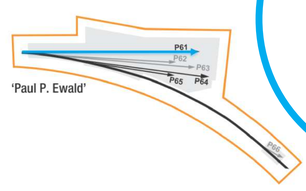
Beamline station P61B: Large Volume Press.

Deutsches Elektronen-Synchrotron DESY
A Research Centre of the Helmholtz Association



Detectors and instrumentation for *in situ* studies at HPHT conditions

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Mission statement

To probe the structure and properties of materials in situ at high pressures and temperatures in a Large Volume Press (LVP) using X-ray diffraction and radiography imaging techniques, as well as other complementary *in situ* techniques.

Applications in geo- and materials sciences:

- Phase relations:
  - Transformation/nucleation
  - Melting curves
  - Equations of state
- Crystallography
- Controlled rock deformation
- Melt viscosity measurements
- Structure of amorphous materials
- Generation of ultra-high pressures (50 GPa) and temperatures (3000 K)

Additional *in situ* methods:

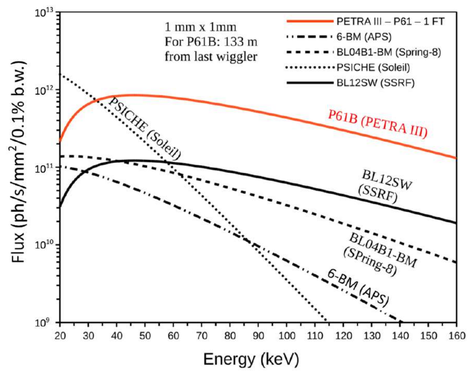
- Ultrasonic wave speed measurements
- Acoustic Emissions detection
- Electrical conductivity measurements

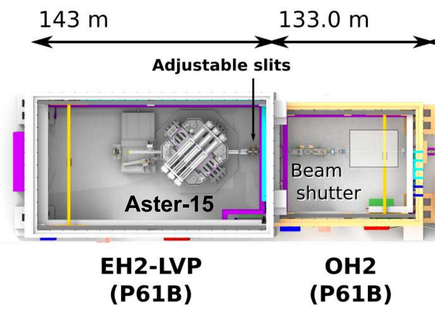
Visit the website!

Beamline paper

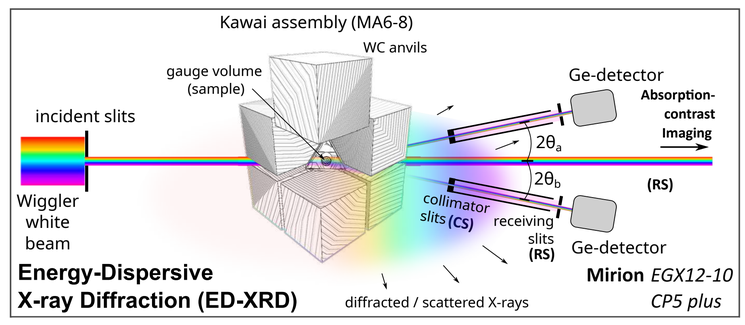
Beamline specifications

Source	10x Damping Wigglers
Array length (m)	60 (10x 4m, 2m gaps)
Period length (mm)	200
# periods	10 x 19
Peak field B0(T)	1.52
Def. parameter K	28.4
Max. power (kW)	10 x 21
At P61B	
Usable energy range	30 - 160 keV
Integrated flux (calc)	~10^{14} ph/s/mm^2/0.1% b.w.
Max. beam size P61B	2.2 mm (h) x 1.6 mm (v)

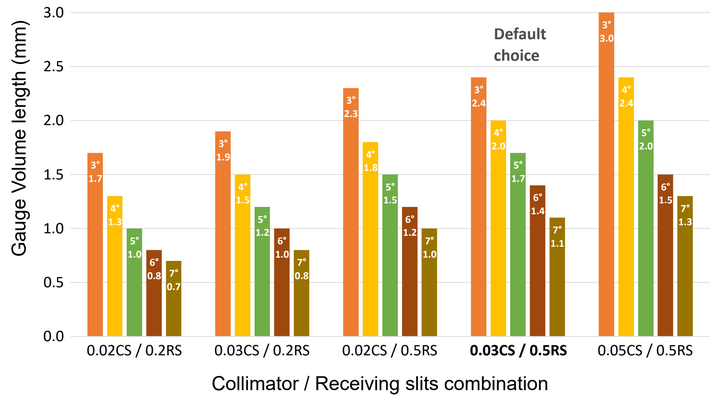




ED-XRD set up using 2x Ge-SSD

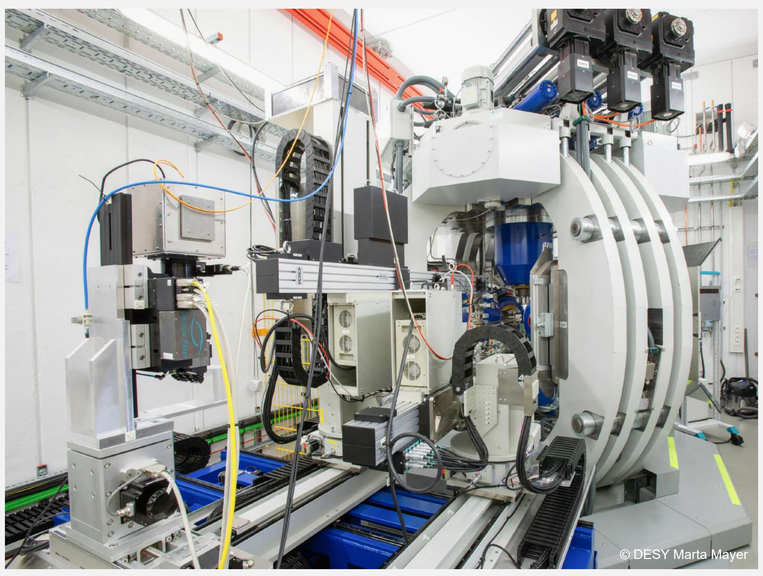


Gauge volume simulations using ray-tracing



High-pressure *in situ* X-ray measurement techniques

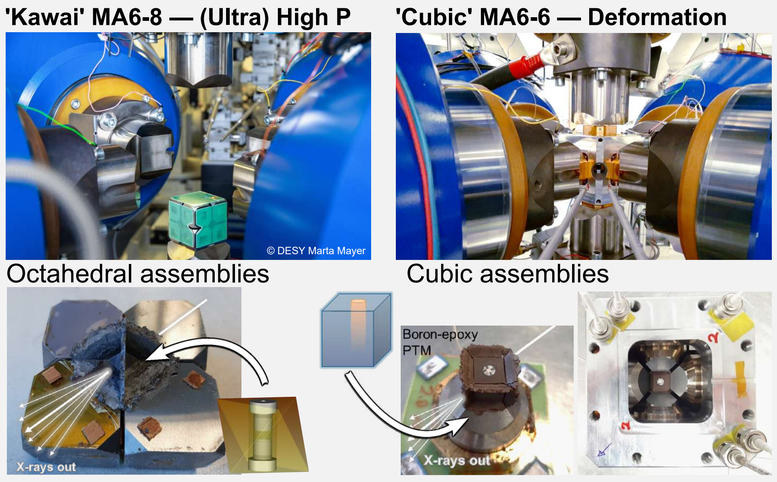
1. The Aster-15 LVP at P61B (ErUM-Pro, Uni. Bayreuth)

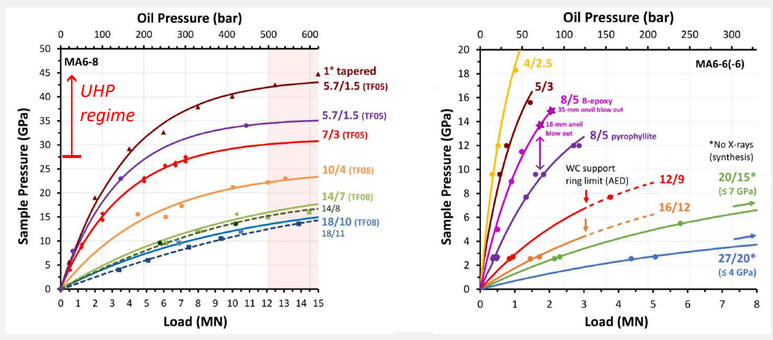


Maximum load	15 MN – 5 MN/axis
Ram position control	1 µm step – 100 mm displacement
Oil pressure control	0.5 bar – 620 bar per ram
Anisotropic compr.	Axial symmetric, triaxial
5-axis stage	x,y1,y2,z (±100 mm), rotation: ±11.5°
Combined weight	ca. 45 ton

2. Versatile compression modes in Aster-15

'Kawai' MA6-8 — (Ultra) High P | **'Cubic' MA6-6 — Deformation**

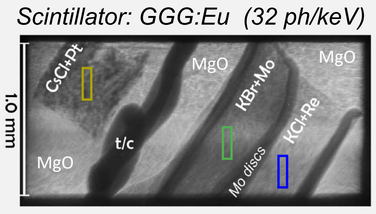




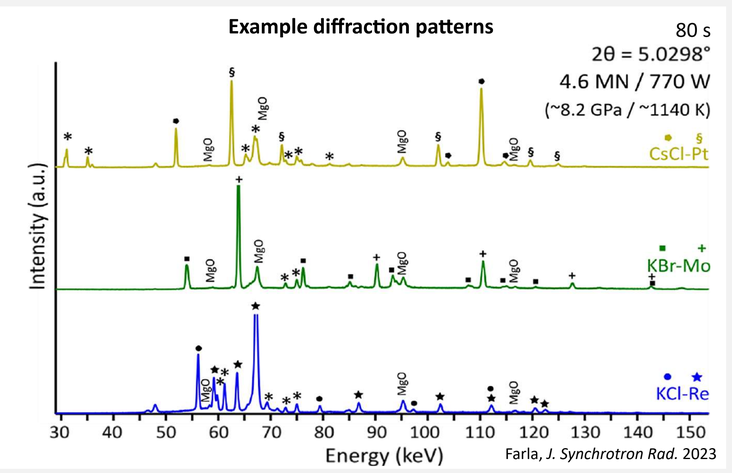
3. Radiography & ED-XRD in LVP Experiments

PCO.Edge
5.5 MP (6.5 µm)
Global shutter

Absorption contrast imaging
Scintillator: GGG:Eu (32 ph/keV)



Example diffraction patterns



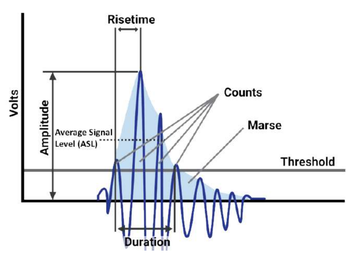
Farla, *J. Synchrotron Rad.* 2023

Multiple materials can be accommodated in the same cell assembly at similar high pressure-temperature (HPHT) conditions for complete structural characterisation by ED-XRD over large ranges of HPHT.

Complementary *in situ* techniques

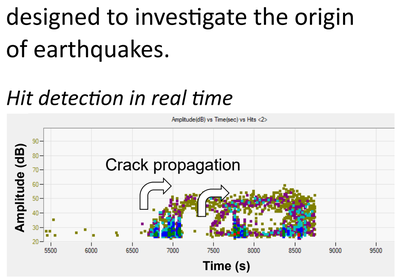
Acoustic Emissions Detection (AED)

AE waveform characteristics

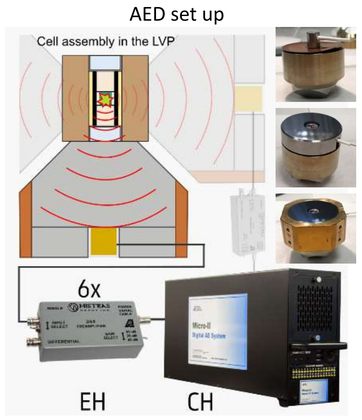


HPHT deformation experiments designed to investigate the origin of earthquakes.

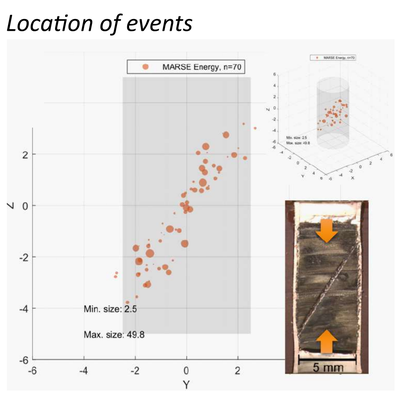
Hit detection in real time



AED set up



Location of events

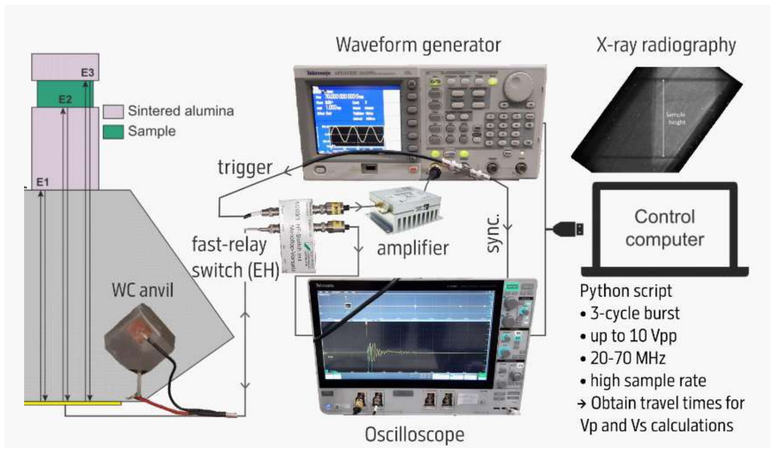


Ma, Gasc, Farla *Rev. Sci. Instrum.* 2023

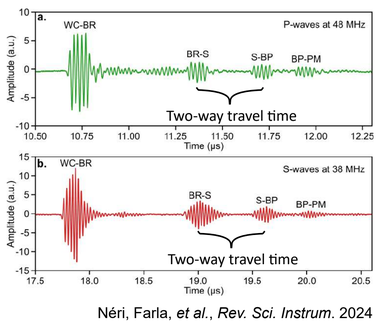
Mistras: Micro-II digital AE system
- 6 Channels, 3 PCI-2 cards
- Sampling rate 40 MS/s. Bandwidth: 0.1 - 3 MHz

Ultrasonic wavespeed measurements (interferometry)

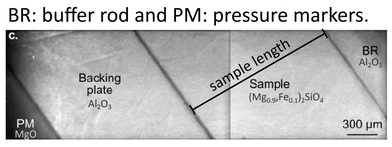
HPHT deformation experiments designed to investigate the seismic structure of the Earth's interior as well as elastic properties of materials.



Right side. Typical P- and S-waveforms in the oscilloscope obtained at 2.9 MN load and 900 °C. *Wavespeed = distance/time!*



Below. Typical radiography image, where BR: buffer rod and PM: pressure markers.



Néri, Farla, *et al.*, *Rev. Sci. Instrum.* 2024

Key science highlight

Depressed 660-km discontinuity caused by akimotoite-bridgmanite transition

Chanyshev *et al. Nature* (2022) **https://doi.org/10.1038/s41586-021-04157-z**

Phase boundaries of $MgSiO_3$ akimotoite-bridgmanite transition (green line) and Mg_2SiO_4 post-spinel transition (purple line) and stable phases in Mg_2SiO_4 system.

Major upcoming development

Laue monochromator for AD-XRD
using a Varex 4343CT & Manta camera

Selectable energies: 45 – 80 keV

Design: Stefan Sonntag

ETA: 2025-II

Bundesministerium für Bildung und Forschung
ErUM-Pro
HELMHOLTZ
BGI Bayerisches Geoinstitut
UNIVERSITÄT LEIPZIG
Universitet Stockholms
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