

# Status & development of P61B LVP at PETRA III, DESY

## Ultra-LVP workshop

Robert Farla  
23-09-2021

### *Acknowledgments:*

Shrikant Bhat, Stefan Sonntag, Artem Chanyshv, 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



**P61B**  
LVP



# P61B LVP Mission

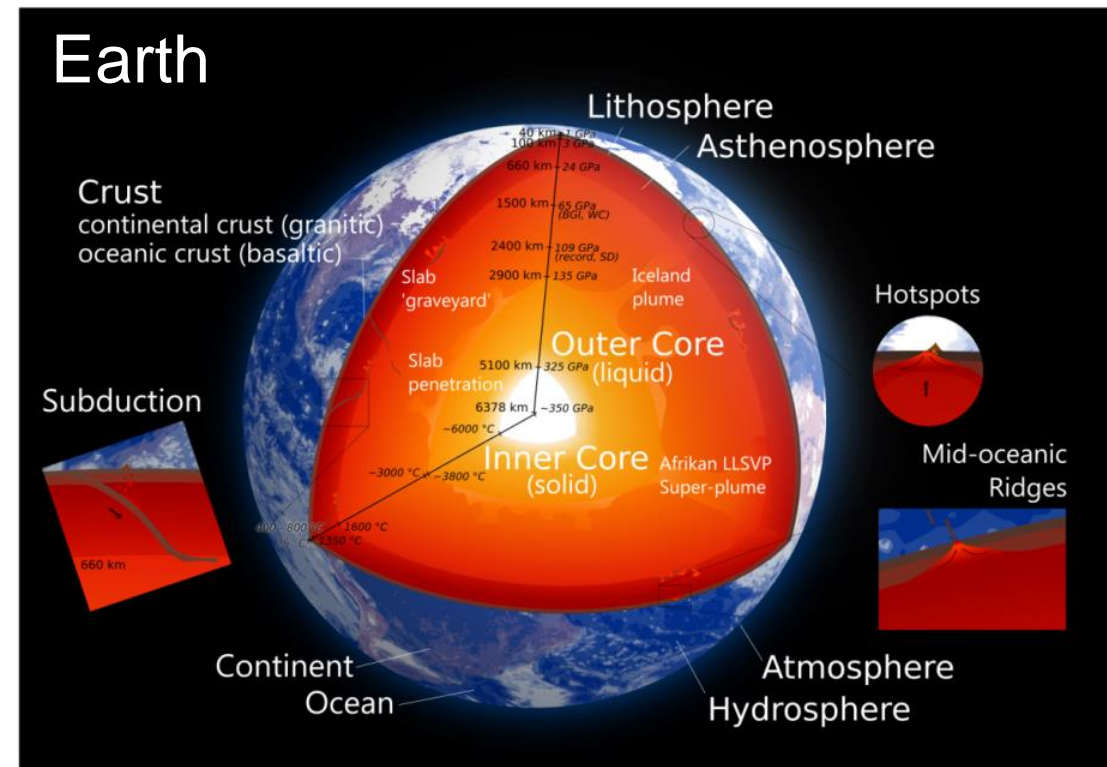
## Applications in geo- and material sciences:

### 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:

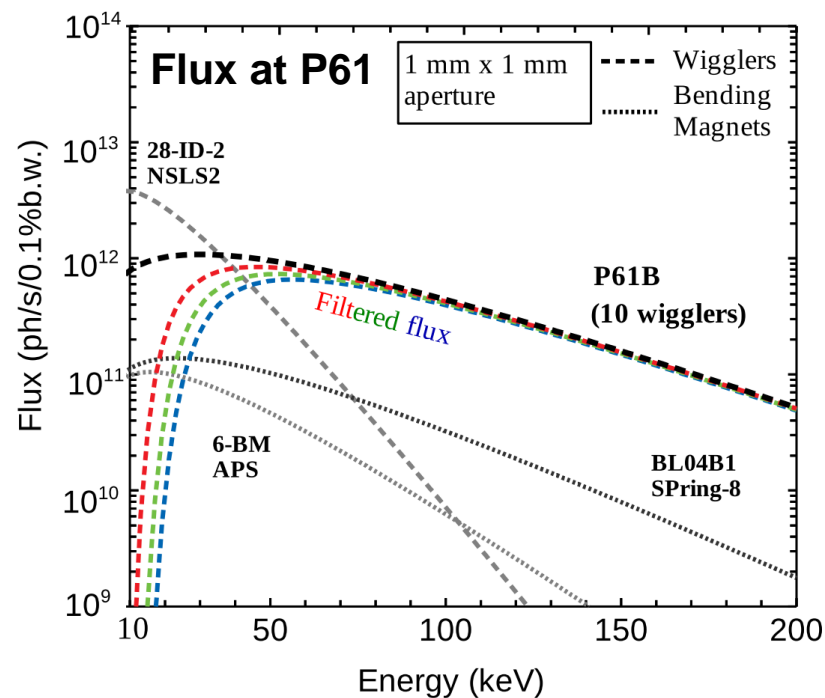
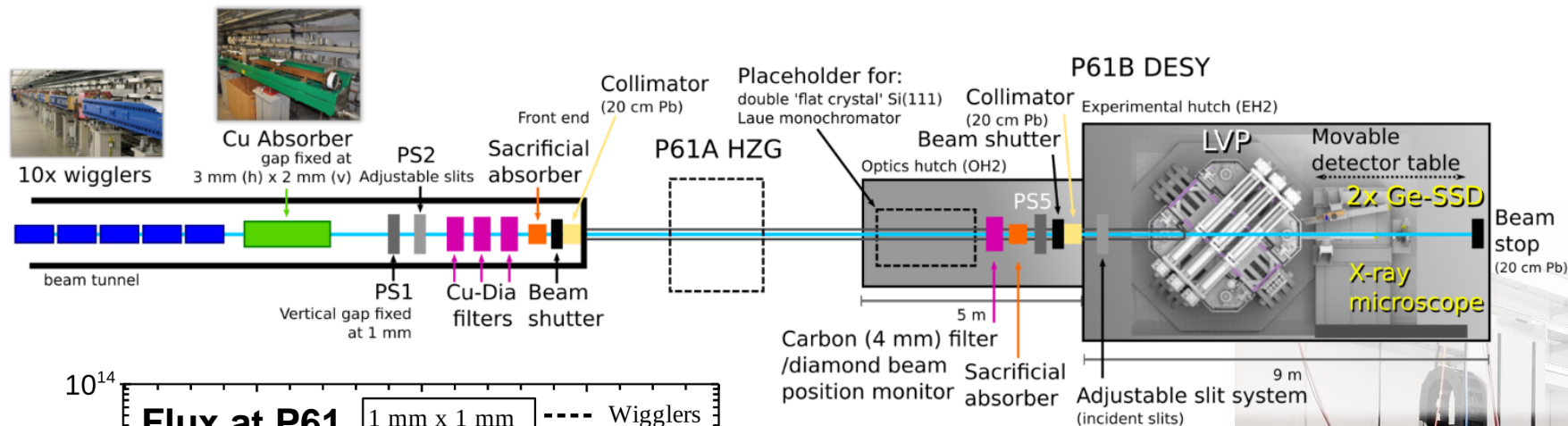
- MA experiments with complementary *in situ* techniques:
  - Ultrasonic interferometry
  - Acoustic Emissions testing
  - Electrical conductivity (upcoming)
- Synthesis of novel (recoverable) materials



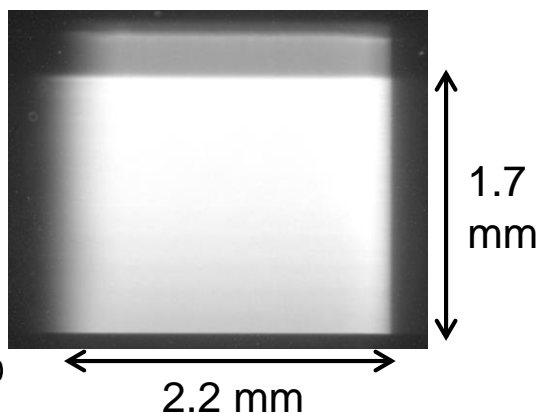


# Beamline layout

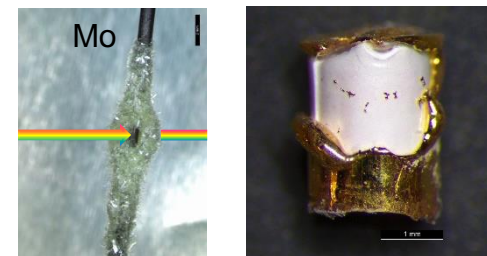
The Large Volume Press (LVP) extreme conditions beamline (50% in situ / 50% ex situ)



Full beam size

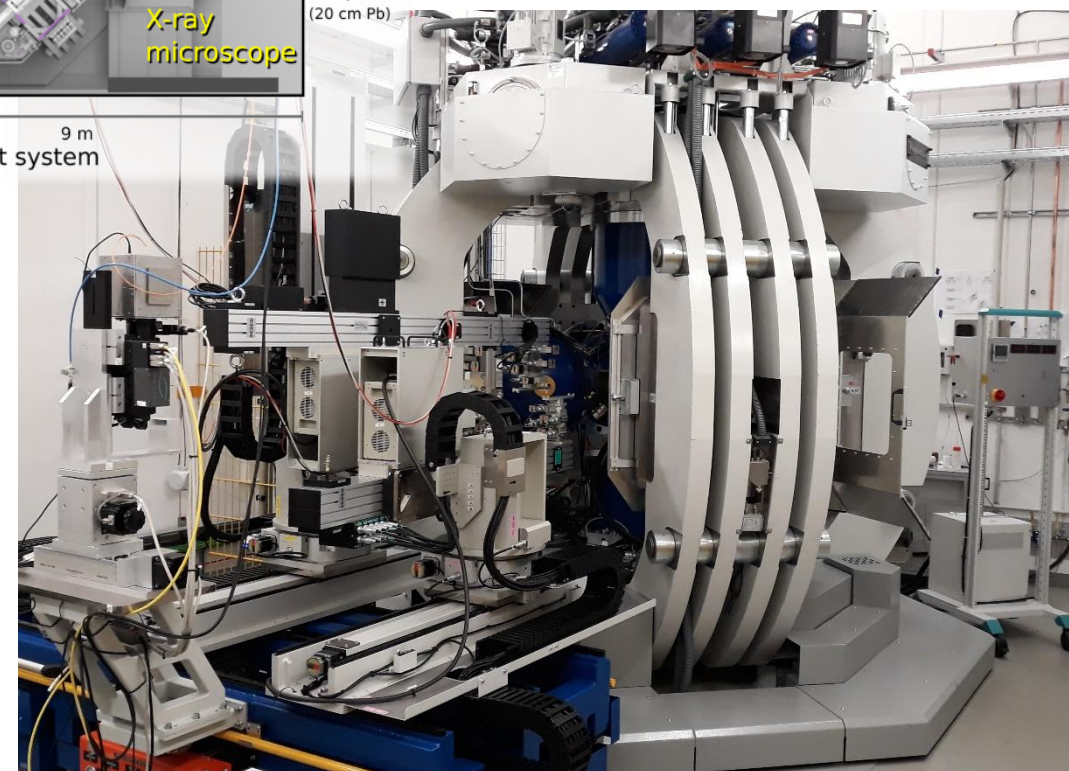


**CAUTION: HOT BEAM!**



Crystal growth of  $\text{MoO}_3$

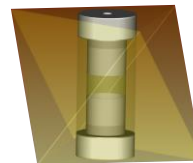
Molten Au capsule



New detector positioning system (since March 2021)

# High-pressure techniques

## Standard assemblies for isotropic compression

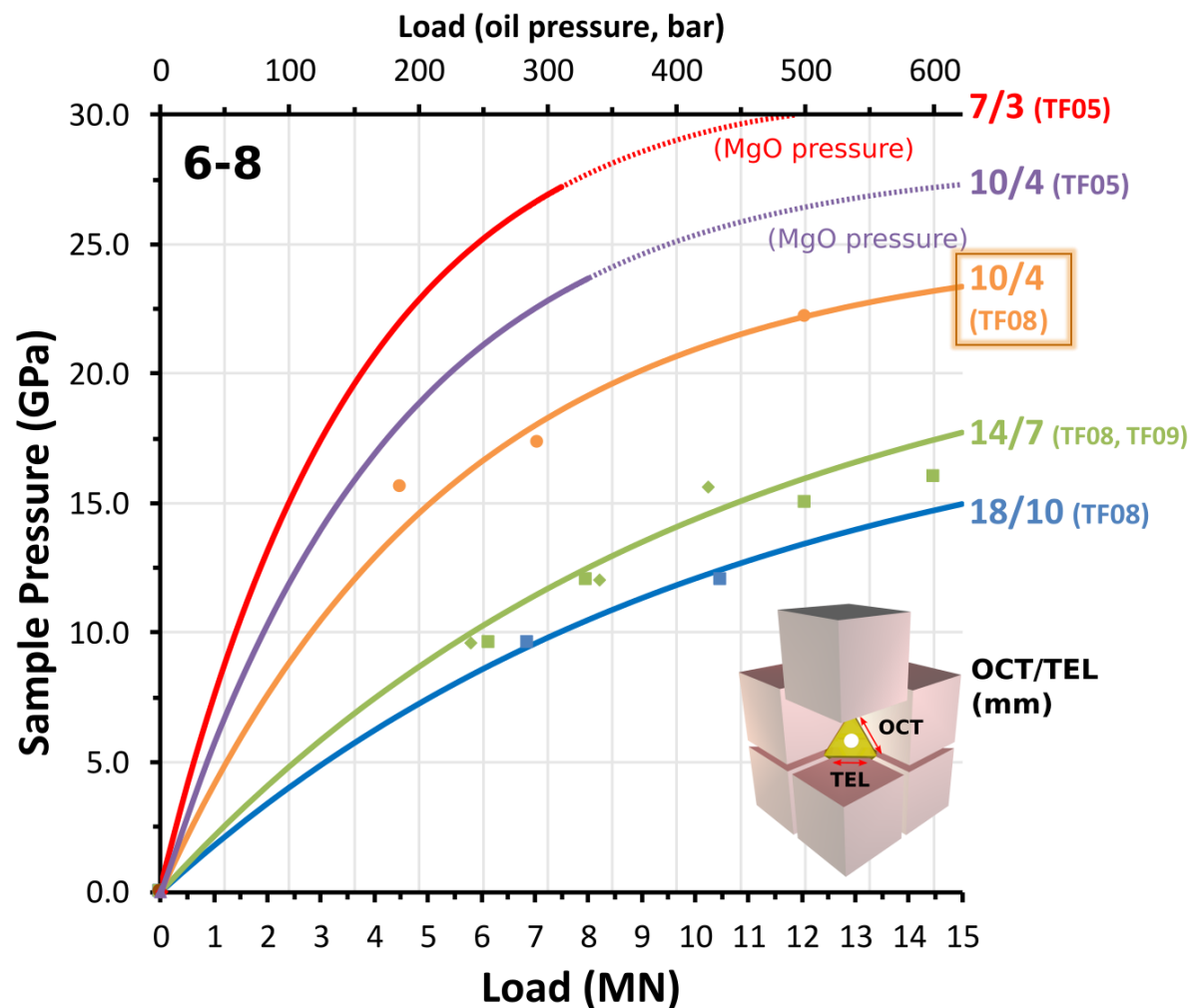
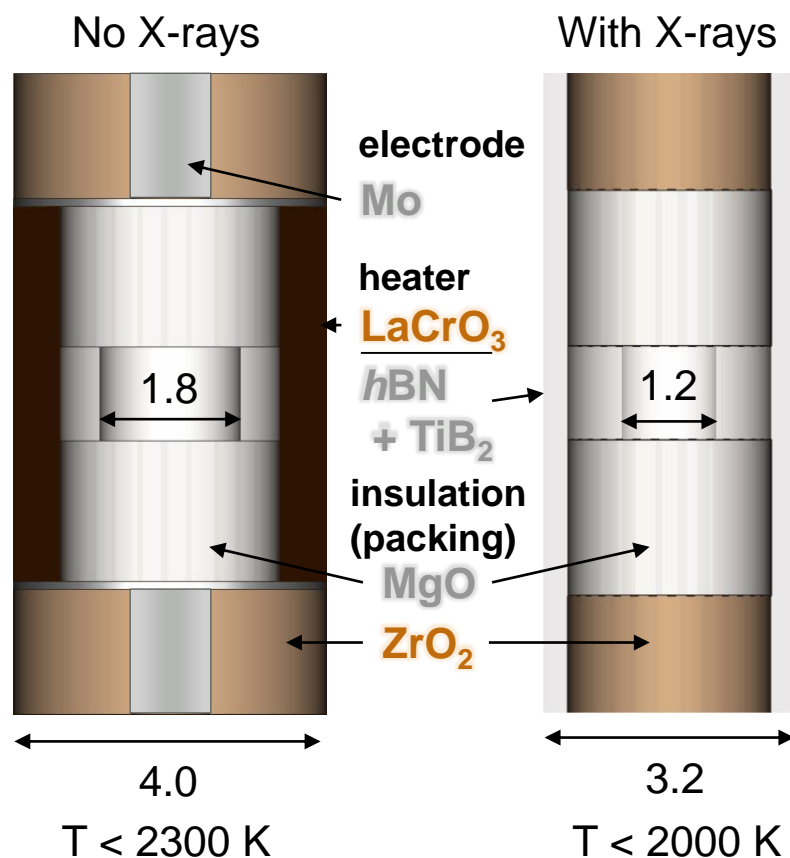


Simultaneous generation of ultrahigh pressure and temperature to 50 GPa and 3300 K in multi-anvil apparatus [also at P61B]

Longjian Xie *et al.* *Rev. Sci. Instrum.* 2021 (*in press*)

### 'Kawai' 6-8 mode

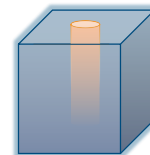
Example assembly "10/4" for  $P \geq 15$  GPa



Original assembly design by Dr. Nishiyama

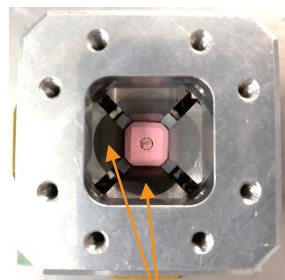
# High-pressure techniques

Standard assemblies for *in situ* studies of rock deformation



'Cubic' 6-6 mode

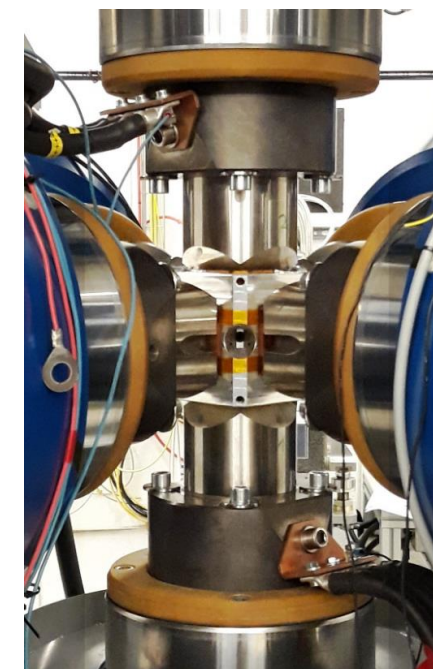
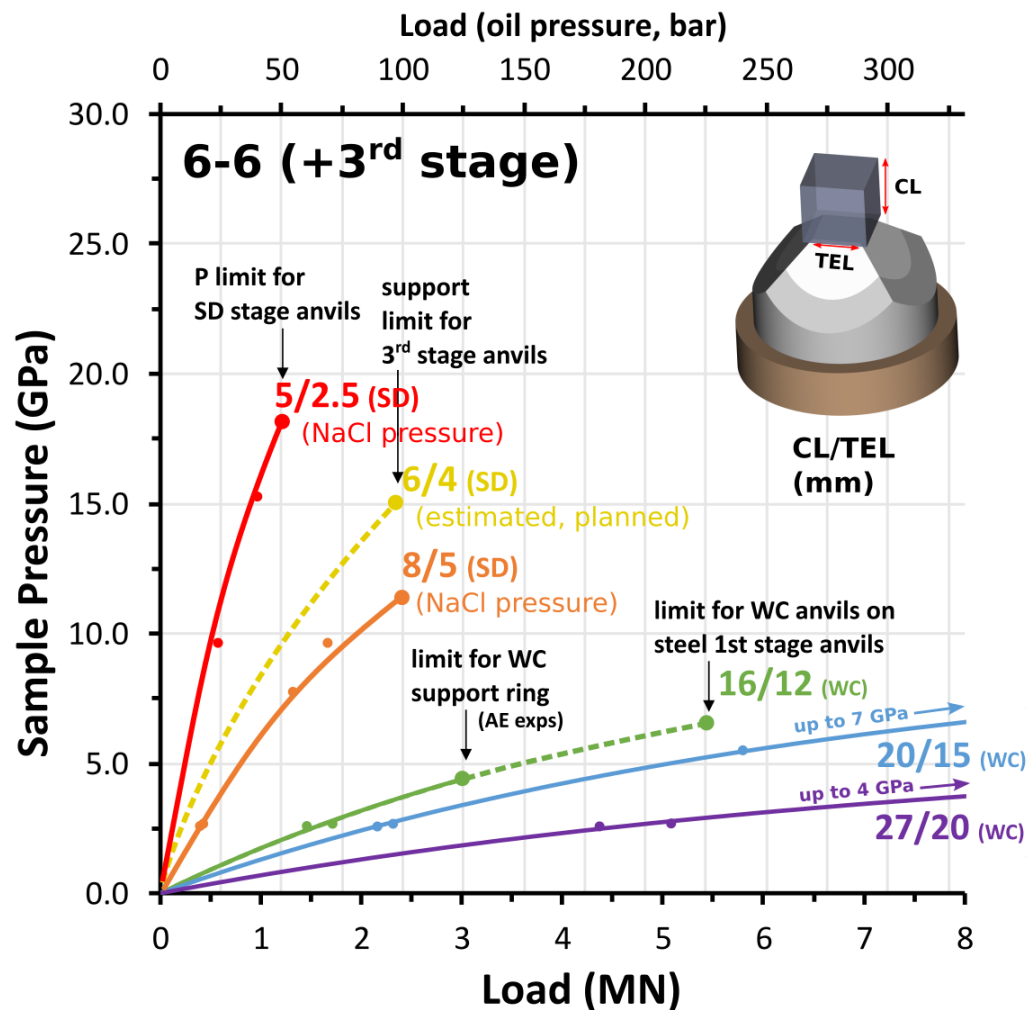
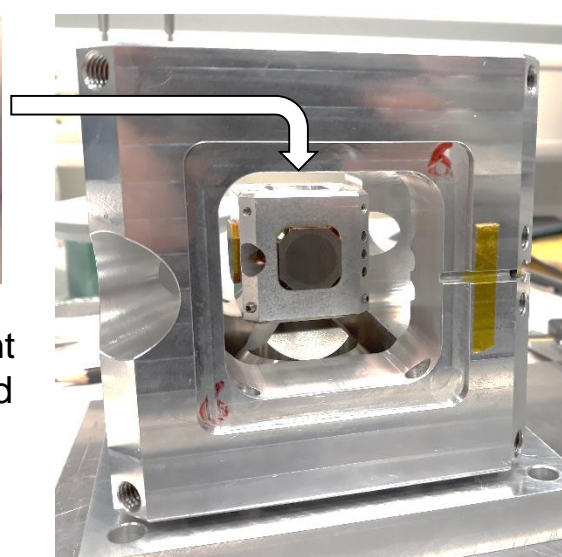
← 38 mm →



X-ray transparent  
sintered diamond  
anvils

New, 3<sup>rd</sup> stage  
design

← 110 mm →



Inside the LVP  
'Aster-15'

Generally for  
*ex-situ* synthesis



# The whitebeam X-ray microscope

## X-ray radiography

- **Double objectives (5x, 10x)** for high-resolution, full beam imaging
- **GGG: Eu / LuAG:Ce scintillators:** 20, 40  $\mu\text{m}$
- **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



Camera Control

Camera Settings | Stage Settings

Filename

File Path: /gfps/local/testCS/

File Name: 0\_1CS5\_Tas008

Comment: test

File Postfix: tif

Counter: 0

Camera Operation

Choose Folder

0\_1CS5\_Tas008+32+

Timing Data Saving

Exposure Time (s): [ ] Start

Delay Time (s): [ ] Stop

Number of Frames: [ ]

Timing Liveview

Exposure Time (s): [ ] Start

Delay Time (s): [ ] Stop

Region of Interest (ROI)

(Full resolution is X = 2560 px, Y = 2160 px)

Xmin: 1

Ymin: 1

Xmax: 2560

Ymax: 2160

Reset

Measurement

Mark No.: Length:  $\mu$  Angle to vertical axis:  $^\circ$

ROI Mark Mark

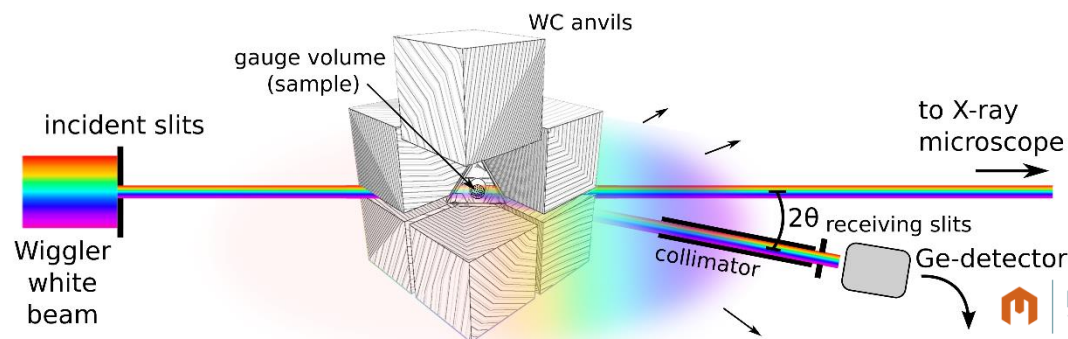
Distance Measurement Save Picture Save Picture As

# X-ray powder diffraction using white beam

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

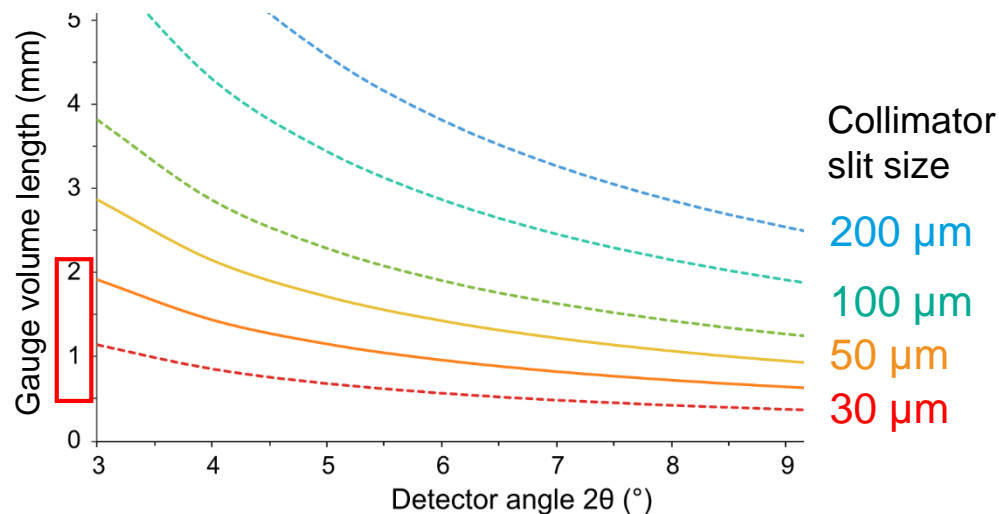
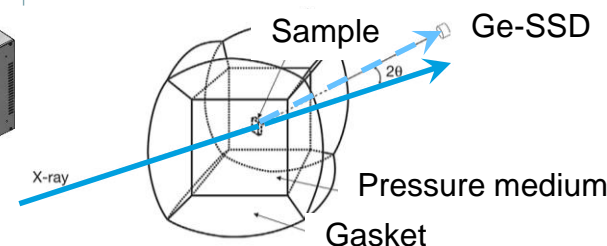
### ED-XRD in the LVP

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 (circled in red)      observable (circled in green)

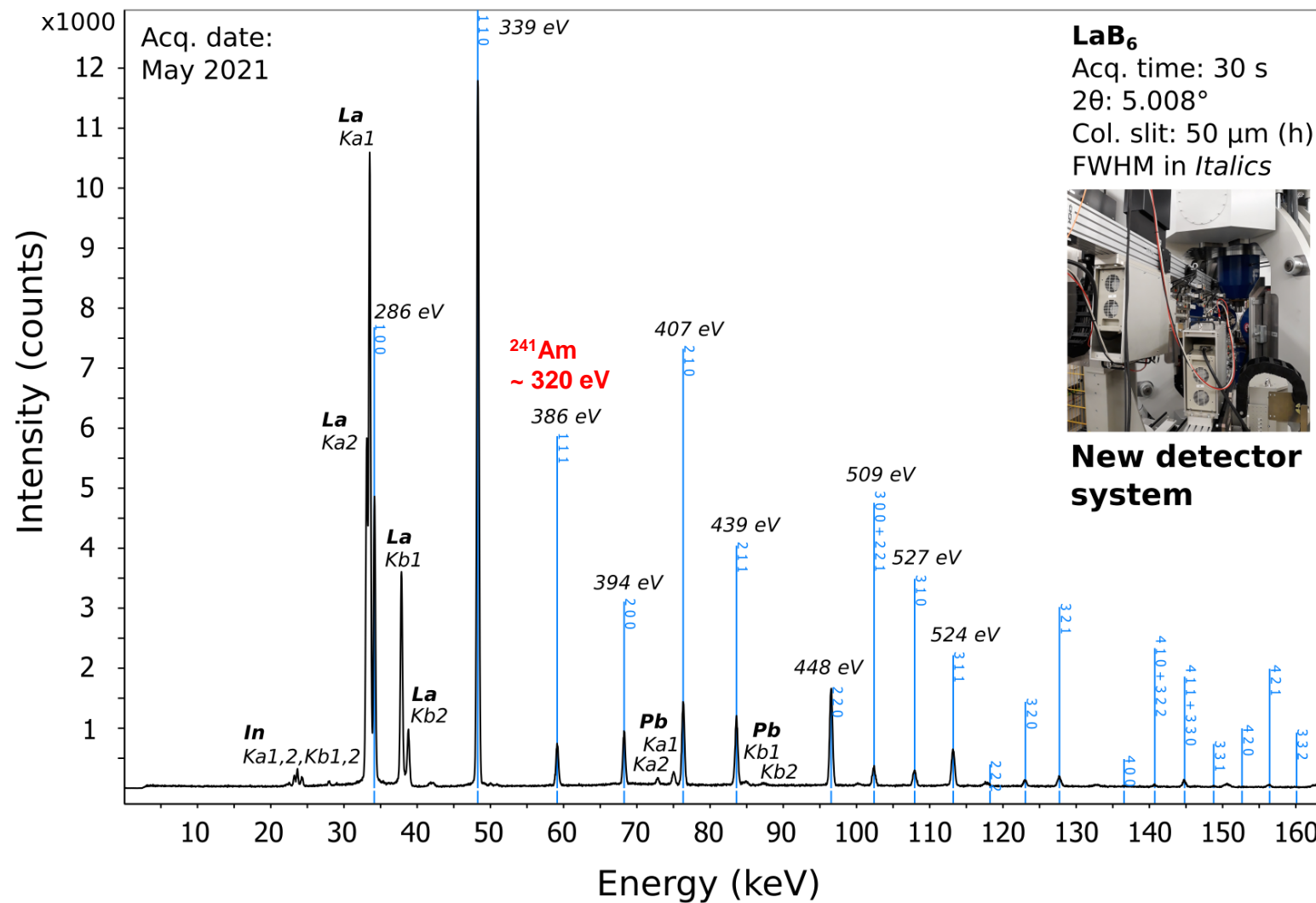


Ge-SSD (2x)	Mirion (Canberra)
Horz. detector positions	1xGe : min 3° - max 20° 2xGe : min 5° - max 10°
Horz. & vert. positions	Ge <sub>vert</sub> : min 7.5° - max 23° Ge <sub>horz</sub> : min 6.5° - max 10°



# 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<sub>6</sub> 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

Simultaneous Pressure and Temperature Determination

Use selected pair Use selected pair Use selected pair Use selected pair Use selected pair Use selected pair Use selected pair Use selected pair Loading takes 5 seconds!

**Steepest crossing**

NaCl(B1) + Pt  SiC + Pt  MgO + NaCl(B1)  hBN + Pt  MgO + Pt  Fe100 + Pt  MgO + Pt + hBN  MgO + W + hBN

NaCl(B1) + Ni  SiC + Ni  MgO + KCl(B2)  hBN + Ni  MgO + Ni  Fe100 + Ni  MgO + Pt + NaCl  MgO + W + NaCl

NaCl(B1) + Au  SiC + Au

NaCl(B1) + Ir  SiC + Ir

KCl(B2) + Pt  MgO + W

KCl(B2) + Ni  NaCl(B1) + Mo

KCl(B2) + Ir  NaCl(B1) + W

KCl(B2) + Mo

Mg Silicon carbide + MgO

	a0	a	V/V0	P (GPa)		P-IC (GPa)	Pmin (GPa)	Pmax (GPa)	$\Delta P$ (GPa)	Isochor $\pm$ (GPa)
SiC	4.3586	4.3586	1.00	-0.000	Calculate	0.000		0.138		0.05
MgO	4.2112	4.2112	1.00	0.000				338.42		0.05

T (K)   
 T-IC (K)   
 Tmax (K)   
  $\Delta T$  (K)

**Equation of State Cross-fit P-T estimation**

Simultaneous pressure and temperature determination by two EoS

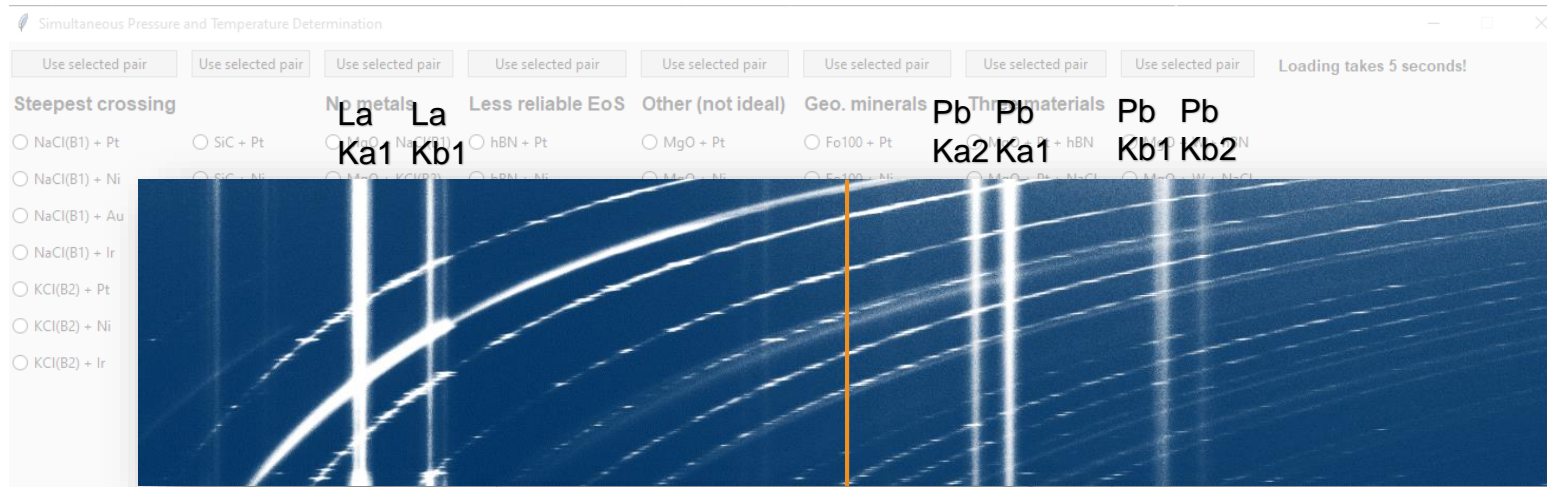
— SiC (Nisr17 / Vinet)  
— MgO (Tange09 / Vinet)

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

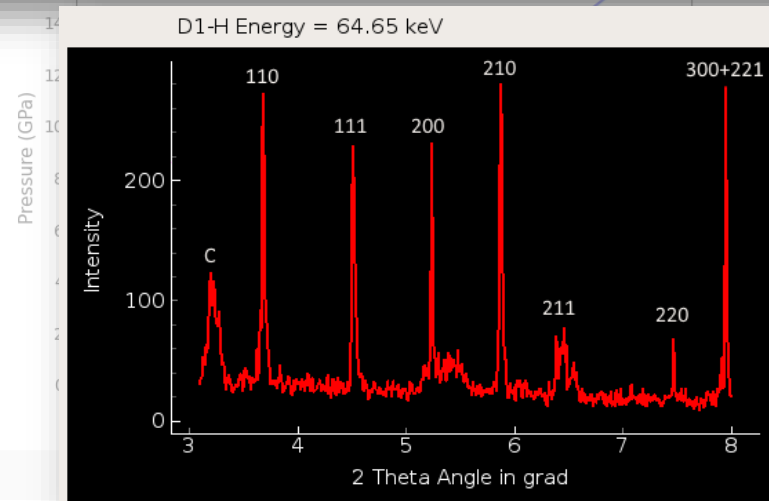


Custom software for simultaneous PT estimation using EoS of pairs of materials (e.g. MgO+SiC)

Angle (deg)

8°

Energy →

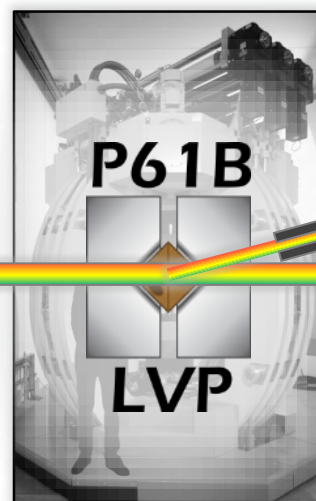


Combined angle and energy-dispersive structural analysis refinement (CAESAR) (Wang et al. 2004).

### First result on $\text{LaB}_6$ .

- 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. Chanyshv (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*

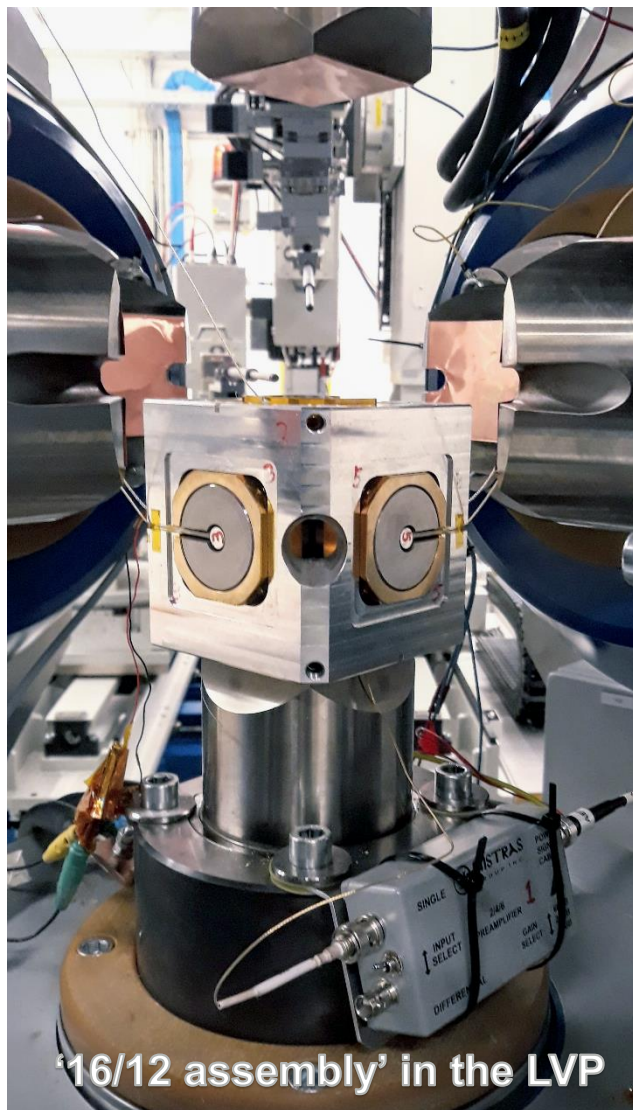
**3. Earth Materials**  
**Acoustic Emissions**  
S. Ma (Jilin Uni, China/DESY)  
J. Gasc (Uni Montpellier)  
S. Incel (Bochum)

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



# Acoustic Emissions testing (*in situ*)

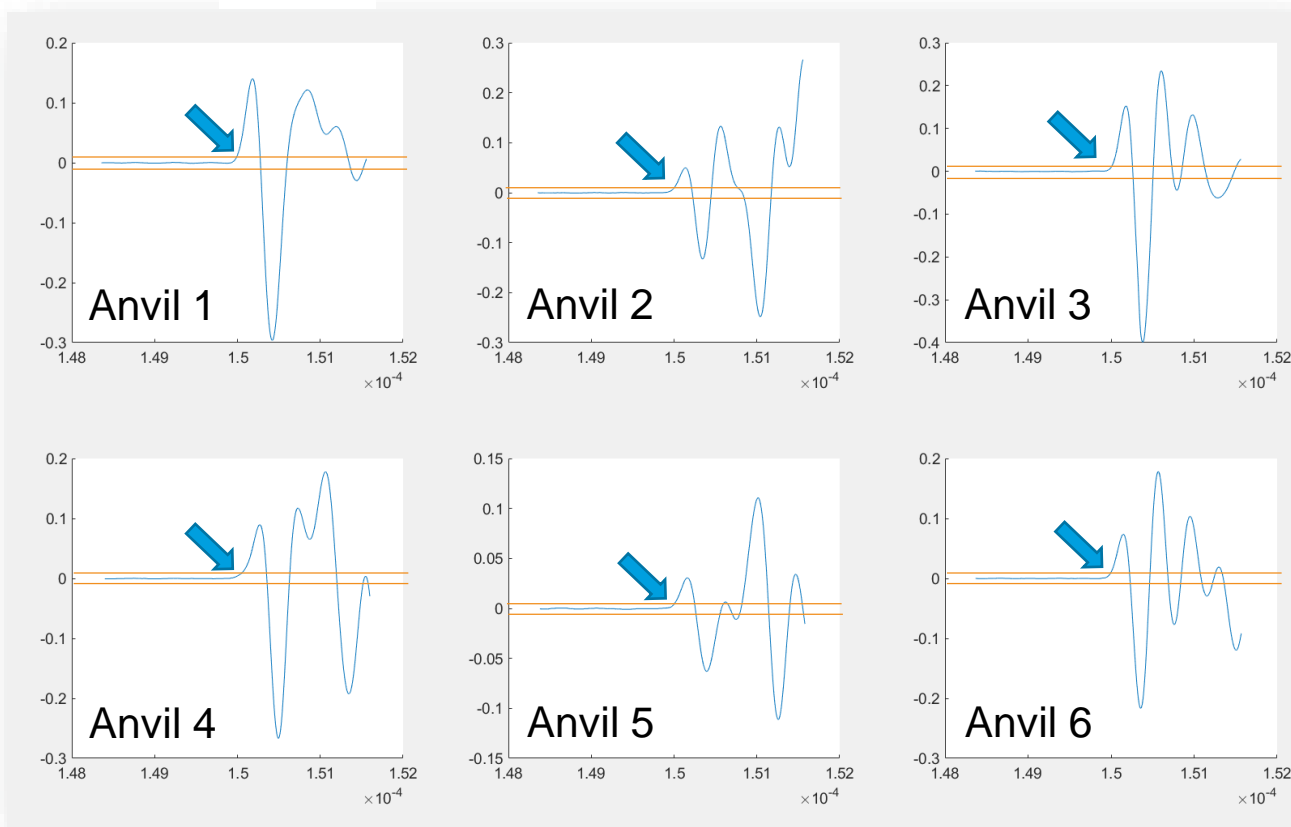
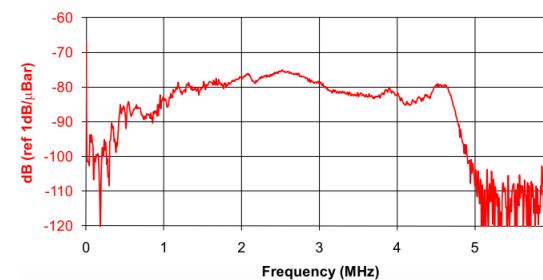
## Methodology (MA6-6 compression)



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.

**Micro200HF Sensor**  
Very Wideband Frequency Miniature Sensor



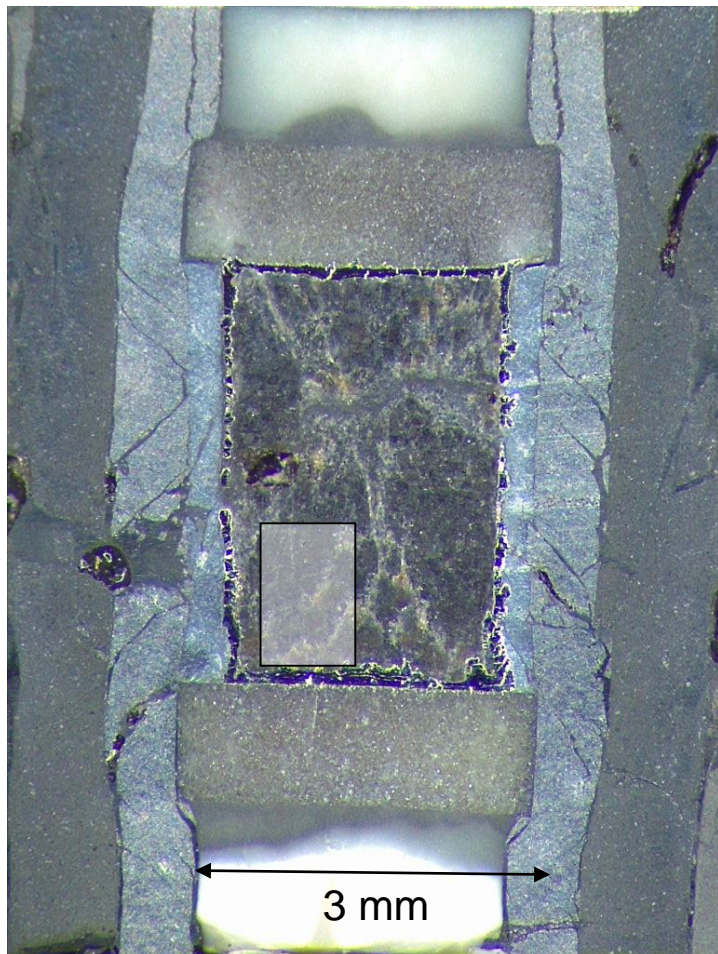
**Acoustic Emissions**  
S. Ma (Jilin Uni,  
China/DESY)  
J. Gasc (Uni Montpellier)  
S. Incel (Bochum)



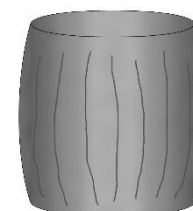
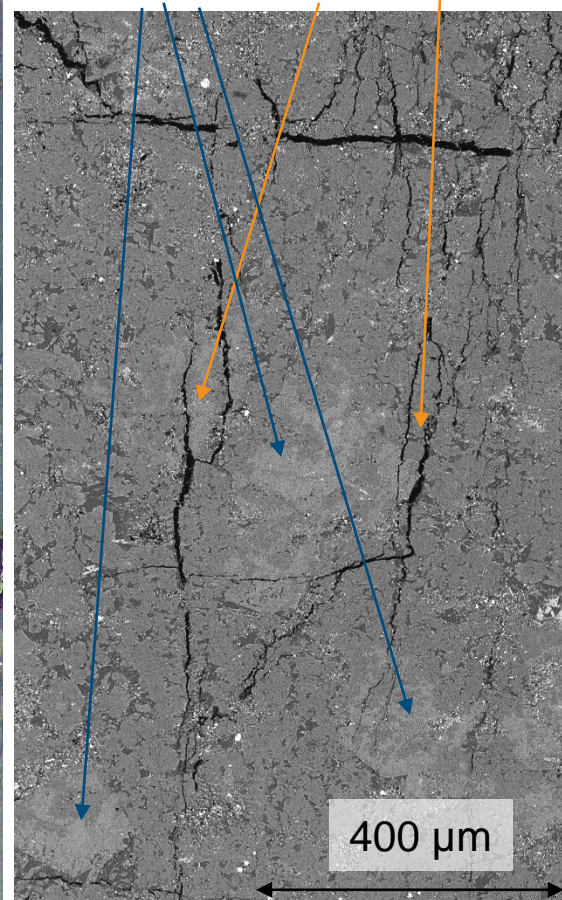
# Acoustic Emissions testing (*in situ*)

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

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

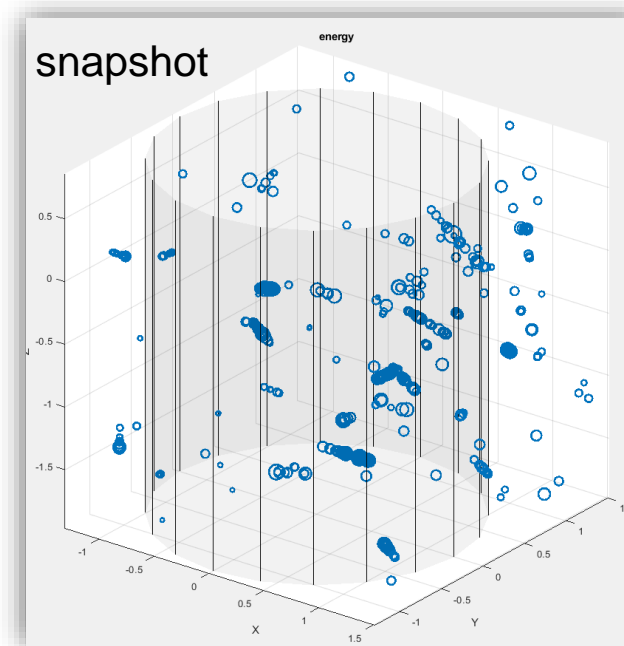


Garnet nucleation  
Mode-I tension cracks?

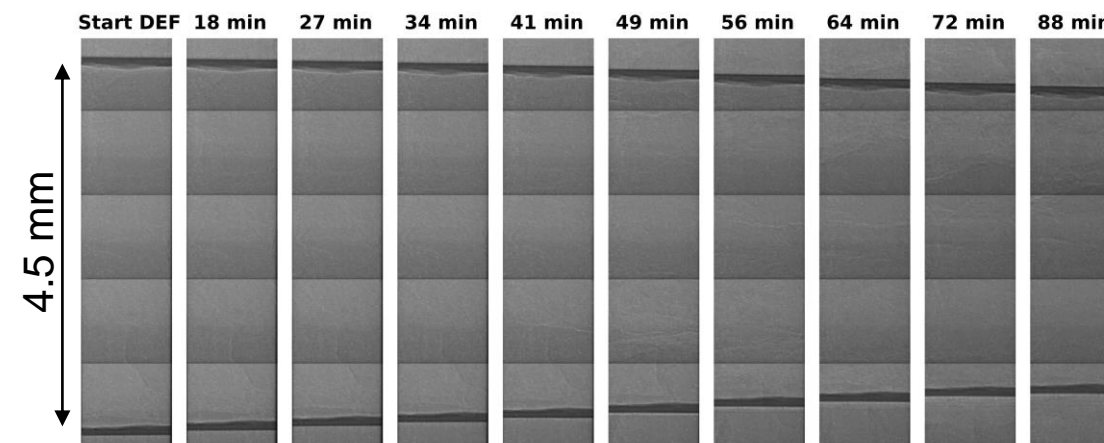


cracked specimen

*Acoustic Emissions:*  
3d event location  
(circle size  $\propto$   
energy release)



Strain history



# Wave speed measurements (*in situ*)

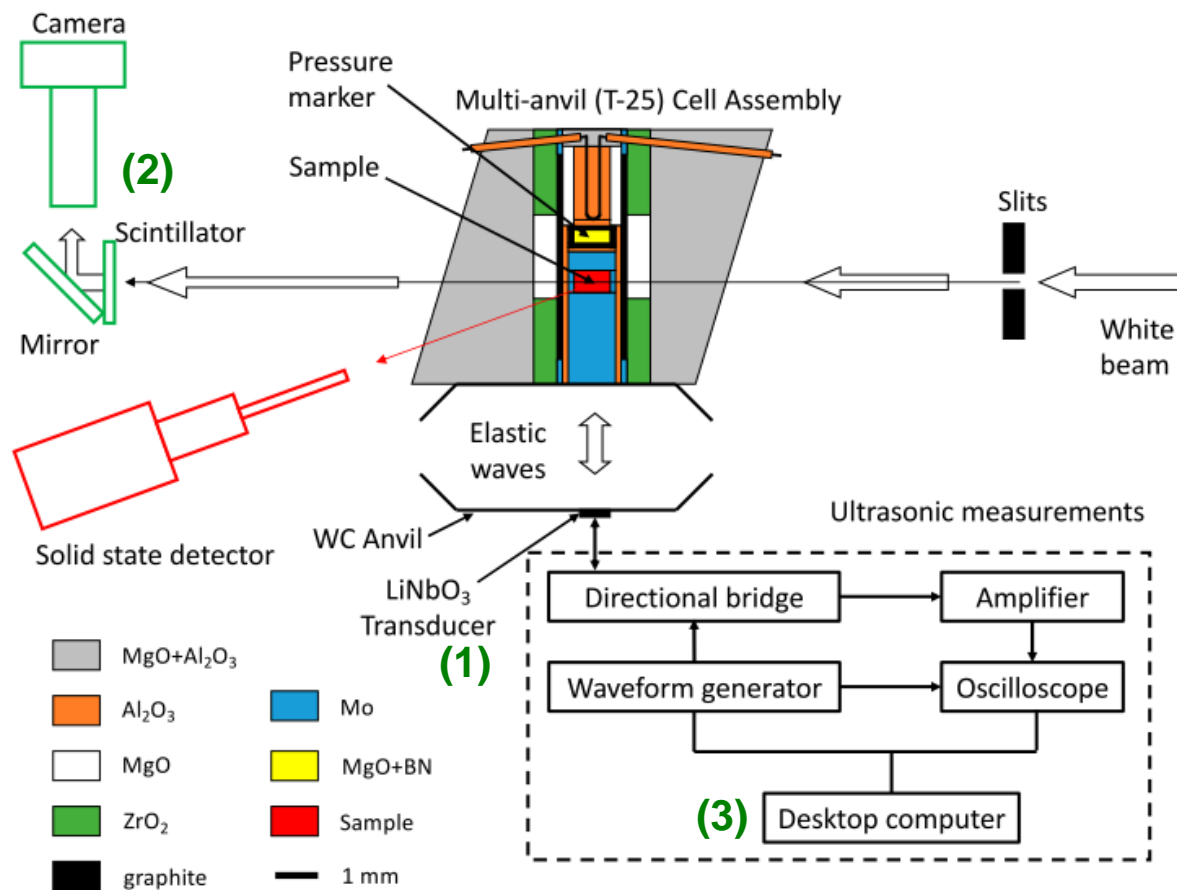
Ultrasonic Interferometry: Now available at P61B

**Ultrasonic Interferometry**

R. Farla (DESY)

A. Neri (BGI)

Lianjie Man (BGI)



## General method

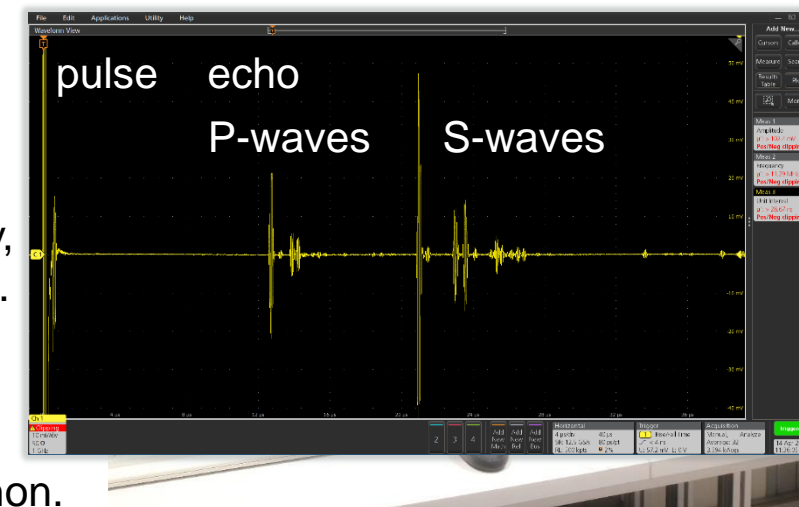
(1) A **LiNbO3 sensor** of choice on the back of a mirror polished anvil, transmits a pulse and receives an echo.

(2) Simultaneous imaging (**radiography**) provides sample length with **sub-pixel resolution** ( $< 1 \mu\text{m}$ ).

(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.

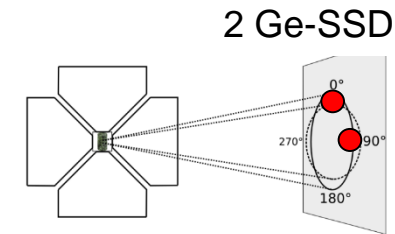




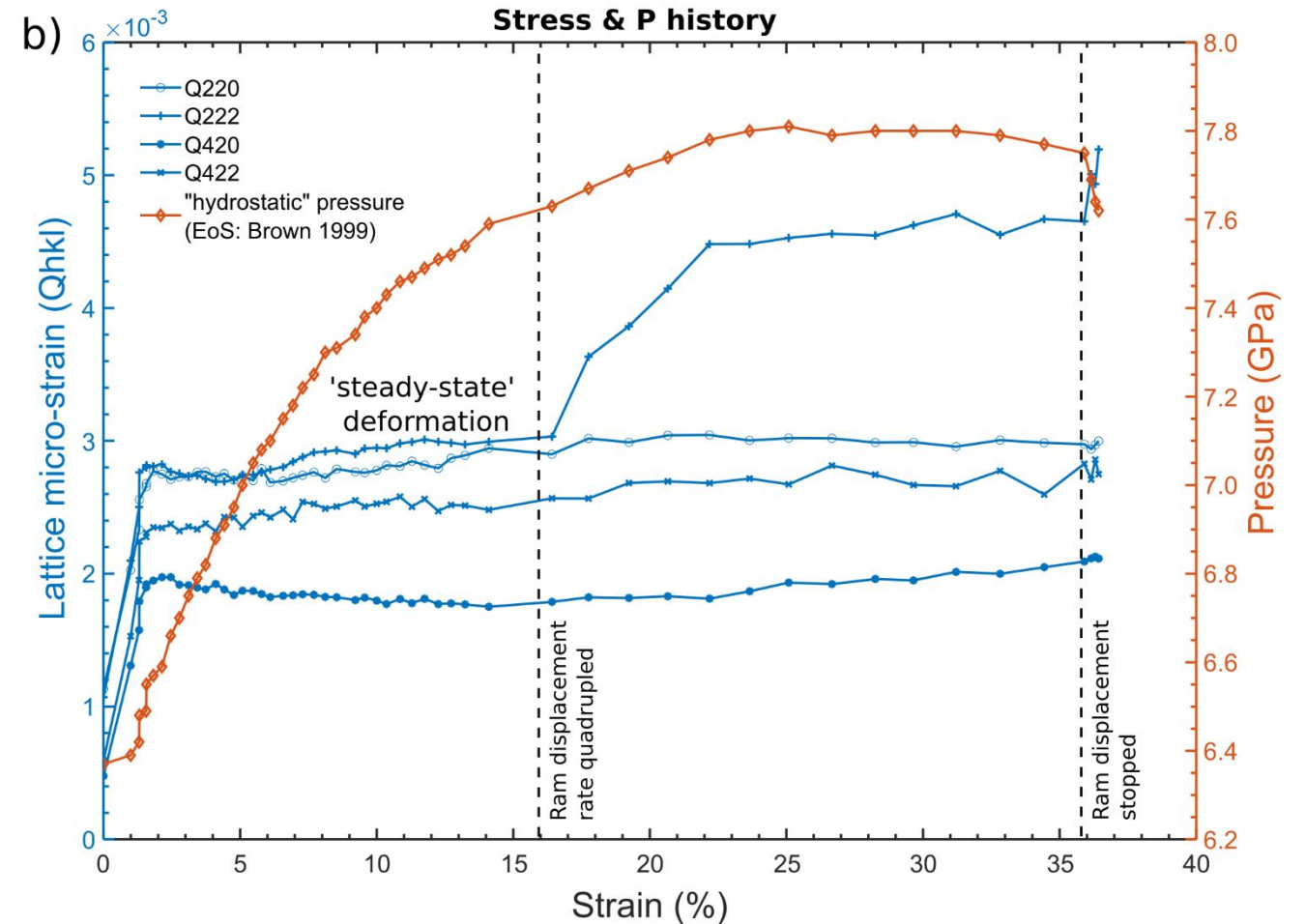
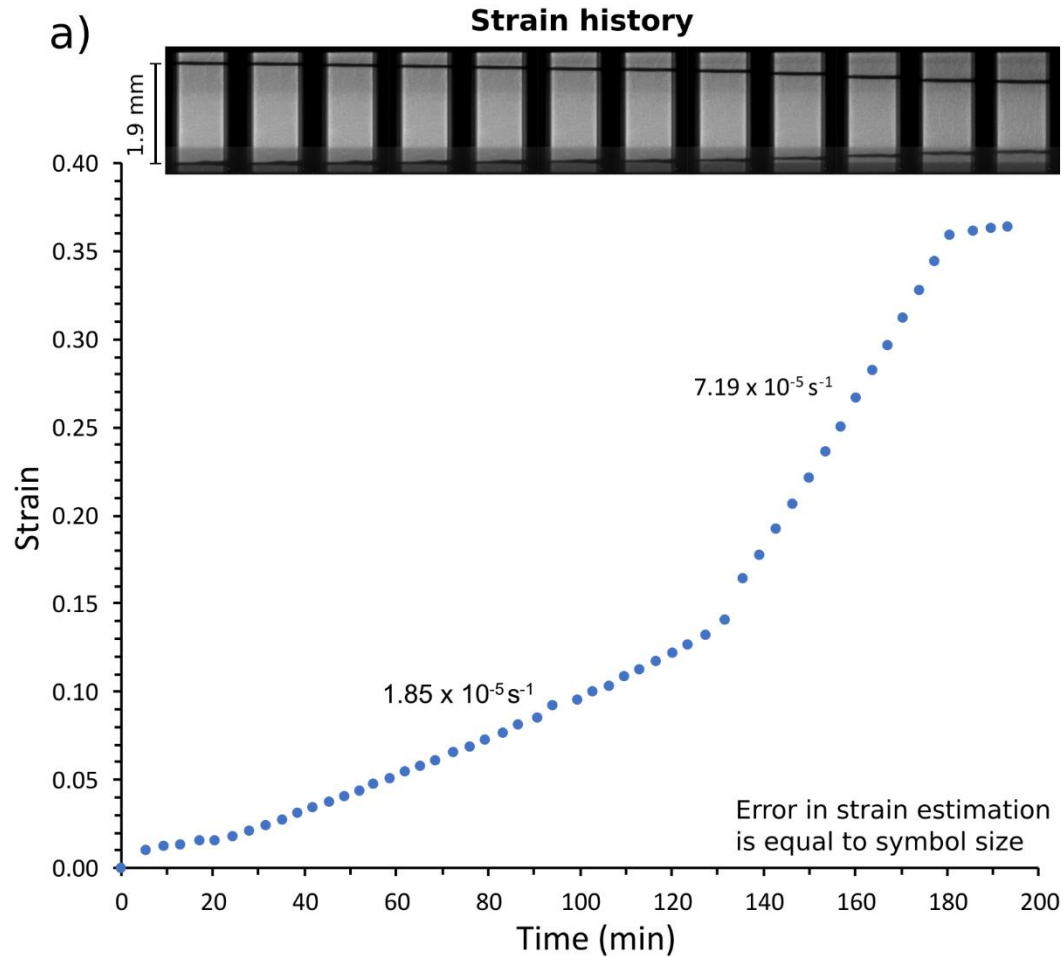
# Rock deformation (*in situ*)

## 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,  $2\theta = 7.5^\circ$ )



**Rock Mechanics**  
R. Farla (DESY)

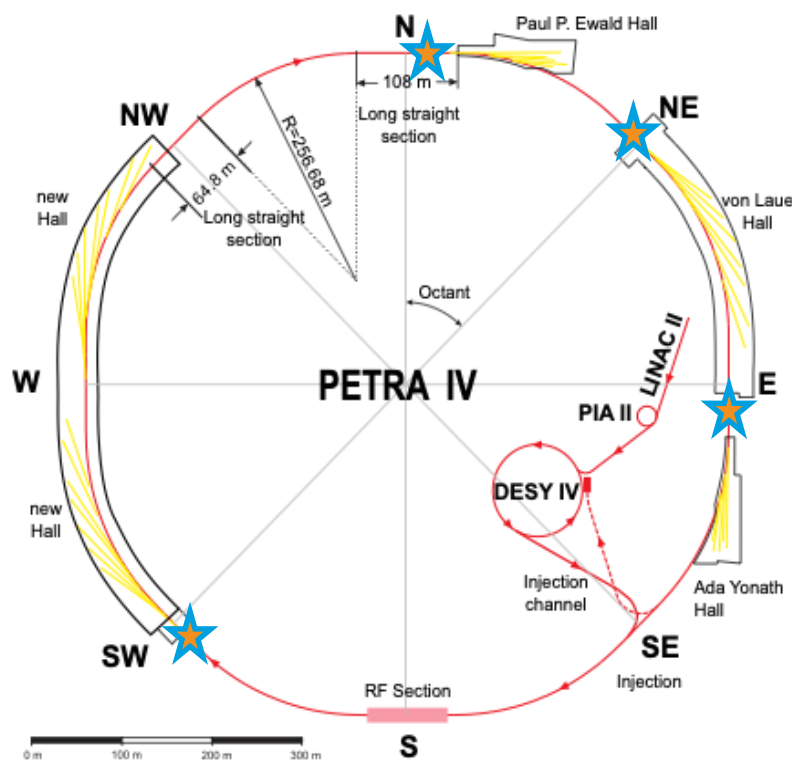


# PETRA IV

**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).

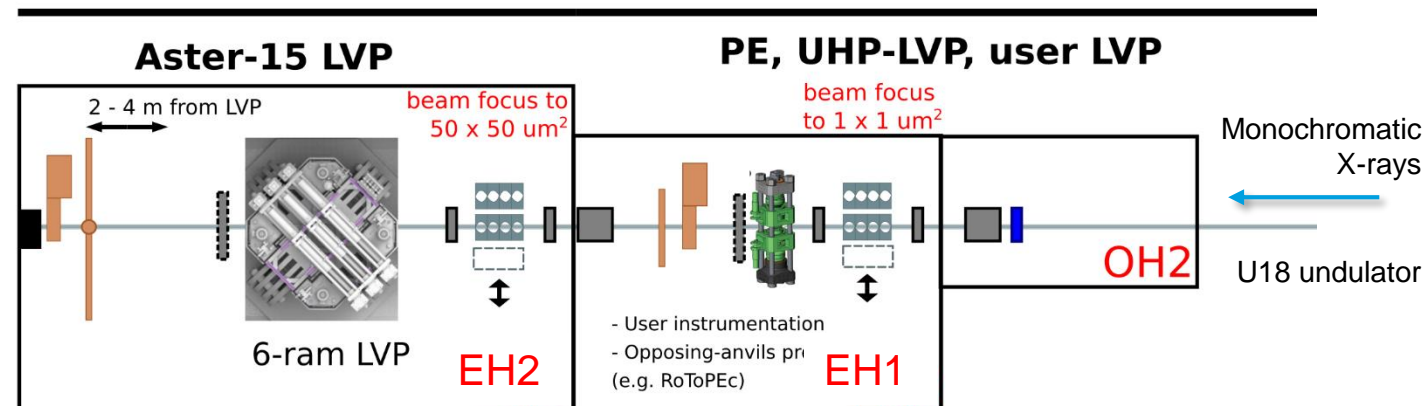
**Possible BL concept:**

115 m

100 m

90 m

83 m



- High energies (40 – 120 keV harmonics)
- High-res PXR / Imaging /  $\mu$ -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!**

- ... Sheet P61B
- ... ions from P61B
- ... ncing P61B
- ... t & Staff
- ... e layout
- ... Data Sheet P61
- SMAT
- ... nced XAFS
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- FLASH SCHEDULES
- ROJECT
- + PROJECT
- FRAStructure
- LAB
- IXFEL
- SORTIA EUROPEAN XFEL
- INSTITUTES ON-SITE
- rchive

- PETRA III >>
- FLASH >>
- EUROPEAN XFEL >>
- CFEL >>
- CSSB >>

- C22B >>
- ... >>

## Announcements

[1] P61B will continue normal operations for the current 2021-I and 2021-II runs. Travel restrictions may apply due to the COVID-19 pandemic. Please stay informed about the DESY COVID rules.

[2] Call for proposals (2022-I beam time):  
Regular proposals: **Mo 19.7. - Wed 1.9.2021 (deadline!)**

## Visiting guidelines

→ Read the following information [here](#), before visiting.

Please decide whether you prefer Collaborative or Independent beam/experiment time.

## News

→ Visit [here](#) for the latest beamline news.

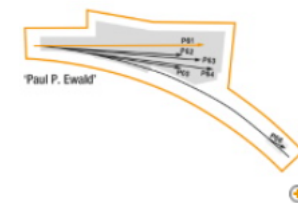
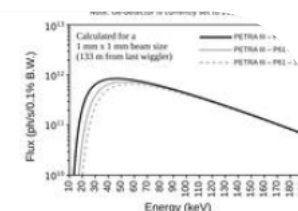
## Application for LVP experiment time

→ **Apply** for standalone LVP use at P61B. Applications can be submitted anytime. Current available dates:

- 10 June - 06 July, 2021
- 19 Aug - 14 Sept, 2021
- 18 Oct - 02 Nov, 2021
- 09 Dec - 21 Dec, 2021

**Visit the beamline website @**  
<http://tiny.cc/petra3p61>

- Announcements
- Calls for proposals
- LVP access w/h X-rays
- Beamline activities
- And more...



Location of beamline P61 and end station P61B in the Paul P. Ewald hall.



## Contact

**DESY.** Deutsches  
Elektronen-Synchrotron

[www.desy.de](http://www.desy.de)

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FS-PETRA-D  
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Tel: 4470