In situ X-ray measurements on mm-sized samples at high p-T in the Large Volume Press at P61B, PETRA III

SRI Conference

PS16.1 New opportunities in high-pressure research

Robert Farla
28.03.2022

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)

All DESY Support Groups are gratefully acknowledged

HELMHOLTZ RESEARCH FOR GRAND CHALLENGES · BGI Bayerisches Geoinstitut · UNIVERSITÄT LEIPZIG

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 (later w/ CAESAR or monochromator)
- Controlled rock deformation
- Melt viscosimetry measurements
- Structure of amorphous materials

For 50% experiment time:

- Synthesis of novel (recoverable) materials
  - Band-gap tuning/semiconductors, optical windows, super-hard/conductive, catalyzers, hydrogen storage
- In-house tests/research
- ‘Rapid access’ (short-term proposal, no external review)

Complementary *in situ* techniques:

- Ultrasonic interferometry
- Acoustic Emissions testing
- Electrical conductivity (pending)

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

Artem Chanyshev[1,2✉], Takayuki Ishii[2,3✉], Dmitry Bondar[2], Shrikant Bh
Robert Farla[1], Keisuke Nishida[2], Zhaodong Liu[2,4], Lin Wang[2,5], Ayano I
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

Longjian Xie,[1,2,a)] Artem Chanyshev,[1,3] Takayuki Ishii,[1,4] Dmitry Bondar,[1] Keisuke Nishida,[1] Zhen Chen,[4] Shrikant Bhat,[3] Robert Farla,[3] Yuji Higo,[5] Yoshinori Tange,[5] Xiaowan Su,[6] BingMin Yan,[4] Shuailin Ma,[3,4] and Tomoo Katsura[1]

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

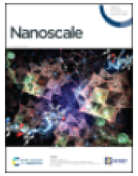
ORIGINAL PAPER

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]

P61B LVP Mission

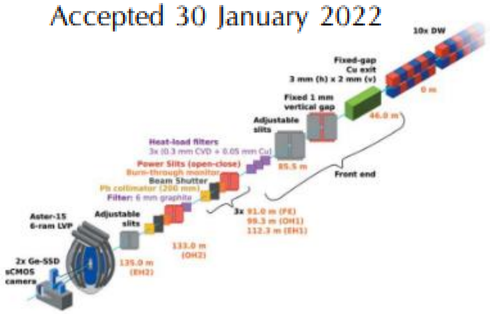
First publications using X-rays

Article
Nature | Vol 601 | 6 January 2022 | 69

Depressed 660-km discontinuity caused by akimotoite–bridgmanite transition

JOURNAL OF SYNCHROTRON RADIATION
ISSN 1600-5775

Accepted 30 January 2022



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

Robert Farla,[a*] Shrikant Bhat,[a] Stefan Sonntag,[a] Artem Chanyshev,[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 Seeck[a] and Tomoo Katsura[b]

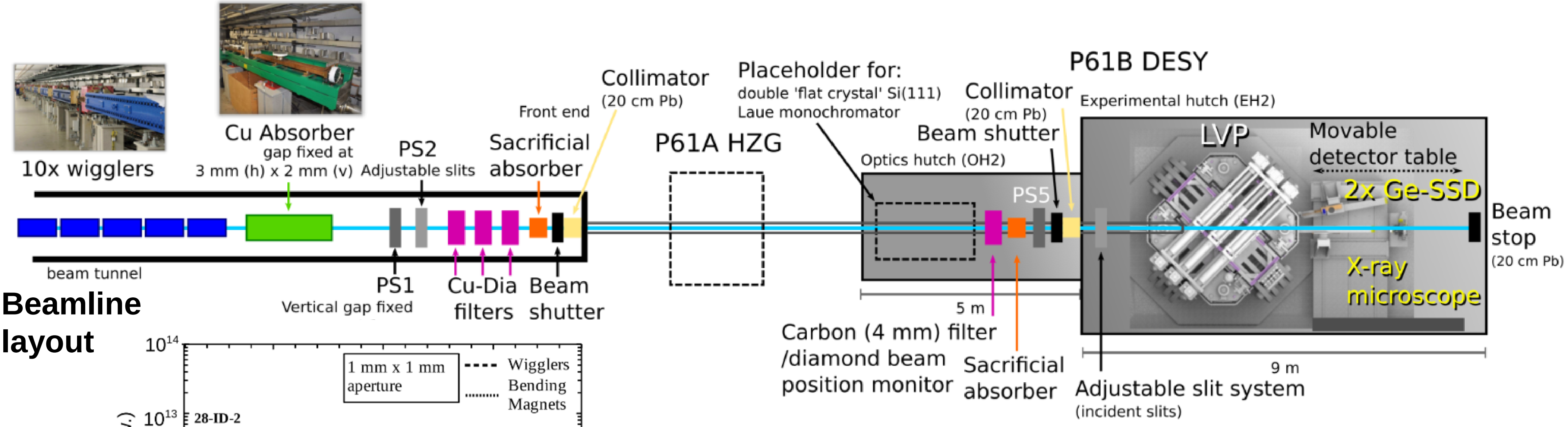
Shrikant Bhat[1] · Robert Farla[1] · Tomoo Katsura[2,3]

Received: 31 March 2021 / Accepted: 11 August 2021

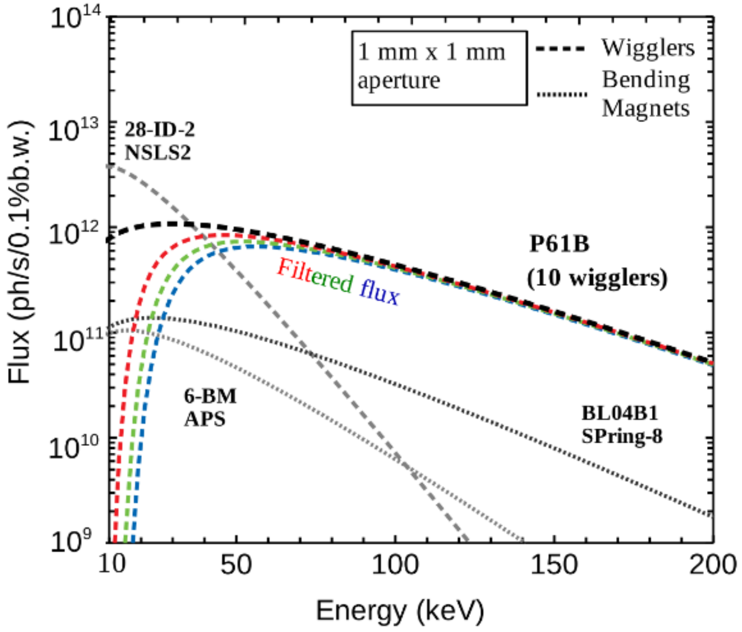
Shuailing Ma,[ab] Robert Farla,[b] Kuo Bao,[*a] Akhil Tayal,[b] Yongsheng Zhao,[ab] Qiang Tao,[a] Xigui Yang,[c] Teng Ma,[a] Pinwen Zhu[a] and Tian Cui[*ad]

Beamline layout

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



Beamline layout



Flux at P61

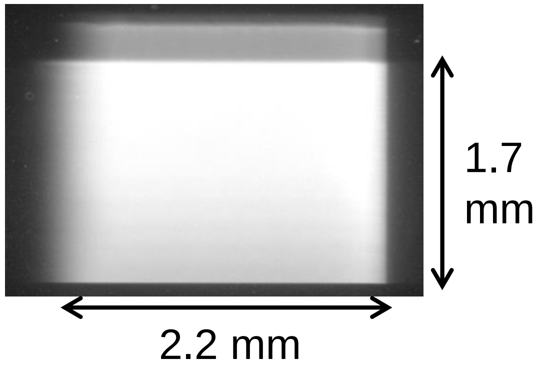
Peak at

~1 x 10^{12} ph/s/mm^2 @ 50 keV

Integrated flux (1FT)

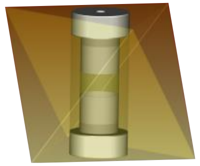
~3 x 10^{14} ph/s/mm^2

Full beam size



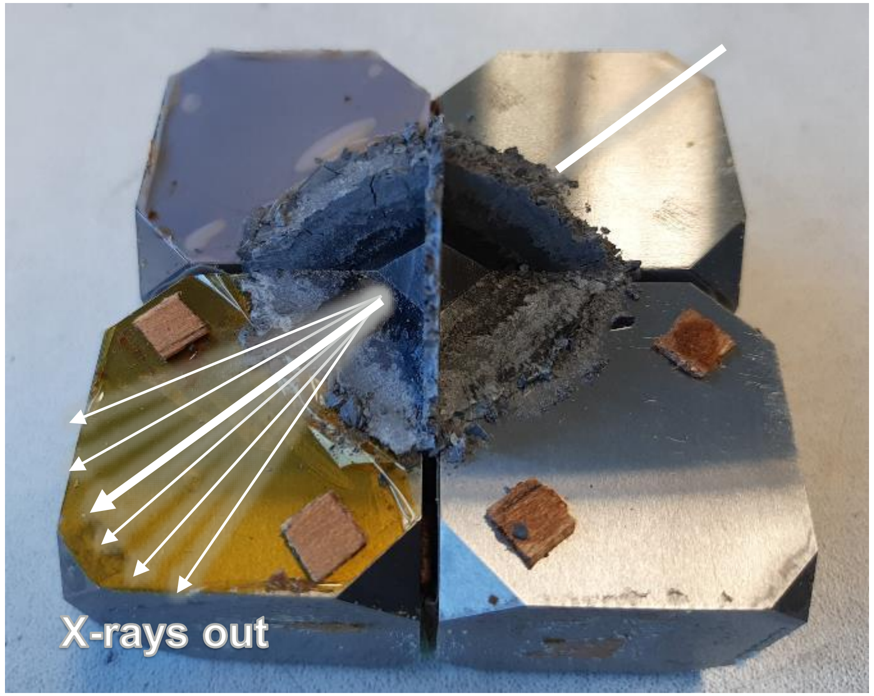
High-pressure techniques

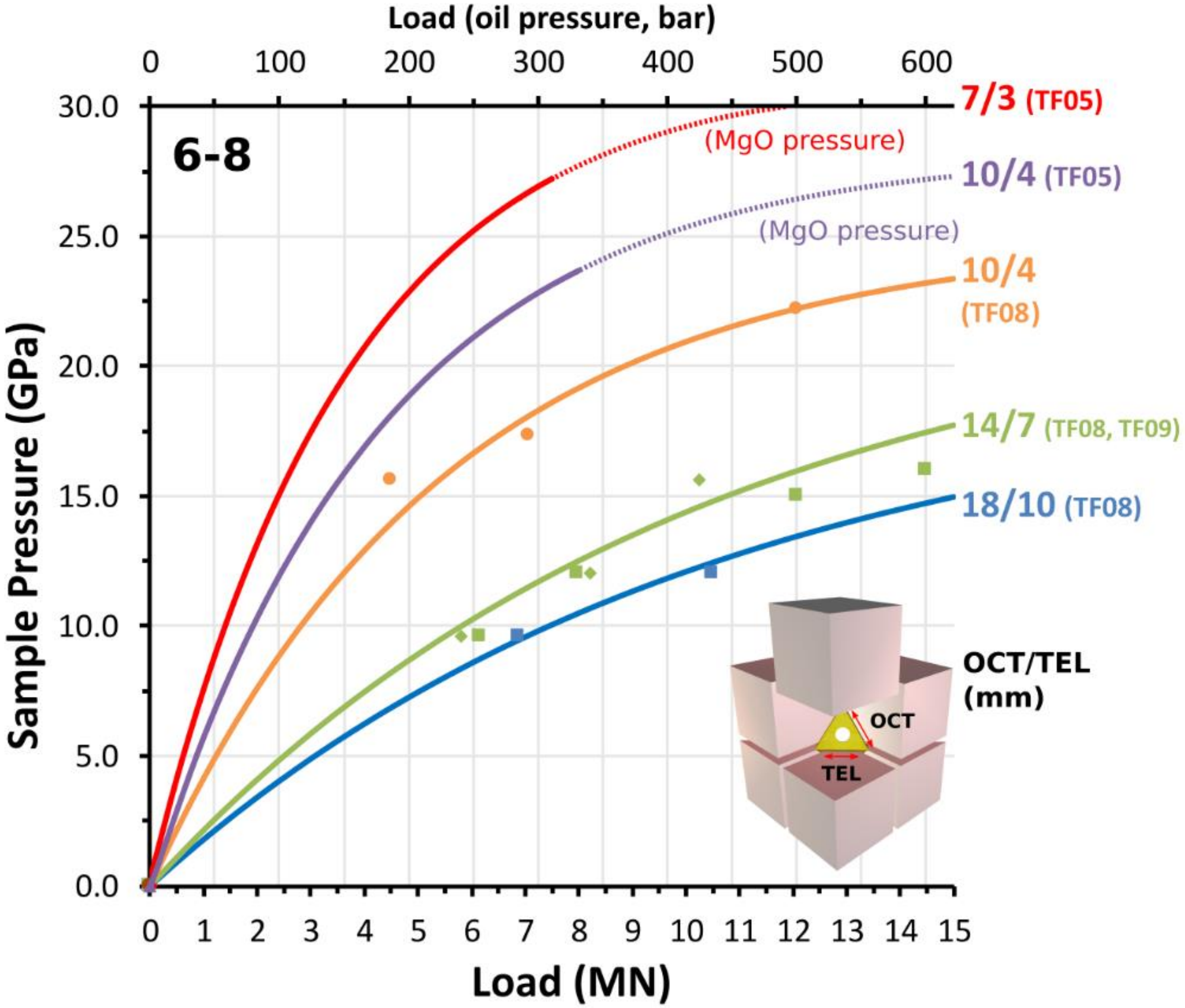
Standard assemblies for *in situ* hydrostatic high-pressure experiments



‘Kawai’ 6-8 mode

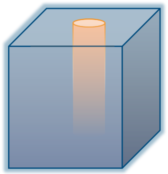
Recovered assembly after compression



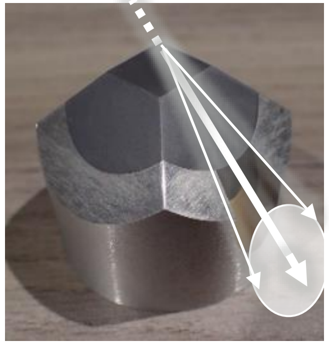


High-pressure techniques

Standard assemblies for *in situ* studies of rock deformation



'Cubic' 6-6 mode (p = 0.5 - 4 GPa)

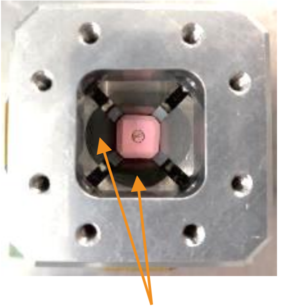


New! Large cBN anvil (X-ray transparent)

Compatible with Acoustic Emissions (AE) detection

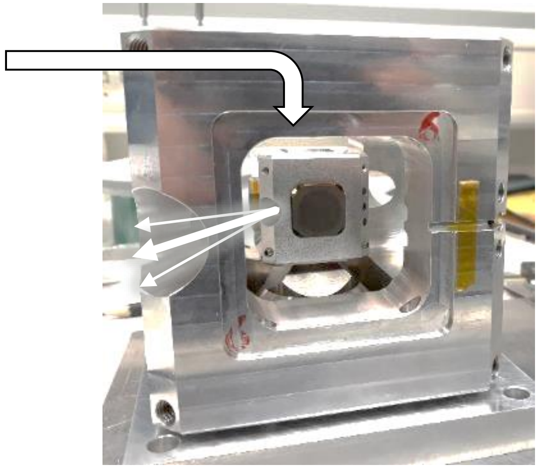
'Cubic' triple-6 mode (p > 5 GPa)

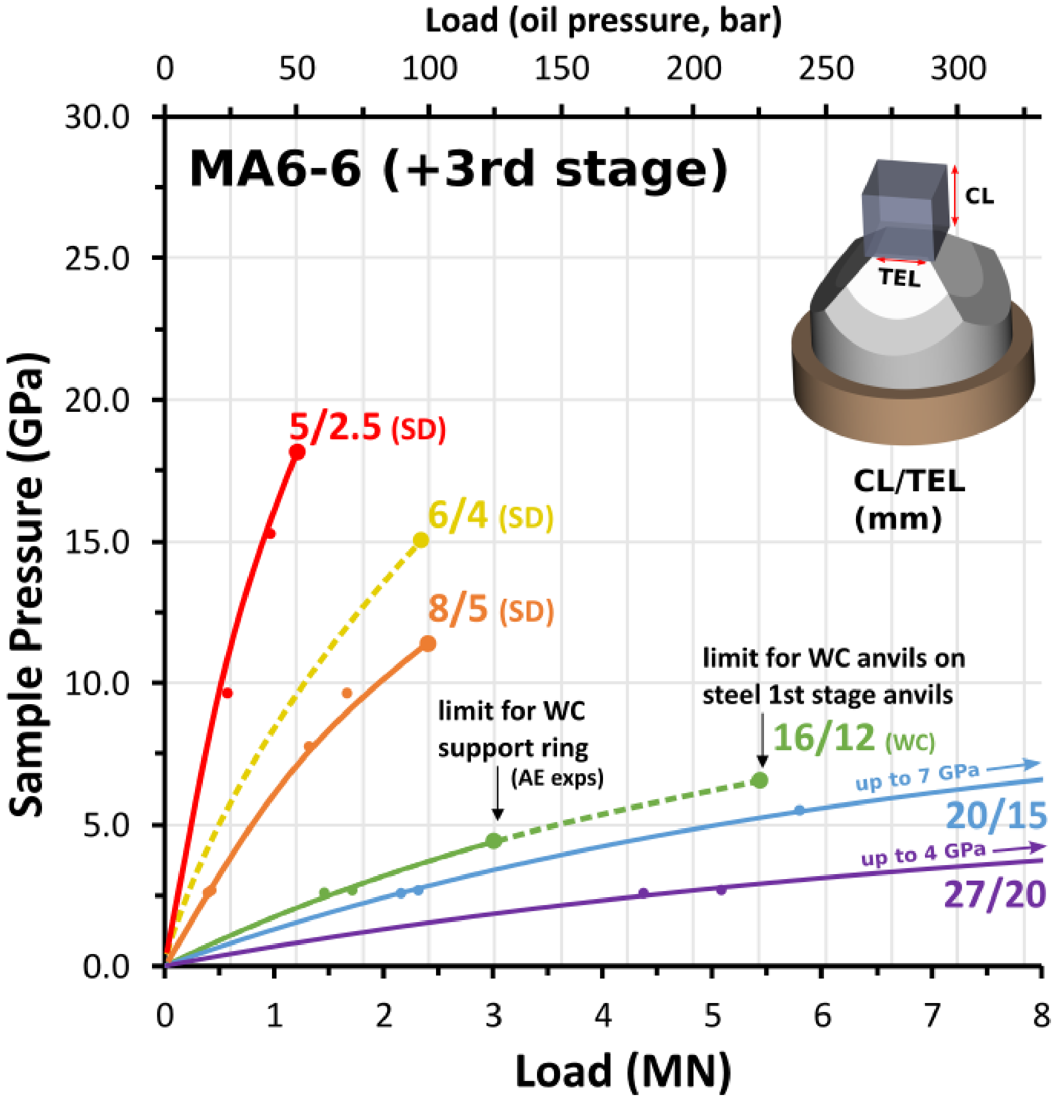
← 38 mm →



X-ray transparent sintered diamond anvils

← 110 mm →





Generally for *ex-situ* synthesis

The whitebeam X-ray microscope

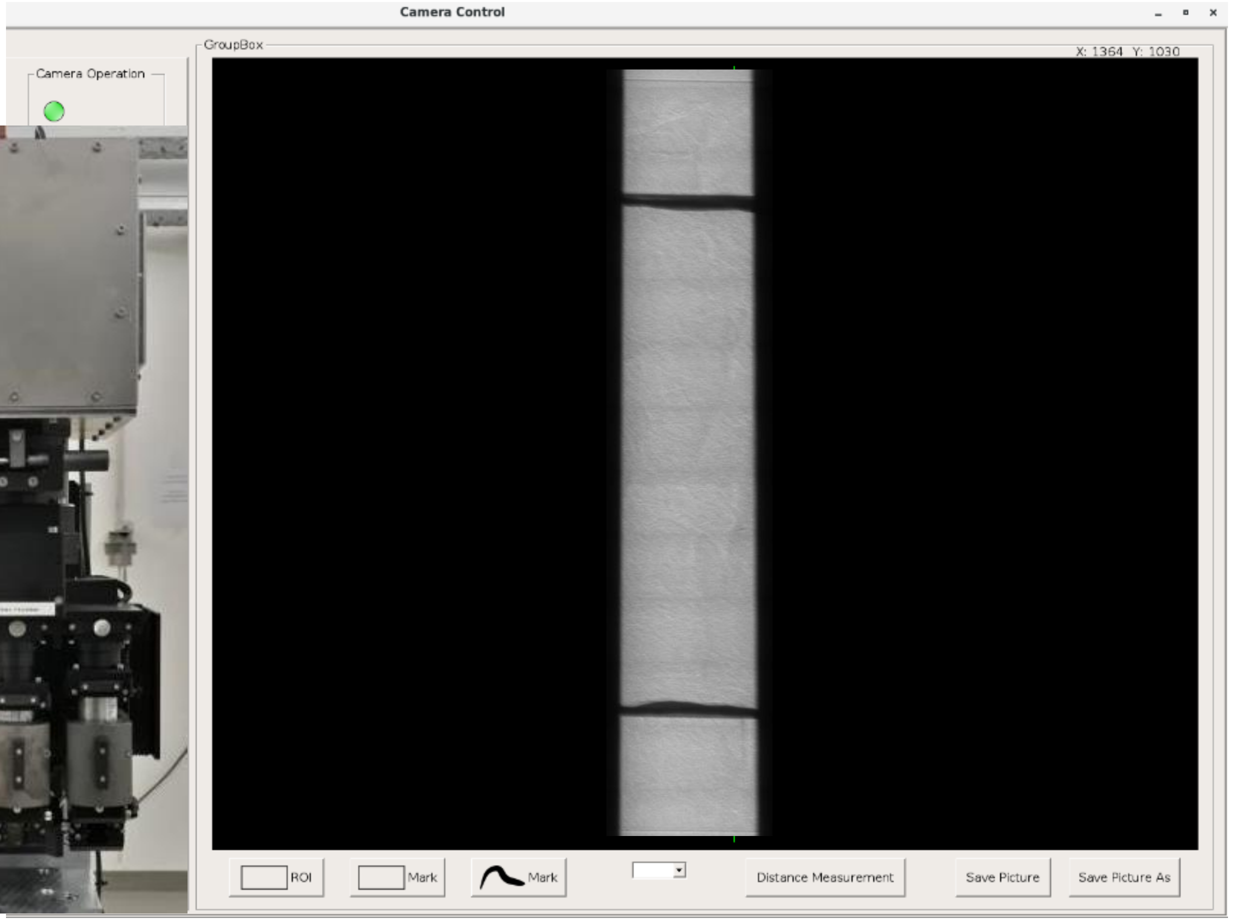
X-ray radiography

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

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

- **Scintillators (thickness):**
  - **GGG:Eu**
    20, 40 µm
  - **LuAG:Ce**
    20, 40 µm
  - **GaGG:Ce-HL**
    150, 200 µm, ultra-bright

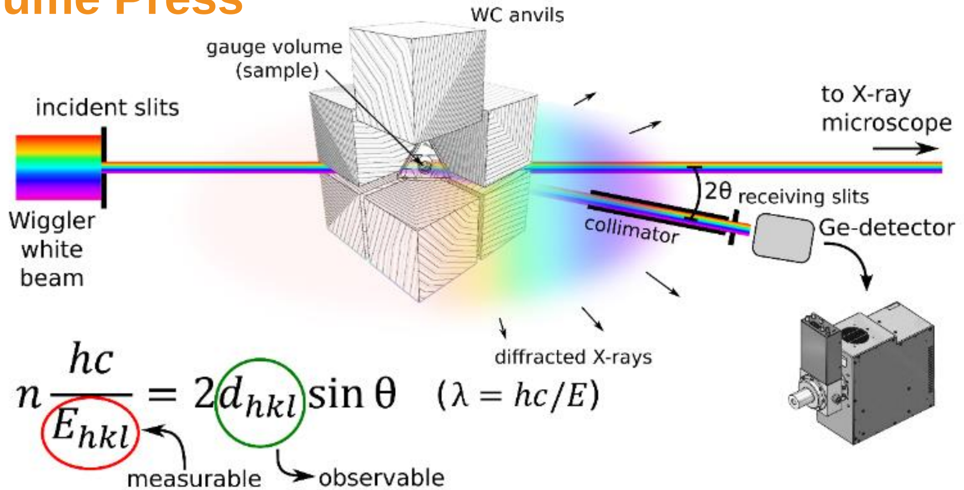
pco. Optique Peter OPTICAL & MECHANICAL ENGINEERING



X-ray powder diffraction using white beam

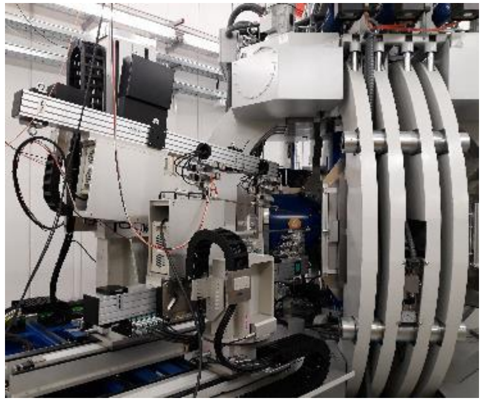
Energy-dispersive X-ray diffraction (ED-XRD) in the Large Volume Press

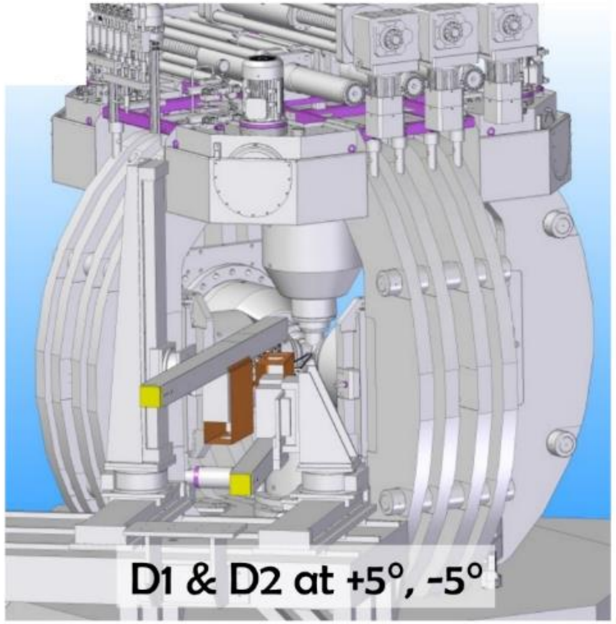
1. High spatial resolution (define gauge volume)
   → avoid high temperature & pressure gradients
   → multiple samples in one experiment
2. Ideal for low-Z (X-ray transparent) samples.
3. Fast acquisition (10-100 s) covering large Q-range.

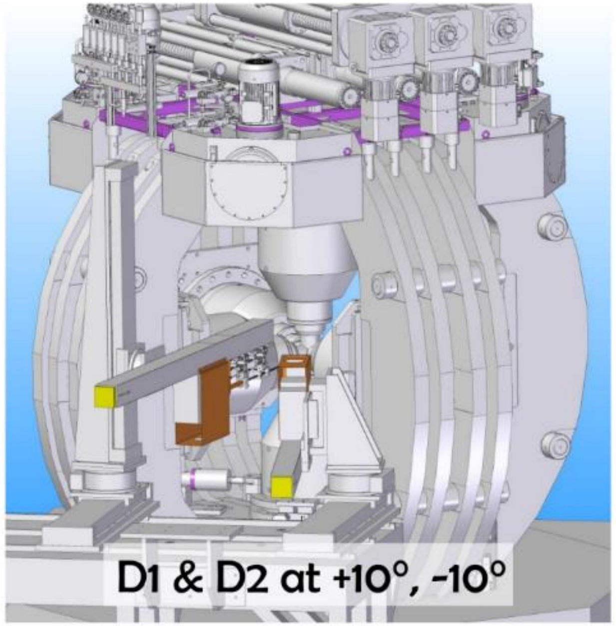


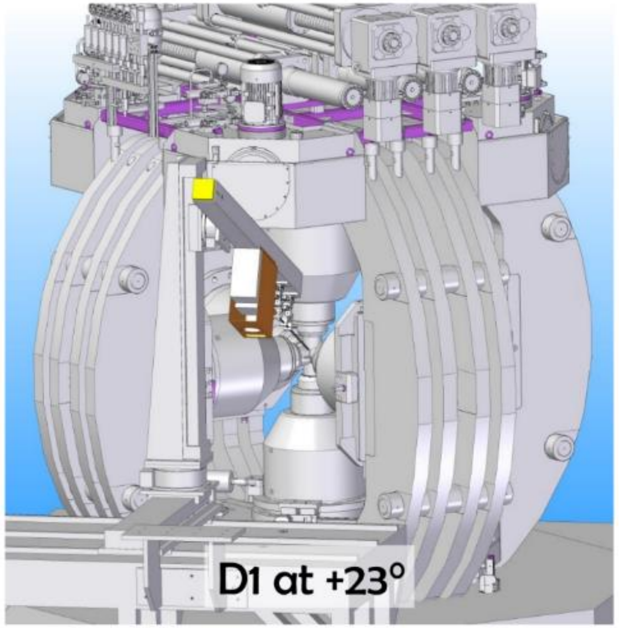
$$n\frac{hc}{E_{hkl}} = 2d_{hkl}\sin\theta \quad (\lambda = hc/E)$$

Various measurement positions





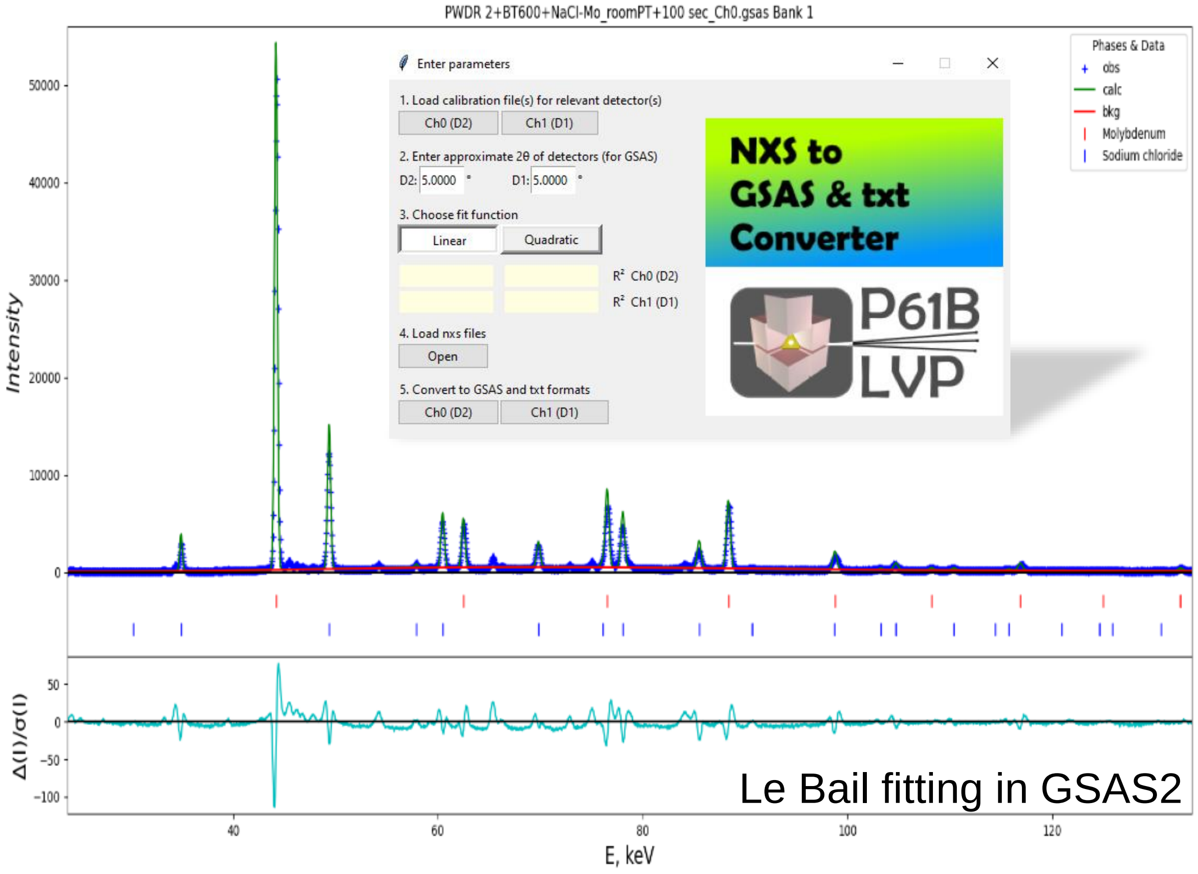




Beamline software tools

Available from the website

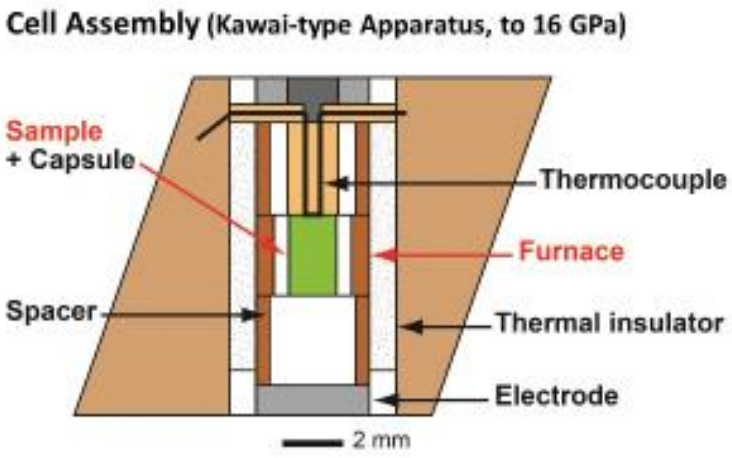
- **Data file conversion**
  HDF5 (nxs) → txt and GSAS2 formats



Le Bail fitting in GSAS2

- **Simultaneous P and T estimation** in a cell assembly using pressure standards

Why? To simplify the HP assembly



Problems with thermocouple

- Can break any time / report false readings
- Does not measure true sample T
  - Typical > 30 °C gradient
- Requires (unknown) pressure correction on emf
- Disturbs pressure distribution / adds stress
  - or hot spots/instability in case of drilled furnace

Beamline software tools

Simultaneous P and T estimation in cell assembly using pressure standards

Ideal PT marker candidates are :

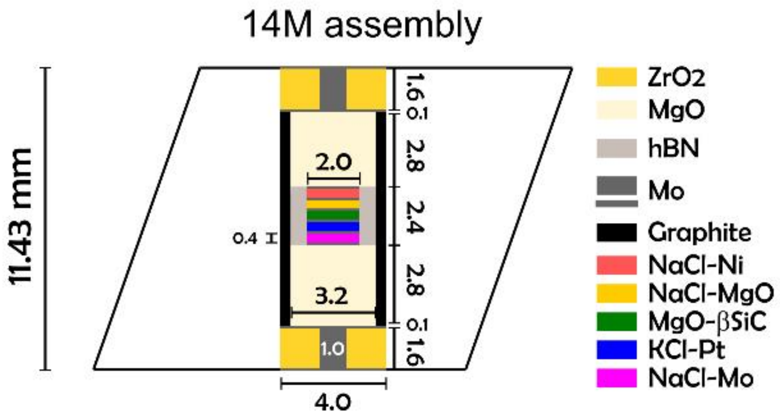
- Highly symmetric (cubic structure),
- Plastically isotropic,
- Non-reactive,
- Stable over large PT ranges,
- Good compressibility or thermal pressure (i.e. expansion)

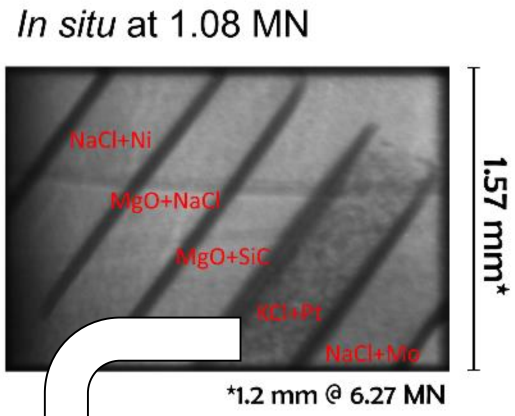
Example combinations:

High compressibility, low thermal pressure
- NaCl (B1/B2), KCl and other salts/halides

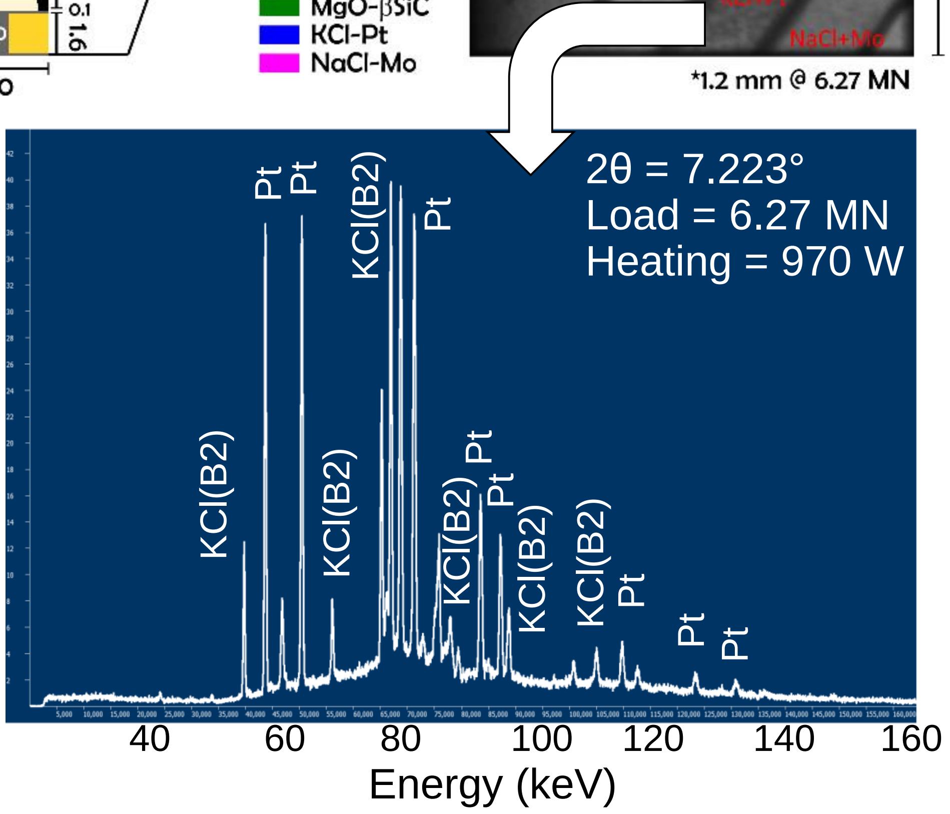
Moderate compressibility, thermal pressure
- SiC (cubic form)
- MgO

Low compressibility, high thermal pressure
- Many metals, including Pt, Au, Ni, …





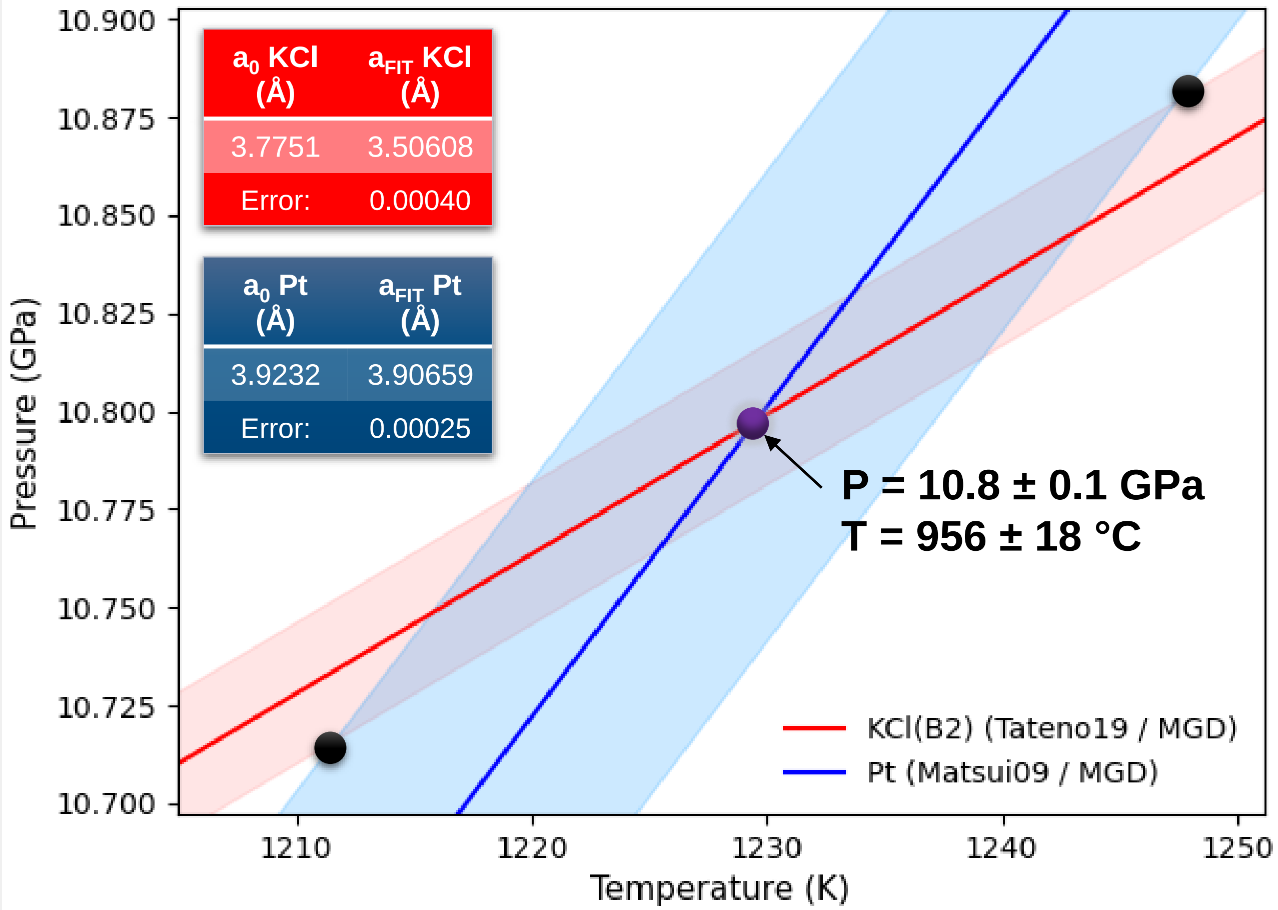
Example experiment



Beamline software tools

Simultaneous P and T estimation in cell assembly using pressure standards

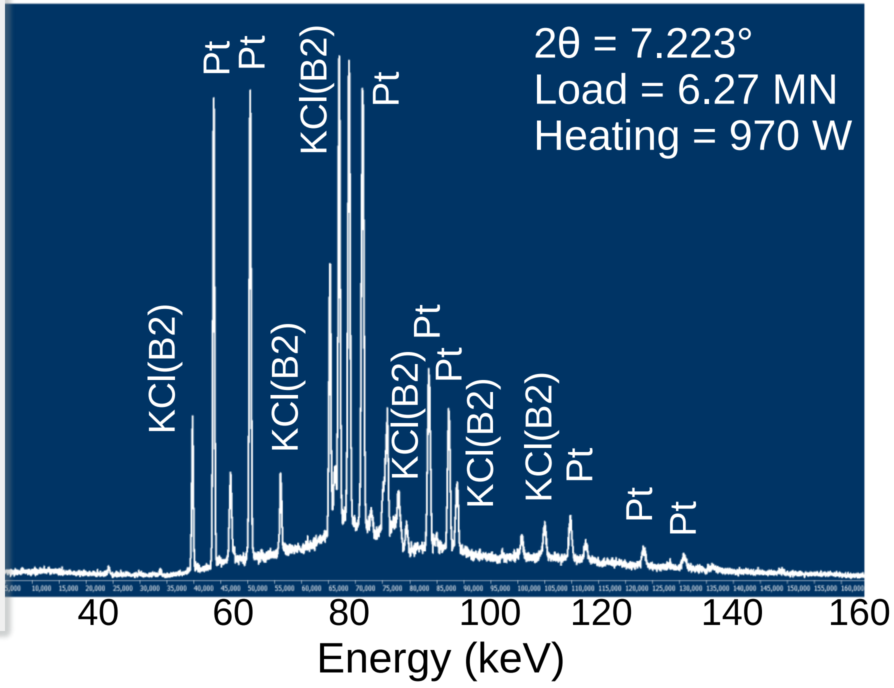
Peak fitting to get lattice parameters and calculate P & T
(KCl: Tateno *et al.* 2019 *Am. Min.* Pt: Matsui *et al.* 2009 *J. Appl. Phys.*)



a_0 KCl (Å)	a_{FIT} KCl (Å)
3.7751	3.50608
Error:	0.00040

a_0 Pt (Å)	a_{FIT} Pt (Å)
3.9232	3.90659
Error:	0.00025

Least parallel isochors minimize PT uncertainties for equivalent errors in the lattice constants from a best fit



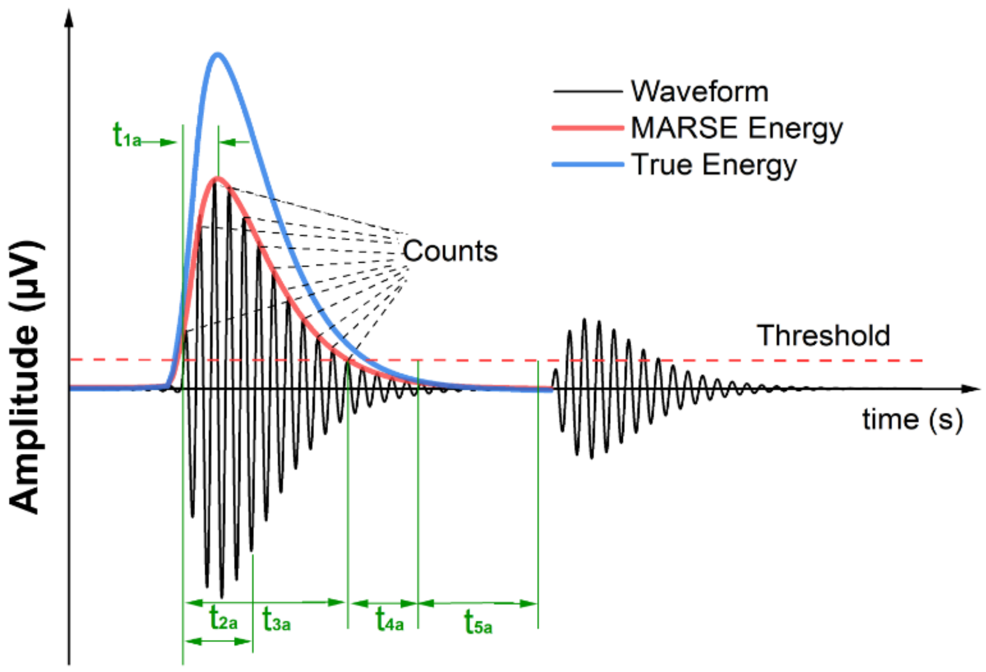
Acoustic Emissions testing

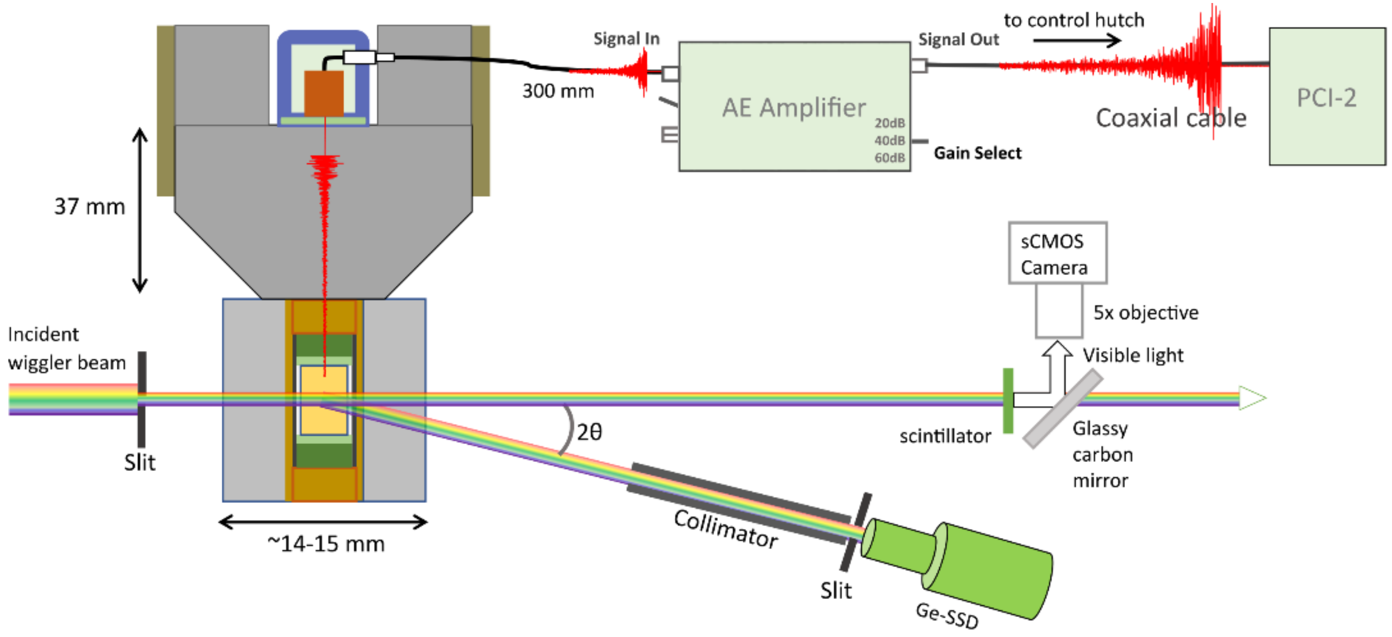
Methodology (MA6-6 compression)

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

AE characteristics

- Amplitude
- MARSE energy
- Counts
- Av frequency
- Rise time
- 1st threshold crossing





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

→ 6 near-simultaneous hits, form an event in 3d space
→ ED-XRD on sample to obtain phase and stress history
→ Radiography to obtain macro-strain history

Explore brittle dehydration reactions in Earth → Earthquakes!

Acoustic Emissions testing

Methodology (MA6-6 compression)

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

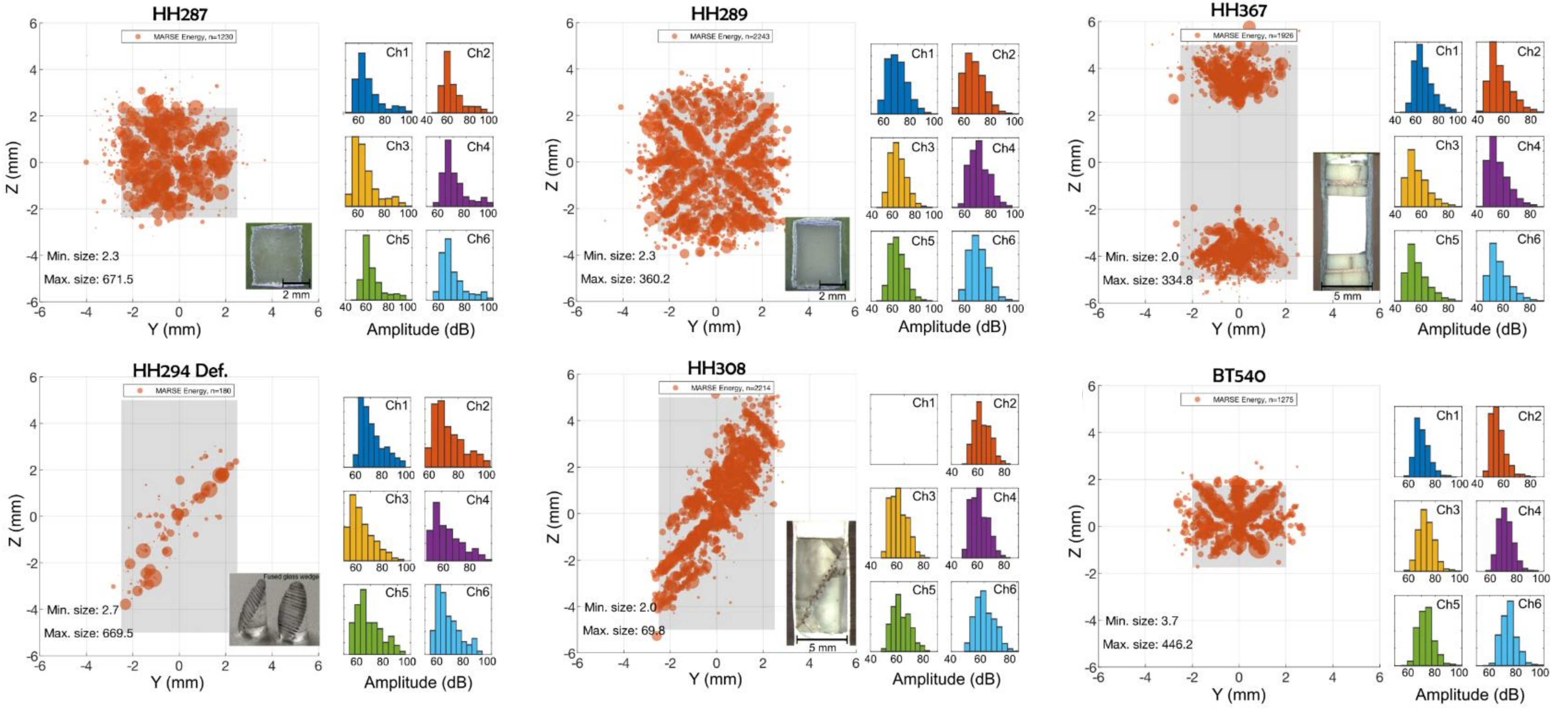
‘16/12’ AE assembly in the LVP

Acoustic Emissions testing

Some results on cracking of silica glass *in situ*

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

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

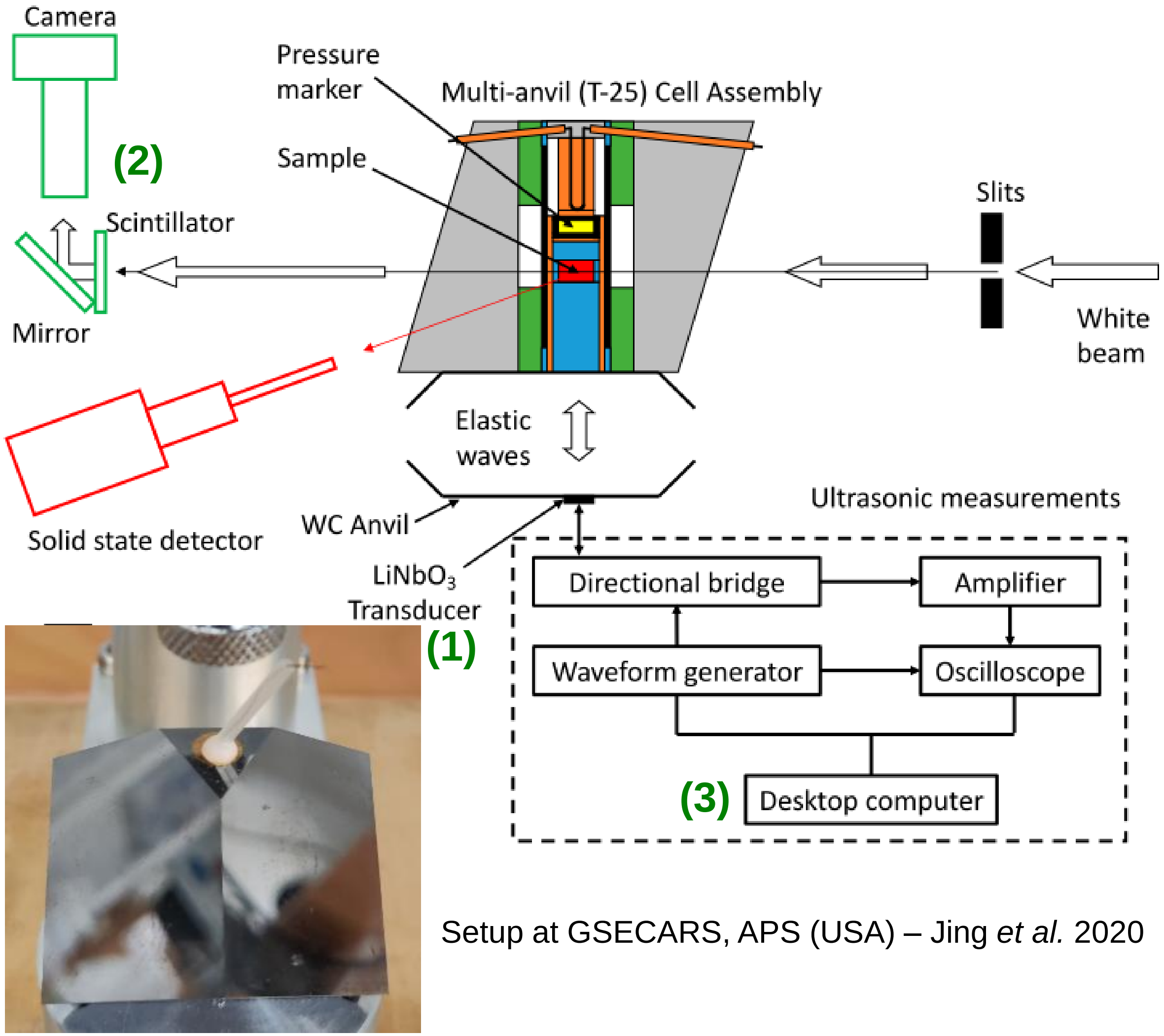


Manuscript in preparation

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

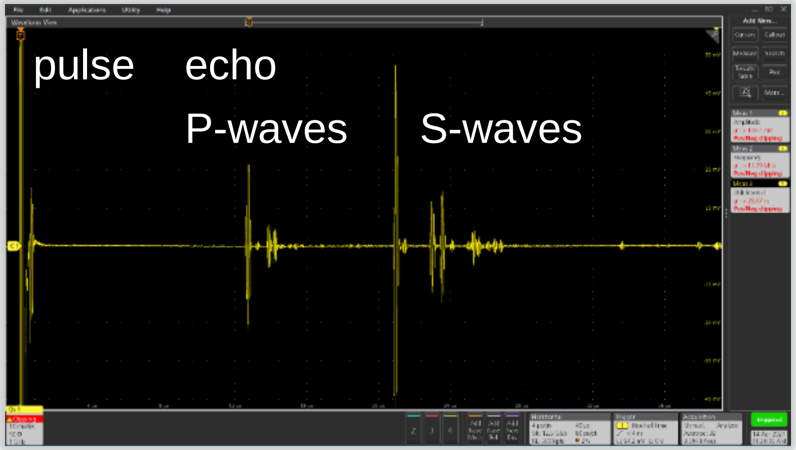
(1) A LiNbO3 sensor of choice on the back of a mirror polished anvil, transmits a pulse and receives an echo.

(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 is scripted using python.



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 (200+ kcps), 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 cubes)
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 activities**
- **And more…**

Thank you for your attention!

Look for a poster by Dr. Christian Lathe in CMWS

Contact

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