



Synchrotron X-ray experiments for studying structure and properties of liquids and glasses at high-pressure and high-temperature conditions in large volume press

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Collaborators

Curtis Kenney-Benson, Changyong Park, Yanbin Wang, Guoyin Shen

-High-pressure experiments using large volume press.

-High-pressure synchrotron X-ray experiments combined with large volume press.

Large volume press

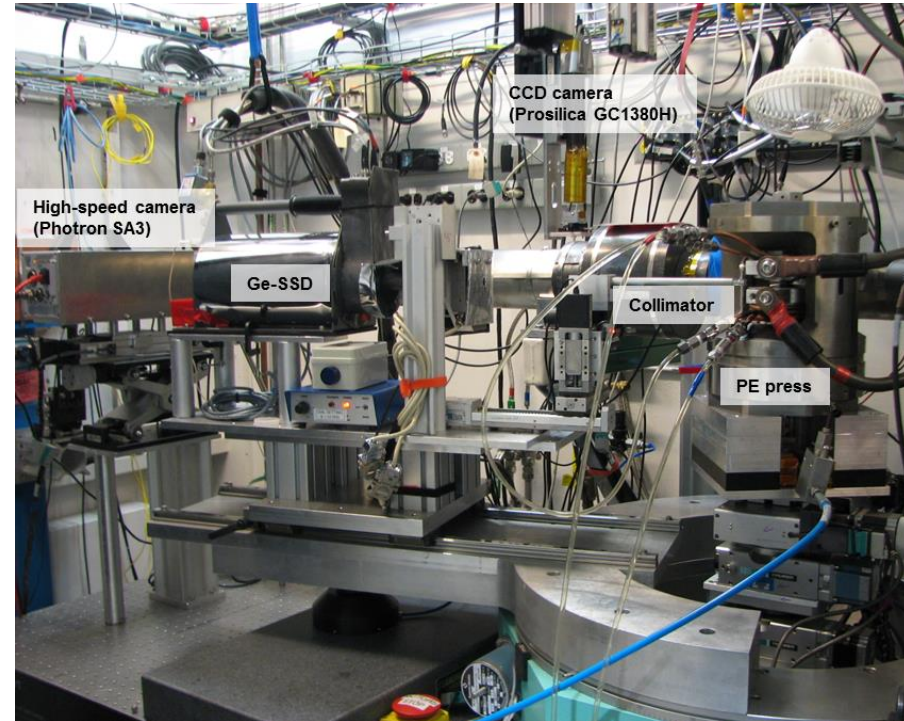
Multi-anvil press

at GRC, Ehime University, Japan

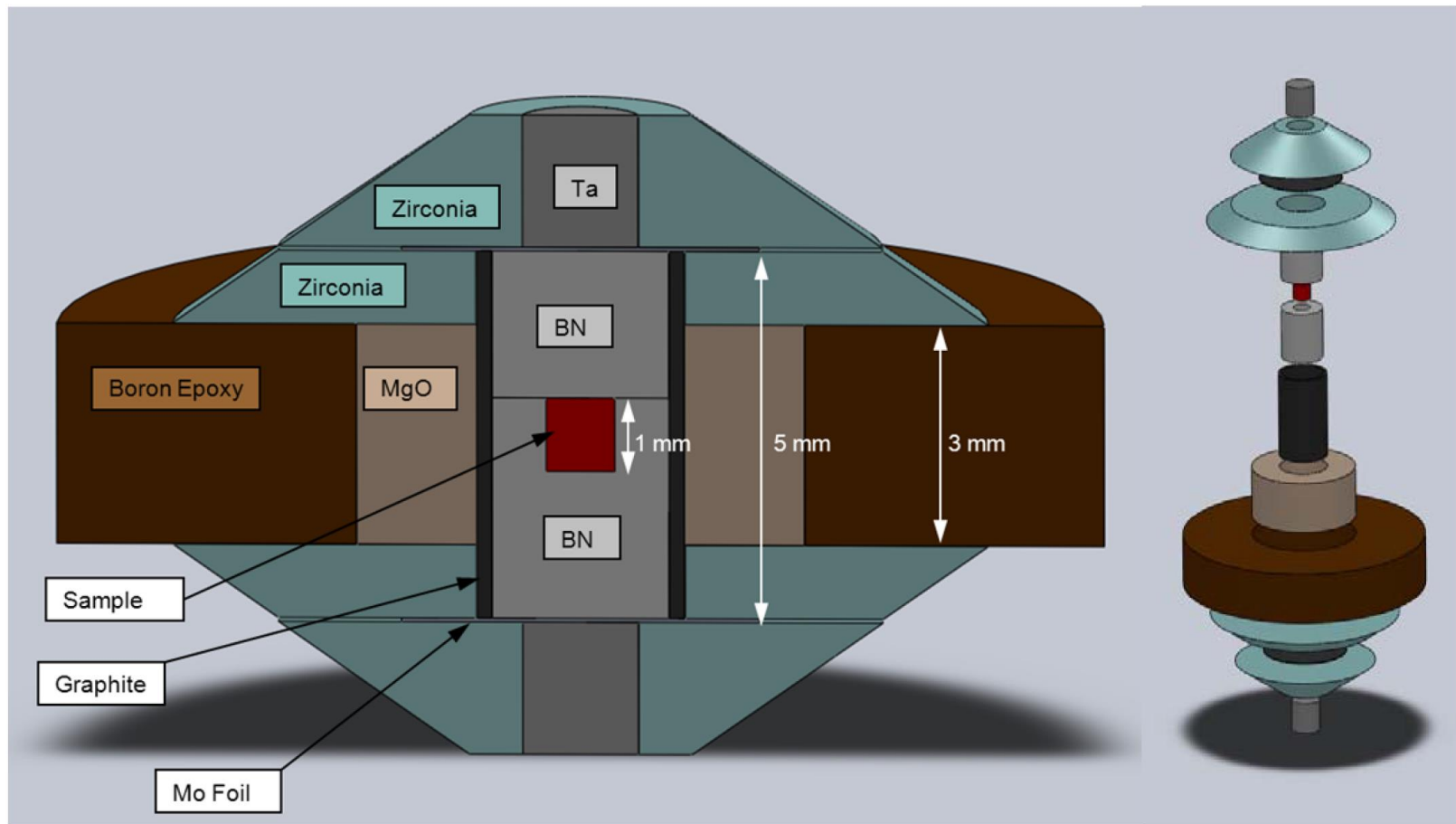


Paris-Edinburgh (PE) press

at HPCAT, Advanced Photon Source, USA



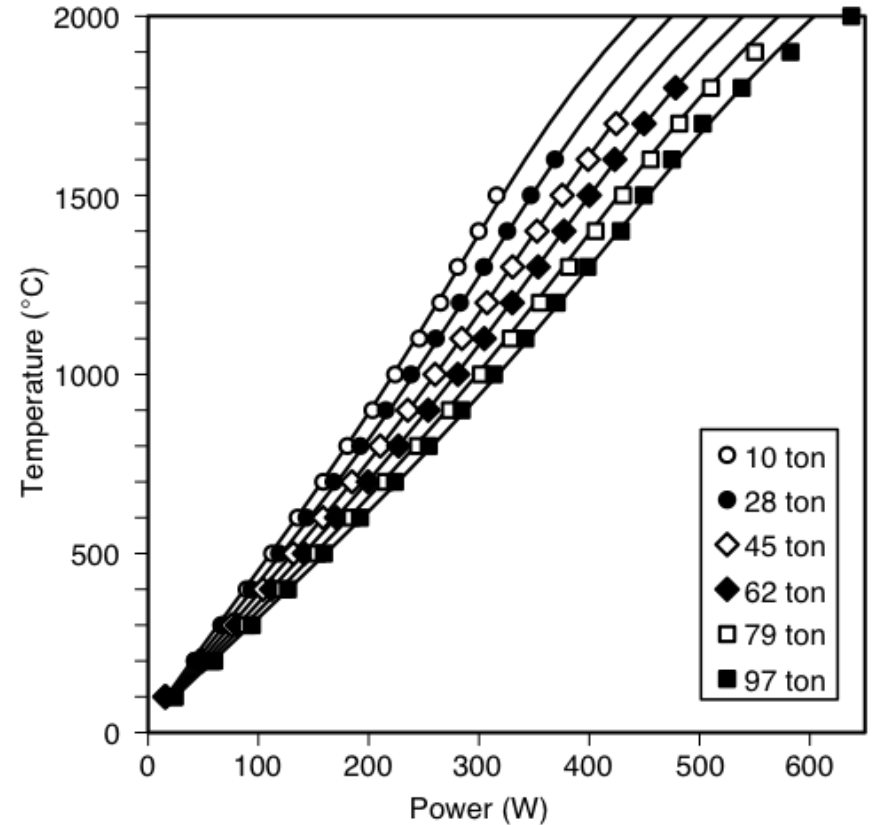
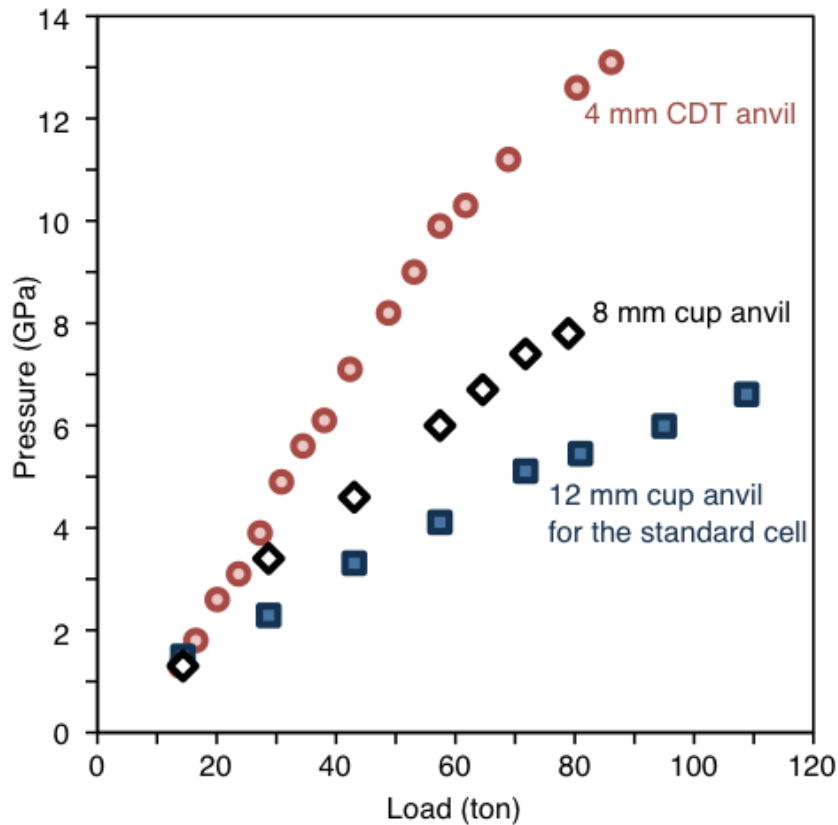
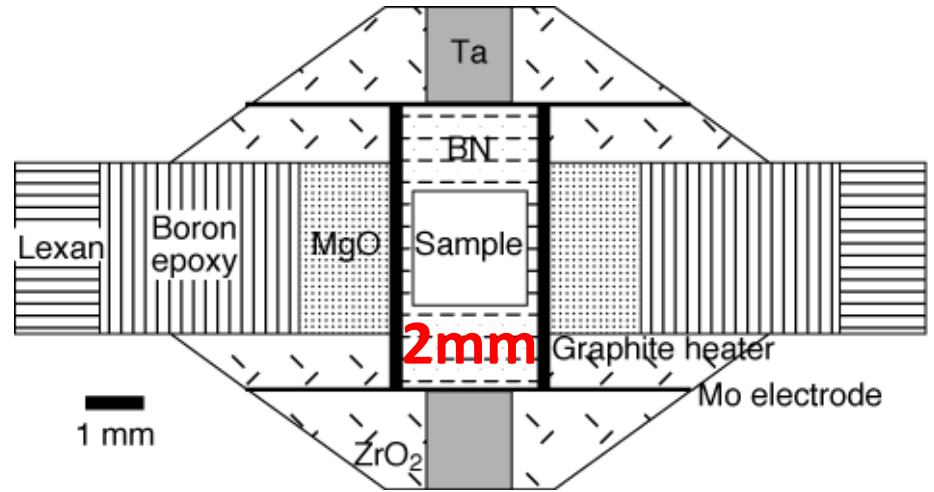
A standard PE cell at HPCAT



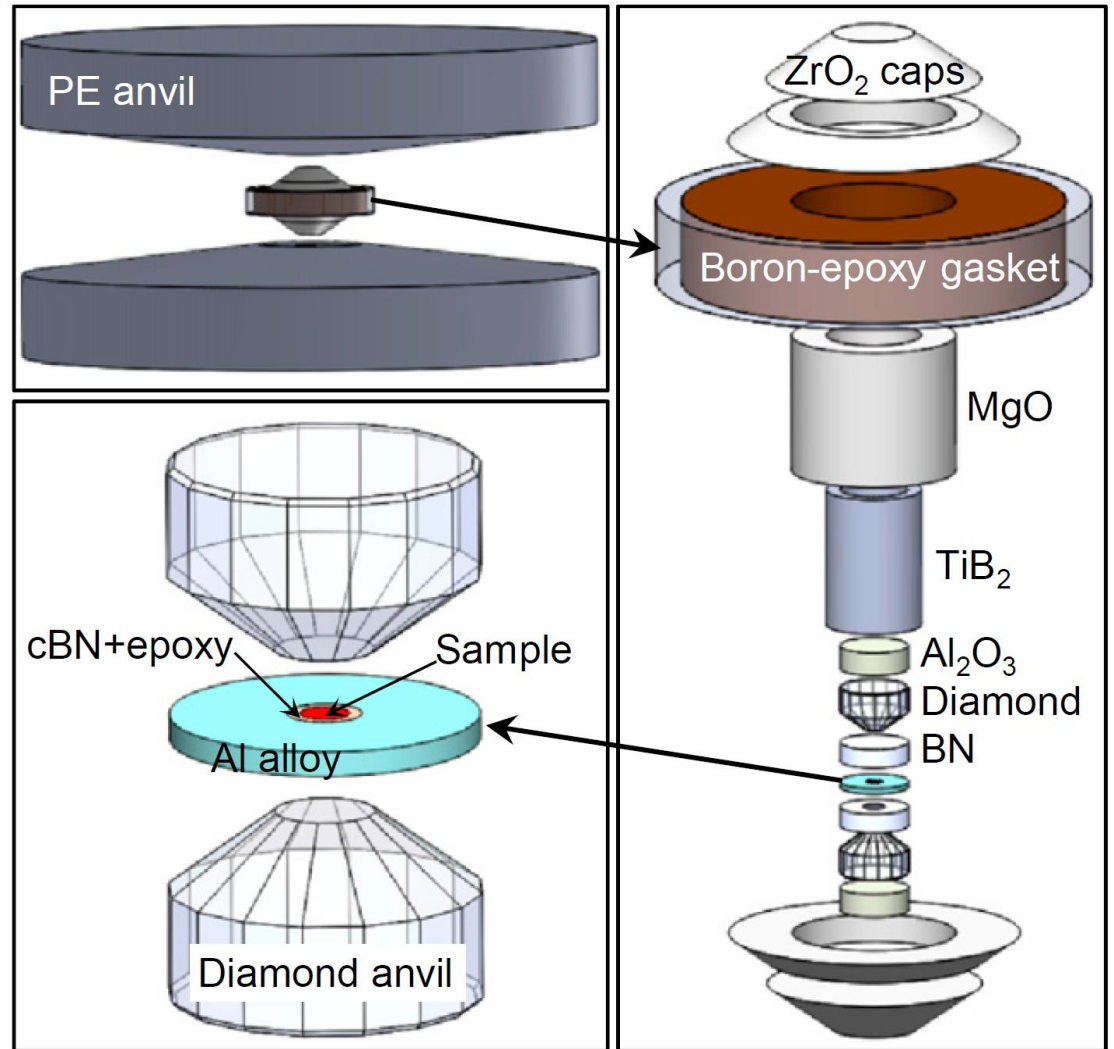
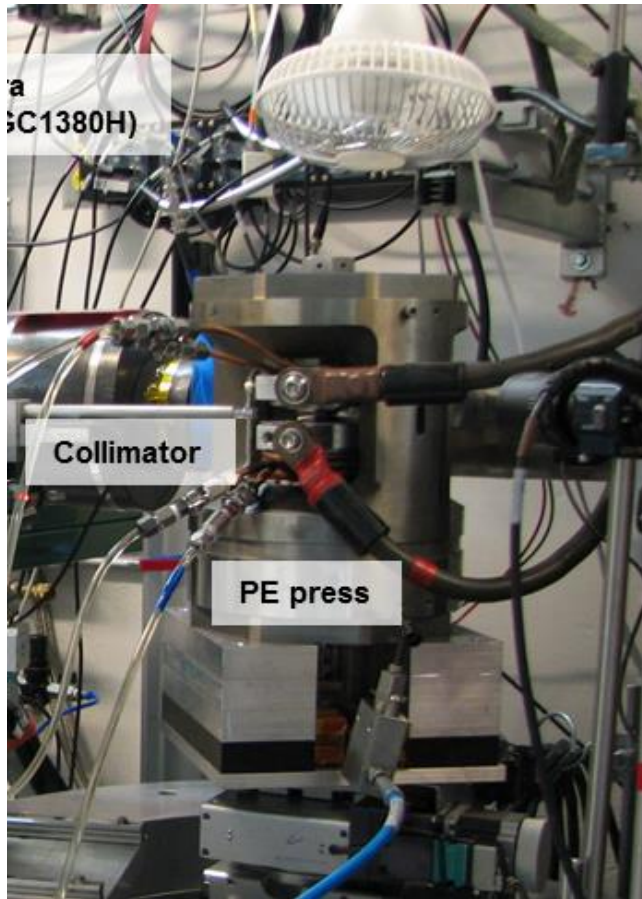
A standard PE cell at HPCAT

Large sample volume

Kono et al. (2014), Phys. Earth Planet. Inter., 228, 269-280



New double-stage large volume cell (Kono et al., 2016)



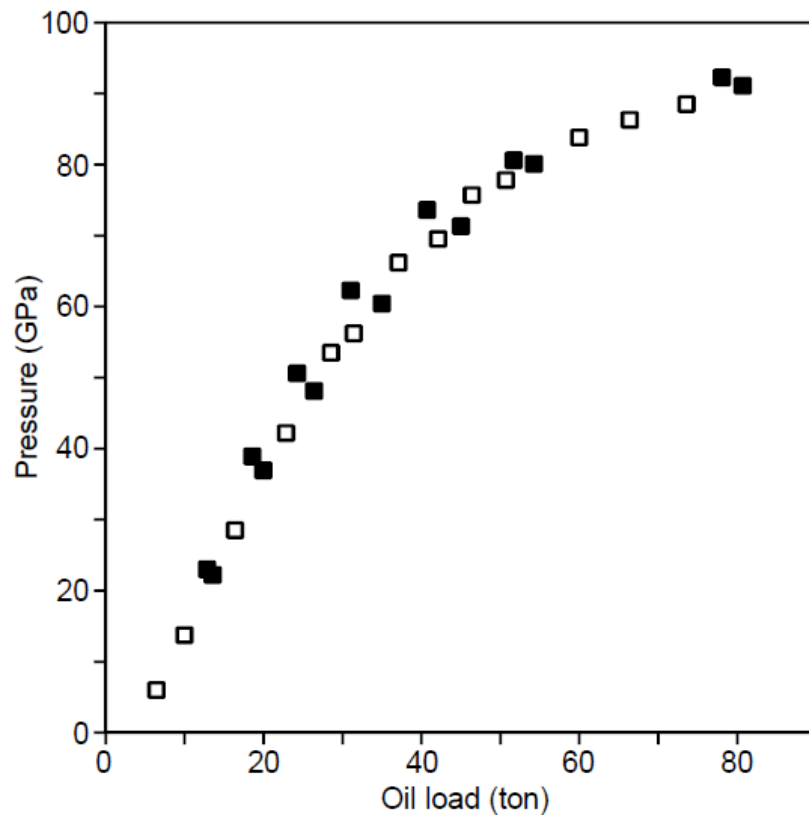
Kono et al. (2016)
PNAS, 113, 3436-3441

Sample size in 0.8 mm culet double-stage cell

Diamond anvil culet: 0.8 mm

Sample diameter: 0.3 mm

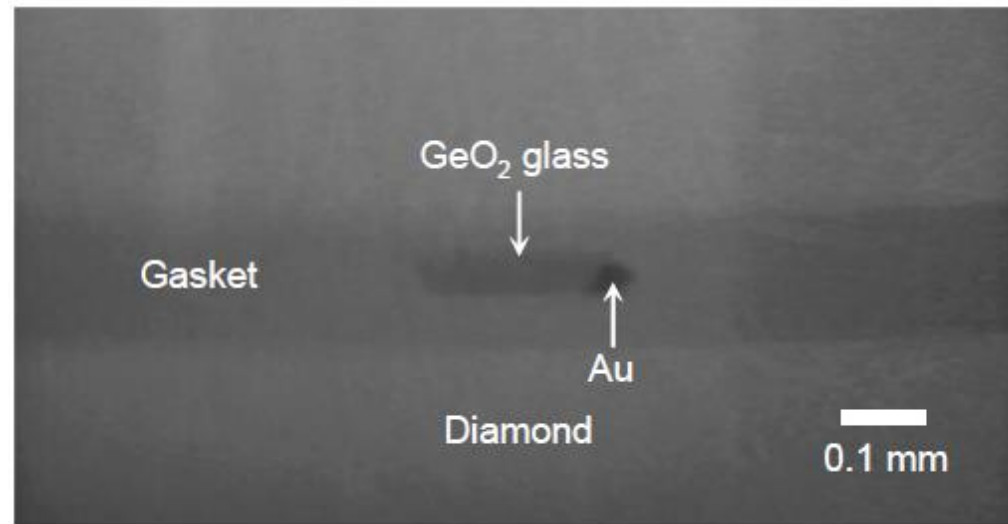
Sample thickness: 0.15 mm



Radiography image at 92 GPa

Sample diameter: 0.24 mm

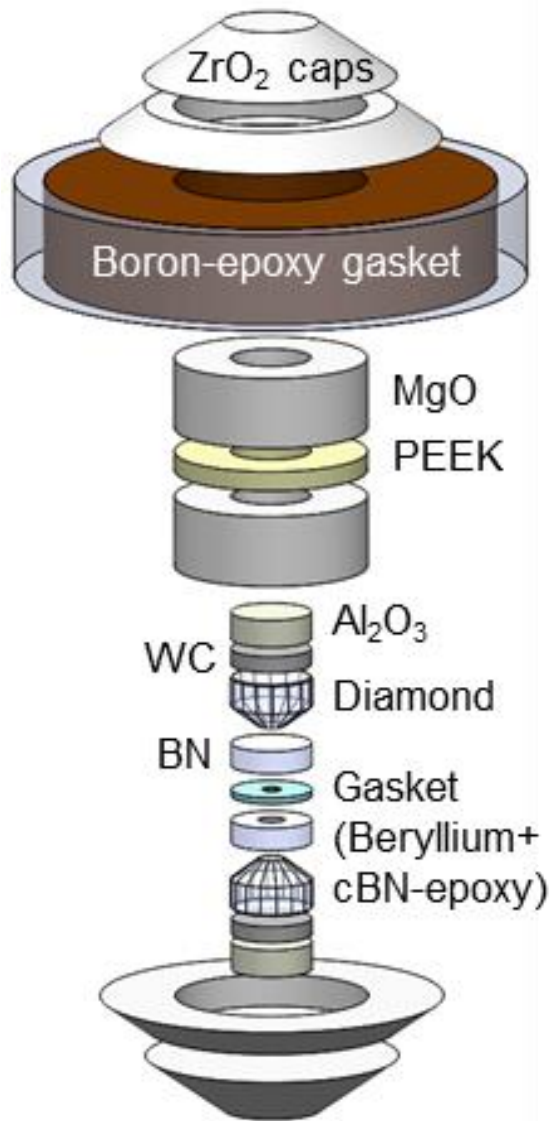
Sample thickness: 0.06 mm



Kono et al. (2016)

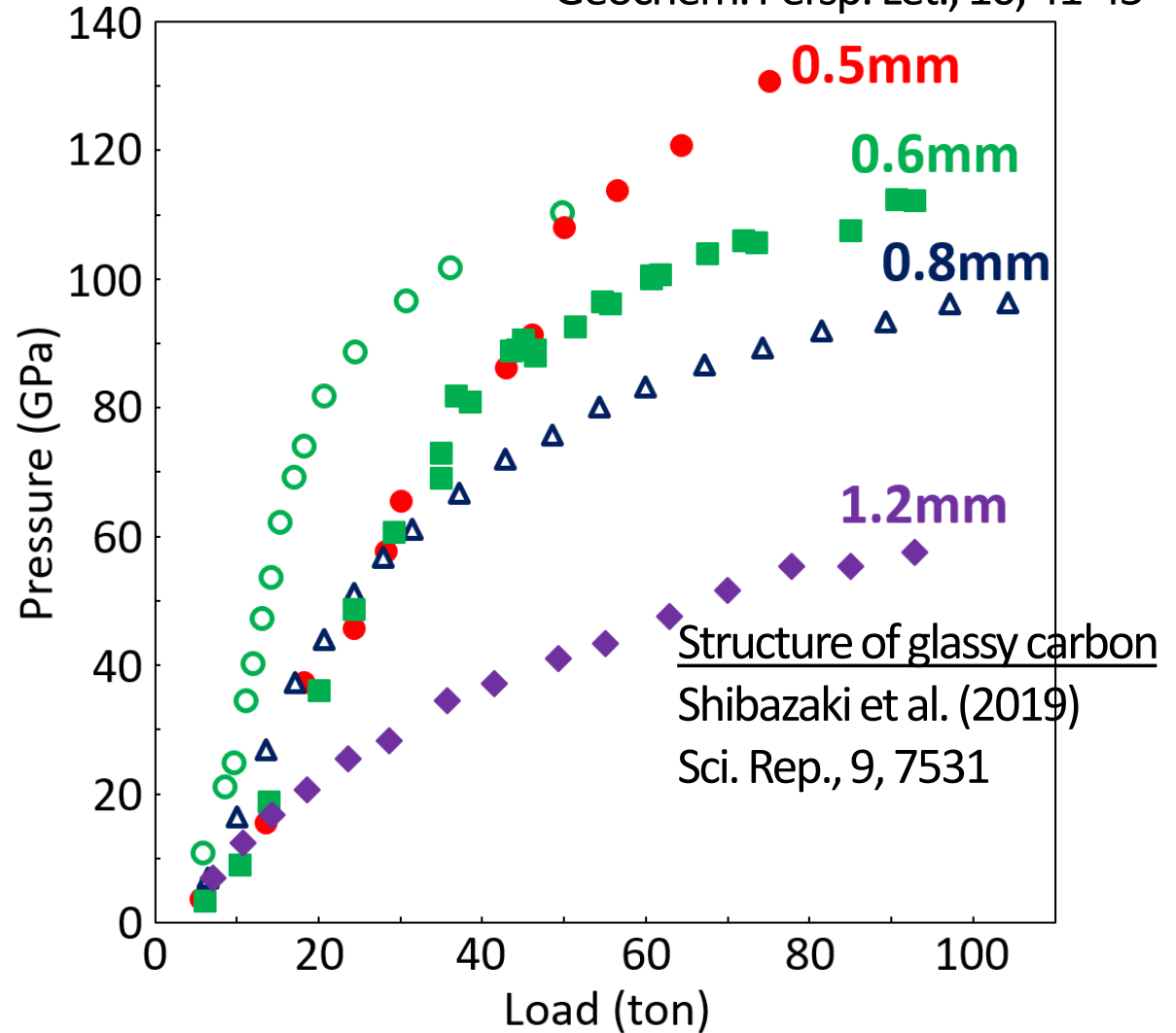
PNAS, 113, 3436-3441

Latest advances in ultrahigh pressure generation with large volume sample



Ohira et al. (2019)

Geochem. Persp. Let., 10, 41-45



Structure of glassy carbon

Shibazaki et al. (2019)

Sci. Rep., 9, 7531

-High-pressure experiments using large volume press.

-High-pressure synchrotron X-ray experiments combined with large volume press.

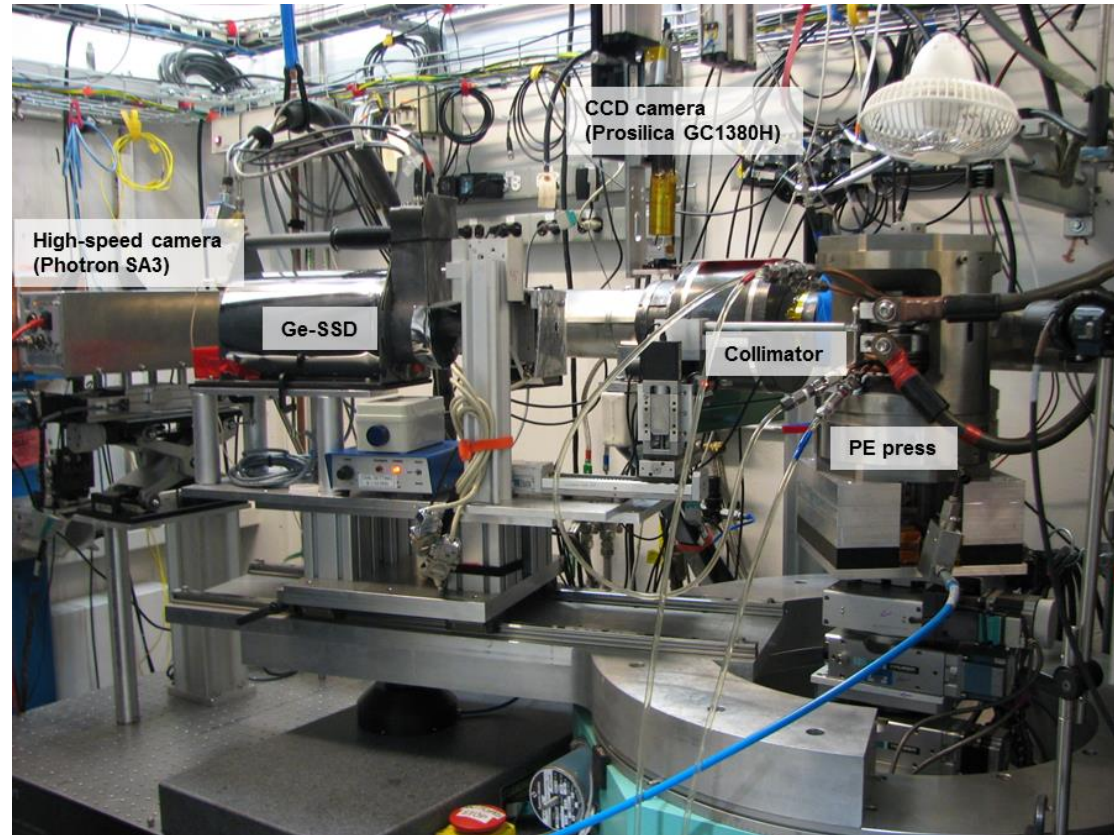
Synchrotron X-ray facilities



APS 16BMB: unique beamline optimized for comprehensive study of liquids and glasses at high pressures using Paris-Edinburgh press

- Structure of liquids and glasses
- Elastic wave velocities
- Viscosity
- Imaging of liquids

at high pressures and high temperatures using Paris-Edinburgh type large volume press.

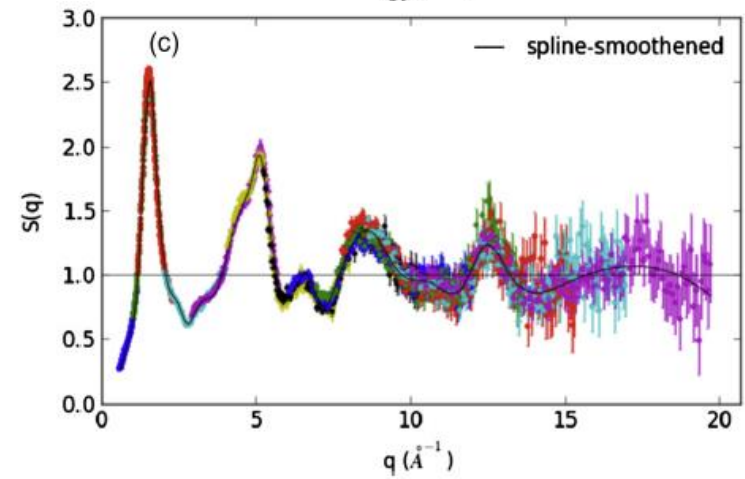
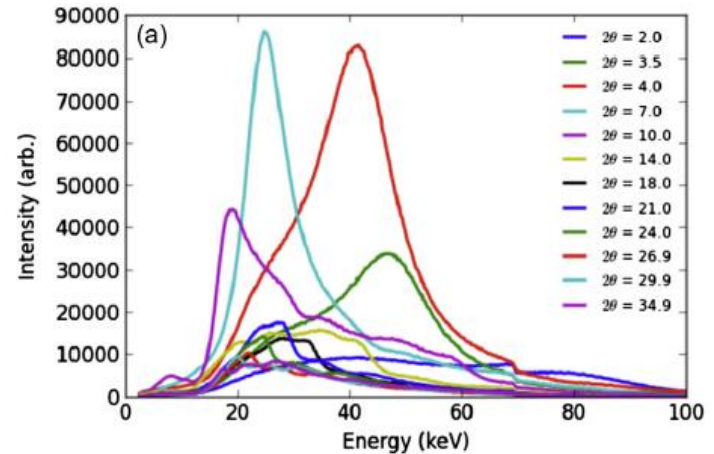
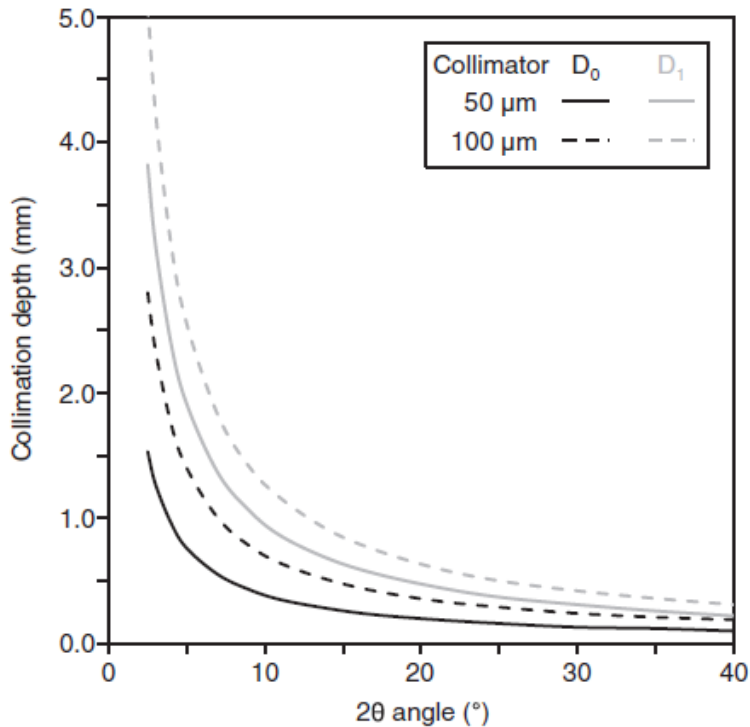
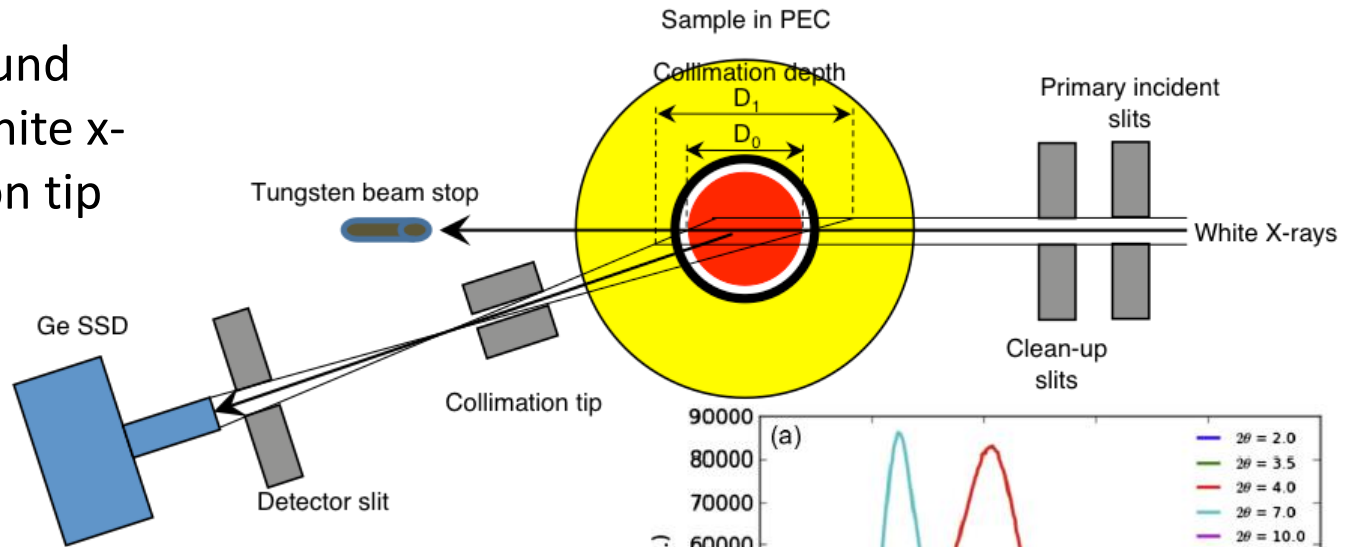


Kono et al. (2014), Phys. Earth Planet. Inter., 228, 269-280

Structure measurement of liquids and amorphous materials

Reducing background signals by using white x-ray with collimation tip

Kono et al. (2014),
Phys. Earth
Planet. Inter., 228,
269-280



Discovery of ultrahigh pressure polyamorphism in GeO_2 glass with coordination number >6

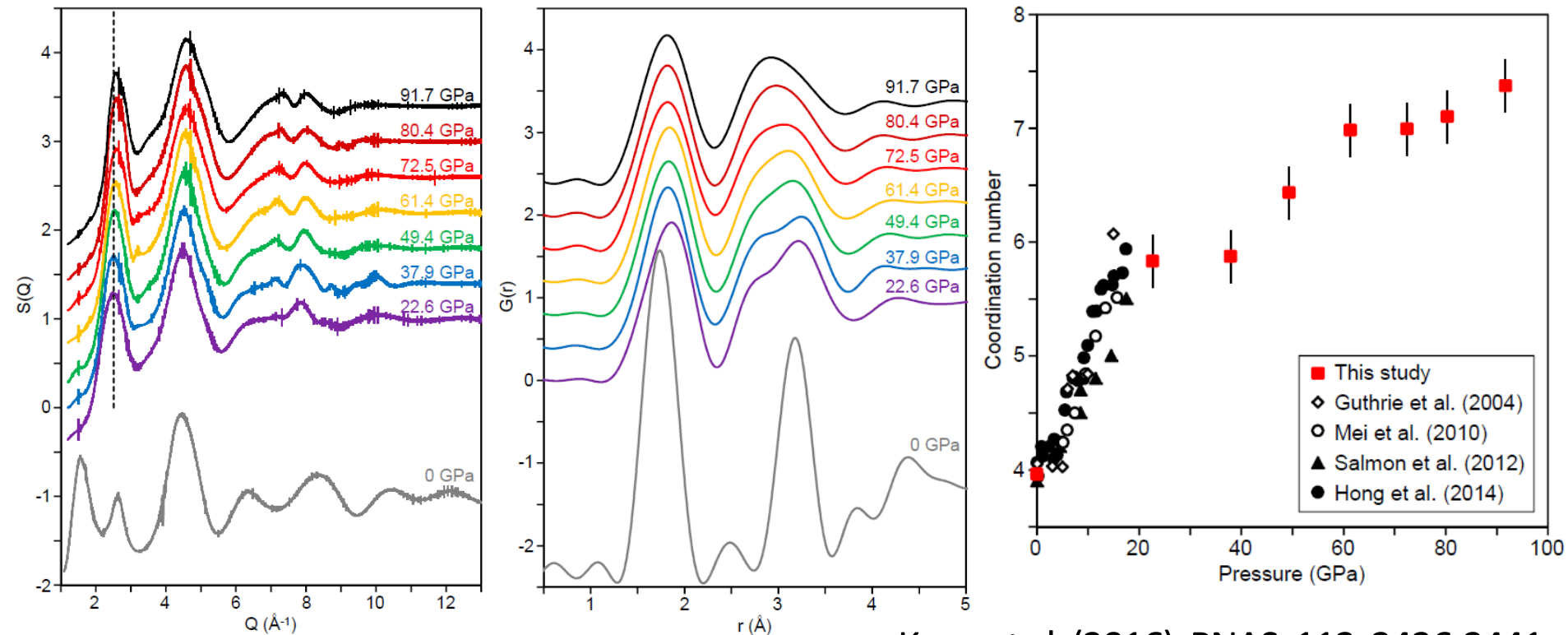
Ultrahigh-pressure polyamorphism in GeO_2 glass with coordination number >6

Yoshio Kono^{a,1}, Curtis Kenney-Benson^a, Daijo Ikuta^a, Yuki Shibazaki^b, Yanbin Wang^c, and Guoyin Shen^a

^aHigh Pressure Collaborative Access Team, Geophysical Laboratory, Carnegie Institution of Washington, Argonne, IL 60439; ^bFrontier Research Institute for Interdisciplinary Sciences, Tohoku University, Aoba-ku, Sendai 980-8578, Japan; and ^cCenter for Advanced Radiation Sources, The University of Chicago, Chicago, IL 60637

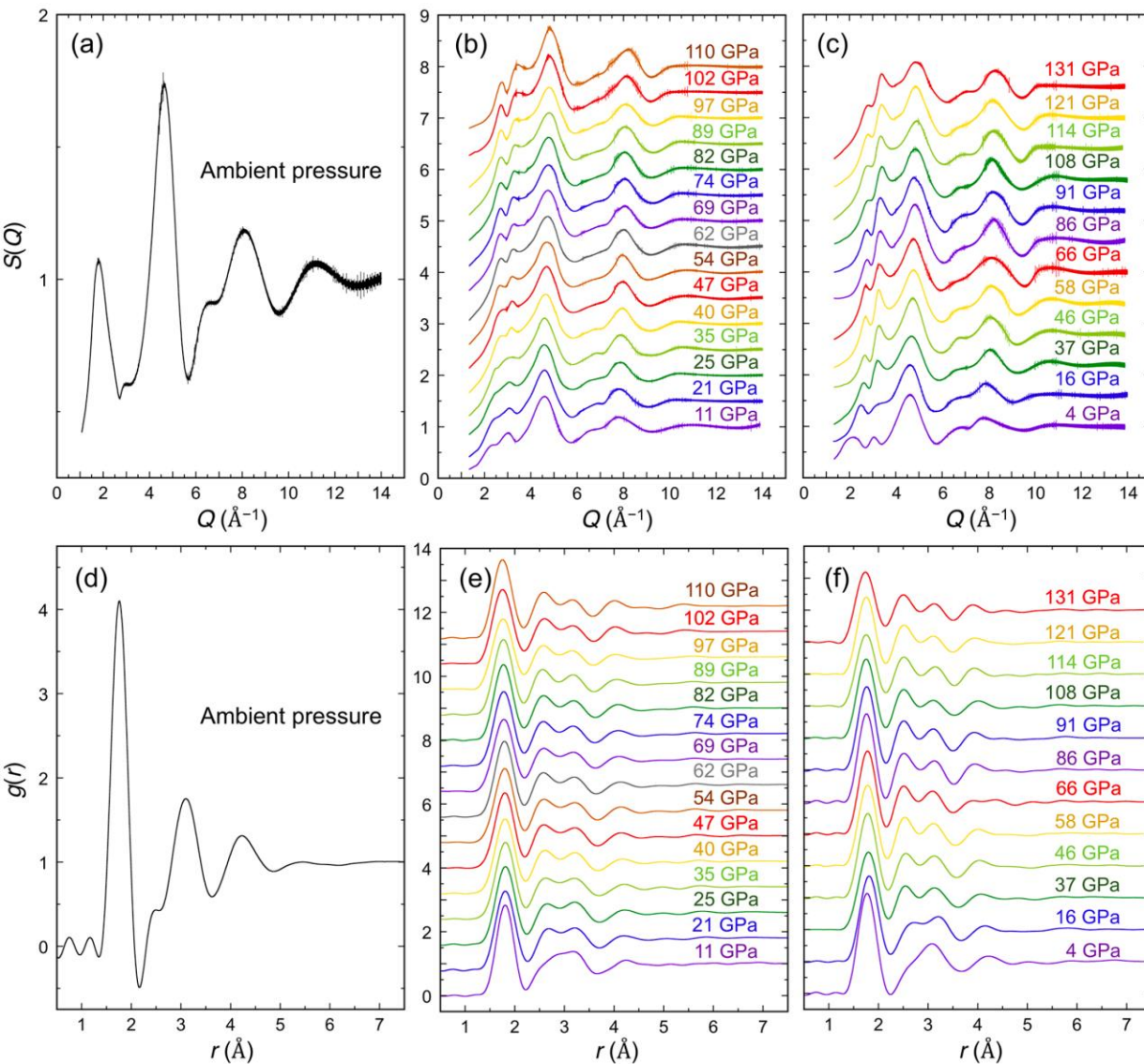
Edited by Alexandra Navrotsky, University of California, Davis, CA, and approved February 17, 2016 (received for review December 9, 2015)

Knowledge of pressure-induced structural changes in glasses is ... Brillouin scattering in a diamond anvil cell (DAC) showed a kink



Kono et al. (2016), PNAS, 113, 3436-3441

Structure of $\text{Al}_2\text{O}_3\text{-SiO}_2$ glass near the pressure of CMB



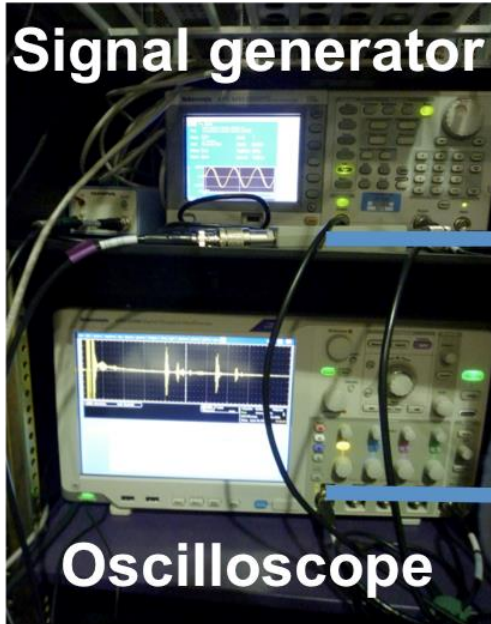
■ Ultrahigh pressure structural changes in a 60 mol. % Al_2O_3 -40 mol. % SiO_2 glass

I. Ohira^{1,2*}, Y. Kono^{1,3}, Y. Shibazaki^{4,5}, C. Kenney-Benson⁶, A. Masuno⁷, G. Shen⁶

Ohira et al. (2019)
Geochem. Persp. Let., 10, 41-45

Ultrasonic measurement

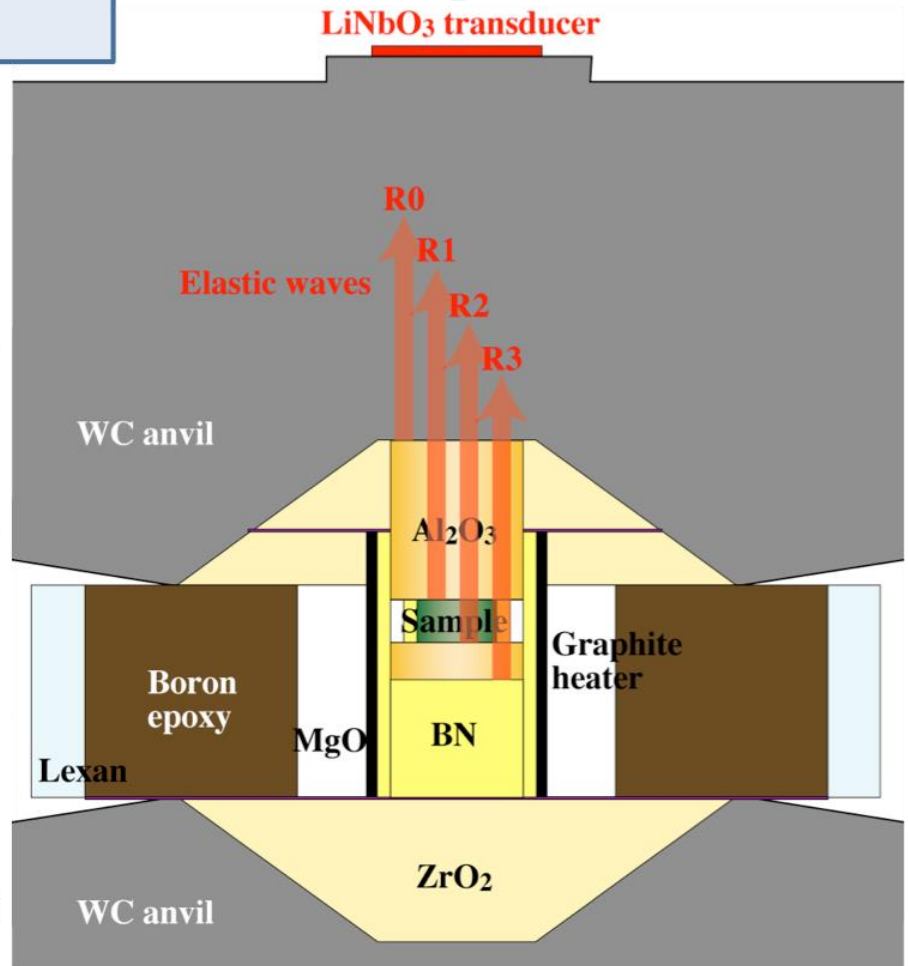
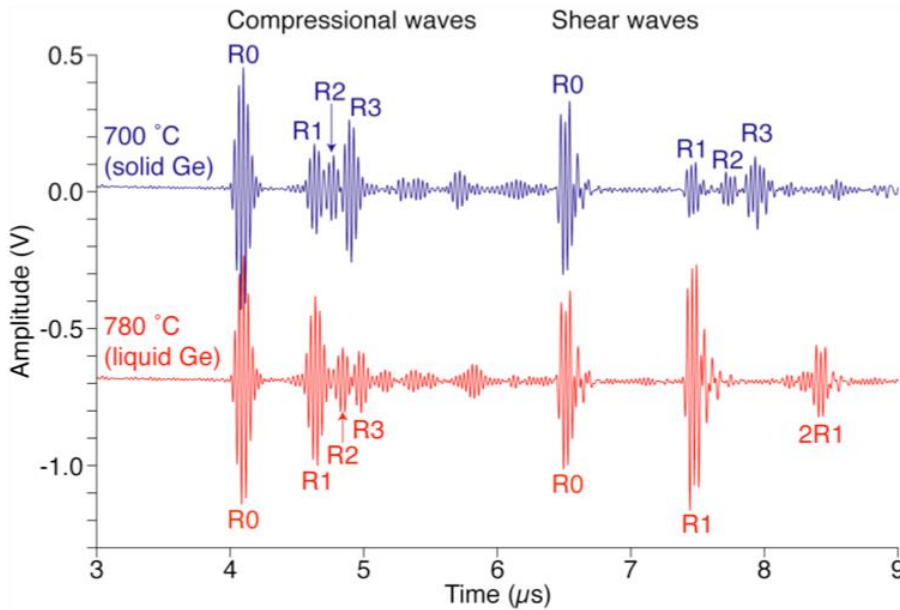
Kono et al. (2012), RSI, 83, 033905



Directional bridge

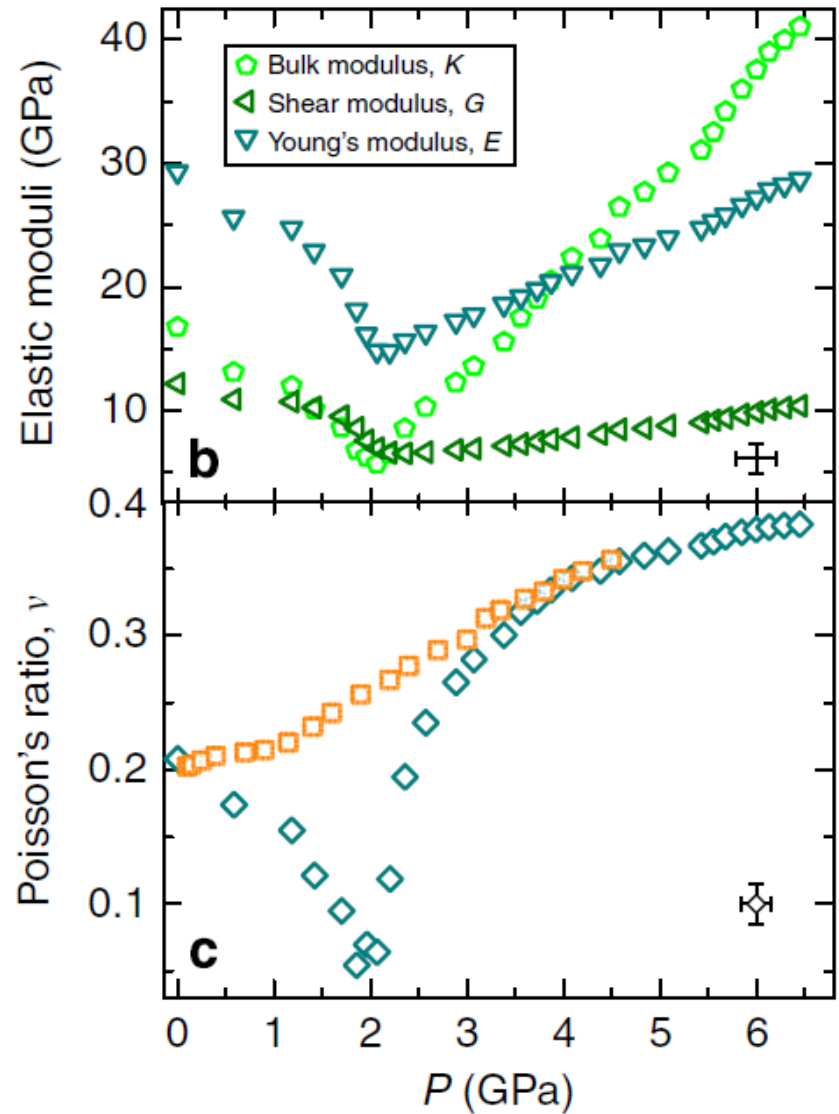
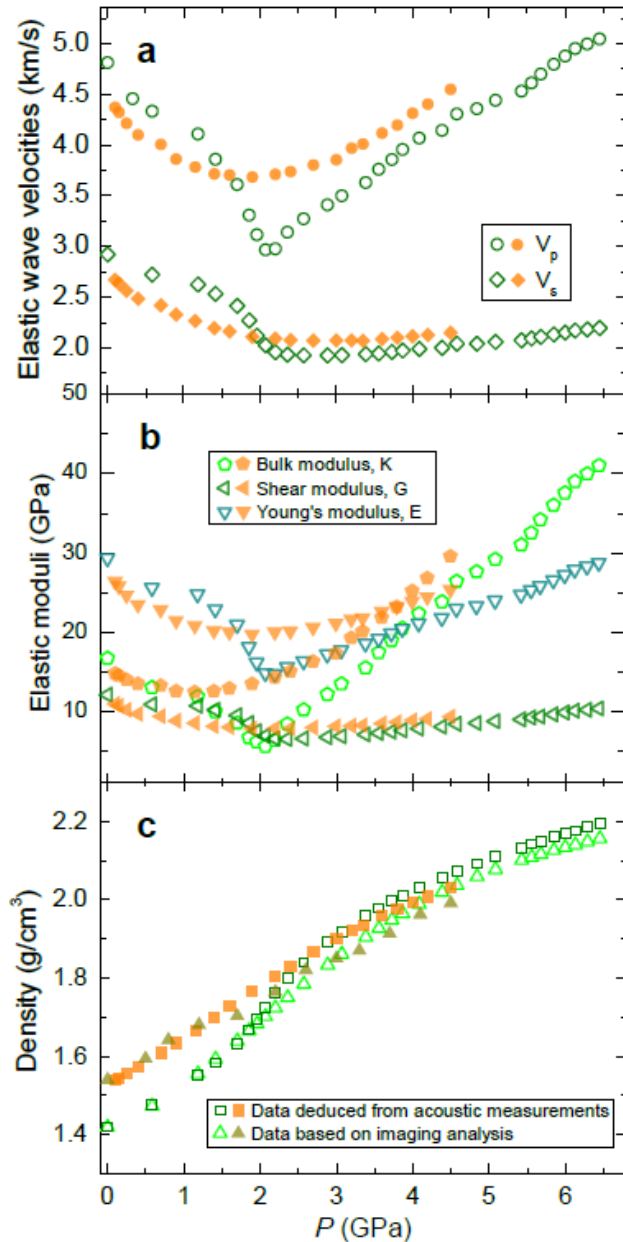
-40 dB

Amplifier
(+40 dB)



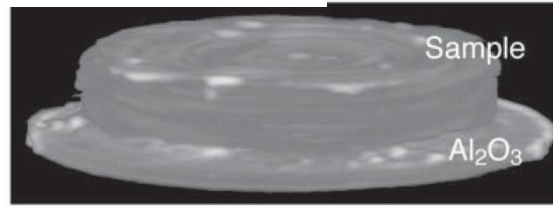
Anomaly in elastic properties in type-II grassy carbon

Type I
Type II



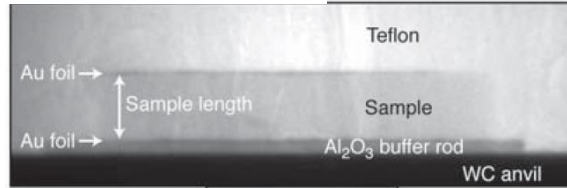
Combined elastic property and 3D tomography measurement at APS, 13BMD

Tomography



装置を回転しながら、2次元ラジオグラフィー像を収集

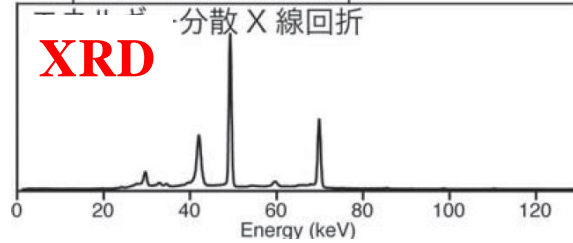
Radiography



CCD camera

Scintillator
Mirror

Solid state detector



Elastic wave velocity

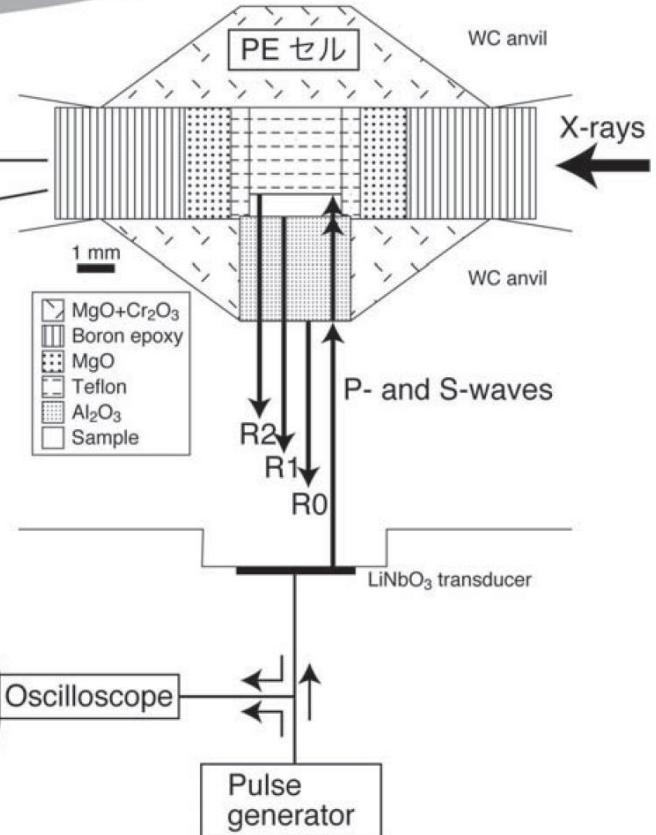
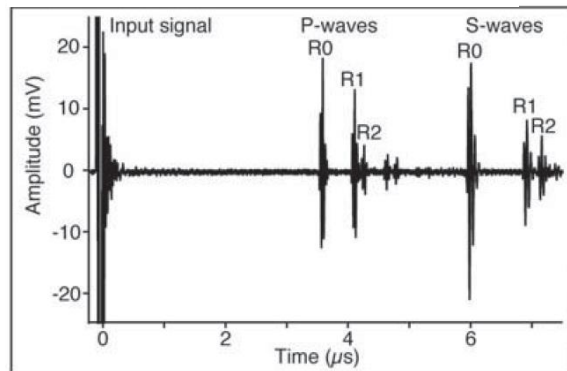


図2 Advanced Photon Source, ビームライン 13BM-Dにおける放射光X線測定と組み合わせた高圧下弾性波速度測定実験の概要。

Kono and Wang (2012),
Chikyū
Kono et al. (2011)
RSI, 82, 023906

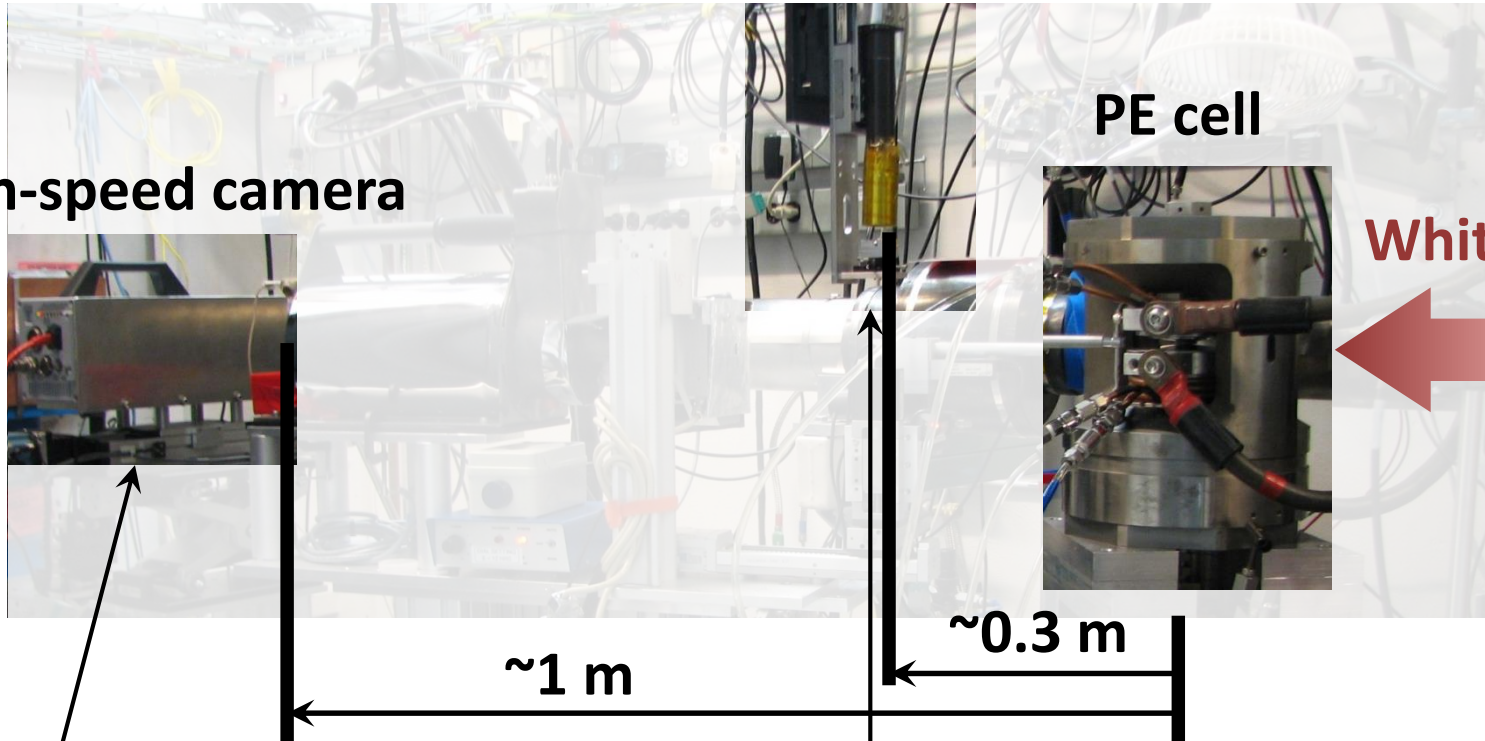
White X-ray imaging

CCD camera

High-speed camera

PE cell

White X-ray



~1 m

~0.3 m

Photron SA3

- typically 2.5 or 5.0 $\mu\text{m}/\text{pixel}$
- up to 120,000 frames/second

Purpose

- Fast imaging (e.g., viscosity)
- Phase contrast image

Prosilica GC1380H

- 0.95 $\mu\text{m}/\text{pixel}$
- <30 frames/second

Purpose

- Static and low speed imaging
- Sample shape determination

Fast imaging capability at 16-BM-B

Exposure time:
(Frame rate)

200 μ s
(5000 fps)

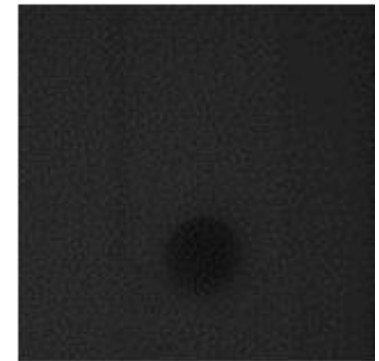
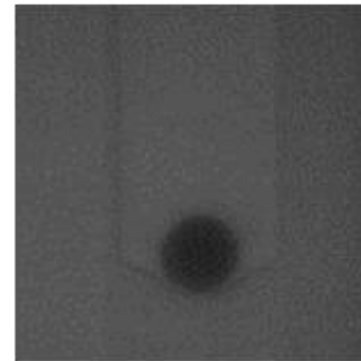
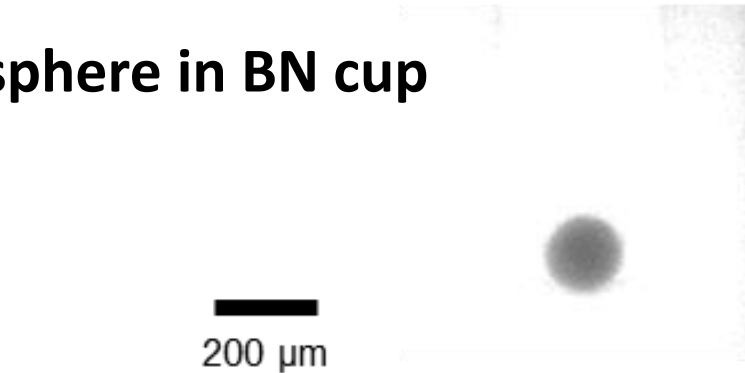
100 μ s
(10000 fps)

25 μ s
(40000 fps)

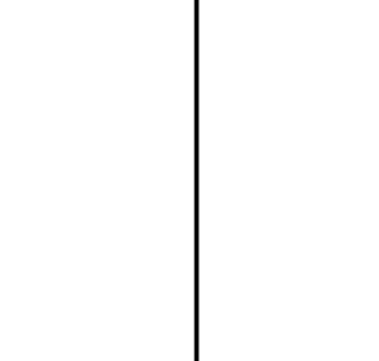
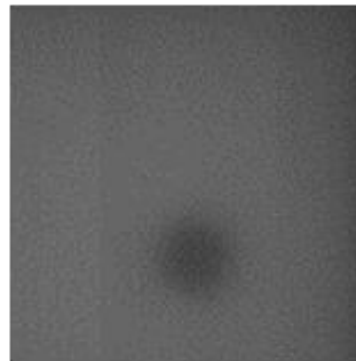
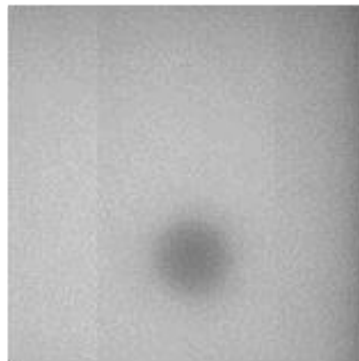
10 μ s
(100000 fps)

Pt sphere in BN cup

No PE cell

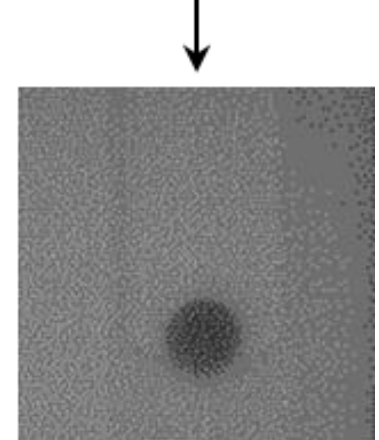
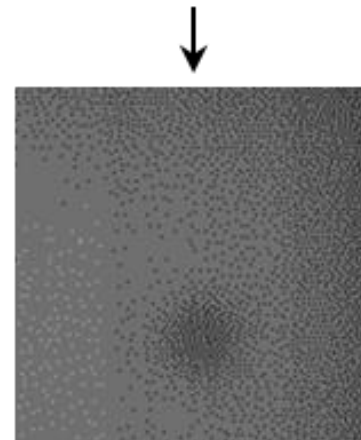


In PE cell



The imaging speed is more than 100 times faster than previous 'high-speed imaging' in large volume press (typically 30-60 fps)

After brightness adjustment



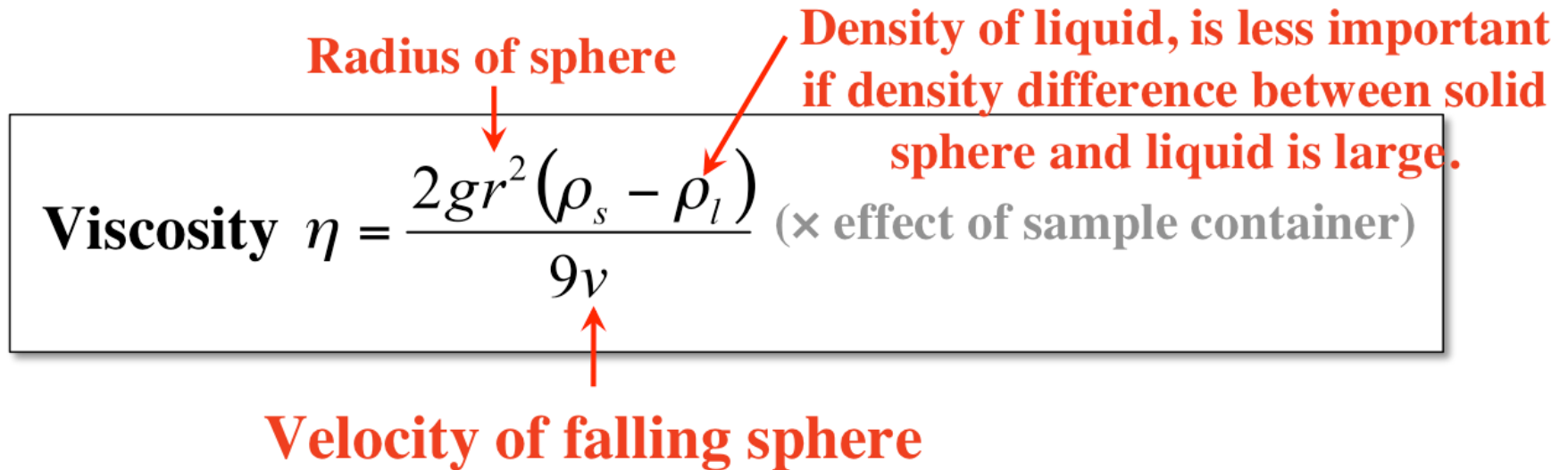
Falling sphere viscosity measurement

Radius of sphere

Density of liquid, is less important if density difference between solid sphere and liquid is large.

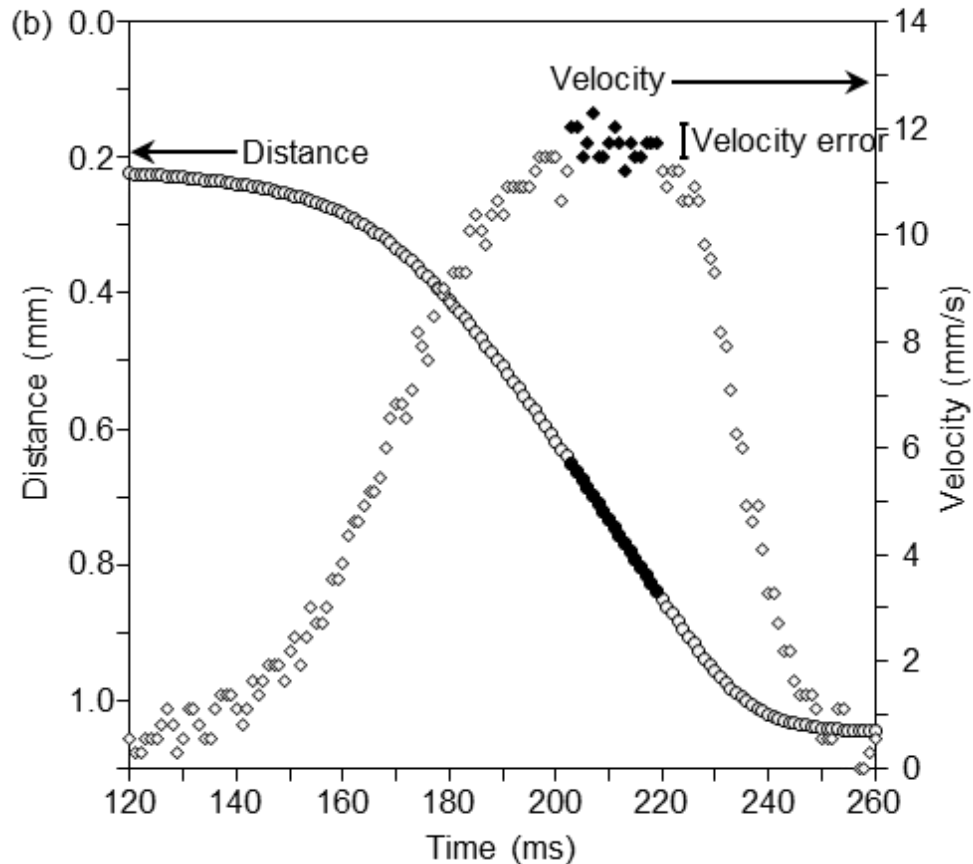
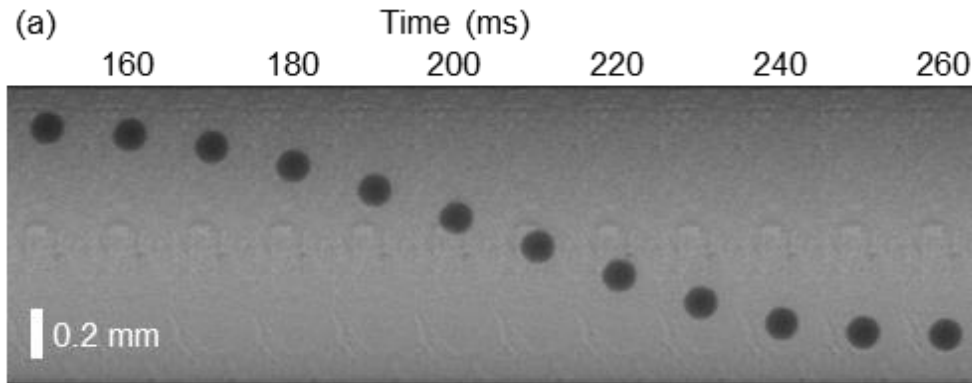
Viscosity $\eta = \frac{2gr^2(\rho_s - \rho_l)}{9v}$ (\times effect of sample container)

Velocity of falling sphere

The diagram shows the equation for viscosity measurement using a falling sphere. The equation is enclosed in a black-bordered box. Three red arrows point from text labels to parts of the equation: one from 'Radius of sphere' to the 'r' in the numerator, one from 'Density of liquid, is less important if density difference between solid sphere and liquid is large.' to the 'rho_l' in the numerator, and one from 'Velocity of falling sphere' to the 'v' in the denominator. The word 'Viscosity' is written in bold black text to the left of the equation.

- Accurate determination of velocity of falling ball is essential to precisely determine viscosity of low-viscosity materials.

Analysis of falling sphere viscosity measurement



Kono et al. (2014)
Nature Comm., 5, 5091

Ultralow viscosity of carbonate melt

Kono et al. (2014)
Nature Comm., 5, 5091

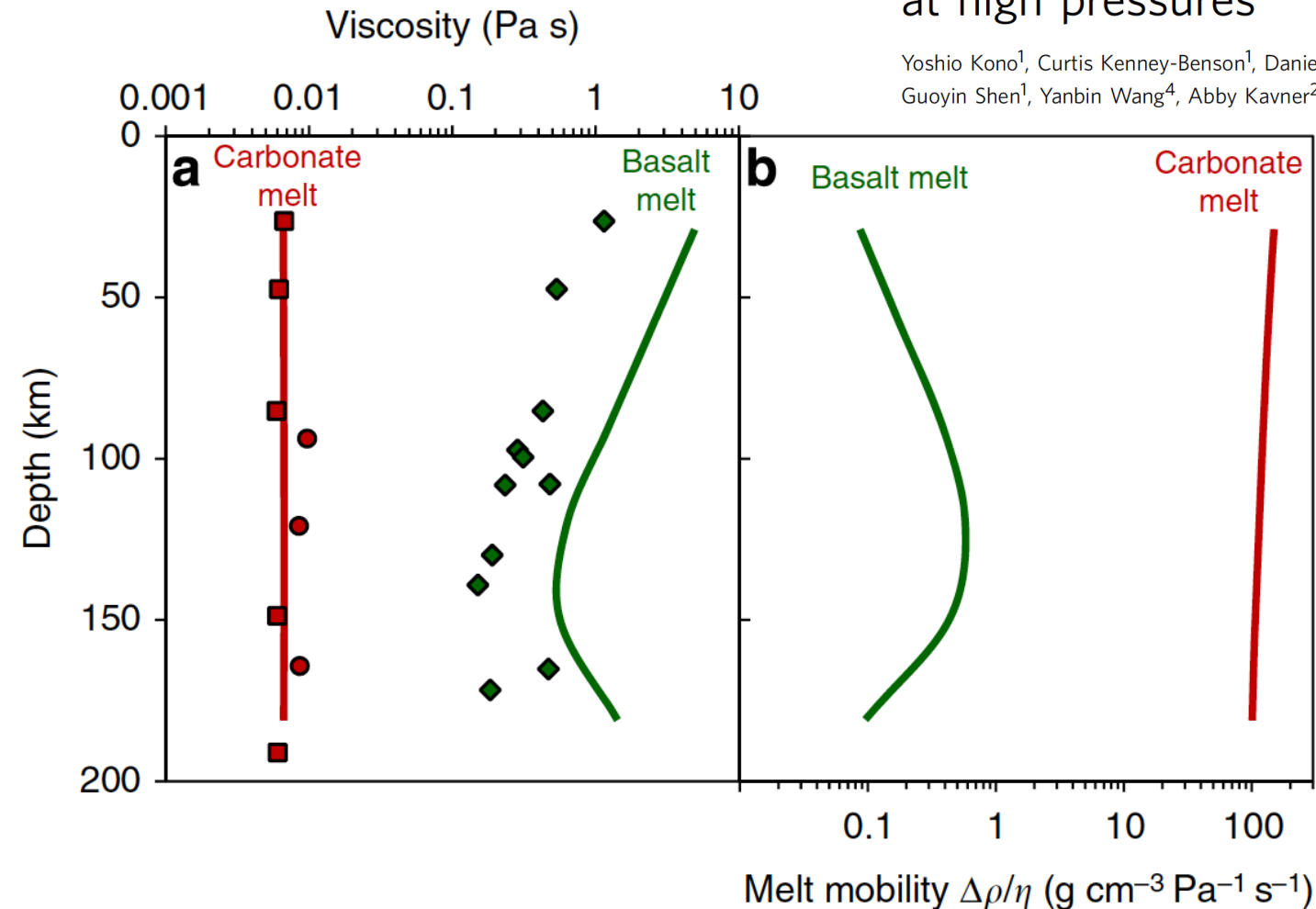
ARTICLE

Received 21 Jan 2014 | Accepted 28 Aug 2014 | Published 14 Oct 2014

DOI: 10.1038/ncomms6091

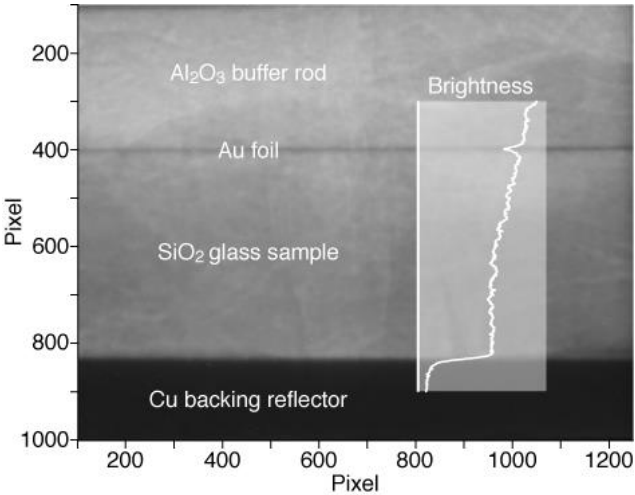
Ultralow viscosity of carbonate melts at high pressures

Yoshio Kono¹, Curtis Kenney-Benson¹, Daniel Hummer², Hiroaki Ohfuji³, Changyong Park¹, Guoyin Shen¹, Yanbin Wang⁴, Abby Kavner² & Craig E. Manning²

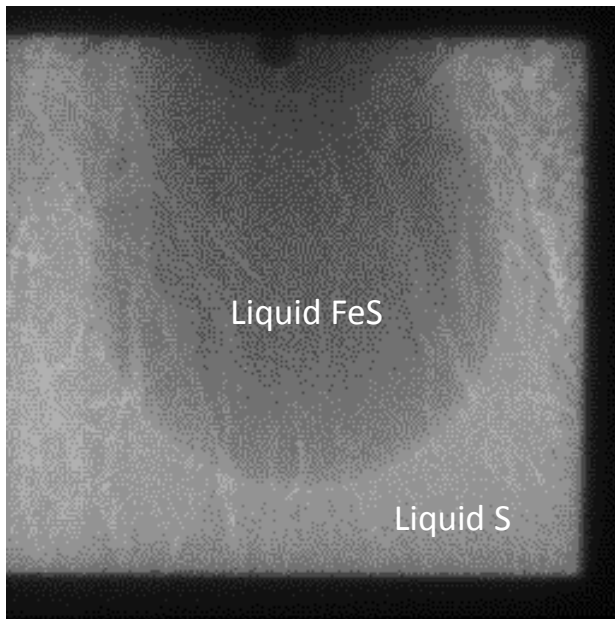


Sample shape

-Sample length

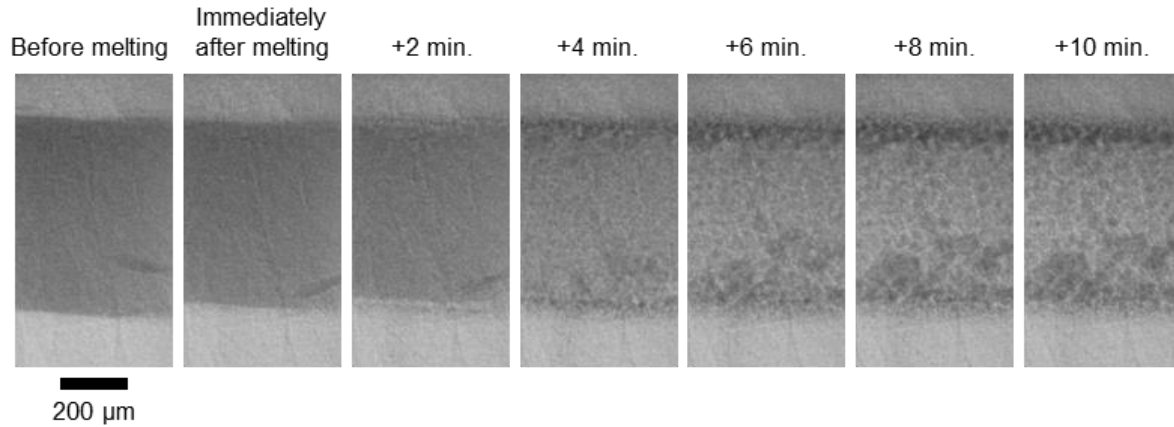


-Surface tension

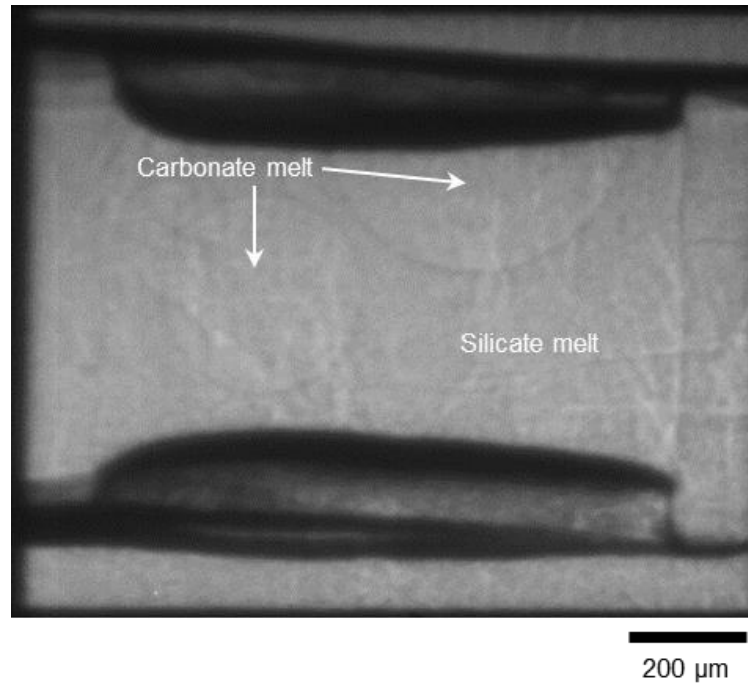


Liquid phase separation

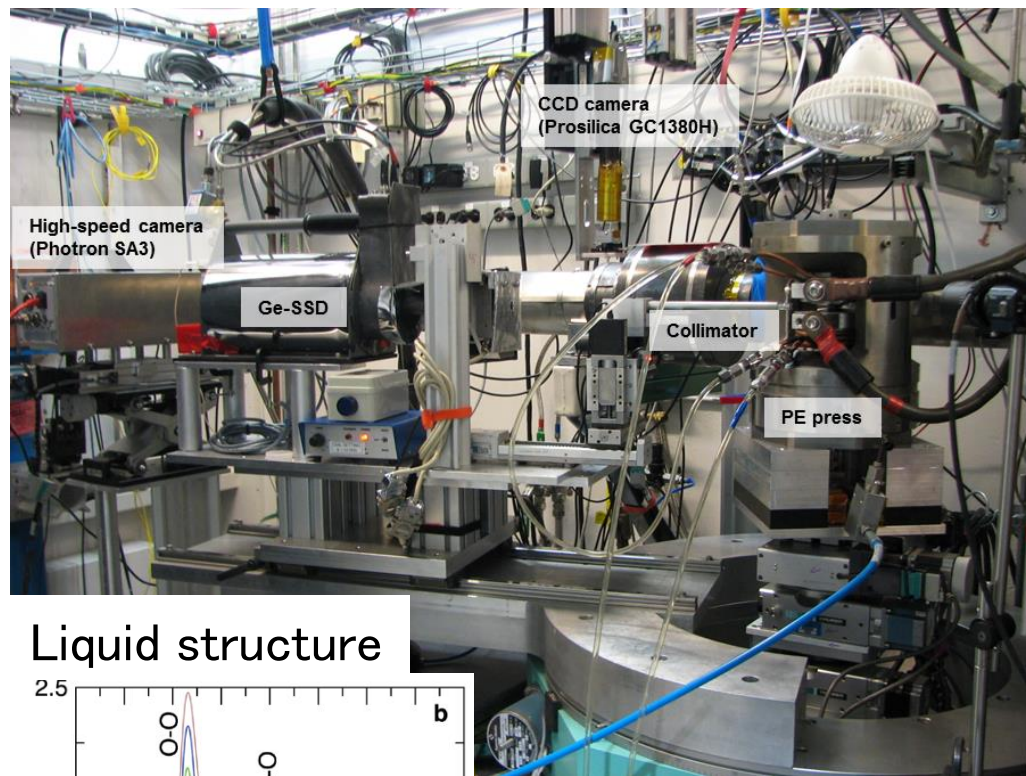
Sample: lithium germanate borate



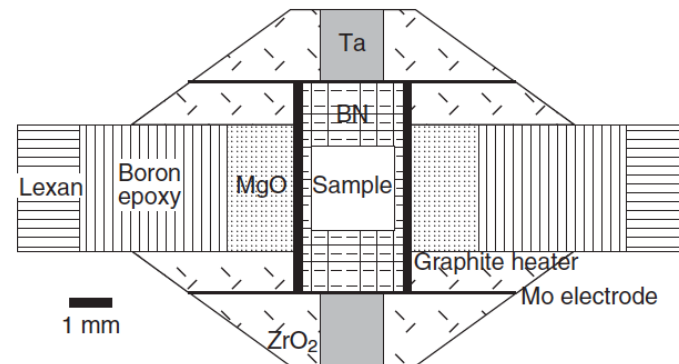
Silicate and carbonate melts immiscibility



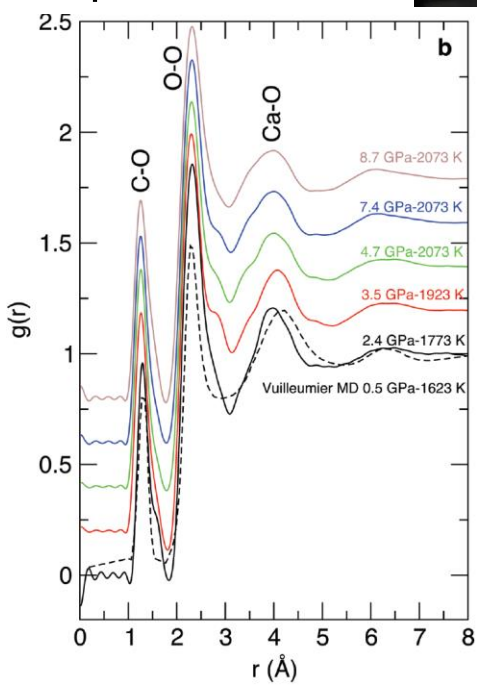
Comprehensive study of liquids under pressure



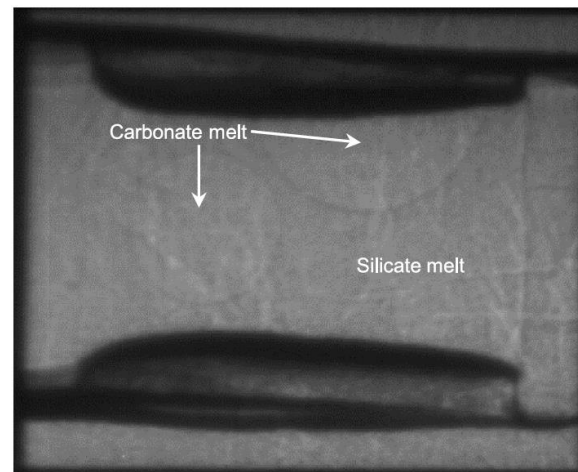
Large volume press experiment



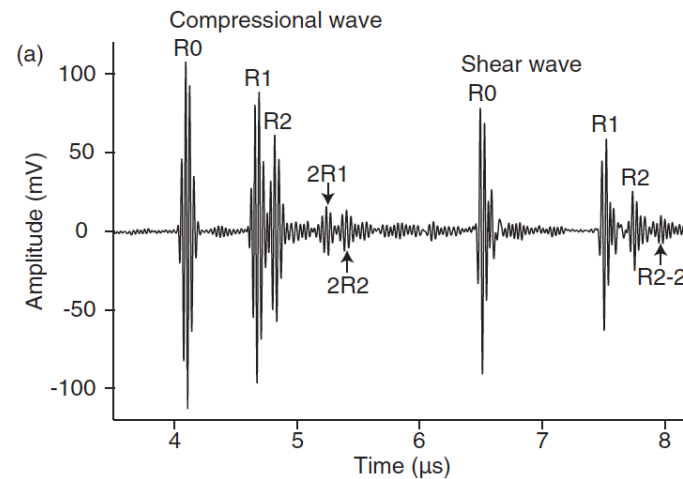
Liquid structure



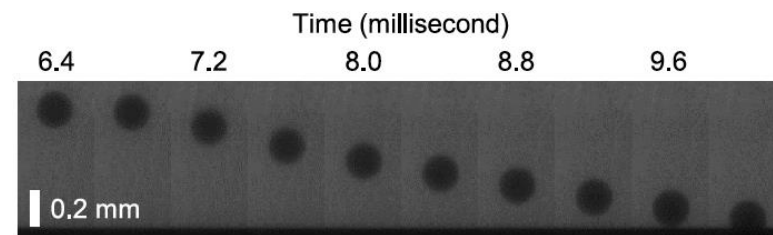
Imaging of magmas



Ultrasonic measurement

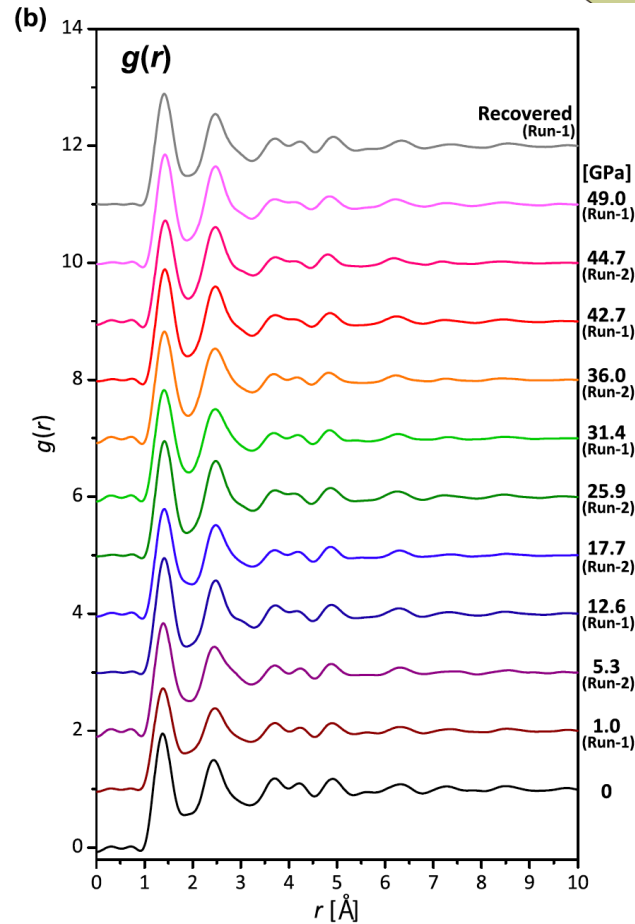
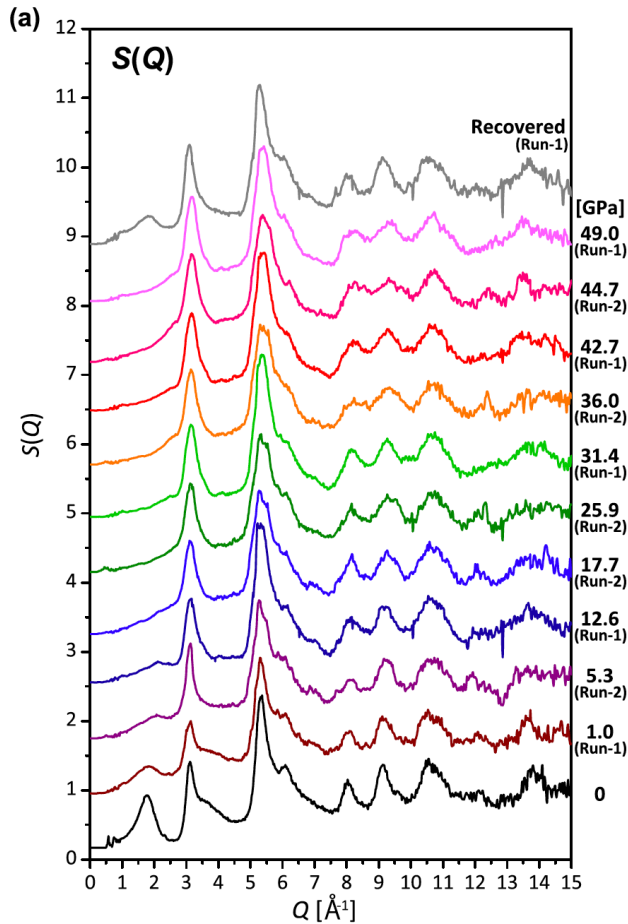
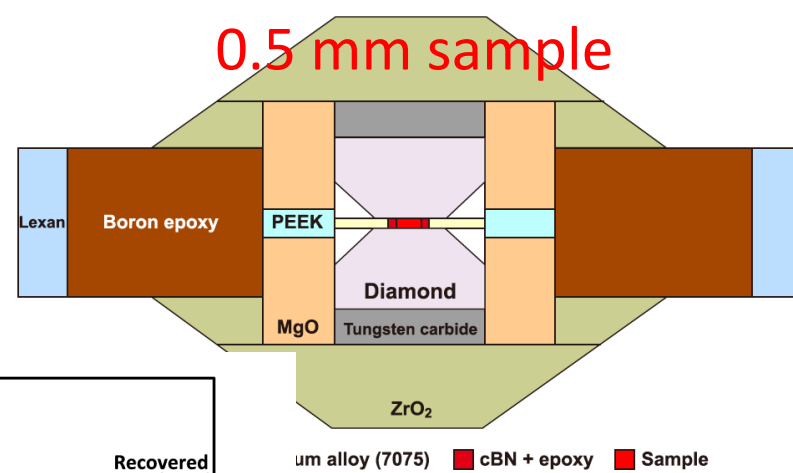


Viscosity of liquids



Measurement of structure of glassy carbon at 49 GPa

9 hours acquisition time for 1 data point



Shibazaki et al. (2019)
Sci. Rep., 9, 7531

Measurement of structure of lower Z amorphous solids and liquids at higher pressures is challenge for the future.

Summary

-Combination of large volume press and synchrotron X-ray measurement is a powerful tool to investigate structure and properties of liquids and glasses at in situ high pressure and high temperature conditions.

-Study of structure of liquids and glasses under extreme conditions are still challenging due to weak X-ray scattering.

-High-brilliance X-ray after the upgrade of synchrotron X-ray facilities such as ESRF-EBS would open more opportunity to investigate liquids and glasses under extreme conditions.

References

[1] Y. Kono and C. Sanloup, Magmas Under Pressure: Advances in High-Pressure Experiments on Structure and Properties of Melts (2018).

[2] Y. Kono, C. Park, C. Kenney-Benson, G. Shen, and Y. Wang, Physics of the Earth and Planetary Interiors 228, 269-280 (2014).