Exploring magmas under pressure using the Paris-Edinburgh press and synchrotron light

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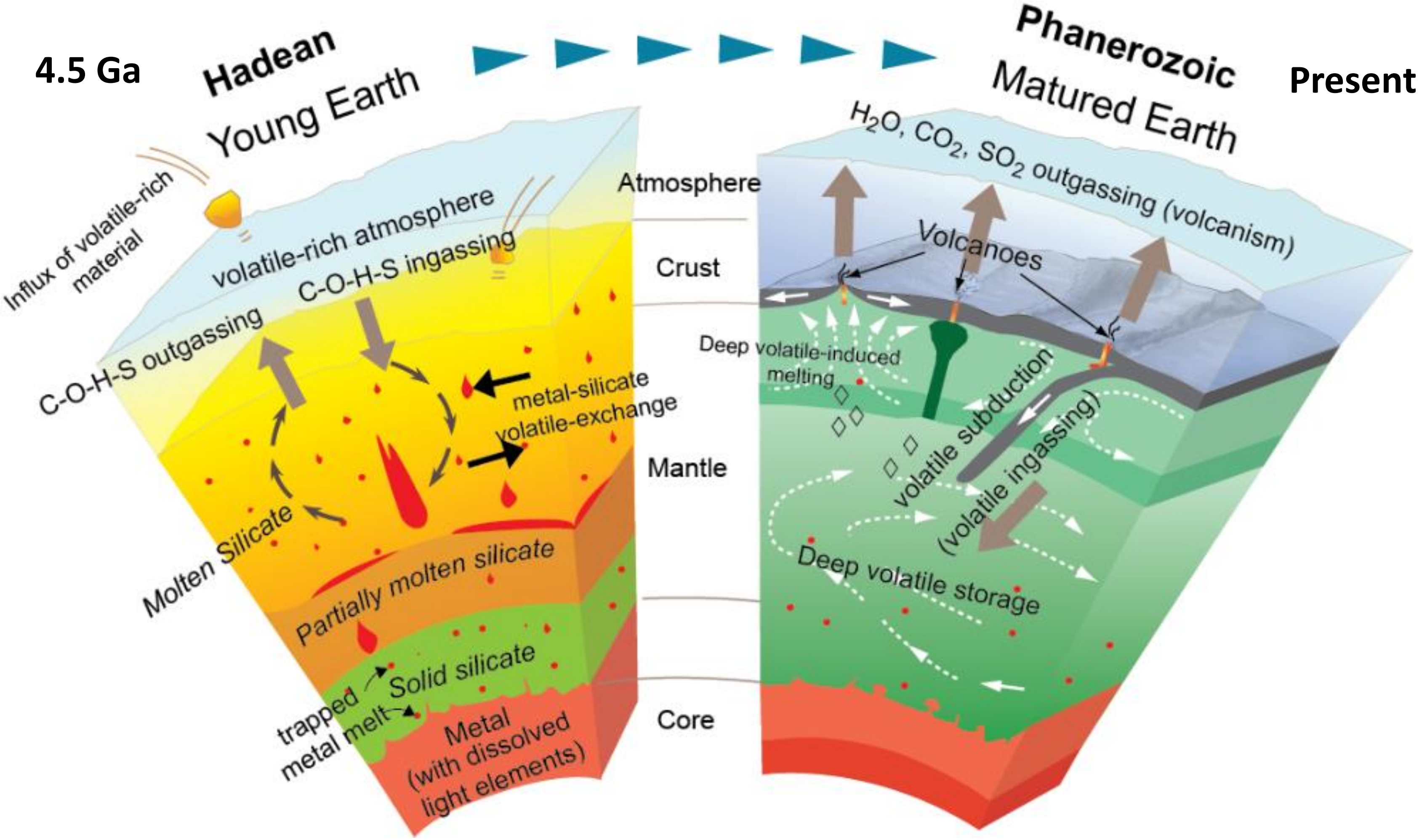
Terre Planètes Environnement

Université Claude Bernard Lyon 1

The key role of magmas

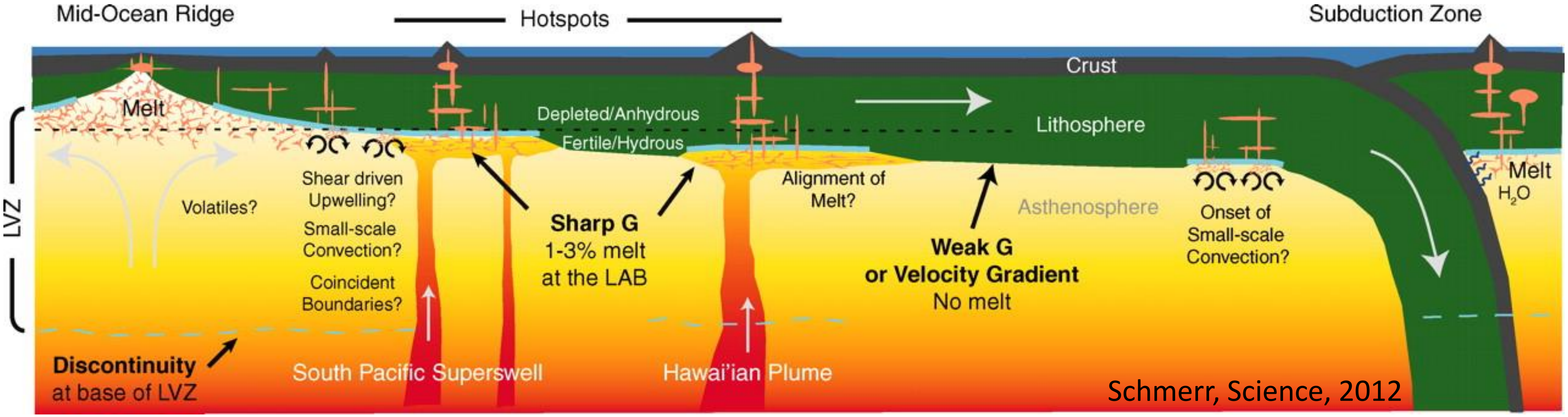


Magmas control the mass and energy (heat) transfers in planetary interiors

The key role of magmas

Diversity of volcanic rocks and eruption styles

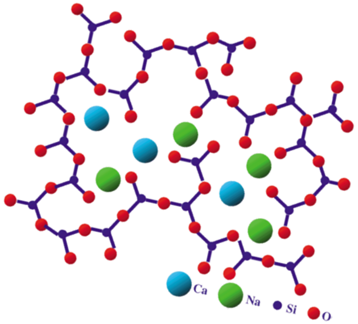
Chemistry
Silicate 40-60 wt% SiO_2
+/- carbonate +/- water



Schmerr, Science, 2012

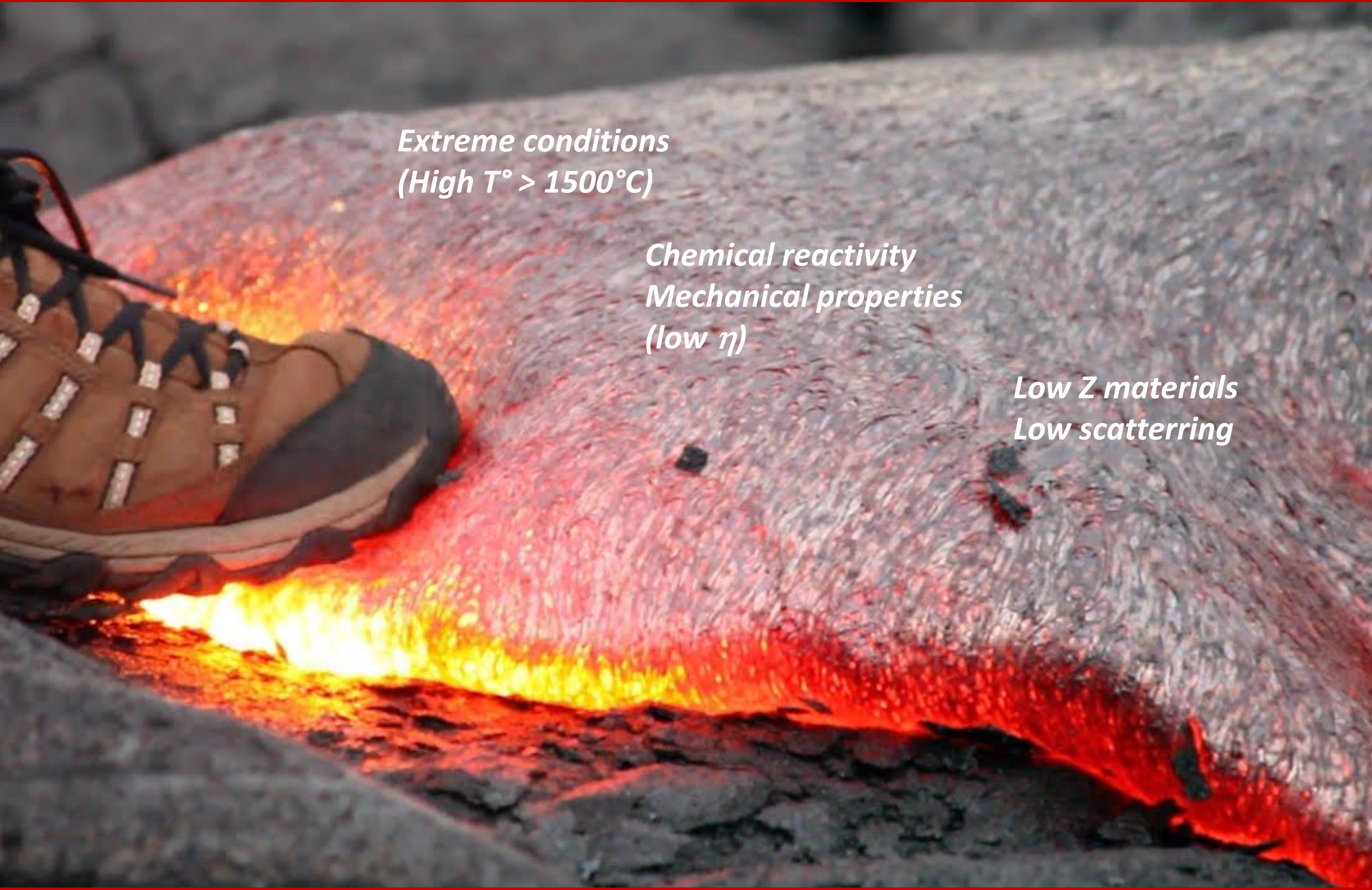
Liquid structure

Network (polymer) of SiO_4 tetrahedra



$$\textit{Dynamics of melt migration \& volcanic eruptions} \sim \frac{\textit{Buoyancy}\ \ \rho_{sol} - \rho_{liq}}{\textit{Viscosity}\ \ \eta_{liq}}$$

Introduction Challenges in the study of magmas under pressure

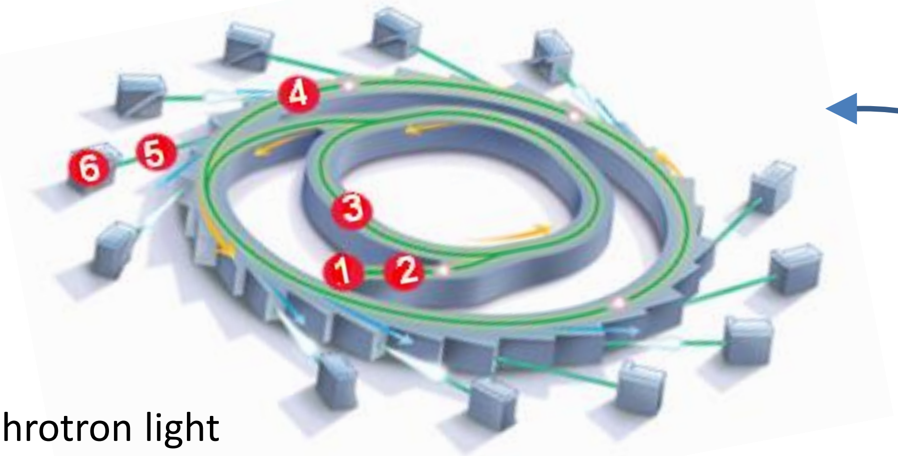


Extreme conditions
(High T° > 1500°C)

Chemical reactivity
Mechanical properties
(low η)

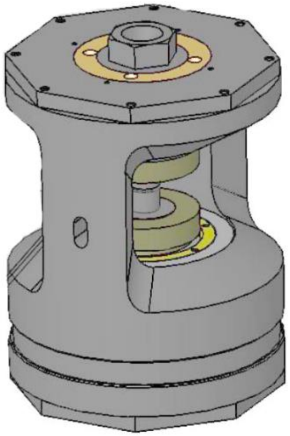
Low Z materials
Low scatterring

Recipes for a good experiment !



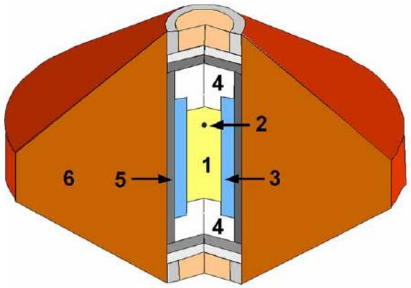
Synchrotron light

X-ray brilliance - high energy
Micro-beams
Panel of available techniques:
Absorption / Diffraction



High PT device
Paris-Edinburgh press
+ resistive heating

Low weight > transportable
Large X-ray opening



Cell assembly

Sample capsule
Confinement
X-ray transparent

Metrology P-T

In situ calibration / thermocouple

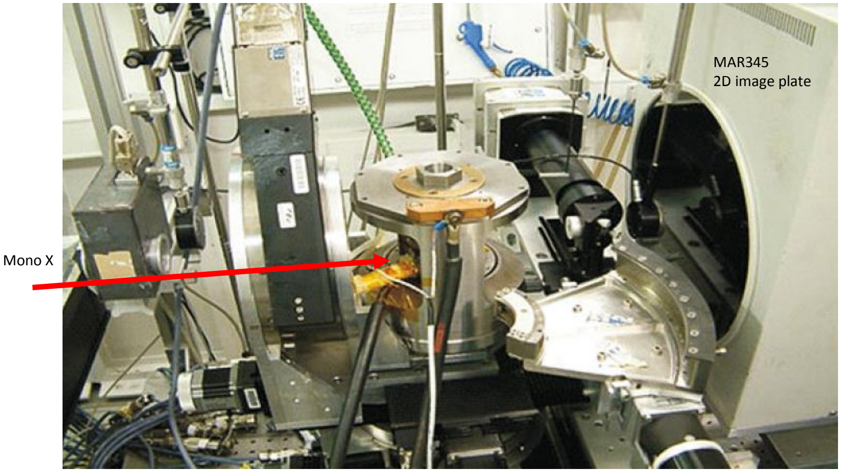
A (brief) summary of available techniques

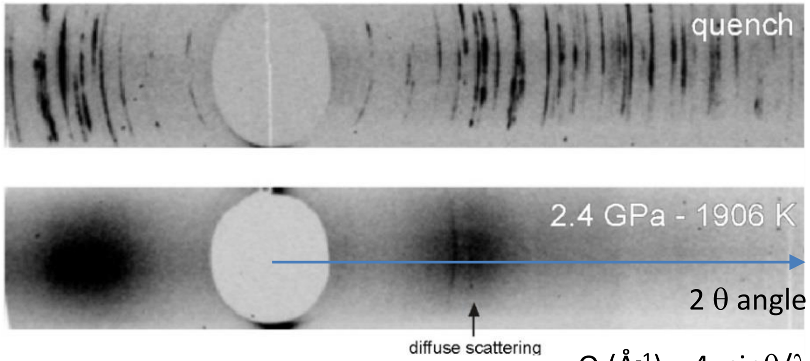
How to measure magmas density? elasticity? viscosity?

1. Density of magmas from X-Ray Diffraction

Cf Y. Kono's lecture

Angle dispersive X-ray diffraction





$Q\ (\text{Å}^{-1}) = 4\pi\sin\theta/\lambda$

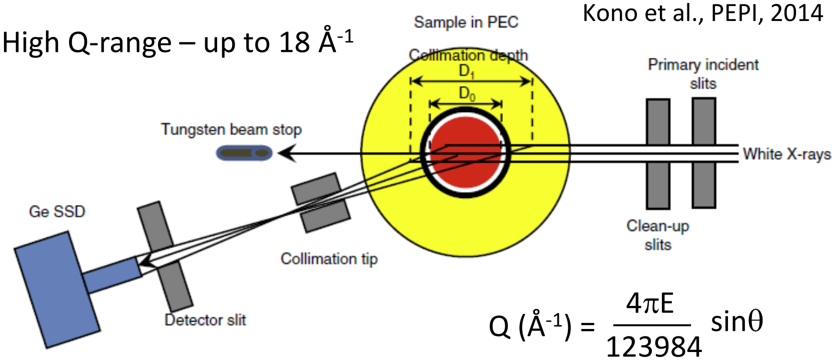
$$I^{\mathrm{obs}}(Q) = aI^{\mathrm{sample}}(Q) + b(Q)I^{\mathrm{cellassembly}}(Q)$$

$$I^{\mathrm{sample}}(Q) = \frac{1}{a\alpha}[\boxed{I_{\mathrm{coh}}(Q)} + I_{\mathrm{incoh}}(Q)]$$

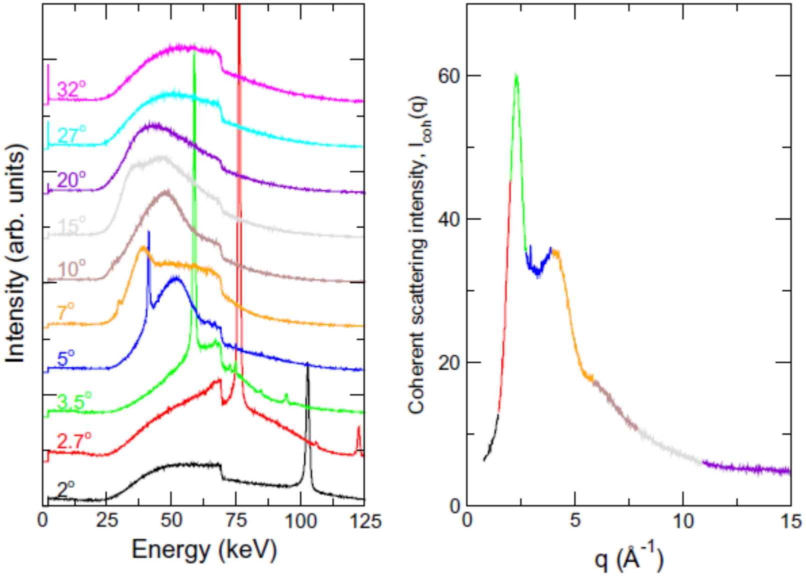
Energy dispersive X-ray diffraction

Kono et al., PEPI, 2014

High Q-range – up to 18 Å^{-1}



$$Q\ (\text{Å}^{-1}) = \frac{4\pi E}{123984}\sin\theta$$



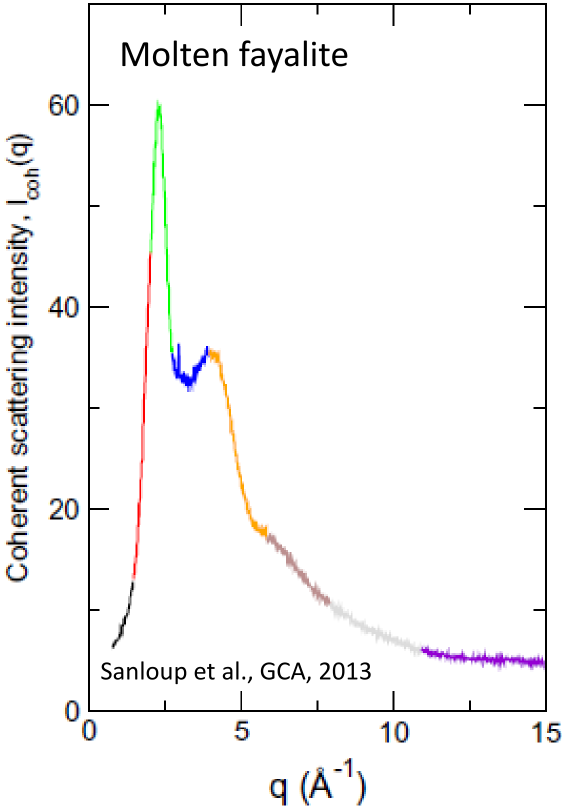
Background substraction/correction

1. Density of magmas from X-Ray Diffraction

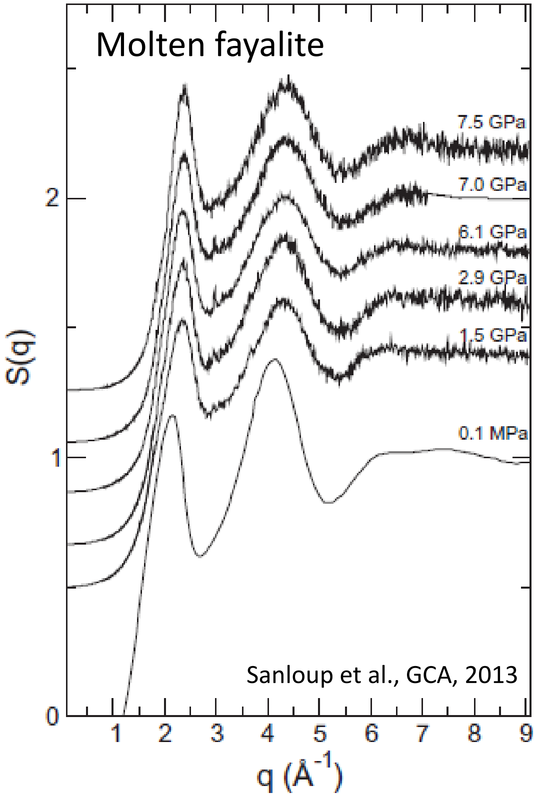
Coherent scattering intensity *Normalization* → Structure factor *Fourier Transform* → Pair distribution function

$$S(q) - 1 = \frac{K[I_s(q) - A(q)I_B(q)] - I_{inc}(q)}{\langle f^2(q) \rangle},$$

$$g(r) - 1 = \frac{1}{\boxed{2\pi^2 r n_0}} \int_0^\infty q[S(q) - 1]\sin(qr)\mathrm{d}q,$$



Sanloup et al., GCA, 2013



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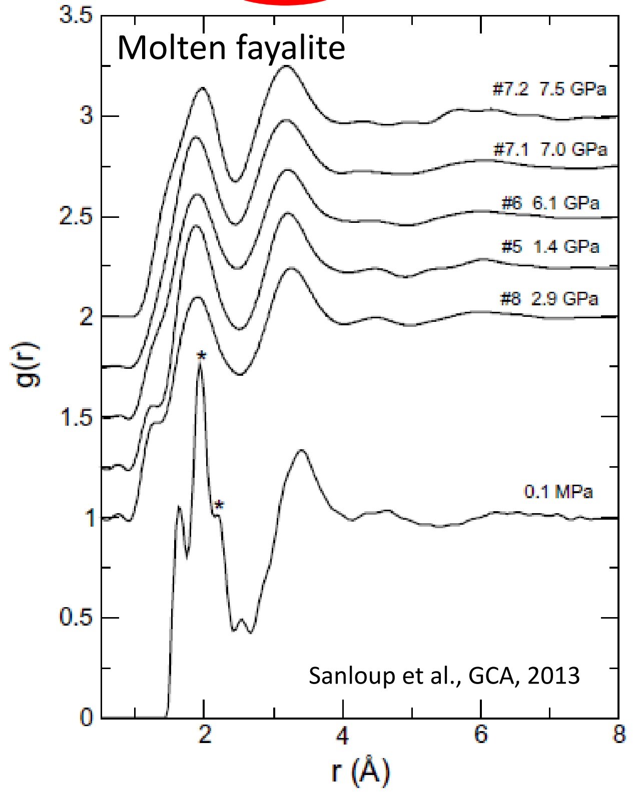

Sanloup et al., GCA, 2013

The atomic density n_0 (nb of atoms per Å^3) is obtained by minimizing the signal in g(r) at distances lower than the interatomic distances, for $0 < r < r_{min}$ (first coordination shell)

Eggert et al, PRB, 2002

1. Density of magmas from X-Ray Diffraction

Coherent scattering intensity *Normalization* → Structure factor *Fourier Transform* → Pair distribution function

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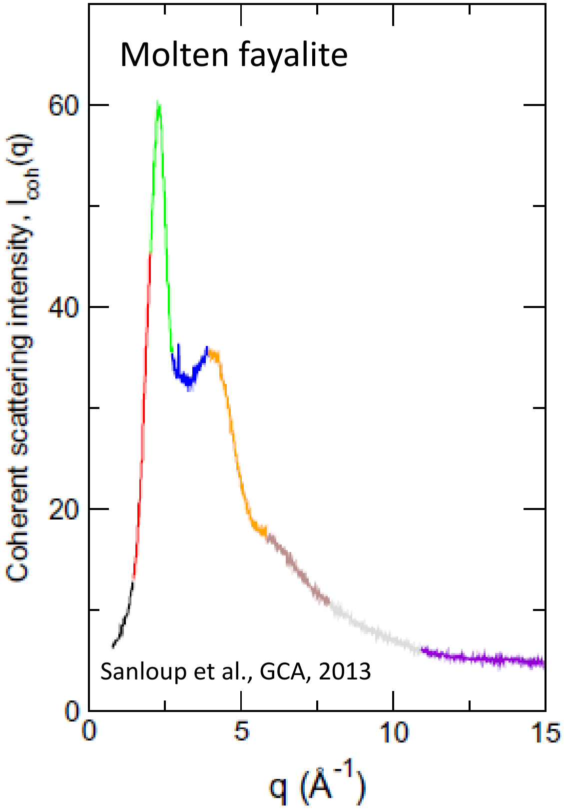
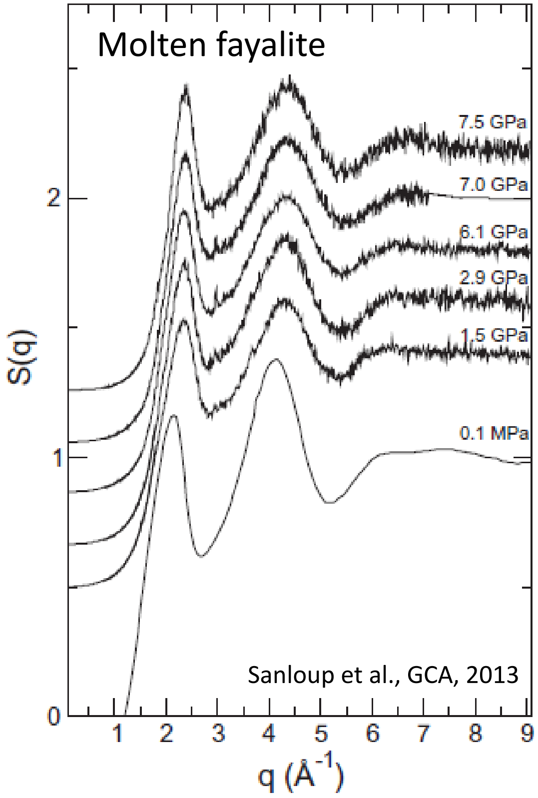


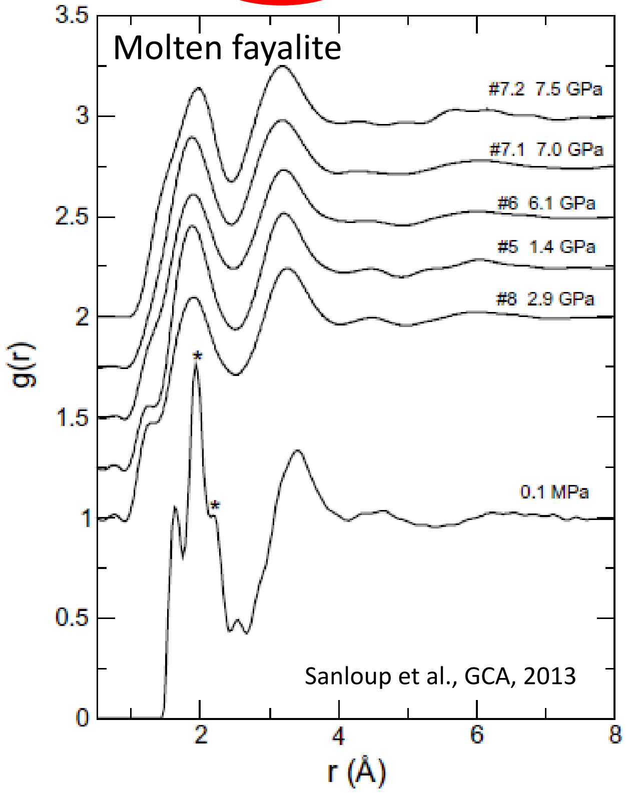
Molten fayalite

Sanloup et al., GCA, 2013



Molten fayalite

Sanloup et al., GCA, 2013



Molten fayalite

Sanloup et al., GCA, 2013

Density & structural information on the melt

High Q range – Good signal to noise ratio even for low Z materials

Complexities in the determination of density

2. Density of magmas from X-Ray Absorption

Principle: *Beer-Lambert's law*

$$I = I_0 \, . \, exp^{-\mu . \rho . x}$$

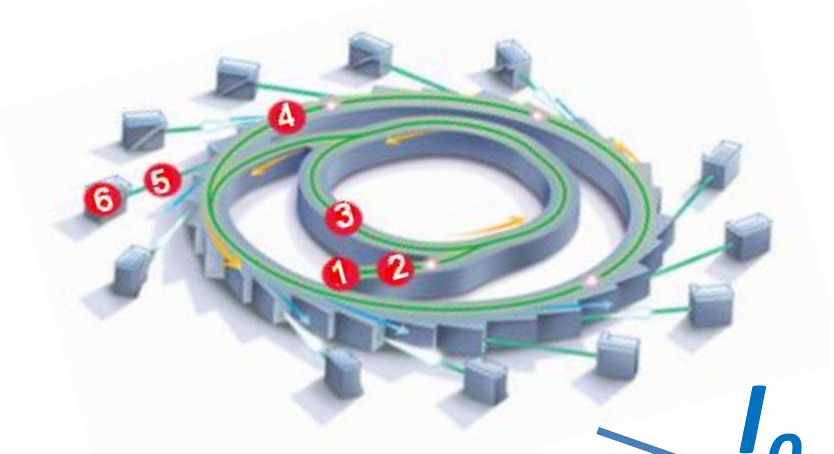
I = intensity of the X-ray beam

I_0 = int. of the incident X-ray beam

μ = mass absorption coefficient (chemistry dependent)

ρ = density of the liquid

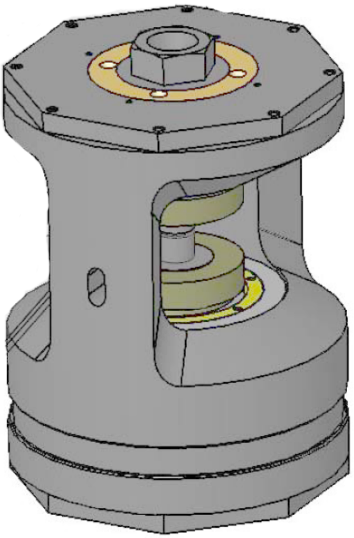
x = sample thickness



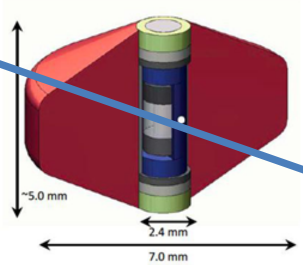
I_0

X-rays (33 keV)

Paris-Edinburg press



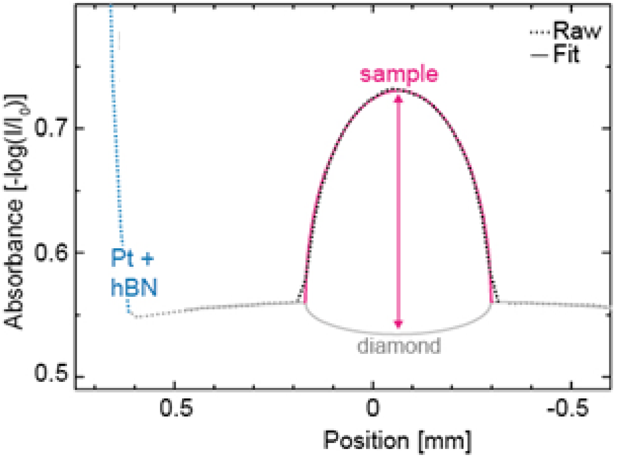
I



Sample assembly

Absorbance

$A = -\log_{10}(I/I_0) = \mu.\rho.x$



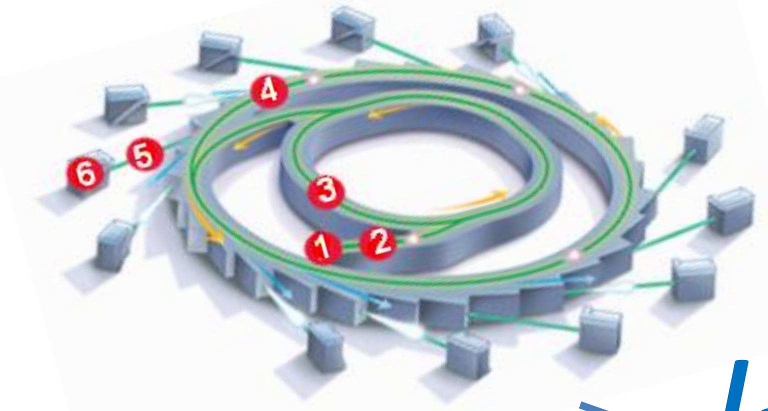
2. Density of magmas from X-Ray Absorption

Principle: *Beer-Lambert's law*

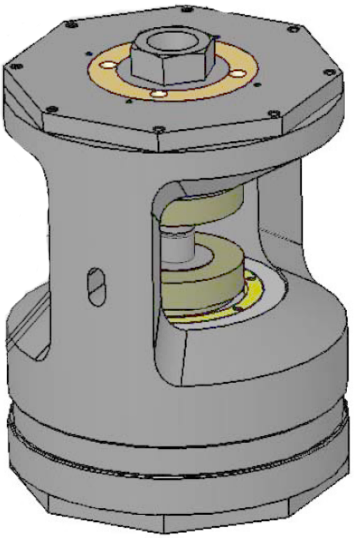
$$I = I_0 \,.\, exp^{-\mu.\rho.x}$$

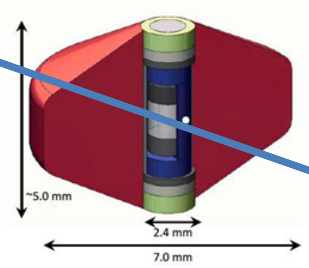
μ = mass absorption coefficient (chemistry dependent)

Additive function of composition, independent of P-T
> Determined at room conditions



Paris-Edinburg press





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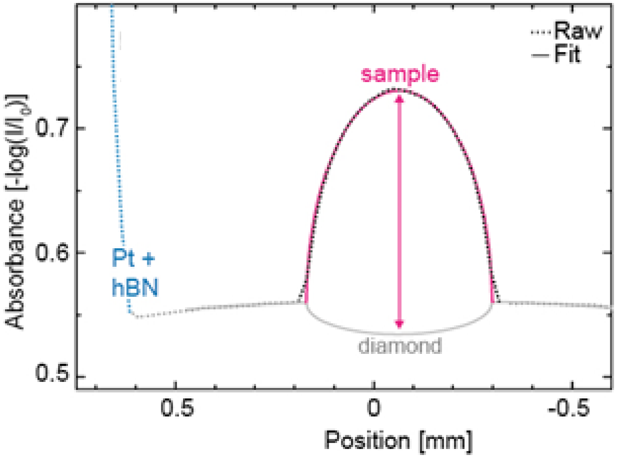

2. Density of magmas from X-Ray Absorption

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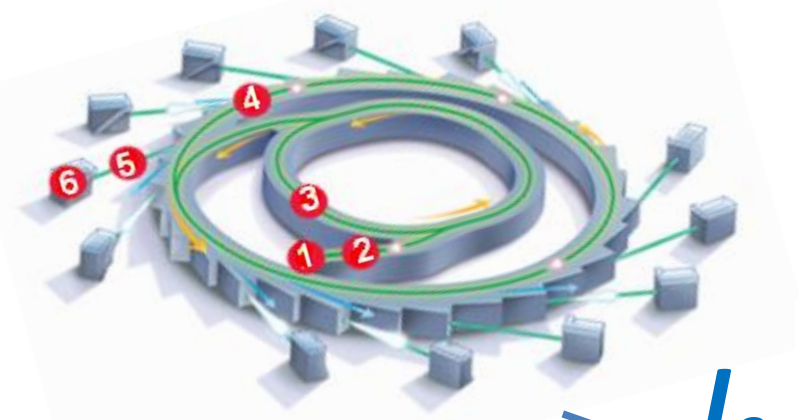
$$I = I_0 \, . \, \exp^{-\mu . \rho . x}$$

x = sample thickness

> diamond capsule



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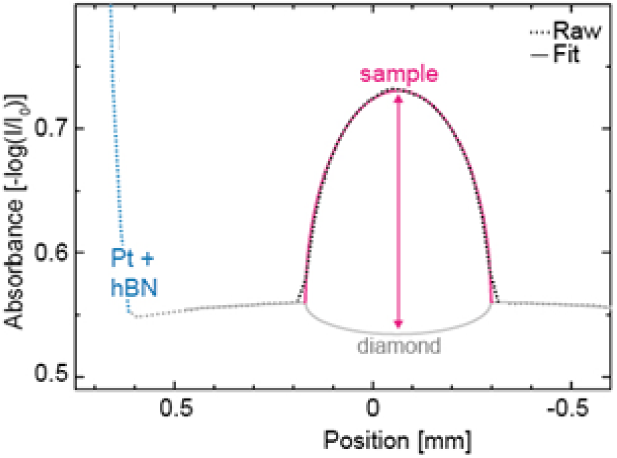
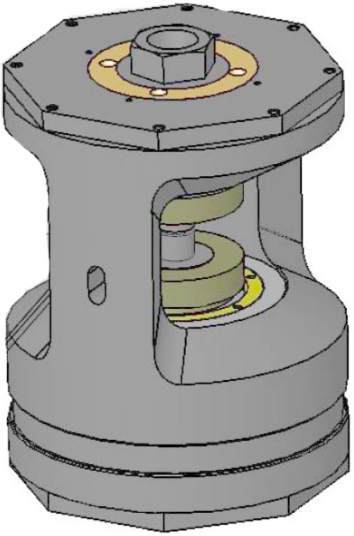
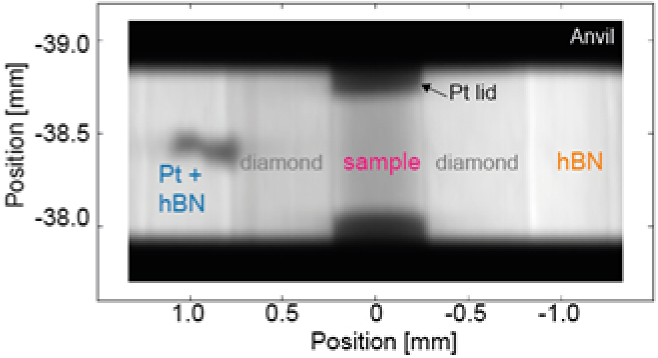


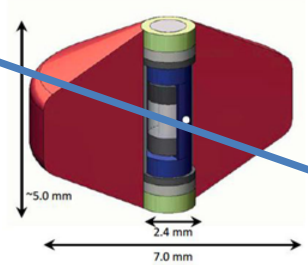
I_0

X-rays (33 keV)

Paris-Edinburg press







I

Sample assembly

2. Density of magmas from X-Ray Absorption

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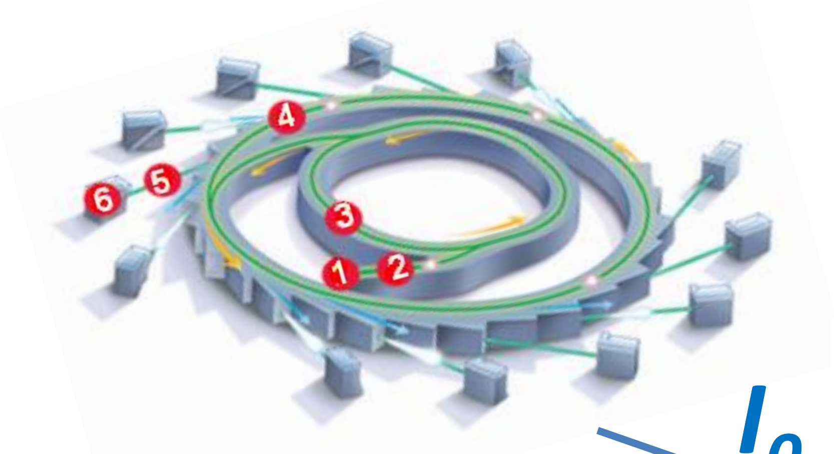
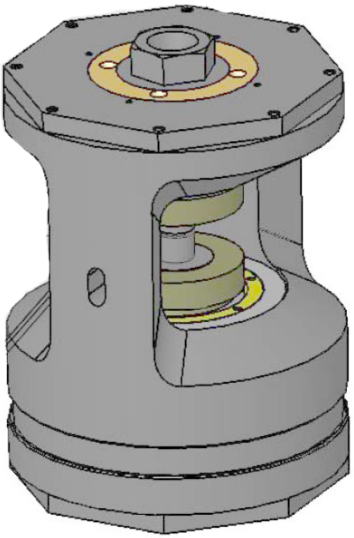


Variations in the absorption profiles with P-T reflect variations in density

Paris-Edinburg press



I_0

X-rays (33 keV)

I

Absorbance

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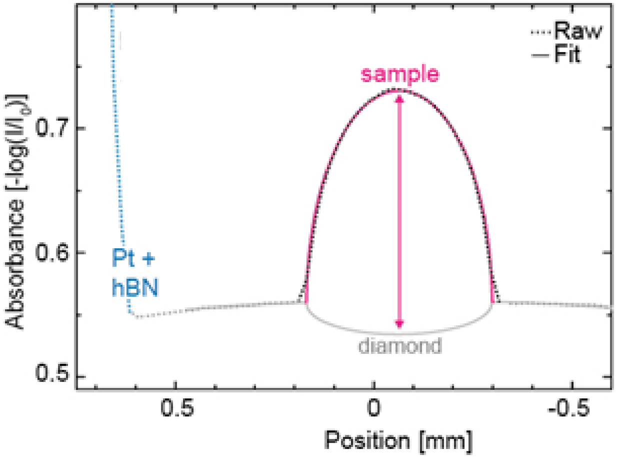
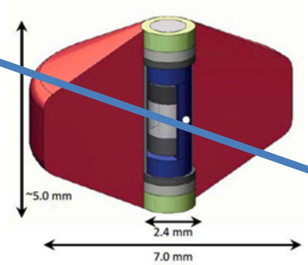
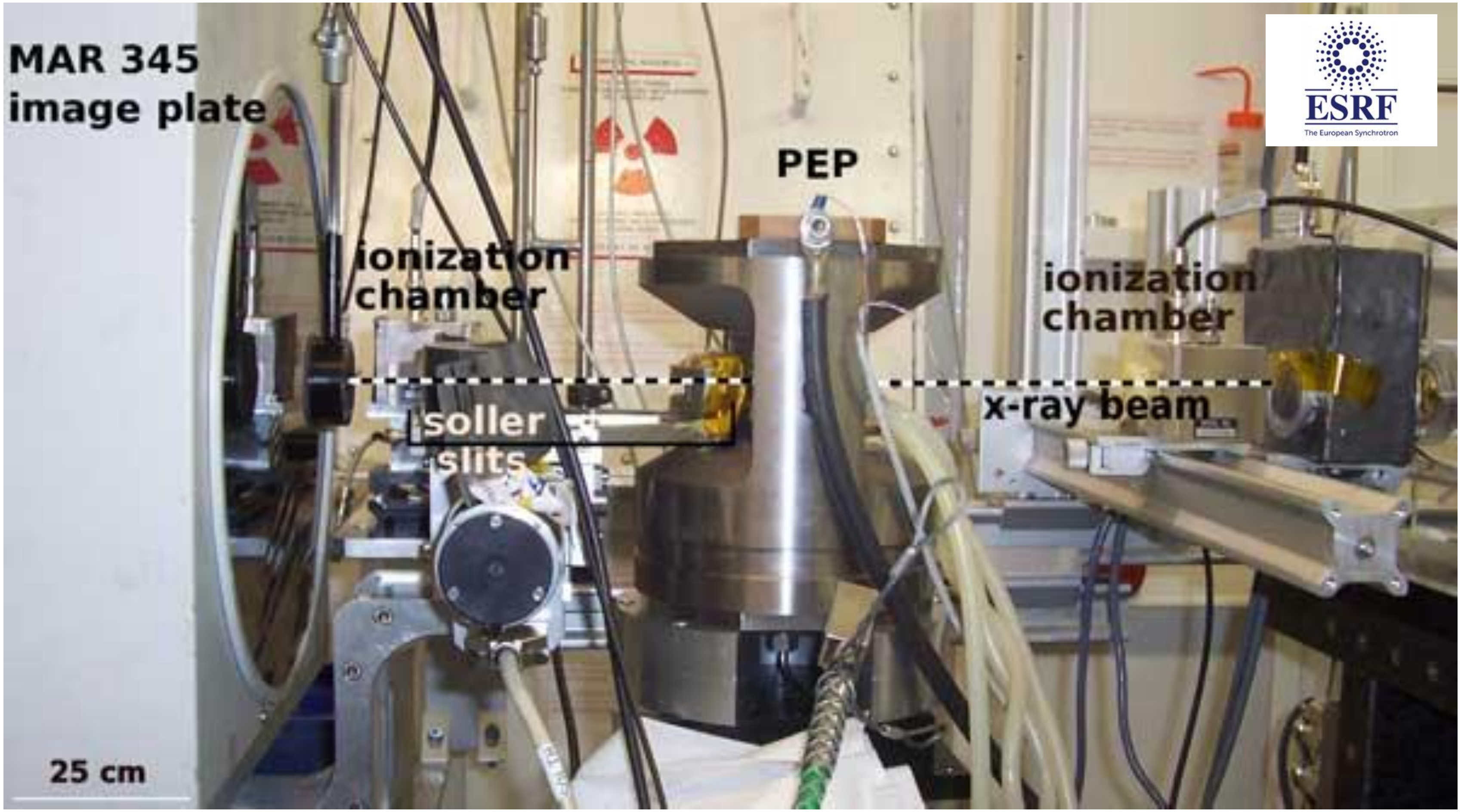




Sample assembly

2. Density of magmas from X-Ray Absorption



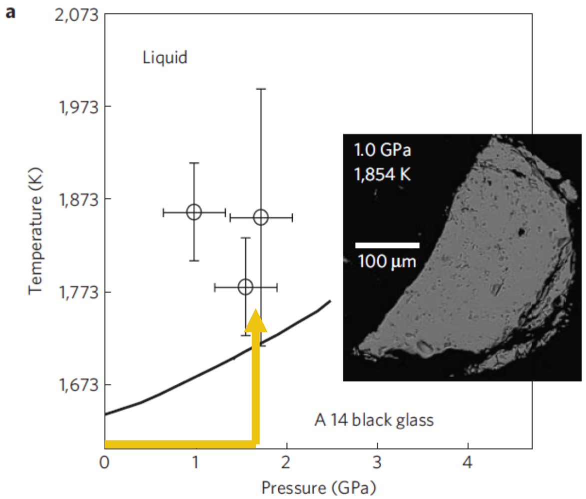
Ionization chambers for I/I_0 measurements

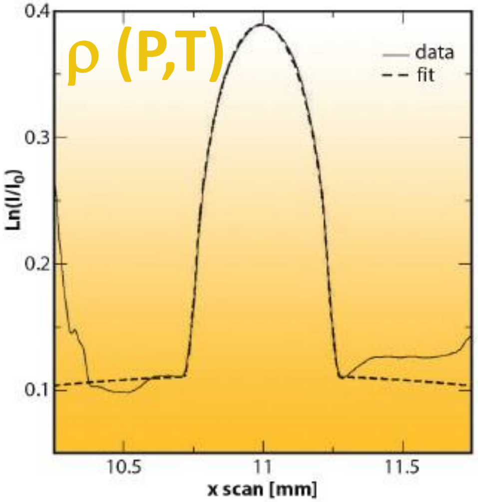
X-ray diffraction for P-T calibration and solid/liquid indentification

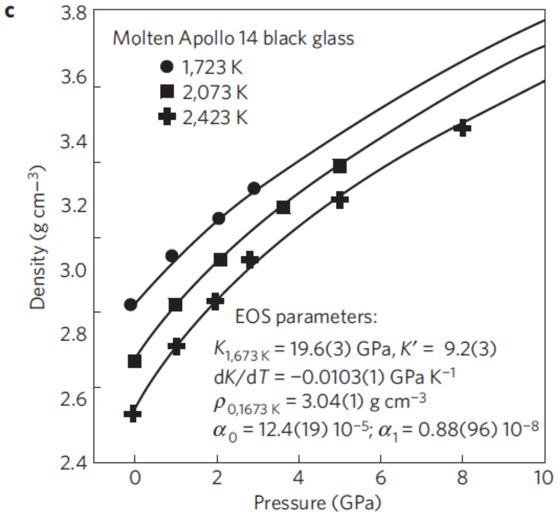
2. Density of magmas from X-Ray Absorption

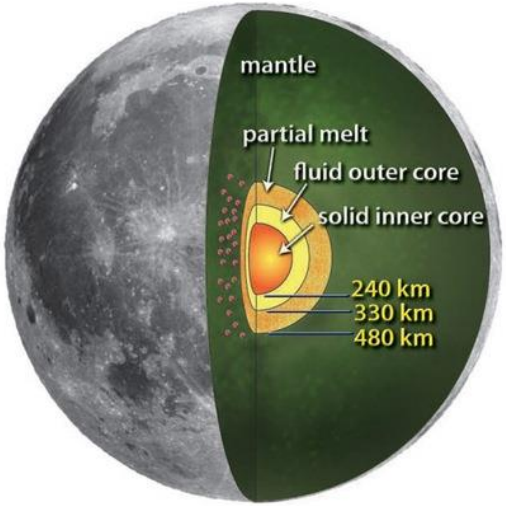
Application: density of lunar basaltic melts

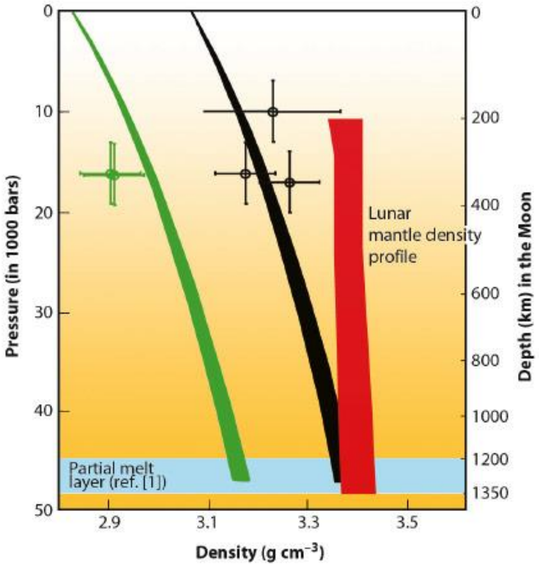
Van Kan Parker et al., Nature Geo., 2012









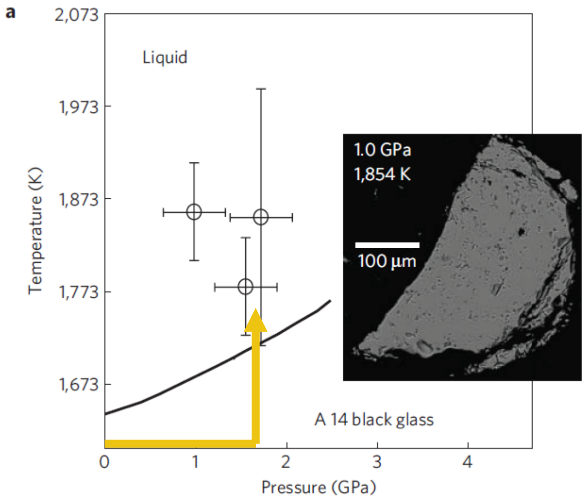


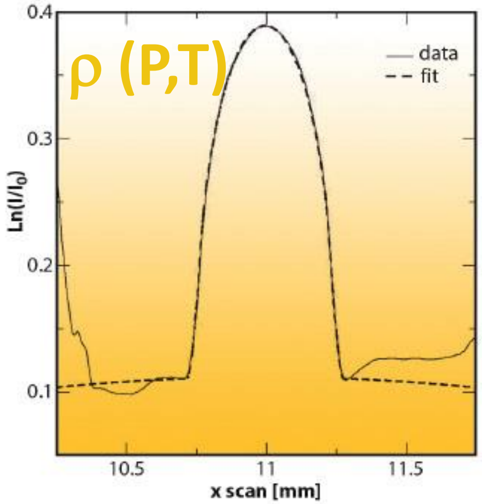
TiO_2 rich basaltic melt can be trapped today at the base of the lunar mantle and explain the partial-melt layer

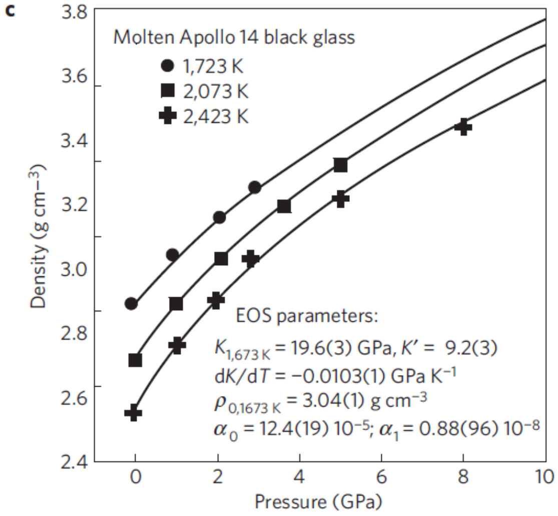
2. Density of magmas from X-Ray Absorption

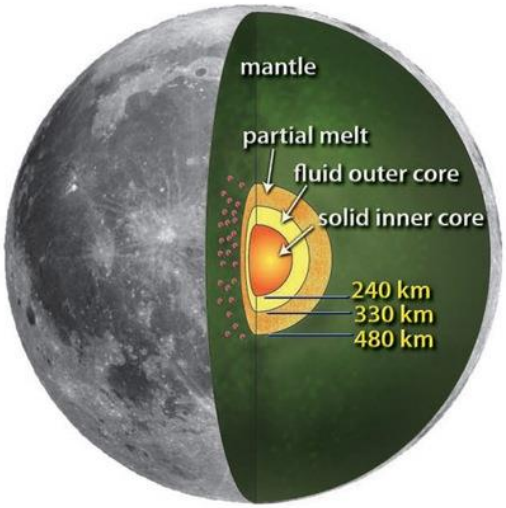
Application: density of lunar basaltic melts

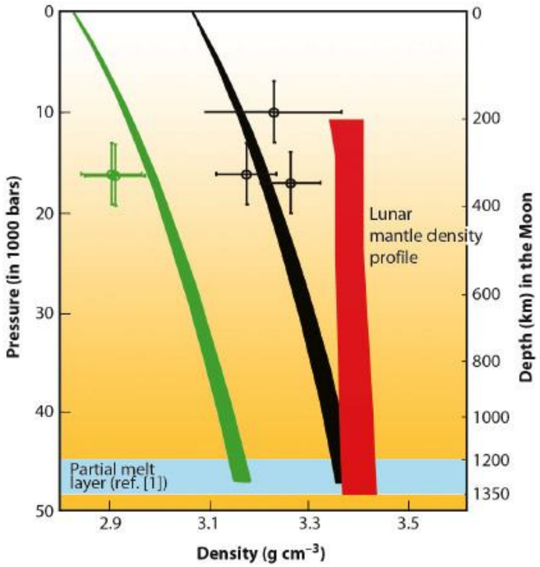
Van Kan Parker et al., Nature Geo., 2012











Robust & accurate technique = density error 1-2%

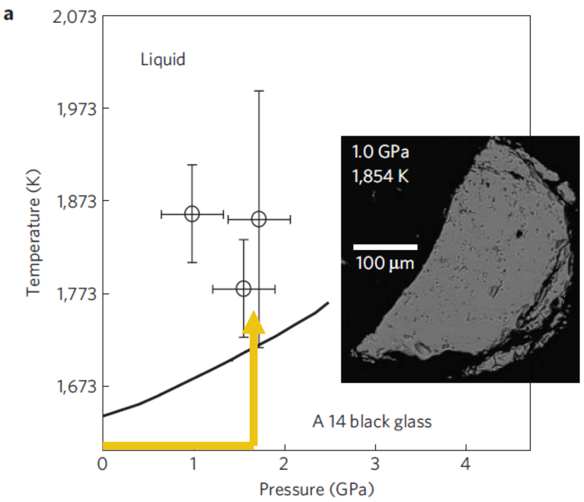
Clear absorption contrast between diamond capsule and sample (low Z) & can be improved by changing E (keV)

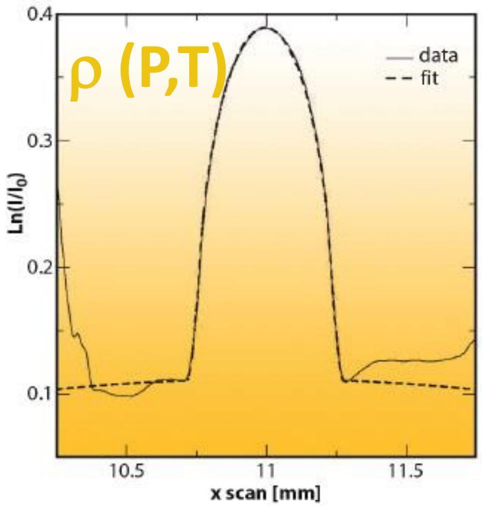
Several P-T points in one run (compressibility, thermal expansion)

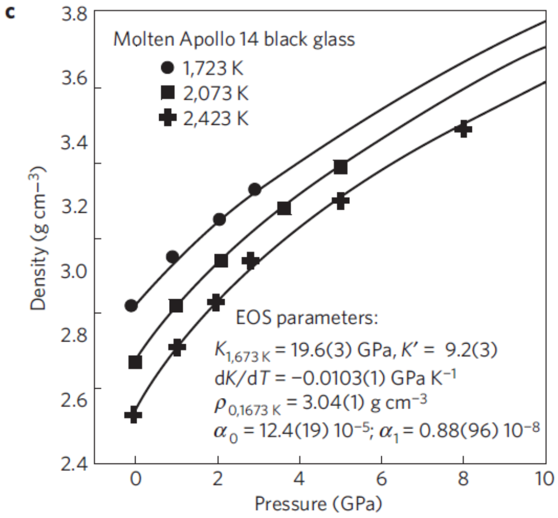
2. Density of magmas from X-Ray Absorption

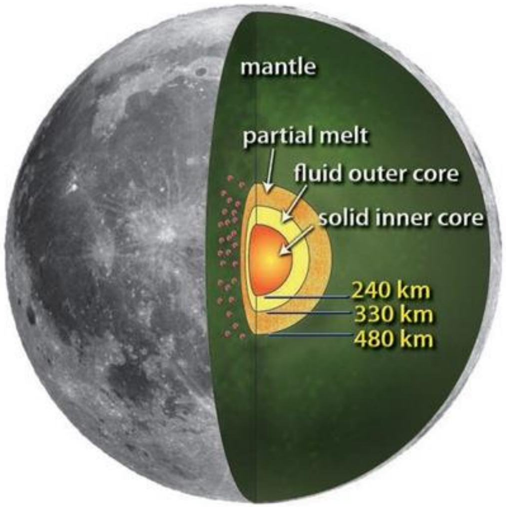
Application: density of lunar basaltic melts

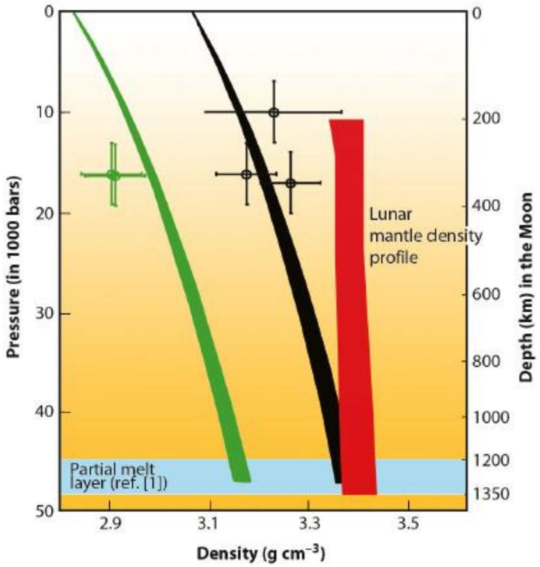
Van Kan Parker et al., Nature Geo., 2012











Difficulties:

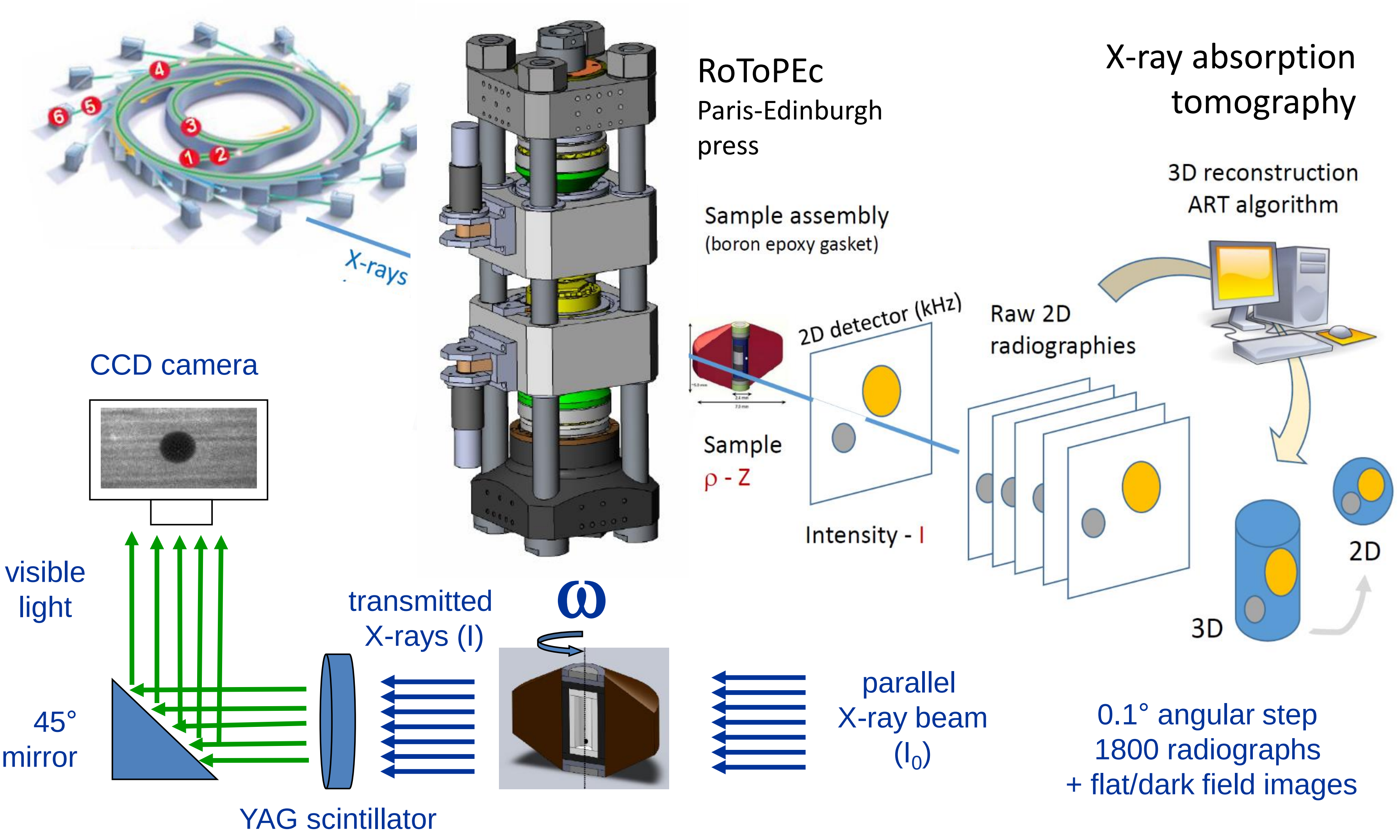
Chemical reactivity with the capsule – lids

Ensure full-melting (XRD)

Check composition on post-mortem samples

3. Density of (magmas) glasses from X-Ray Tomography

Principle: follow directly the volume change of the sample under P-T



RoToPEc

Paris-Edinburgh press

X-ray absorption tomography

3D reconstruction ART algorithm

Sample assembly (boron epoxy gasket)

2D detector (kHz)

Raw 2D radiographies

Sample

ρ - Z

Intensity - I

2D

3D

CCD camera

visible light

45° mirror

transmitted X-rays (I)

ω

YAG scintillator

parallel X-ray beam (I_0)

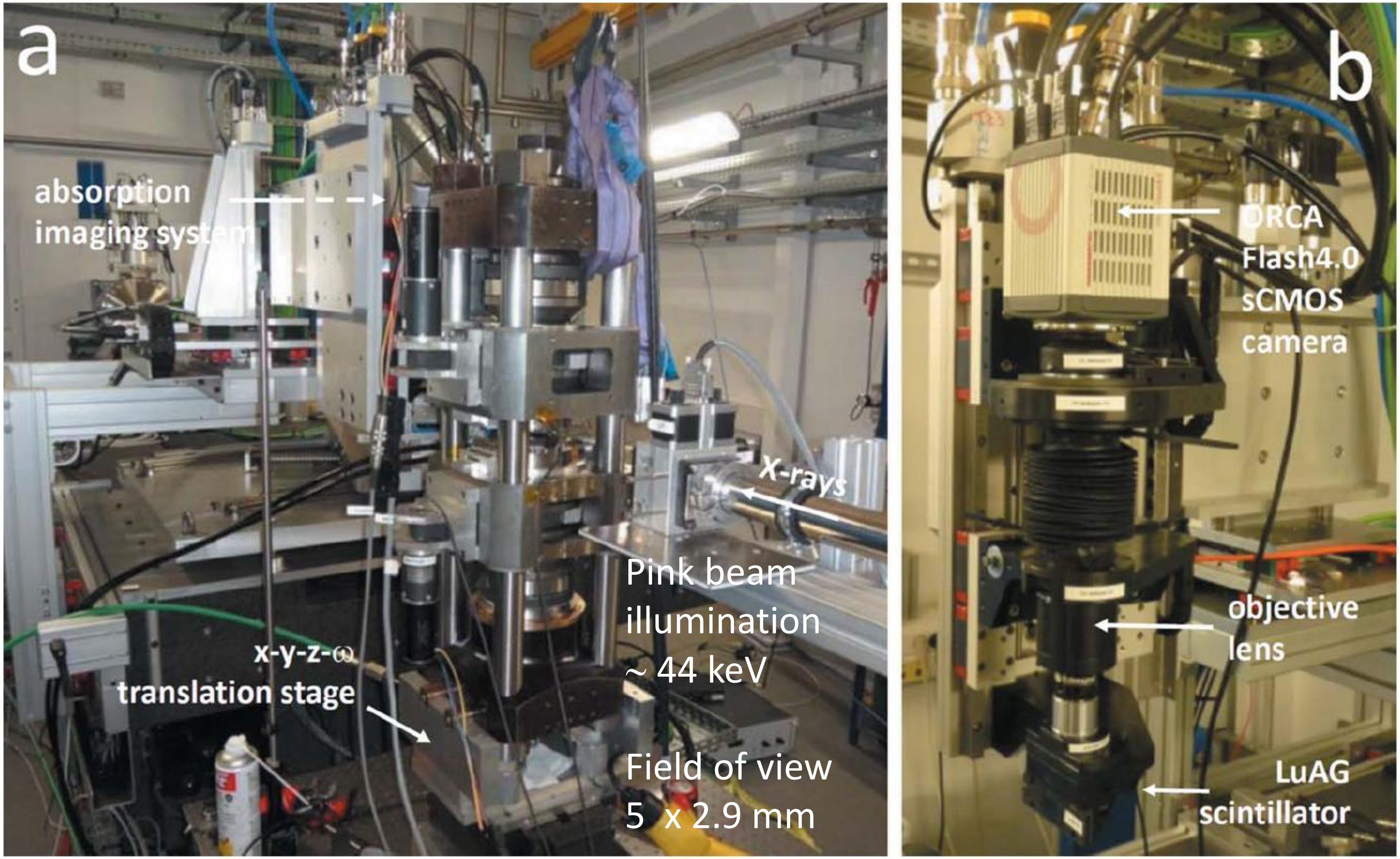
0.1° angular step

1800 radiographs

+ flat/dark field images

3. Density of (magmas) glasses from X-Ray Tomography

Experimental set-up @ PSICHE (SOLEIL)



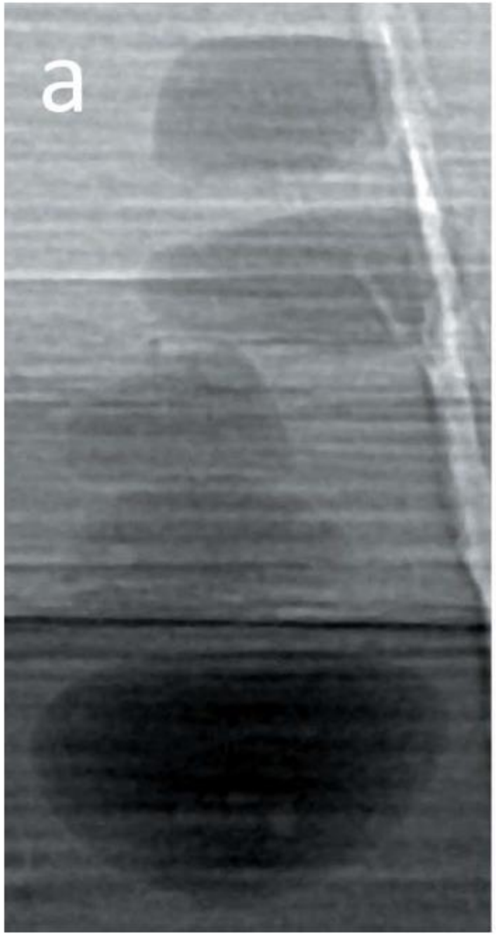
1800 images every 0.1° > collection time ~ 20 min (limited by the rotation speed of the anvils)

3. Density of (magmas) glasses from X-Ray Tomography

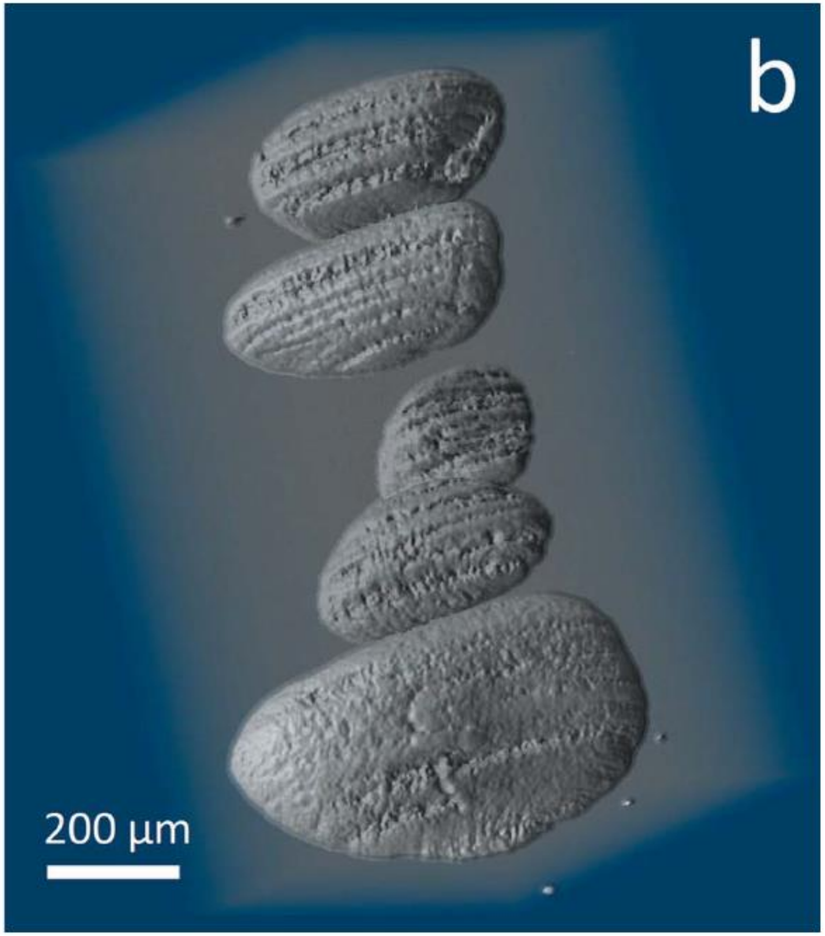
Application: compressibility of a basaltic glass

Alvarez-Murga et al., J. Synchro. Rad., 2017

2D radiography



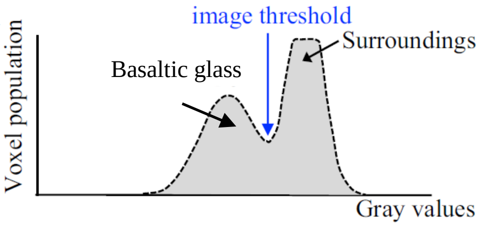
3D reconstruction



Voxel size ~ 3 μm^3

1- Segmentation

Classification of voxels from their gray values (absorption)



2 - Quantification

Number of voxels for each blob

> Volume (μm^3)

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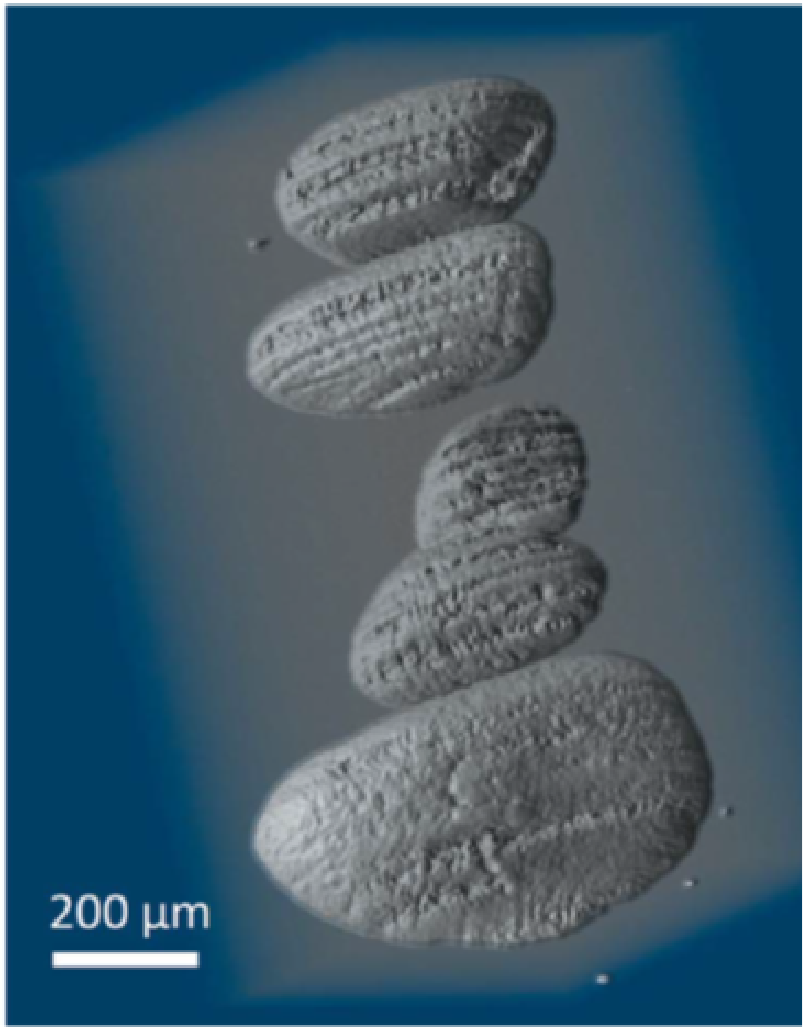
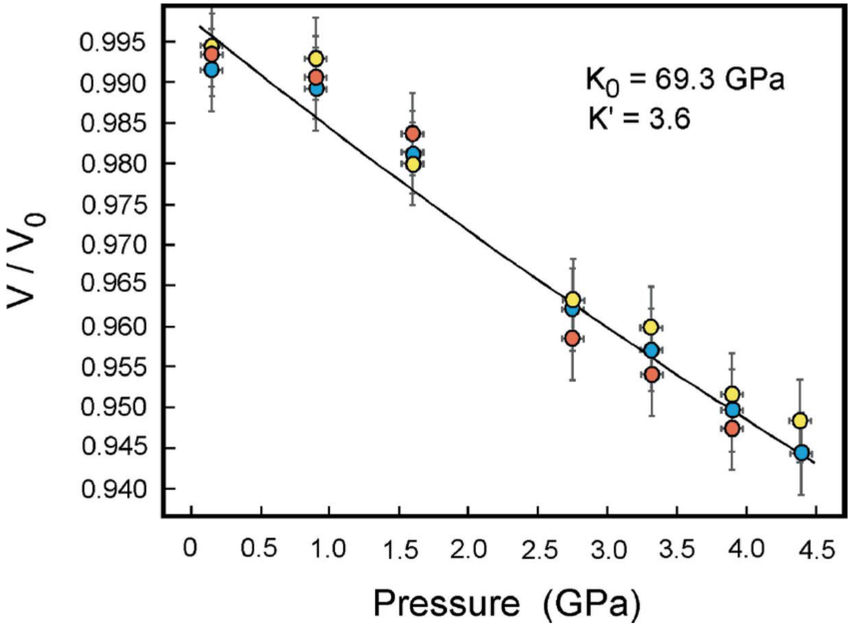
Application: compressibility of a basaltic glass

Alvarez-Murga et al., J. Synchro. Rad., 2017





Robust technique – Direct measurement of volume

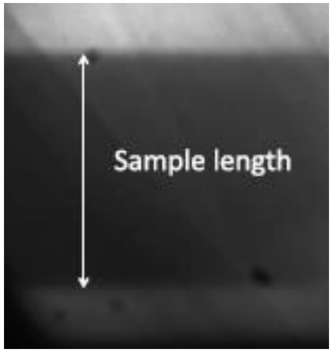
Density error 2-3 %
(image resolution, segmentation)

To date, only applied on glasses…

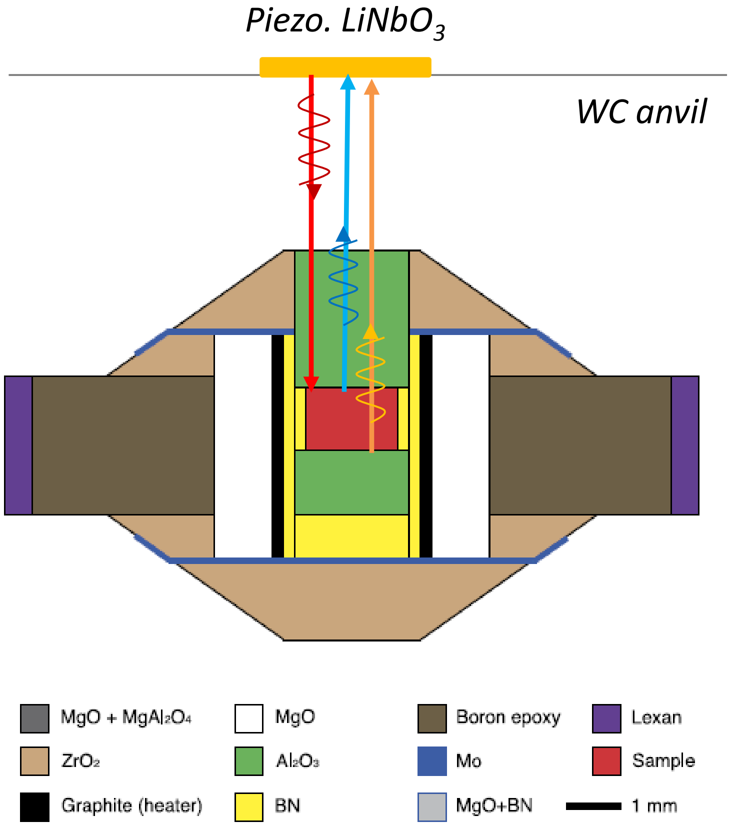
4. Sound velocity and elasticity from ultrasonic method

Principle: velocities of elastic waves in the sample from travel-times & sample length

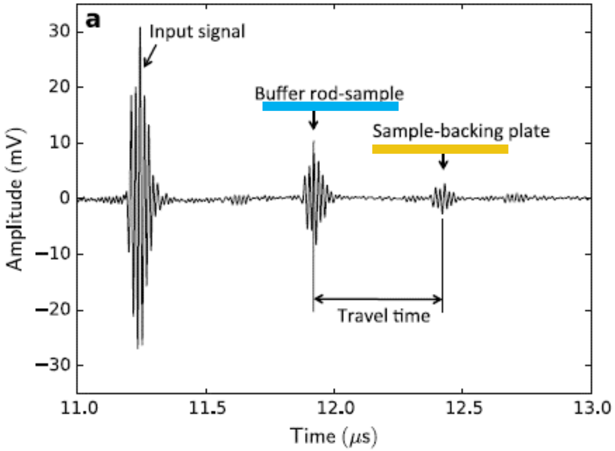
X-ray radiography



l = *sample length (μm)*



Acoustic signal



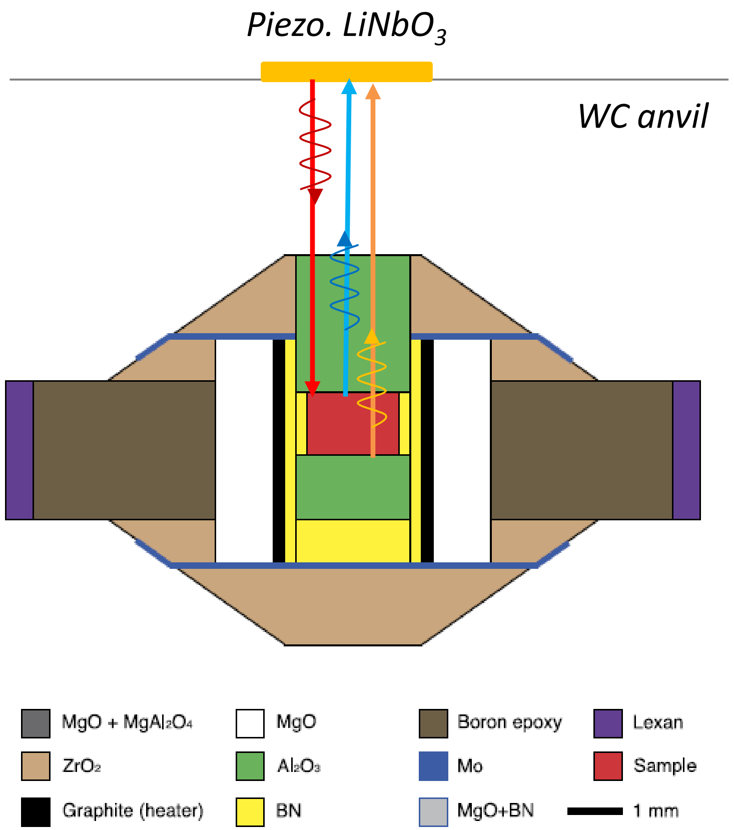
$t_{P,}$ = travel time – delay (μs)

V_P = acoustic velocity (km/s)

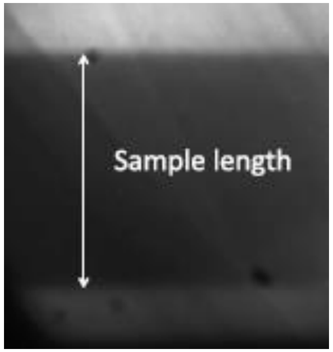
$$V_P = 2*l \,/\, t_P$$

4. Sound velocity and elasticity from ultrasonic method

Principle: velocities of elastic waves in the sample from travel-times & sample length

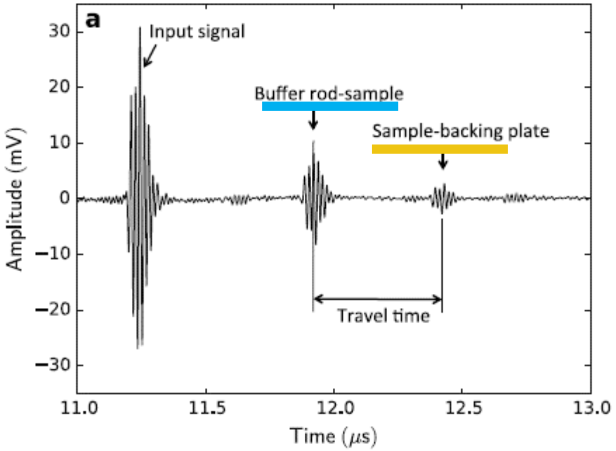


X-ray radiography



l = sample length (μm)

Acoustic signal - MHz



$t_{P,}$ = travel time – delay (μs)

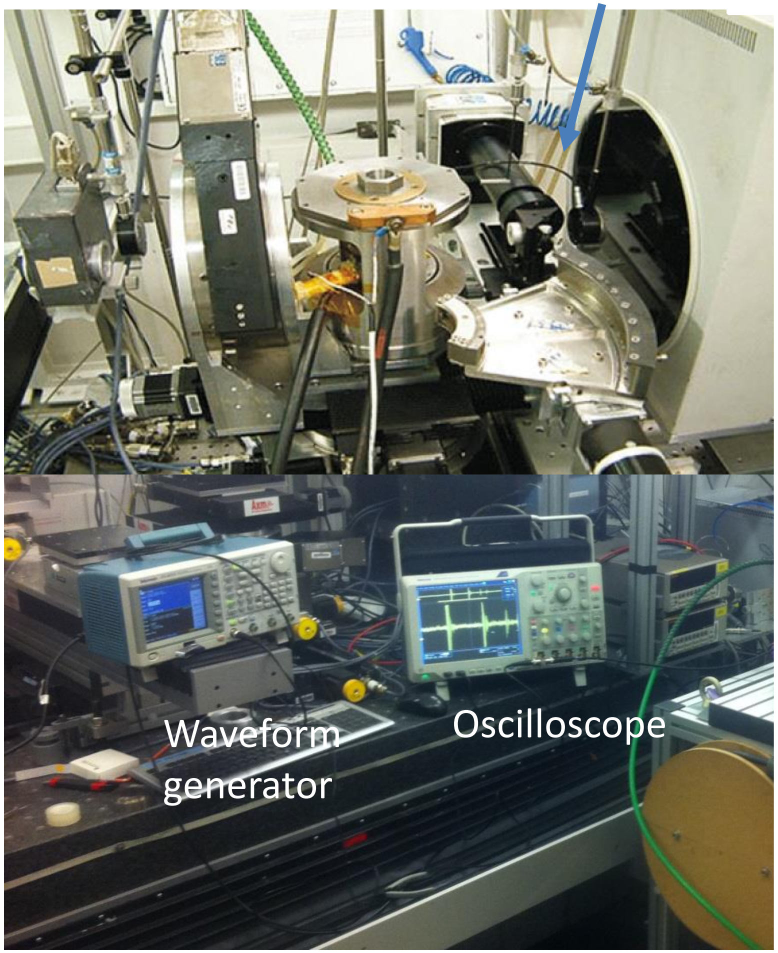
$$K_s = \rho . V_P{}^2$$

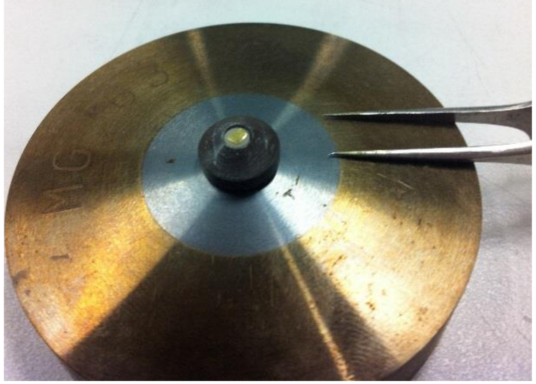
$$\rho = \rho_0\left(1+\frac{K'_S}{K_{S0}}P\right)^{1/K'_S}$$

$$V_P{}^2 = \sqrt{\frac{K_{S0}}{\rho_0}}\left(1+\frac{K'_S}{K_{S0}}P\right)^{\frac{1}{2}-\frac{1}{2K'_S}}$$

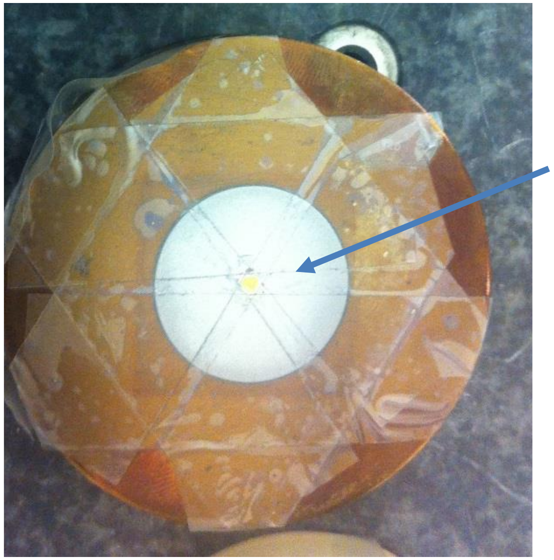
4. Sound velocity and elasticity from ultrasonic method

Experimental set-up





Cell assembly
(gold foil to improve MHz wave transmission)



$LiNbO_3$ crystal
(glued on the backside of the anvil)

X-ray diffraction for P-T calibration and solid/liquid indentification

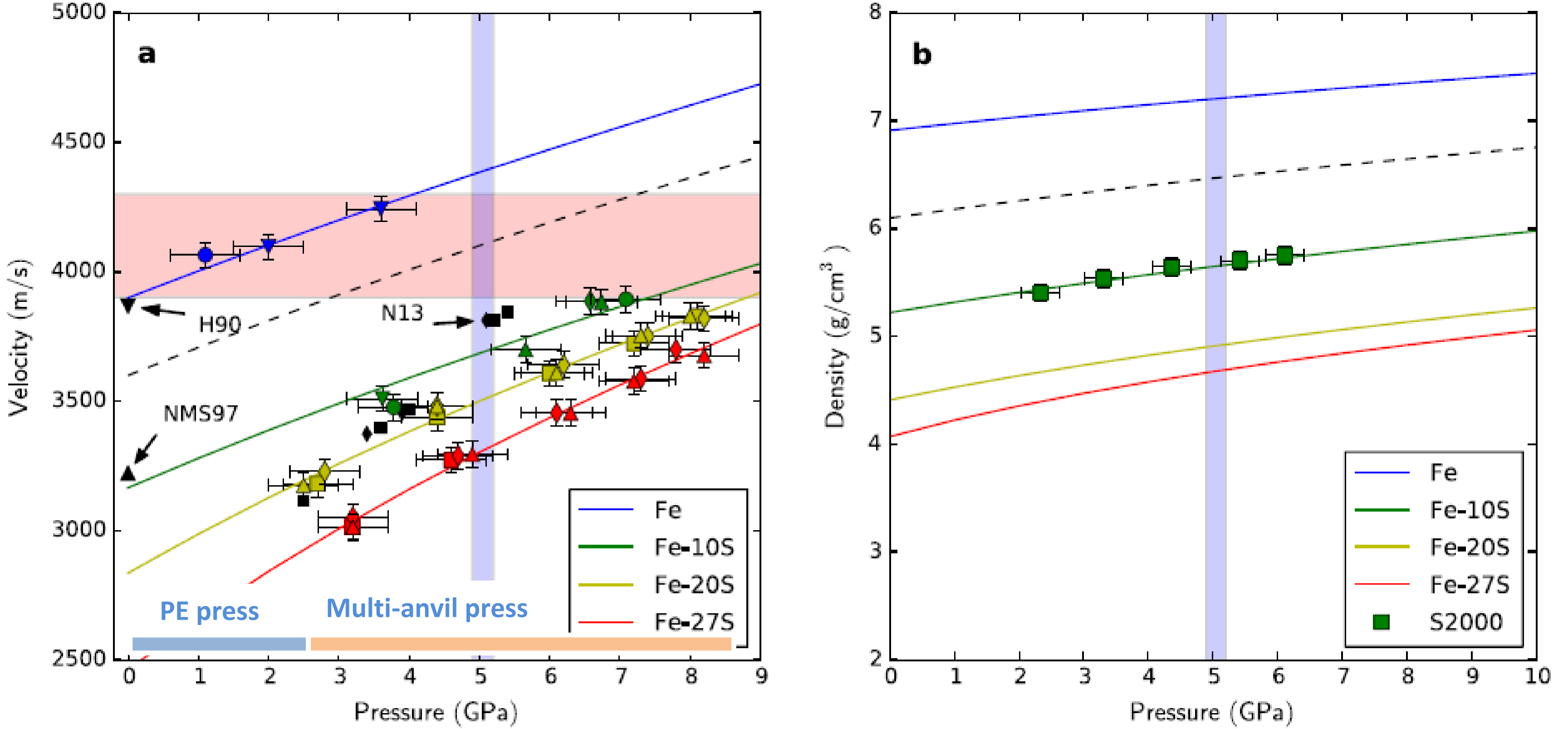
4. Sound velocity and elasticity from ultrasonic method

Application: sound velocity of Fe–S liquids & implications for the Moon’s molten outer core

Jing et al., EPSL, 2014

Argonne NATIONAL LABORATORY

HPCAT – 16-BM-B



Comparison with lunar seismology data = sulfur content, density, temperature of the moon’s outer core

4. Sound velocity and elasticity from ultrasonic method

Application: Sound velocity of Fe–S liquids & Implications for the Moon's molten outer core

Jing et al., EPSL, 2014

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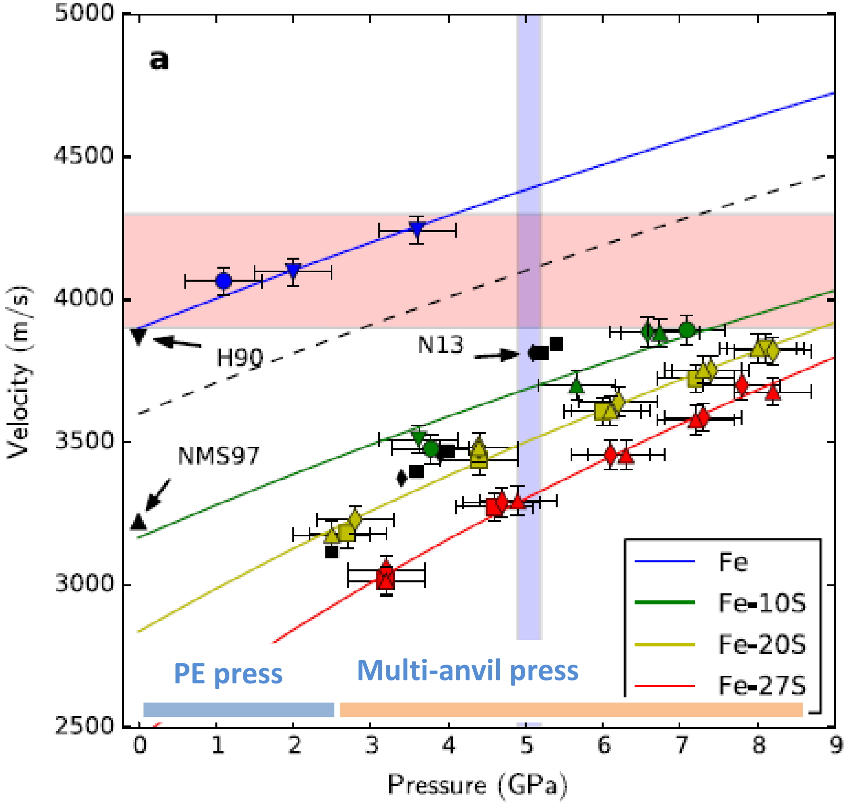


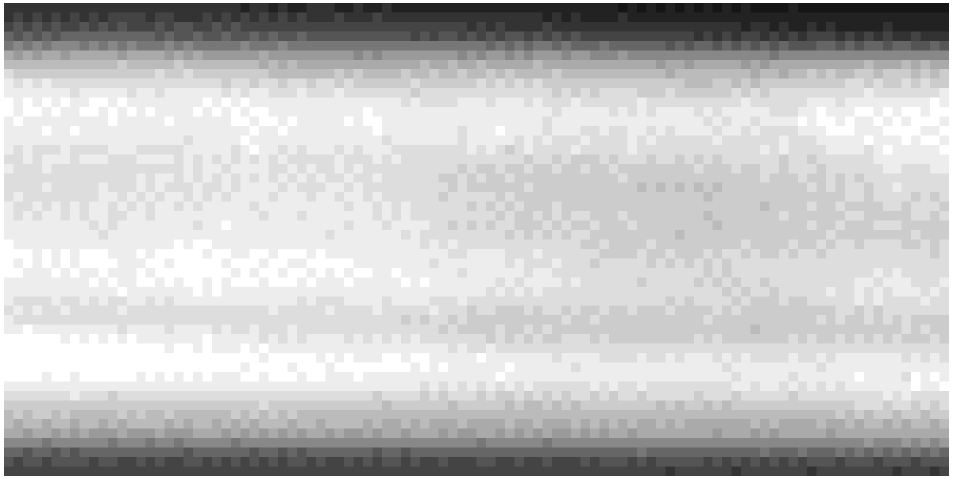
Direct method to measure sound velocities
! Frequency dependence !
(viscoelastic relaxation)

Accuracy
= depend on signal/noise ratio of elastic waves
(transmission vs reflection, impedenace contrast)
= sample length

Mostly applied for solids, or metal alloys to date…

5. Viscosity of magmas

Principle: « Stoke’s law » - Measure the settling velocity of a dense metallic sphere within the liquid under HP-HT – « **falling sphere** » technique



Time-resolved radiography

High-speed CCD camera – 1 frame/15 ms

Good spatial resolution – 2.5 µm/pix

Dense sphere (Pt, Re, WC) – ∅ = 50-200µm

Suitable for viscosity measurements down to 10^{-2}-10^{-3} Pa.s

$$\eta = \frac{2 g r_s^2 (\rho_s - \rho_l)}{9V} \cdot \frac{F}{K}$$

V = settling velocity (m/s)

r = radius of the metallic sphere (m)

ρ_s = density of the metallic sphere (kg/m^3)

ρ_l = density of the liquid (kg/m^3)

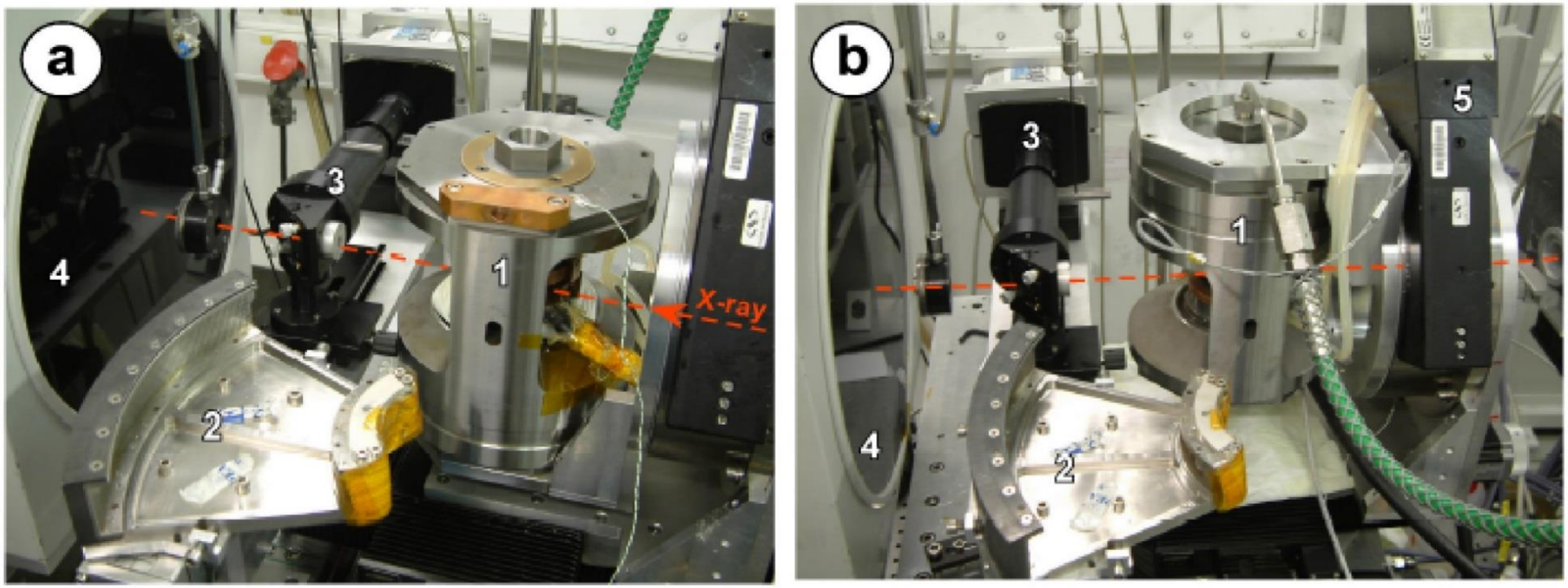
F - K = wall effect & finite length corrections

ρ_s, ρ_m, R_s = f (P,T)

5. Viscosity of magmas

Experimental set-up @ ID27 ESRF

Perrillat et al., High Pres. Res. (2010)



Upside down rotation of the press for multiple viscosity measurements

X-ray radiography

Full beam: 1.2 x 1.5 mm

High space & time resolution

X-ray diffraction

Collimated beam: 25x25 μm

P-T calibration

Structure of melts (Soller slits)

X-ray absorption

Ion chambers

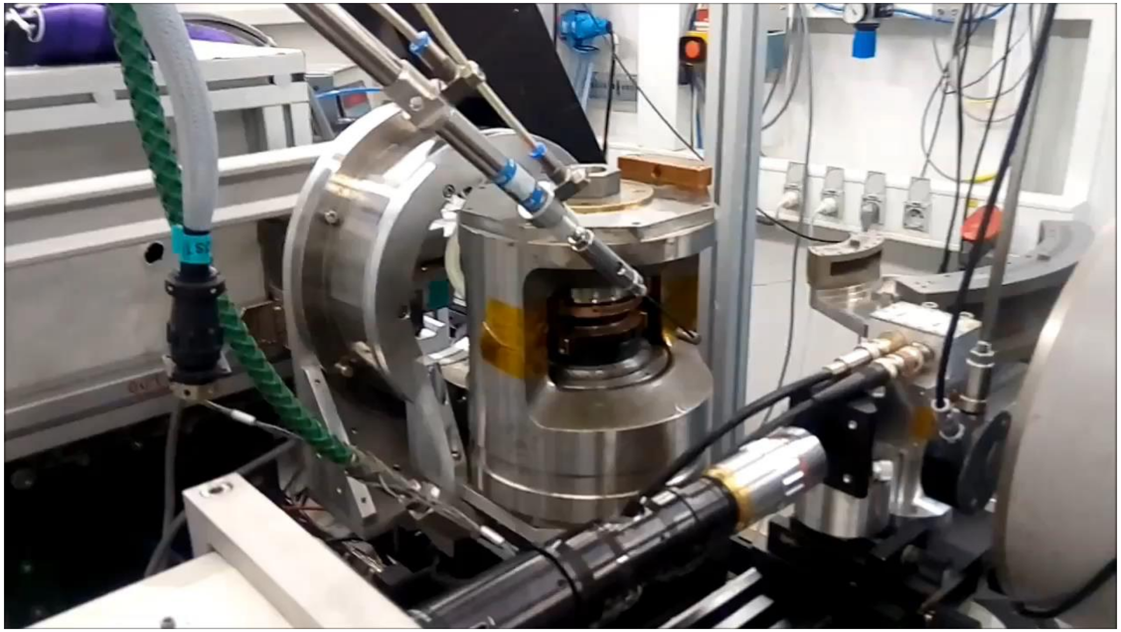
I/I_0 scans

Density of melts

5. Viscosity of magmas

Experimental set-up @ ID27 ESRF

Perrillat et al., High Pres. Res. (2010)



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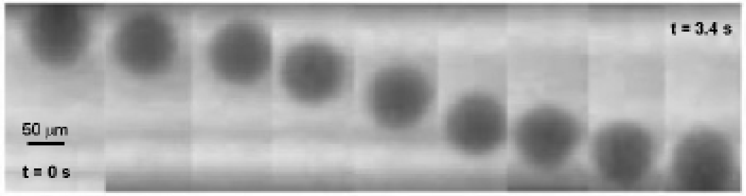
5. Viscosity of magmas

Application: viscosity of primitive lunar magmas

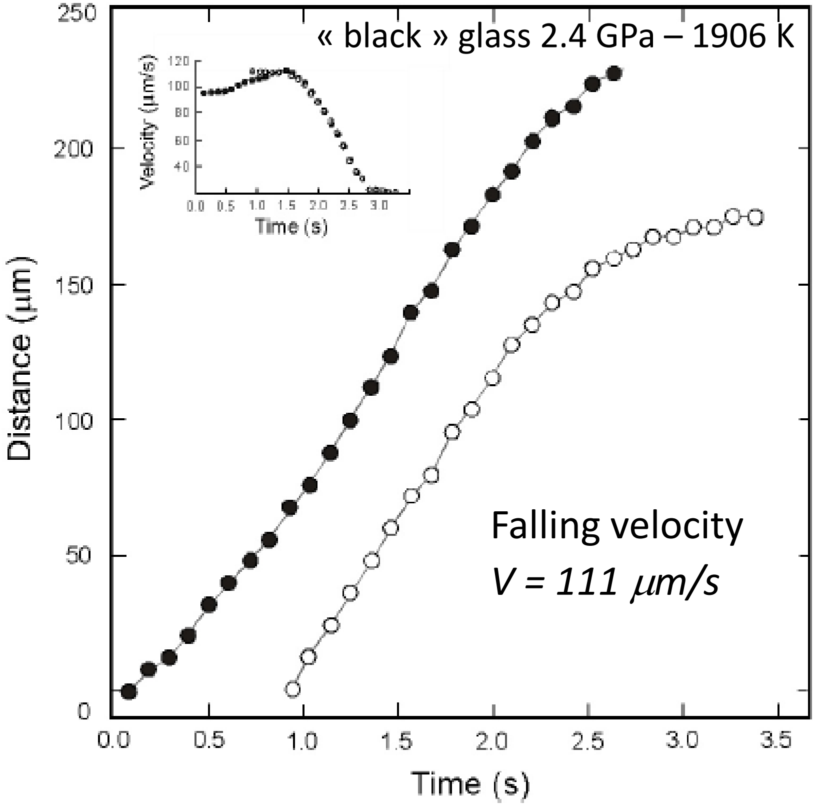
Rai et al., Frontiers, 2019



measured

measured or EoS

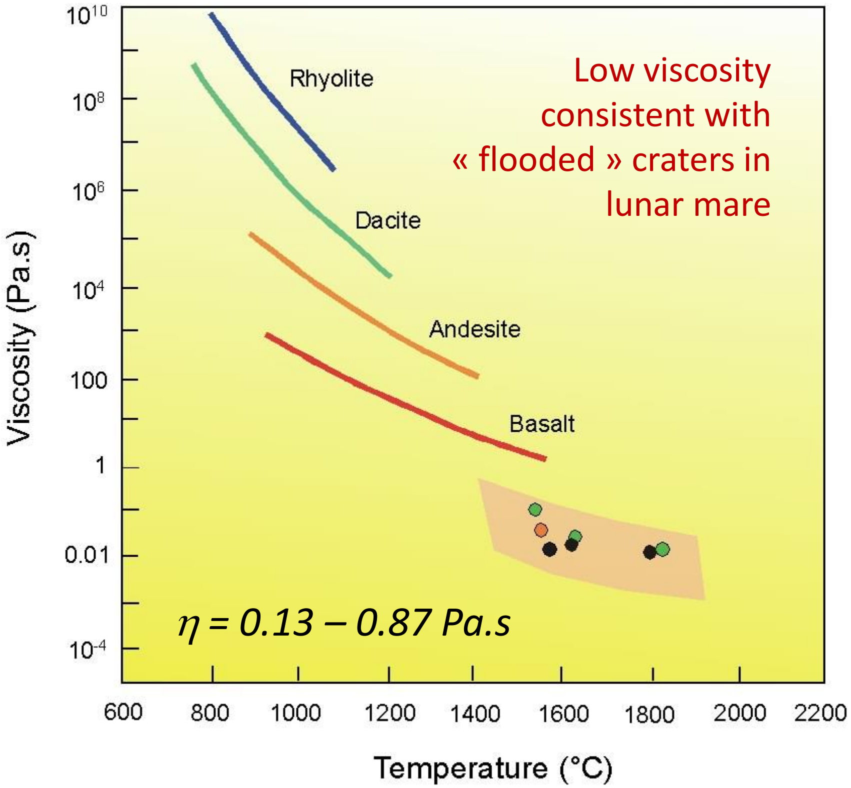
$$\eta = \frac{2 g r_s^2 (\rho_s - \rho_l)}{9V} \cdot \frac{F}{K}$$



« black » glass 2.4 GPa – 1906 K

Falling velocity

V = 111 µm/s



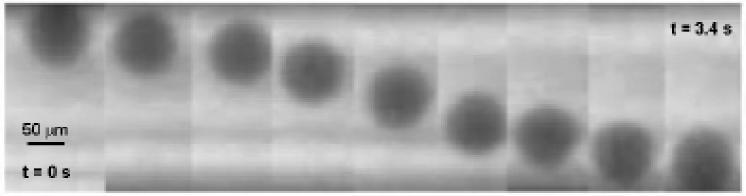
Low viscosity consistent with « flooded » craters in lunar mare

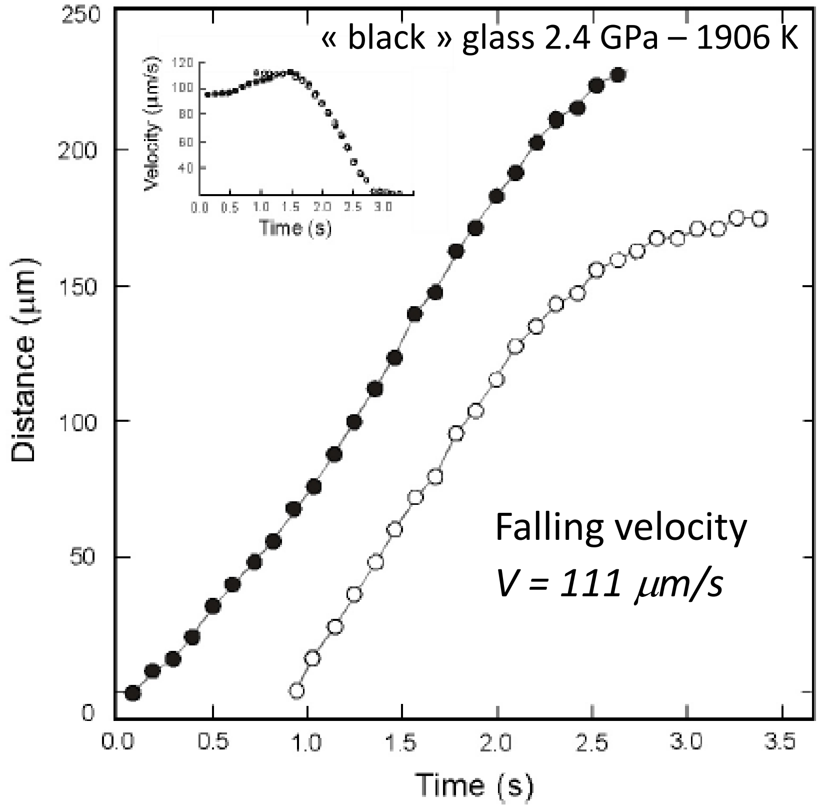
$\eta = 0.13 - 0.87$ *Pa.s*

5. Viscosity of magmas

Application: viscosity of primitive lunar magmas

Rai et al., Frontiers, 2019





« black » glass 2.4 GPa – 1906 K

Falling velocity

V = 111 *μm/s*

measured

measured or EoS

$$\eta = \frac{2gr_s^2(\rho_s - \rho_l)}{9V} \cdot \frac{F}{K}$$

Viscosity error
mostly from V, since ρ_s *-*ρ_l *is large*

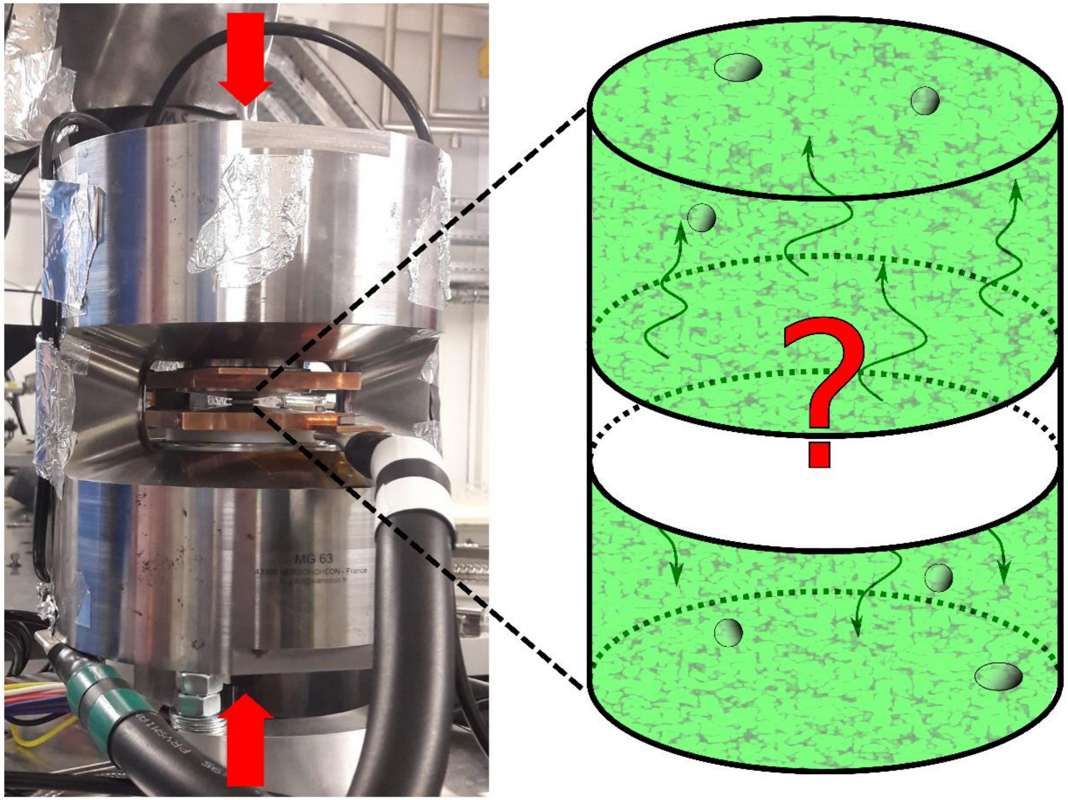
Suitable for viscosity measurements from 10^{-3} *to* 10^{2} *Pa.s*

Ensure full melting at falling
> Fast heating ramp
> Sphere-trap assembly
(the dense sphere is enclosed in a material with higher melting T° than the sample)

Recent progress & Perspectives

Magmas migration from 4D Tomography

2 GPa
T > 835°C



Polycristalline
olivine

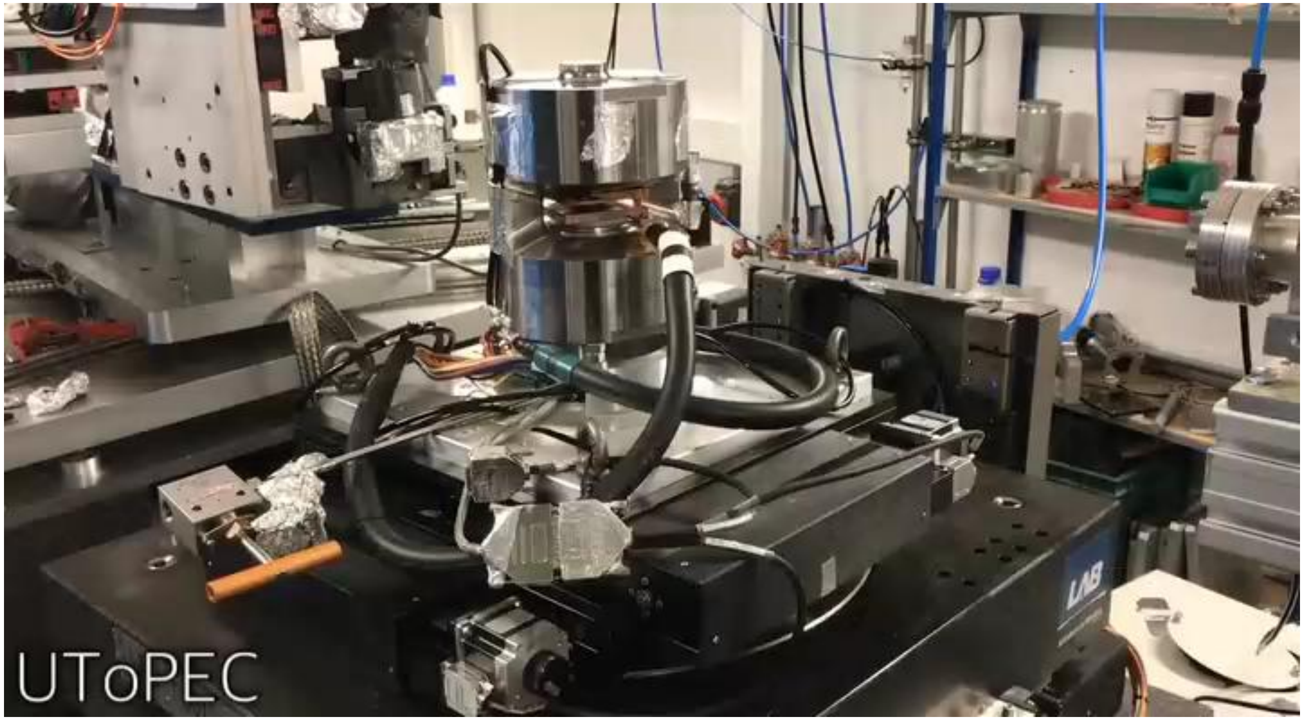
$BaCO_3$ carbonate
(melting at T > 800°C)

Giovenco et al., 2019

Kinetics of carbonate magmas migration within the Earth's mantle
Microsctructure & geometry of the melt phase (permeability, wetting angle, porosity)

Recent progress & Perspectives

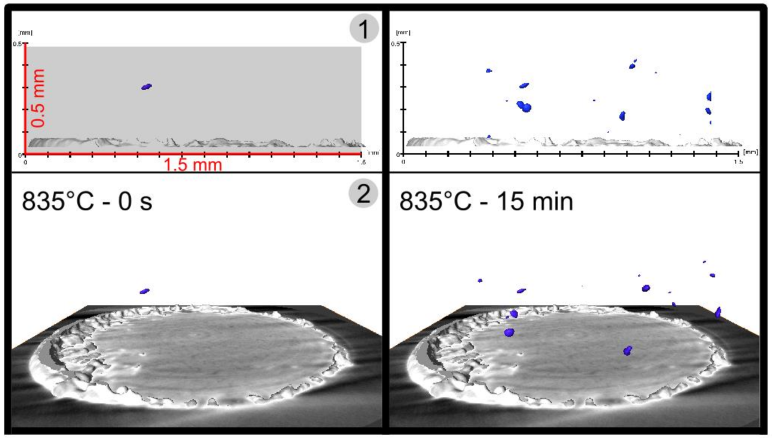
Magmas migration from 4D Tomography



1 CT scan / second > suitable for fast migration of low viscosity fluid

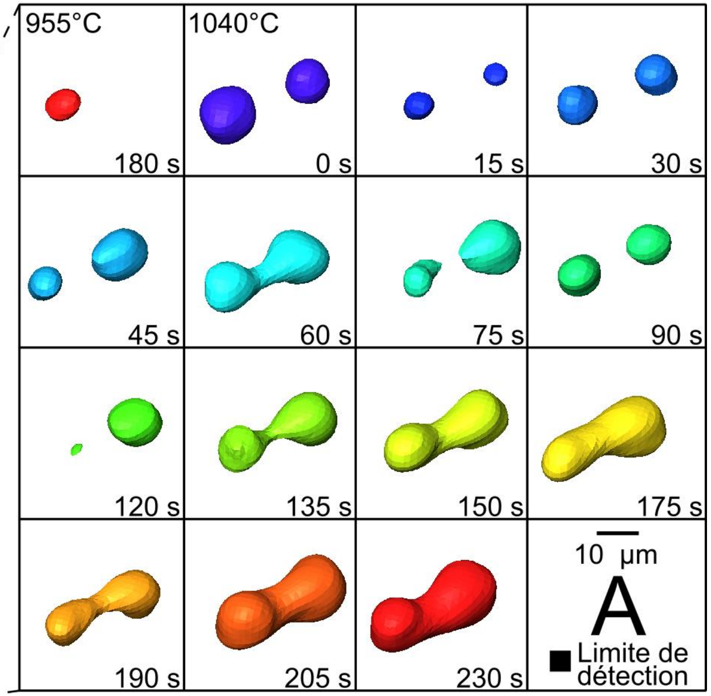
Magmas migration from 4D Tomography

Fast impregnation = 2 mm/h

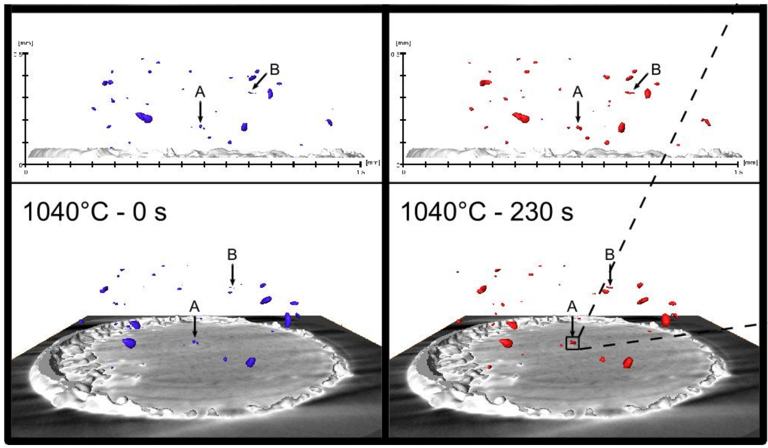


Geometry of the melt phase and evolution with time

Giovenco et al., 2019



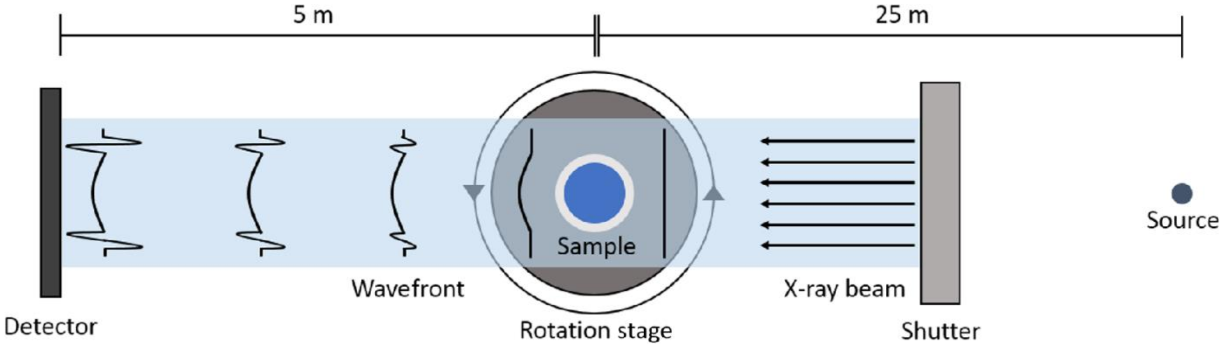
Densification of the liquid network with time



Recent progress & Perspectives

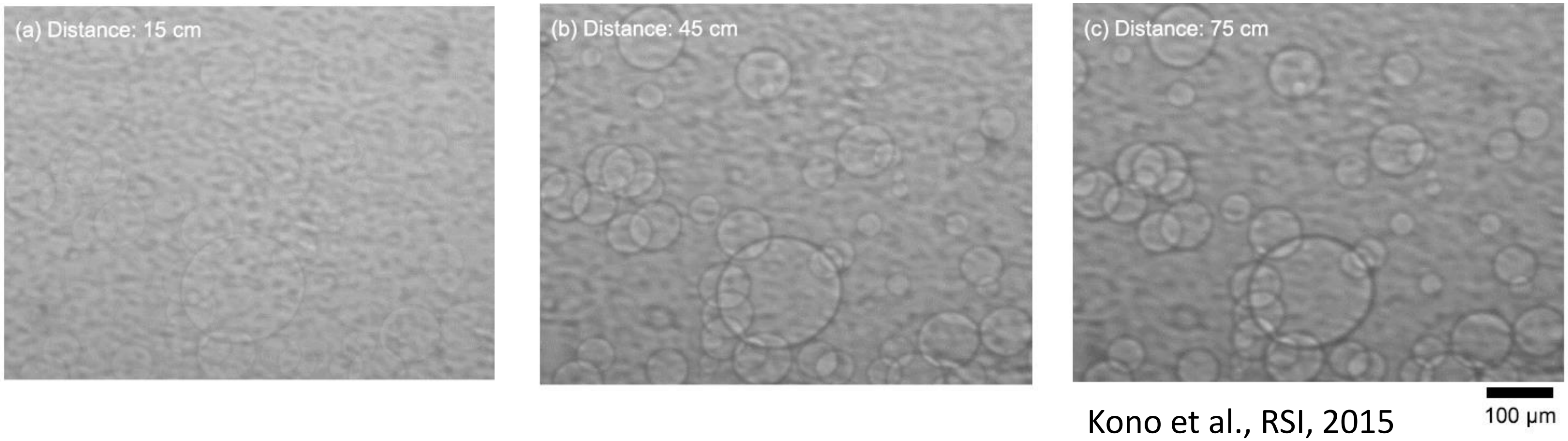
Use of the high coherence of the beam = Phase Contrast Imaging

Propagation based phase contrast imaging



$Dc = \lambda .L / 2\sigma$ L = source to sample distance σ = source size

Holotomography = reconstruction of the local phase – phase retrieval algorithm

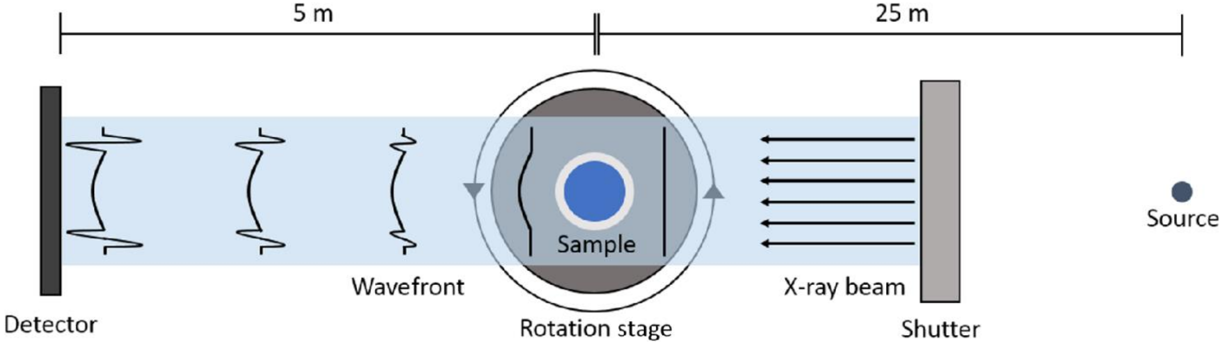


Kono et al., RSI, 2015

Recent progress & Perspectives

Use of the high coherence of the beam = Phase Contrast Imaging

Propagation based phase contrast imaging

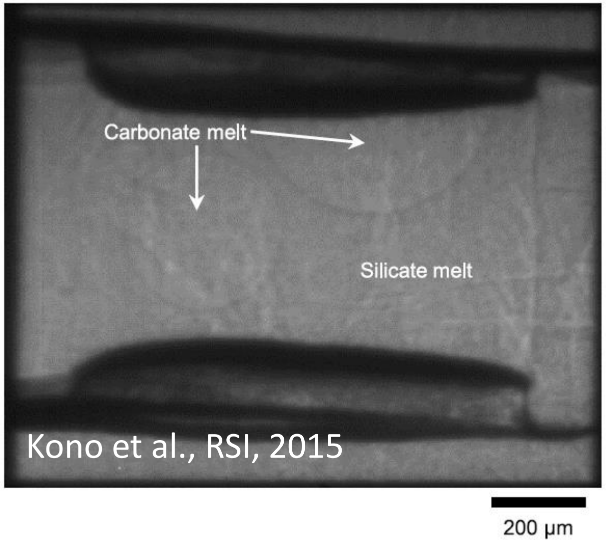


$Dc = \lambda . L / 2\sigma$ L = source to sample distance σ = source size

Holotomography = reconstruction of the local phase – phase retrieval algorithm

Imaging of materials of similar absorption (otherwise uniform)

Ex: immiscibility between magmas of different compositions



Kono et al., RSI, 2015

Thanks for your attention !