Status & development of the LVP beamline P61B at PETRA III

Workshop presentation

DESY user meeting 2021

Robert Farla (beamline leader)
01-02-2021

Acknowledgments:
Stefan Sonntag, Shrikant Bhat, 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

P61B LVP

DESY.

Outline of the talk

I. **Introduction**
- Beamline mission

II. **Beamline layout and characteristics of P61**

III. **High-pressure techniques**

IV. **Commissioning with X-ray beam**
- White beam X-ray microscope *for radiography*
- 2x Ge-detectors *for energy-dispersive X-ray diffraction (ED-XRD)*
- Graphical user interfaces for data acquisition and visualization

V. **Research and Development**
- Overview beamline activities and active collaborations
  - Acoustic Emissions & embrittlement of rocks
  - Ultrasonic interferometry & *in situ* wave speed measurements
  - *In situ* studies of Rock Deformation

VI. **Planned work**
- Installation glovebox
- Development of a monochromator for AD XRD

VII. **PETRA IV**
- Status and concept beamline for TDR

VIII. **Summary**

P61B LVP Mission

Applications in geo- and material sciences:

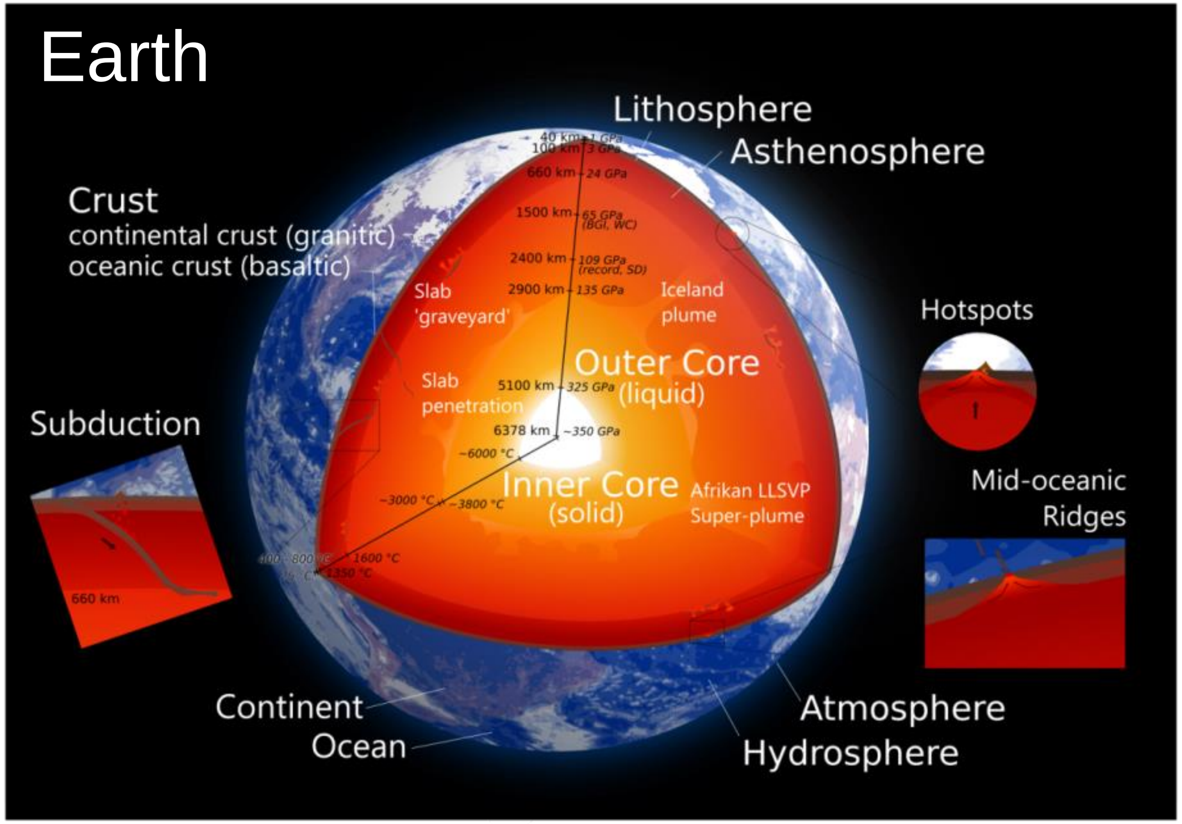
- Phase relations:
  - Transformation/nucleation
  - Melting curves (solidus/liquidus)
  - Equations of state
- Crystallography (w/ CAESAR or mono)
- Controlled rock deformation
- Melt viscosity measurements
- Structure of amorphous materials

Complementary *in situ* techniques:

- Ultrasonic interferometry
- Acoustic Emissions testing
- Electrical conductivity

Synthesis of novel functional materials

- Production feasibility (industry?)



P02.2 ECB

- Extreme pressures (1 TPa)
- Small (0,001 mm³) samples
- Single phase (typically)

P61B

Ultra-high pressures (60±0.1 GPa)
Large (100 mm³) samples
Polymineralic rock
→ Study of grain boundary transport properties (conduction, diffusion, rheology) !

Beamlines are complementary!

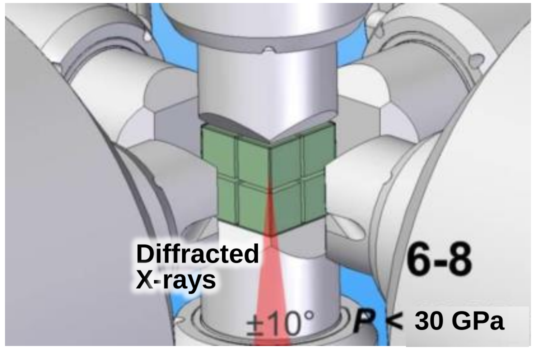
P61B LVP Status

Quick summary

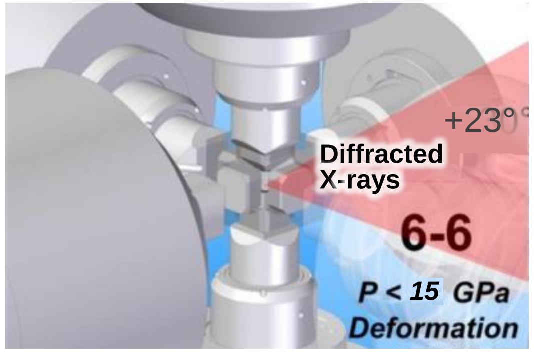
- ✓ User runs started on Aug. 2020.
- ✓ Temporary 1-detector setup for ED-XRD was successful for many users.
- ✓ New, 2-detector positioning system under commissioning (March 2021).
- ✓ Radiography 'works'.
  - Viscometry (falling sphere) and deformation exps. to be explored.
  - LVP z-stage scanning to be added.
- ✓ Cubic compression (MA666) currently tested using sintered diamond anvils for *in situ* rock deformation experiments.
- ✓ Acoustic Emissions setup *nearly ready* to be combined with X-ray measurements.
- ✓ Ultrasonic interferometry (wave speed measurements) setup ready, to be commissioned.

Versatile compression modes

Kawai-type (8-6):
Extreme pressures,
reliable geometry

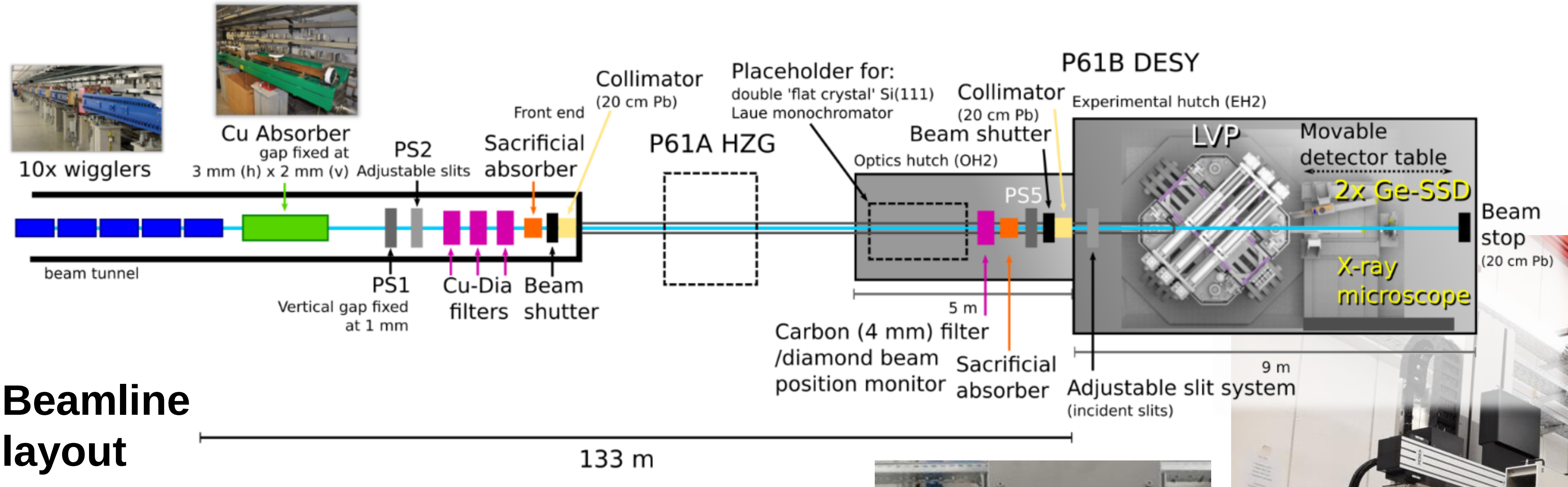


Cubic (6-6):
Large X-ray window,
anisotropic compression

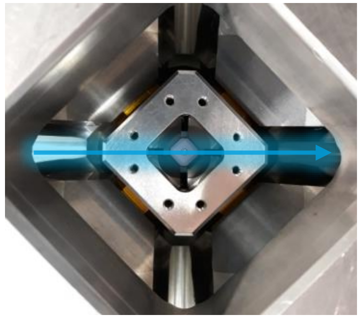


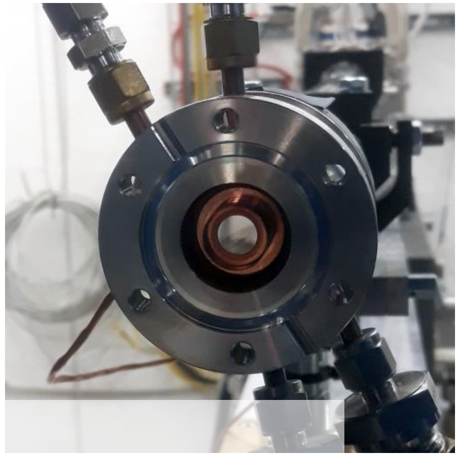
Beamline layout

The Large Volume Press (LVP) extreme conditions beamline

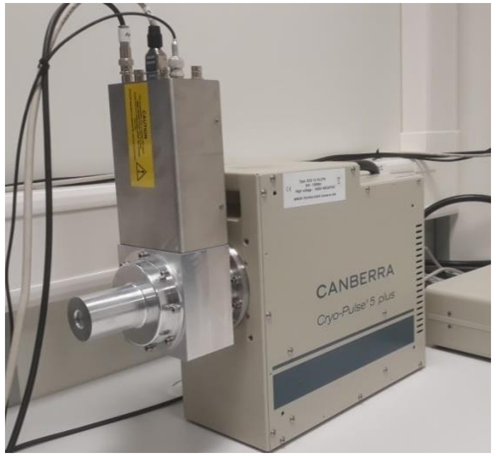


Beamline layout



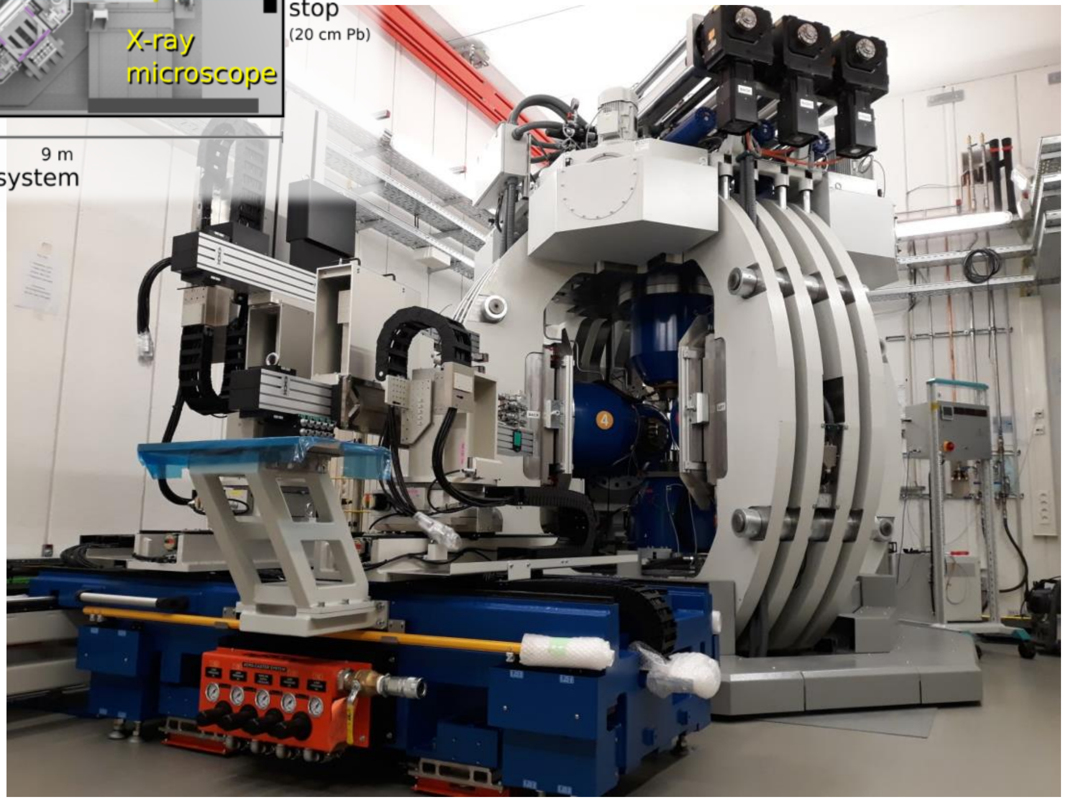


CVD-diamond exit window



HP Ge-SSD w/ electric cryostat

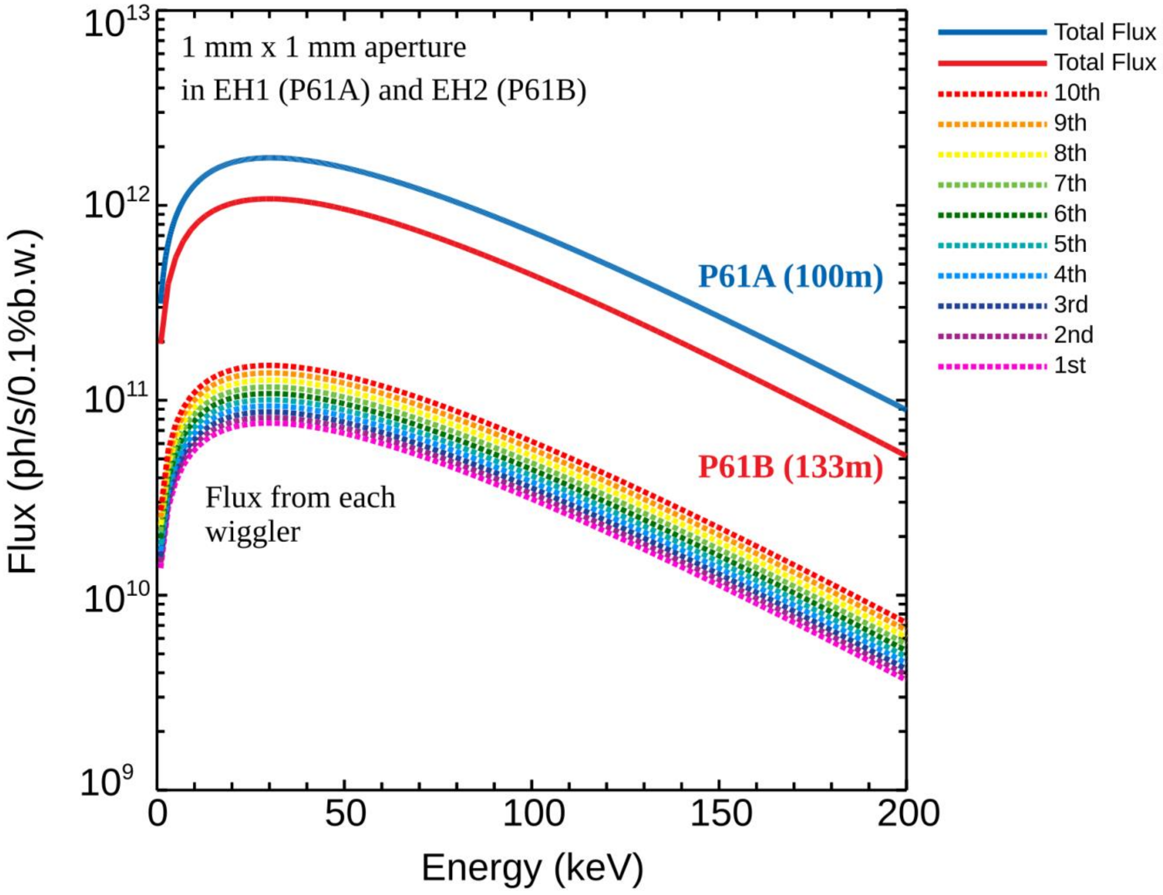
X-ray microscope

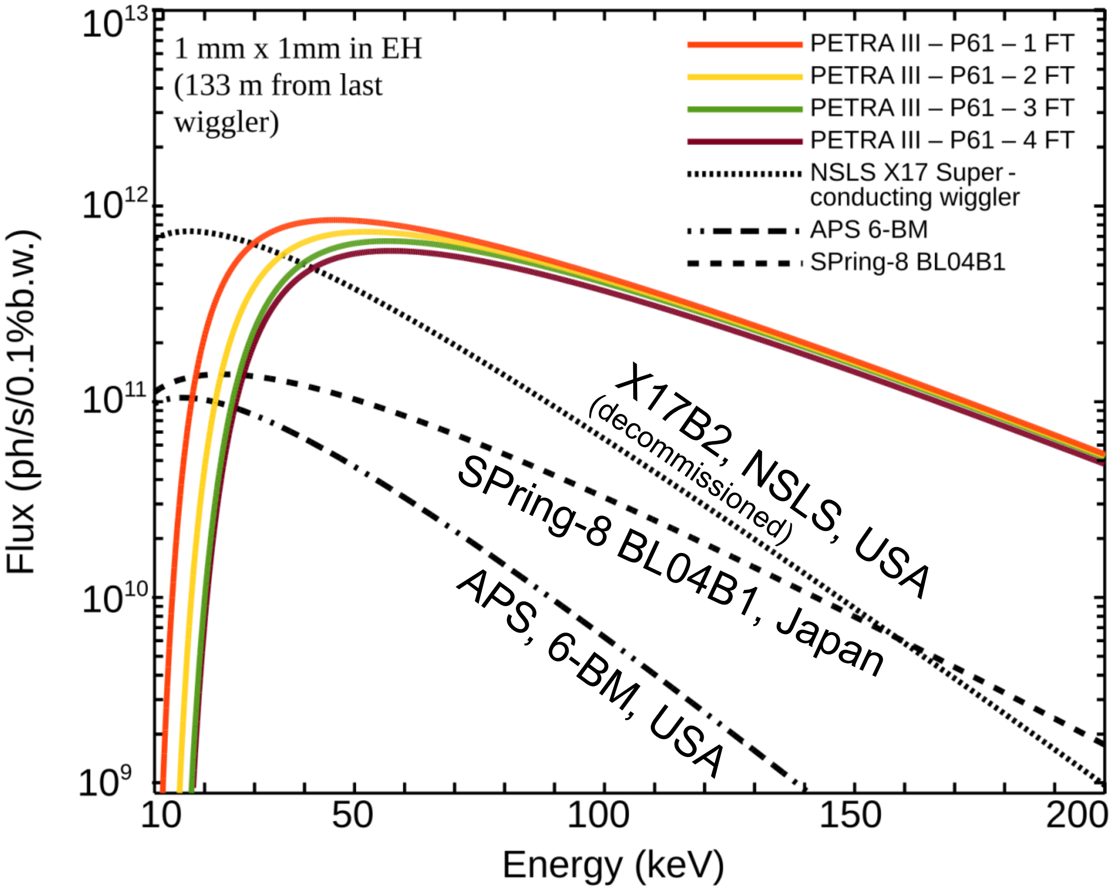


New 2-detector positioning system, in commissioning

Flux and power

Calculations of photon flux at P61B





Flux and power

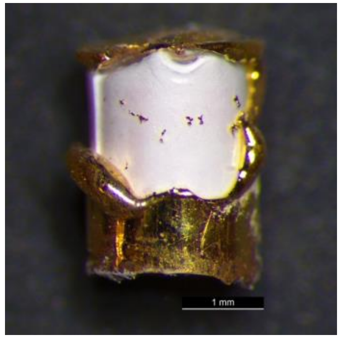
Beamline optics, beam size and power

Adjustable 1 cm thick tungsten slits in EH2

- Unfiltered beam: **16 W/mm²**
- 3 FE filters: **10 W/mm²**
- Variable Cu absorber: **< 1 W/mm²** (flux loss below 70 keV)

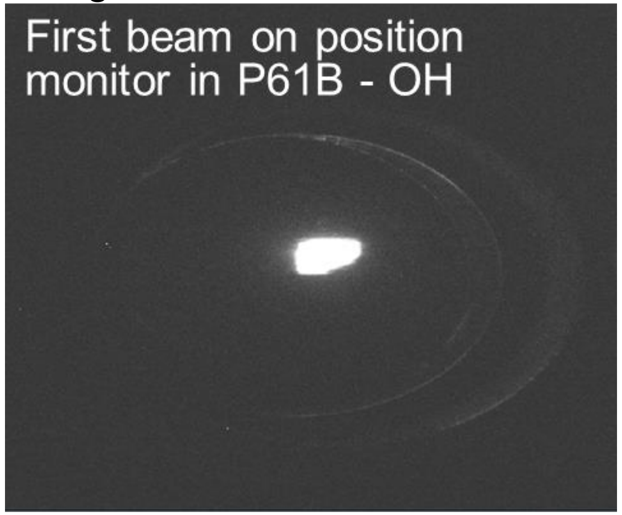
CAUTION: HOT BEAM!

Crystal growth of MoO_3 (> 600 °C)

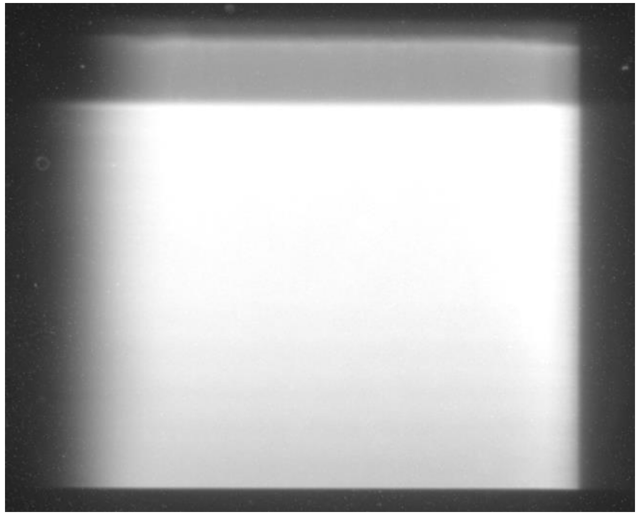


Instantly molten Au capsule (> 1064 °C)

1 August 2019

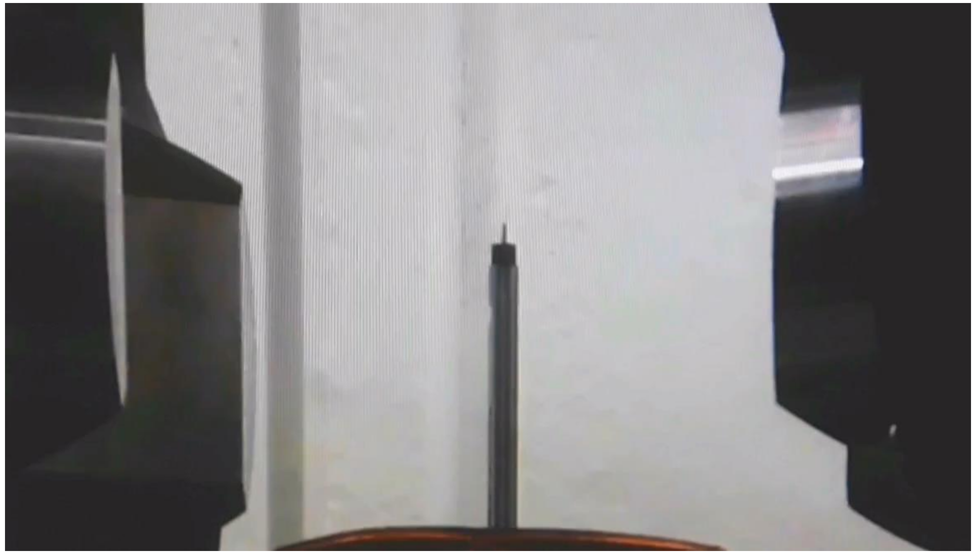


Current



1.7 mm

2 mm



Lighting a (W wire) candle using synchrotron X-rays

High-pressure techniques

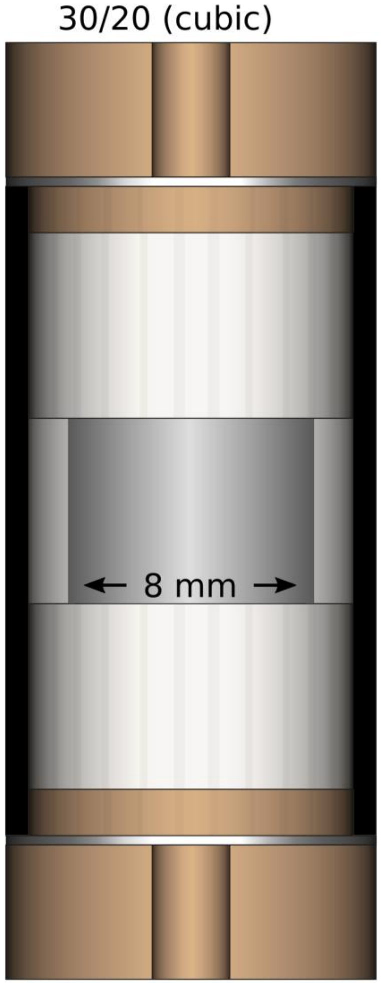
Standard assemblies / anvils for users (without resources)

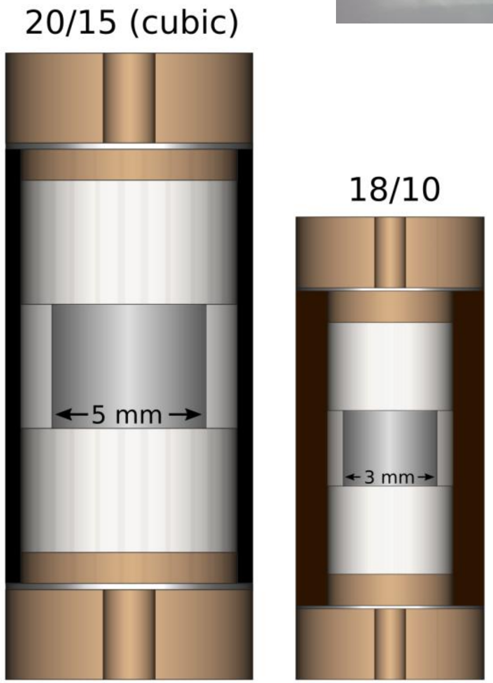
ZrO_2

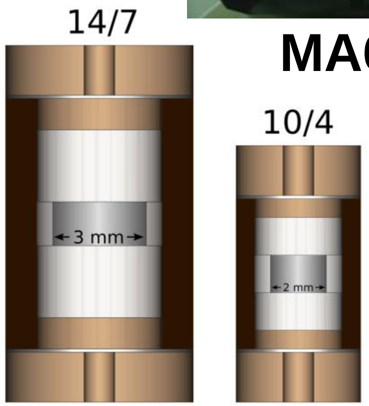
MgO

Furnace (graphite, $LaCrO_3$)

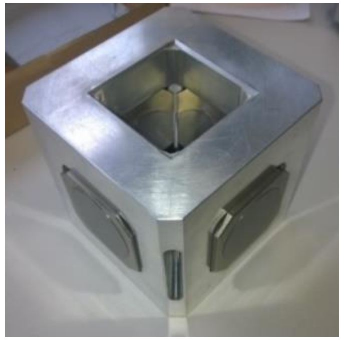
MA6-6







30/20 (cubic)	20/15 (cubic)	18/10	14/7	10/4
P_{max} = 4 GPa	P_{max} = 7 GPa	12 GPa	16 GPa	20 GPa
V ≈ 300 mm^3	V ≈ 78 mm^3	17 mm^3	10 mm^3	4 mm^3

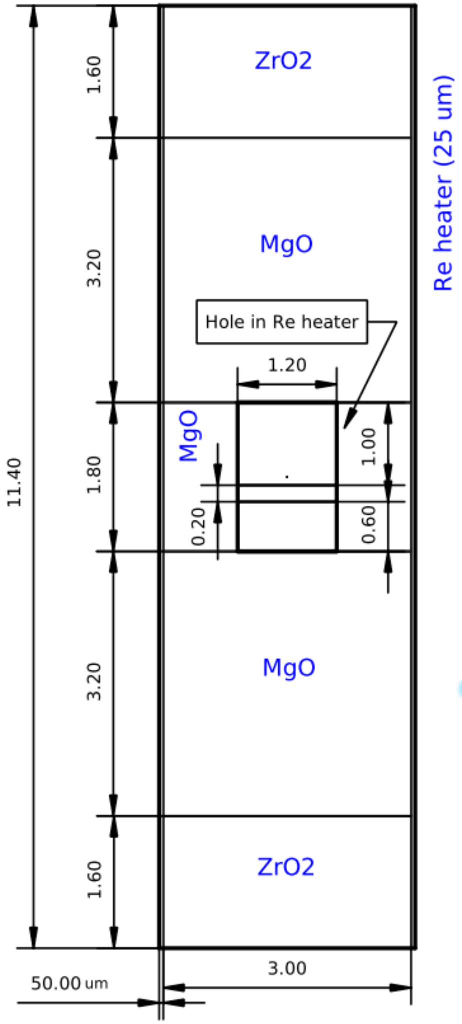


No X-rays
Maximize sample volume

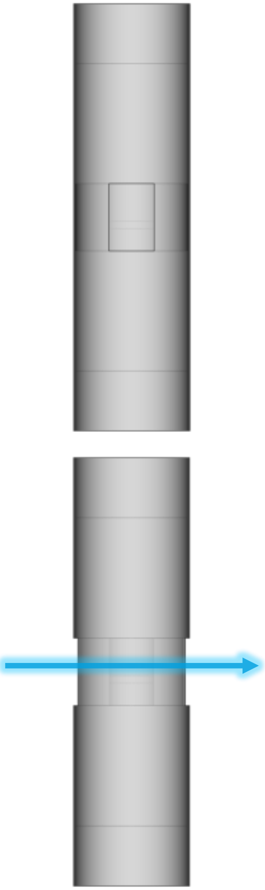
MA6-8

Original assembly design by Dr. Nishiyama

P > 10 GPa, T up to 1600 °C (for now)



Re heater



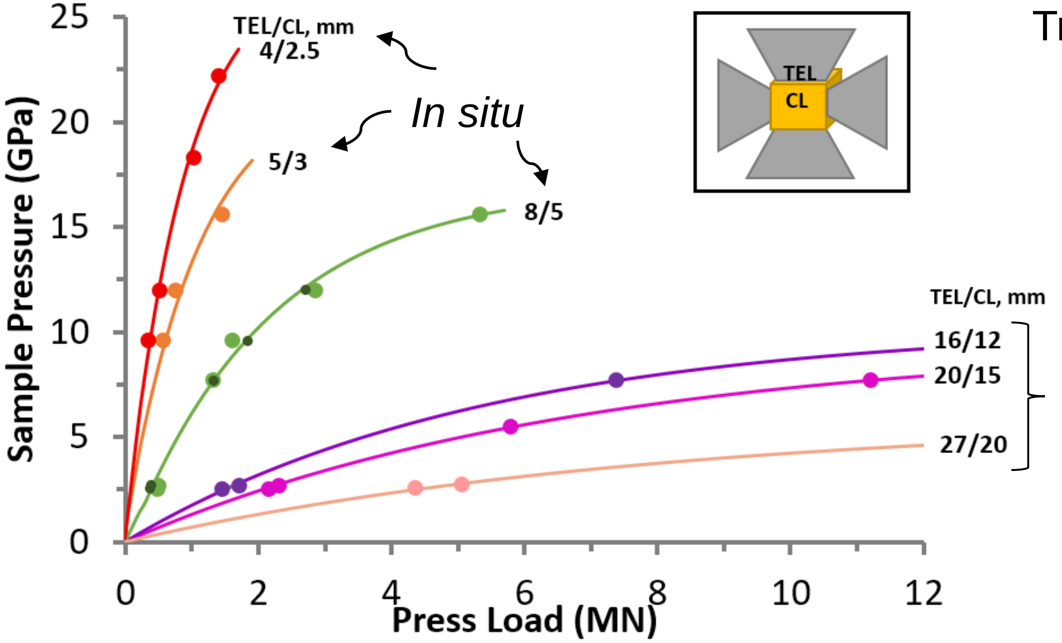
14M, 10M, & 7M

With X-rays
Optimise diffraction condition

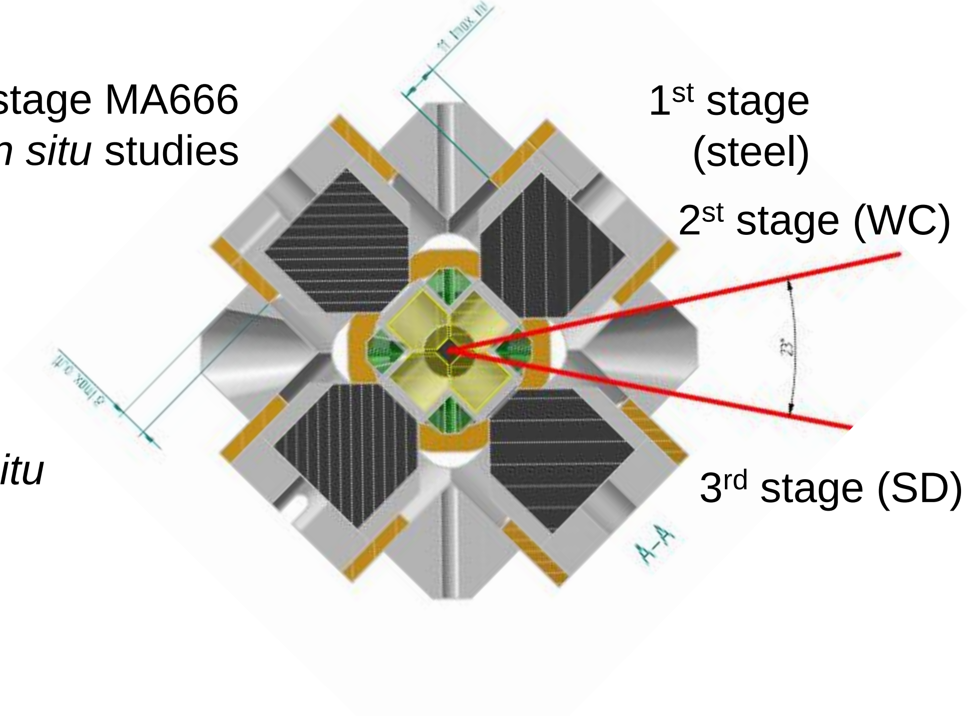
High-pressure techniques

Standard anvils for *ex situ* and *in situ* rock deformation studies

Pressure generation cubic system

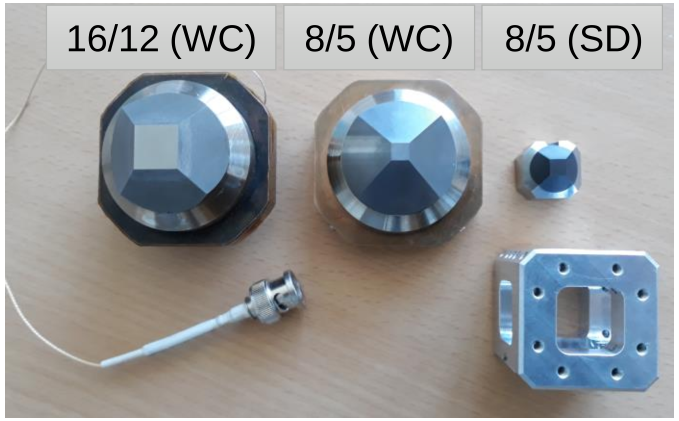


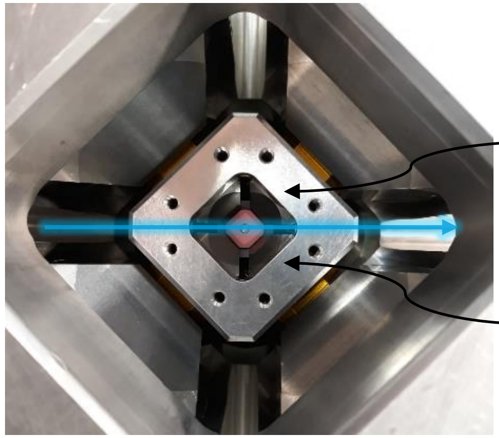
Triple stage MA666 for *in situ* studies



Sample size:

- [< 7 GPa] 10 mm to 6 mm (*ex situ, synthesis*)
- [< 18 GPa] 2 mm to 0.5 mm (*ex/in situ*, deformation)





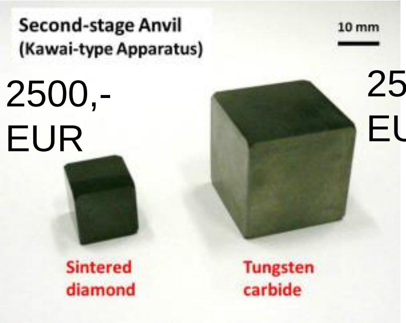
X-ray transparent, sintered diamond (SD) anvils

Ultra-high pressure techniques

Recent developments

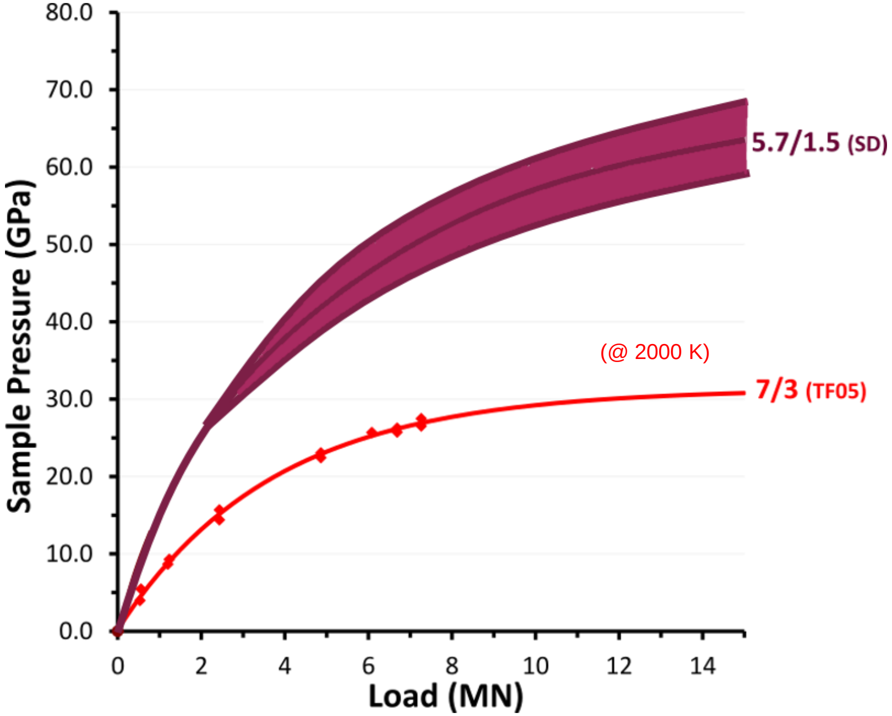
Pressure in LVP is limited by the hardness of anvil materials

- WC (tungsten carbide) – 30 GPa limit, up to 45 GPa/2000 K with special shape.
- Sintered diamond and cBN much harder and X-ray transparent!
  → Also very expensive, smaller and brittle!



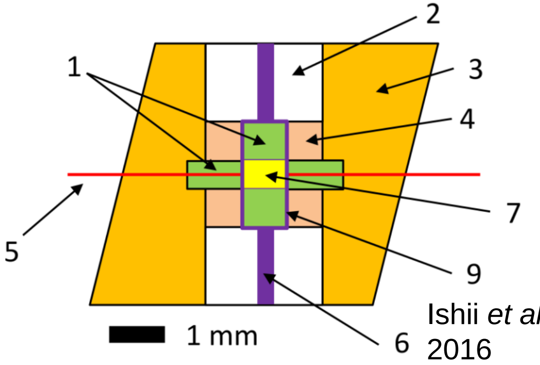
2500,- EUR

250,- EUR



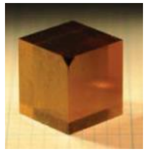
Aim: routine experiments without breakage of SD anvils after 1st run (at least up to 60 GPa)

A) 5.7/1.5 cell assembly



Ishii *et al.* 2016

1. Most reliable, generate highest pressures:
14 mm SD anvils with **cobalt** binder
2. Most transparent to X-rays, lower pressures:
14 mm SD anvils with **SiC** binder
3. Hardest/Extreme HP/most expensive:
Binderless **nano-polycrystalline diamond**

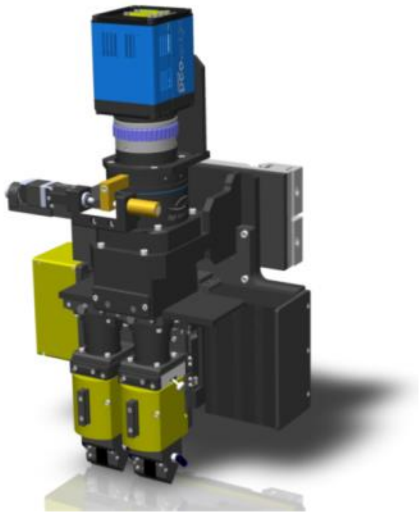


- Anvil truncation: typically 1.5 mm for UHP
- Sample size: up to 0.5 mm.

The whitebeam X-ray microscope

Optique Peter
OPTICAL & MECHANICAL ENGINEERING

pco.



The X-ray microscope is ready for use at P61B

- **Double objectives (5x, 10x)** for high resolution, full beam imaging
- **GGG: Eu scintillators:** 10, 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 or frame capture via Tango

X-ray powder diffraction using white beam

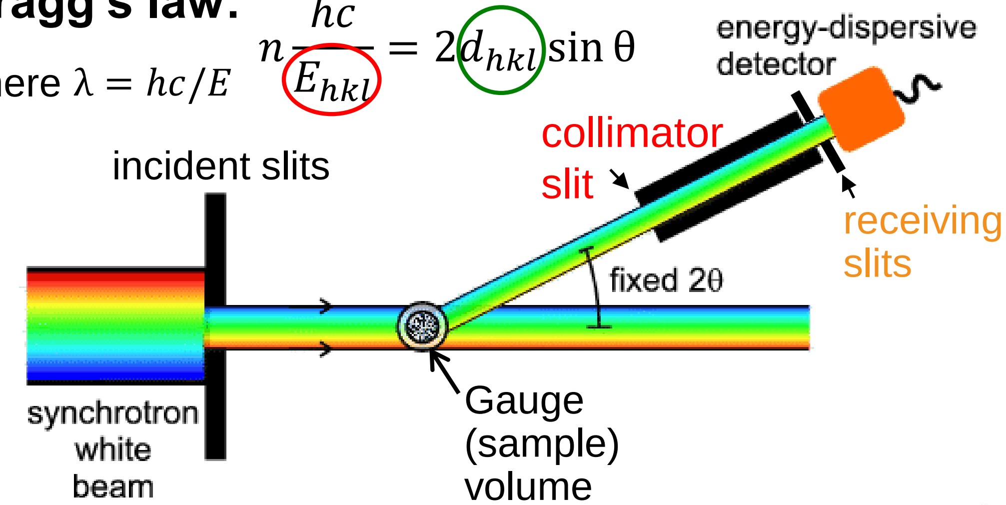
Energy-dispersive X-ray diffraction (ED-XRD) in the Large Volume Press

MIRION TECHNOLOGIES

Ge-detector (2x) – ED XRD	Mirion (Canberra)
Collimator slit (mm)	0.03, **0.05**, 0.1, 0.2
Receiving slits (mm)	0.05, 0.1, 0.2, 0.5, 1.0, 2.0
Horz. detector positions	1xGe: min 4° - max 20° 2xGe: min 5° - max 10°
Horz. & vert. positions	Ge_{vert}: min 7.5° - max 23° Ge_{horz}: min 6.5° - max 10°

Bragg's law: $n\frac{hc}{E_{hkl}} = 2d_{hkl}\sin\theta$

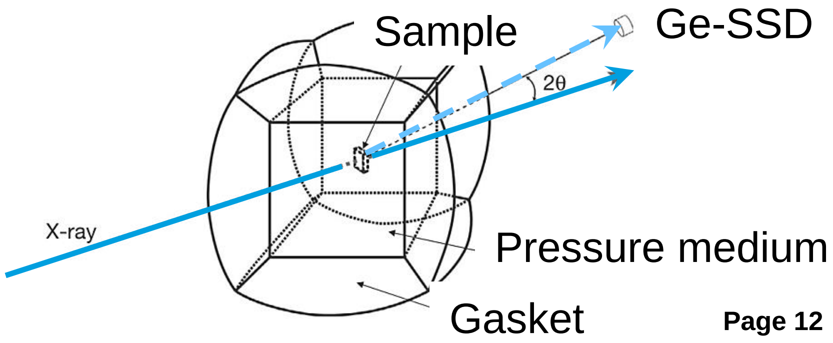
where $\lambda = hc/E$



http://pd.chem.ucl.ac.uk/

Why ED-XRD with LVP?

1. High spatial resolution (small gauge volume)
   → avoid high temperature/pressure gradients
   → multiple samples in one experiment
2. Low absorption
   → useful for low-Z materials
3. No scanning, information in wide range of d-spacing collected at once
   → great for reaction kinetics experiments

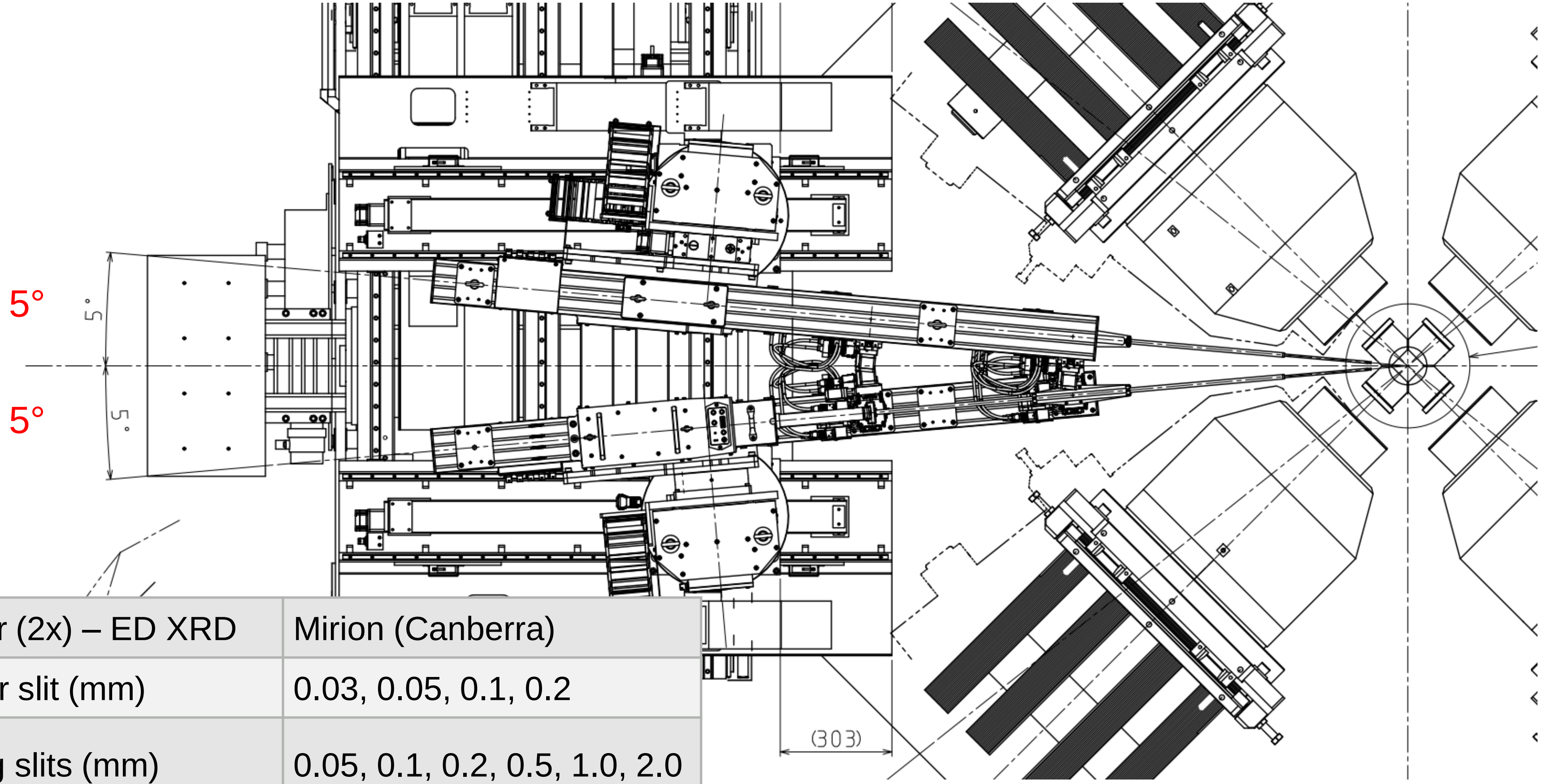


X-ray powder diffraction using white beam

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

MIRION TECHNOLOGIES

The position of both detectors in the horizontal plane limits the smallest angle to 5° each, and largest angle to 10 ° each.



Ge-detector (2x) – ED XRD	Mirion (Canberra)
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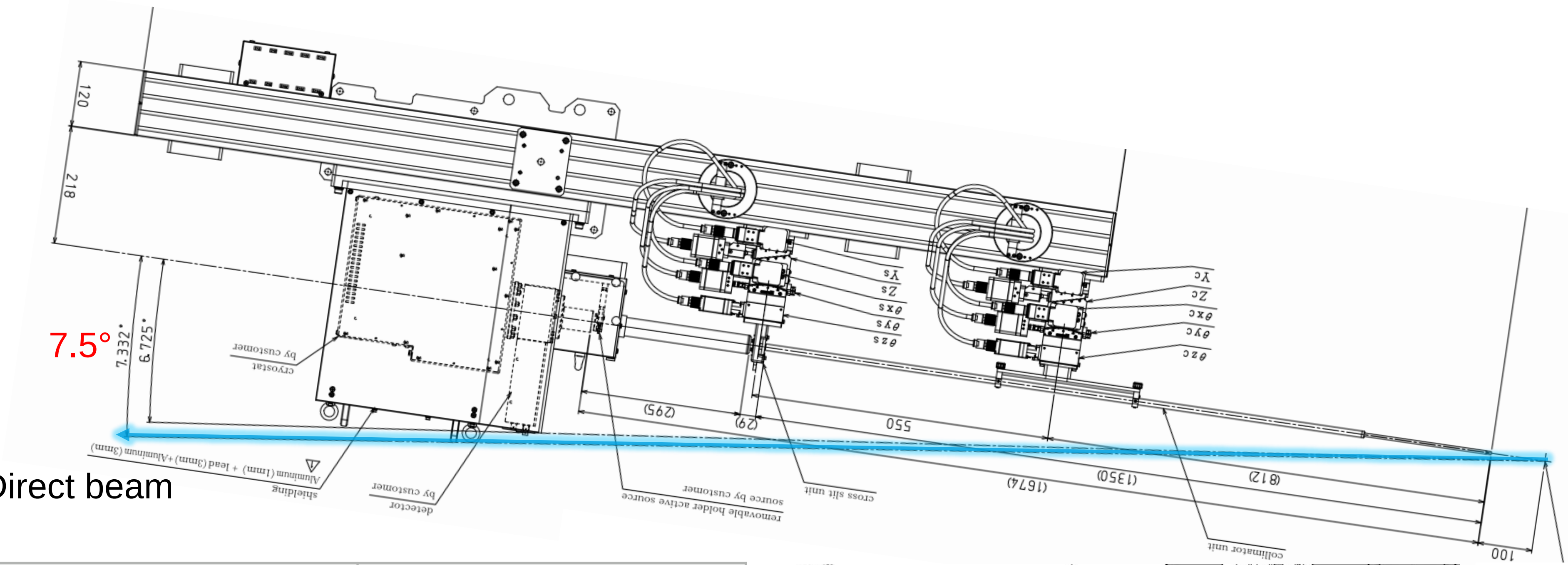
X-ray powder diffraction using white beam

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

MIRION TECHNOLOGIES

The position of the vertical detector disallows XRD at angles less than ~7.5 °



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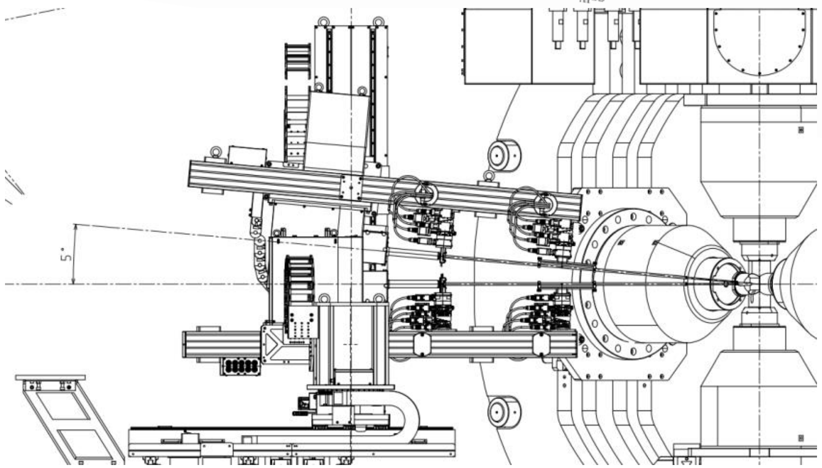


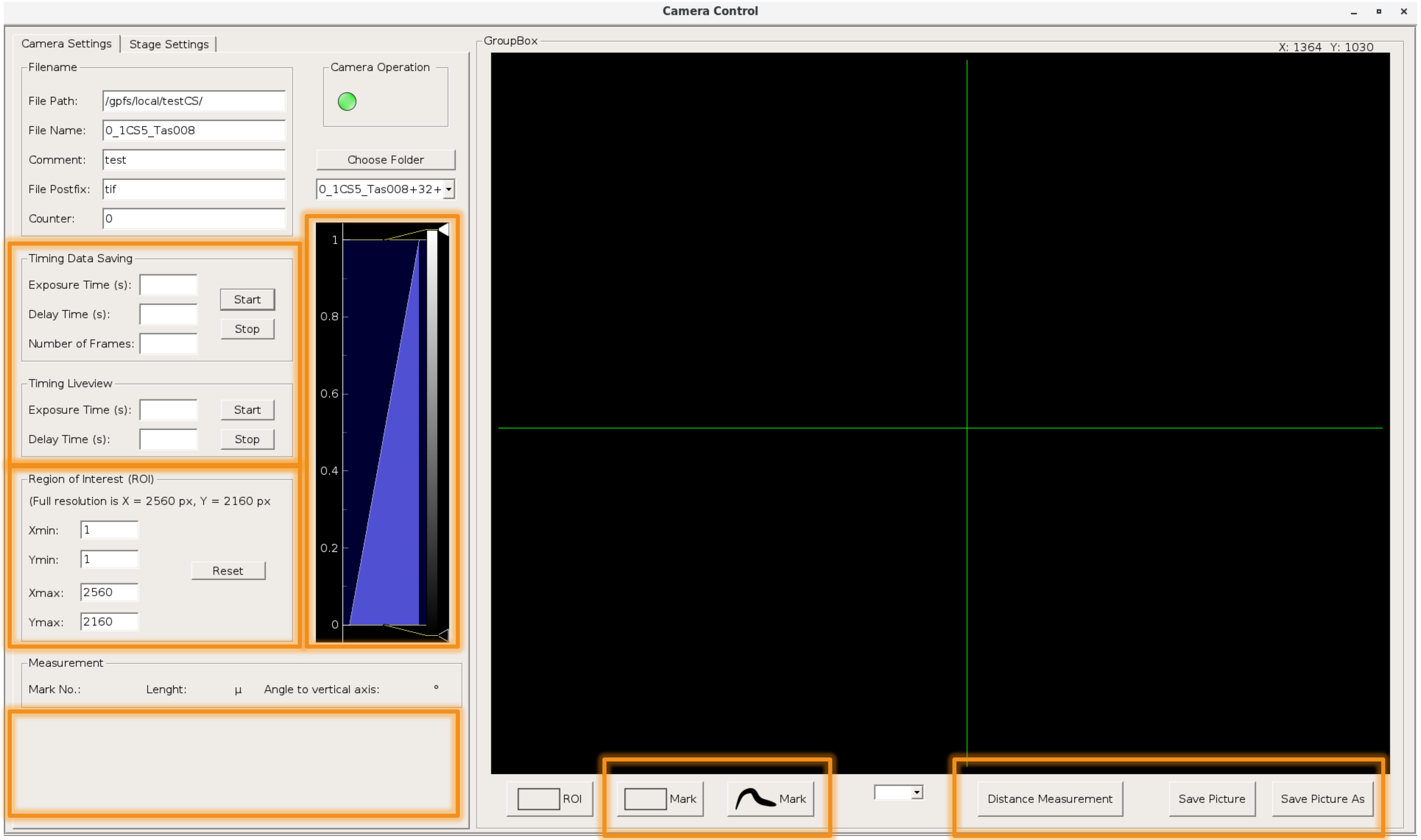
Software development

Data acquisition and control systems

Credit: Stefan Sonntag

Interface for Radiography (absorption contrast)

- Mark spot on sample for ED XRD pencil beam.
- Adjust slider to increase contrast.
- Switch between acquisition and live mode easily.
- Select ROI for fast frame rates.
- (pending) acquisition during z stage scan and montage.



Software development

Data acquisition and control systems

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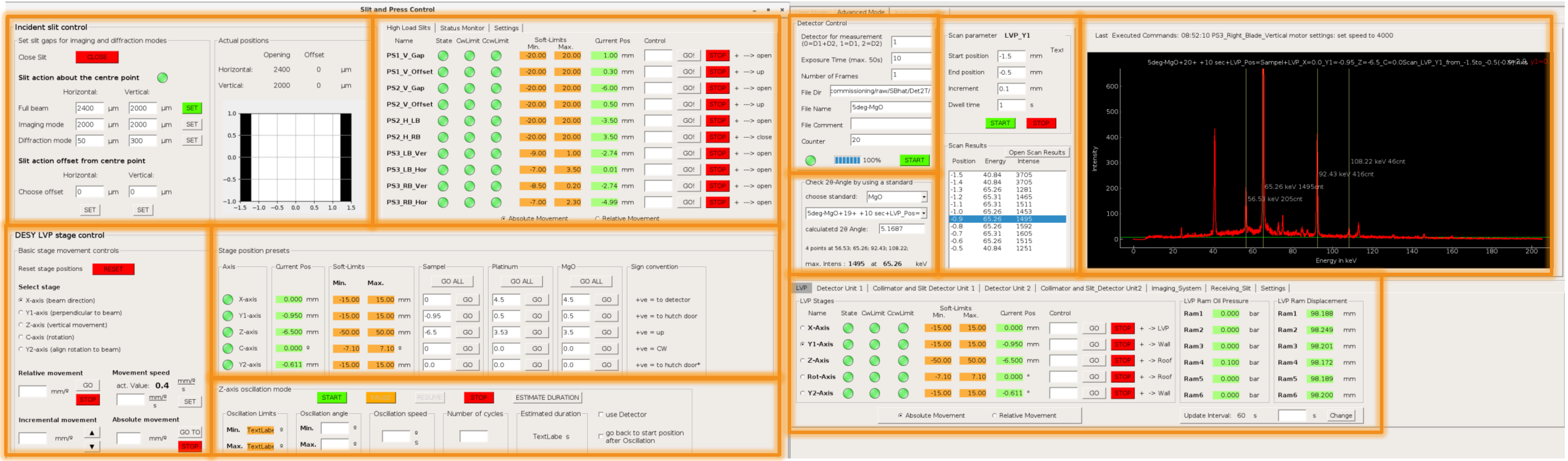


Interface for slits and LVP stage movements

- Quickly switch beam size for imaging and XRD.
- Adjust stage movement by axis selection or…
- …from stage presets on sample(s) and P-marker
- Use LVP stage rotation to improve XRD patterns
- Information area for stage/slit positions and beam status

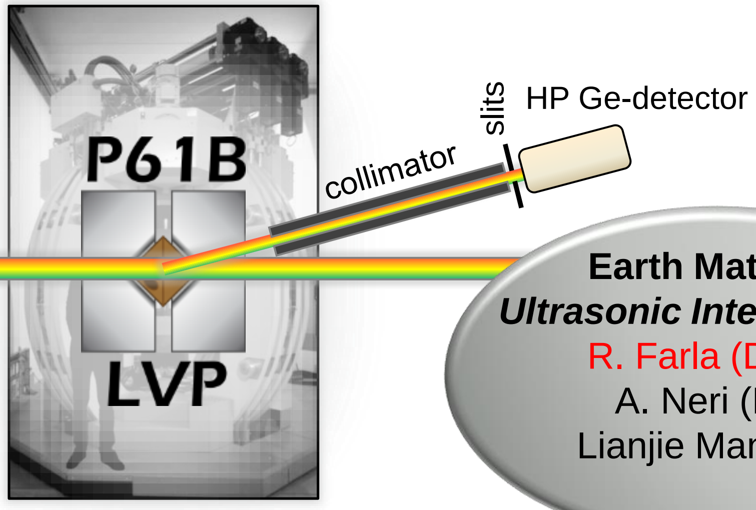
Interface for XRD acquisition and detector stages

- Easily acquire/visualize data using 1 or 2 Ge-detectors
- Automatic 2θ angle calculation on P standards
- LVP stage scanning using Ge-detector (to find sample)
- Tabbed interface for LVP stage, detector stage, and microscope stage movements, *etc*.

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



Earth Materials
UHP Silicates
A. Chanyshev (BGI/DESY)
T. Katsura (BGI)

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

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

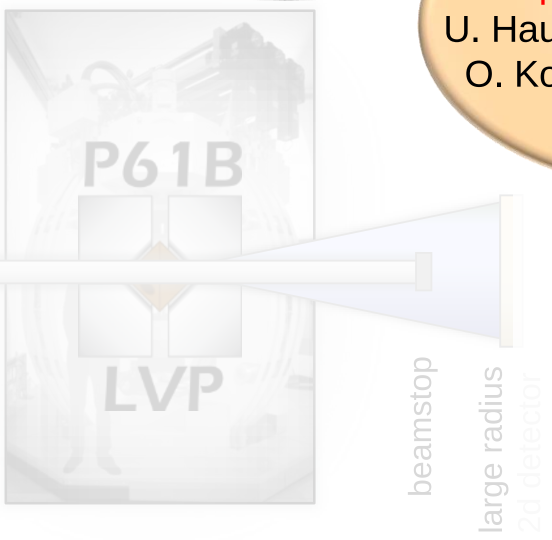
CMWS
Hydrous phases
C. Lathe (DESY/GFZ)
M. Koch Müller (GFZ)
M. Sieber (Potsdam/GFZ)

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

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

Unfocused monochromatic X-rays for AD-XRD

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



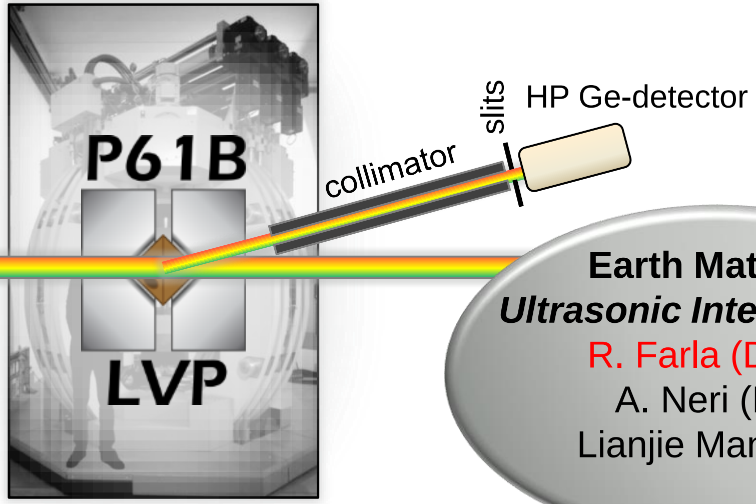
Earth Materials
Rock Mechanics
R. Farla (DESY)
A. Chanyshev (BGI/DESY)
N. Hilairet (Lille Uni)

Development of BCN X-ray transparent 'windows'

P61B LVP Research & Development

Unfocused white X-rays
for ED-XRD

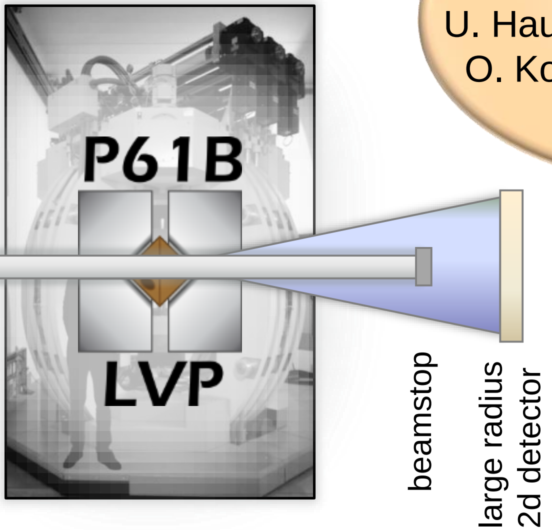
2 mm (h) x 1.7 mm (v)
to 0.03 mm x 0.03 mm



Development is delayed – 1st tests in 2021-I

Unfocused monochromatic X-rays for AD-XRD

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



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T. Katsura (BGI)

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

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.)

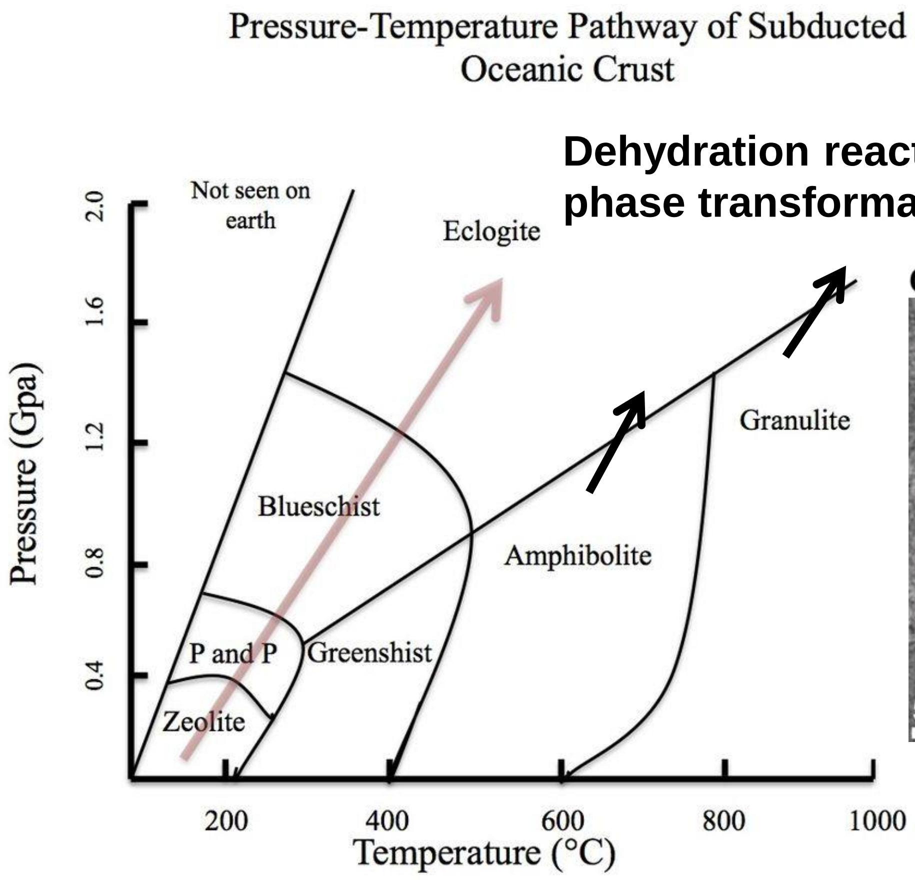
Earth Materials
Acoustic Emissions
S. Ma (Jilin Uni, China/DESY)
J. Gasc (Uni Montpellier)
S. Incel (Bochum)

Earth Materials
Rock Mechanics
R. Farla (DESY)
A. Chanyshev (BGI/DESY)
N. Hilairet (Lille Uni)

Development of BCN X-ray transparent ‘windows’

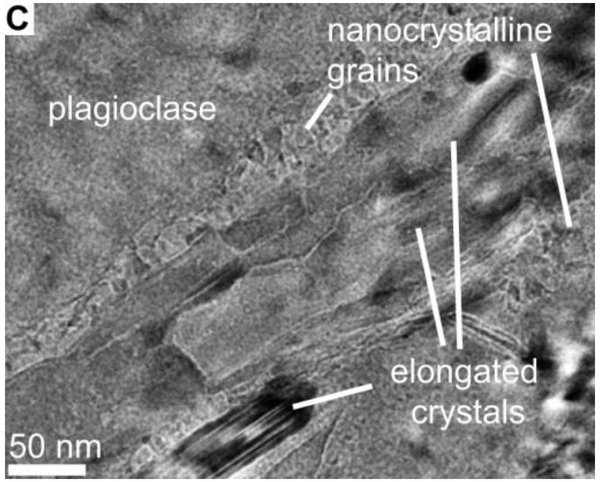
1. Acoustic Emissions testing (*in situ*)

Motivation: Dehydration-induced embrittlement → earthquake !?



Dehydration reactions from phase transformations

https://upload.wikimedia.org/wikipedia/en/d/d1/Metamorphic_pathway_of_pressure-temperature_conditions_in_subduction_zones.jpg

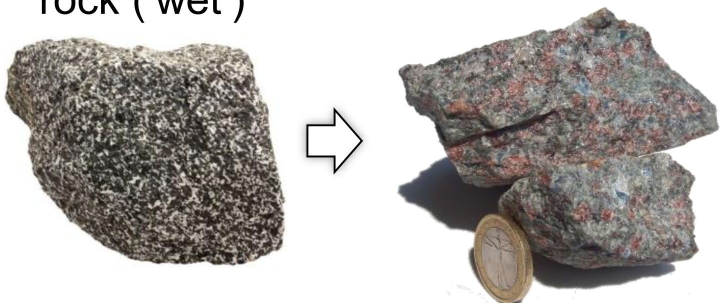


Incel, S., et al., 2019. *Geology*, **47:**235–238

- Fluid-induced plagioclase breakdown under eclogite-facies conditions is exothermic and produces a negative volume change.

Amphibolite rock (‘wet’)

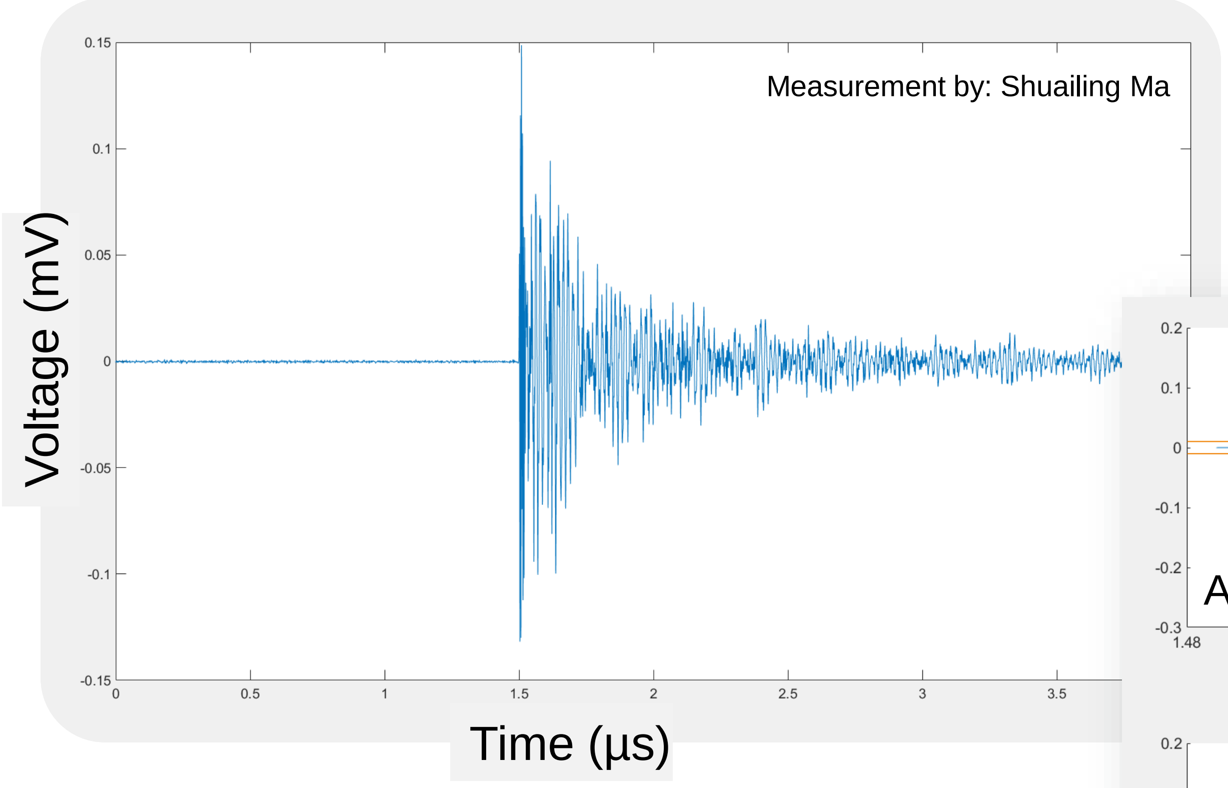
Eclogite rock (‘dry’)



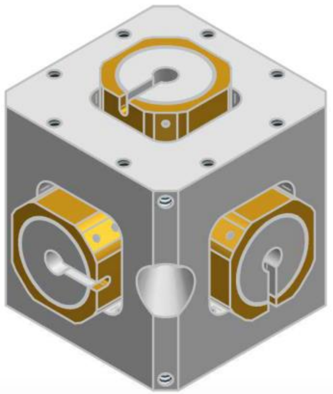
- **P too high, ductile**
- **T too low, no reaction**
- **T too high, reaction too fast**
- **P-T-$\dot{\varepsilon}$ just right, embrittlement and AE**

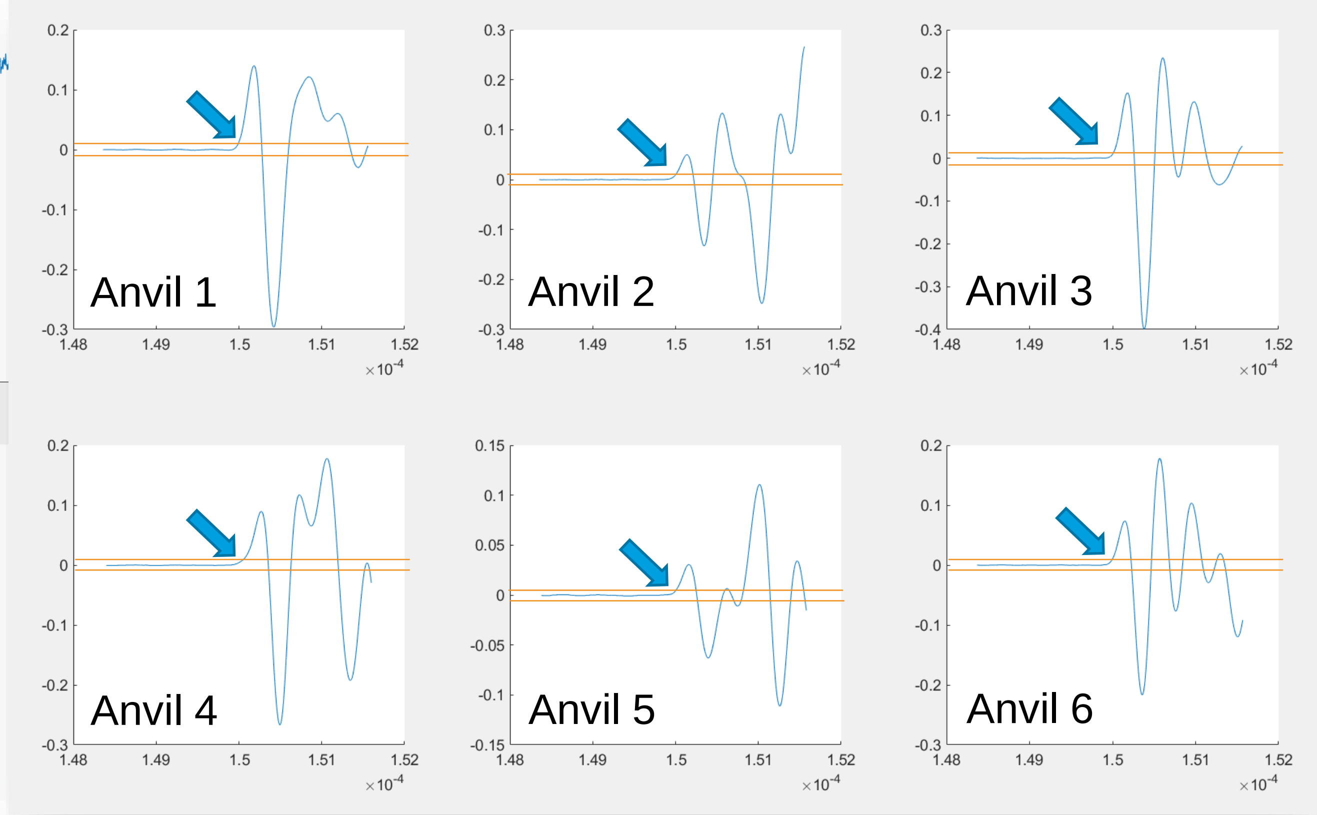
1. Acoustic Emissions testing (*in situ*)

Commissioning progress



Waveform of a typical 'hit' measured by a sensor on the back of an anvil. Excellent signal to noise level is useful for investigations into focal mechanisms & radiated energy.



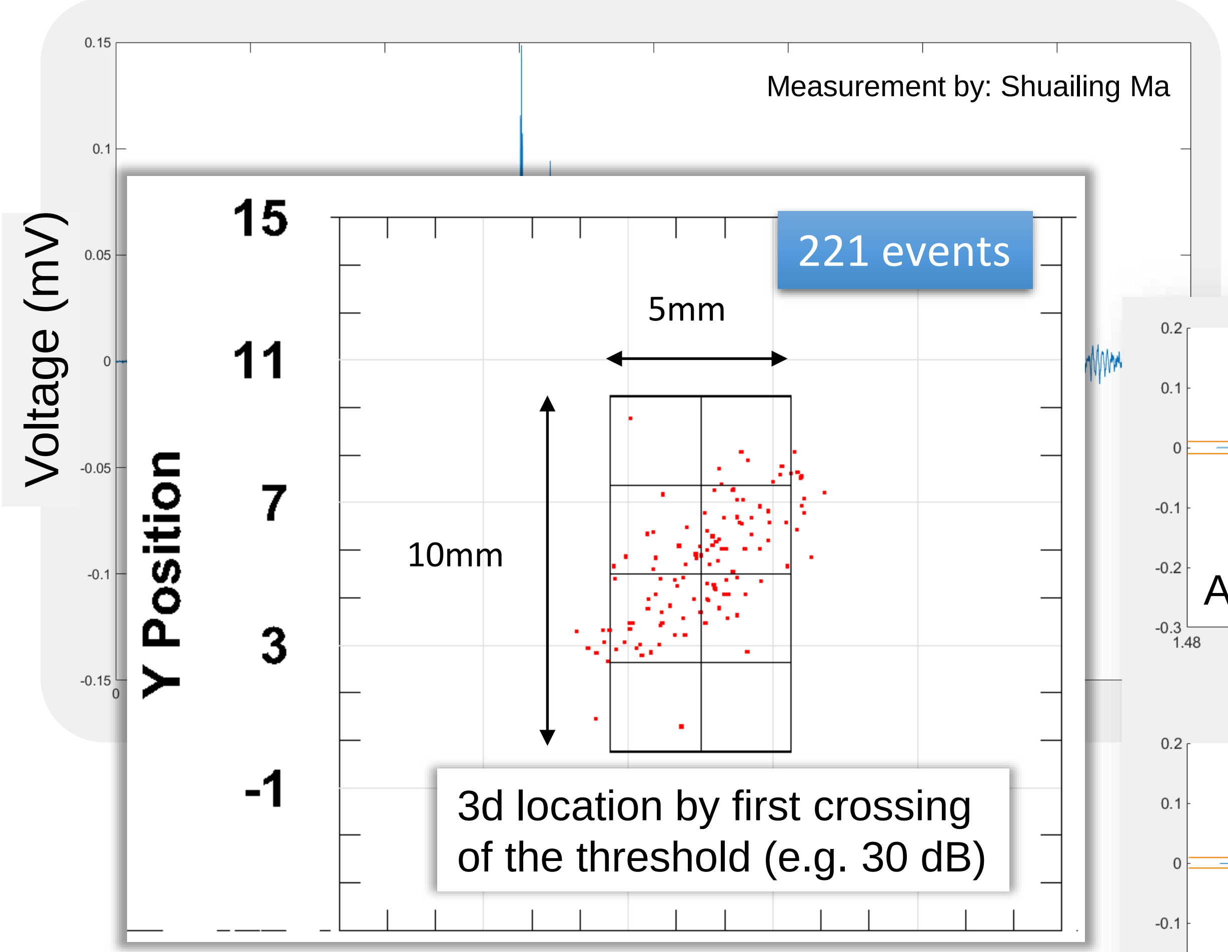


Magnified portion of an 'event', a measurement of nearly-simultaneous hits on each of 6 anvils.

Matlab data processing enabled by Dr. Julien Gasc (Montpellier)

1. Acoustic Emissions testing (*in situ*)

Commissioning progress



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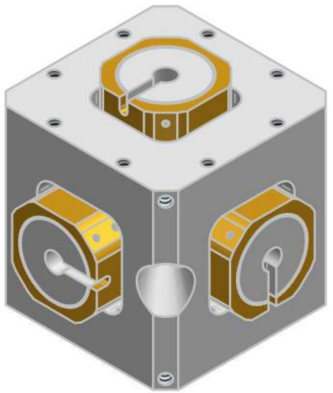
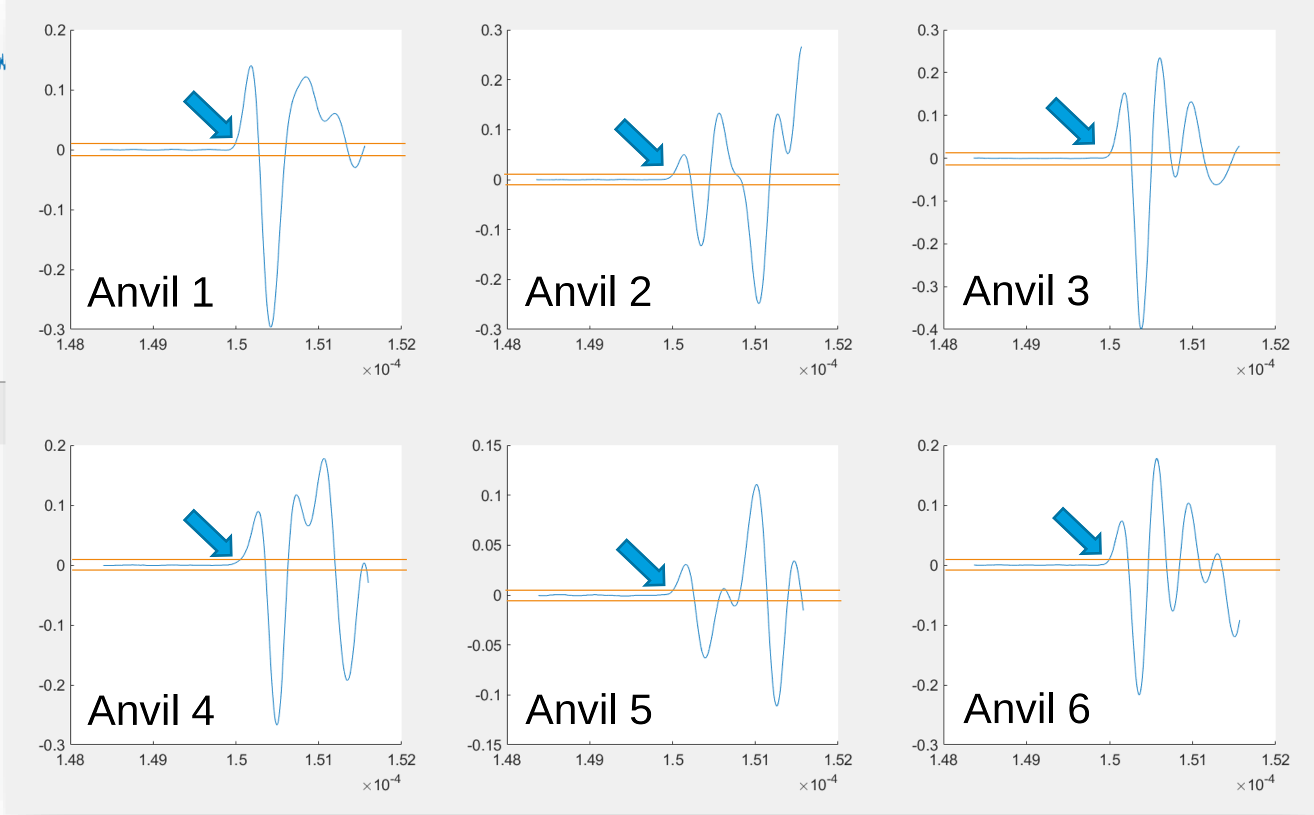
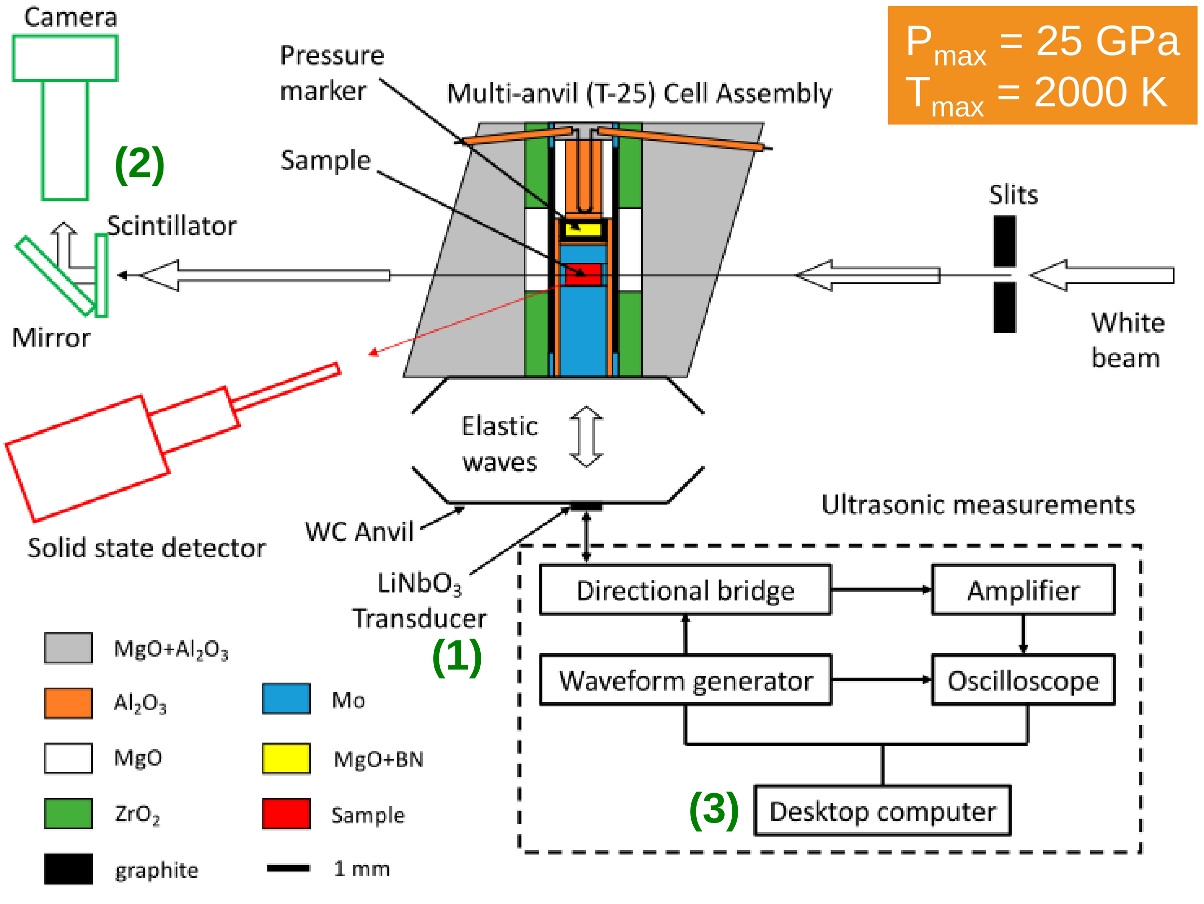




Matlab data processing enabled by Dr. Julien Gasc (Montpellier)

2. Wave speed measurements (*in situ*)

Ultrasonic Interferometry: Equipment and planning



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

General method

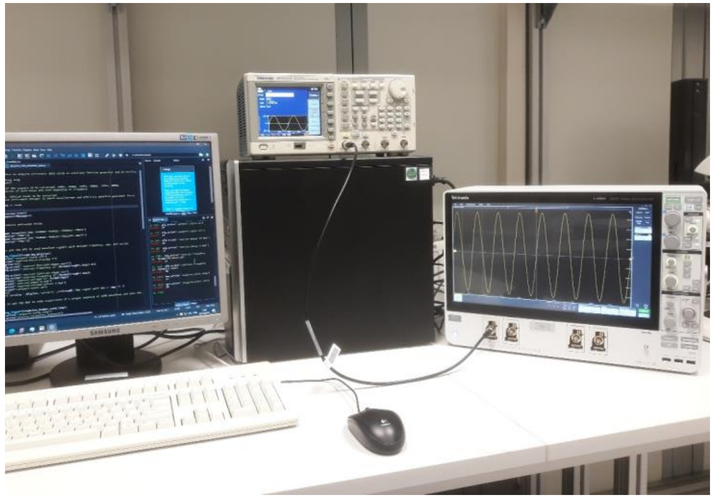
(1) A LiNbO3 sensor of choice on the back of a mirror polished anvil, transmits a pulse and receives an echo.

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

(3) Wave speed at given P,T is calculated for determination of elastic moduli (with density information).

→ Simultaneous measurement Of elastic P and S wave travel times, density, and sample length.

→ Measurement routine can be scripted using python.



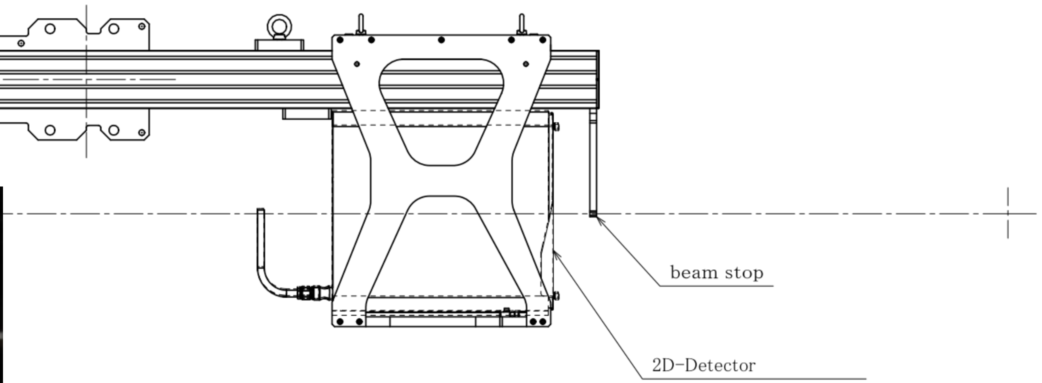
3. Rock deformation (*in situ*)

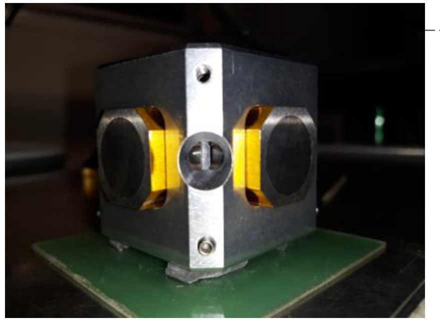
Understanding the mechanical properties of materials at high P-T

Experiment parameters:

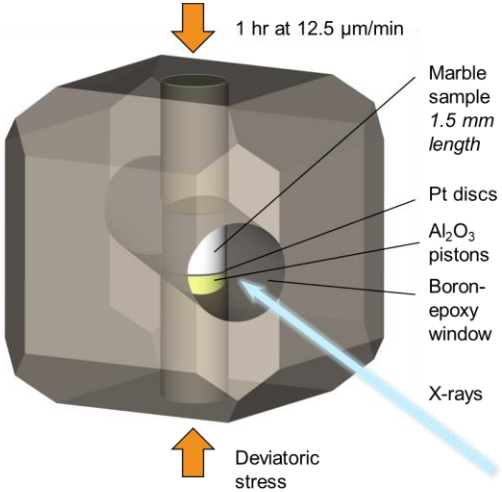
- P_{max} ≈ 10 GPa (8/5 assy)
  P_{max} ≈ 18 GPa (5/2.5 assy)
- T_{max} = 2000 K
- $Strain_{max}$ ≈ 30%
  (in axial symmetric compression)
- Detector requirement:
  - 2 Ge-detectors for ED-XRD or….
  - Integrating area detector for AD-XRD
    (Varex 4343 CT)

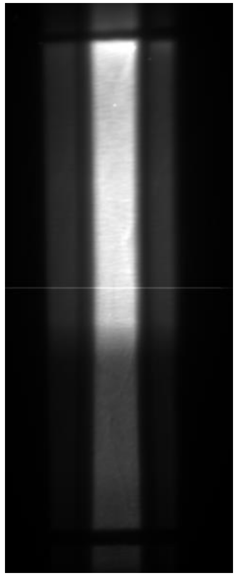
Replacement Unit for the
Detector Positioning System (DPS)



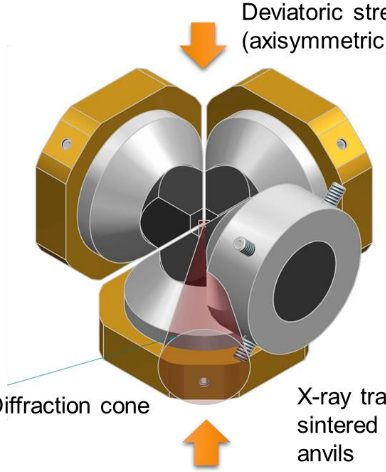


Radiography - Strain

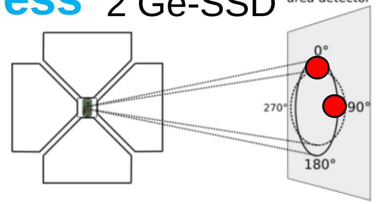


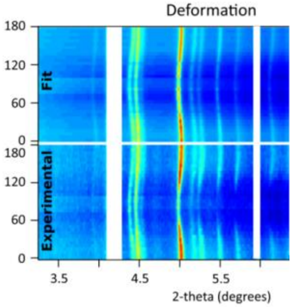


ED & AD XRD - Stress



2 Ge-SSD





Future outlook

Addition of a glovebox

Just delivered,
financed via RAC/BMBF
by Prof. Häussermann
& Kohlmann

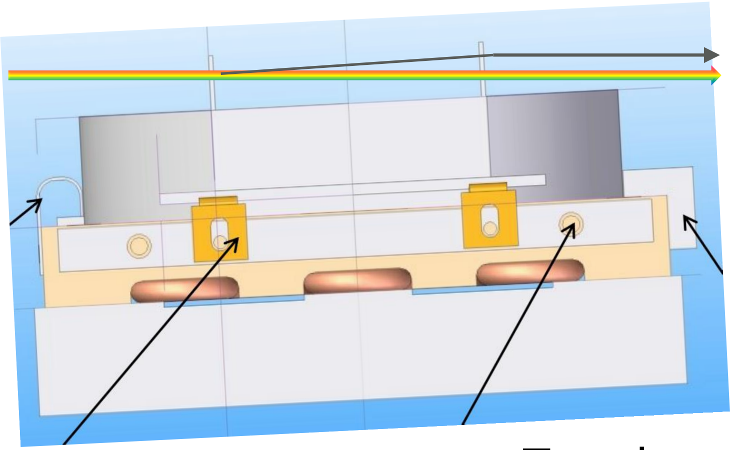
Future outlook

Addition of a monochromator

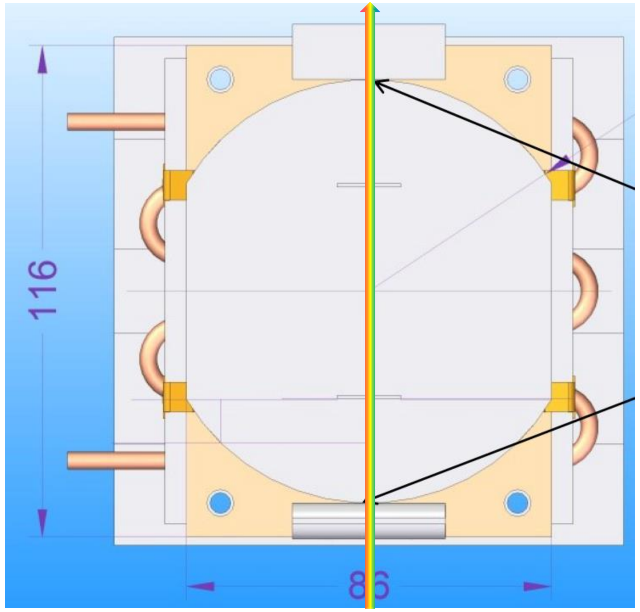
Possible design for Si111 Laue monochromator

- Energy range: **65 - 110 keV**
- Beam offset: **1.3 mm – 2 mm**

2 lamellae (35 mm separation)



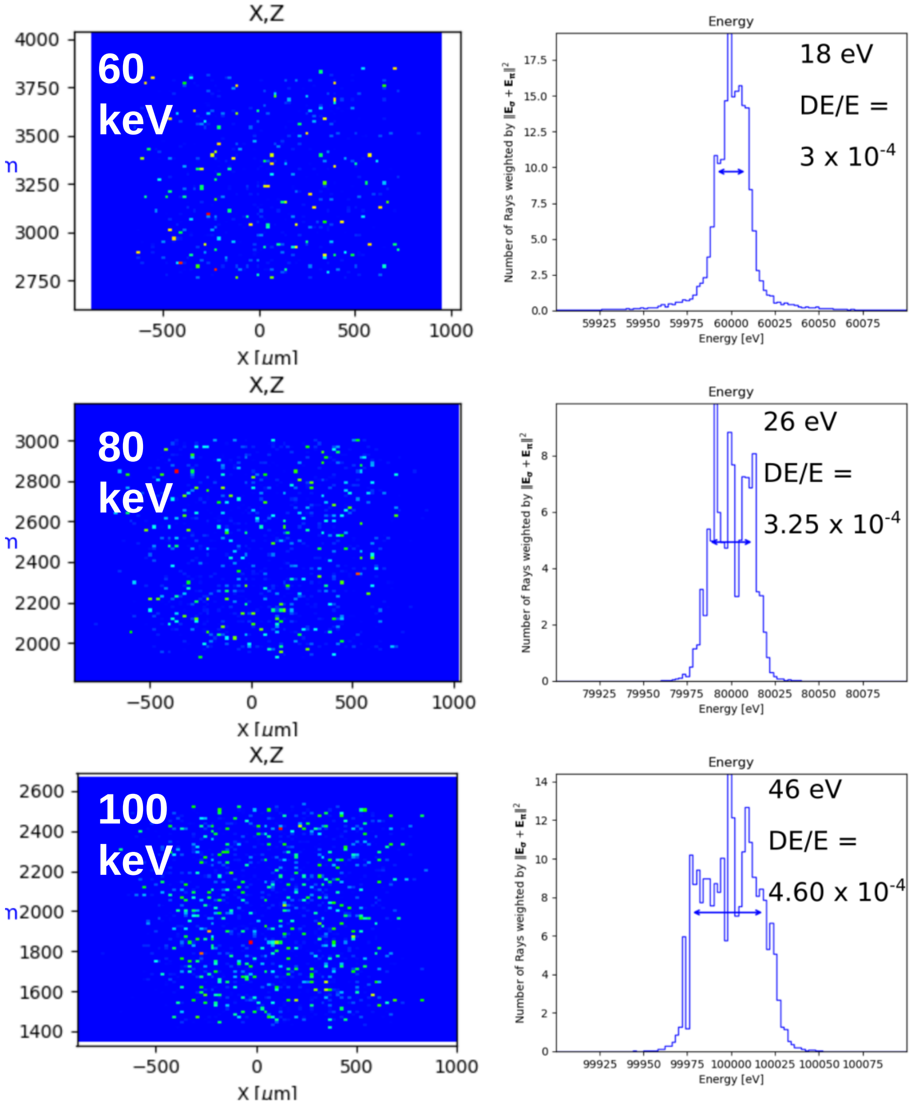
Top view



Side view

Credit: Stefan Sonntag

Beam size after 2nd lamellae | Energy resolution | Flux throughput



1.47 %
1.15×10^{10}
ph/s/0.1% b.w.

1.46 %
8.82×10^{9}
ph/s/0.1% b.w.

2.62 %
1.12×10^{10}
ph/s/0.1% b.w.

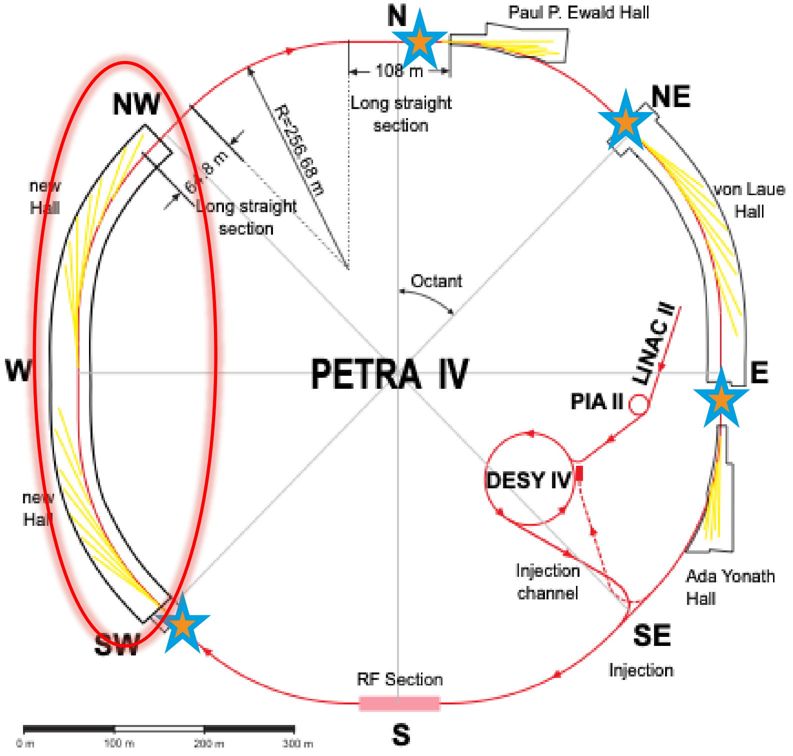
Outlook of PETRA IV.

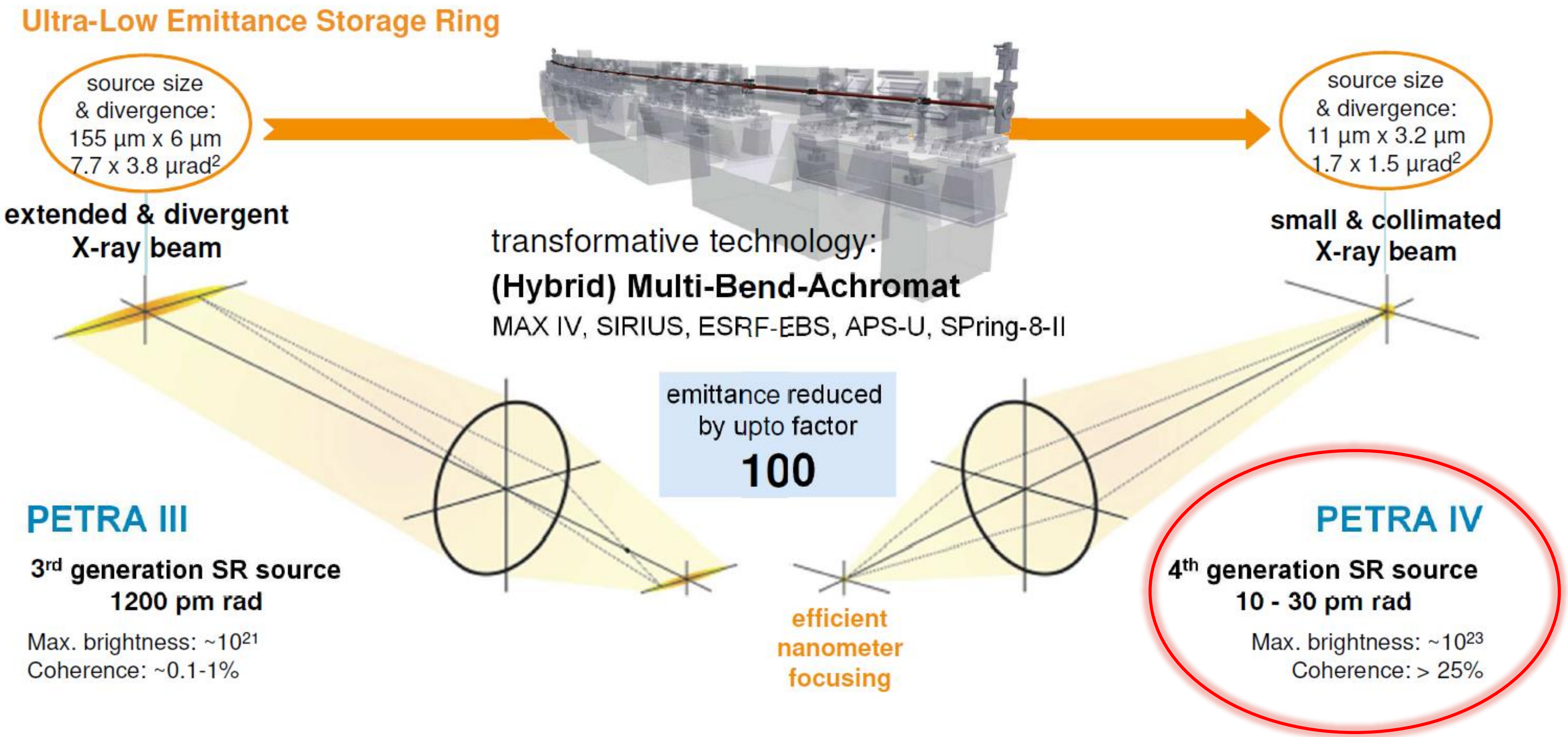
Conceptual Design of Storage Ring and Accelerator Complex

PETRA IV.

Design lattice:
Hybrid 7 Bend Achromat (H7BA)
adopted from ESRF-EBS

> **On-Axis Injection** using fast kickers
> Optimized **insertion devices** in long straight sections

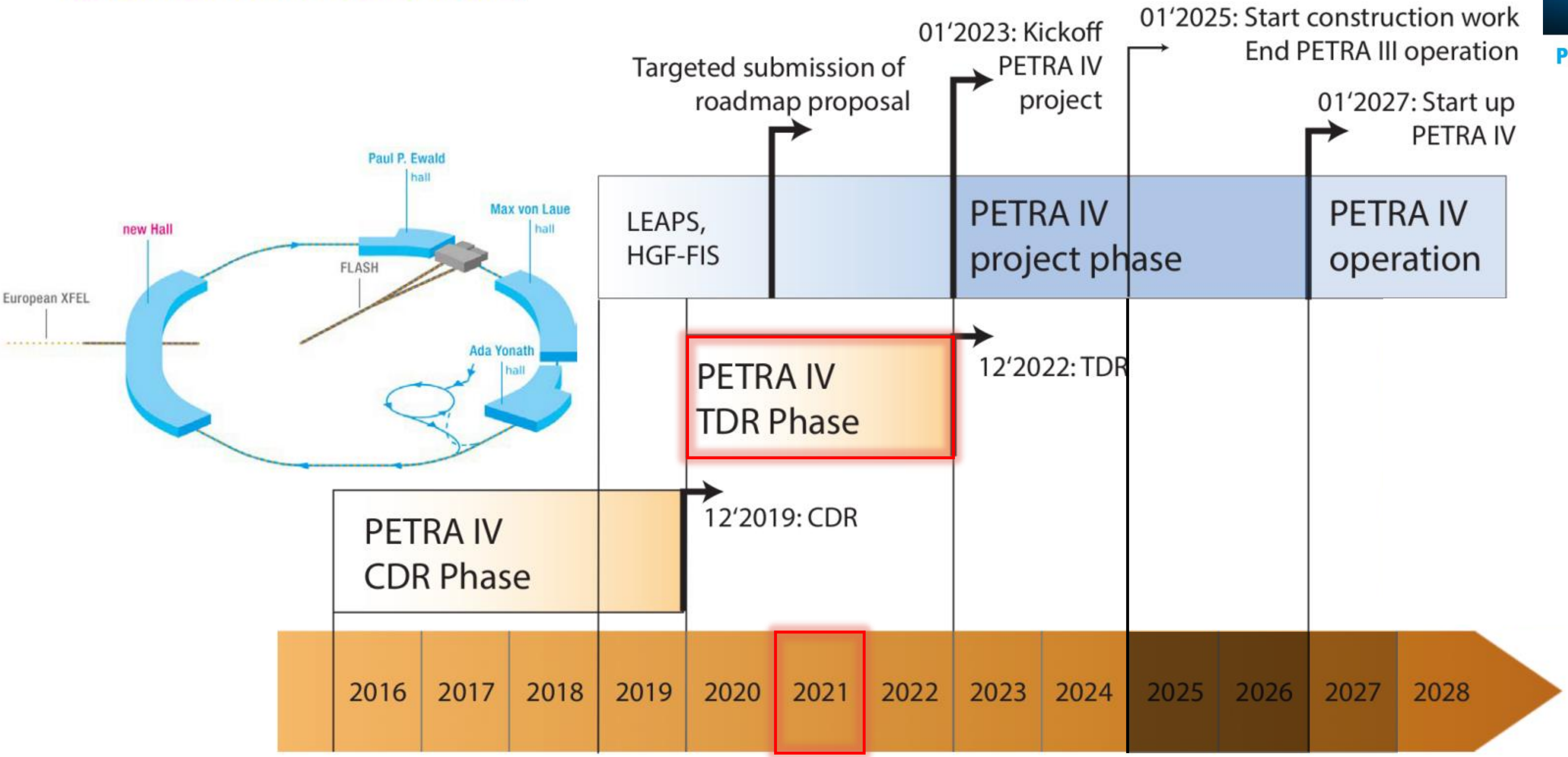




Outlook of PETRA IV.

Task ahead: From Idea to Reality

PETRA IV.



Outlook of PETRA IV.

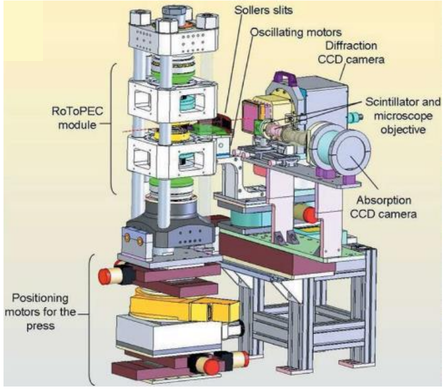
Evaluation of Scientific Instruments Proposals (SIPs)

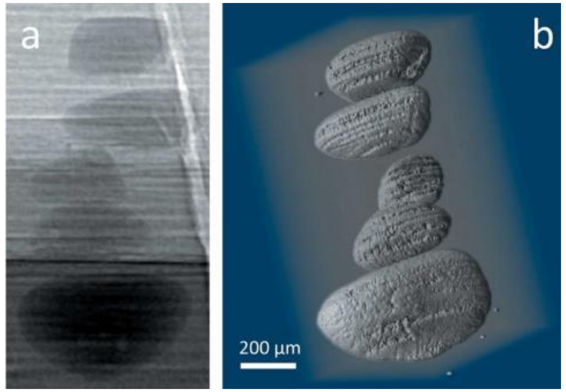
PETRA IV.

5 Proposals for LVP instrumentation submitted:

1. *In-situ* XRD & imaging at high pressure and temperature using the **6-ram LVP** at PETRA IV (PI: Dr. Sieber *et al.*)
2. Synthesis and characterization of novel materials by combination of **the large volume press** and high-density X-ray beams (PI: Prof. Katsura)
3. High-pressure-temperature deformation experiment using X-ray stress analysis and **6-ram LVP** (PI: Prof. Katsura)
4. Reliable investigation of **[ultra]** high P-T phase transitions by combination of *in situ* X-ray diffraction and advanced multi-anvil technique [**Uniaxial DIA-type press**] (PI: Prof. Katsura)
5. Dedicated LVPs for time-resolved, high-resolution, 3D, X-ray Imaging under Extreme Conditions at PETRA IV [using a **PE-type press**]. (PI: Dr. Sieber *et al.*)

RoToPEc





ESRF data:
(a) Raw X-ray radiograph of 5 glass blobs in hBN pressure medium
(b) 3D rendered volume of µCT scan at 0.9 GPa 300 K.
J. Philippe, et al. 2016 / Alvarez-Murga et al. 2017

New LVP@PETRA IV objectives:

- **Synchrotron X-ray absorption/phase contrast (micro-) tomography.**
- **Diffraction/scattering computed tomography, DSCT** (e.g. 3x3 µm^2 focused beam).
- **AD-XRD for powder & single crystal.**
- **Near-field and/or far-field High-Energy Diffraction Microscopy (HEDM).**

Next steps

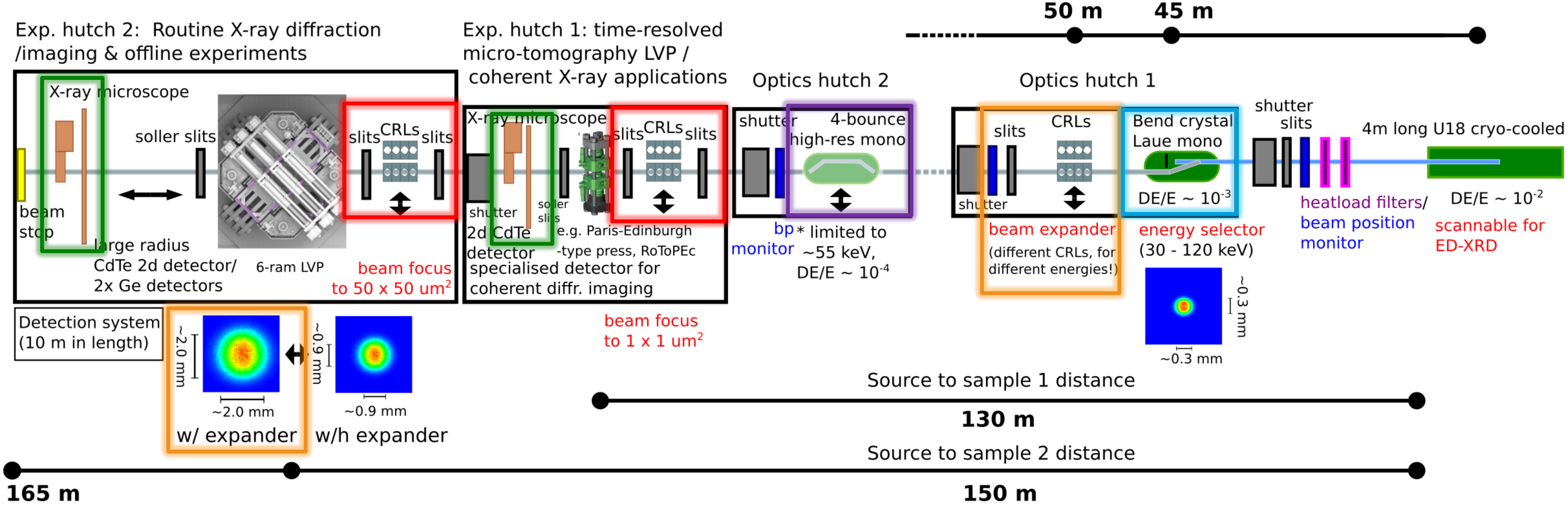
- ✓ The expert members have received the allocated SIPs and details of the Scientific Evaluation Process.
- ✓ External review now finished (1 Feb 2021).
- ❑ Internal review and concept beamline proposals to be prepared (due 1 March 2021).
- ❑ Feedback loop
- ❑ 08 Oct: FS committee submits final beamline recommendation to Directorate.
- ❑ Approved before end of 2021.

Outlook of PETRA IV.

6-ram LVP (and other LVP) at a PETRA IV beamline

- High-flux, high-energy (monochromatic) X-rays: **selectable energy (30 – 120 keV)**
- Fast detection, high pixel resolution**: state-of-the-art CdTe detector(s)**
- Option for **scanning of harmonics** to perform **ED-XRD**
- Ample space for complementary *in situ* sample environments / measurement systems

Concept LVP beamline for PETRA IV



Summary:

Beamline collaboration partners

- UHP geo-research (BGI, Bayreuth)
- Ternary hydrides (Stockholm/Leipzig Uni)
- Water research in CMWS (GFZ/Potsdam)
- 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 providing excellent XRD data quality, high count rate (200+ kcps), low acquisition times.
- Development of user-friendly GUIs.

Upcoming

- Commissioning of 2 Ge-detector positioning system (March 2021).
- New experiments possible! Incl. rock deformation
- **Submit proposals today for 2021-II period (Show me a draft! Deadline 1 March)**

Thank you for your attention!

Special interest!
Looking for expertise in search for candidate materials for quantum computing via HP route.

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

Contact

DESY. Deutsches Elektronen-Synchrotron

www.desy.de

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