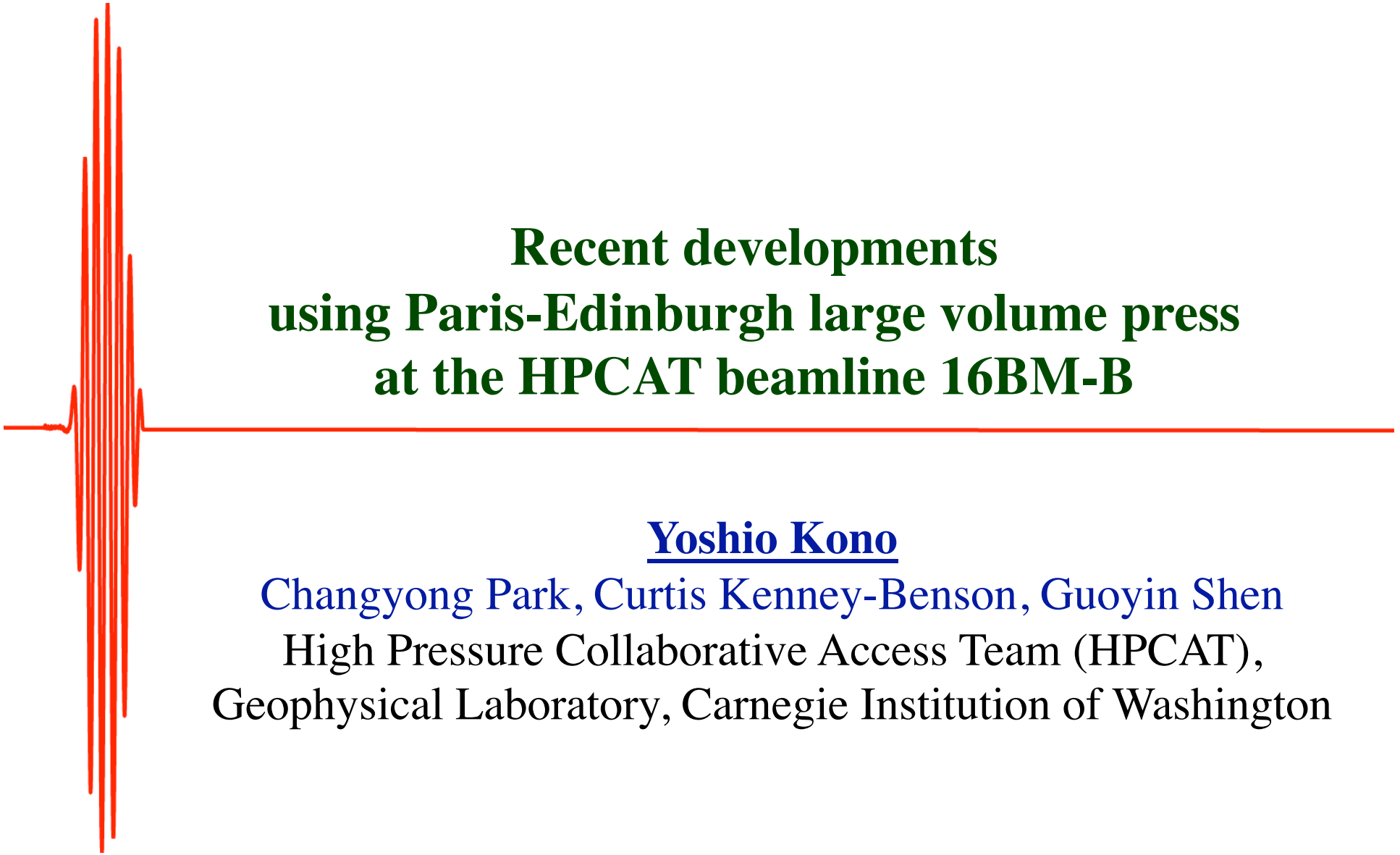

Recent developments using Paris-Edinburgh large volume press at the HPCAT beamline 16BM-B

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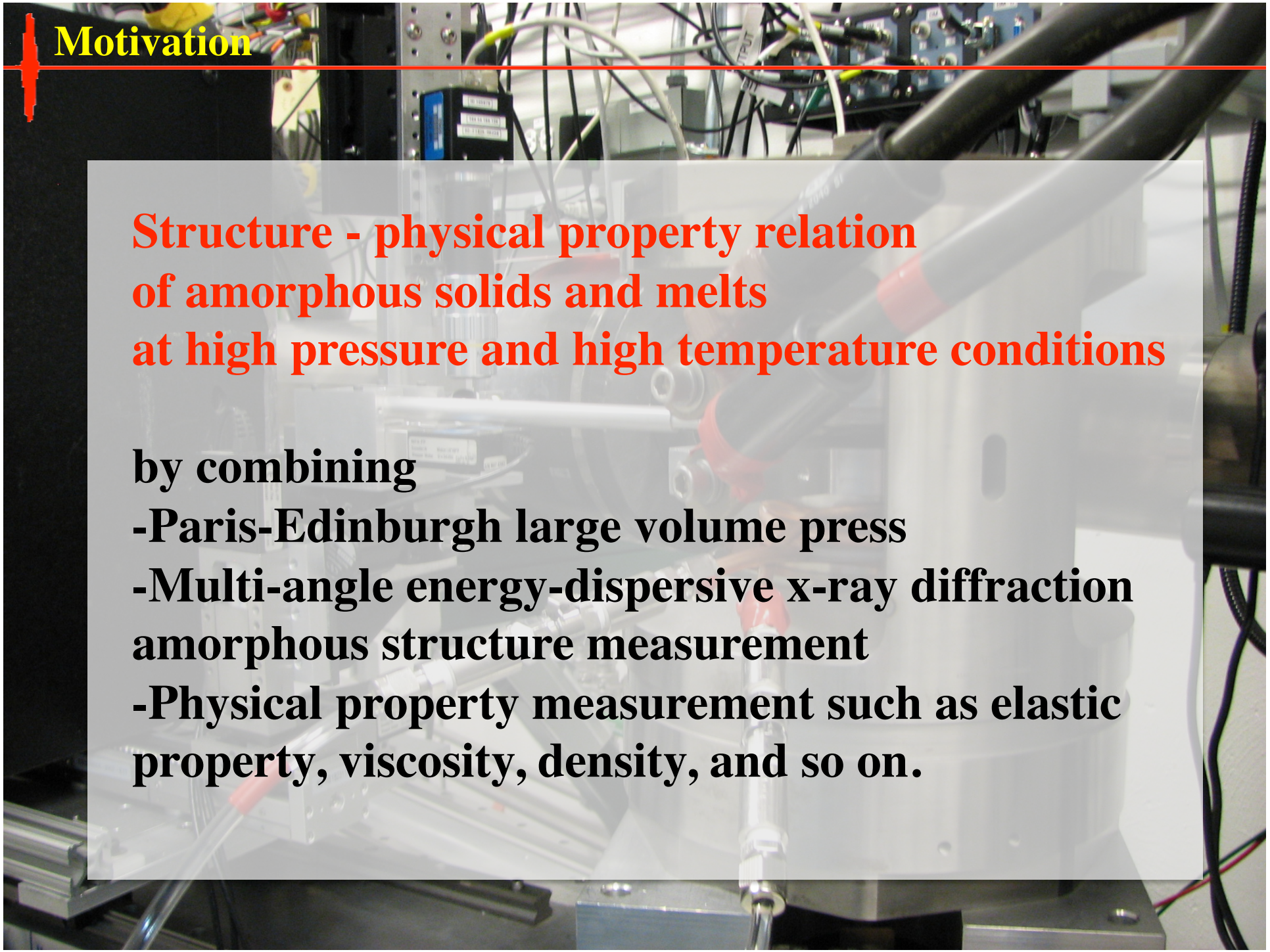


Motivation

Structure - physical property relation of amorphous solids and melts at high pressure and high temperature conditions

by combining
-Paris-Edinburgh large volume press
-Multi-angle energy-dispersive x-ray diffraction amorphous structure measurement
-Physical property measurement such as elastic property, viscosity, density, and so on.

Established technique and recent development

Established technique:

-High-pressure amorphous structure measurement in Paris-Edinburgh cell (PEC) up to 7 GPa and 2000 ˚C

Recent development: Physical property measurements at high PT

-Ultrasonic elastic wave velocity measurement

-Liquid viscosity measurement

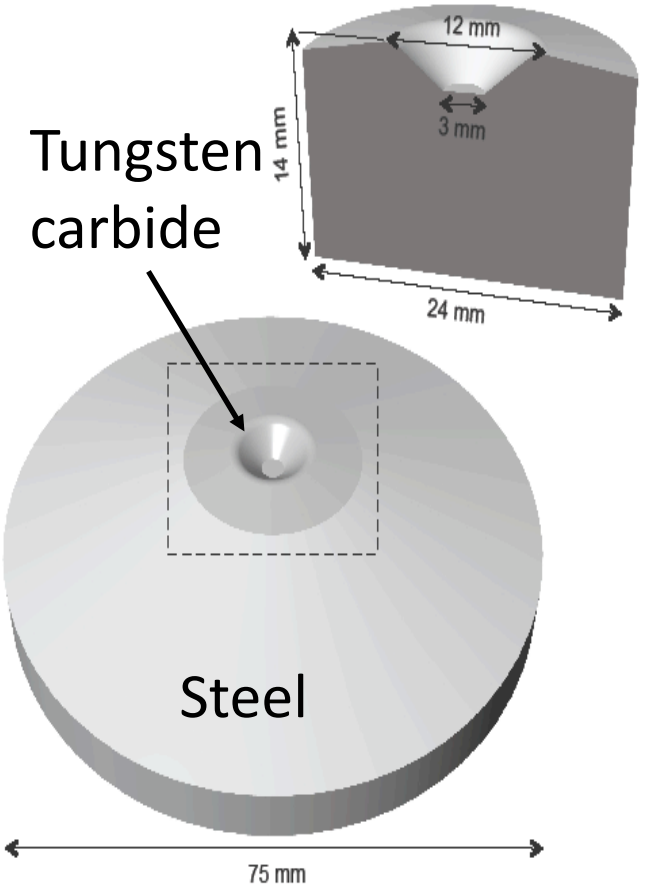
Paris-Edinburgh large volume press at 16BM-B, HPCAT

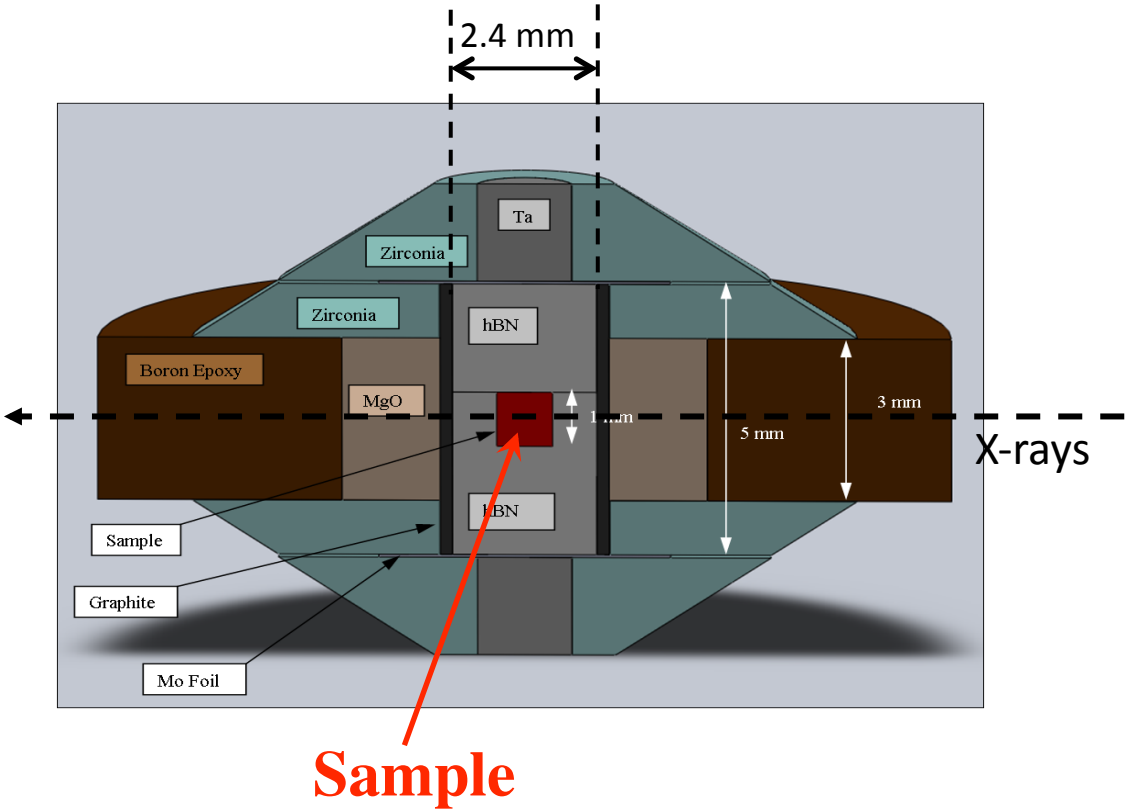
-Large sample volume for amorphous scattering

-Wide angle accessibility (2θ to ~40˚) for high Q

-Stable (>3 days) high pressure and high temperature experiment to ~7 GPa and ~2000 ˚C

WC anvil and sample assembly





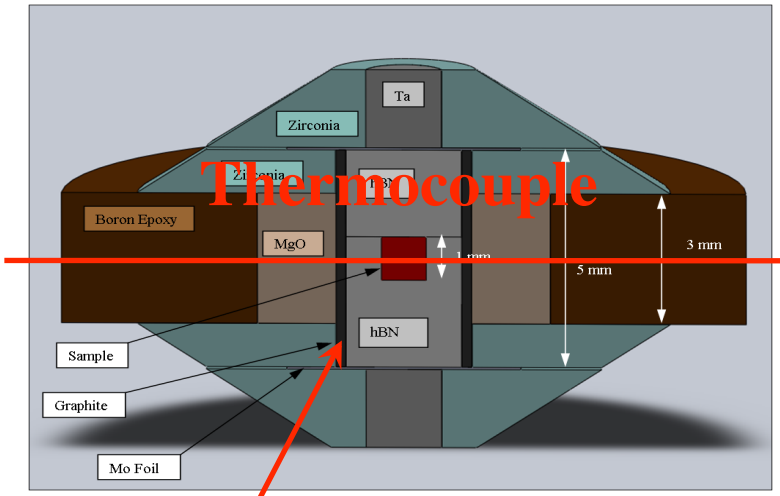
Large sample volume:

Diameter: up to 2 mm

Height: ~1.0 mm height at ~2 GPa

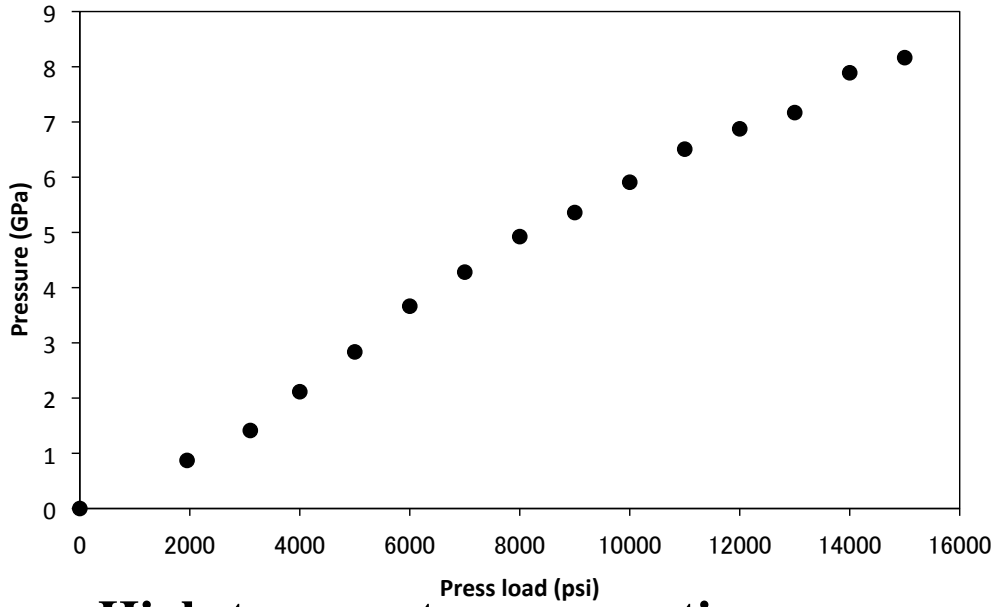
~0.5 mm height at ~7 GPa

High pressure and high temperature generation

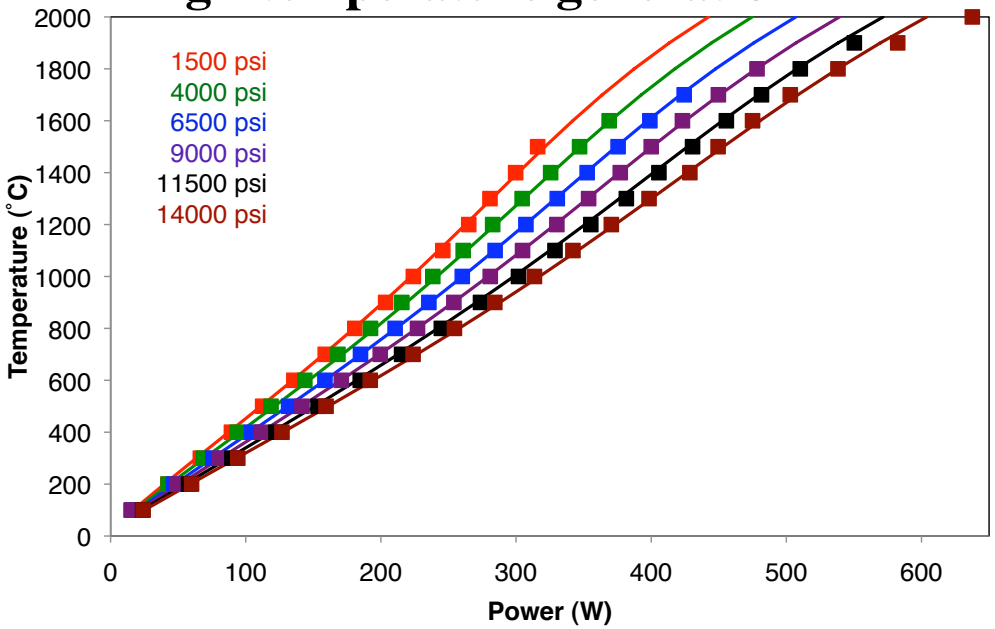


Graphite heater

High-pressure generation at room T



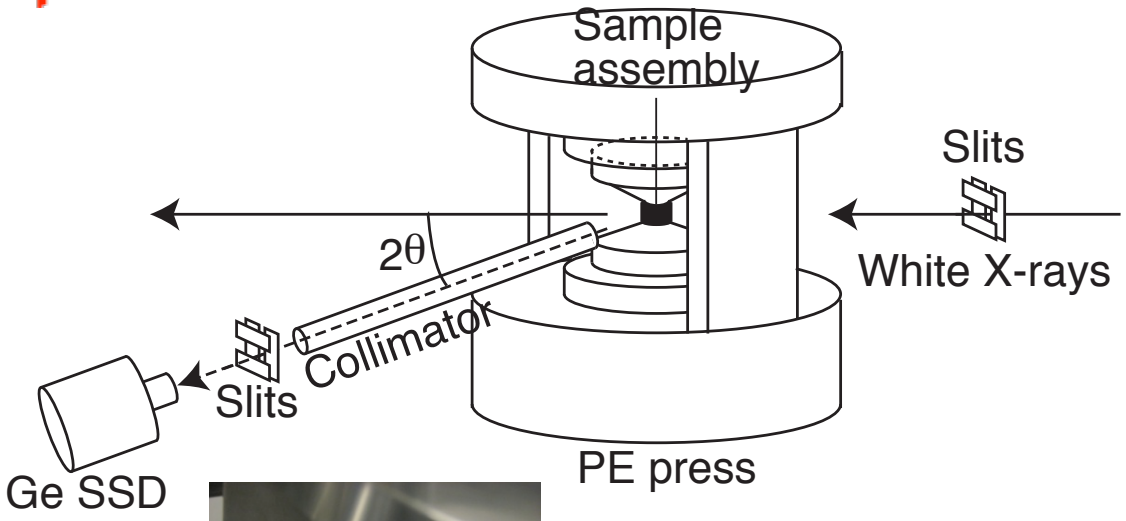
High-temperature generation



Contents

High-pressure amorphous structure measurement in Paris-Edinburgh cell (PEC)

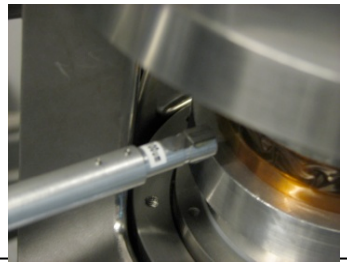
Amorphous structure measurement by multi-angle energy-dispersive x-ray diffraction

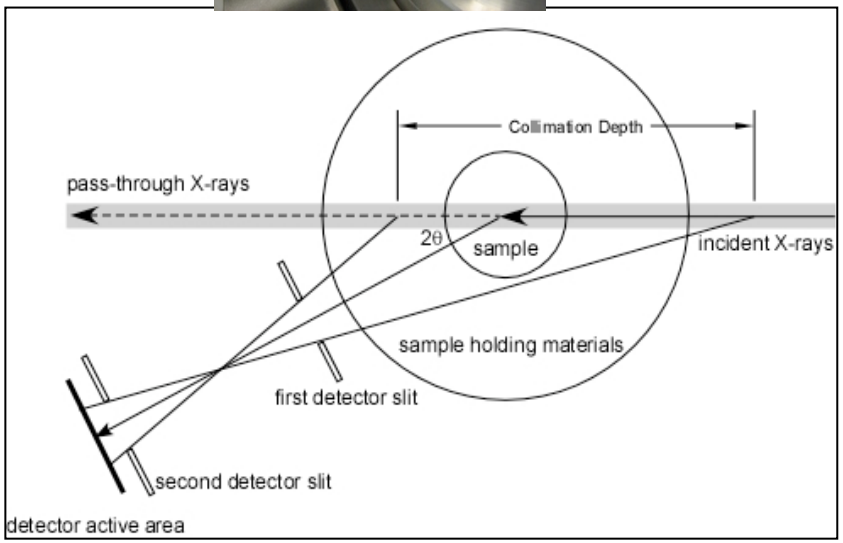


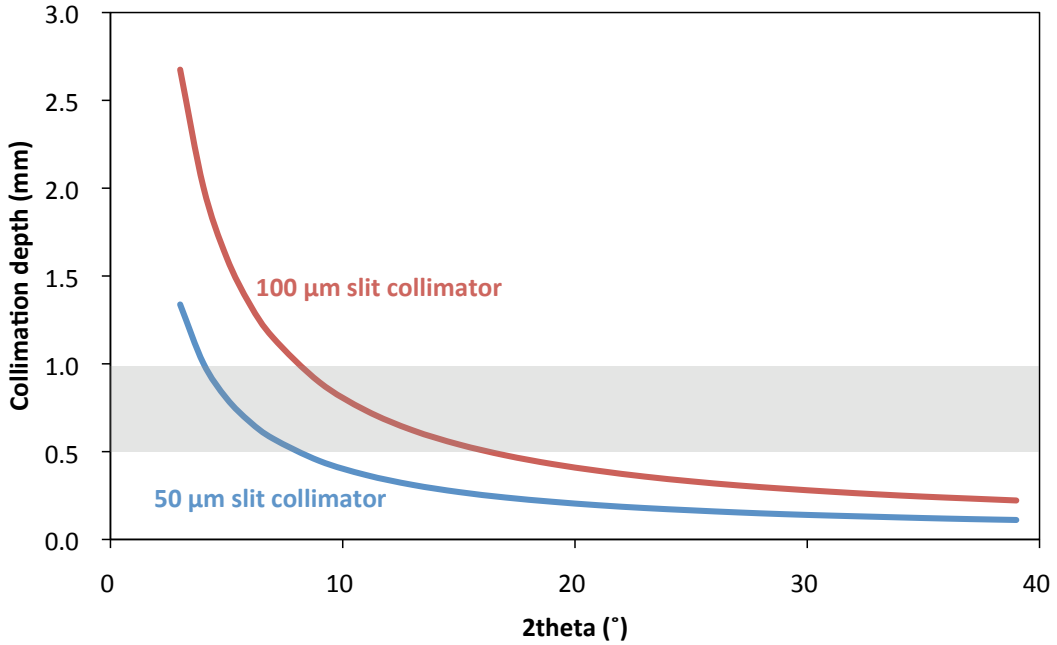
-BM white beam

-2θ up to ~40˚

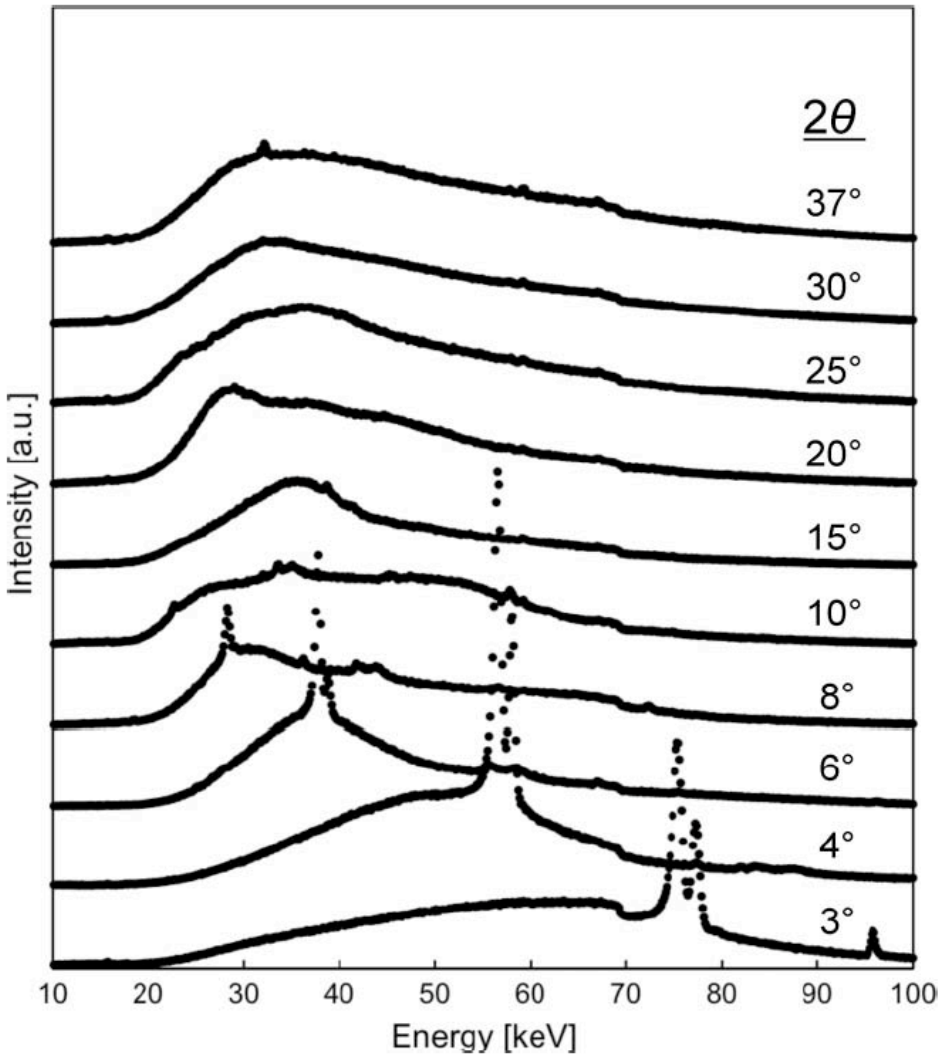
-Collimation depth control with collimator gaps (50, 100 μm)





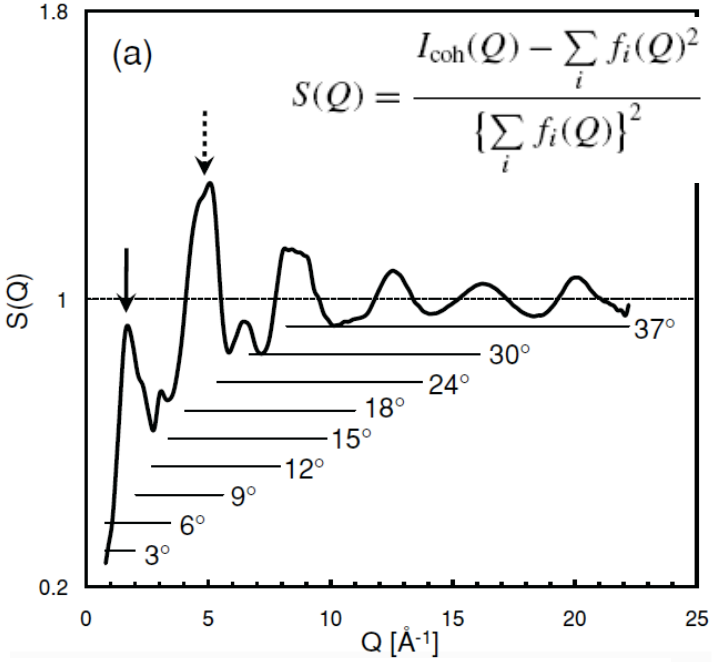


Structure measurement of molten albite ($NaAlSi_3O_8$) at 5.3 GPa and 1600 ˚C (Yamada et al., 2011, RSI)

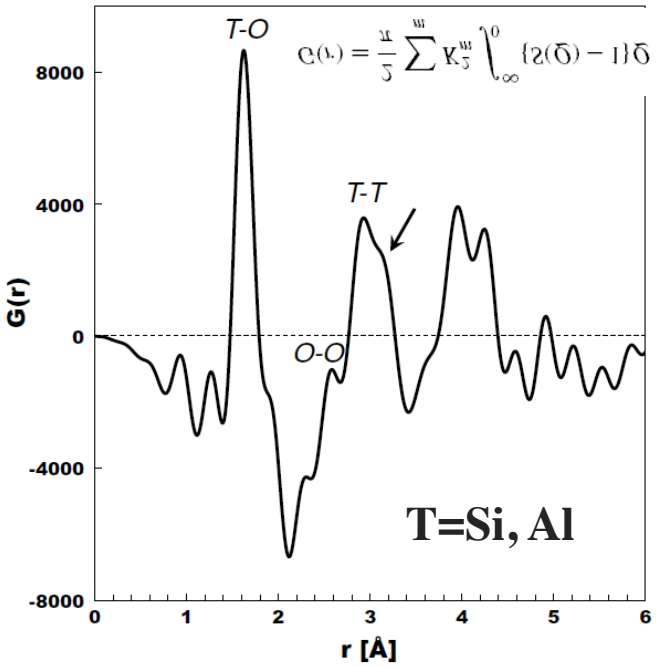


Structure factor

$$S(Q) = \frac{I_{coh}(Q) - \sum_i f_i(Q)^2}{\left\{\sum_i f_i(Q)\right\}^2} + 1$$



Reduced radial distribution function



Contents

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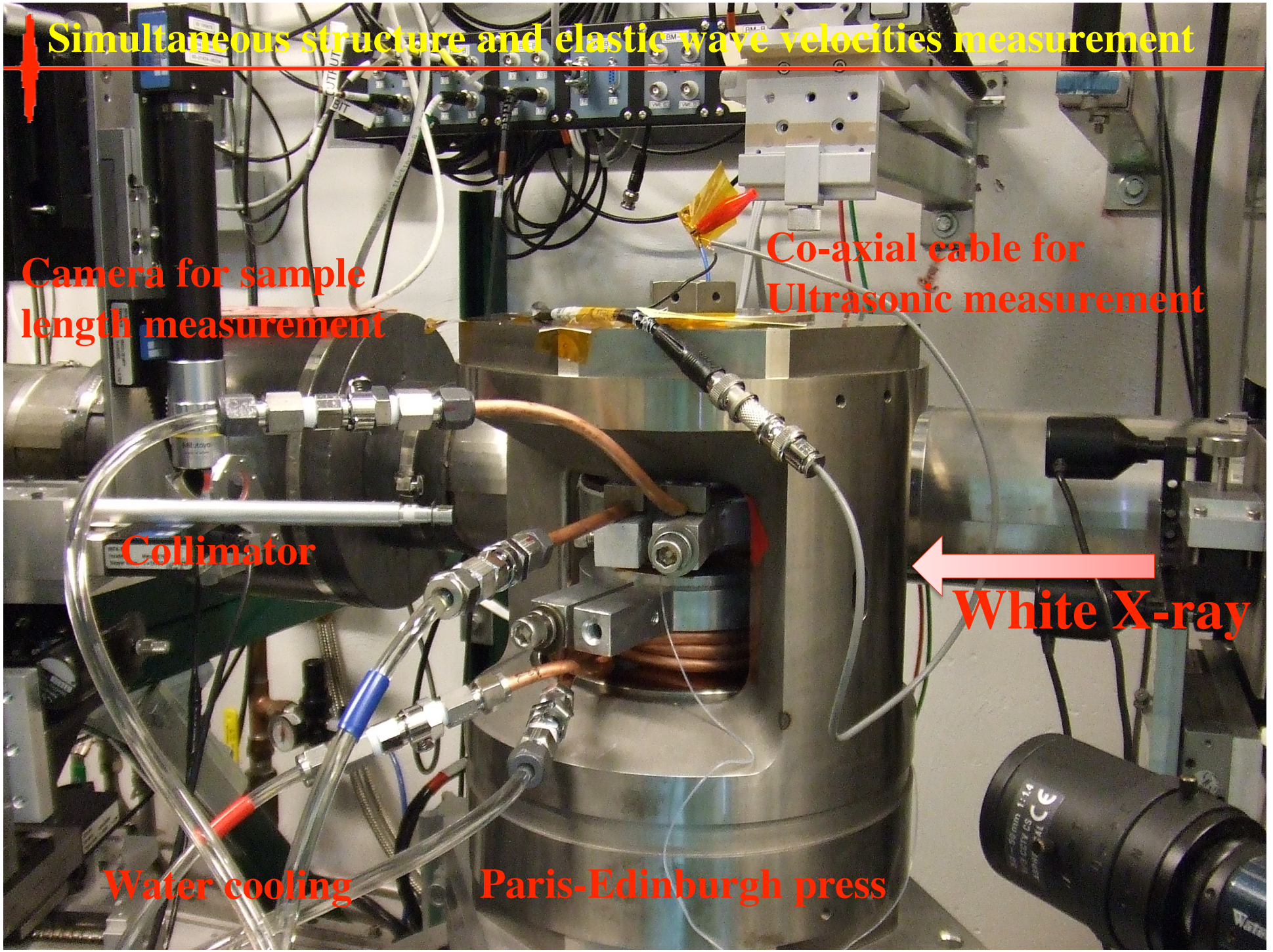
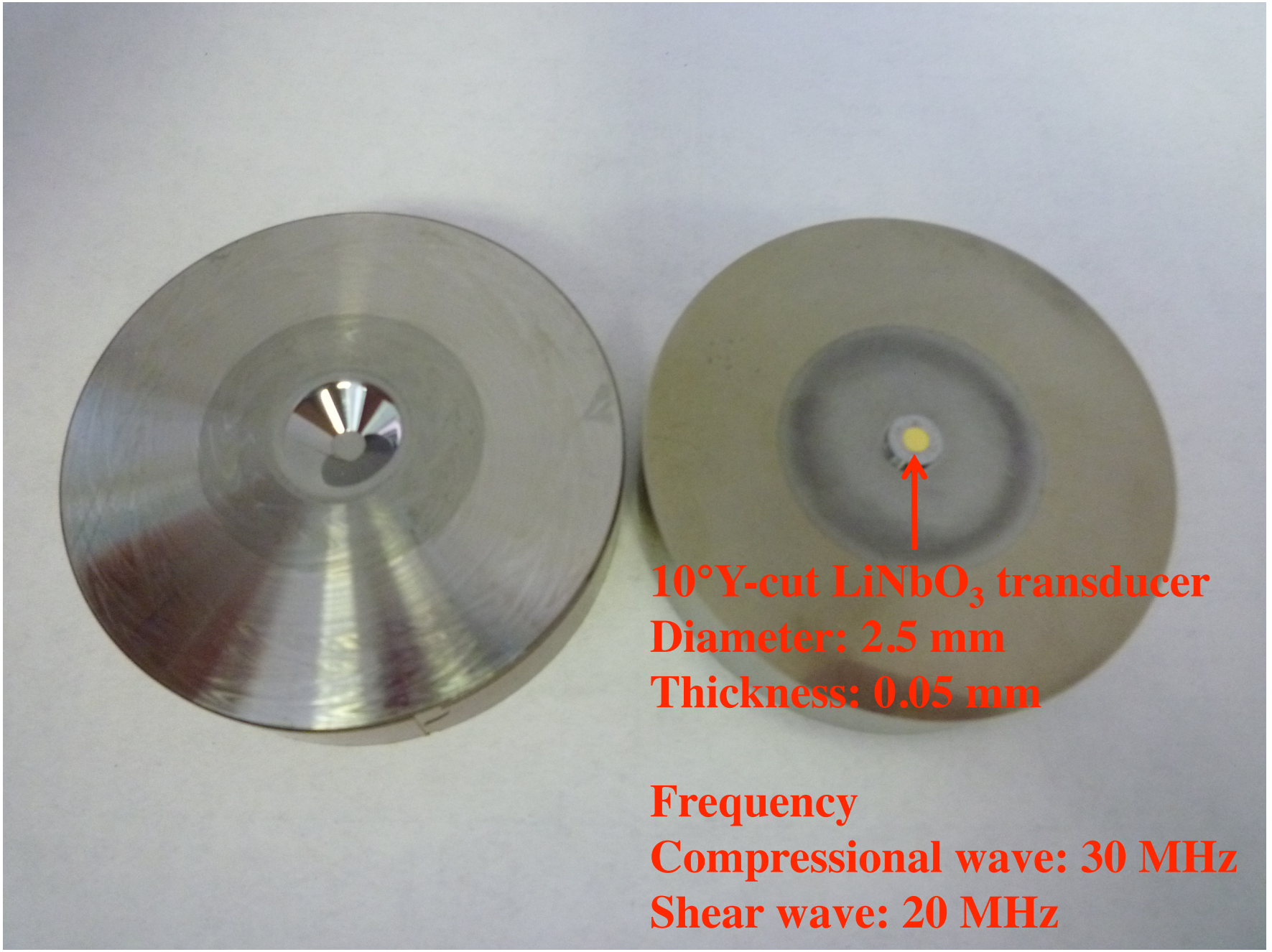
2. Viscosity measurement of silicate melt

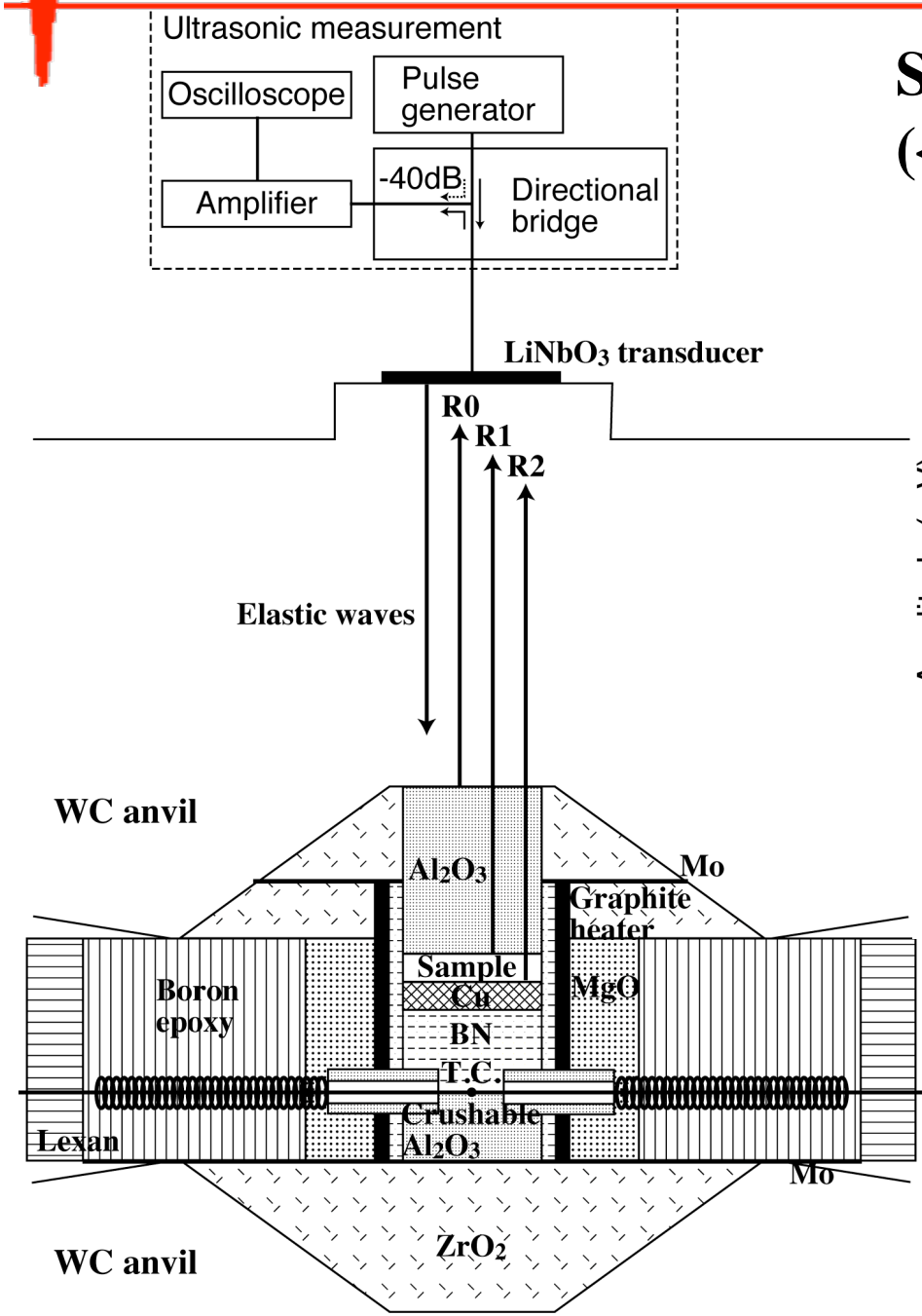
Simultaneous structure and elastic wave velocities measurement



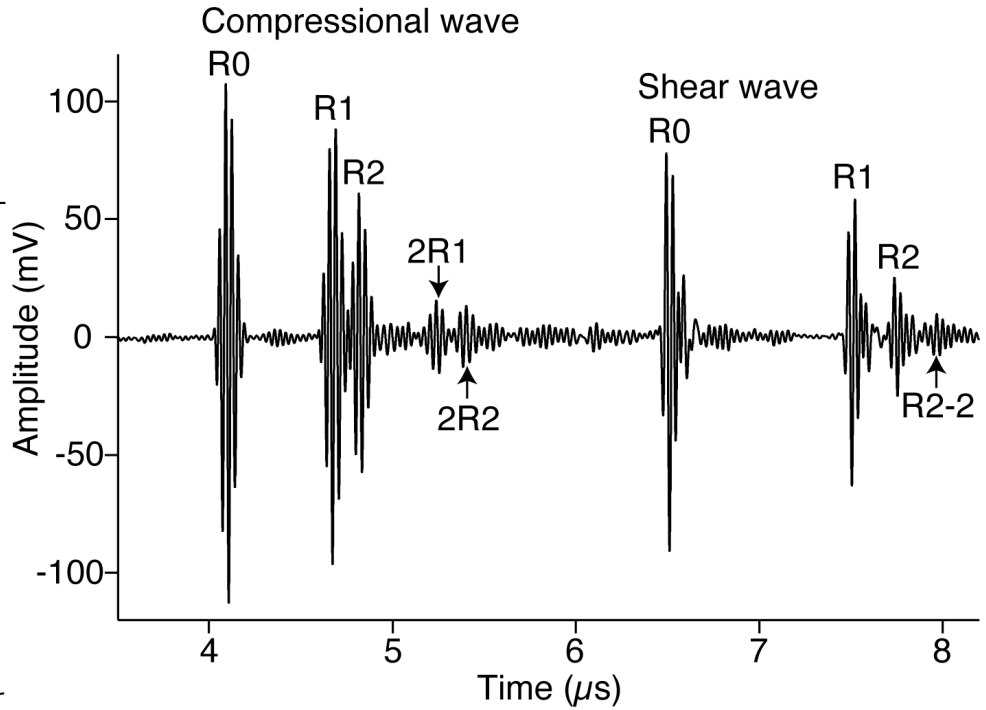
Ultrasonic transducer on PE anvil



Elastic wave velocity measurement

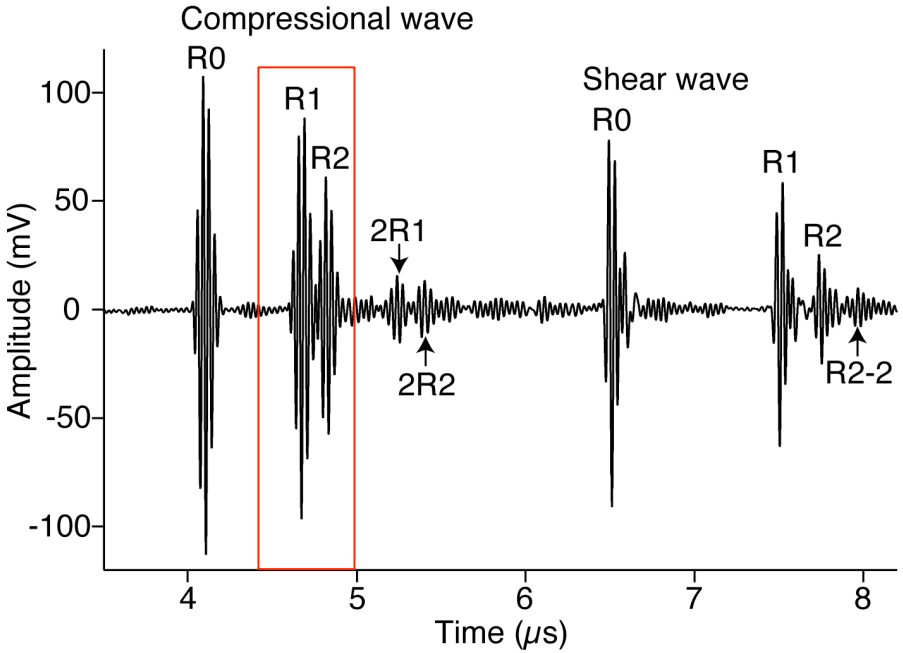


Sampling rate: 10 Gsample/second (<0.1 % of elastic wave travel time)

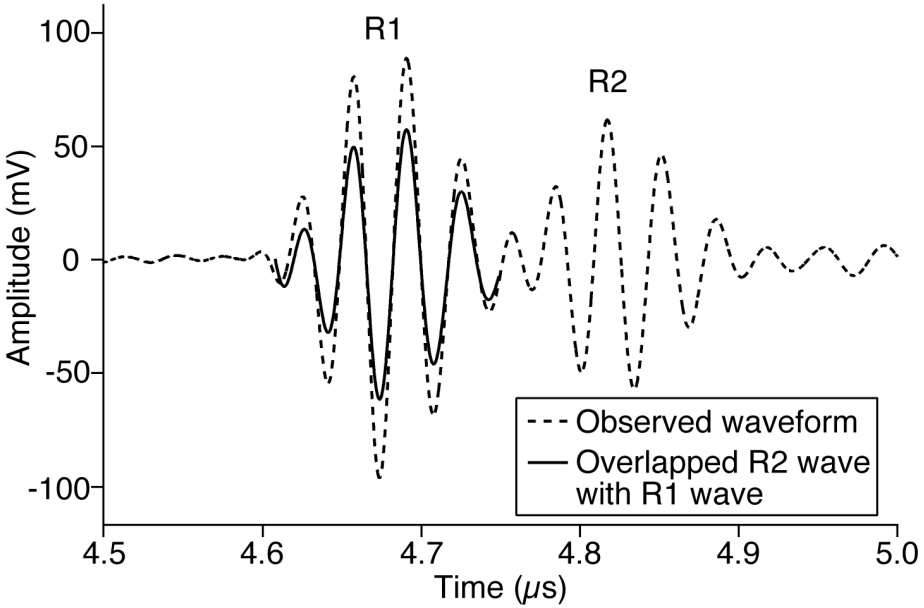


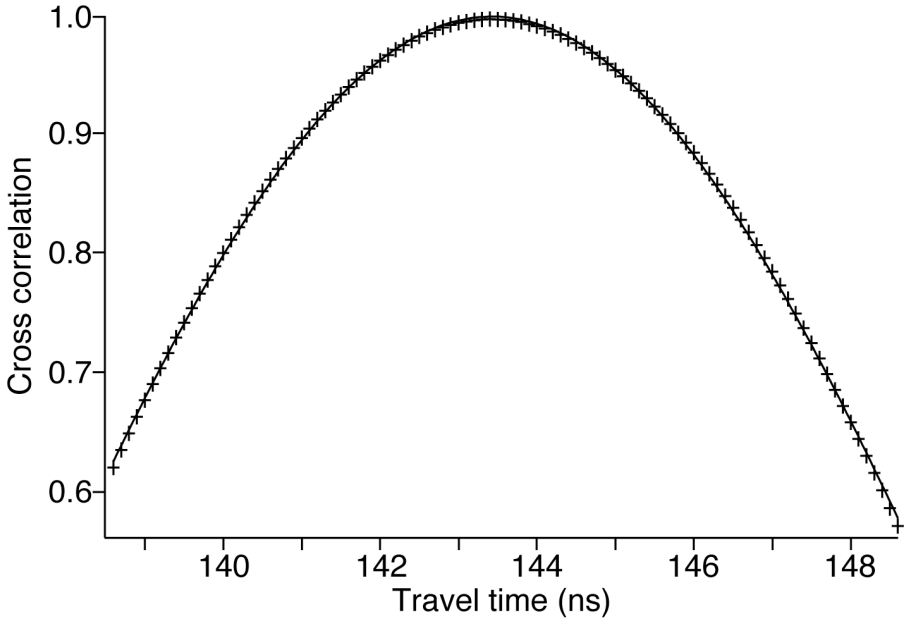
Kono et al. (under review)

Elastic wave travel time determination



$$2\Delta T = t(R2) - t(R1)$$





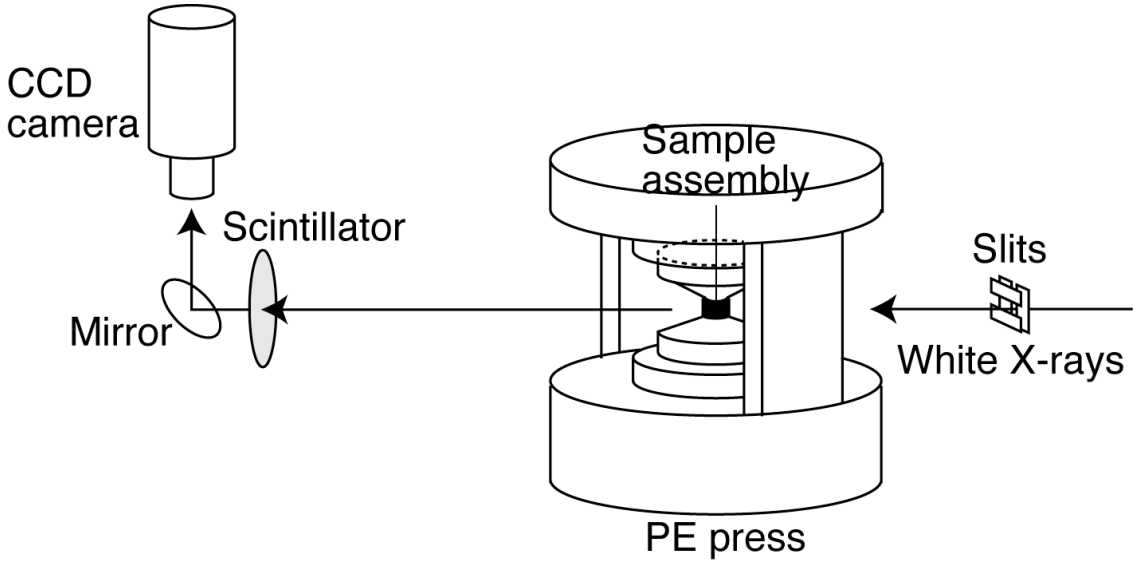
Sample length measurement by white x-ray radiography

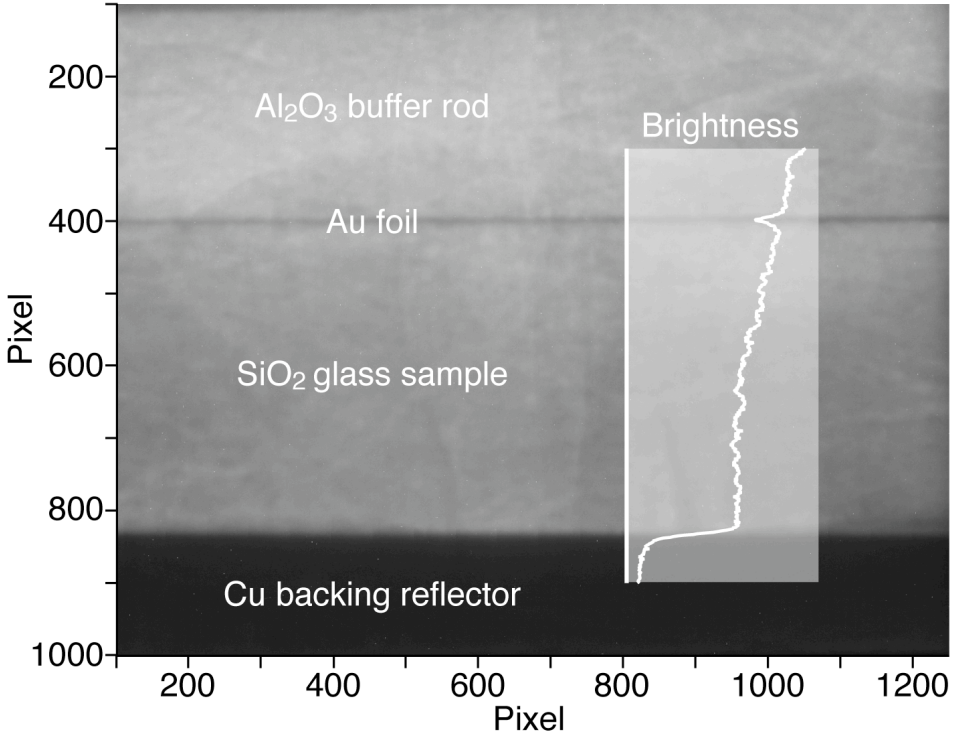
GGG:Tb thin film scintillator

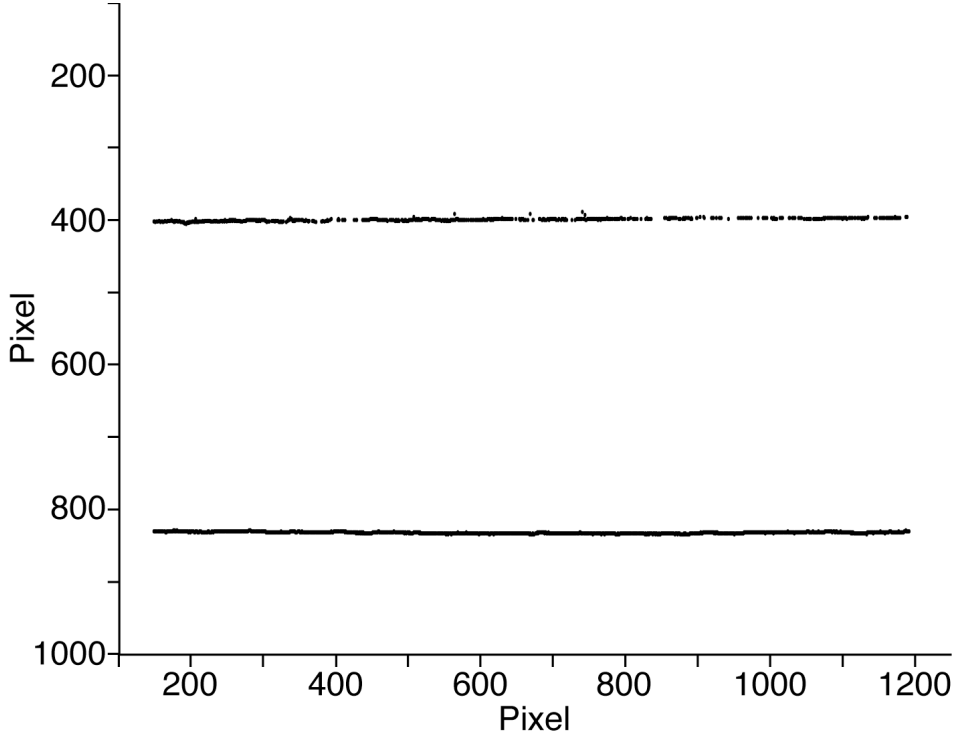
10x optical lens

CCD camera

Pixel resolution: 0.948 μm/pixel







Elastic wave velocity measurement

Ultrasonic measurement

Compressional and shear wave travel times: $2\Delta Tp$ and $2\Delta Ts$

X-ray radiography

Sample length: L

Compressional wave velocity $Vp = 2L/2\Delta Tp$

Shear wave velocity $Vs = 2L/2\Delta Ts$

Poisson's ration $= \frac{1}{2}\left[1 - \frac{1}{(Vp/Vs)^2 - 1}\right]$

Density: ρ

Bulk modulus $= \rho Vp^2 - (4/3)\rho Vs^2$

Shear modulus $= \rho Vs^2$

Young's modulus $= \rho Vp^2(3Vp^2 - 4Vs^2)(Vp^2 - Vs^2)$

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Falling sphere viscometer based on Stokes' law

Stokes' law of friction on sphere in flow

Radius of sphere

Frictional force $F_R = 6\pi\eta r v$ ← Velocity of falling sphere

Viscosity

Velocity of falling sphere follows from the balance between the frictional force and the weight force reduced by the buoyant force

Weight force — Buoyant force

Frictional force $6\pi\eta r v = \frac{4}{3}\pi r^3 \rho_s g - \frac{4}{3}\pi r^3 \rho_l g$

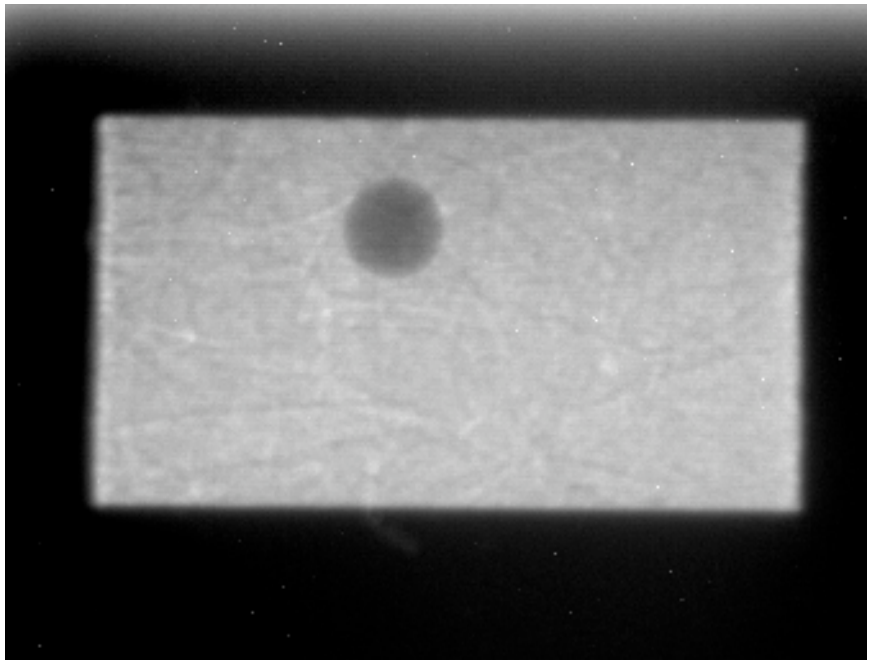
Viscosity $\eta = \frac{2gr^2(\rho_s - \rho_l)}{9v}$ (× effect of diameter and height of sample container)

Velocity of falling sphere

A preliminary test (falling Pt sphere in a silicate melt)

Pressure: ~7 GPa
Temperature: ~1700 ˚C

Density of melt: ~3 g/cm^3
Density of Pt sphere: ~21.5 g/cm^3
Diameter of Pt sphere: 140 μm



Estimation of sphere-falling time

η (Pa s)	v (μm/s)	500 μm falling time (s)
10^{-1}	2550.6	0.3
10^{0}	255.0	3.4
10^{1}	25.5	33.9
10^{2}	2.6	339.0

Summary

Paris-Edinburgh cell experiment at the beamline 16BM-B is currently capable of investigating

-Amorphous structure

-Elastic wave velocity
-Melt viscosity

at high pressures to ~7 GPa
and high temperatures to ~2000 ˚C