# Large Volume Press (LVP)

## Exploration of high pressures and temperatures on mm-sized samples at PETRA III and IV

**R. Farla**
Hamburg, 03/11/2020

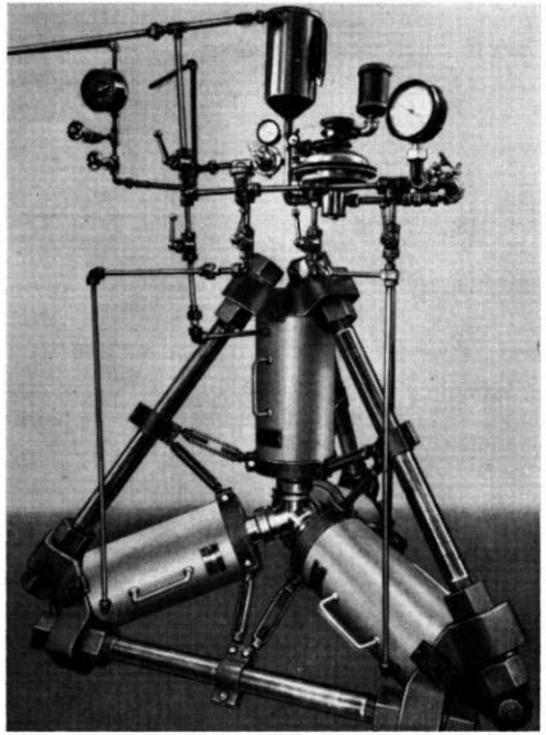
PETRA IV Workshop - Earth, Environment, and Materials for Nanoscience and Information Technology

HELMHOLTZ RESEARCH FOR GRAND CHALLENGES

DESY.

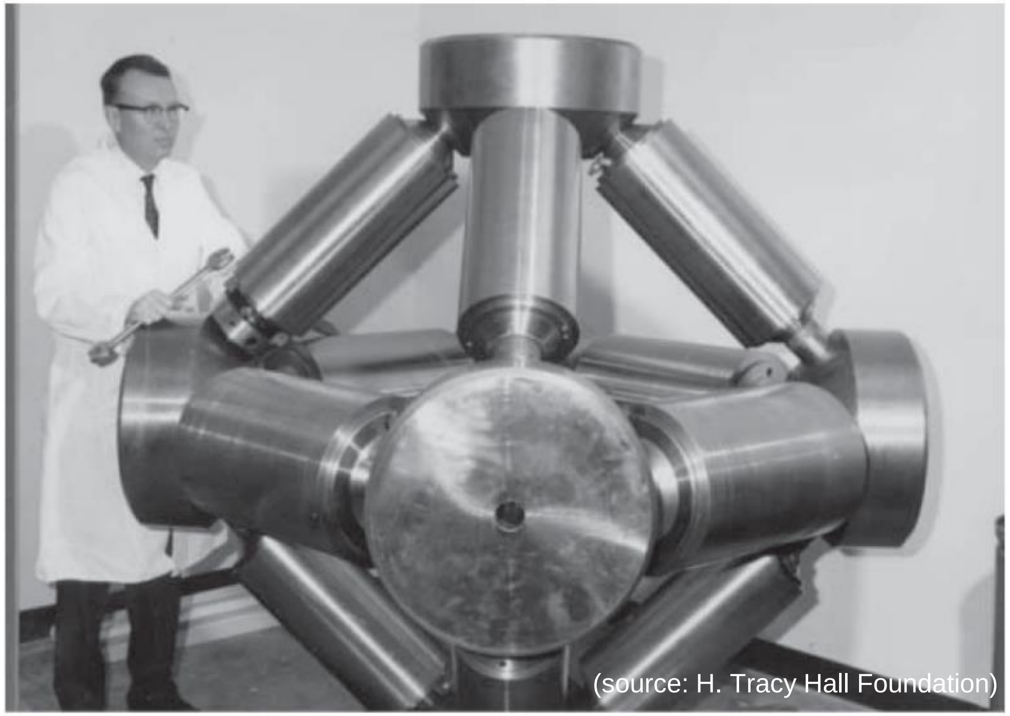
# A brief history of multi anvil, high-pressure devices

## From diamond synthesis to multi-disciplinary research using synchrotron radiation



**1958**. The original tetrahedral-anvil apparatus built by H. Tracy Hall at Brigham Young University (BYU)

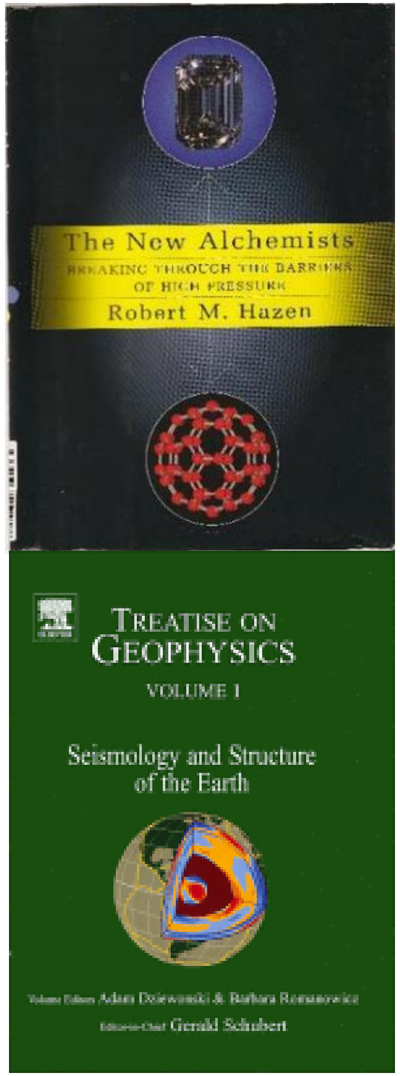
H. Tracy Hall worked at GE in 1955. When he returned to BYU, he was banned to use the belt-type apparatus to synthesize diamond, so he invented the first generation of multi-anvil apparatus (above and to the right)



(source: H. Tracy Hall Foundation)

**1967**, H. Tracy Hall & the first cubic anvil apparatus developed at BYU, USA to produce diamonds without the belt apparatus

$P_{max}$ = 10 GPa
$T_{max}$ > 3000 K



# A brief history of multi anvil, high-pressure devices

## From diamond synthesis to multi-disciplinary research using synchrotron radiation

### Brief history of meetings

- 1955: first **GRC** (Gordon Research Conferences), USA
- 1963: first **EHPRG** (European High Pressure Research Group) meeting, Harlow, UK.
- 1965: first European/International effort, **AIRAPT** ("Association Internationale pour Avancement de la Recherche et de la Technologie aux Hautes Pression")
- 1976: first US–Japan bilateral seminars on high pressure mineral physics, now International meeting, **HPMPS**.

1968. First Japanese tetrahedral LVP
(photo taken in 1991 at ISSP)

MAX-80: multi-anvil-type X-ray system, 1980

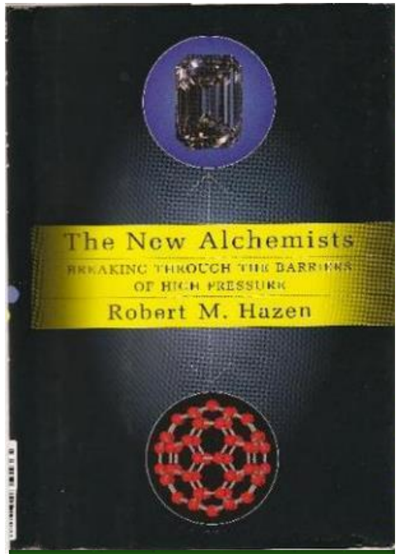
## A brief history of advances

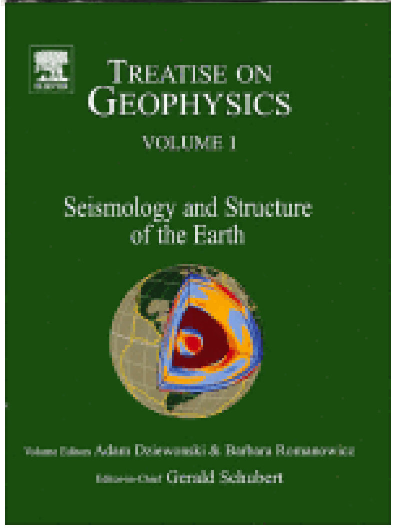
1964, USA, Barnett and Hall interfaced the tetrahedral-anvil apparatus with a laboratory X-ray source to perform *in situ* X-ray diffraction studies for the first time.

1968, Japan, Akimoto wanted to study the Earth's transition zone (400 – 700 km, P > 13 GPa) and built the first Japanese tetrahedral press (on the left).

1979, Japan, Ohtani et al. achieved *in situ* XRD using Philips Mo target tube to calibrate target pressures up to 22 GPa (GaP transition).

Switch to synchrotron sources for *in situ* XRD in LVP:
1980, MAX-80 at PF (KEK), Japan
1991, MAX-80 at DORIS III, HASYLAB, Hamburg
1992, SAM-85 at X17B2, NSLS, USA
1998, SPEED-1500 / Mk.II at SPring-8, Japan



# A brief history of multi anvil, high-pressure devices

## From diamond synthesis to multi-disciplinary research using synchrotron radiation

### Primary motivation:

The desire to study the behaviour of materials at simultaneous pressures and temperatures.

**→ Earth Science community aims to replicate in the laboratory the P–T conditions of the Earth’s deep interior.**

### The Diamond Anvil Cell (DAC) VS LVP

The race to the highest pressures was won by use of DAC (in 1976). However, more parameters are important:

- uniformity and volume of uniform pressure
- hydrostaticity, deviatoric stress (when desirable)
- pressure gradient (when desirable)
- accuracy, uniformity & temporal constancy of temperature, gradients (when desirable)
- access for X-rays

### The Hall-type LVP at PETRA III, DESY

MAVO press LPQ6-1500-100
Built by Voggenreiter, GmbH
(Mainleus, Germany)

Installed at P61 since 2015 featuring 6 independently controlled, hydraulically driven rams.

Specs:

- Max. 5MN force per axis, at 620 bar.
- Ram stroke: 100 mm.
- Control accuracy: +/- 1 um / 0.5 bar
- Compress rate: 0 – 100 bar/min
- 5-axis stage below press

# P61B LVP Mission

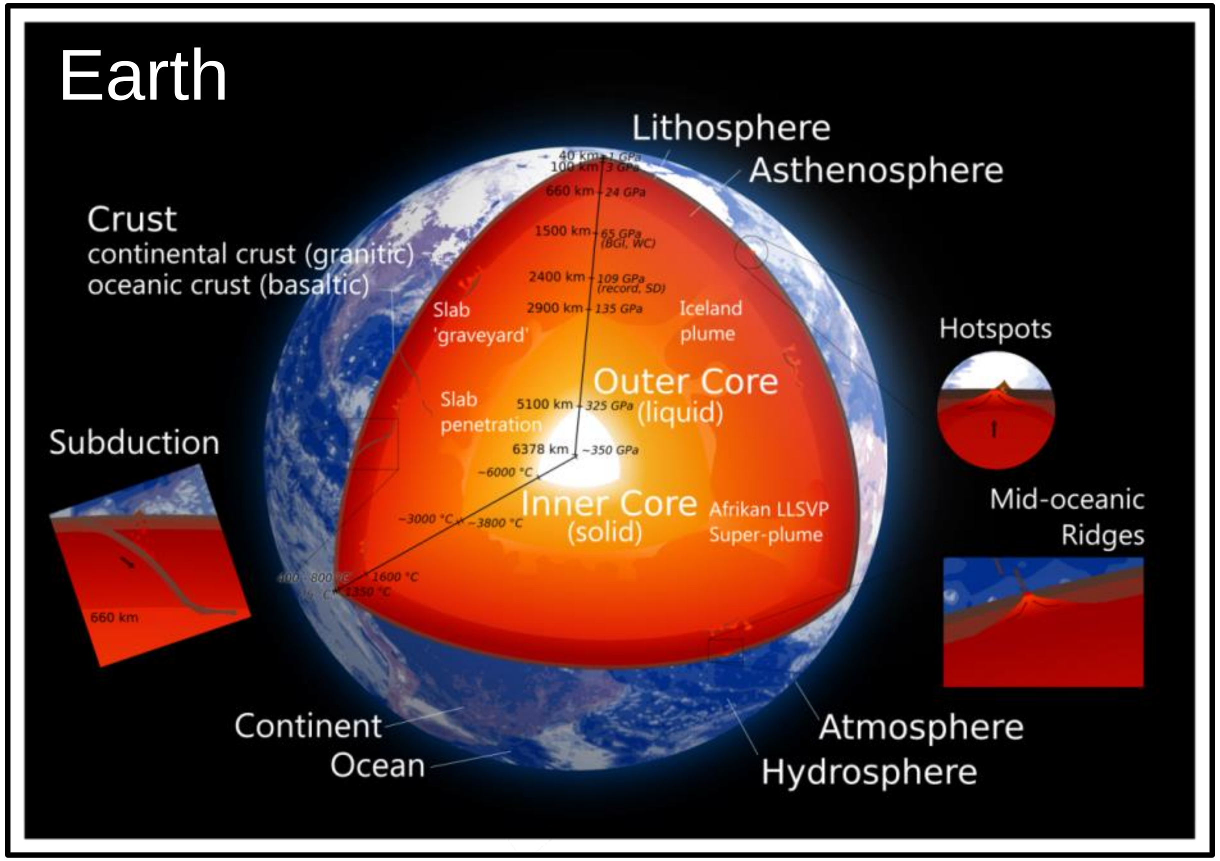
**XRD and imaging in geo- and material sciences:**

- 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




**P02.2 ECB**

- Extreme pressures (1 TPa)
- Small (0,001 mm$^3$) samples
- Single phase (typically)

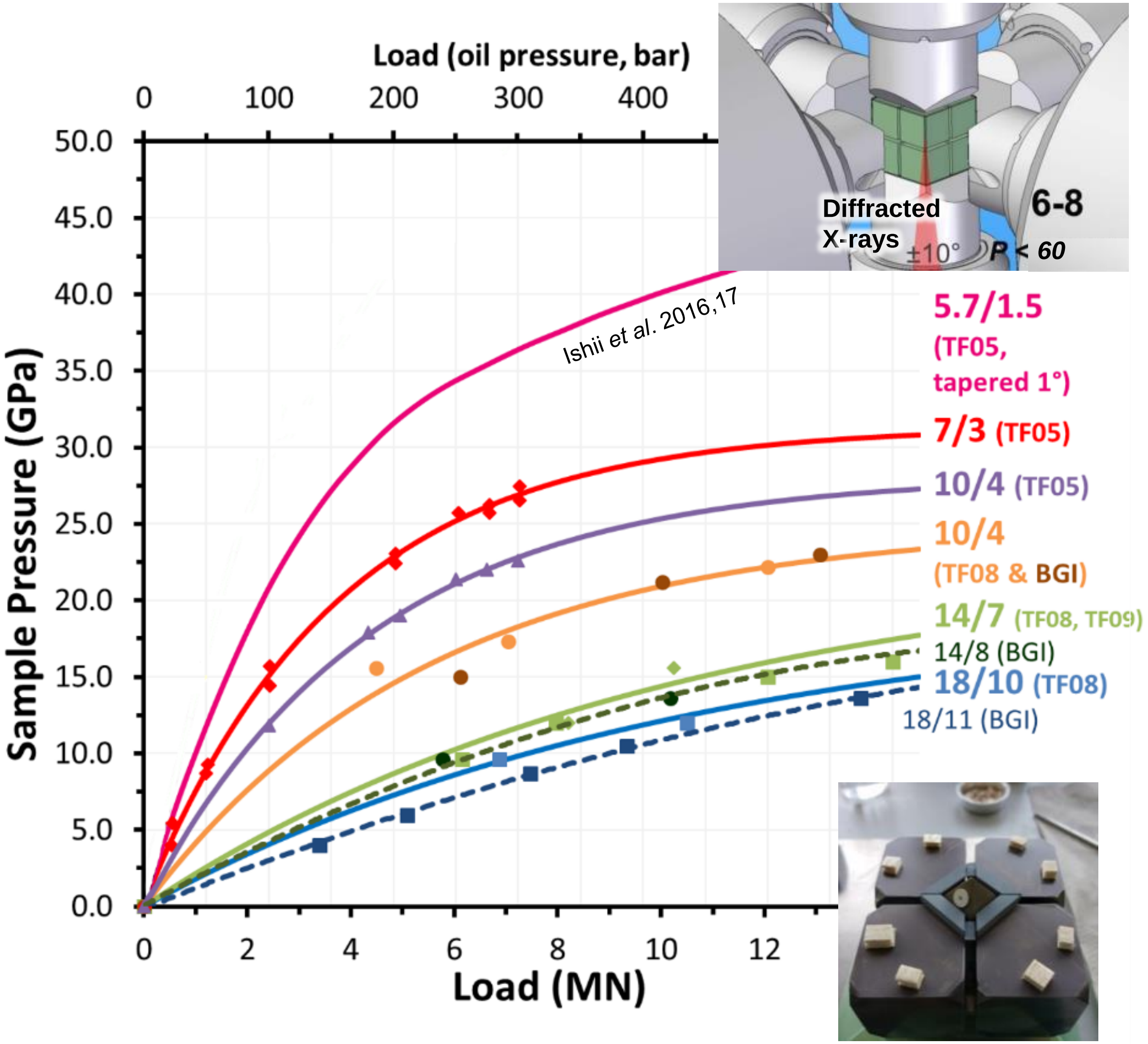
**Beamlines are complementary!**

**P61B**

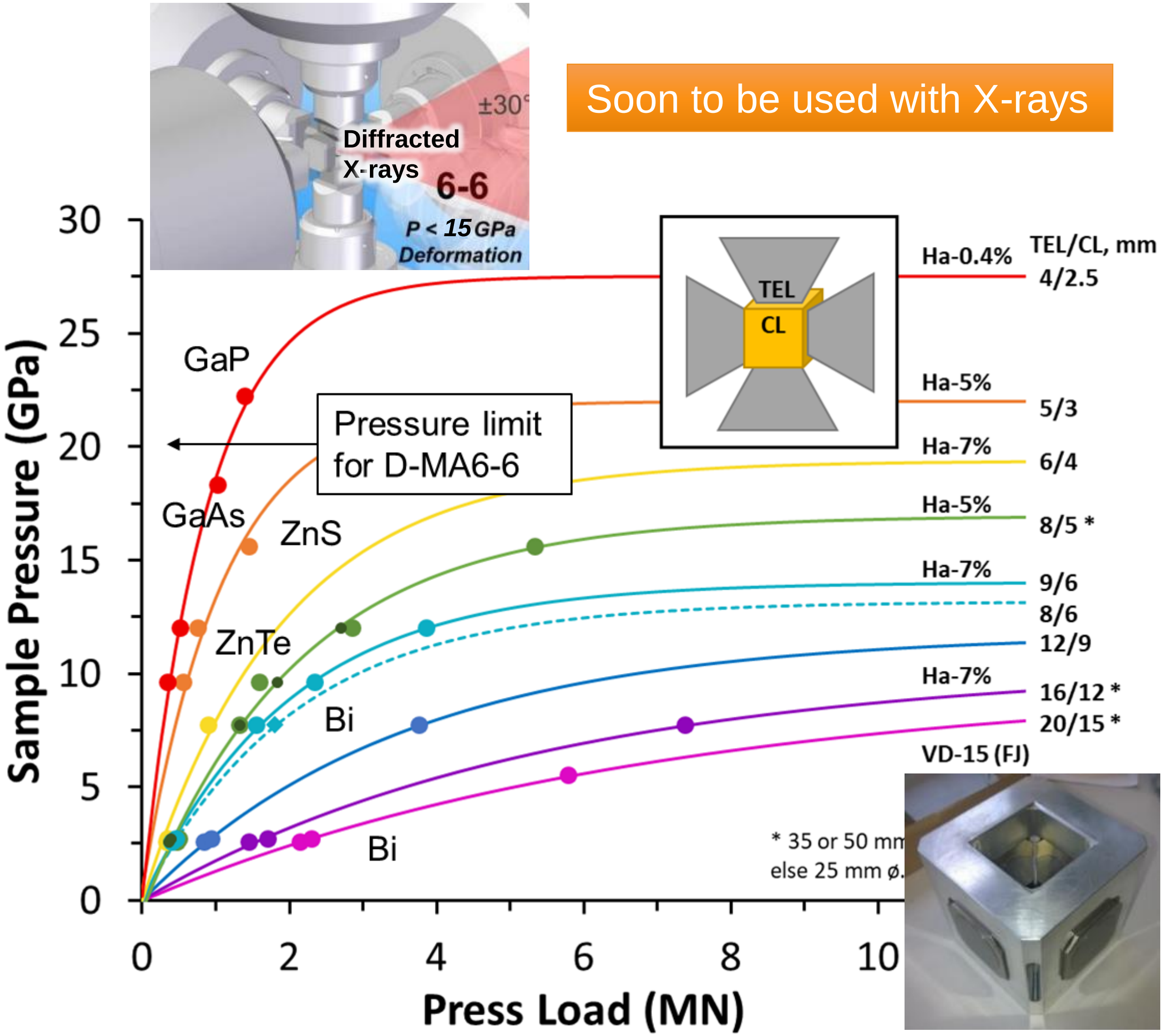
Ultra-high pressures (60±0.1 GPa)
Large (100 mm$^3$) samples
Polymineralic rock
→ Study of grain boundary transport properties (conduction, diffusion, rheology) !

# Pressure generation in the DESY ‘Hall-type’ 6-ram LVP

**Isotropic compression:** for *in situ* phase relations studies to ultra-high pressures (60+ GPa) and temperatures (3500 K)

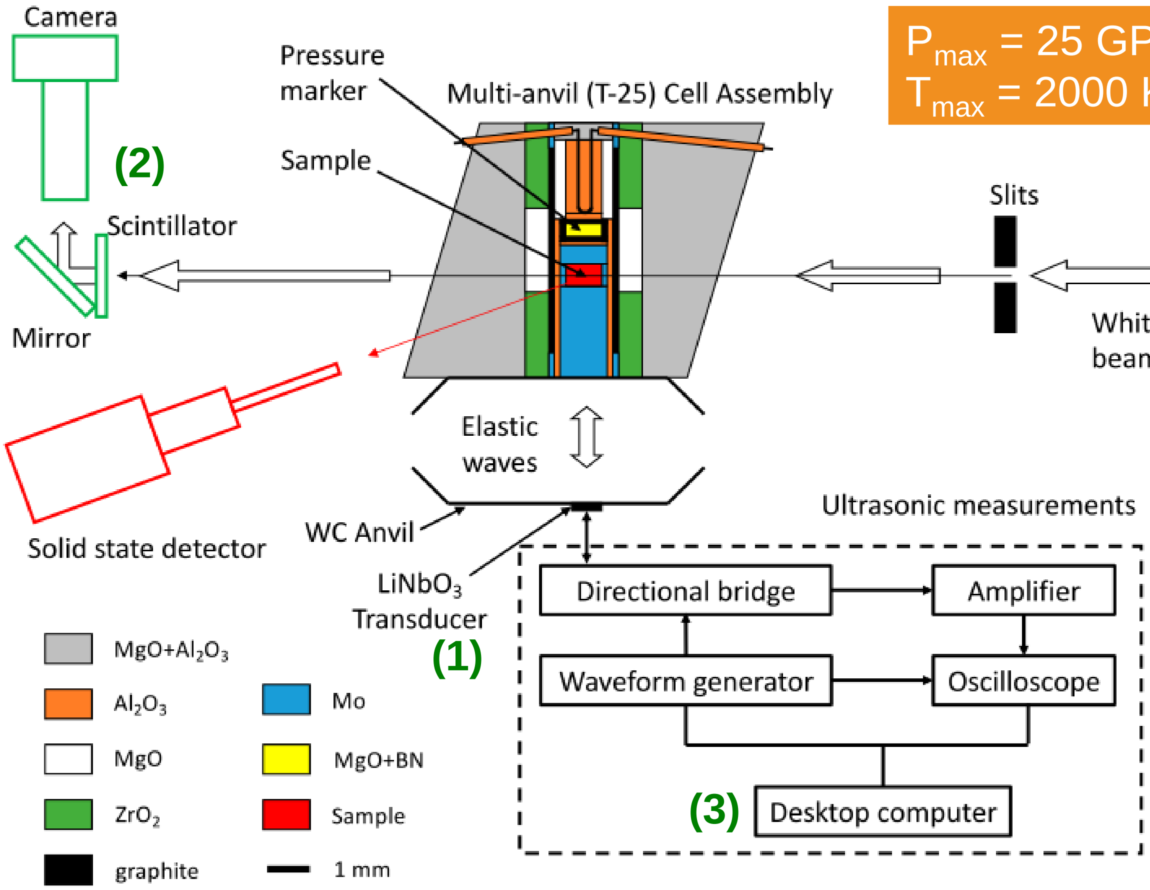



**Anisotropic compression:** for *in situ* rock deformation studies using MA6-6 set ups (max. 15 GPa / 2000 K)



# *In situ* wave speed measurements

## Ultrasonic interferometry technique combined with *in situ* X-ray diffraction and imaging




$P_{max}$ = 25 GPa
$T_{max}$ = 2000 K

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

This standard technique will be reproduced at P61B

**Since mid-1990s**. Measurement of two-way travel time of ultrasonic waves in a sample at high P and T.

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

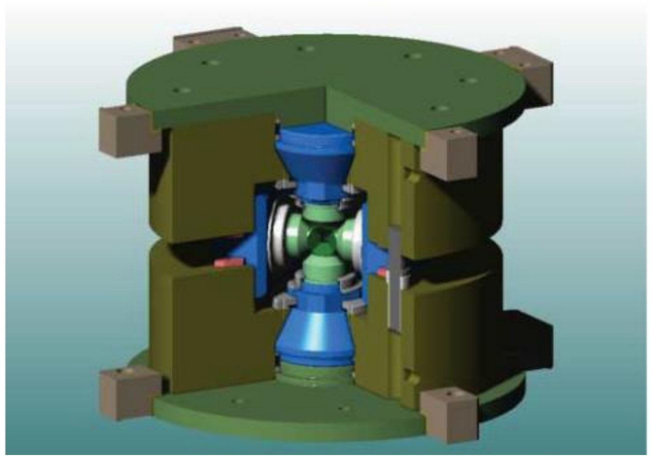
(2) Simultaneous imaging (**radiography**) provides sample length with sub-pixel resolution (< 1 µm).

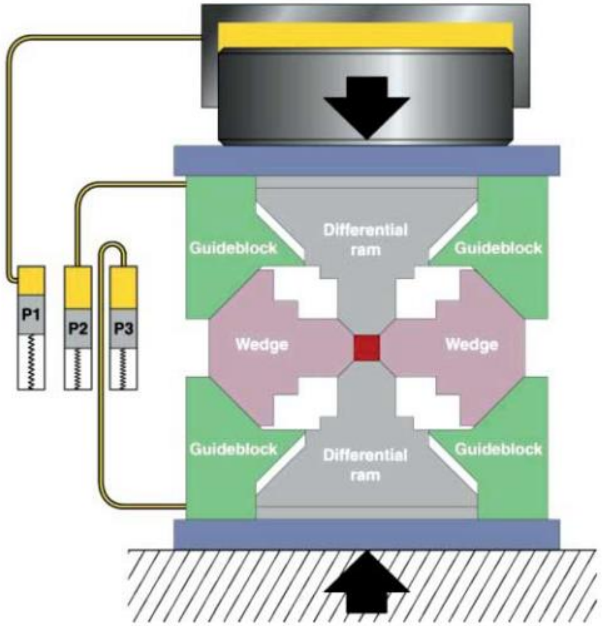
(3) Wave speed at given P,T is calculated for determination of elastic moduli (with density information).

Simultaneously measurement of elastic P and S wave travel times, density, and sample length in a LVP combined with synchrotron X-ray radiation techniques enables direct determination of the **cell pressure** and **seismic properties of materials**.

# *In situ* rock deformation studies

## Understanding the mechanical properties of materials at high P-T





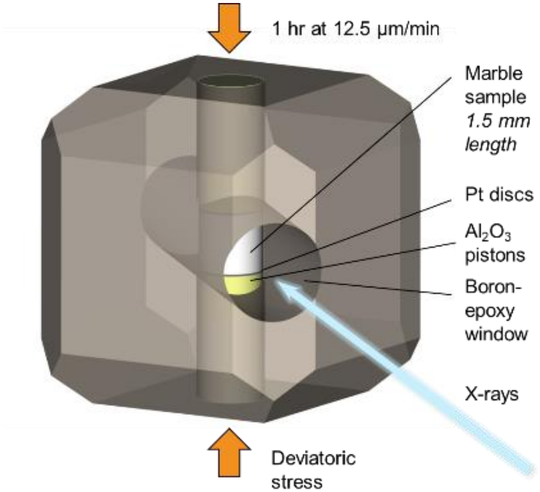

**Since 2002**. The first D-DIA type module in a LVP installed at a synchrotron source (GSECARS, APS, USA).

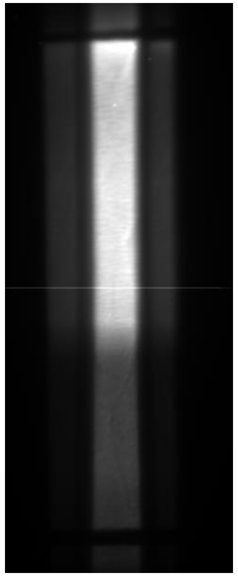
**Since 2019**, now also at P61B, PETRA III, DESY.

### Experiment limitations:

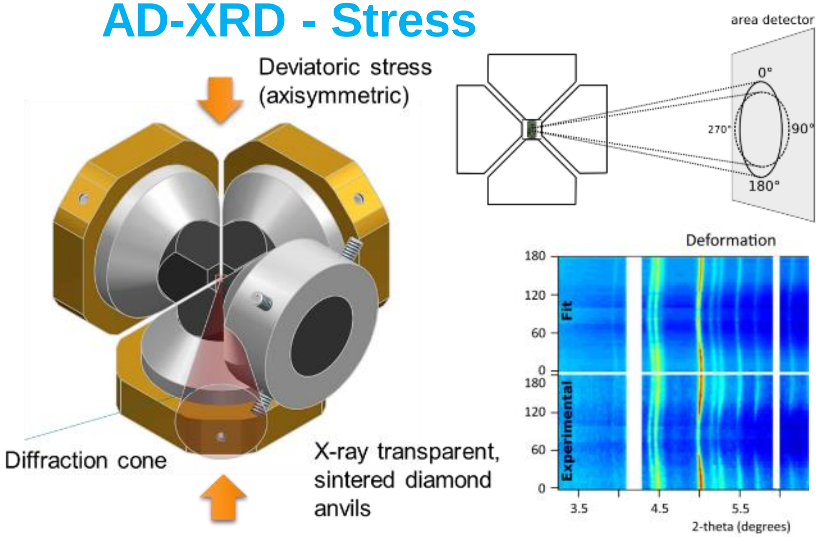
- $P_{max}$ ≈ 18 GPa (with additional 1-2 GPa differential stress)
- $T_{max}$ = e.g. 2000 K
- $Strain_{max}$ ≈ 30% (in compression, more in simple shear geometry)
- Requires min. 2 Ge-detectors (for ED-XRD with white X-rays) or… a large-radius area detector for AD-XRD (better)

### Radiography - Strain



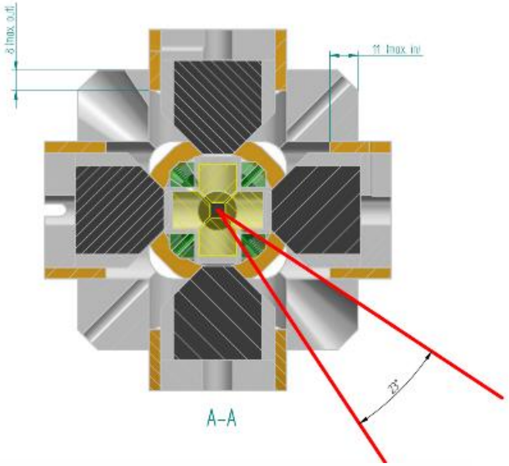
### AD-XRD - Stress




Wang *et al. Rev. Sci. Instrum.* 2003

# *In situ* Acoustic Emissions studies

## Assembly design and development (since 2010s)




Simultaneous acoustic emissions monitoring and synchrotron X-ray diffraction at high pressure and temperature: Calibration and application to serpentinite dehydration

Julien Gasc[a,*], Alexandre Schubnel[a], Fabrice Brunet[a], Sophie Guillon[a], Hans-J. Mueller[b], Christian Lathe[b]

[a] Laboratoire de Géologie, CNRS – École Normale Supérieure, 24 rue Lhomond, 75005 Paris, France
[b] Helmholtz Centre Potsdam, GFZ German Research Centre for Geosciences, Telegraphenberg, D-14473 Potsdam, Germany

**2011 DORIS III @ DESY**

REPORT

Deep-Focus Earthquake Analogs Recorded at High Pressure and Temperature in the Laboratory

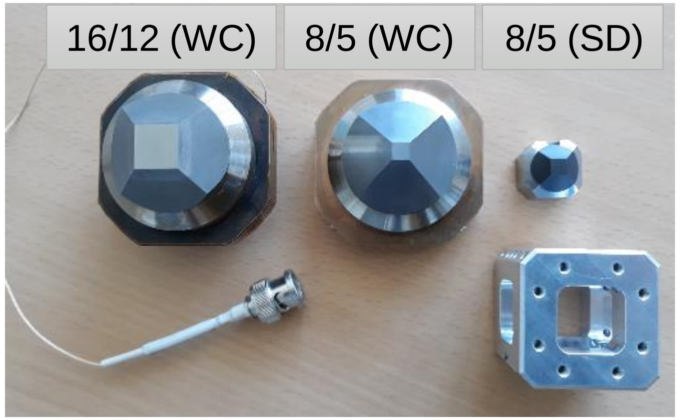
Alexandre Schubnel[1,*], Fabrice Brunet[2], Nadège Hilairet[3,†], Julien Gasc[3], Yanbin Wang[3], Harry W. Green II[4]

+ See all authors and affiliations

*Science* 20 Sep 2013:
Vol. 341, Issue 6152, pp. 1377-1380
DOI: 10.1126/science.1240206

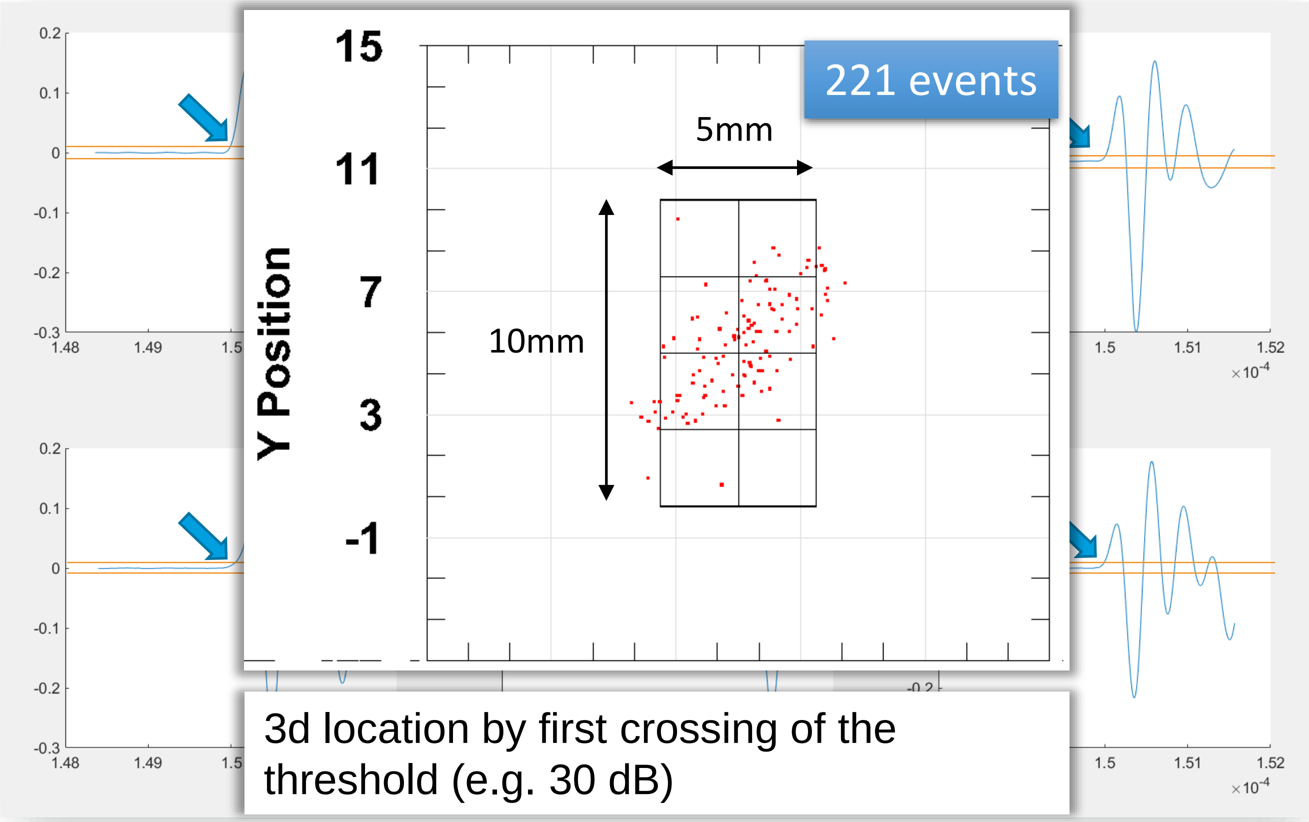
Science

**2013 @ GSECARS, APS**




**2020 PETRA III @ DESY**

Anvil configurations for MA6-6




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

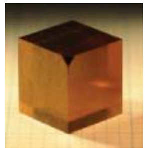
# Ultra-high pressures using sintered diamond anvils

## Recent developments

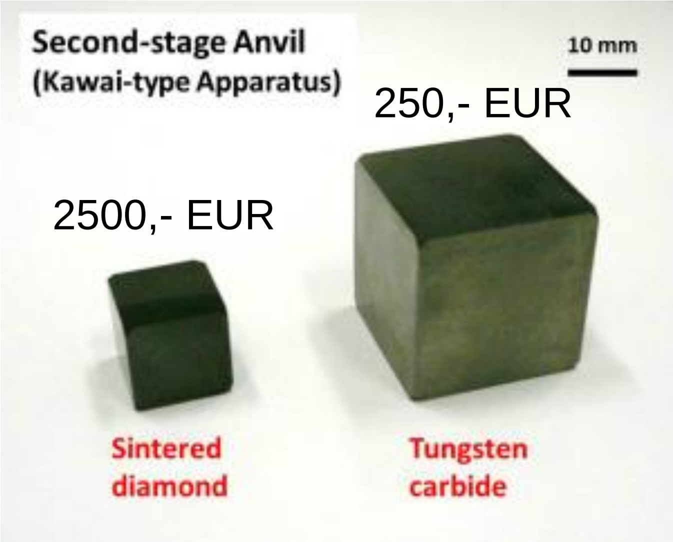
**Pressure in LVP is limited by the hardness of anvil materials**

- 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!

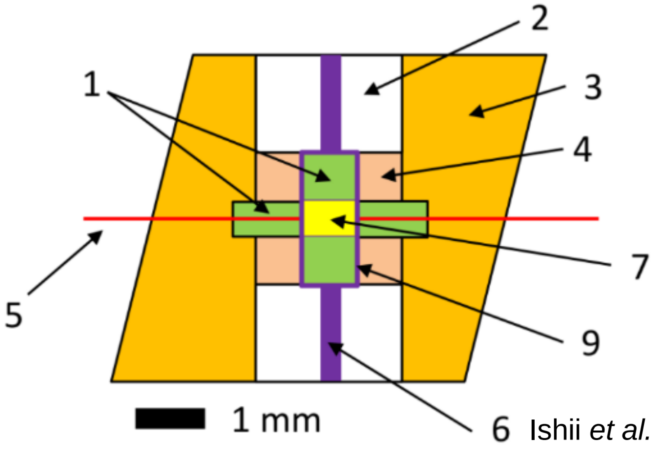
**1. Most reliable, generate highest pressures:**
14 mm SD anvils with **cobalt** binder
**2. Most transparent to X-rays, lower pressures:**
14 mm SD anvils with **SiC** binder
**3. Hardest/Extreme HP/most expensive:**
Binderless **nano-polycrystalline diamond**



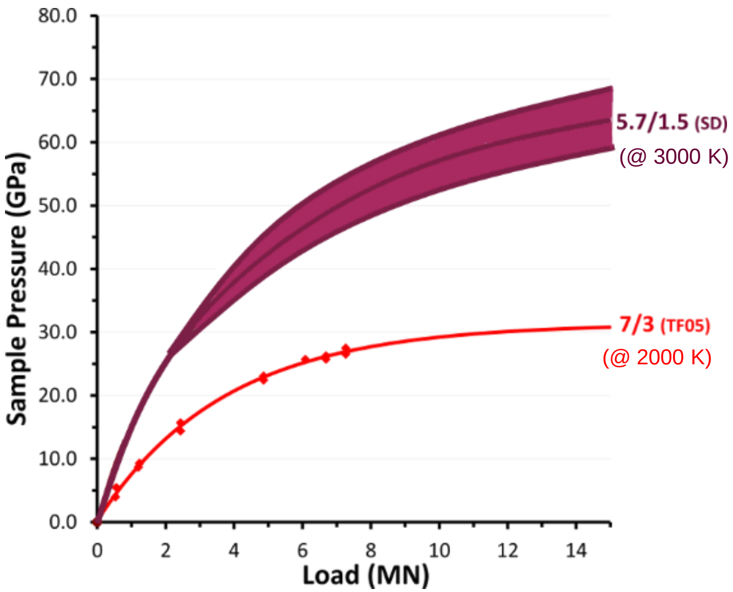
Anvil truncation is typically 1.5 mm for UHP experiments in the LVP using Kawai-8 cubic anvils set ups. Sample size is up to 0.5 mm.




5.7/1.5 cell assembly




Ishii *et al.* 2016




Up to 100 GPa, possible!

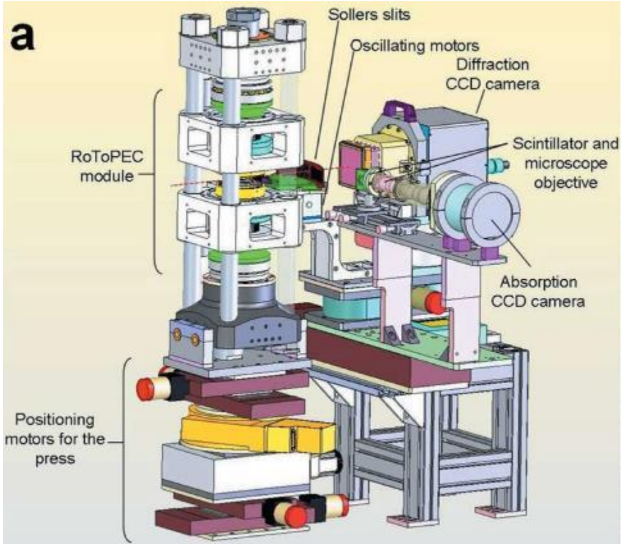
Routine experiments with Option 1 allow for recovery of SD anvils without breakage after 1$^{st}$ run (at least up to 60 GPa)

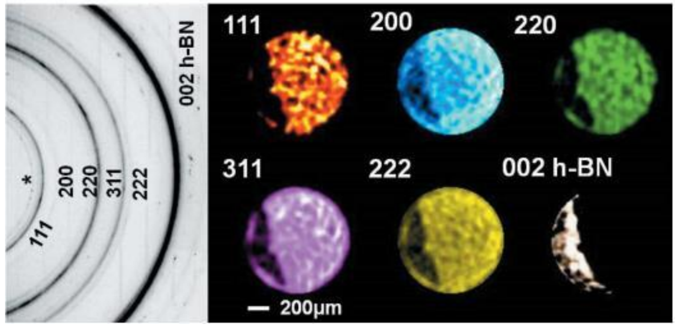
# Synchrotron radiation for studies in a Paris-Edinburgh LVP

## Different type LVP for different research objectives!

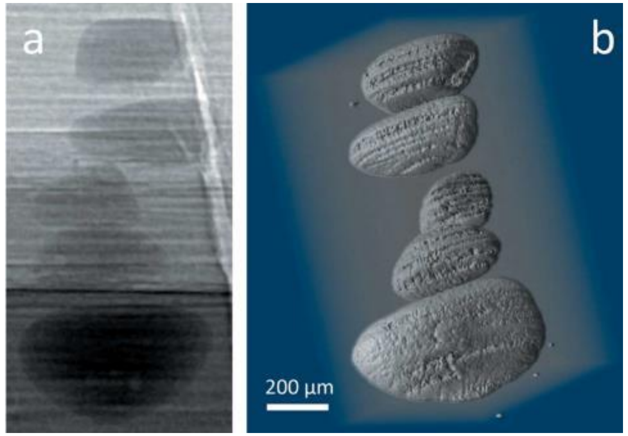
Time resolved 3d-microtomography in a 450 t torsion press, the RoToPEc

*Near-full angular access:* 360°/0.02° precision, *PT range*: 15 GPa/ 2500 K, *Max. load*: 4.5 MN, *torsional deformation by anvil rotation.*


2D XRD pattern and 2D reconstructed slices from *d*-spacing/2θ of different reflections ($C_{60}$ polymerization under pressure).




(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 PETRA IV objectives for:

- Synchrotron X-ray absorption/phase contrast micro-tomography (2x2 $mm^2$ beam) at extreme conditions.
- Diffraction/scattering computed tomography, DSCT (e.g. 3x3 $\mu m^2$ focused beam) at extreme conditions.

**More details on science cases for the 6-ram LVP and PE**
**@ talk of Dr. Sieber at 16:00 today,**

**…and at the satellite meeting 5-6$^{th}$ of November**

# Synchrotron radiation for studies in the Diamond Anvil Cell

## It is easier to say what one cannot do in a DAC

**Probed materials:**

- Single crystal
- Powder diffraction
- Amorphous state (liquid and solid)

**Probing techniques (lab):**

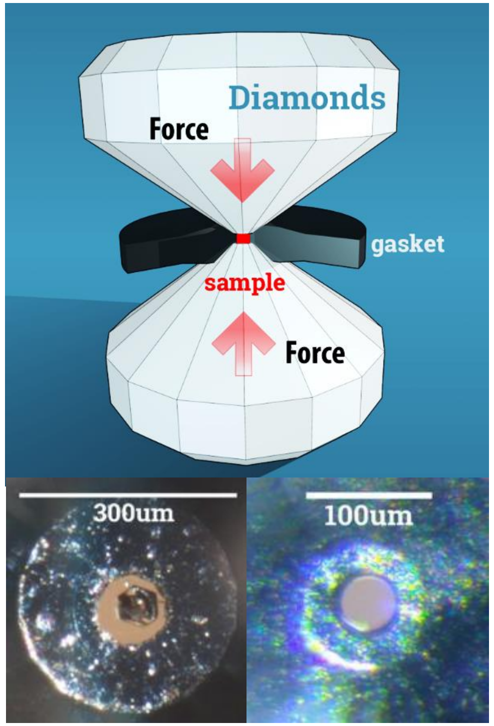
- Luminescence, Raman, UV
- Resistance measurement
- NMR/EPR and etc.

More details – talks of Dr. C. Prescher, Dr. T. Meier, Dr. C. Sternemann, Prof. Dr. I. Kupenko, and Prof. Dr. D. Kraus starting at 16:20 today

and at the satellite meeting 5-6th of November

**Probing techniques (synchrotron based):**

- X-ray diffraction (WAXS, SAXS, resonant scattering, uniaxial, radial, high resolution)
- X-ray spectroscopy
  - X-ray Absorption
  - X-ray Emission
  - X-ray Inelastic Scattering
  - X-ray Mössbauer
  - X-ray Raman
- X-ray imaging (phase contrast, CDI, scanning, full field)



# PETRAIII - LVP + DAC

## Working together for material science

**Techniques are complementary & synergetic:**

- **Synthesized in DAC – produced in LVP (larger quantities)**
- **Produced in LVP – characterized in DAC**


ChemPubSoc Europe

DOI: 10.1002/chem.201904529

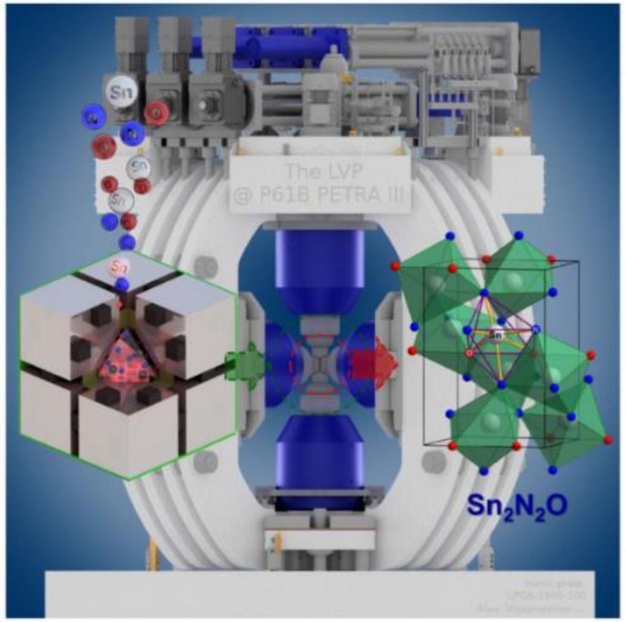
CHEMISTRY A European Journal Full Paper

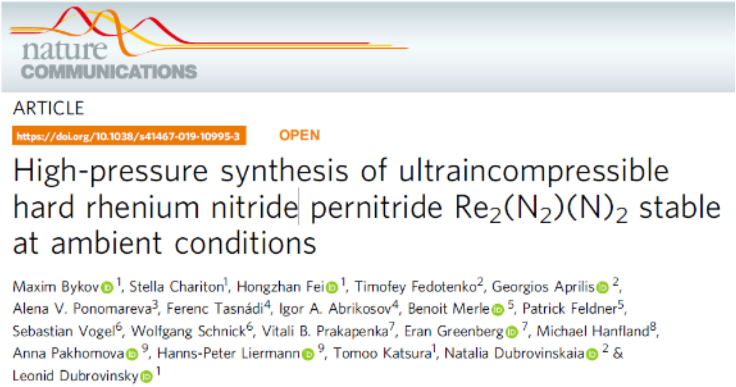
High-Pressure Synthesis | Hot Paper |

A Novel High-Pressure Tin Oxynitride $Sn_2N_2O$

Shrikant Bhat,*[a, c] 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]

**$Sn_2N_2O$ is produced in LVP**
**Elasticity is characterized in DAC**





nature COMMUNICATIONS

ARTICLE

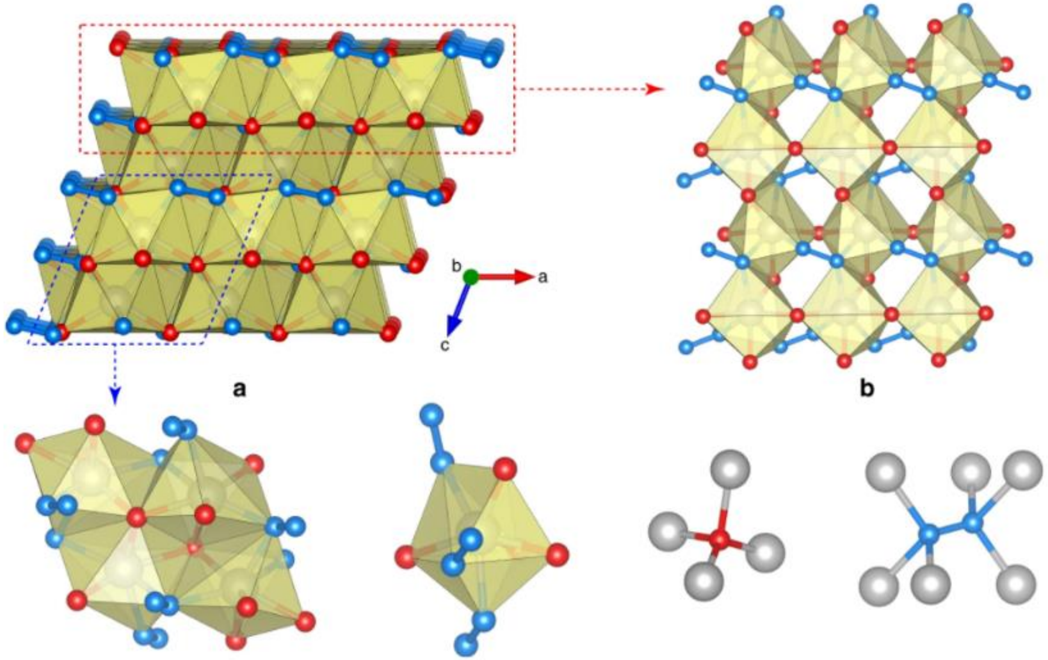
https://doi.org/10.1038/s41467-019-10995-3 OPEN

High-pressure synthesis of ultraincompressible hard rhenium nitride pernitride $Re_2(N_2)(N)_2$ stable at ambient conditions

Maxim Bykov[1], Stella Chariton[1], Hongzhan Fei[1], Timofey Fedotenko[2], Georgios Aprilis[2], Alena V. Ponomareva[3], Ferenc Tasnádi[4], Igor A. Abrikosov[4], Benoit Merle[5], Patrick Feldner[5], Sebastian Vogel[6], Wolfgang Schnick[6], Vitali B. Prakapenka[7], Eran Greenberg[7], Michael Hanfland[8], Anna Pakhomova[9], Hanns-Peter Liermann[9], Tomoo Katsura[1], Natalia Dubrovinskaia[2] & Leonid Dubrovinsky[1]

**$Re_2N_2(N)_2$ is produced and quenched in DAC**

**Produced in larger quantity in LVP**



# New, Expanded, Extreme Conditions Research at PETRA IV

## Thank you for your attention!