



In situ rock deformation and liquids under extreme conditions - the value of specialized LVPs at PETRA IV



M. J. Sieber

T. Katsura
Th 9:20 am

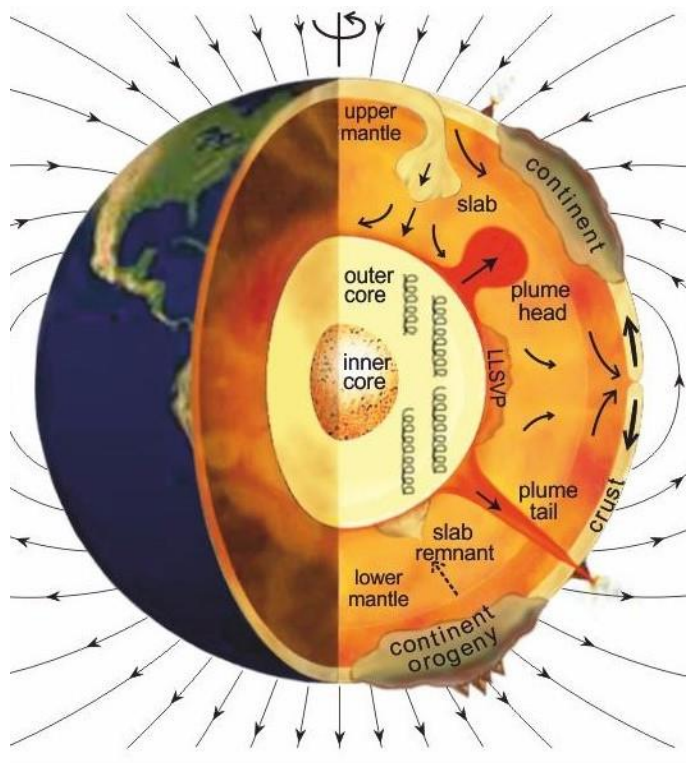
N. Hilaret
Th 9:40 am

R. Farla
Th 10:45 am

Rheology studies in situ rock deformation

Why interesting?

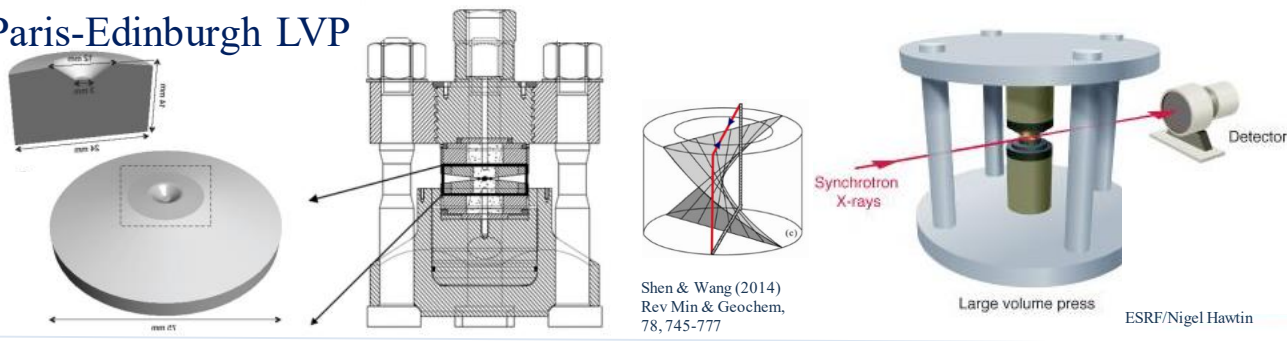
- mantle convections
 - driving tectonics, causing volcanic and seismic activities



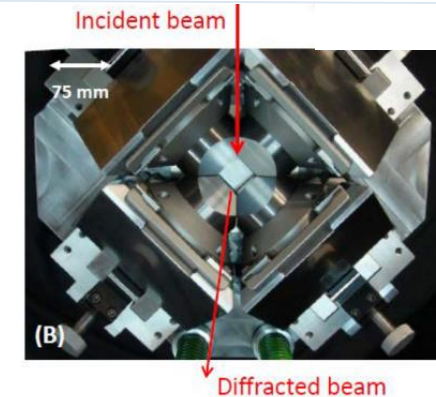
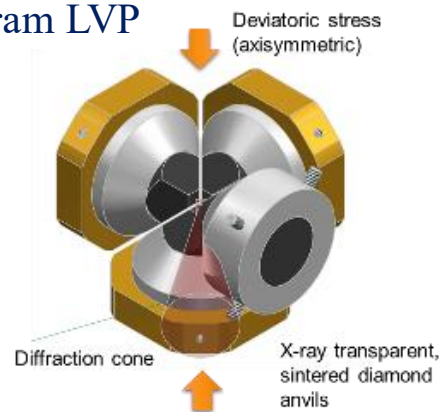
Why are rheology studies under extreme conditions in a LVP needed?

- stress & strain can be monitored & measured
- sufficient number of grains
- uniform & stable PT-conditions over wide strain rates

Paris-Edinburgh LVP



6-ram LVP



in situ rock deformation (2D) AD-XRD & radiographic imaging

Talk N. Hilairet
Th 9:40 am



Angle dispersive X-ray diffraction: stress
X-ray radiographic imaging: axial strain



Deformation of mantle olivine

Hilairet et al. 2012

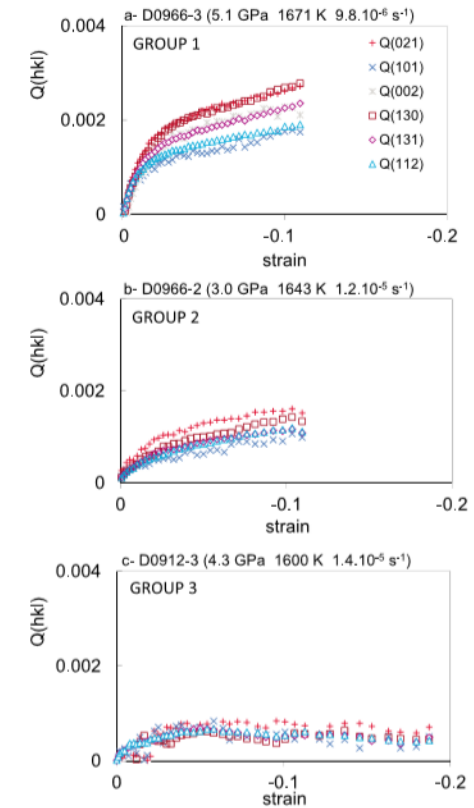
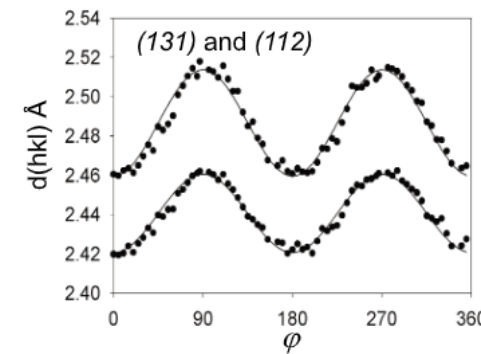
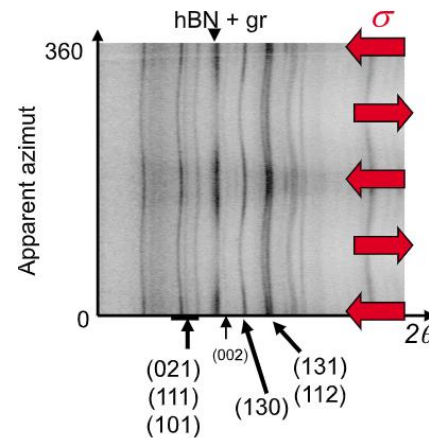
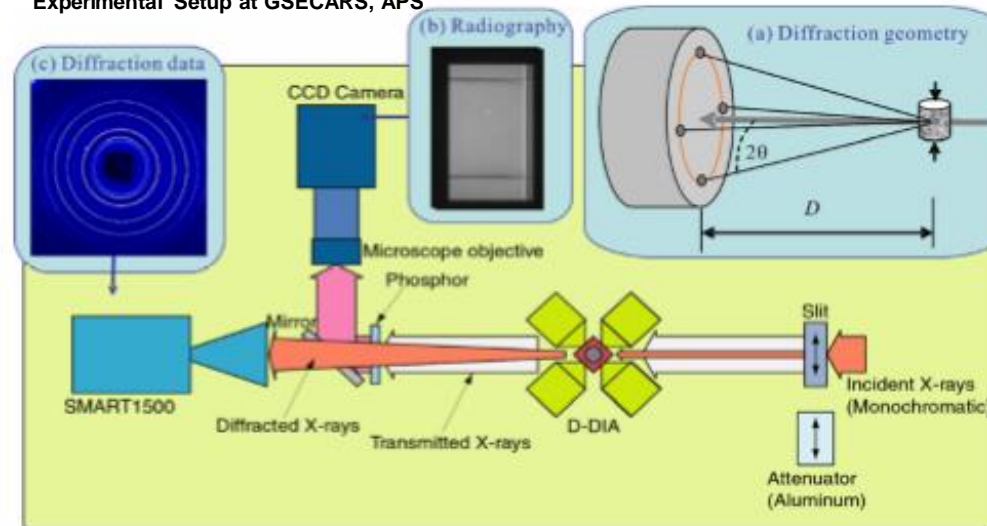
- polycrystalline olivine
- $P=2.8-7.8$ GPa; $T\sim 900-1400$ °C
- stress rates: 7.10^{-6} to 3.10^{-5} s⁻¹



D-DIA

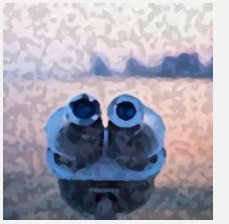
installed @ GSECARS, APS





Experimental Setup at GSECARS, APS





Large Volume Press @ PETRA IV

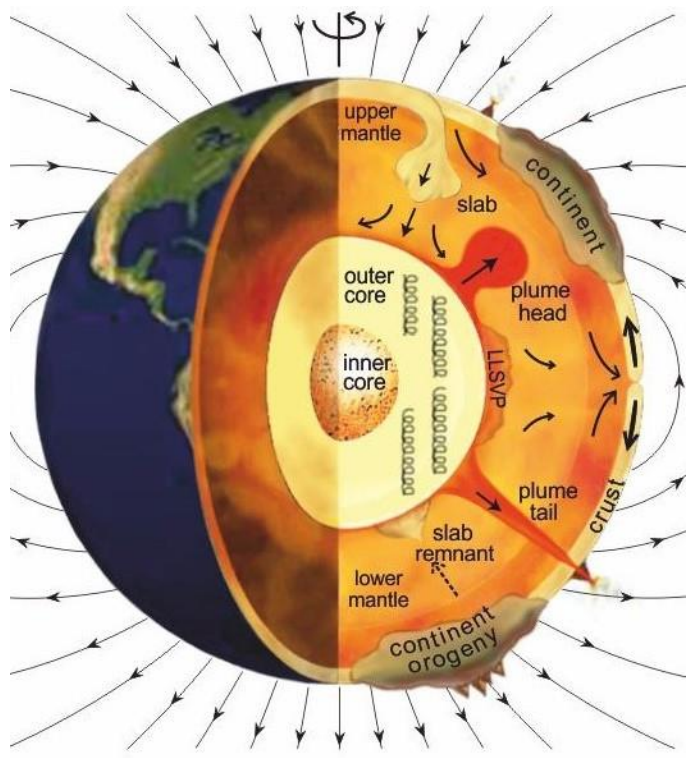


	6-ram MA (P61.B) 20 GPa, 2000 K (40 GPa; 2000 K)
 Research Project	➤ axial deformation (rheology)
	➤ (2D) AD-XRD ➤ imaging (radiography)
 MEET MY NEEDS	➤ improved resolution in stress and strain measurements (also for rocks) ➤ directional, monochromatic X-ray beam with high coherence and low-emittance <ul style="list-style-type: none">➤ $\Delta E/E \sim 10^{-3}$ to 10^{-4}➤ 40-100 keV➤ large-radius area detector➤ highly focused beam

Liquids under extreme conditions

Why interesting?

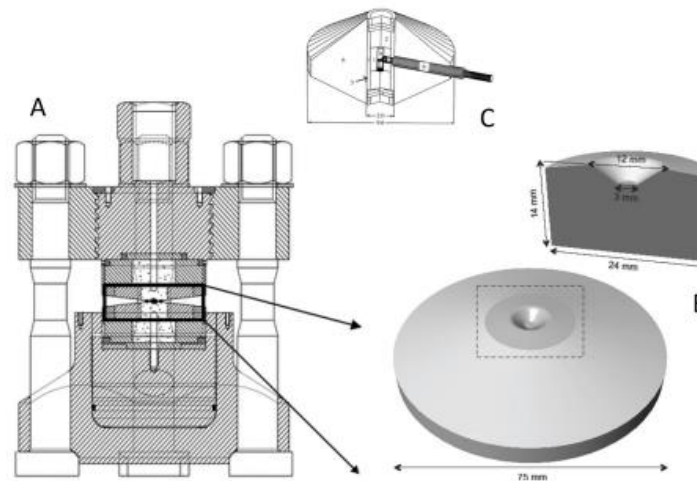
- relevant to the origin and evolution of planets
- heat and mass transfer influence the chemical and thermal history of Earth's interior



Why are studies of liquids under extreme conditions in a (PE) LVP needed?

- some liquids are non-quenchable
- e.g. viscosity, density, structure of a melt but also melting point and melt propagation can best be studied in situ
- precise PT control (over time)
- post mortem characterization possible
- composite materials (multi-grain rocks)

Paris-Edinburgh LVP advantage: wide opening angle



Shen & Wang (2014)
Rev Min & Geochem,
78, 745-777

Liquids under extreme conditions X-ray absorption imaging / tomography



X-ray absorption imaging / tomography



Research Project

Rhyolite melt percolation through the mantle

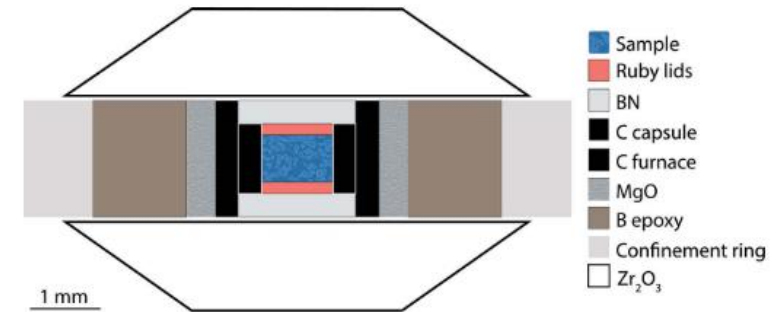
Boulard E., *et al.* 2018

- finely ground rhyolite & olivine crystals
- $P=3$ GPa; ≤ 1600 K
- **rotation 135°** , 10 msec exposure time
→ **10 sec for a tomogram**

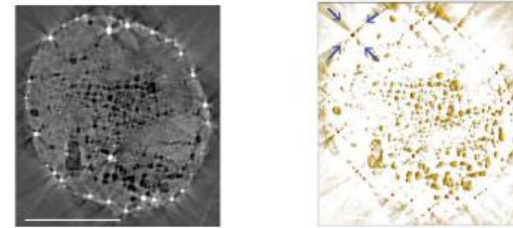


PE

installed @ PSICHE, SOLEIL

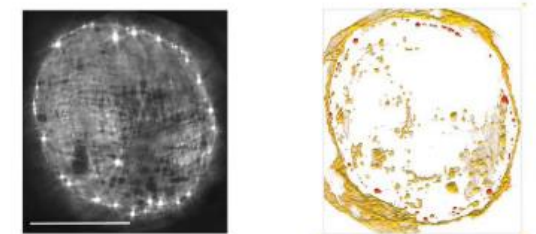


in situ measurement - FBP reconstruction



Number of iron beads: 964
Mean size of iron beads: $812 \mu\text{m}^3$
Total volume of iron: 0.0007mm^3

in situ measurement - ART reconstruction



Number of iron beads: 609
Mean size of iron beads: $1948 \mu\text{m}^3$
Total volume of iron: 0.0012mm^3

Liquids under extreme conditions X-ray absorption imaging / tomography



X-ray absorption imaging / tomography

Compressibility of basaltic glass

Álvarez-Murga M., *et al.* 2017

- 5 basaltic glass beads in h-BN
- P=0-4.4 GPa; RT
- rotation 180°, 0.15° s⁻¹
- ➔ 20 min for a tomogram

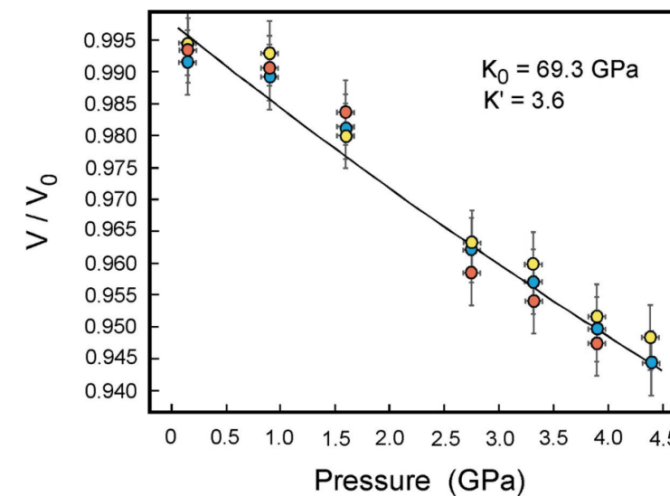
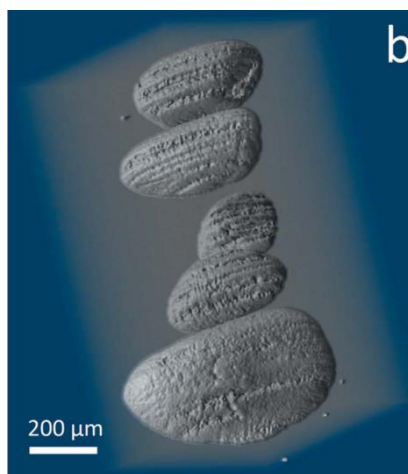
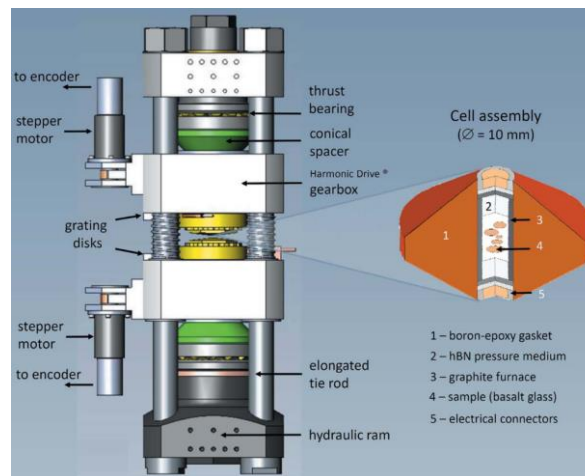


Research Project



RoToPEc

installed @ PSICHE, SOLEIL



Liquids under extreme conditions

X-ray absorption imaging / tomography



X-ray absorption imaging / tomography

Compressibility of basaltic glass

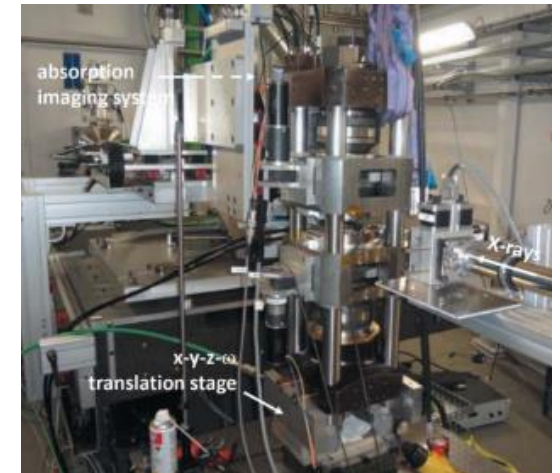
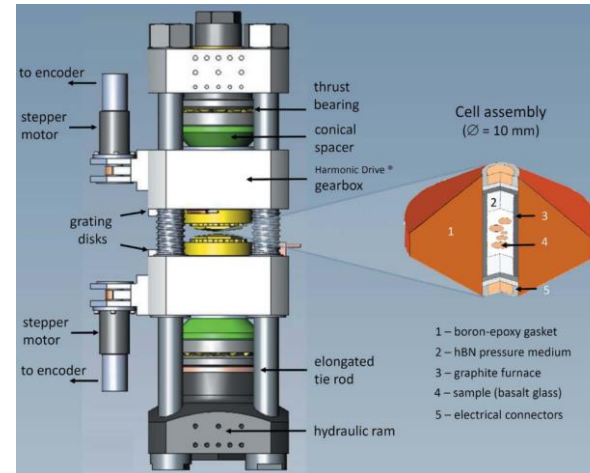
Álvarez-Murga M., *et al.* 2017

- 5 basaltic glass beads in h-BN
- P=0-4.4 GPa; RT
- **rotation 180°, 0.15° s⁻¹**
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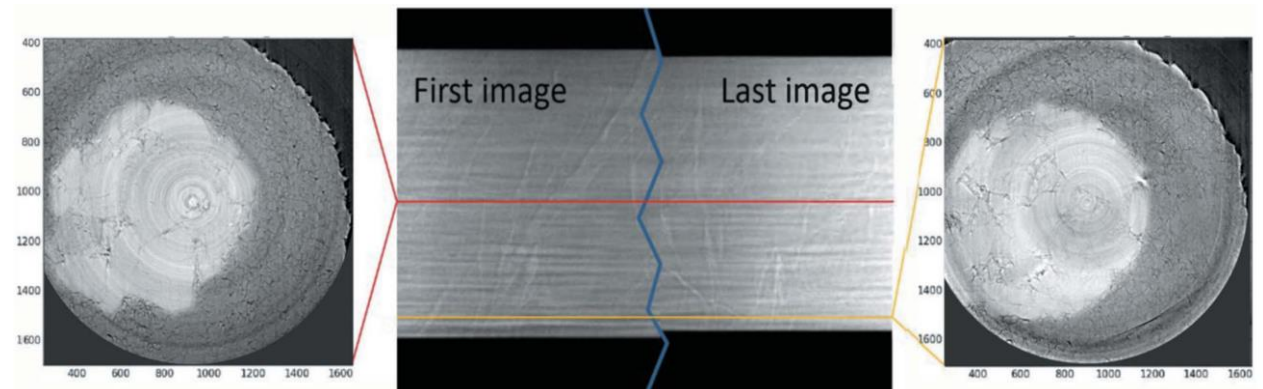


RoToPEc

installed @ PSICHE, SOLEIL



3 GPa 1800 K



Liquids under extreme conditions X-ray phase contrast imaging



X-ray phase contrast imaging



Liquid phase separation and liquid-liquid immiscibility

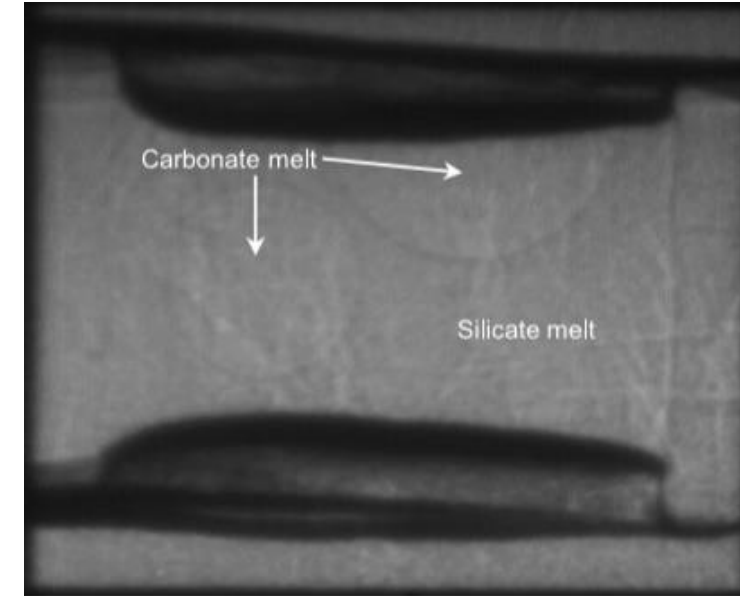
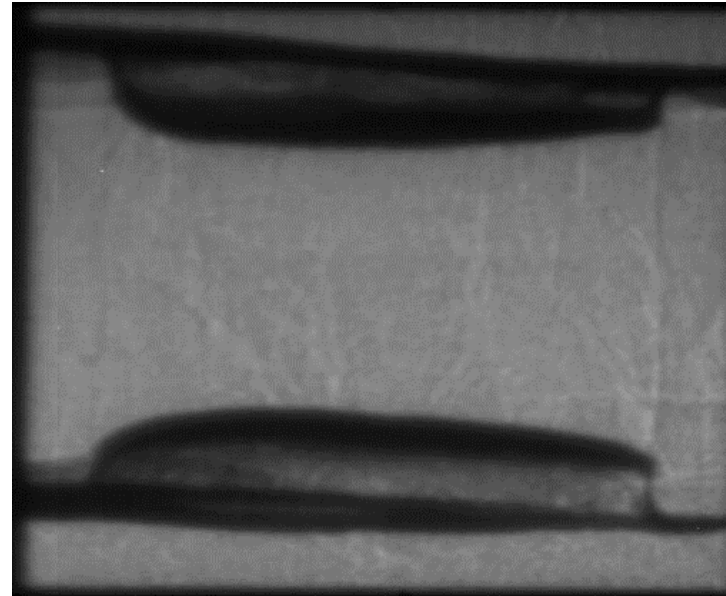
Kono Y., *et al.* 2015

- 40 wt% albite & 60 wt% calcite
- $P=2.5$ GPa; 1350-1500 °C
- movie: 60 frames/sec



PE

installed @ 16-BM-B, APS



200 μm

X-ray diffraction / scattering μ -tomography



X-ray diffraction / scattering μ -tomography

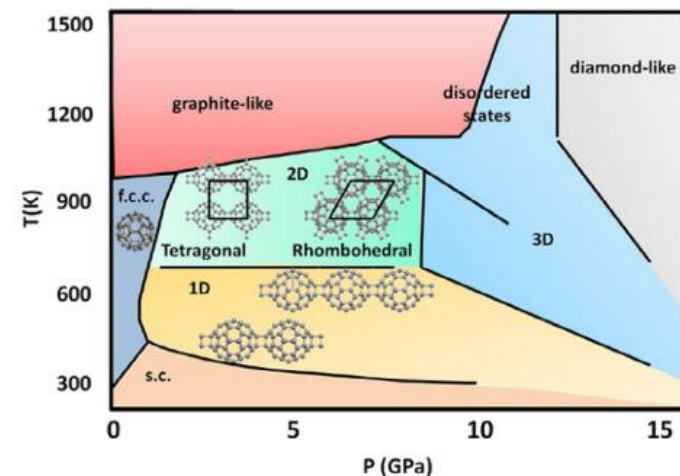
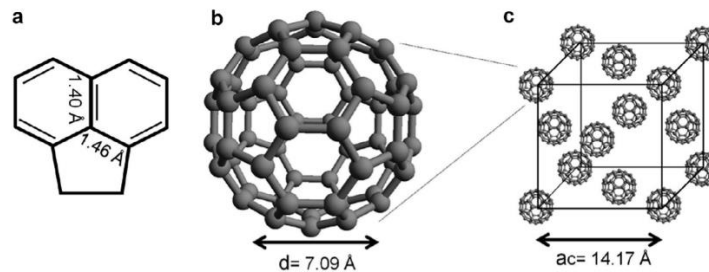


Research Project

C₆₀ polymerization & phase transitions

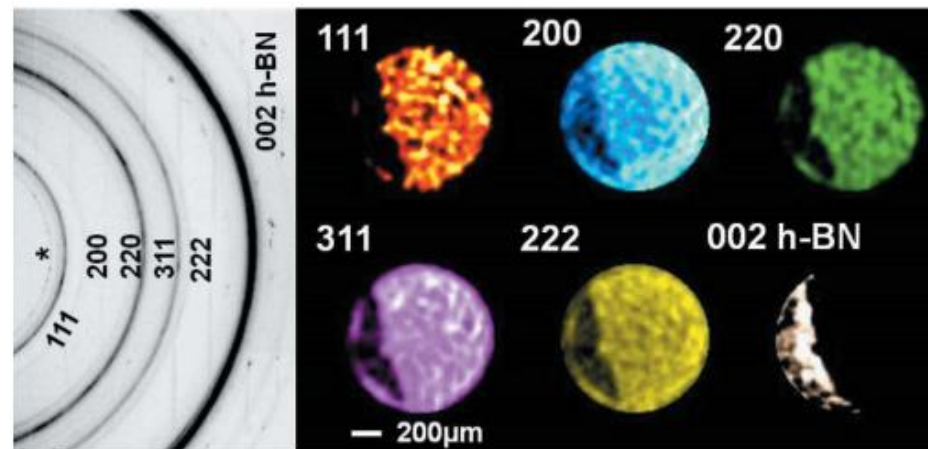
Álvarez-Murga M., et al. 2017

- fullerene
- monochromatic beam
 - 2.4 μm x 2.0 μm ; 4.0 mm x 2.0 mm
- for 1.35 mm profile: 2350 2D-XRD patterns \rightarrow 8h



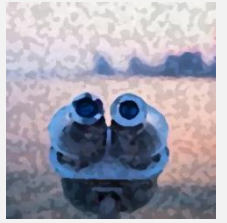
RoToPEc





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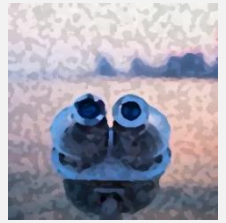
Large Volume Press @ PETRA IV







		<p>PE-LVP (former P02.2); RoToPEc (new) 10 GPa, 1700 K (20 GPa, 1700 K)</p>
 <p>Research Project</p>		<ul style="list-style-type: none"> ➤ structure, density and viscosity of liquids
		<ul style="list-style-type: none"> ➤ (4D) AD-XRD ➤ DSCT (Diffraction Scattering X-ray Tomography) ➤ imaging (μ-tomography & radiography & phase contrast)
 <p>MEET MY NEEDS</p>		<ul style="list-style-type: none"> ➤ improved energy resolution (10^{-4} for $E > 40$ keV) ➤ fast acquisition and high resolution (for 4D imaging) <ul style="list-style-type: none"> ➤ tunable spot size ($\sim 1 \times 200 \mu\text{m}$ to $\sim 2 \times 2 \text{ mm}$) <ul style="list-style-type: none"> ➤ large radius area detector ➤ X-ray microscope (for radiography/tomography)
		<ul style="list-style-type: none"> ➤ big data-storage: ~ 1 TB/day (more?)



Large Volume Press @ PETRA IV



	<p>6-ram MA (P61.B) 20 GPa, 2000 K (40 GPa; 2000 K)</p>	<p>PE-LVP (former P02.2); RoToPEc (new) 10 GPa, 1700 K (20 GPa, 1700 K)</p>	<p>Uniaxial LVP UHP</p>
 Research Project	<ul style="list-style-type: none"> ➤ structural characterization & phase transitions of e.g. hydrous minerals and carbonates ➤ axial deformation (rheology) 	<ul style="list-style-type: none"> ➤ structure, density and viscosity of liquids ➤ shear deformation (rheology) ➤ evolution of microstructures & phase distribution 	<ul style="list-style-type: none"> ➤ pushing the pressure limit
	<ul style="list-style-type: none"> ➤ (1D/2D) AD-XRD ➤ ED-XRD ➤ imaging (radiography & phase contrast) 	<ul style="list-style-type: none"> ➤ (4D) AD-XRD ➤ DSCT (Diffraction Scattering X-ray Tomography) ➤ imaging (μ-tomography & radiography & phase contrast) 	<ul style="list-style-type: none"> ➤ ED-XRD
 MEET MY NEEDS	<ul style="list-style-type: none"> ➤ improved energy resolution (10^{-4} for $E > 40$ keV) ➤ fast acquisition and high resolution (for kinetics / 4D imaging) <ul style="list-style-type: none"> ➤ tunable spot size ($\sim 1 \times 200 \mu\text{m}$ to $\sim 2 \times 2 \text{ mm}$) ➤ large radius area detector ➤ X-ray microscope (for radiography/tomography) 		<div style="background-color: #003366; color: white; padding: 10px; border-radius: 15px; text-align: center;"> <p>T. Katsura Th 9:20 am</p> </div>
<ul style="list-style-type: none"> ➤ additive in situ characterization (e.g. ultrasound-velocity, electrical-conductivity measurements) 	<ul style="list-style-type: none"> ➤ big data-storage: ~ 1 TB/day (more?) 		

Thanks

Denis Andrault

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Kai Bagschik

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Marcus Schwarz

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Hans-Peter Liermann

Kristina Spektor

Shrikant Bhat

Nadege Hilairet

Geeth Manthilake

Sergio Speziale

Artem Chanyshv

Astrid Holzheid

Jean-Philippe Perrillat

Leonore Wiehl

Dominique de Ligny

Tomo Katsura

David Rafaja

Max Wilke

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Sindy Fuhrmann

Eleonora Kulik

Franziska Scheffler