

Status & development of P61B LVP at PETRA III, DESY



Beamline satellite workshop

26 Jan 2023

Robert Farla

Collaborators and support:

Shrikant Bhat, Stefan Sonntag, Artem Chanyshv (BGI), Shuailing Ma (Ningbo), Christian Lathe (GFZ), Kristina Spektor (Leipzig), Adrien Néri (BGI), Lianjie Man (BGI), Tomoo Katsura (BGI), Ulrich Häussermann (Stockholm), Holger Kohlmann (Leipzig)

Many thanks to all DESY Support Groups (FS-TI, -EC, -BT, Machine...)

HELMHOLTZ RESEARCH FOR
GRAND CHALLENGES



UNIVERSITÄT
LEIPZIG



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

Review of Scientific Instruments

<https://doi.org/10.1038/s41586-020-2020-2>

Received: 26 November 2020

Accepted: 18 October 2021

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Open access

Contributions to Mineralogy and

<https://doi.org/10.1007/s00410-021-00410-0>

ORIGINAL PAPER

Determination of the 660-km discontinuity in the Mg_2SiO_4 – Fe_2SiO_4 system at 1740 K using modern multi-anvil techniques

Artem Chanyshv^{1,2} · Dmitry Bondar² · Hongzhan Fei² · Narangoo Purevjav² · Takayuki Ishii^{2,3} · Keisuke Nishida² · Shrikant Bhat¹ · Robert Farla¹ · Tomoo Katsura^{2,3}

Received: 31 March 2021 / Accepted: 11 August 2021

European Journal of Mineralogy

Open Access




Eur. J. Mineral., 34, 201–213, 2022

<https://doi.org/10.5194/ejm-34-201-2022>

In situ reinvestigation of reaction phase A plus high-pressure clinoenstatite to forsterite plus water in the system MgO – SiO_2 – H_2O (MSH)

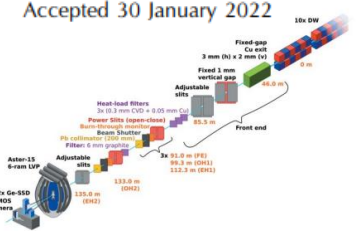
Christian Lathe^{1,2}, Monika Koch-Müller¹, Bernd Wunder¹, Oona Appelt¹, Shrikant Bhat², and Robert Farla²



JOURNAL OF SYNCHROTRON RADIATION

ISSN 1600-5775

Accepted 30 January 2022



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

Robert Farla,^{a*} Shrikant Bhat,^a Stefan Sonntag,^a Artem Chanyshv,^{a,b} Shuailing Ma,^{a,c} Takayuki Ishii,^{b,d} Zhaodong Liu,^{b,c} Adrien Néri,^b Norimasa Nishiyama,^{a,e} Guilherme Abreu Faria,^f Thomas Wroblewski,^{a,f} Horst Schulte-Schrepping,^a Wolfgang Drube,^a Oliver Seck^a and Tomoo Katsura^b

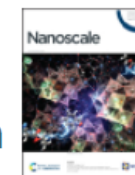
essure



ida,¹ Zhen Chen,⁴
h Yan,⁴ Shuailin Ma,^{3,4}

ferromagnetic nano-structure manganese mono-boride with high Vickers hardness†

Shuailing Ma,^{ab} Robert Farla,^b Kuo Bao,^{*a} Akhil Tayal,^b Yongsheng Zhao,^{ab} Qiang Tao,^a Xigui Yang,^{id} Teng Ma,^a Pinwen Zhu^a and Tian Cui^{id} *ad

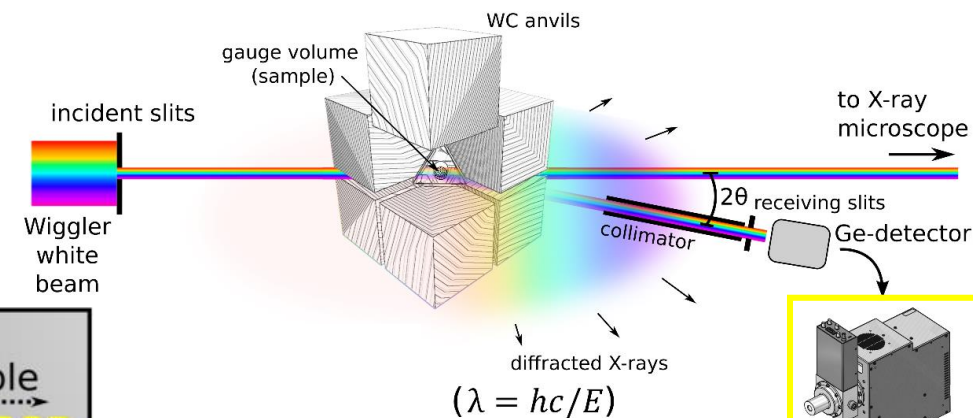
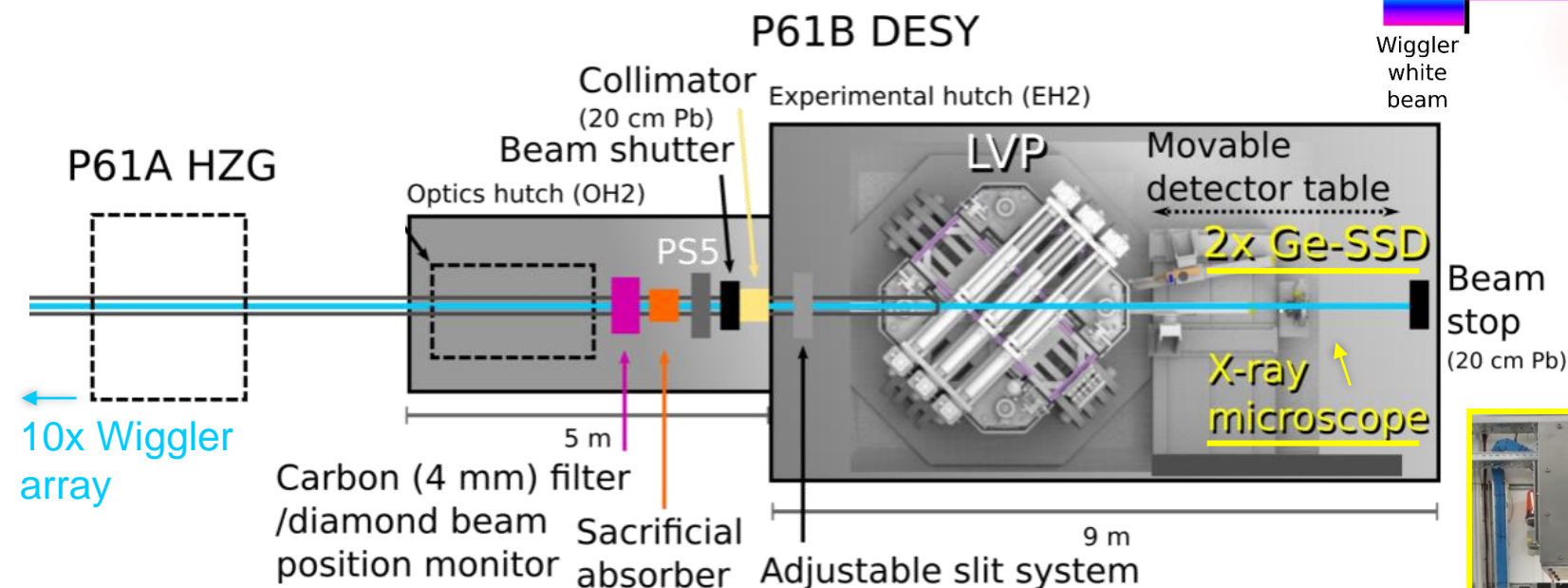


From the journal:
Nanoscale

Beamline layout

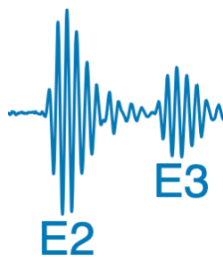
The Large Volume Press (LVP) extreme conditions beamline (50% X-rays, 50% offline operation)

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

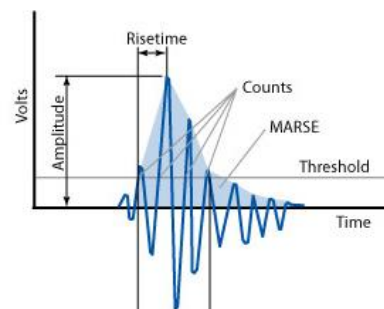


Energy-Dispersive XRD

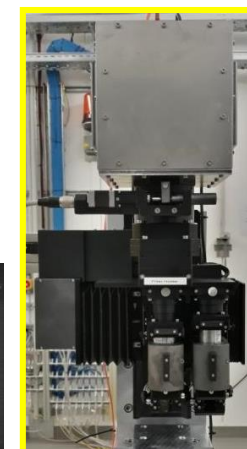
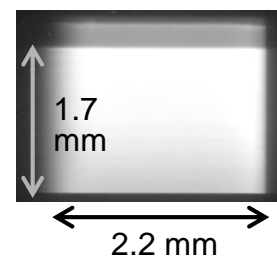
Ultrasonic velocity measurements



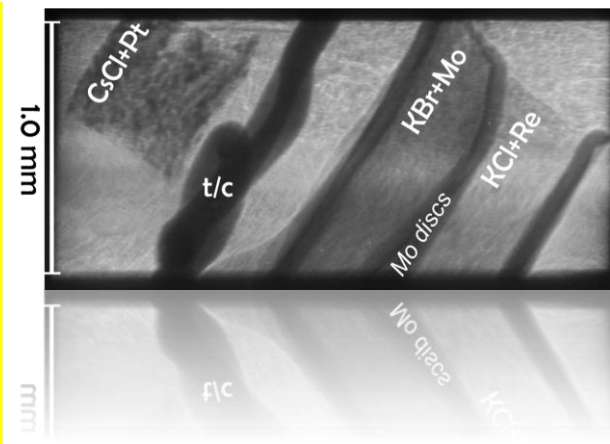
Acoustic Emission detection



Full beam size



High resolution imaging

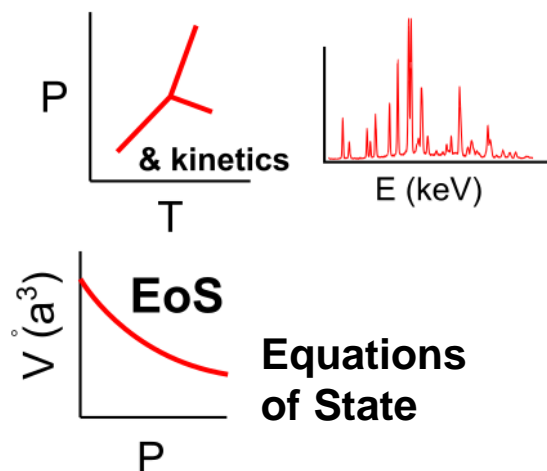


P61B LVP Mission

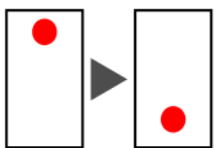
Access modes

For 50% beam availability (submit by: 01.03.2023)

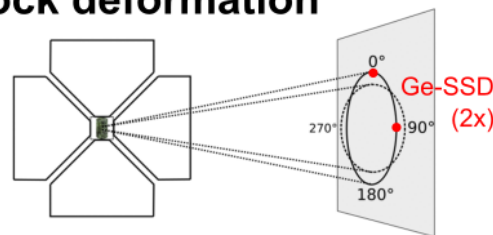
1 Phase relations & discovery of new HP phases



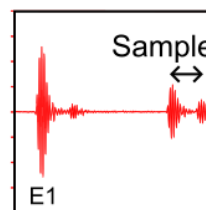
2 Falling-sphere viscosimetry



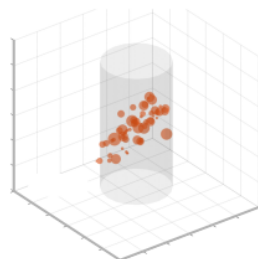
3 Controlled rock deformation



4 Ultrasonic Interferometry (10-60 MHz)



5 Acoustic Emission detection (0.1 - 4 MHz)



For 50% LVP availability (no X-rays)

- 'Rapid access'
 - Submit proposal any time, no external review
- Synthesis of novel (recoverable) materials
 - Band-gap tuning/semiconductors, optical windows, super-hard/conductive, catalyzers, hydrogen storage
 - Trial experiments before (scheduled) beam time

Looking forward



to your proposals

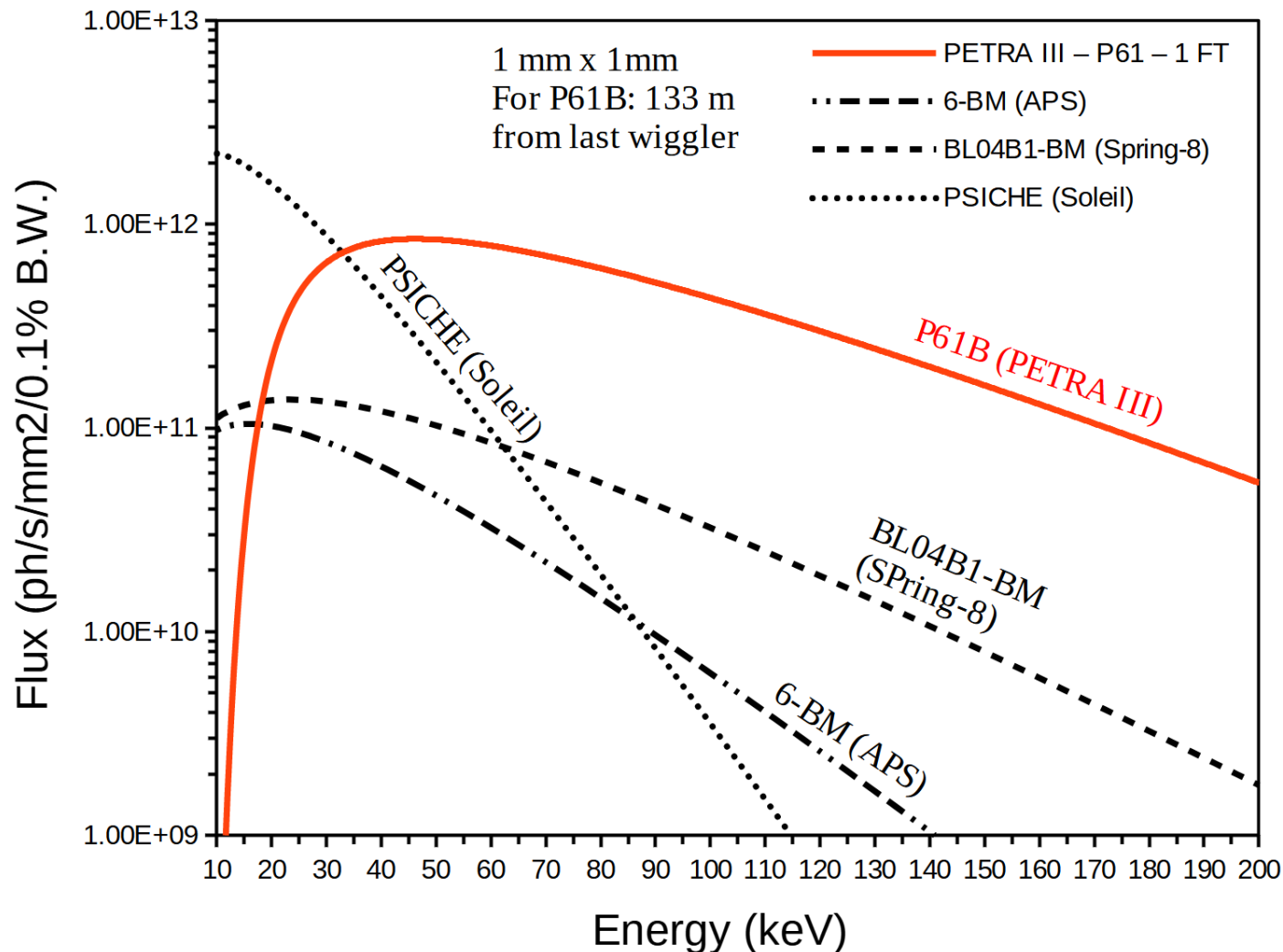
Aster-15



LVP-P61B

Beamline layout

Flux comparison with other white X-ray, LVP beam lines



Flux at P61

Peak at @ 50 keV

~10¹² ph/s/mm²

Integrated flux (1FT)

~10¹⁴ ph/s/mm²

If you need white X-rays < 40 keV,
then PSICHÉ (Soleil) can be interesting.

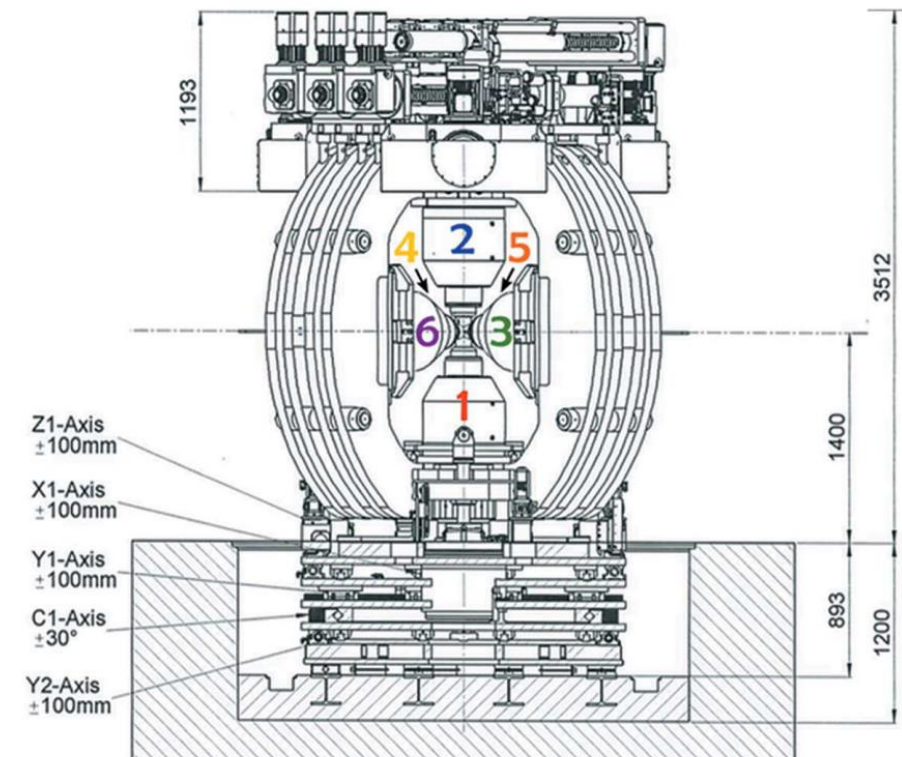
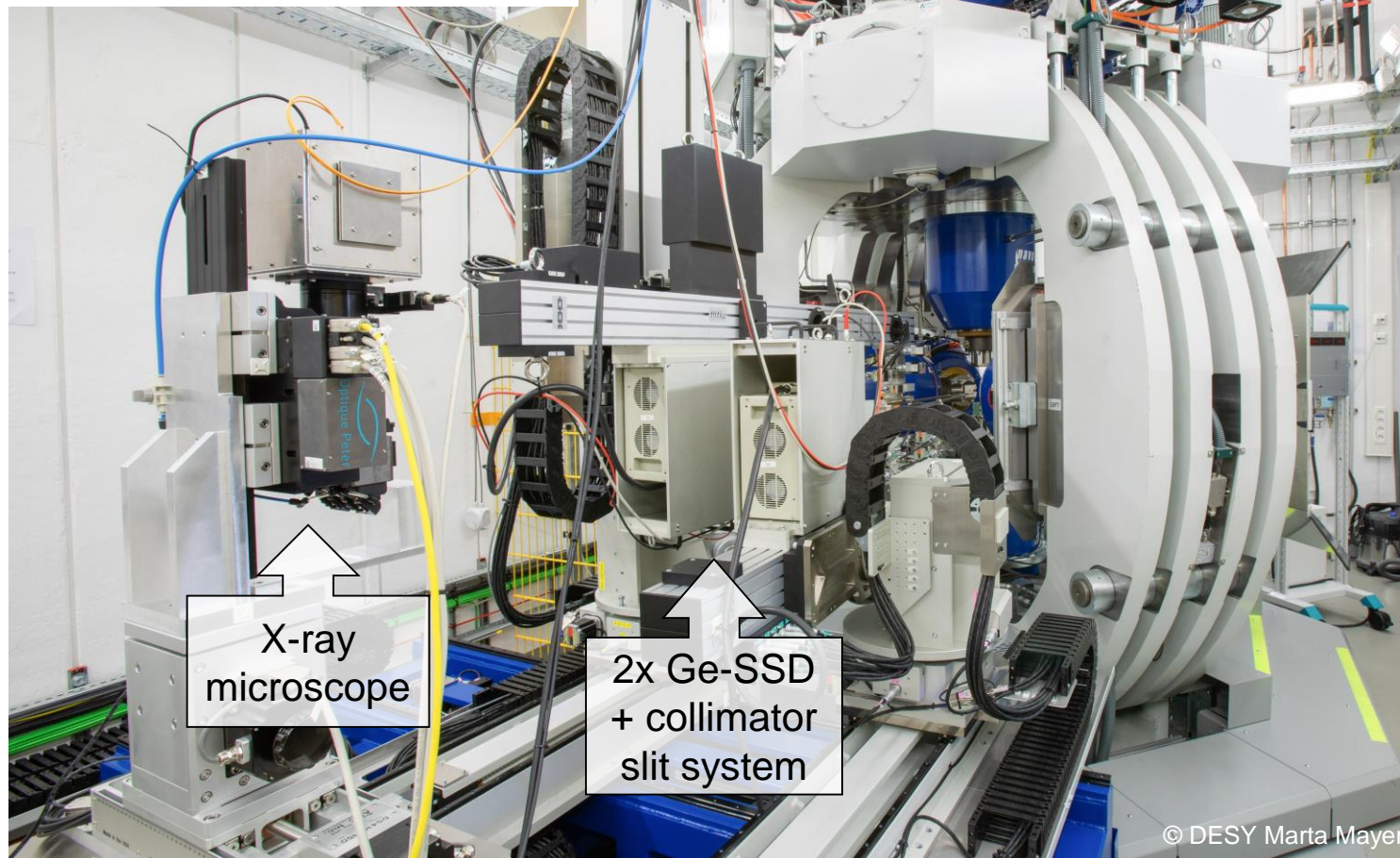
**For all other needs (high-energy white X-rays),
P61B LVP excels in the world.**

The LVP 'Aster-15'

Key specifications

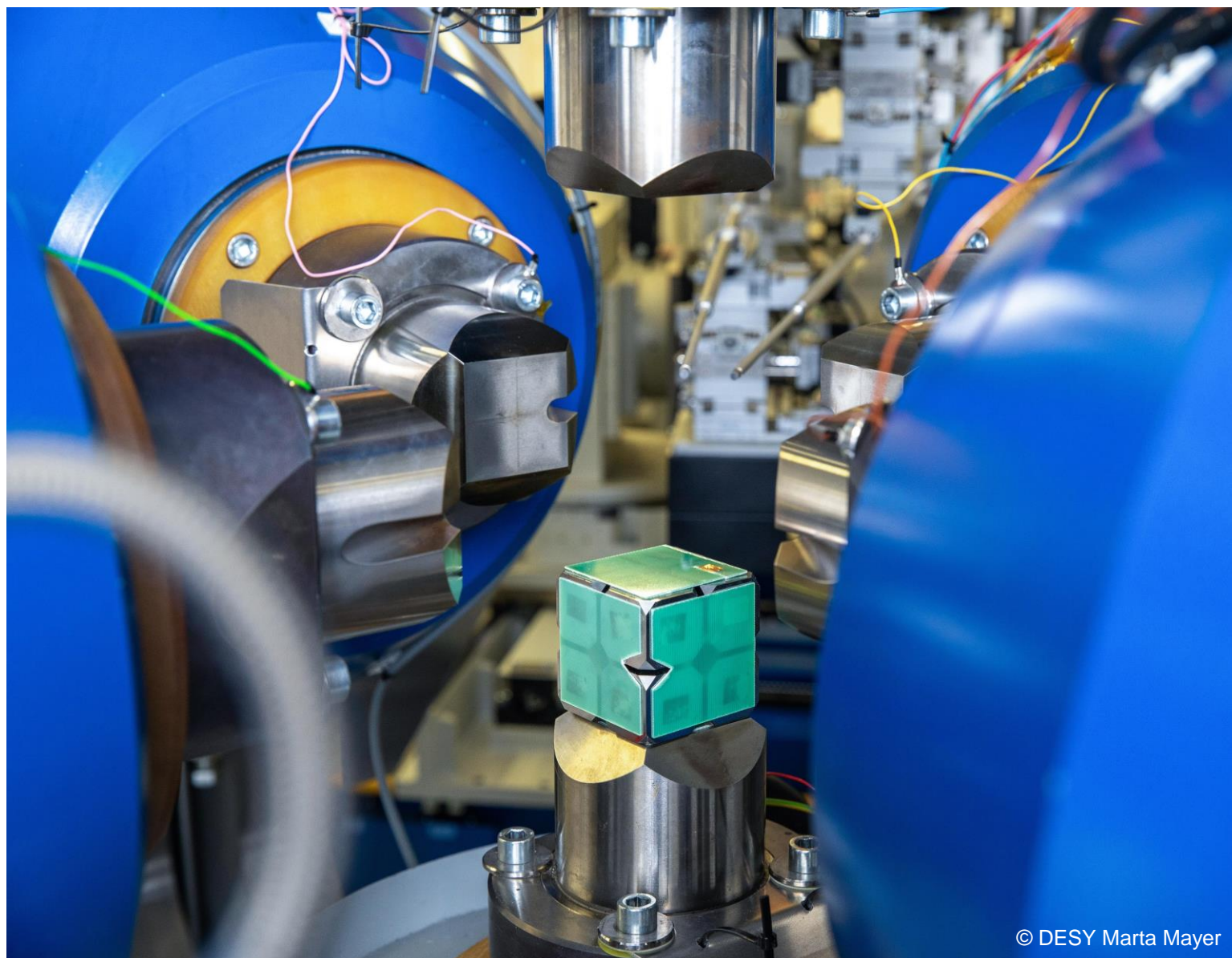
'Aster-15' mavo press LPQ6-1500-100

+ Detector positioning system



Maximum load	15 MN – 5 MN/axis
Ram position control	1 μ m step – 100 mm
Oil pressure control	0.5 bar – 620 bar/ram
Anisotropic compr.	Axial symmetric, triaxial
5-axis stage	x,y ₁ ,y ₂ ,z (\pm 100 mm), rotation: \pm 11.5°
Combined weight	ca. 45 ton

High-pressure techniques

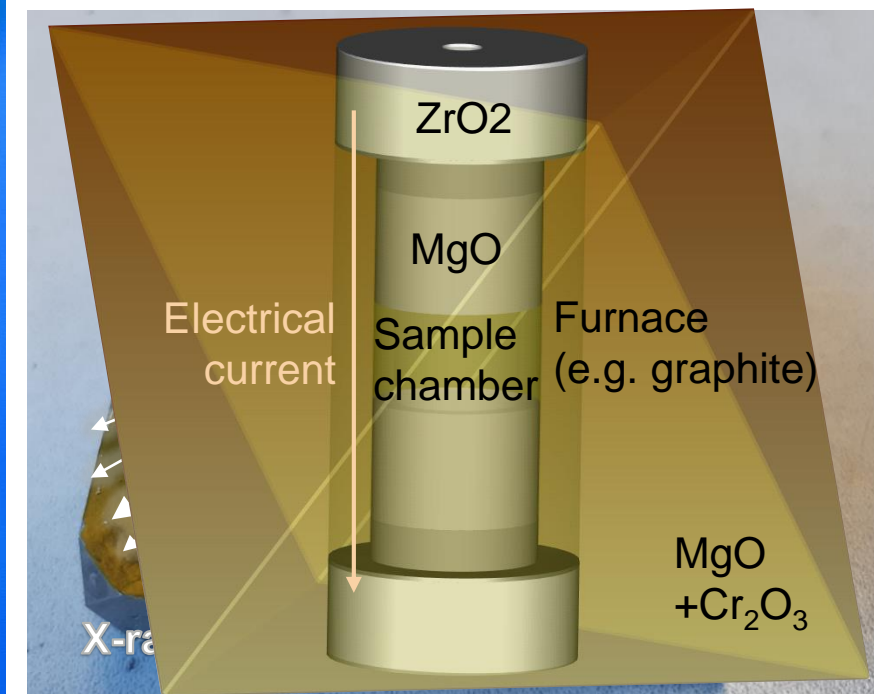


© DESY Marta Mayer

'Kawai' 6-8 mode (octahedral PTM)

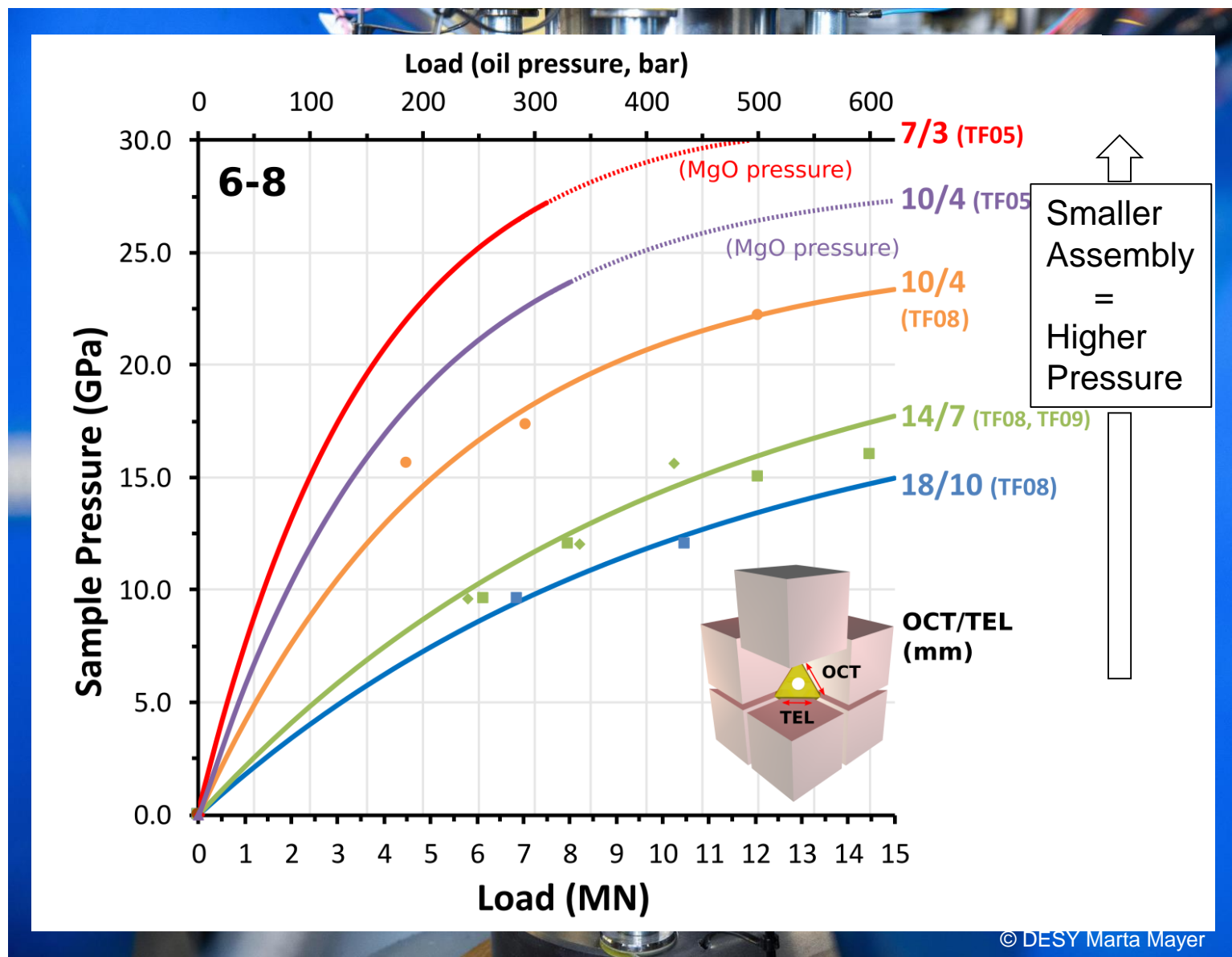
Typical assembly

Recovered assembly after compression



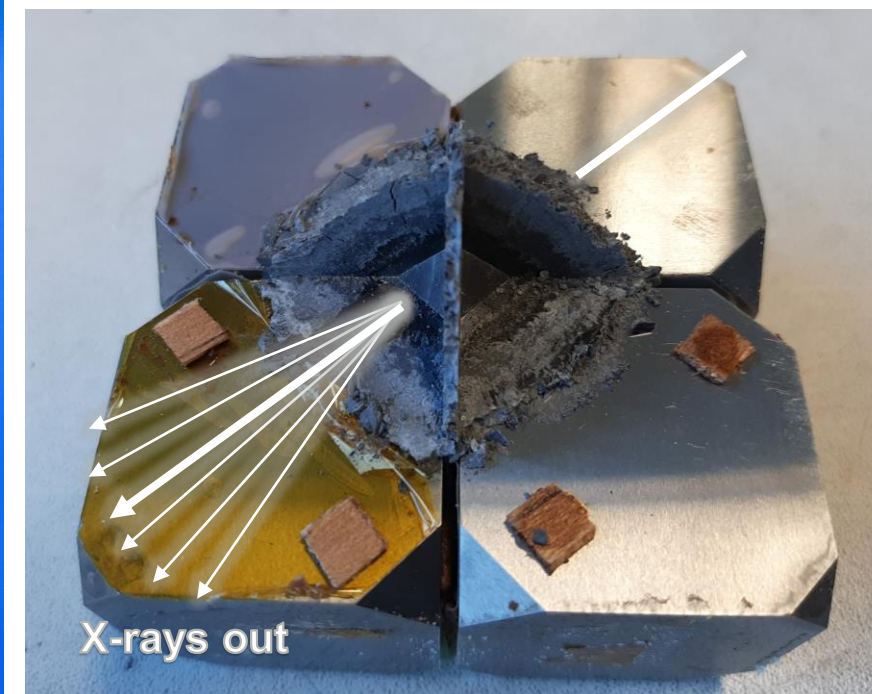
← PTM sizes from 18 mm to 7 mm →

High-pressure techniques



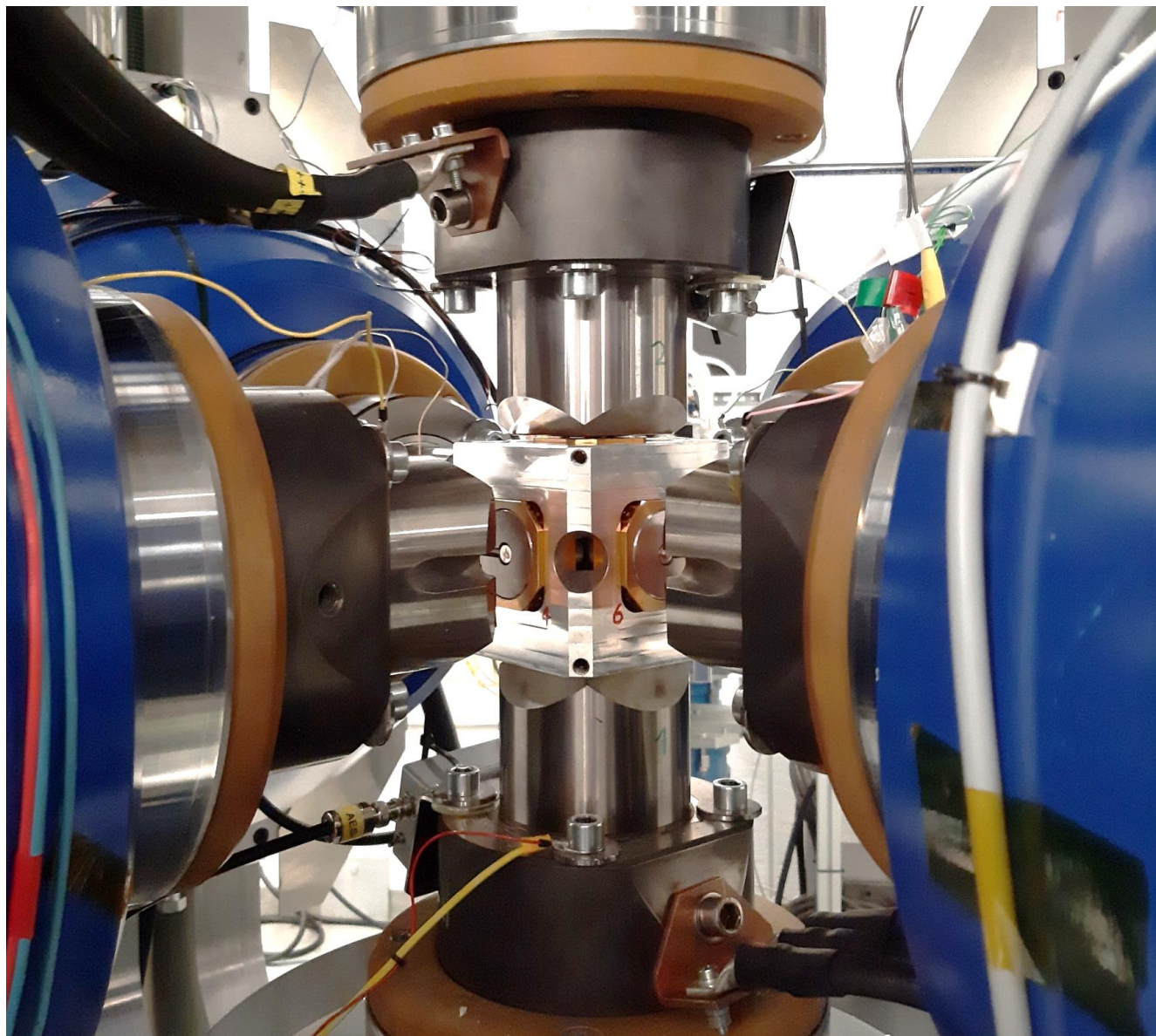
**'Kawai' 6-8 mode
(octahedral PTM)**

Recovered assembly after compression



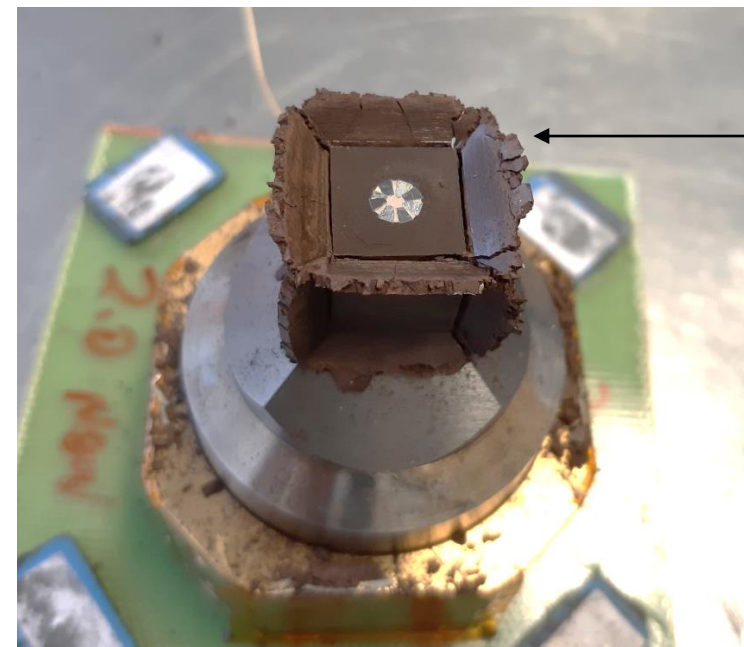
**Useful for standard isotropic
compression and UHP studies.**

High-pressure techniques

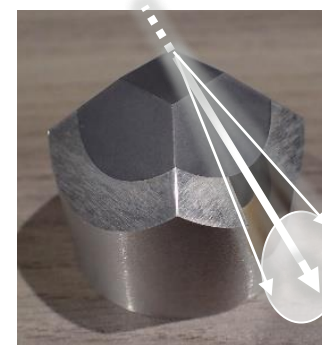


'Hall' 6-6 mode (cubic PTM)

Recovered assembly after compression

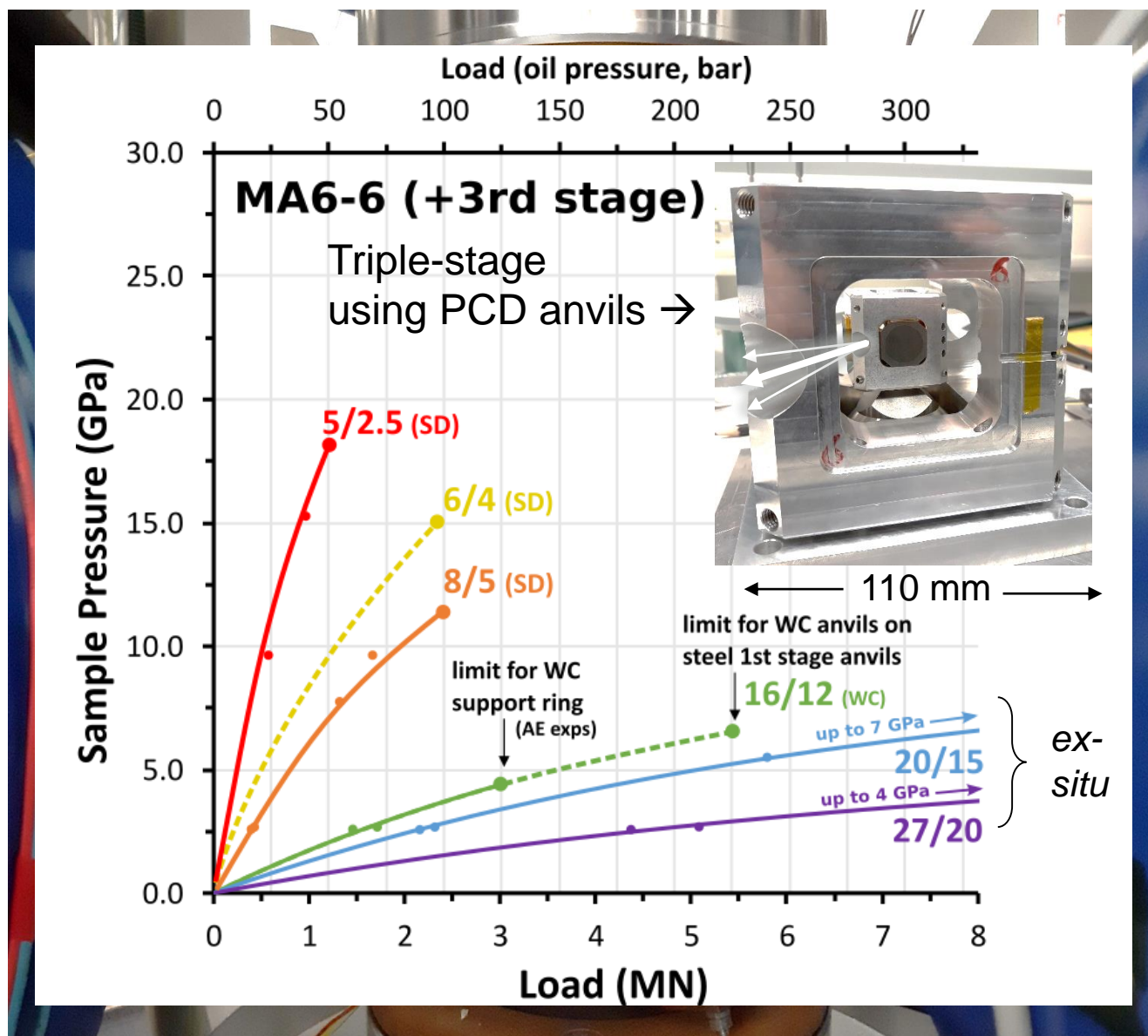


Boron-epoxy
PTM



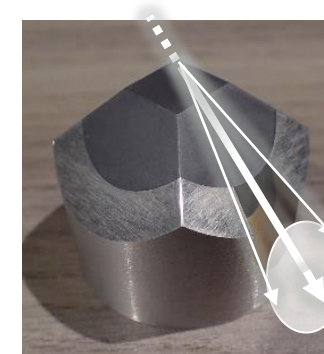
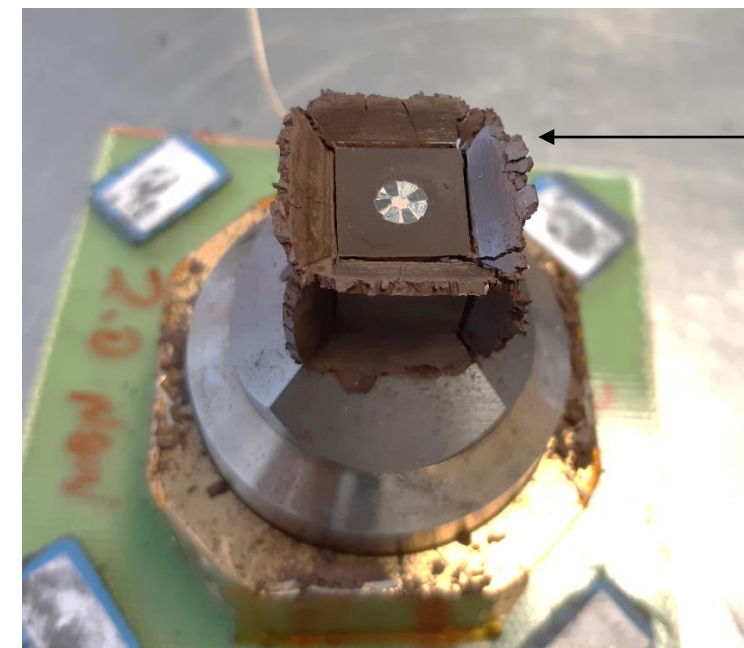
← New!
Large cBN
anvil.

High-pressure techniques



'Hall' 6-6 mode (cubic PTM)

Recovered assembly after compression



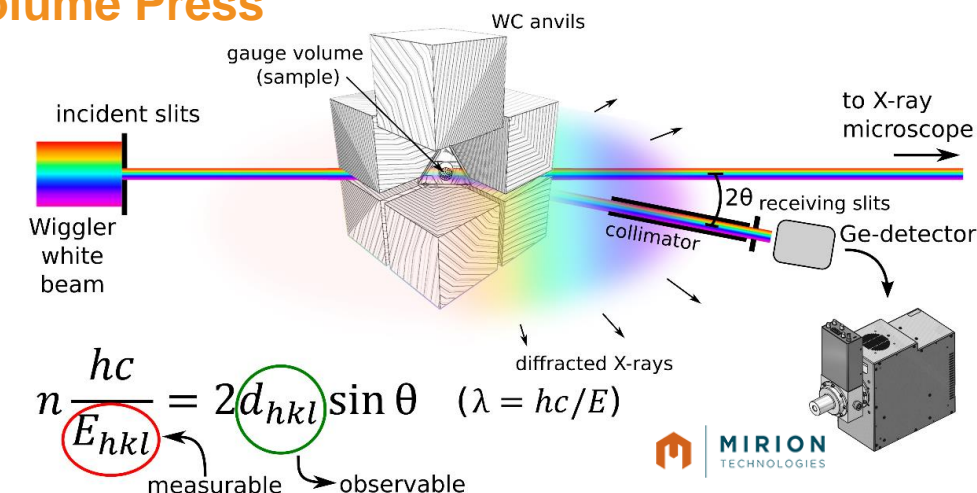
← **New!**
Large cBN anvil.

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

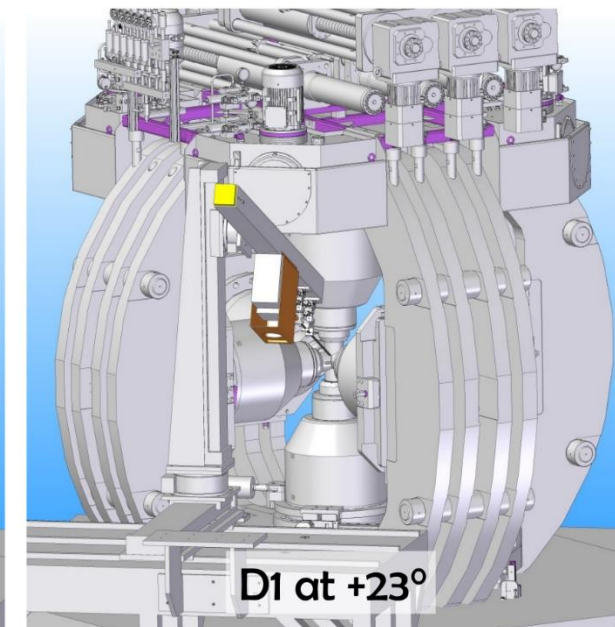
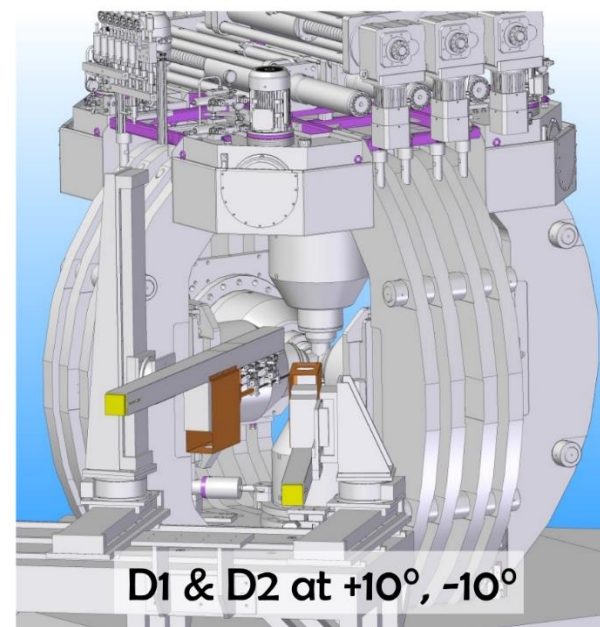
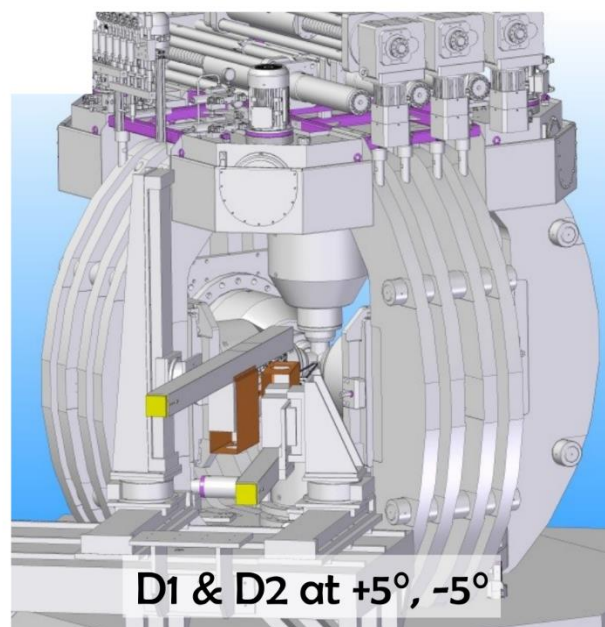
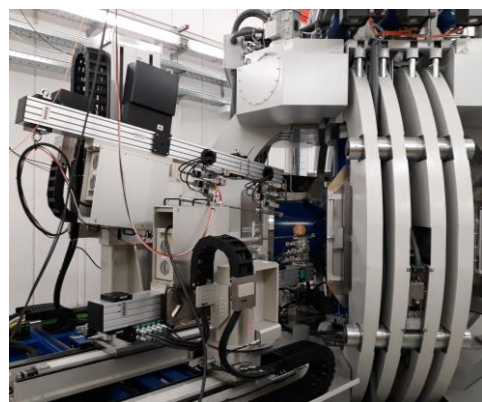
X-ray powder diffraction using white beam

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

1. High spatial resolution (define gauge volume)
 - avoid high temperature & pressure gradients
 - no diffracted X-rays from sample environment
2. Fast acquisition (can be < 10 s)
3. Large Q-range (1 – 12 Å⁻¹, special case up to 25 Å⁻¹)



Various measurement positions of Ge-detectors (x2)



The whitebeam X-ray microscope

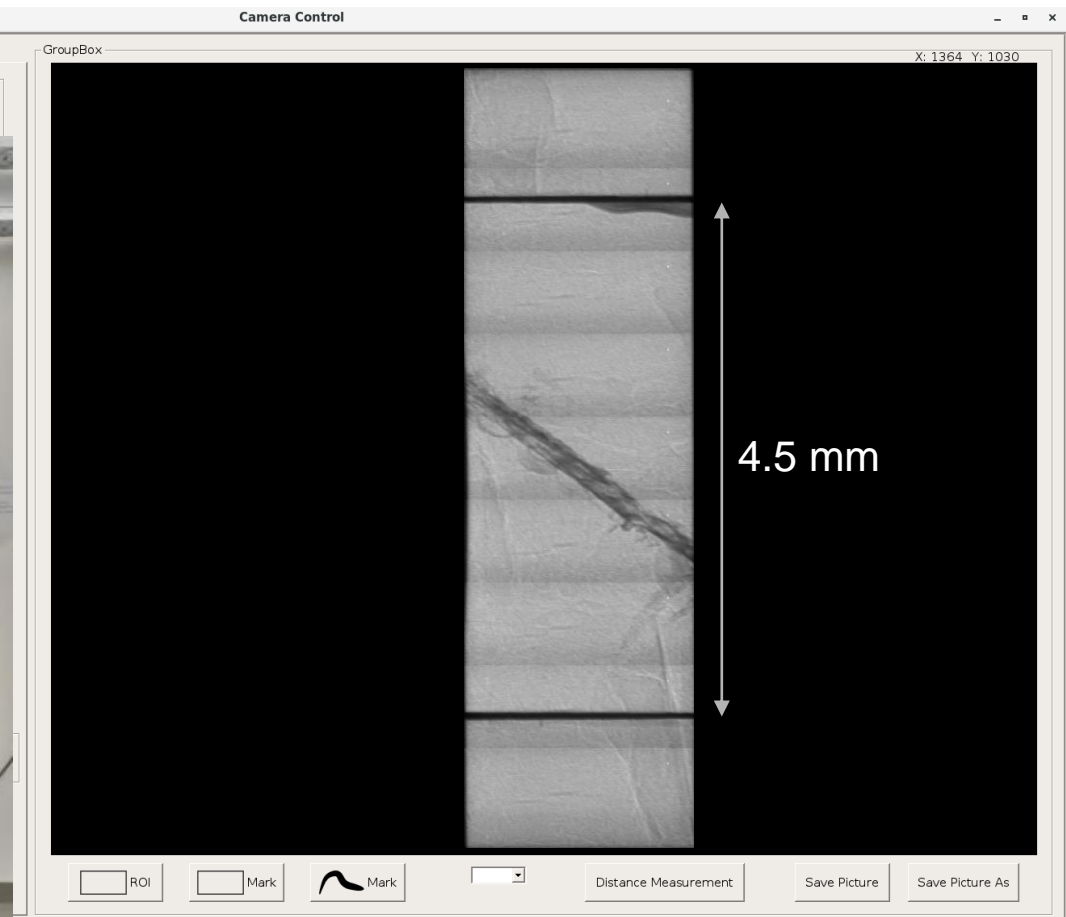
X-ray radiography

- **PCO.edge 5.5 MP sCMOS**
 - True global & rolling shutter
 - 100 fps @ full-resolution (up to 1000 fps for ROI)
 - Live view & frame capture
- **Double objectives (5x, 10x)**
- **LVP Z-stage imaging scan**
- **Scintillators (thickness):**
 - **GGG:Eu (32 ph/keV)**
20, 40 μm , **best resolution**
 - **GaGG:Ce-HL (54 ph/keV)**
150, 200 μm , **ultra-bright**



pco.

Optique Peter
OPTICAL & MECHANICAL ENGINEERING



HPHT synthesis of novel nitrides

World of Nitrides



DOI: 10.1002/chem.201904529

CHEMISTRY
A European Journal
Full Paper

High-Pressure Synthesis | *Hot Paper*

A Novel High-Pressure Tin Oxynitride $\text{Sn}_2\text{N}_2\text{O}$

Shrikant Bhat,^{*,[a, e]} Leonore Wiehl,^[b] Shariq Haseen,^[c] Peter Kroll,^[c] Konstantin Glazyrin,^[a] Philipp Gollé-Leidreiter,^[b] Ute Kolb,^[b, d] Robert Farla,^[a] Jo-Chi Tseng,^[a] Emanuel Ionescu,^[b] Tomoo Katsura,^[e] and Ralf Riedel^[b]

SCIENTIFIC
REPORTS

nature research



OPEN

Discovery of Ternary Silicon Titanium Nitride with Spinel-Type Structure

Shrikant Bhat^{1,✉}, Abhijeet Lale², Samuel Bernard², Wei Zhang³, Ryo Ishikawa^{3,4}, Shariq Haseen⁵, Peter Kroll⁵, Leonore Wiehl⁷, Robert Farla¹, Tomoo Katsura⁶, Yuichi Ikuhara³ & Ralf Riedel⁷

Nitrides

S. Bhat (DESY)

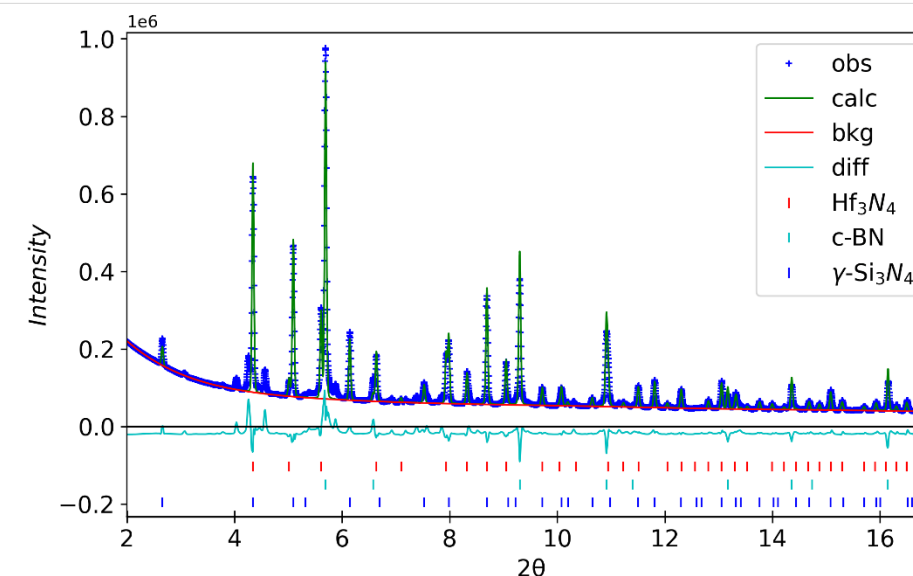
Commercial ALON™ products

Novel High-pressure $\gamma\text{-Si}_3\text{N}_4/\text{Hf}_3\text{N}_4$ ceramic nanocomposites with excellent mechanical properties

Wei Li *et al.*

Submitted 09.11.2022

Acta Materialia



Information provided by Dr. S. Bhat

HPHT synthesis of novel hydrides

World of Hydrides

PHASE 1

Exploration of ternary hydrides at P61B

BH_3NH_3 (the internal hydrogen source)

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

PHASE 2

Investigation of unique properties

Potential superconductivity ?
(at relatively low P compared to DAC studies)

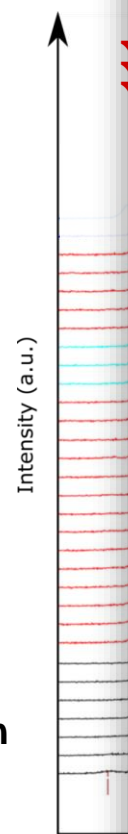
heating to
590 °C

cooling to
370 °C

heating to
410 °C

compression

BaS

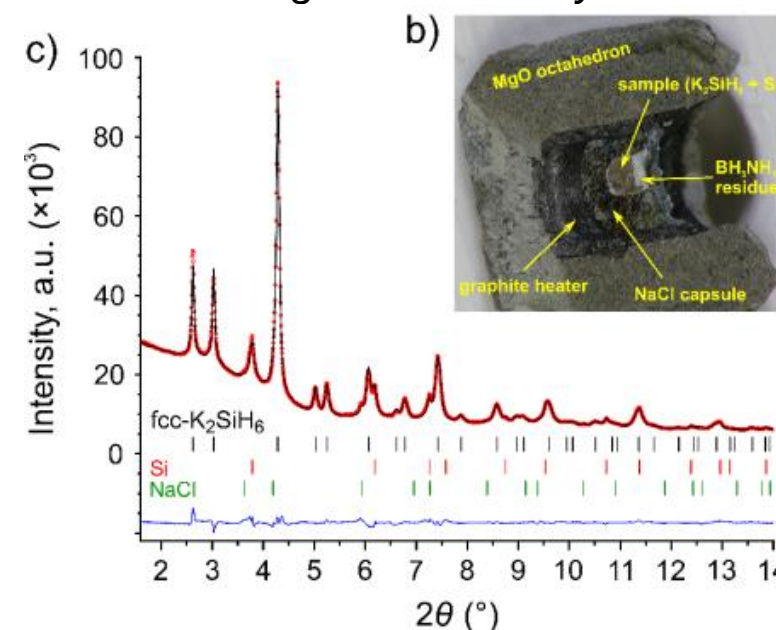


Formation and polymorphism of semiconducting K_2SiH_6 and strategy for metallization

Vekilova *et al.*

Submitted 14.12.2022

Inorganic Chemistry



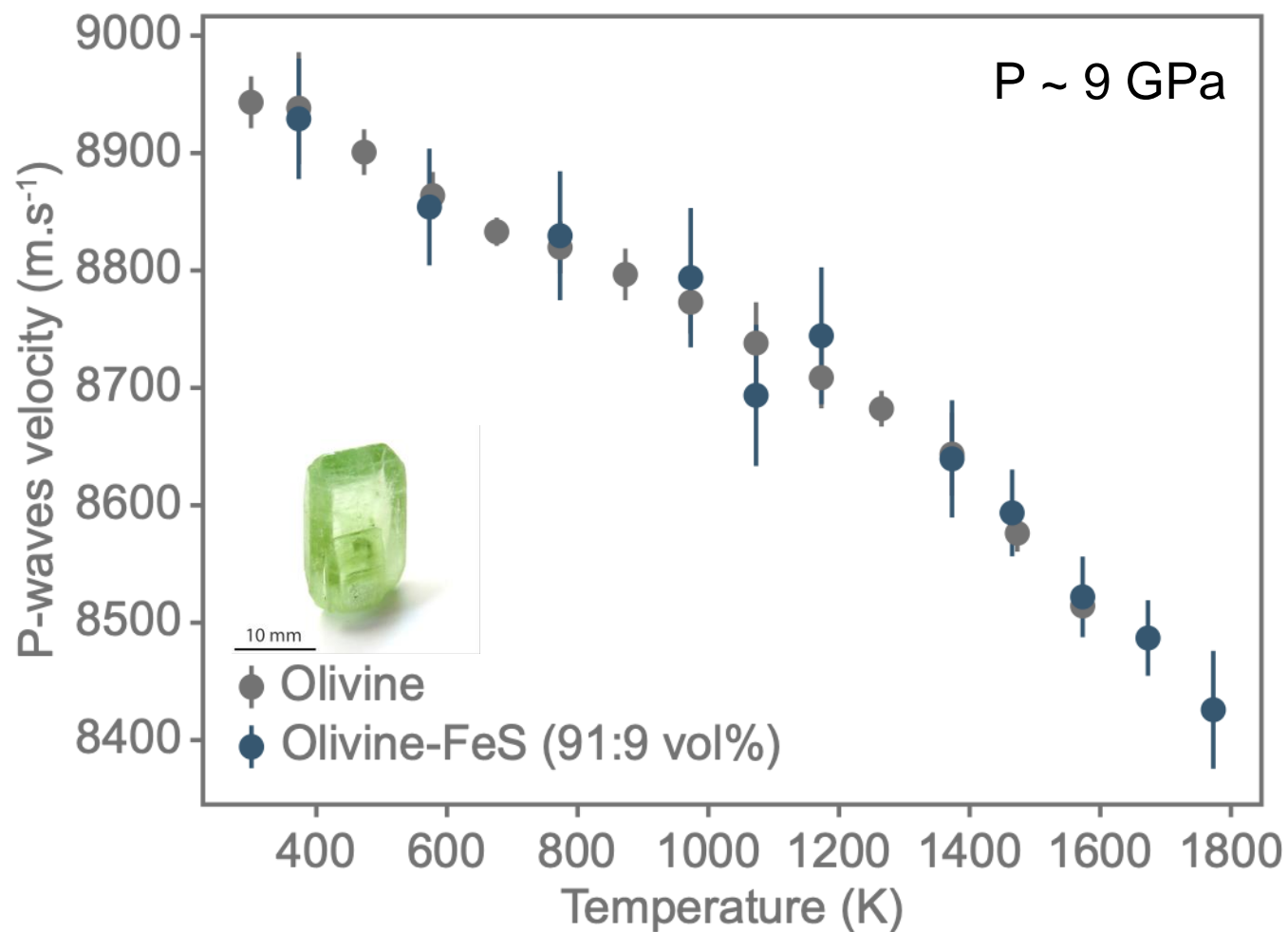
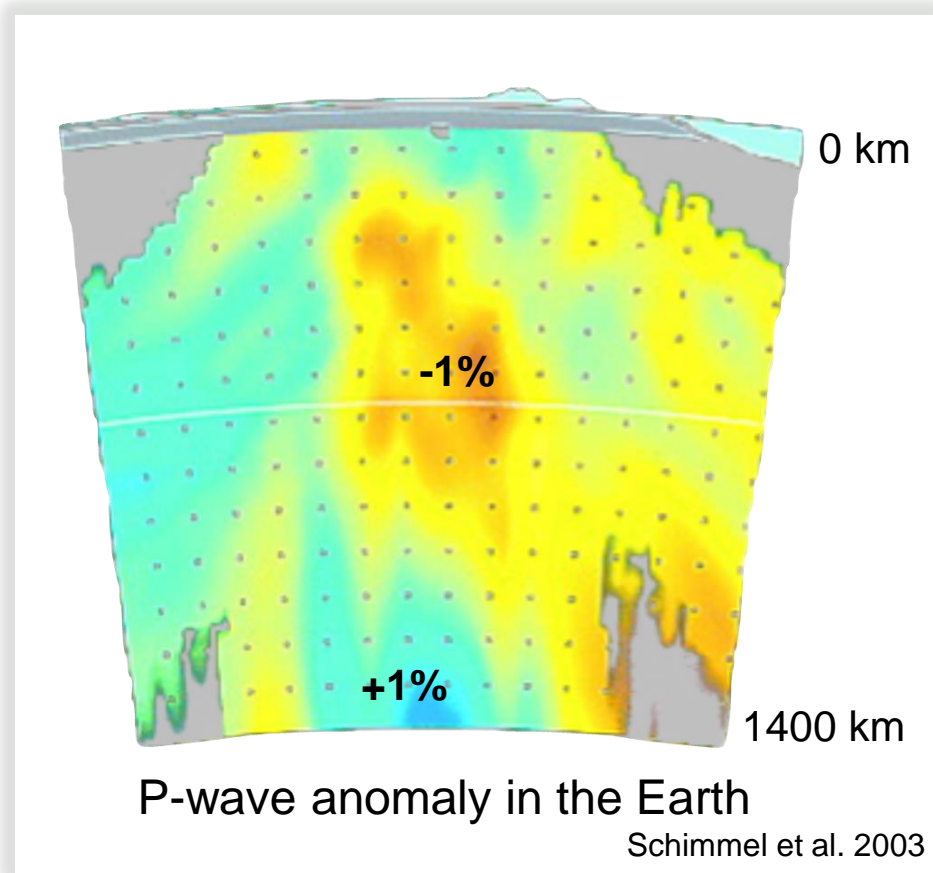
Information provided by Dr. K. Spektor

Wave speed measurements

Ultrasonic Interferometry: the Pulse – Echo technique

Ultrasonic Interferometry

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



Information provided by Dr. A. Néri

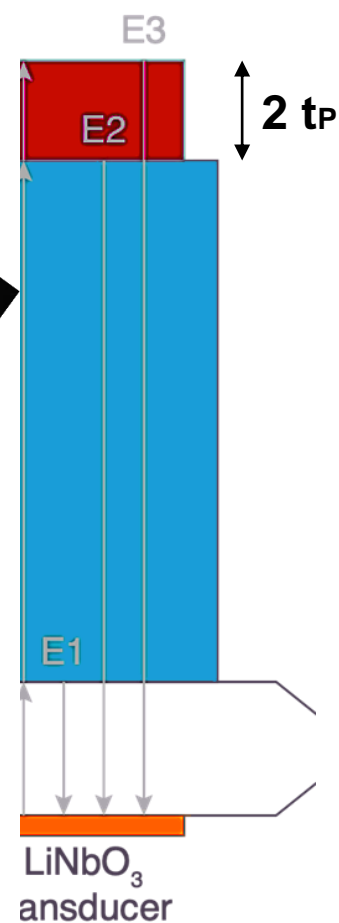
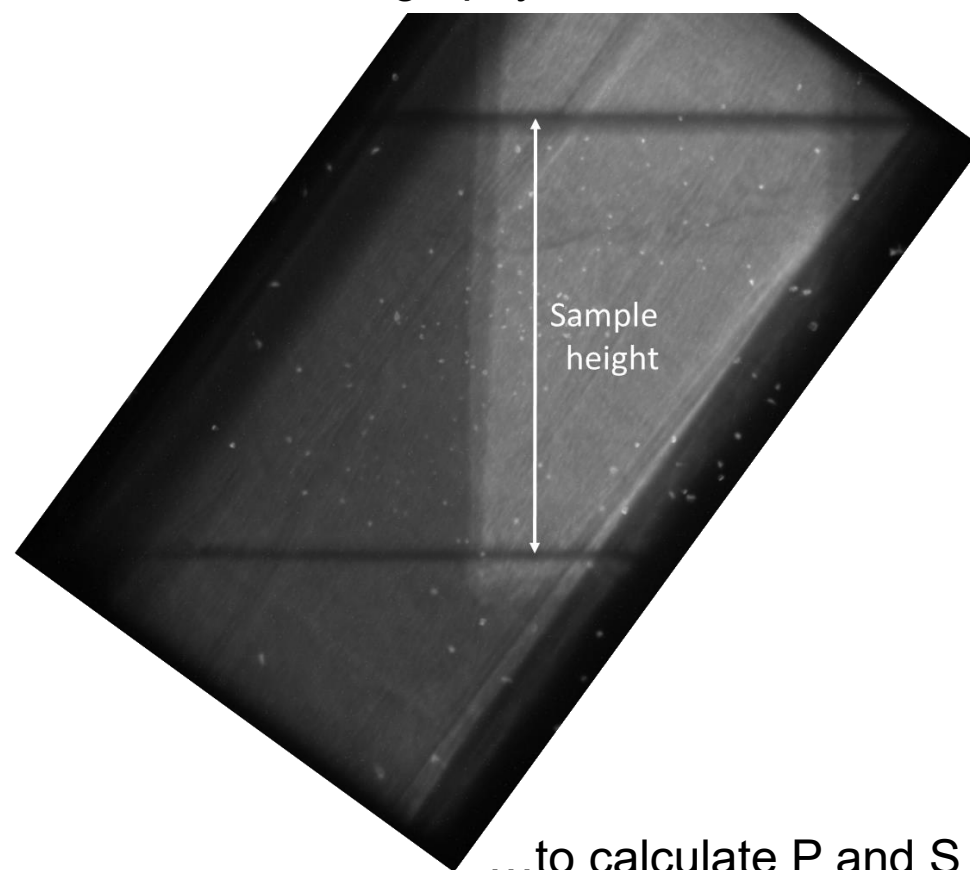
Wave speed measurements

Ultrasonic Interferometry

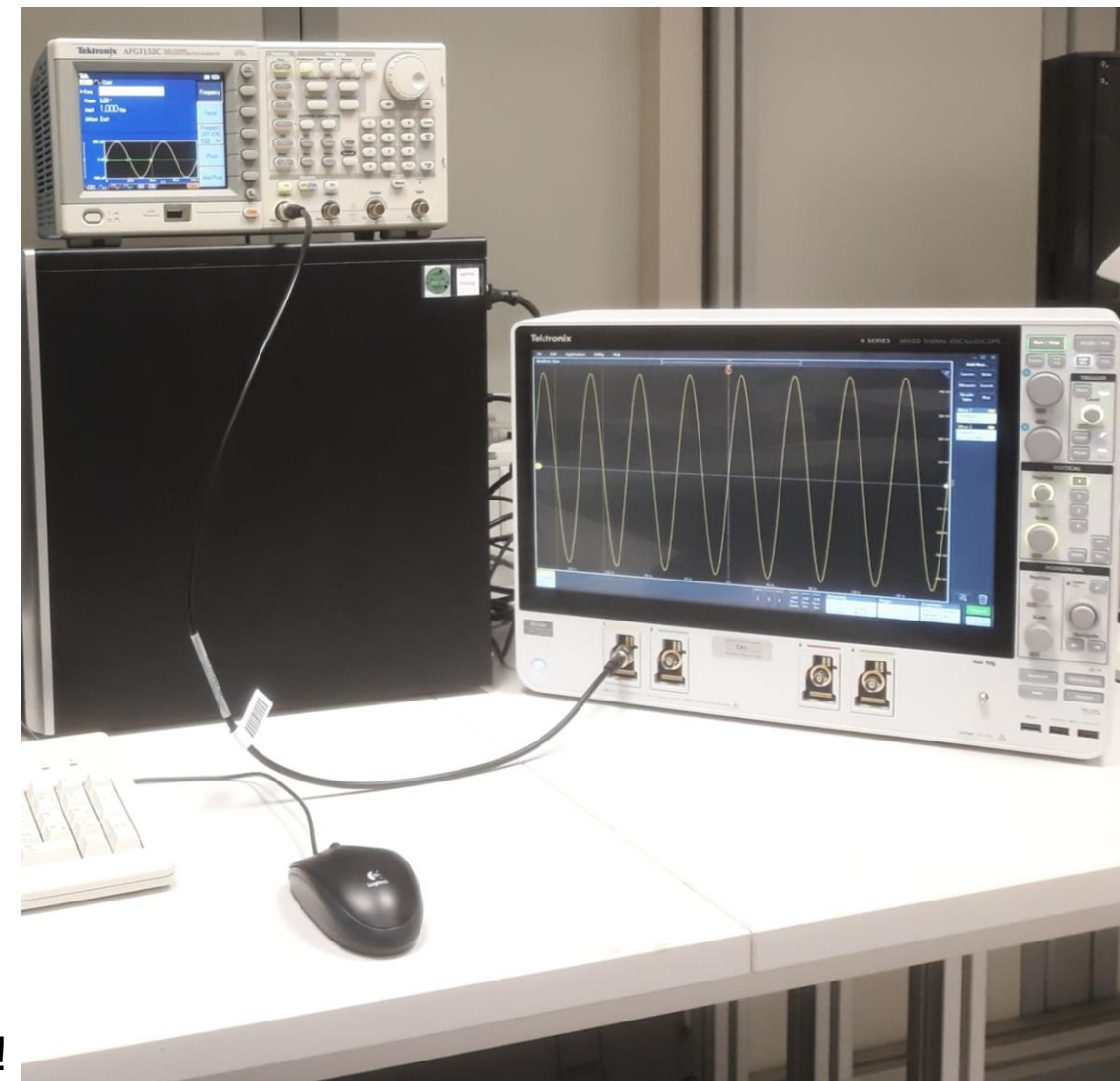
A. Néri, L. Man & R. Farla (BGI / DESY)

Ultrasonic Interferometry: the Pulse – Echo technique

$2t_P$...plus sample length
measurement by
Radiography...



...to calculate P and S wave velocities!

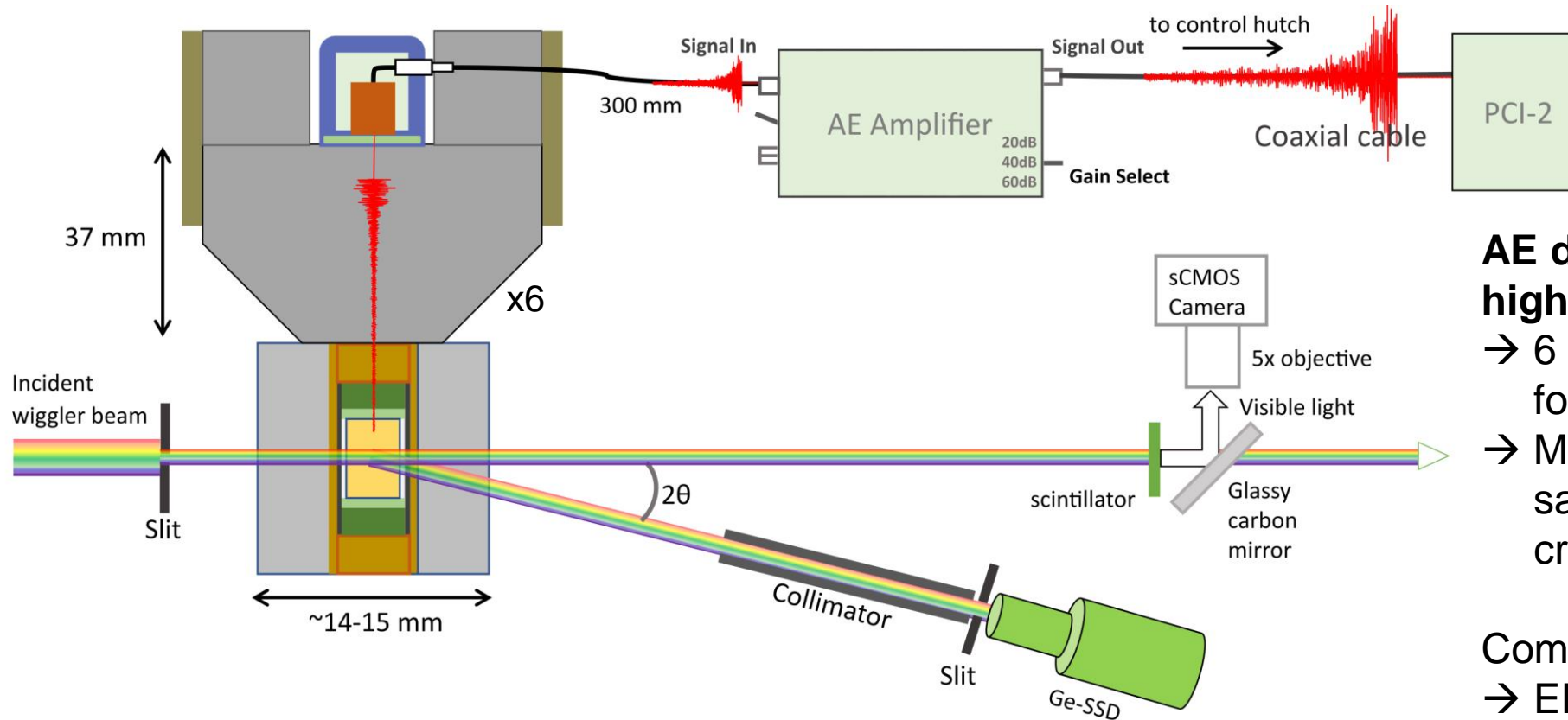


Acoustic Emissions testing

Exploring causes for Earthquakes at the laboratory scale

Acoustic Emissions

S. Ma & R. Farla
(Uni Ningbo / DESY)



AE detection in the LVP at high P, T & Stress

- 6 near-simultaneous hits, form an event.
- Multiple events are located in sample volume, indicates cracking.

Combine with:

- ED-XRD on sample to obtain phase and stress history
- Radiography to obtain the macro-strain history

Brittle dehydration reactions and phase transformations in the Earth
→ possible origin of earthquakes!

Acoustic Emissions testing

Benchmark tests on the *in situ* detection of silica glass fractures

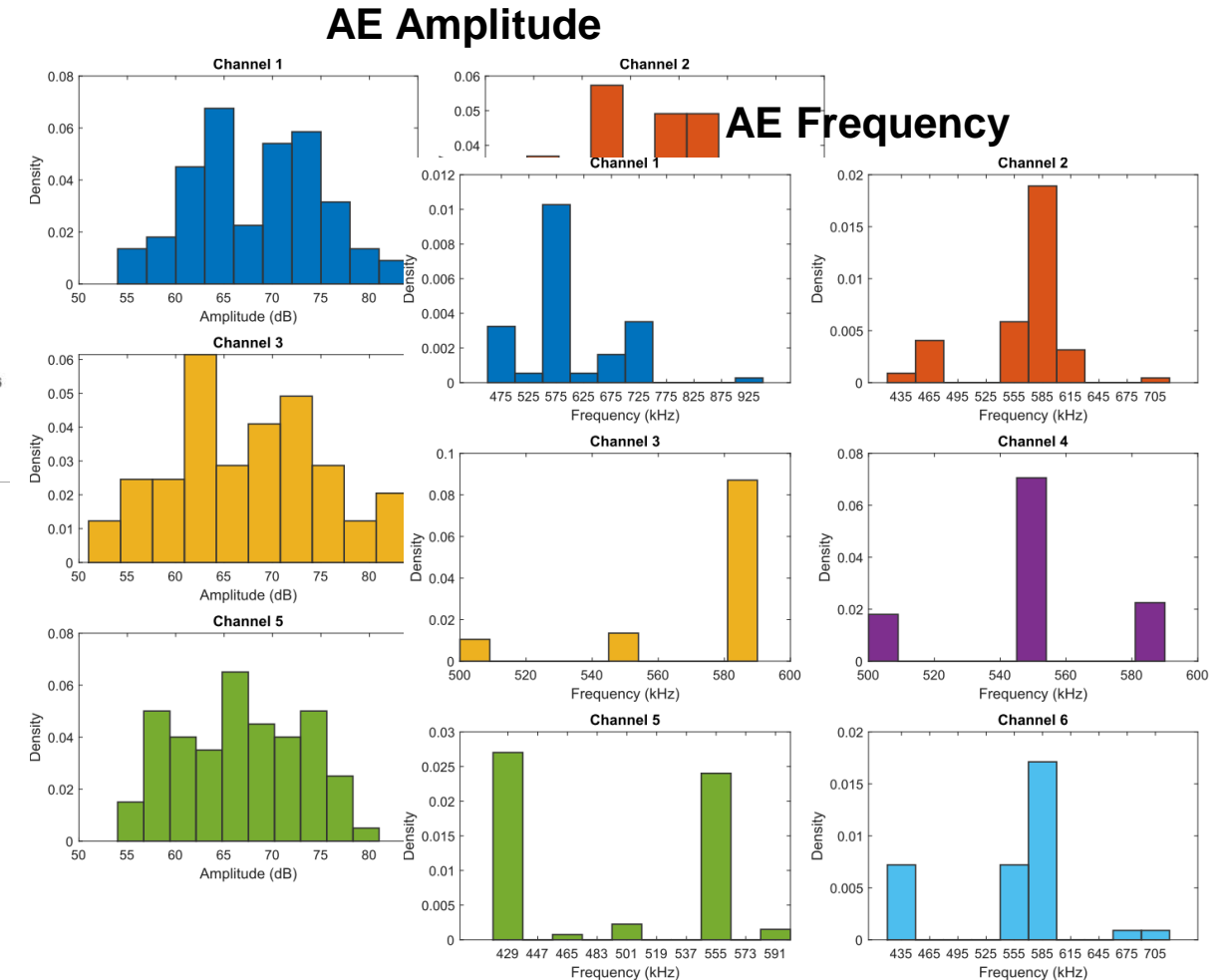
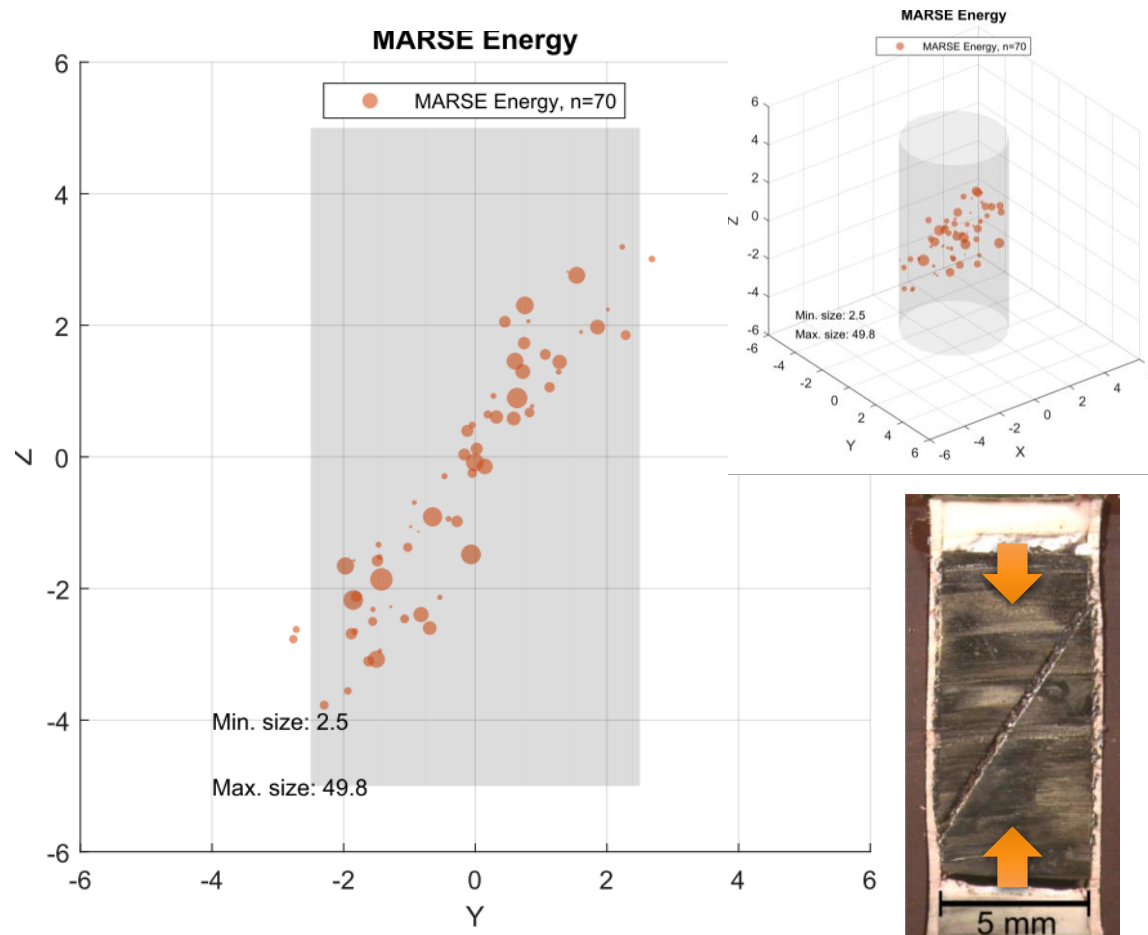
Acoustic Emissions

S. Ma & R. Farla

(Uni Ningbo / DESY)

- Adaptable MATLAB scripts for processing using:
triggered (raw) waveforms and pre-calculated **AE characteristics of events**.

Manuscript accepted in
Review of Scientific Instruments



Beamline development

Beamline software

Load-pressure conversion

Assembly	Sample pressure	Press load (max. 15 MN)	Oil pressure (max. 620 bar)
MA6-8 · 7/3 (TF05)	GPa	0.000 MN	0.0 bar
MA6-8 · 10/4 (TF05)	GPa	0.000 MN	0.0 bar
MA6-8 · 10/4 (TF08)	GPa	0.000 MN	0.0 bar
MA6-8 · 14/7 (TF08)	GPa	0.000 MN	0.0 bar
MA6-8 · 14/8 (BG1) 14/7 (TF09)	GPa	0.000 MN	0.0 bar
MA6-8 · 18/10 (TF08)	GPa	0.000 MN	0.0 bar
MA6-8 · 18/11 (BG1)	GPa	0.000 MN	0.0 bar

Enter parameters

- OPTIONAL: Load Ch-En calibration file(s) for relevant detector(s)
Ch0 (D2) Ch1 (D1)
- Enter approximate 2θ of detectors (for GSAS)
D2: 5.0000 ° D1: 5.0000 °
- Choose fit function
Linear Quadratic
R² Ch0 (D2)
R² Ch1 (D1)
- Load nxs files
Open
- Convert to GSAS and txt formats
Ch0 (D2) Ch1 (D1)

NXS to GSAS & txt Converter

P61B LVP

NXS to GSAS(-II) conversion

CsCl + Platinum

Enter lattice parameters

CsCl

a0	a	± a(err)	V/V0	P (GPa)
4.123	4.123	0.001	1.00	0.000

Pt

a0	a	± a(err)	V/V0	P (GPa)
3.9231	3.9231	0.0001	1.00	0.000

Calculate P above for T (K) = 300

Calculate...!

P-T estimation

P	-0.000	±	0.027	GPa
T	300.00	±	6.46	K

Save as...! Oil P & Power: 0.0 0.0

Note: .txt extension is automatically applied. bar W

Message: Isochors intersect

Simultaneous pressure and temperature determination by two EoS

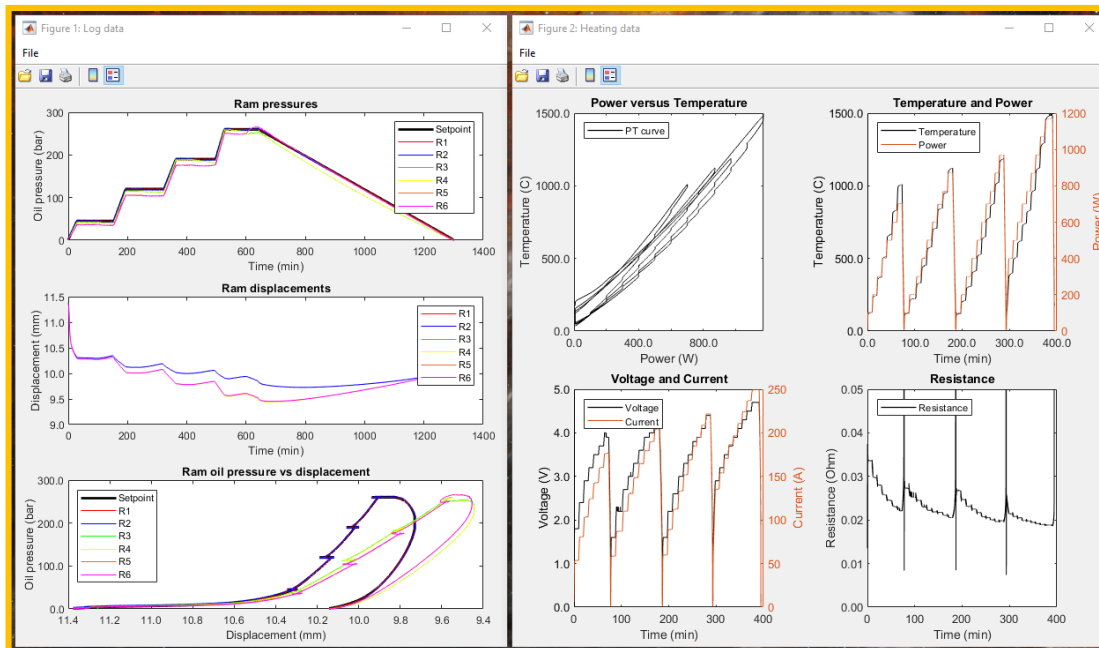
Pressure (GPa)

Temperature (K)

— CsCl (Decker71(modified)/ MGD)
— Pt (Matsui09 / MGD)

x=687, y=22.15

Aster-15 Log-viewer



▲ Simultaneous P & T estimation (manuscript in prep.)

DTA extract

Enter experiment run number
BT_

Extract waveforms and AE data

0 Enter Ch # to remove
6 Arrival time precision

Load DTA file Extract event data

Extract waveforms and AE data

Load DTA file Extract all data

Written by R. Farla at P61B

AE tools

DTA event waveviewer

Enter Ch # to remove ('0' is none): 0 Arrival time precision (# of decimal places, max 8): 6

Then load a Miras DTA file and view the AE waveforms

Load DTA file Total hits: 480 Total events: 80

Viewing hits: 7 - 12 | Event: 2 First threshold crossing (FTC): 30 dB

Amp: 67 dB Abs. Energy: 8.71e+02 Crit: 366 Freq: 585 Ch: 1	Amp: 67 dB Abs. Energy: 1.30e+03 Crit: 402 Freq: 546 Ch: 2
Amp: 63 dB Abs. Energy: 9.79e+02 Crit: 316 Freq: 546 Ch: 3	Amp: 64 dB Abs. Energy: 9.69e+02 Crit: 310 Freq: 585 Ch: 4
Amp: 66 dB Abs. Energy: 1.35e+03 Crit: 283 Freq: 468 Ch: 5	Amp: 64 dB Abs. Energy: 9.20e+02 Crit: 322 Freq: 546 Ch: 6

Arrival time: 295.75713500 s

Arrival time: 295.75713500 s

Arrival time: 295.75713500 s

Arrival time: 295.75713475 s

Arrival time: 295.75713475 s

Arrival time: 295.75713475 s

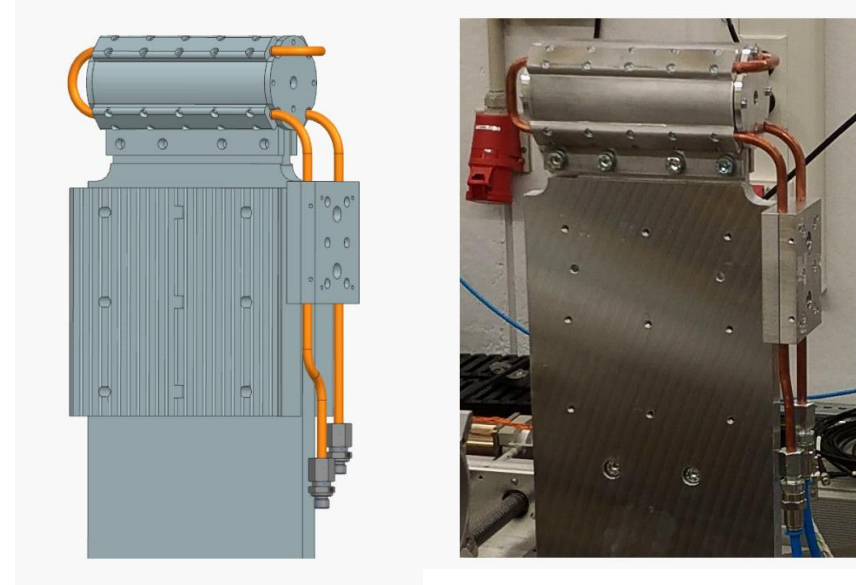
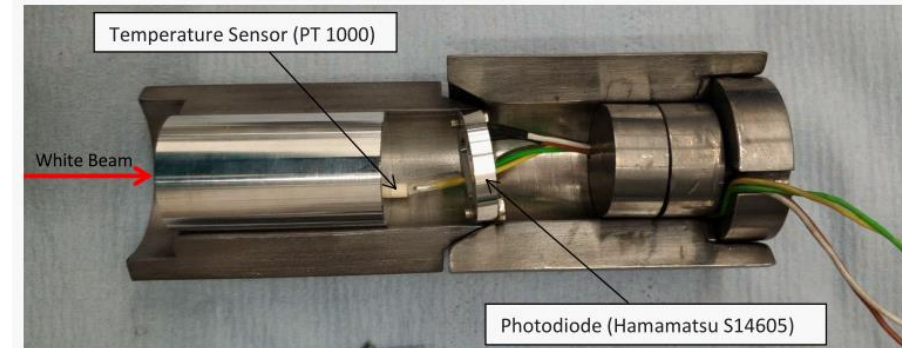
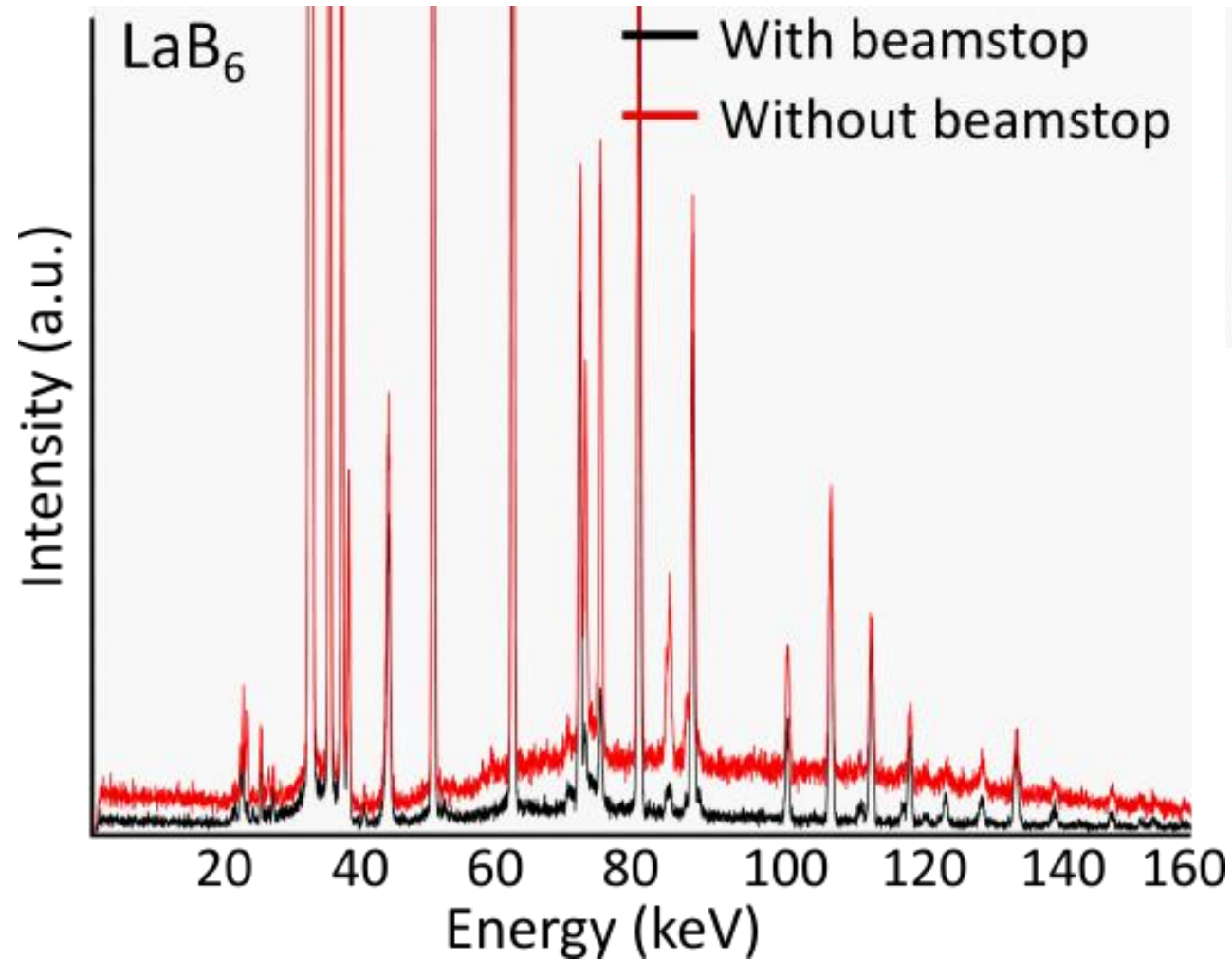
Arrival time: 295.75713475 s

Time (microseconds)

Written by R. Farla at P61B 16.12.2022 v1.0

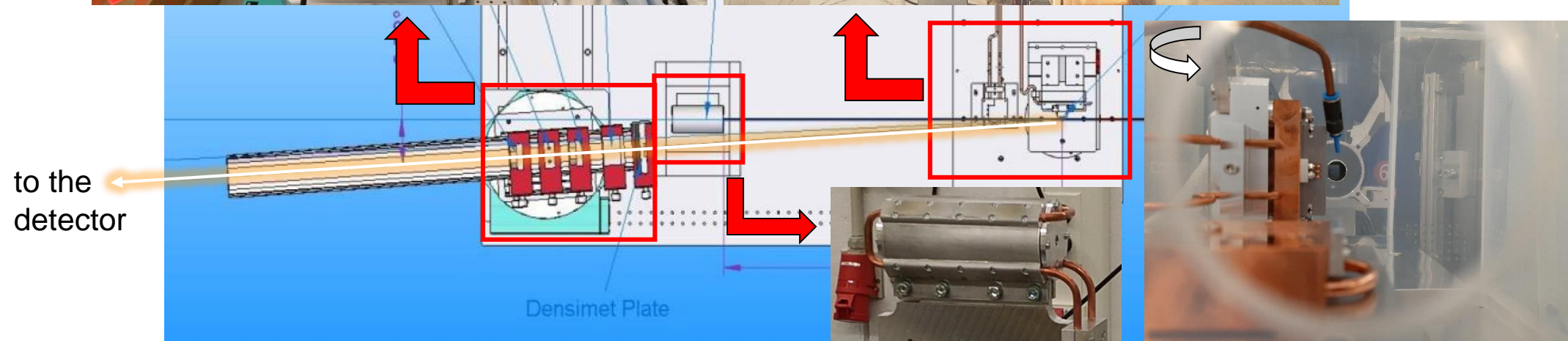
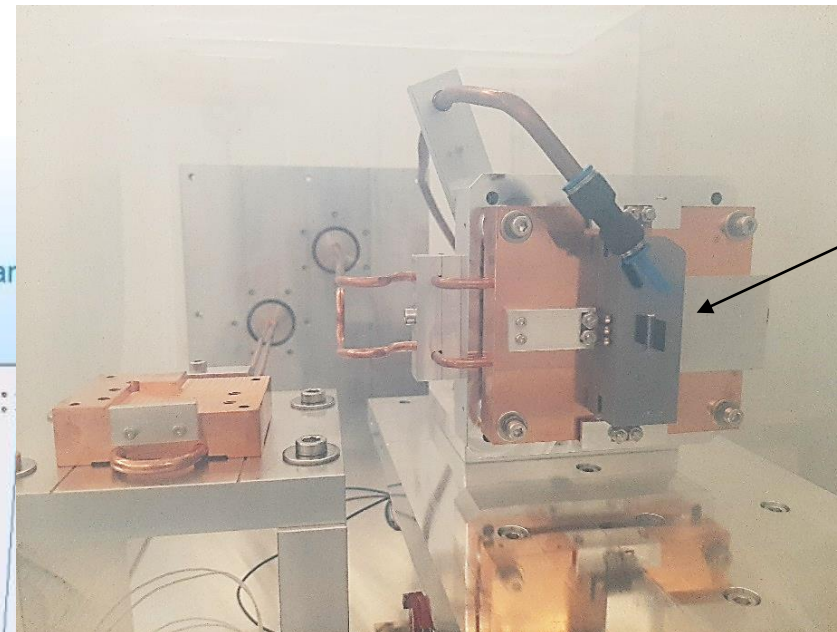
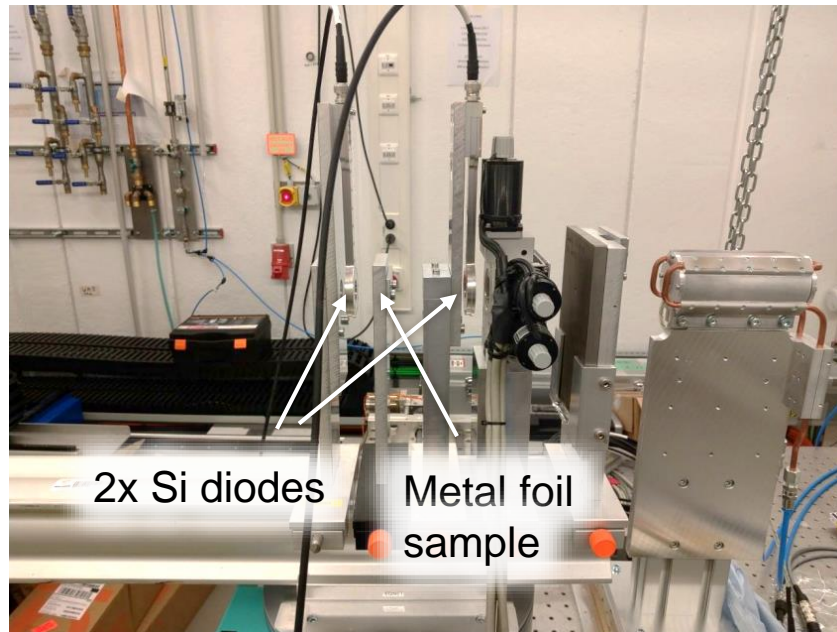
Beamline development

Beamline hardware (1/2) – the beam stop



Beamline development

Beamline hardware (2/2) – monochromator update

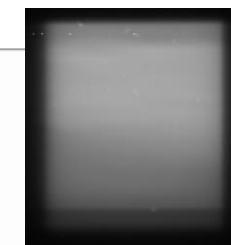


Beamline development

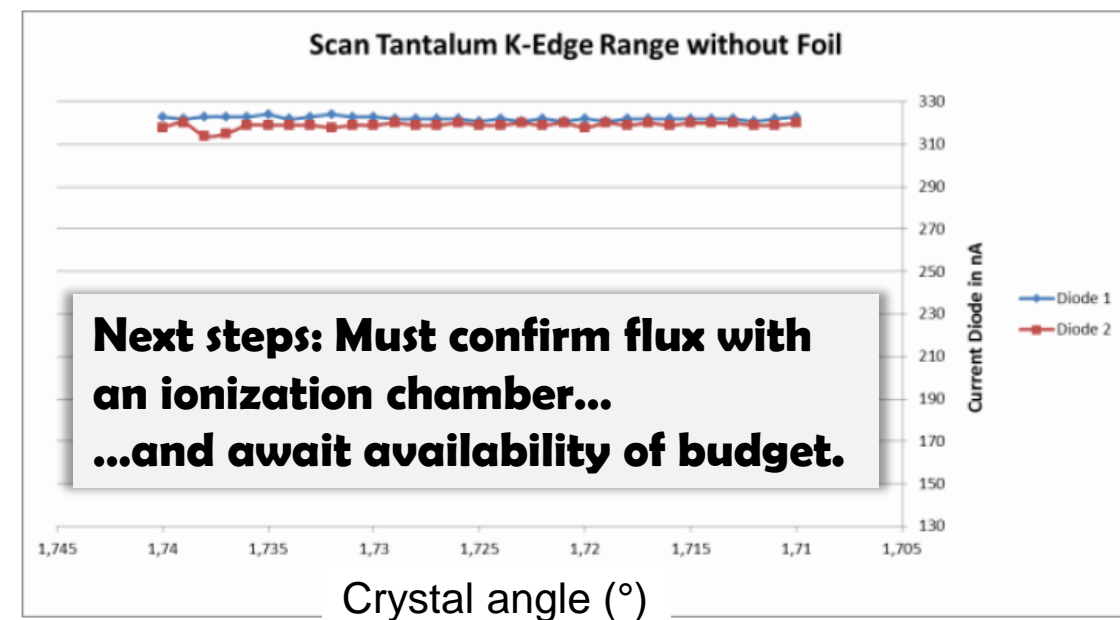
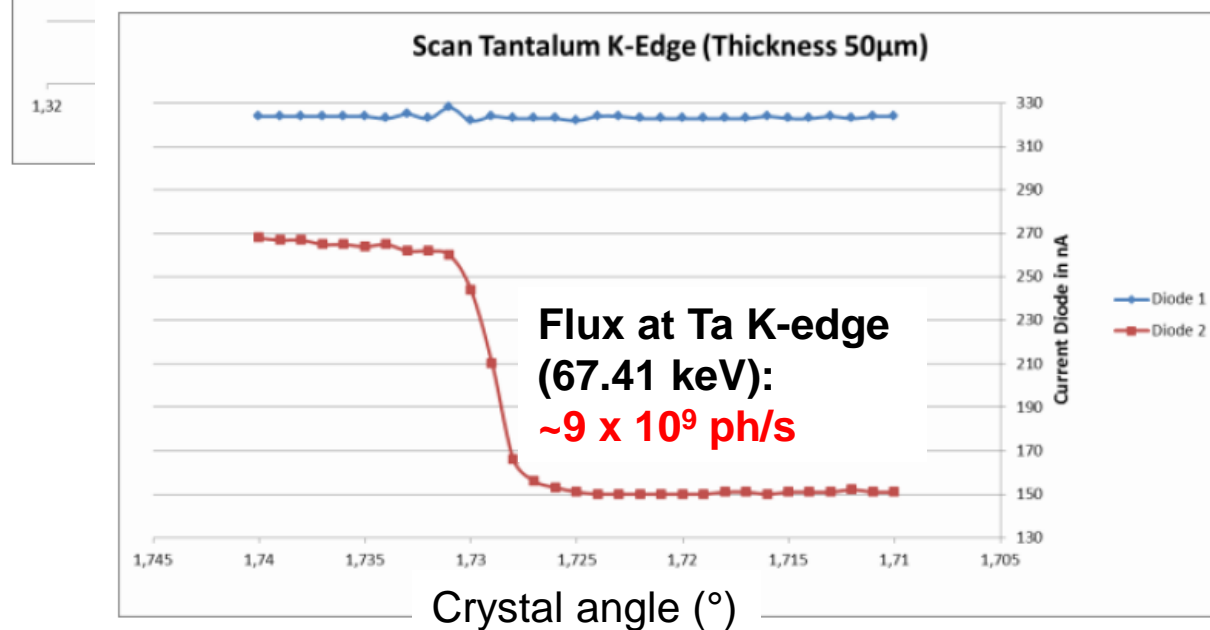
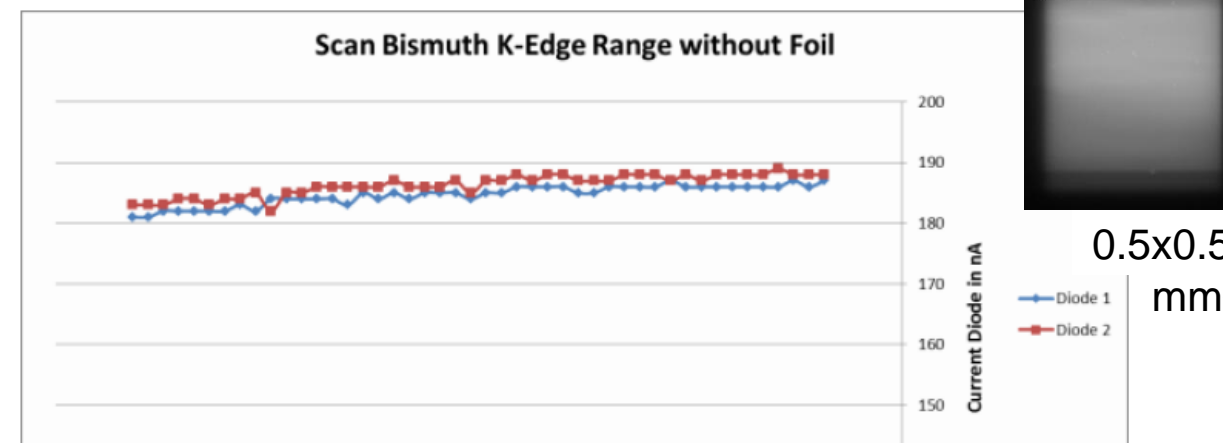
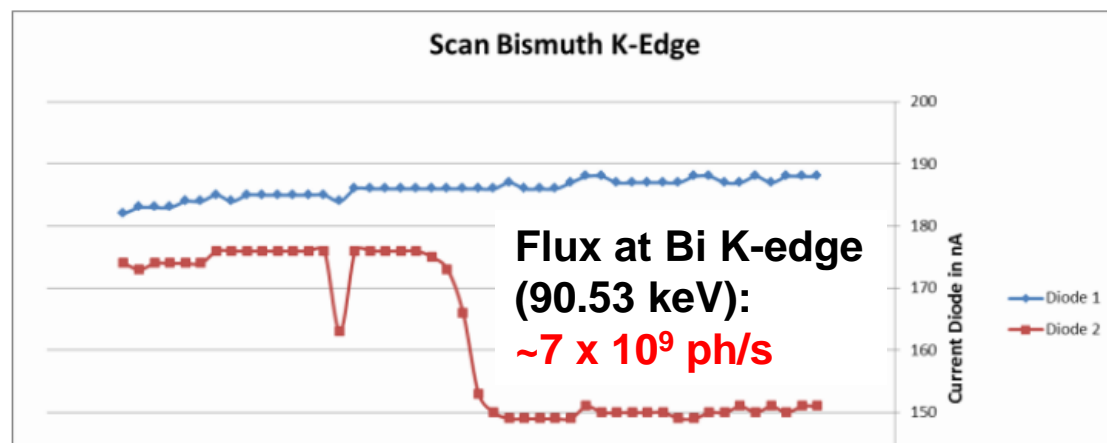
Beamline hardware (2/2) – monochromator update

N.B. crystal angle \neq Bragg angle

Mono beam



0.5x0.5
mm²



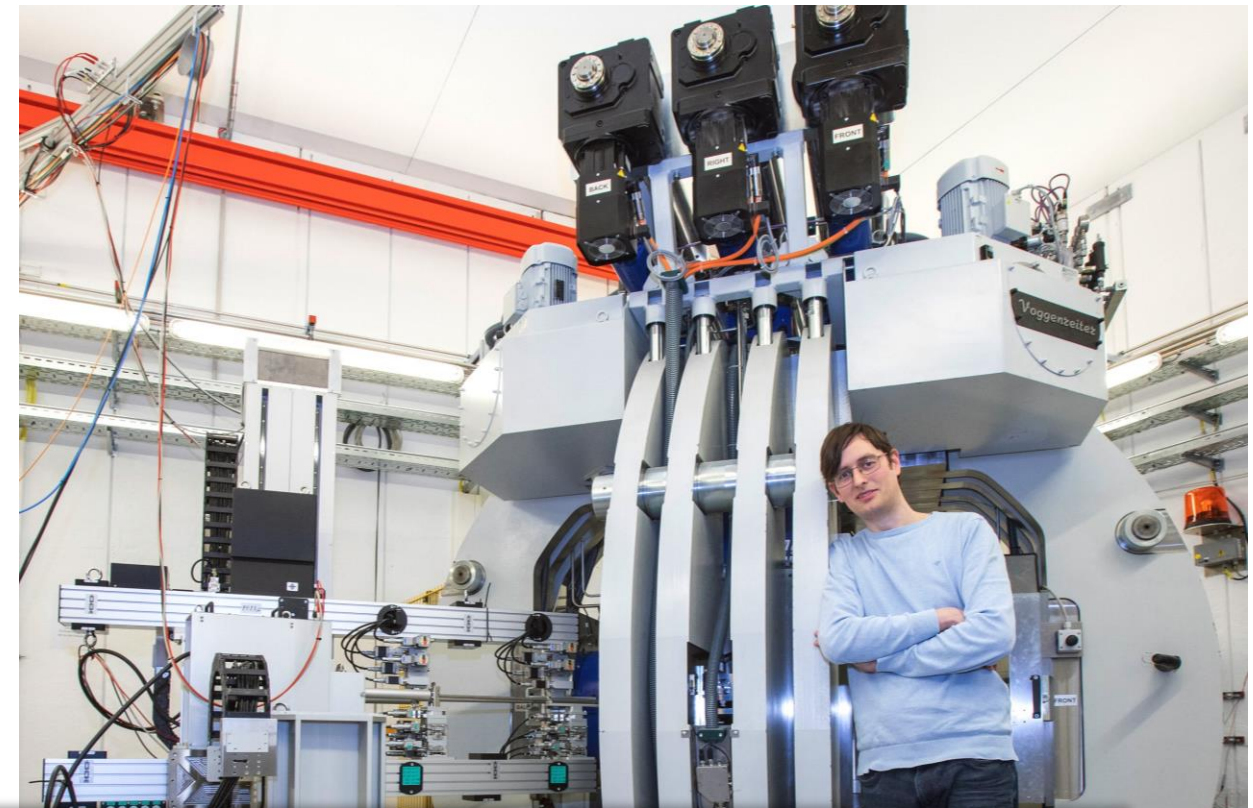
Summary

Dedicated user operation at P61B

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

Support for new *in situ* experiments

1. Controlled rock deformation (2 Ge-SSD)
2. Acoustic Emissions (AE) w/ deformation
3. Ultrasonic wave speed measurements (using 26 mm or 32 mm WC anvils)
4. Falling sphere viscosimetry (w/ GaGG:Ce scint.)



Visit the beamline website

<http://tiny.cc/petra3p61>

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

Looking forward



to your proposals

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**Thank you
for your attention!**