

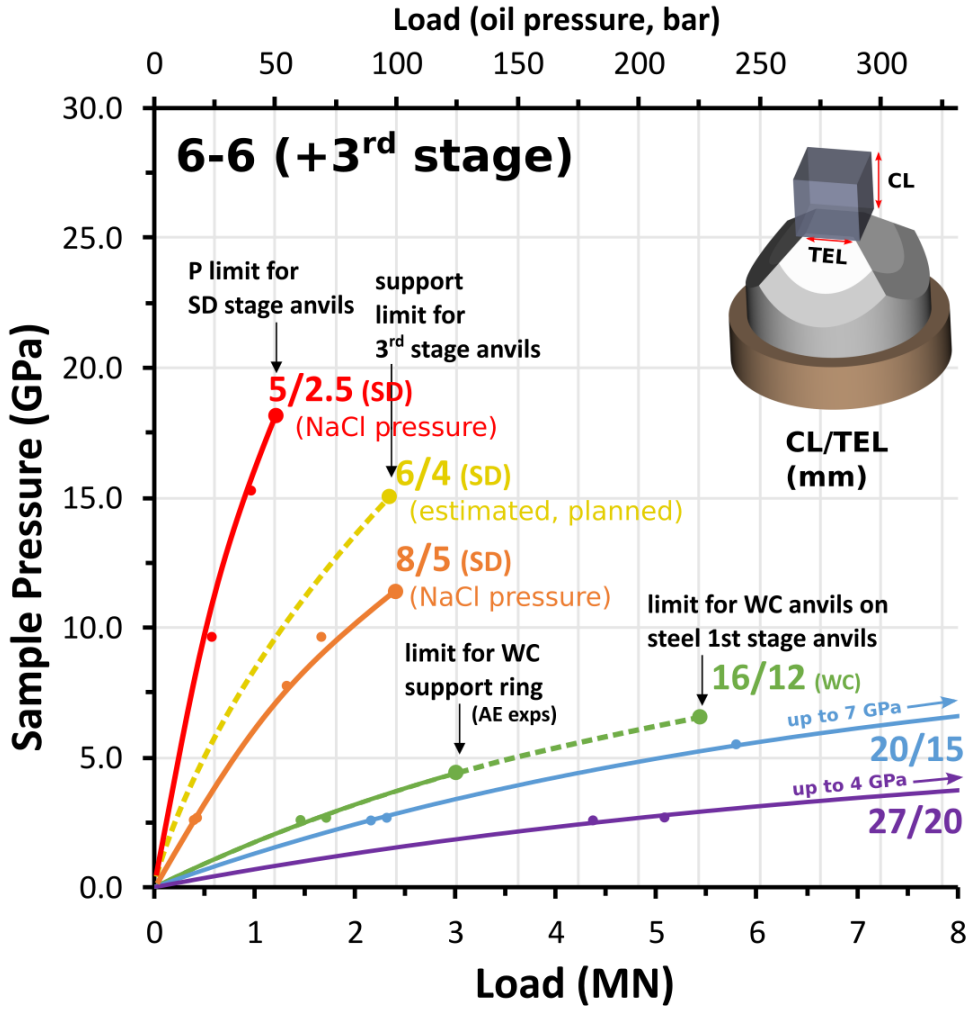


- Compatible with WC anvils
  - **12 mm TEL → 0.5 - 4 GPa**
  - 9 mm TEL evaluated
- Compatible with Acoustic Emissions detection.

‘Cubic’ triple-6 mode (p > 5 GPa)



X-ray transparent sintered diamond anvils



Anvils are provided!

Assemblies designed with your input!

Generally for *ex-situ* synthesis

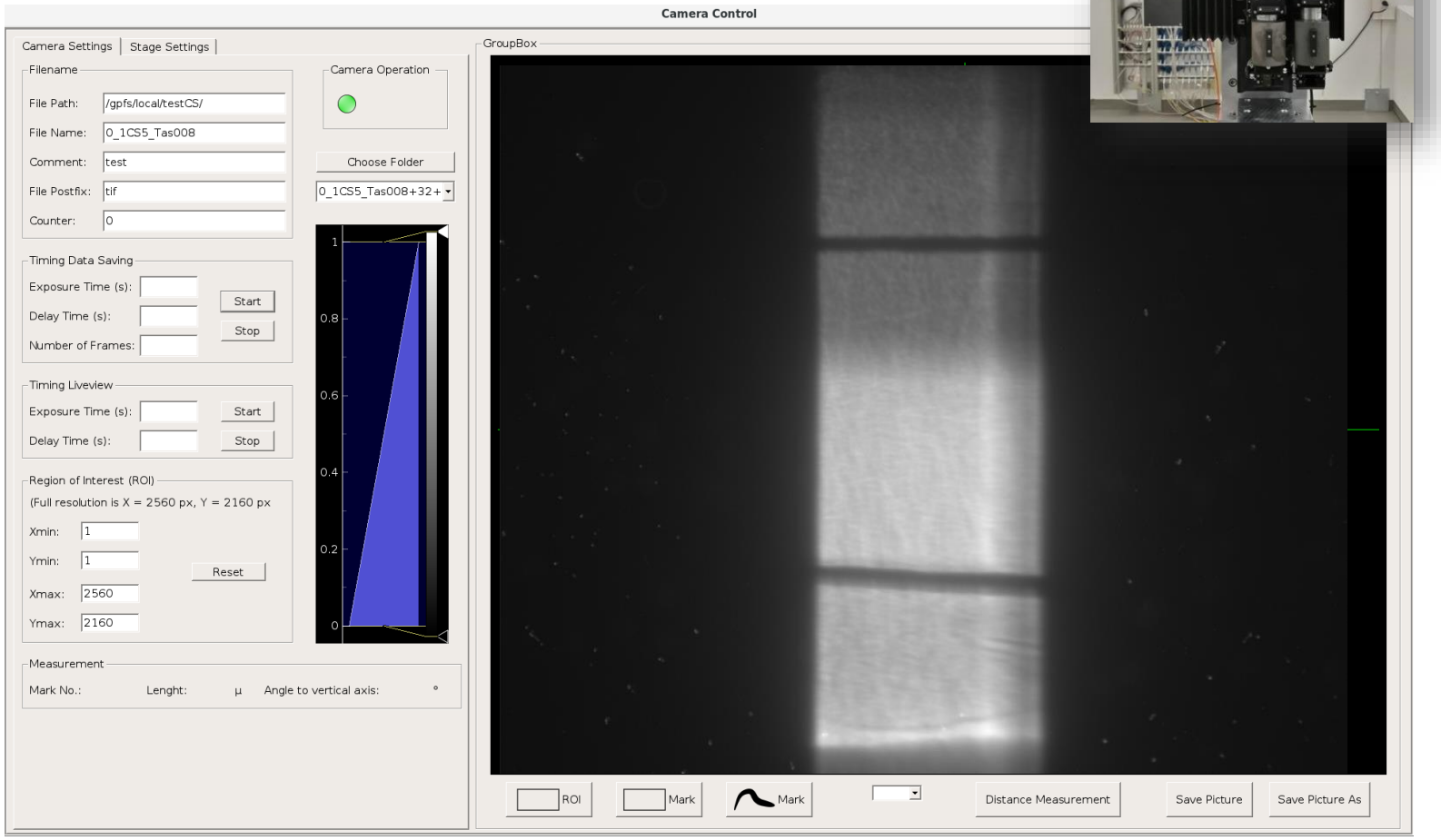
The whitebeam X-ray microscope

X-ray radiography

- **Double objectives (5x, 10x)** for high-resolution, full beam imaging

- **Scintillators:**
  - (*in use*) **GGG:Eu** – 20, 40 µm
  - **LuAG:Ce** – 20, 40 µm
  - **GaGG:Ce-HL** – 150, 200 µm
    New! Ultra-bright e.g. for viscosimetry

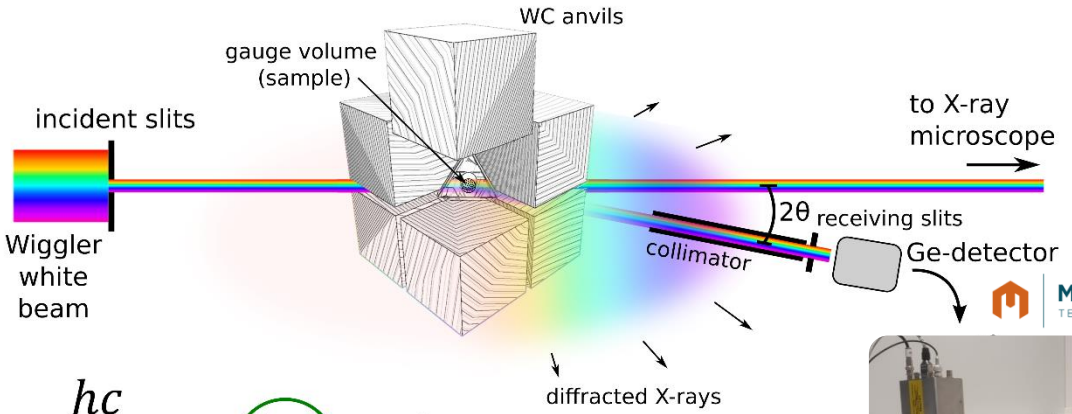
- **PCO.edge 5.5 MP sCMOS camera**
  - True global & rolling shutter
  - 100 fps @ full-resolution (up to 1000 fps for ROI)
  - Live view & frame capture
  - LVP Z-stage imaging scan



X-ray powder diffraction using white beam

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

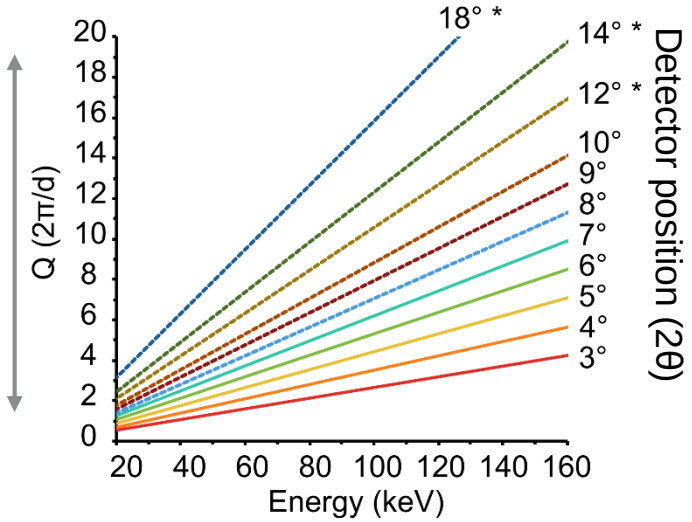
1. High spatial resolution (define gauge volume)
   → avoid high temperature & pressure gradients
   → multiple samples in one experiment
2. Ideal for low-Z (X-ray transparent) samples.
3. Fast acquisition (10-100 s) covering large Q-range.

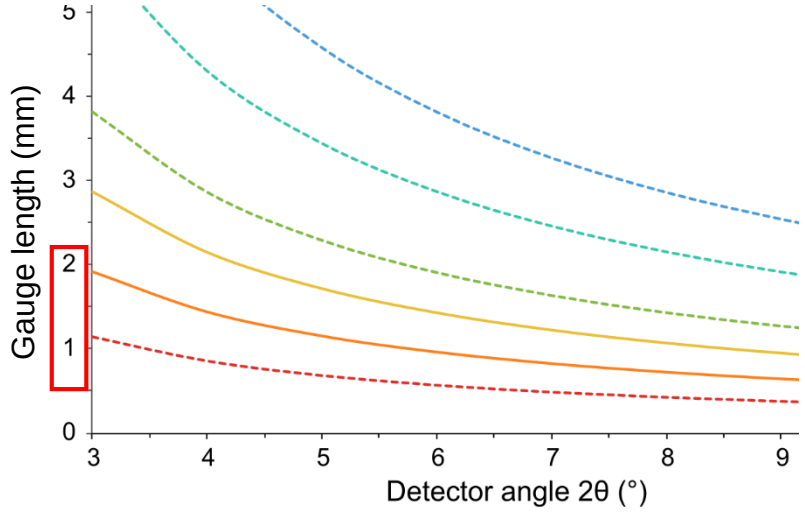


$$n\frac{hc}{E_{hkl}} = 2d_{hkl}\sin\theta \quad (\lambda = hc/E)$$

measurable → observable

2x Ge-SSD, electronic cryostat, CMOS pre-amp, 4k digital analyzer (millions of cps)





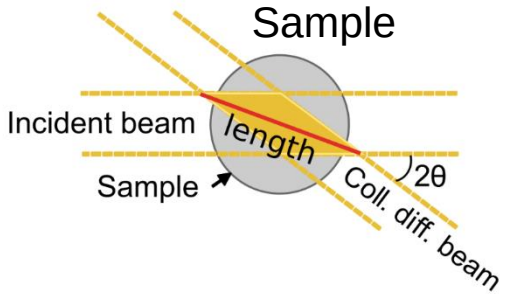
Collimator slit size

200 µm

100 µm

50 µm

30 µm

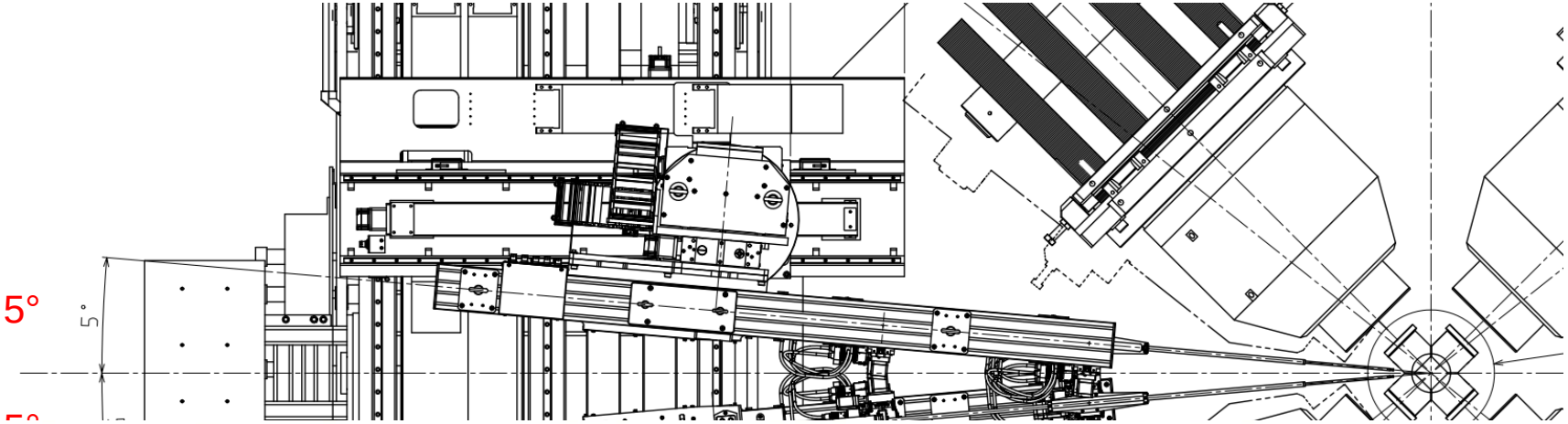


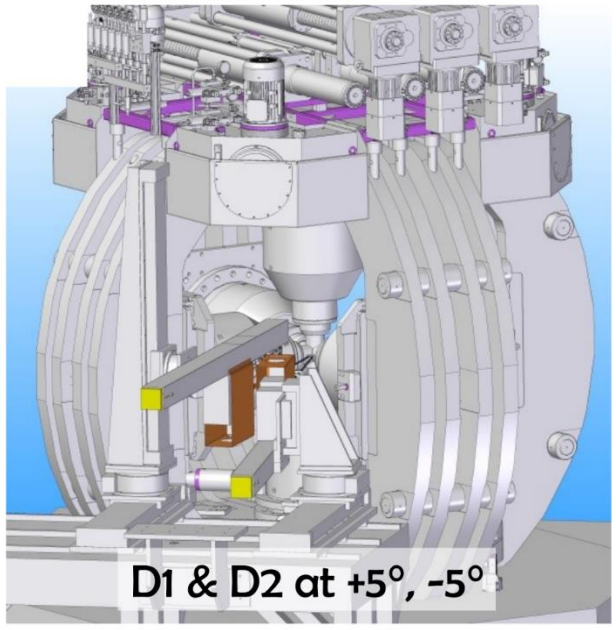
X-ray powder diffraction using white beam

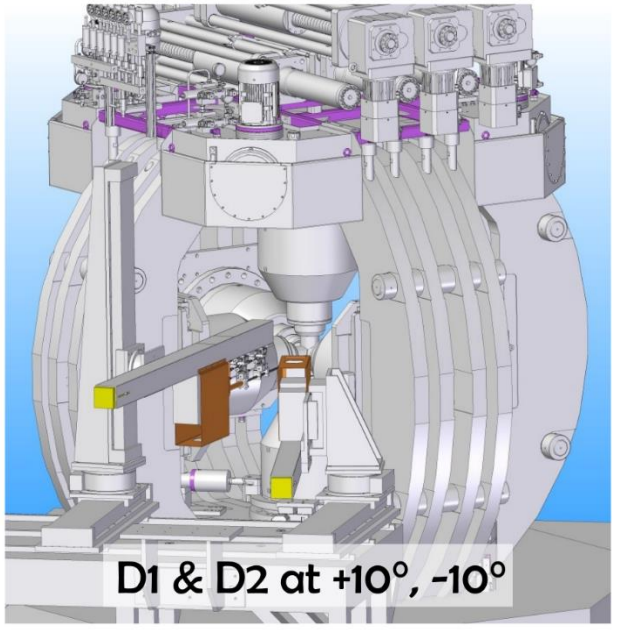
Energy-dispersive X-ray diffraction (ED-XRD) in the Large Volume Press

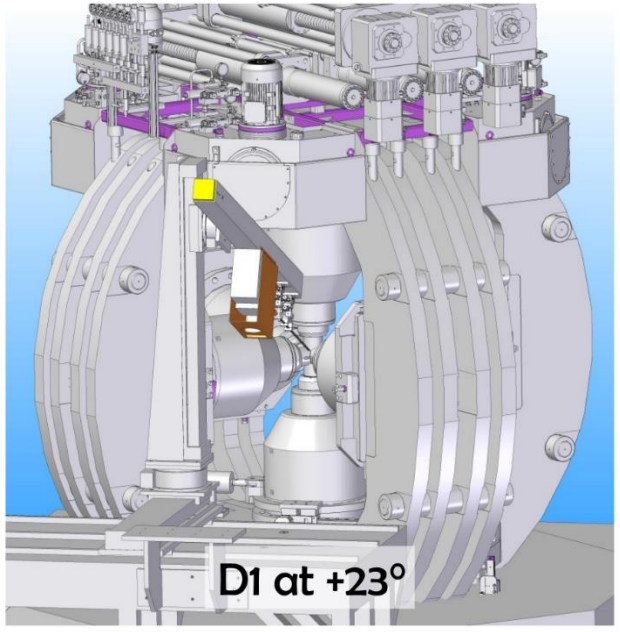
MIRION TECHNOLOGIES

Measurement positions
→ A world of possibilities!



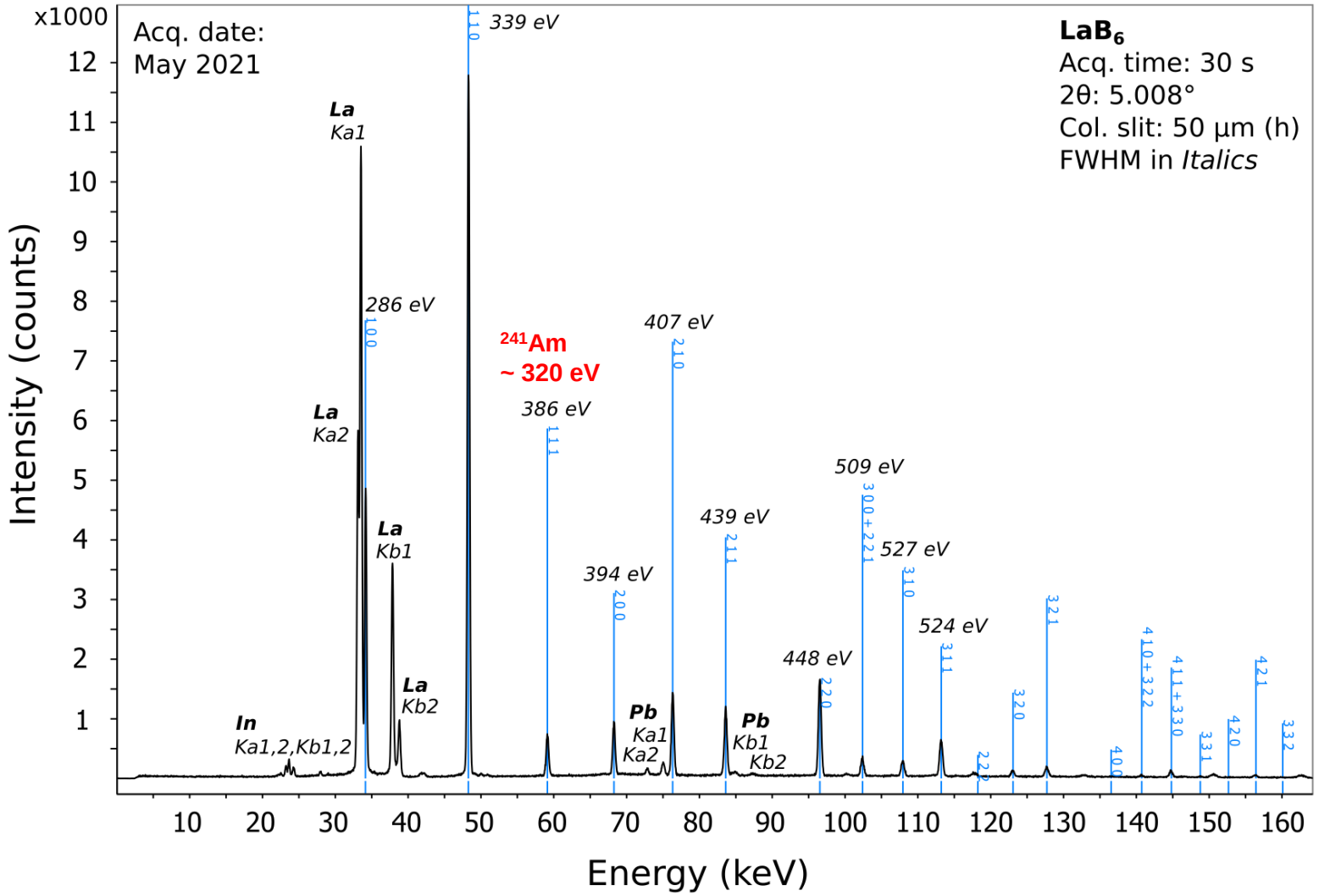






X-ray powder diffraction using white beam

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



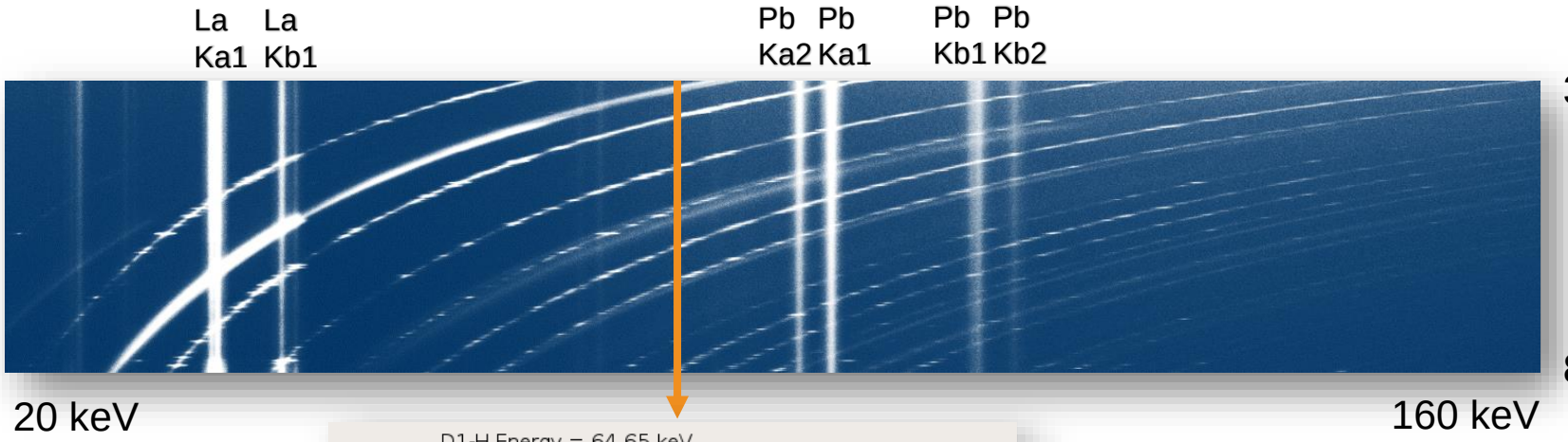
ED-XRD resolution test on NIST powder(s) e.g. LaB_6 in a 0.8 mm diameter capillary tube.

- Good resolution!
- Low dead time / high count rates (up to 400 cps).

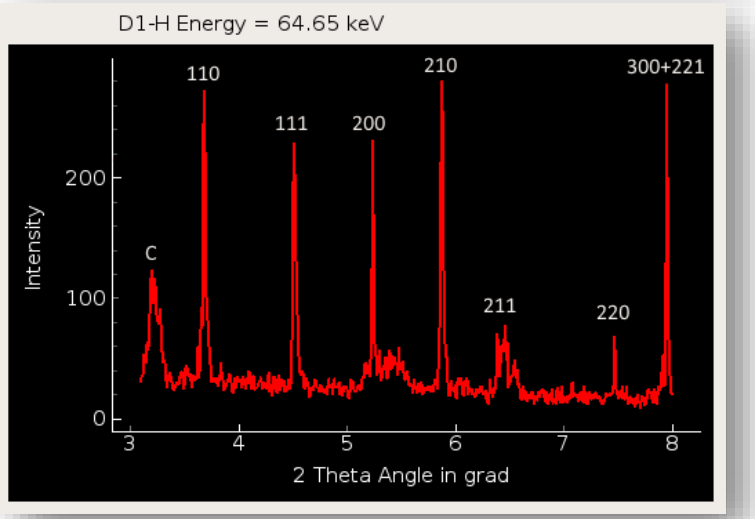
X-ray powder diffraction using white beam

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

First preliminary result



Detector scan
0.01° increments
20 s per scan
→ 2.7 hours.



Resulting AD-XRD pattern of LaB_6 sample

- Spotty pattern to be improved by simultaneous press rotation.
- Detector stage movements require unique calibration to keep gauge volume in sample.

X-ray powder diffraction using white beam

Beamline software tools (available from the website)

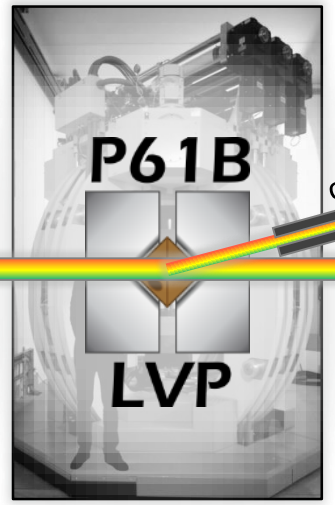
1. **Data file conversion** HDF5 (nxs) → csv and GSAS1 formats.

2. **Detector energy calibration** using radionuclides → obtain linear or quadratic relation between channel # and energy.

3. **2θ calculator** for pressure standards (MgO, NaCl) as well as other materials (e.g. Ni and Al_2O_3).

4. **Simultaneous P and T estimation** using Equation of State of pairs of materials in cell assembly.

P61B LVP Research



Unfocused white X-rays for ED-XRD

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

slits

HP Ge-detector

collimator

Development of BCN X-ray transparent 'windows'

Monochromator development continues…

Unfocused monochromatic X-rays for AD-XRD

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

beamstop

large radius 2d detector

1. Earth Materials
HP Silicates
A. Chanyshev (BGI/DESY)
T. Katsura (BGI),
et al.

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

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

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

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

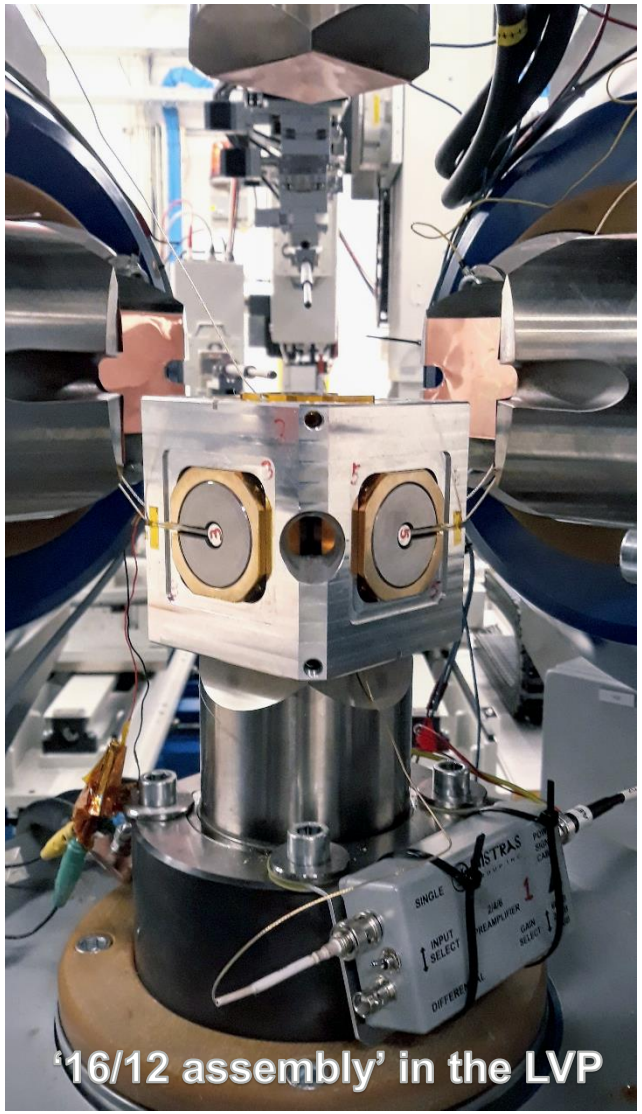
3. Earth Materials
Acoustic Emissions
S. Ma (Jilin Uni, China/DESY)
J. Gasc (Uni Montpellier)
S. Incel (Bochum)

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

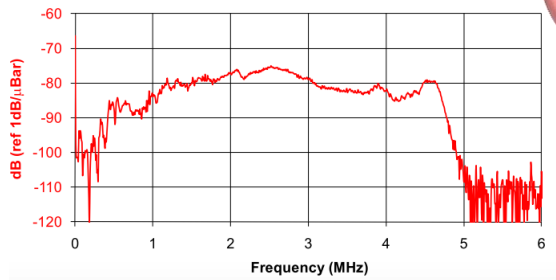
Acoustic Emissions testing

Methodology (MA6-6 compression)

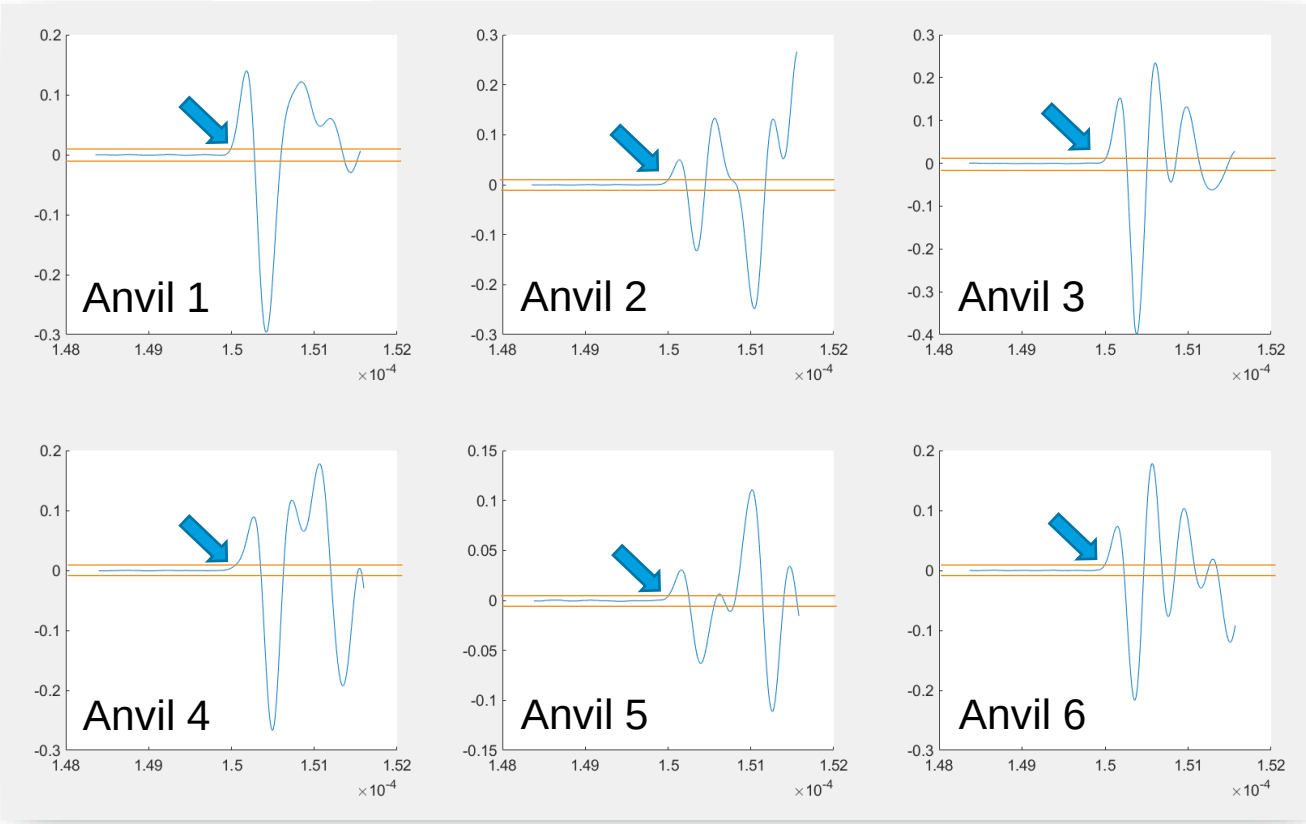
Acoustic Emissions
S. Ma (Jilin Uni, China/DESY)
J. Gasc (Uni Montpellier)
S. Incel (Bochum)



Micro200HF Sensor
Very Wideband Frequency Miniature Sensor



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

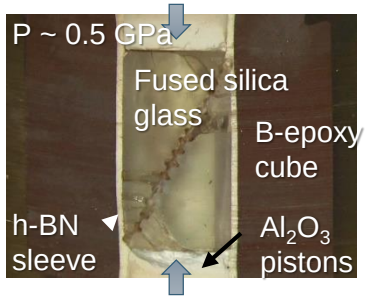


Aim: to investigate focal mechanisms & radiated energy of crack propagation.

Acoustic Emissions testing

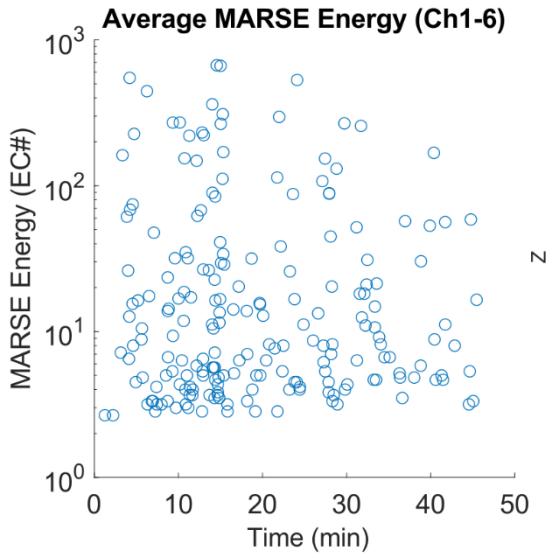
New, fast data processing techniques using MATLAB, incl. improved 3D location

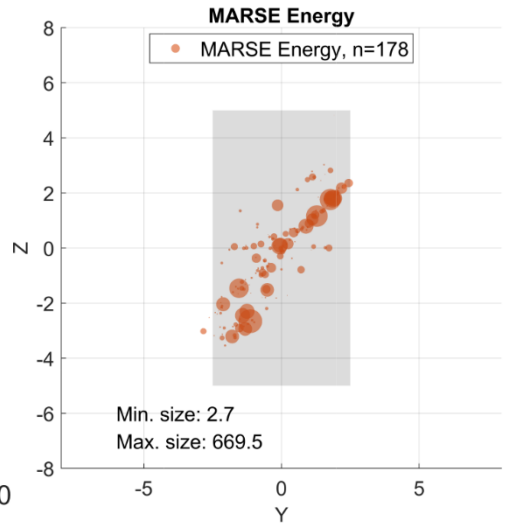
Acoustic Emissions
S. Ma (Jilin Uni, China/DESY)
J. Gasc (Uni Montpellier)
S. Incel (Bochum)

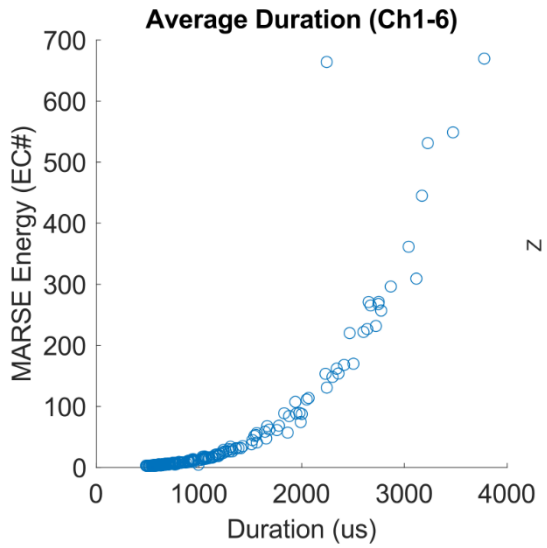


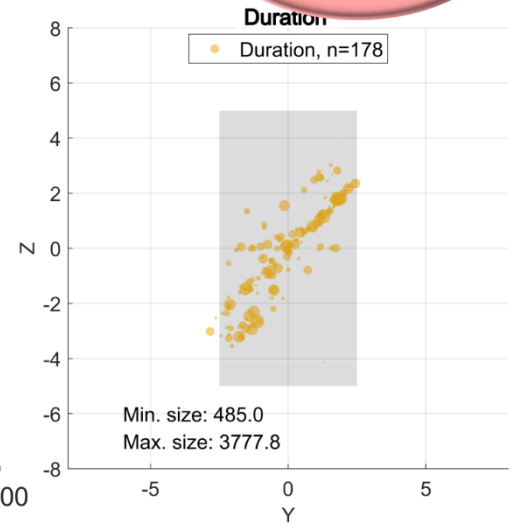
Adaptable scripts for processing AE data available.

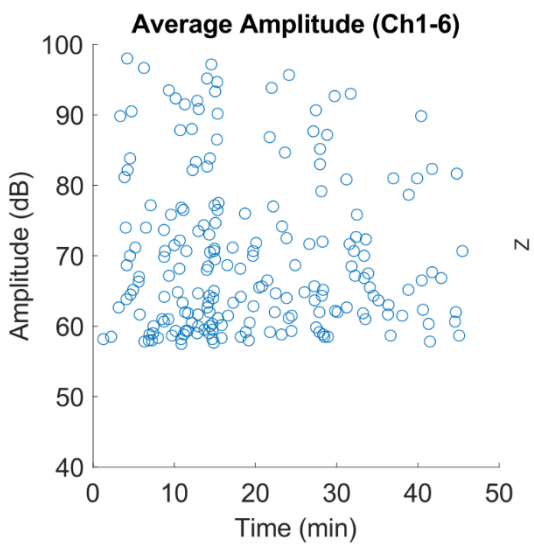
Requires only **extraction of triggered (raw) waveforms** of events and **AE characteristics** calculated by AEwin software.

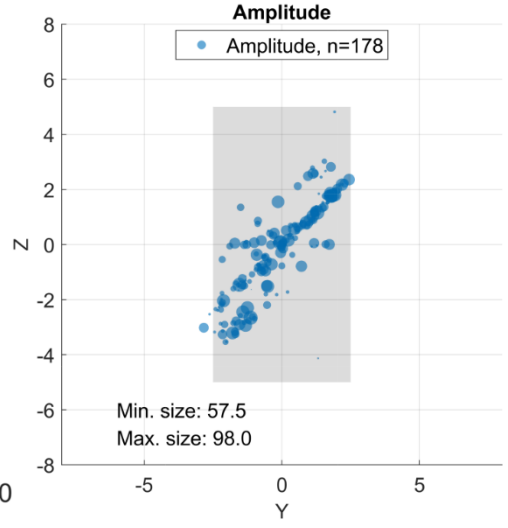


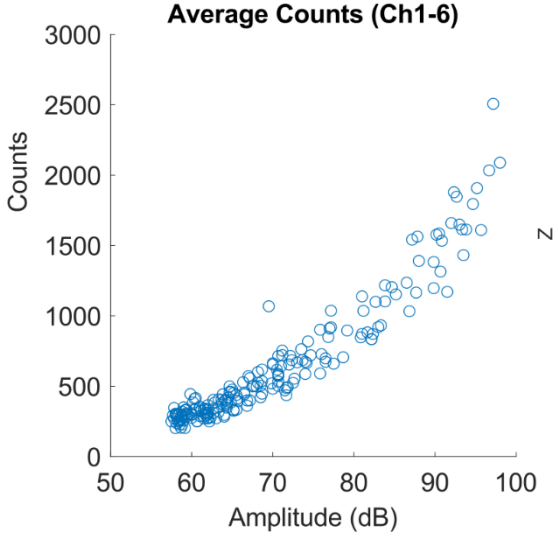


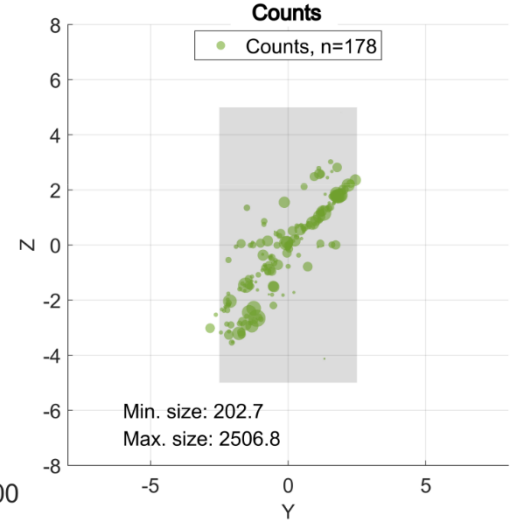








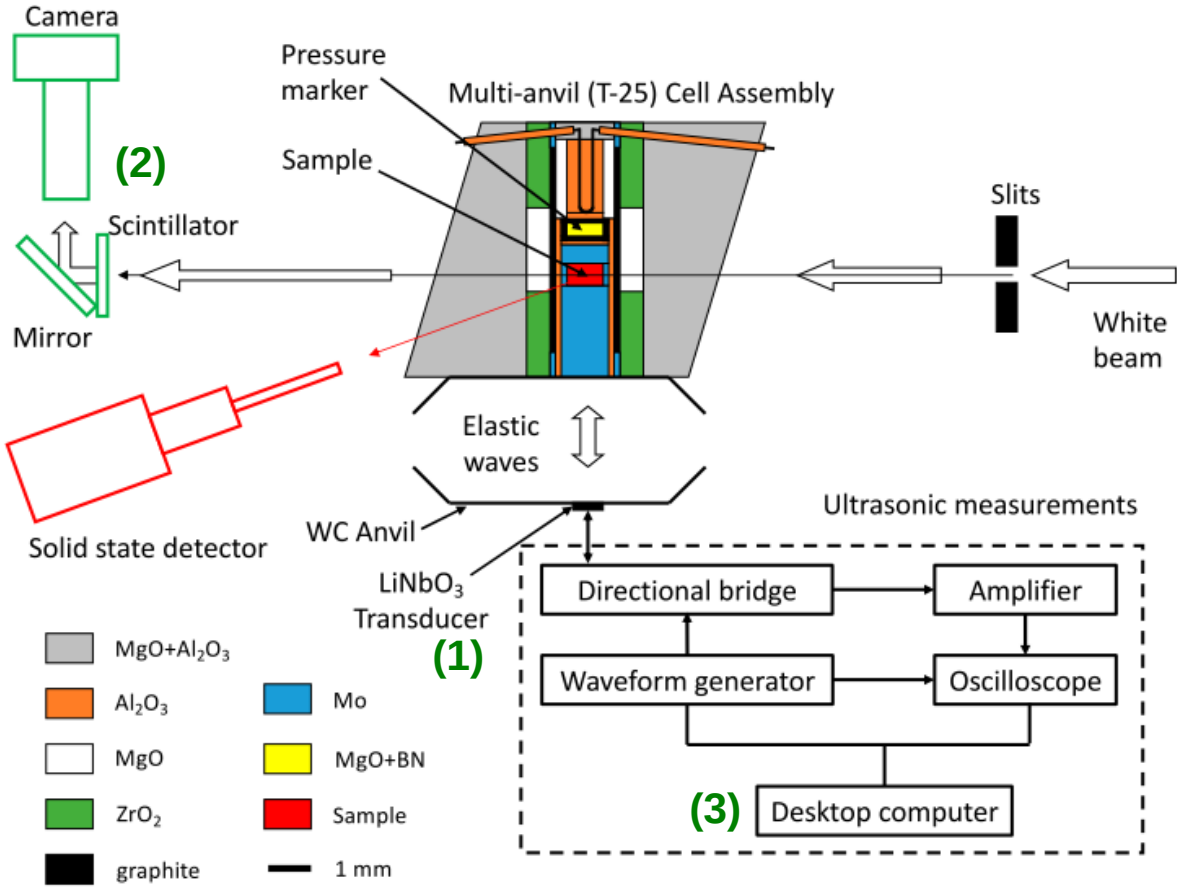




Wave speed measurements

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

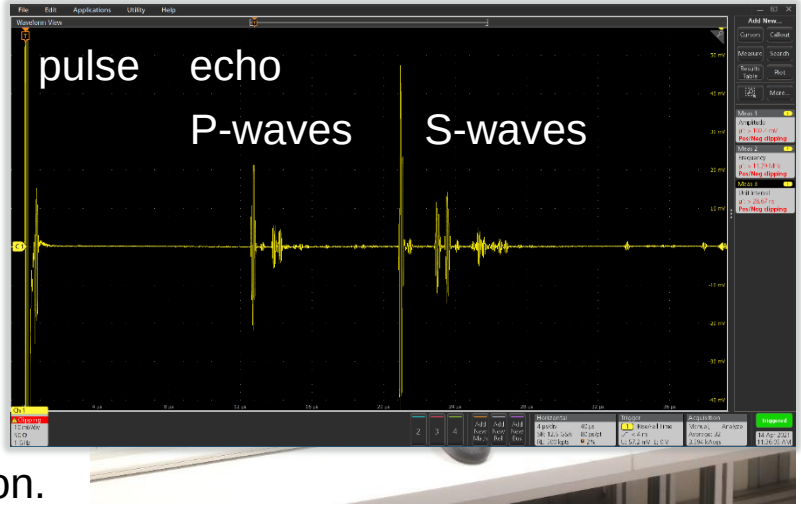
General method

(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.

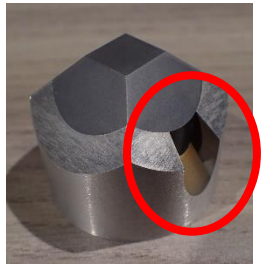
Rock deformation (*in situ*)

Anvil-assembly design for *in situ* studies of rock deformation

Rock Mechanics
R. Farla (DESY)

‘Cubic’ 6-6 mode (p = 0.5 - 4 GPa)

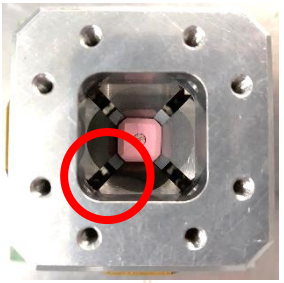
New! Large cBN anvil (X-ray transparent)



- Compatible with W carbide anvils
-

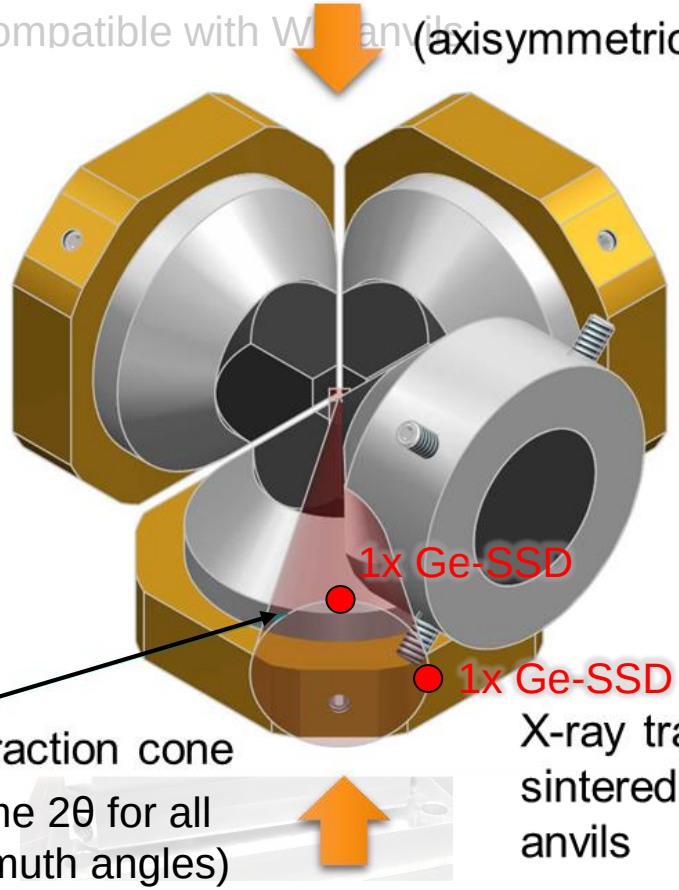
‘Cubic’ triple-6

⟵ 38 mm ⟶

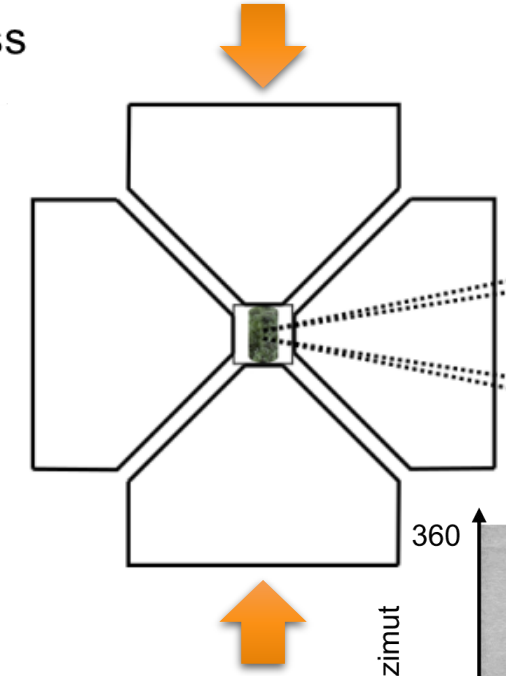


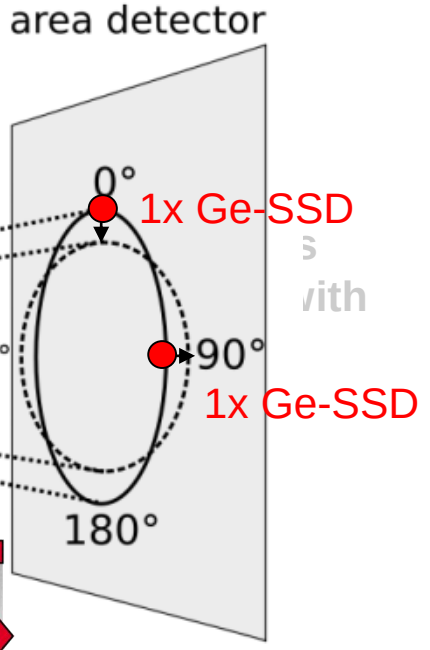
X-ray transparent sintered diamond anvils

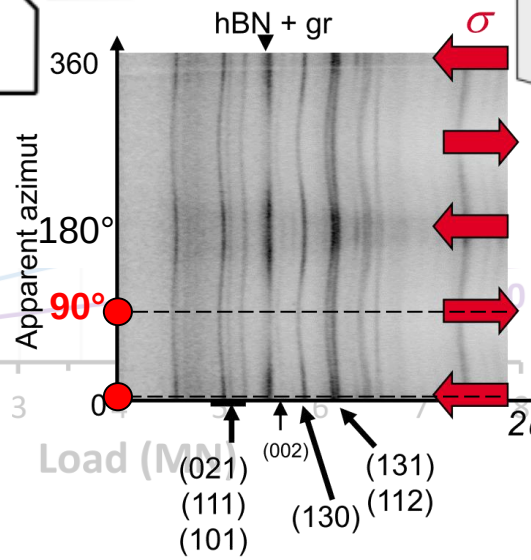
Only for axisymmetric compression



X-ray transparent, sintered diamond anvils







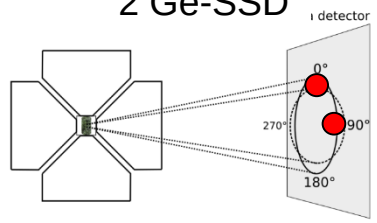
lattice micro-strain = stress

for ex-situ synthesis

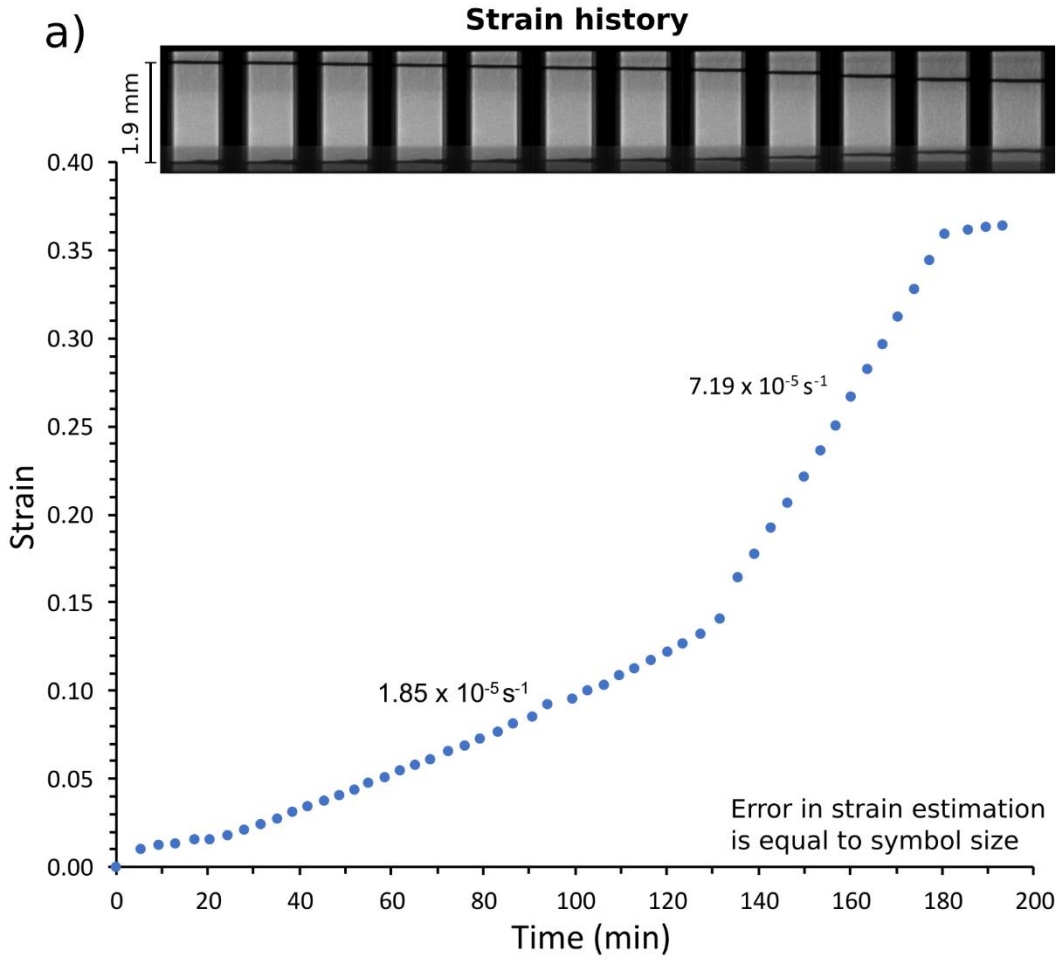
Rock deformation (*in situ*)

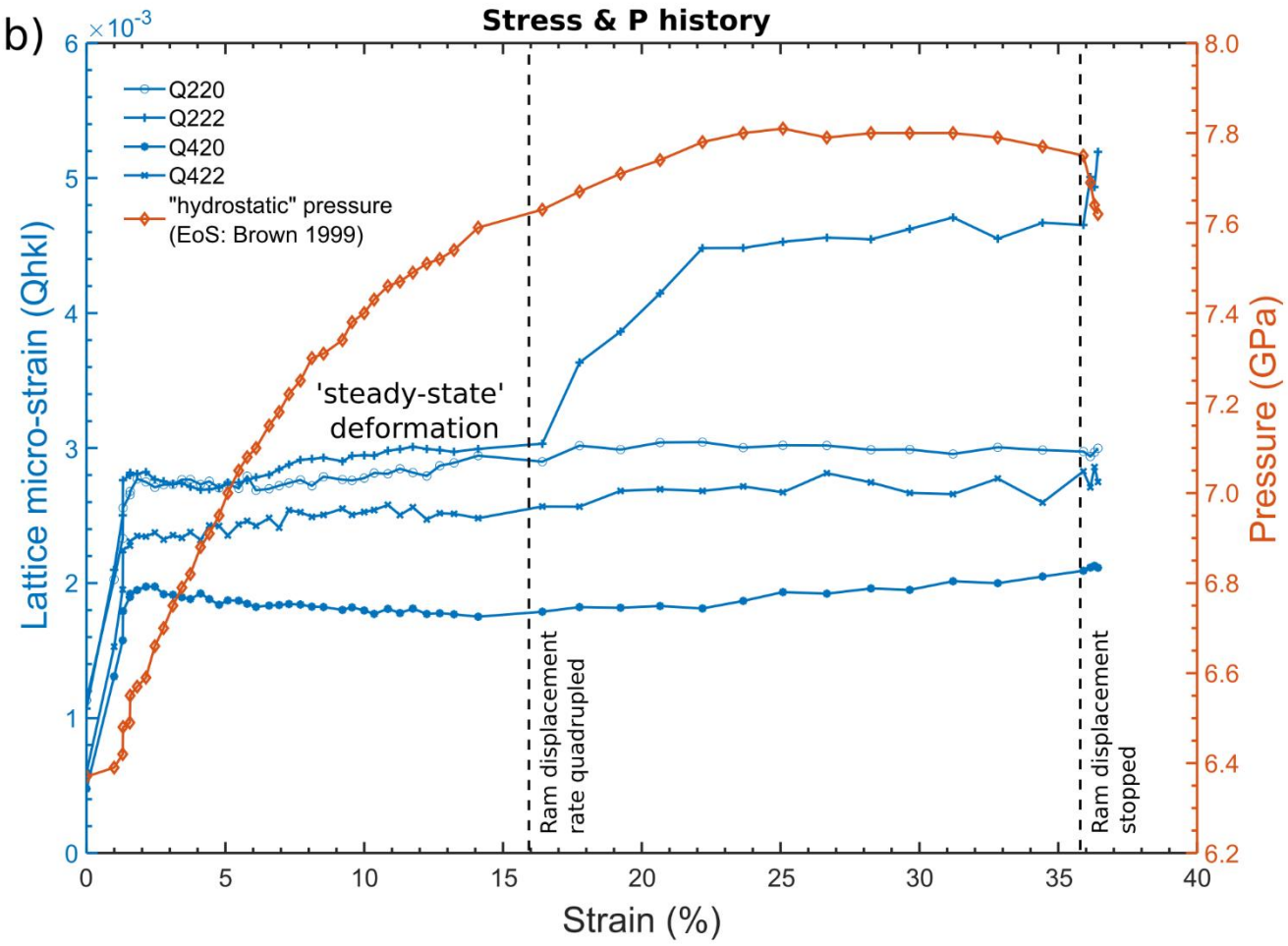
Concept experiment: deformation of NaCl

- 1.9 mm sample deformed at room T and high P (8/5 assembly). Diffraction acq. using 2 Ge-SSD (at 0°, 90° azimuth, 2θ = 7.5°)



Rock Mechanics
R. Farla (DESY)





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)

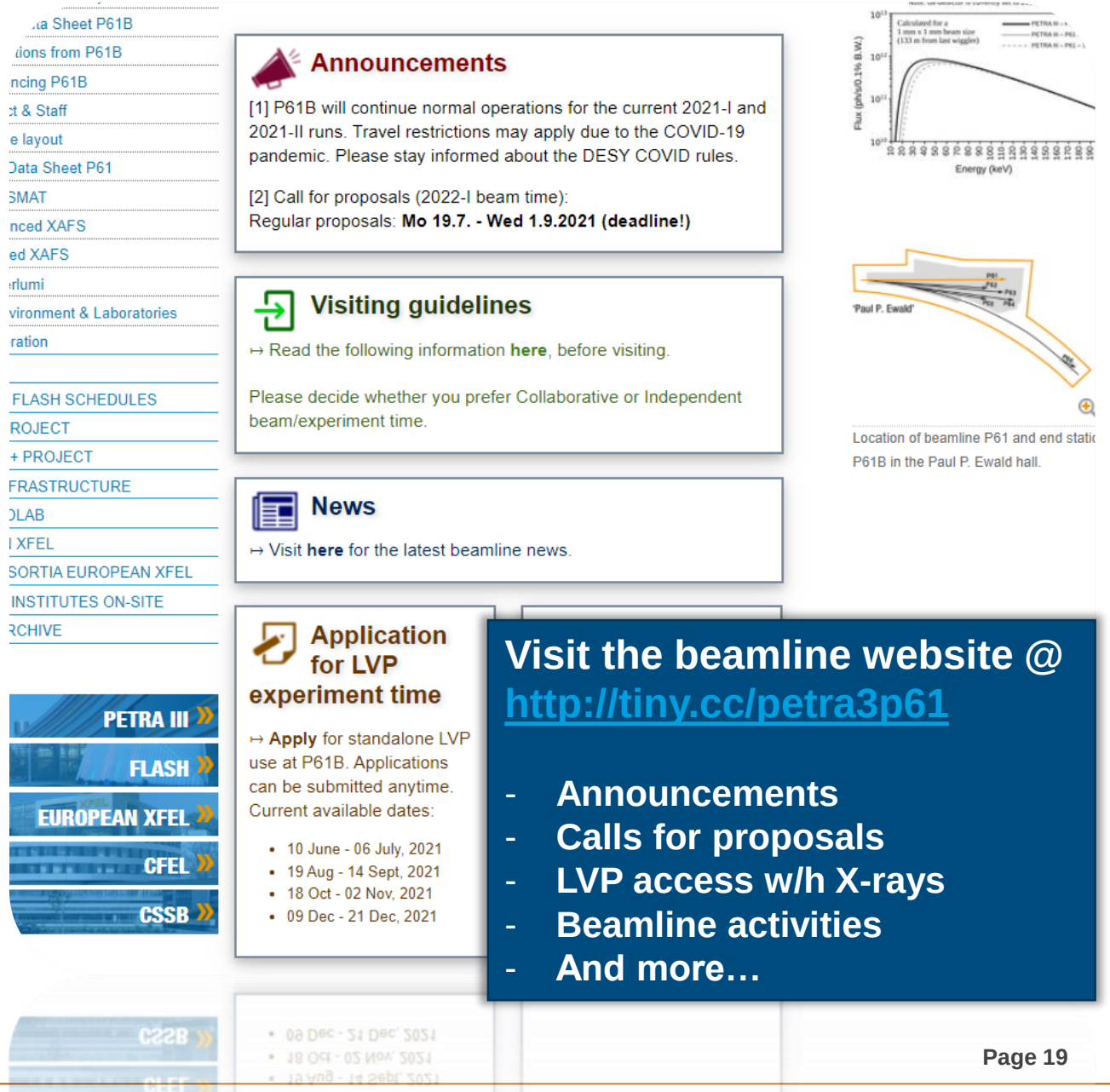
User operation started at P61B

- 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.

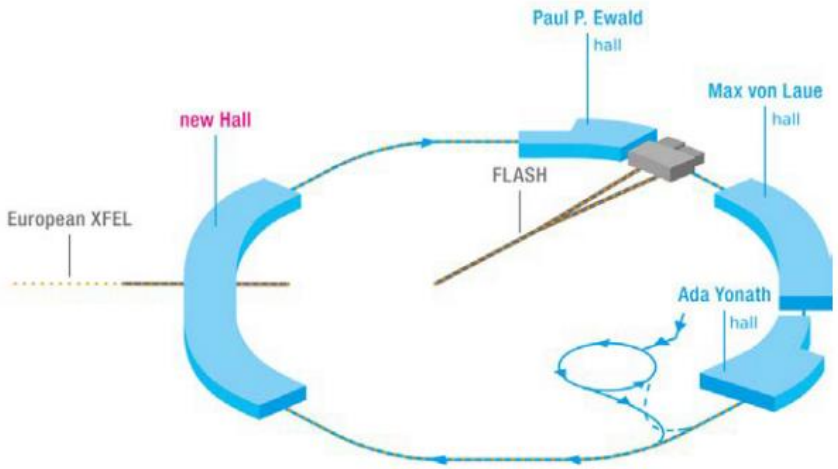
Support for new *in situ* experiments

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 cubes)
4. Falling sphere viscosimetry (w/ GaGG:Ce scint.)

Thank you for your attention!



PETRA IV. NEW DIMENSIONS — Timeline (updated)



Targeted submission of roadmap proposal

01'2023: Kickoff PETRA IV project

01'2025: Start construction work
End PETRA III operation

01'2027: Start up PETRA IV

LEAPS, HGF-FIS

PETRA IV project phase

PETRA IV operation

PETRA IV TDR Phase

mid-2023 TDR complete

PETRA IV CDR Phase

12'2019: CDR

Today

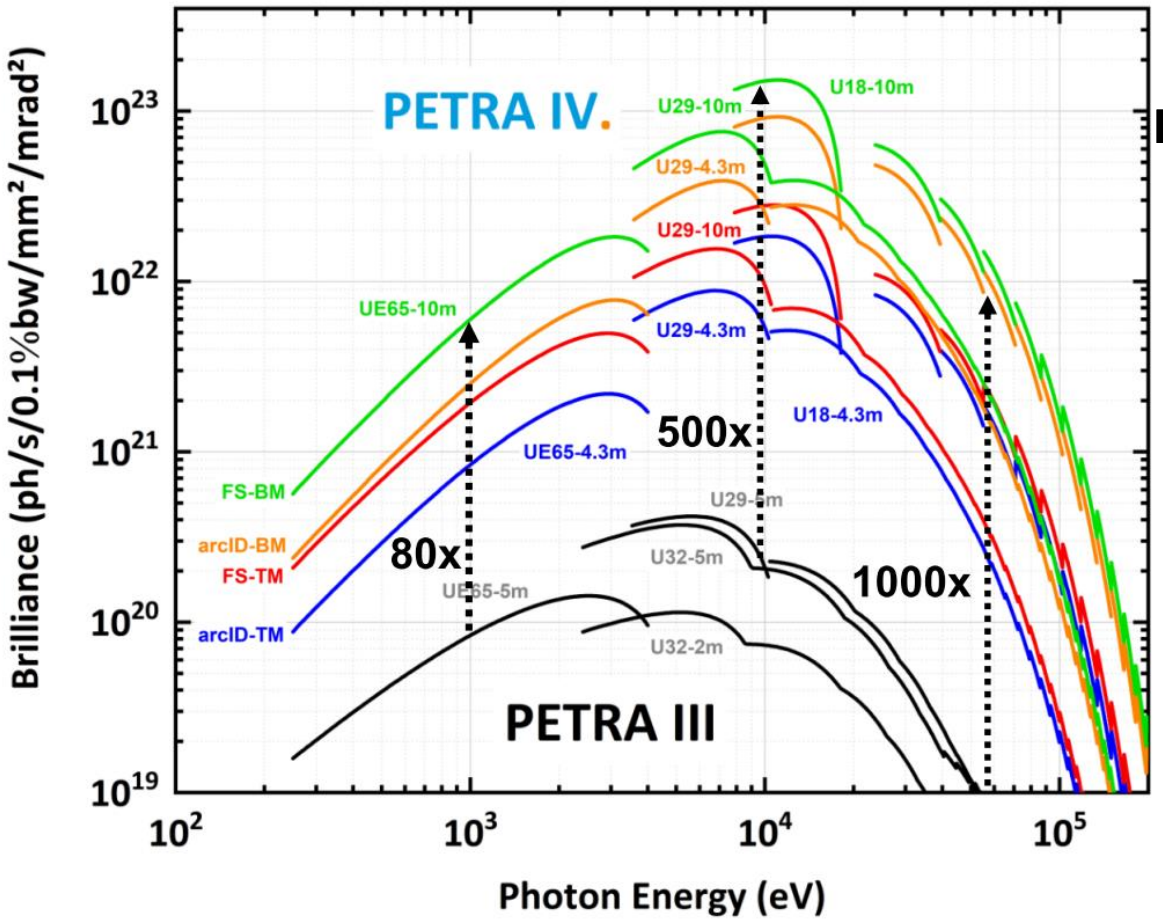
Shutdown

2016 2017 2018 2019 2020 2021 2022 2023 2024 2025 2026 2027 2028

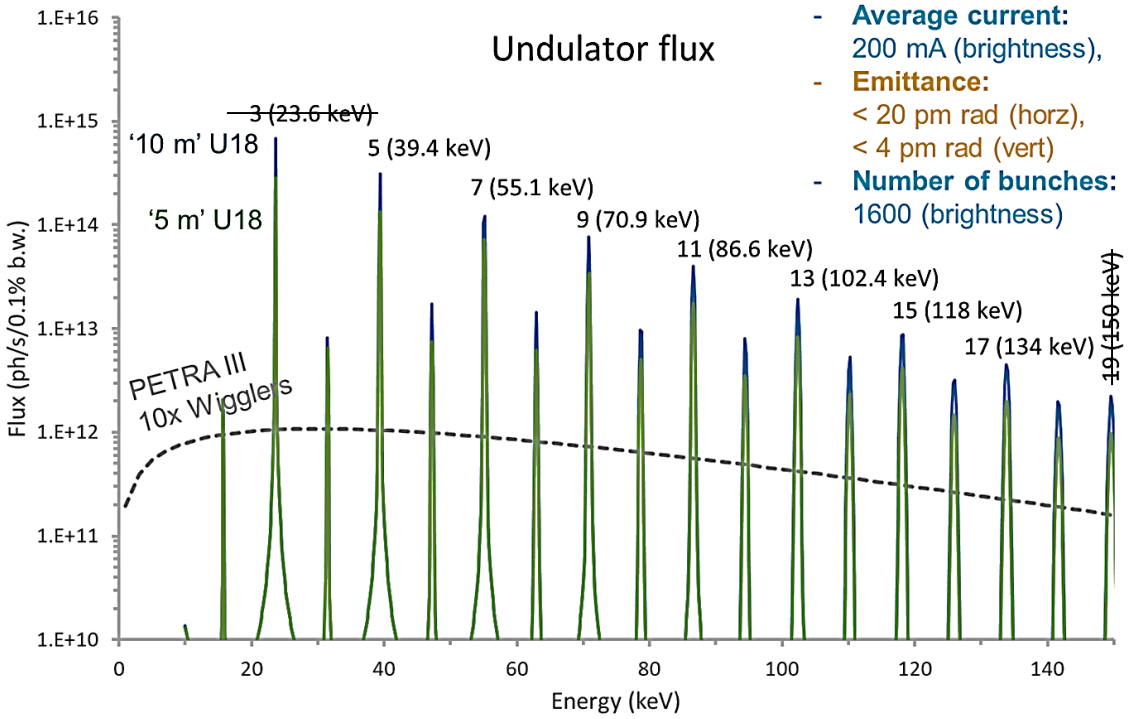
Beamline portfolio complete

PETRA IV. Calculations

Tuning curves for various undulators



- Major increase in brilliance compared to PETRA III
  - high energies: U18 undulator **with Laue mono**.
  - new *in situ* LVP beamline **not flagship**, hence location/undulator length **not yet decided**.
  - Harmonic flux **significantly** higher than P III Wigglers



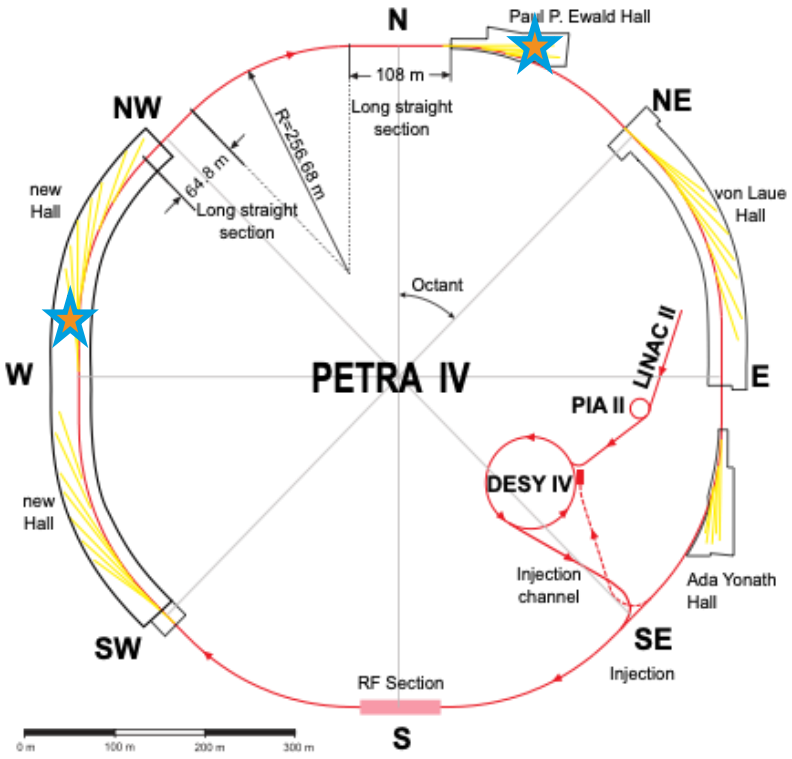
PETRA IV. 5 W's & How

Who? Us!

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!!!

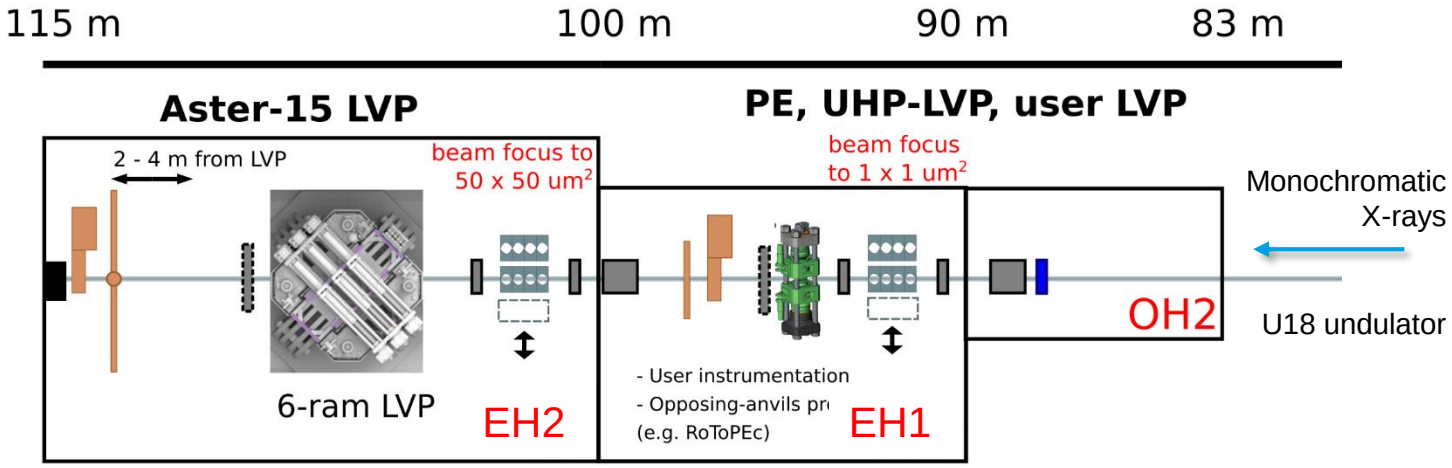
Where? LVP position to be decided:



When? **PETRA III end operation: early 2026**

PETRA IV BL operation: **2028** (existing halls), **2029-30** (West hall).

How? **Possible BL concept:**



- *High energies (40 – 120 keV harmonics) / ultra-high brilliance*
- *New detectors: High-res PXRD / Imaging / µ-tomography*
- *CRLs for expanding/focusing beam*

- EH1: X-rays ~30-50% for smaller (portable) LVPs:
  (1) PE-Press / (2) ultra-high P LVP / (3) other, e.g. RoToPEc.
- EH2: X-rays ~50-70% for Aster 6-ram LVP
  (plus 50-30% offline operation)

Contact

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Elektronen-Synchrotron

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