# First experiments with D-DIA apparatus on XAS

Jeremy Wykes

*Macquarie University & Australian Synchrotron*

# Acknowledgements


- AS Controls group
  - William Rambold, Nader Ashfar, Andy Staritt
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- GSECARS
  - Yanbin Wang, Julien Gasc, Mark Rivers
- COMPRES
  - Kurt Leinenweber
- MQ
  - Tracy Rushmer, Simon Turner, Simon Clark, George Amulele, Anthony Lanatti, Bruno Colas

# Timeline

- December 2011: ARC LIEF LE120100076
    - *The first Australian high pressure Synchrotron facility for geoscience research*
    - *Rushmer, O'Neill, Cruden, Turner.*
- August 2013: Apparatus delivered
- April 2014: Mark Rivers writes initial control software
- March 2015: Jeremy hired
- March 2016: First high P run
- May 2016: First *in situ* high P XANES measurement
- June 2016: First XANES measurement of silicate liquid
- July 2016: First imaging/falling sphere experiment

# What is D-DIA?

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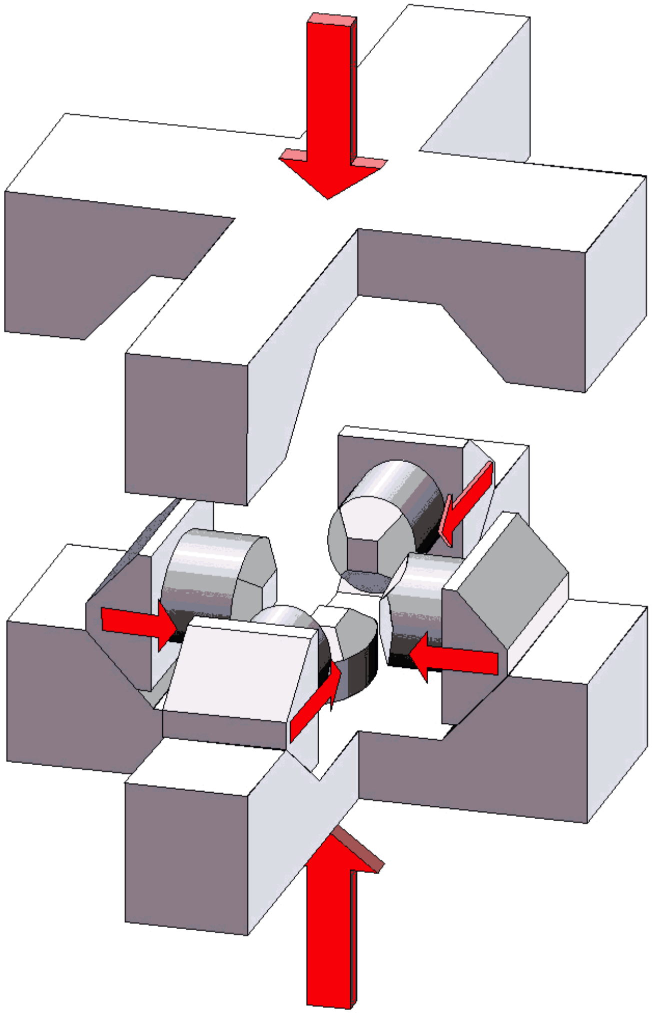
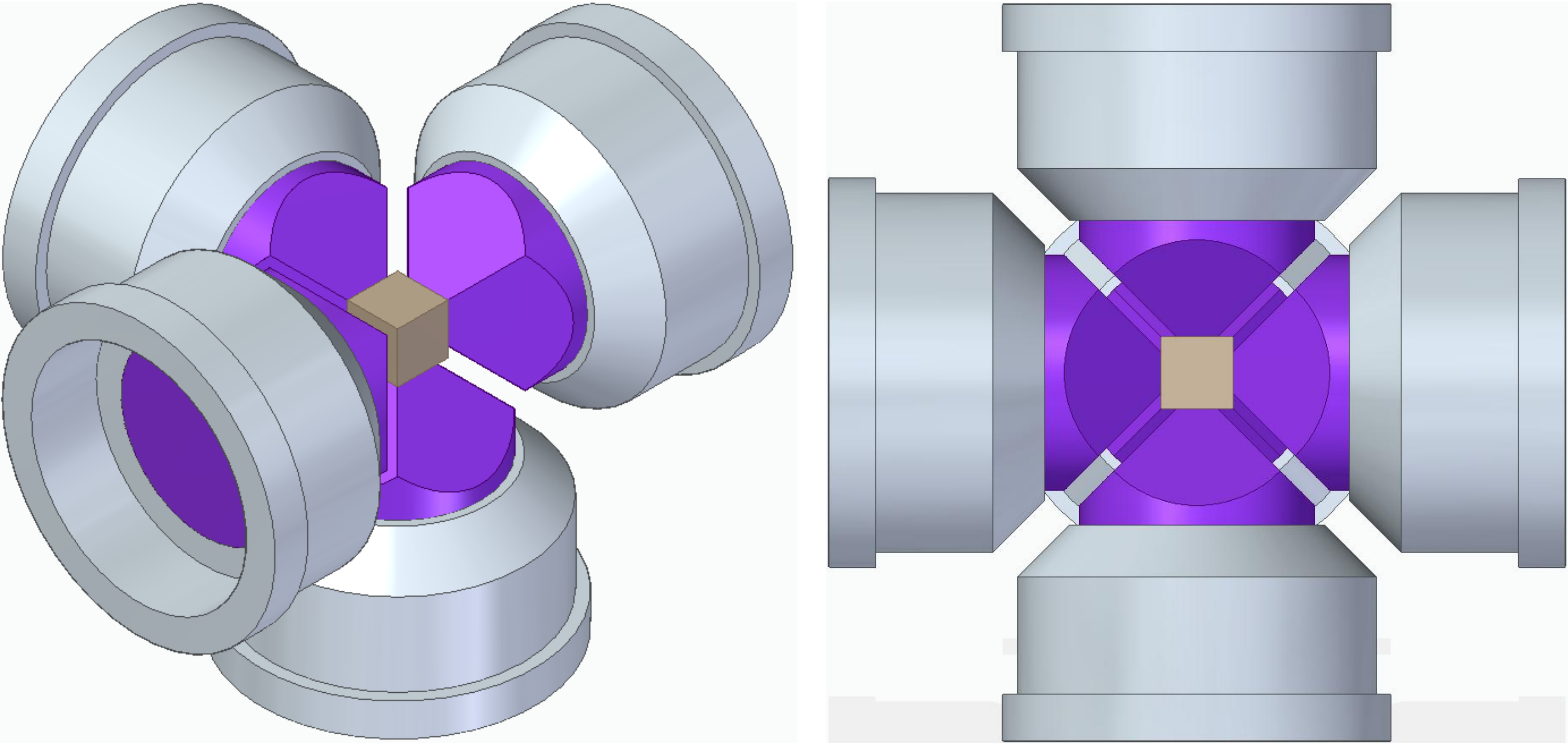
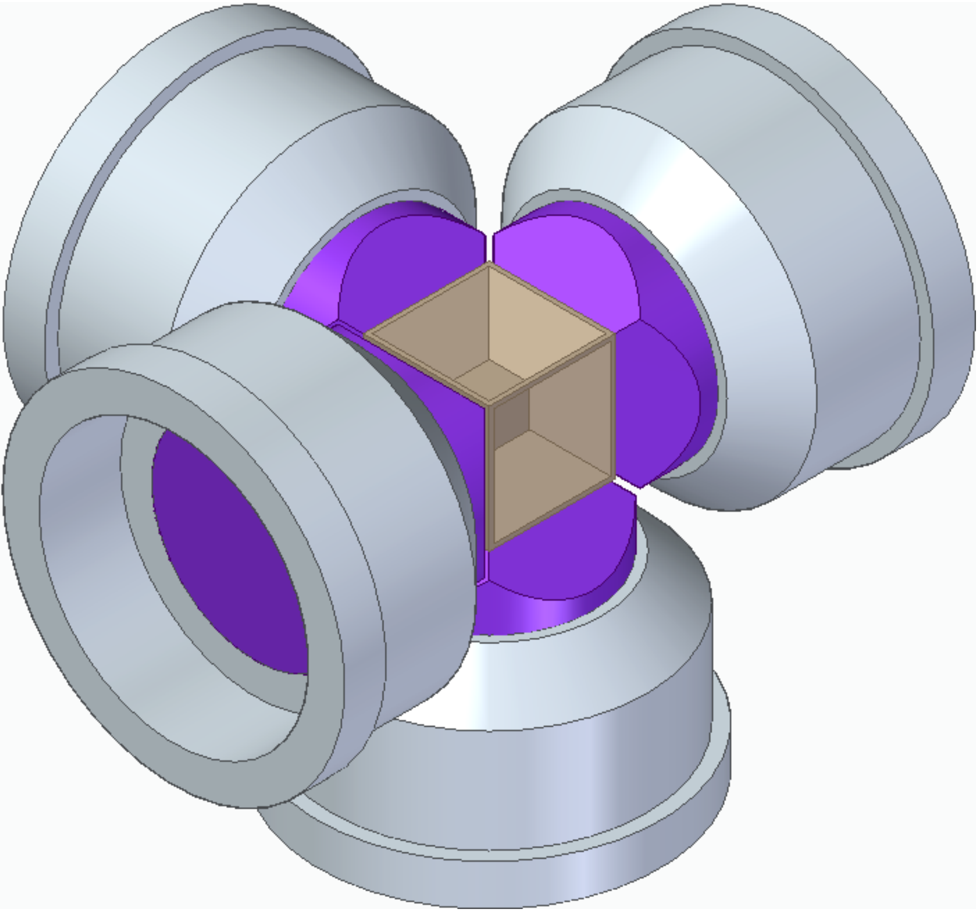
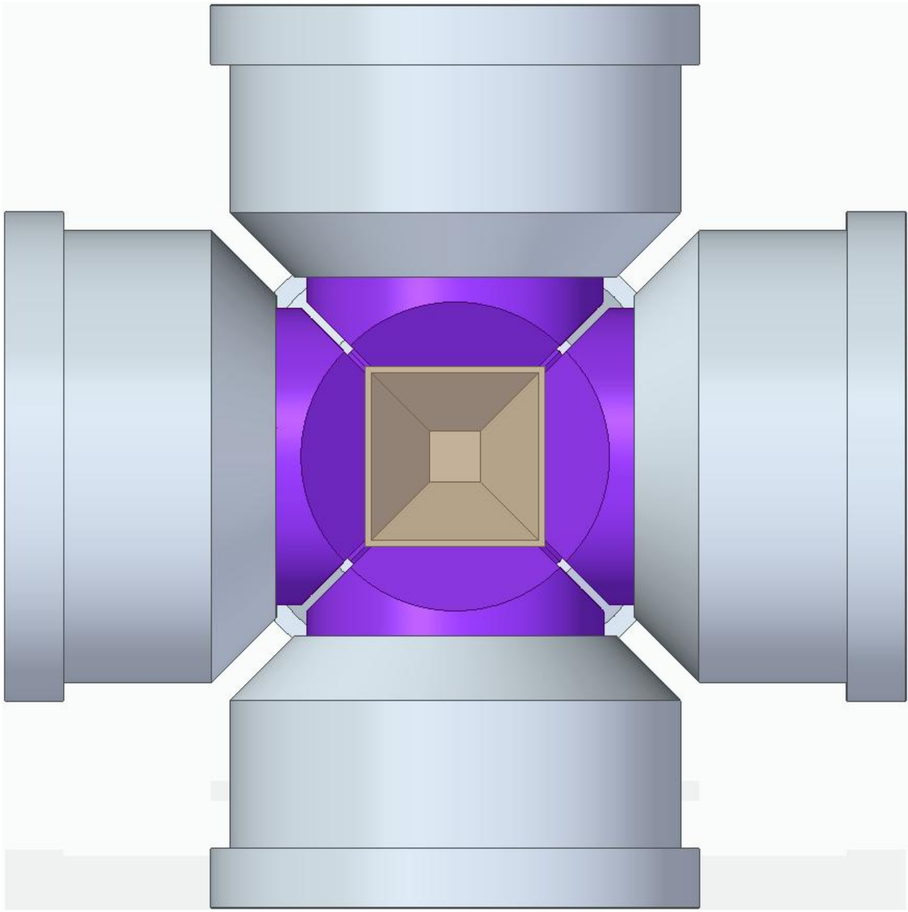
- Multi-anvil apparatus
    - Anvils remain in compression
    - Principle of massive support
- 6 anvils
- Cubic sample volume
    – Cubic multi-anvil





7 mm cube
4 mm anvil truncation





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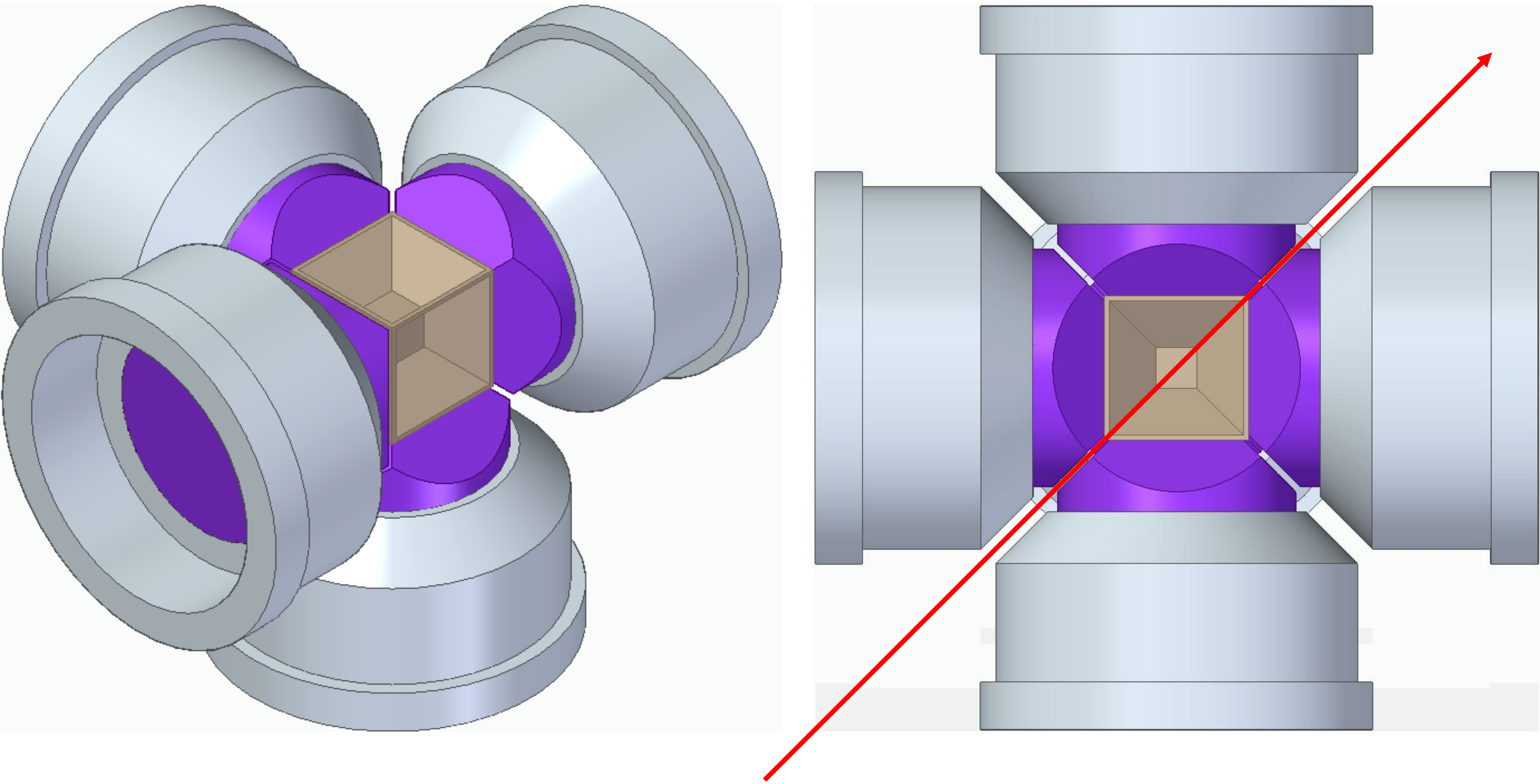

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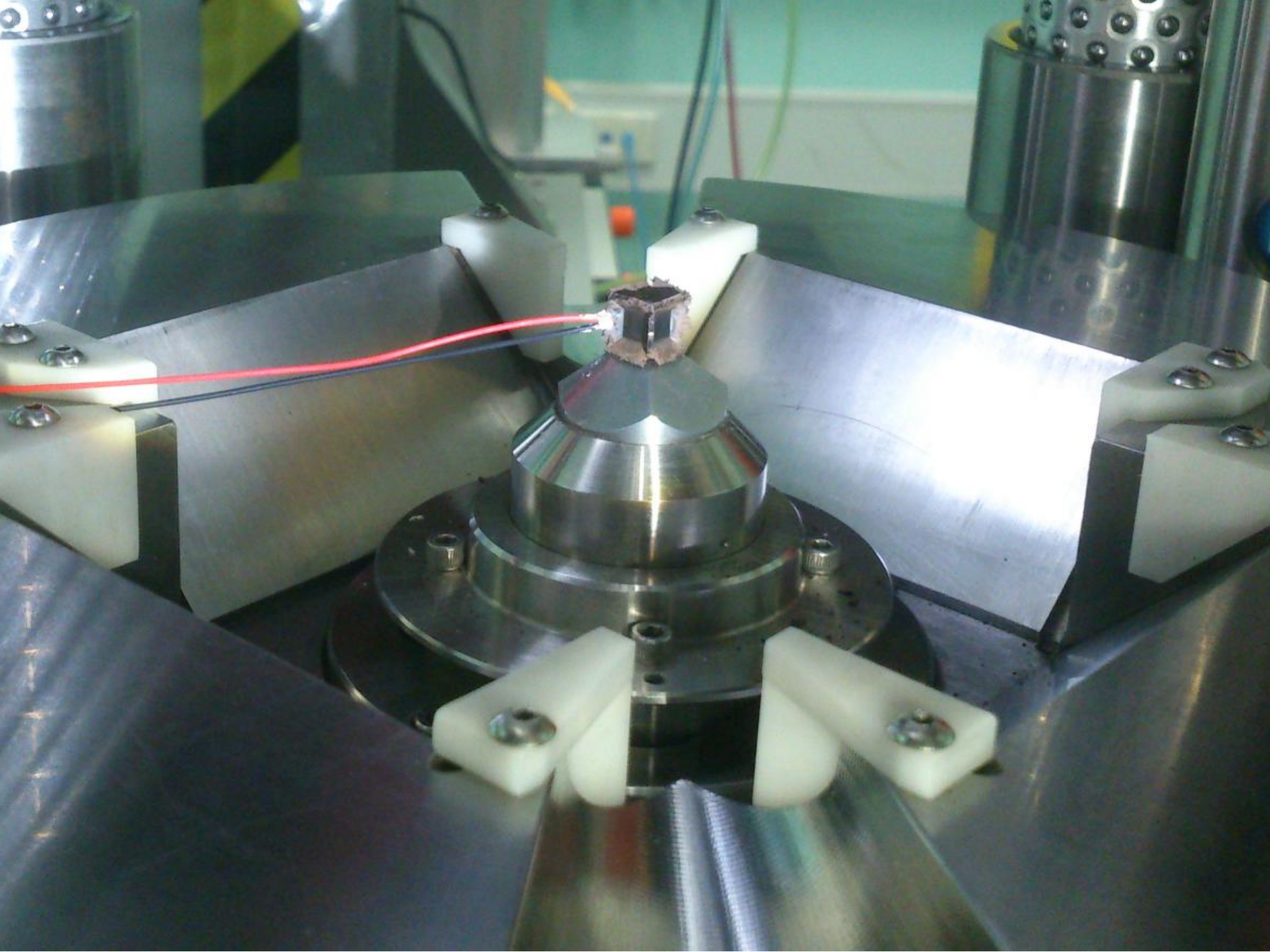
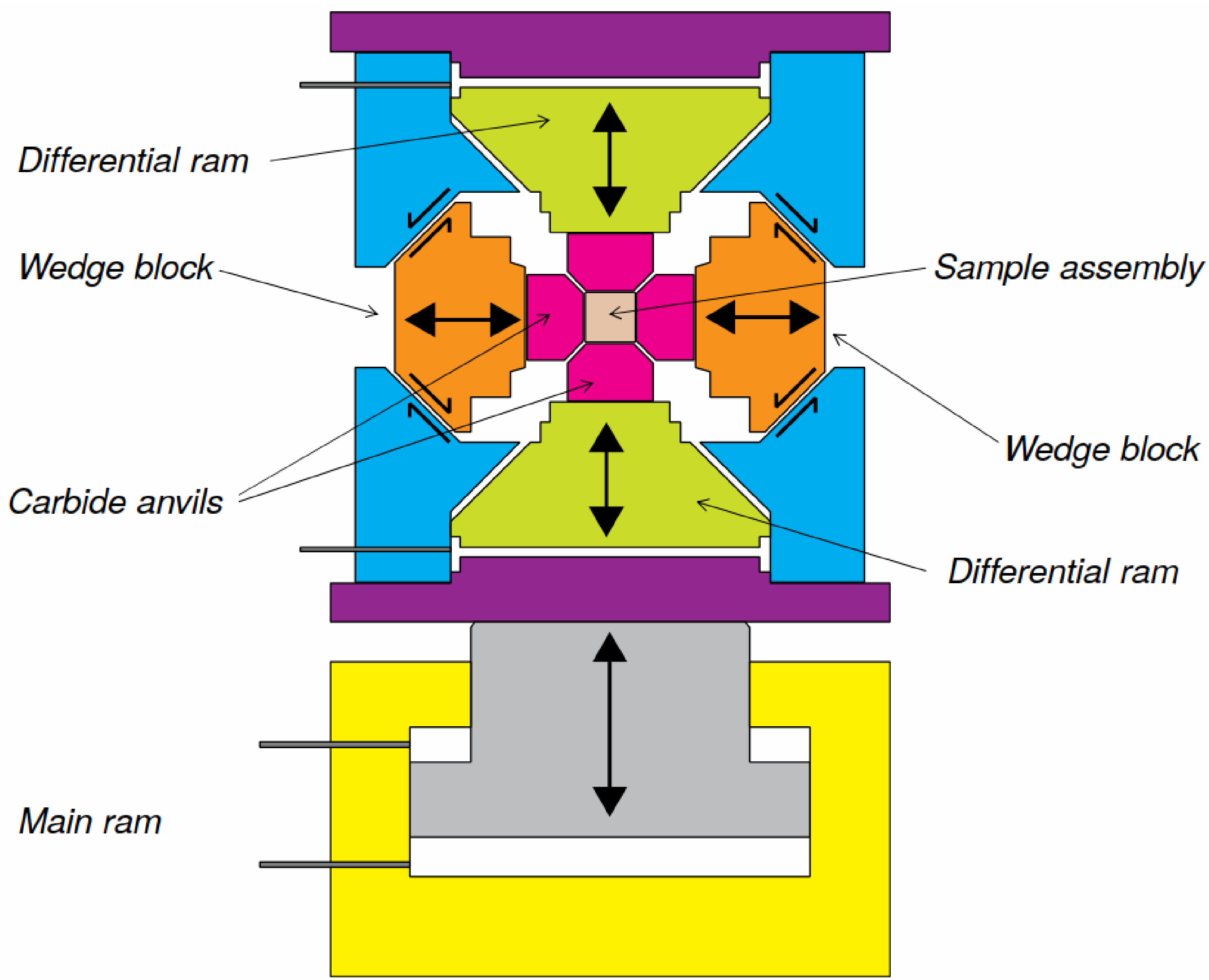


# Deformation-DIA apparatus



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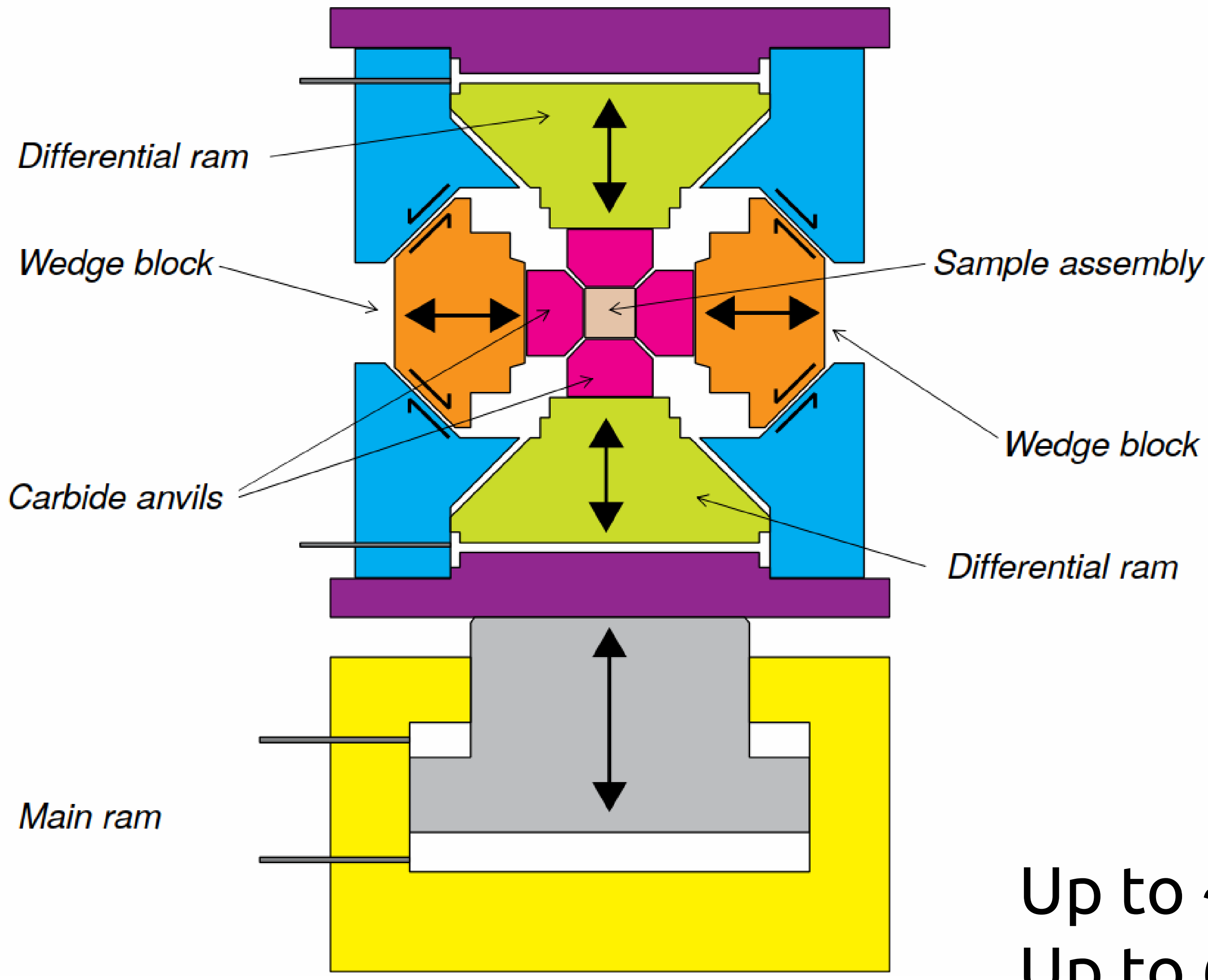
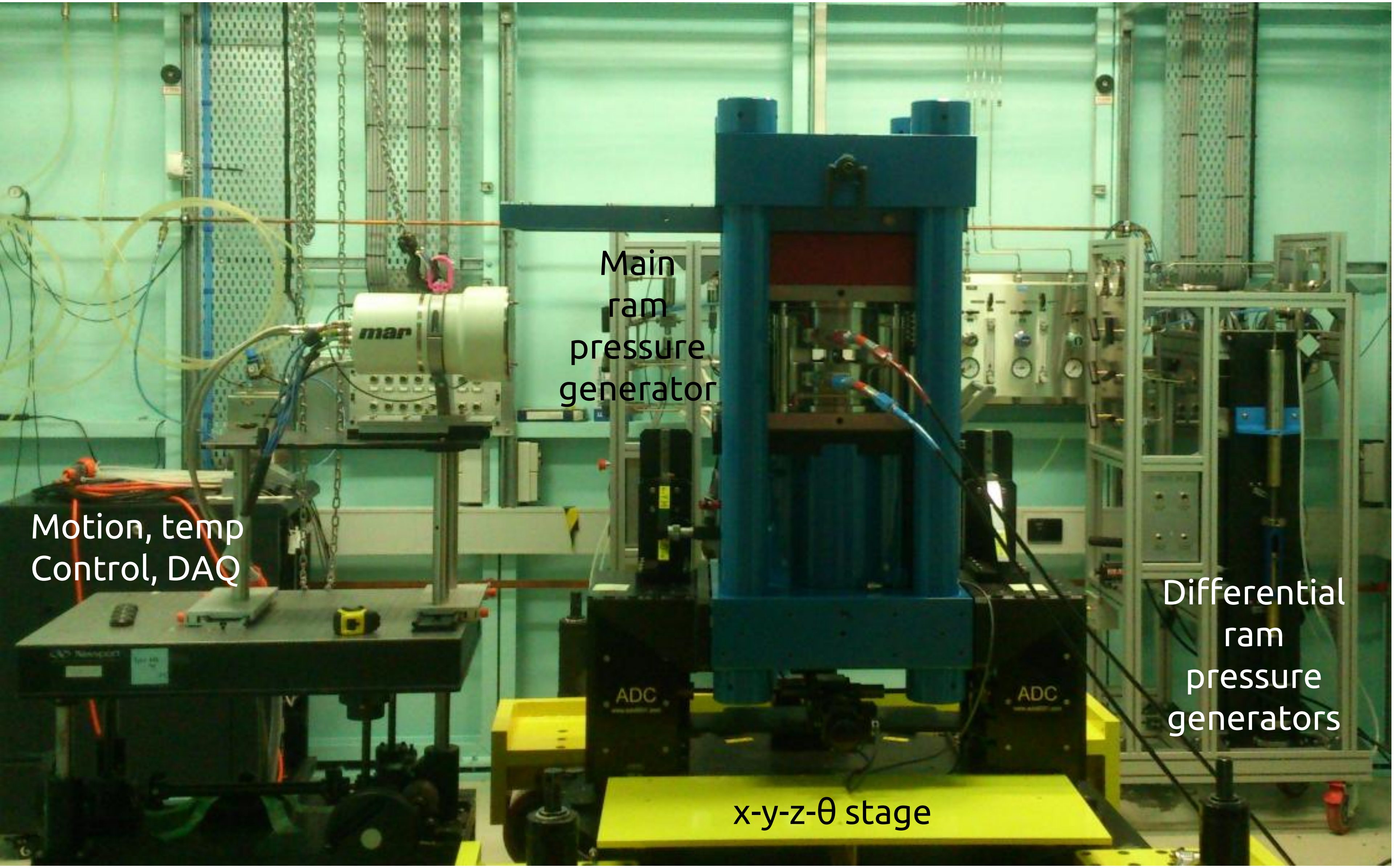



Up to ~1500°C
Up to 6 GPa

May 2016



Main
ram
pressure
generator
Motion, temp
Control, DAQ
Differential
ram
pressure
generators
x-y-z-θ stage
mar
ADC
ADC

# First user experiment

- **Proof of concept**: Can we collect XANES from silicate liquids at high P and T?

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- **Proof of concept**: U and Th $L_3$-edge XANES in MORB (mid-ocean ridge basalt) liquid

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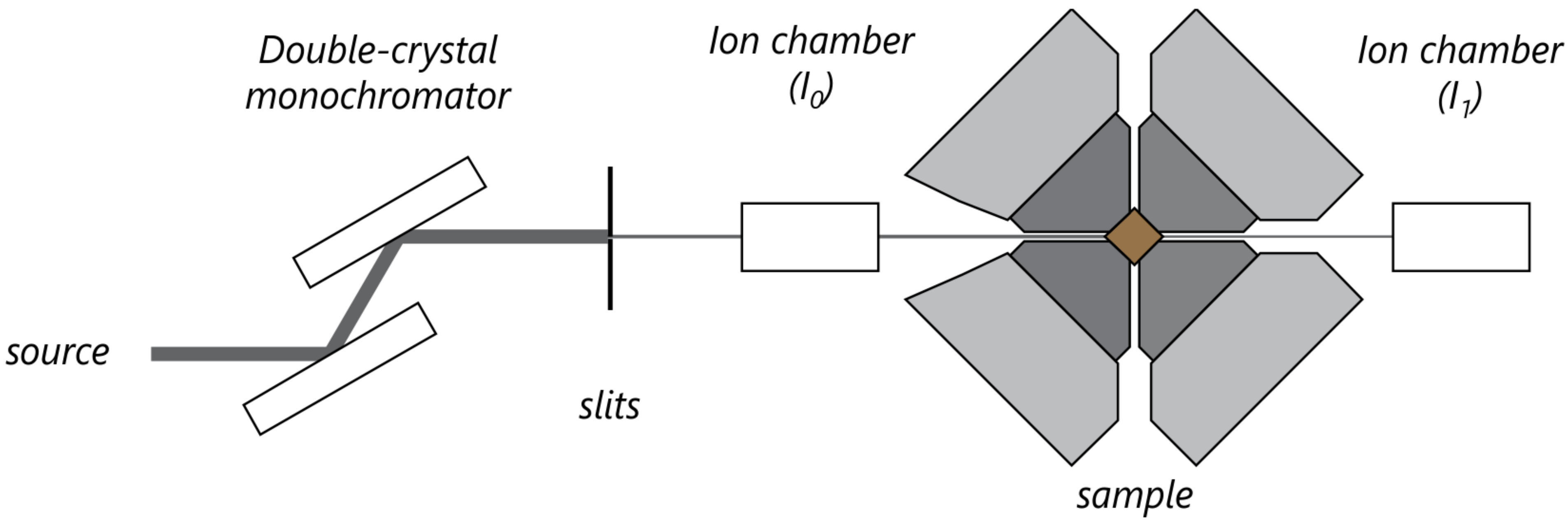
- **Proof of concept**: U and Th $L_3$-edge XANES in MORB liquid
- U-series disequilibria observed in igneous rocks
- Chemical separation of U and daughters during melting leads to disequilibrium
- With assumptions, U-series disequilibria are used to infer timescales of magmatic processes
- Assumptions include valence state of U
- Experiments suggest pressure-induced valence change of U

# Experiment schematic



# First user experiment

- **Proof of concept**: U and Th $L_3$-edge XANES in MORB liquid
- Contain silicate liquid at experimental conditions
- Control chemical potential of oxygen ($fO_2$)
- Permit sufficient beam transmission
- $\Delta\mu d$ sufficient for XANES (*i.e.* not $<0.1$)

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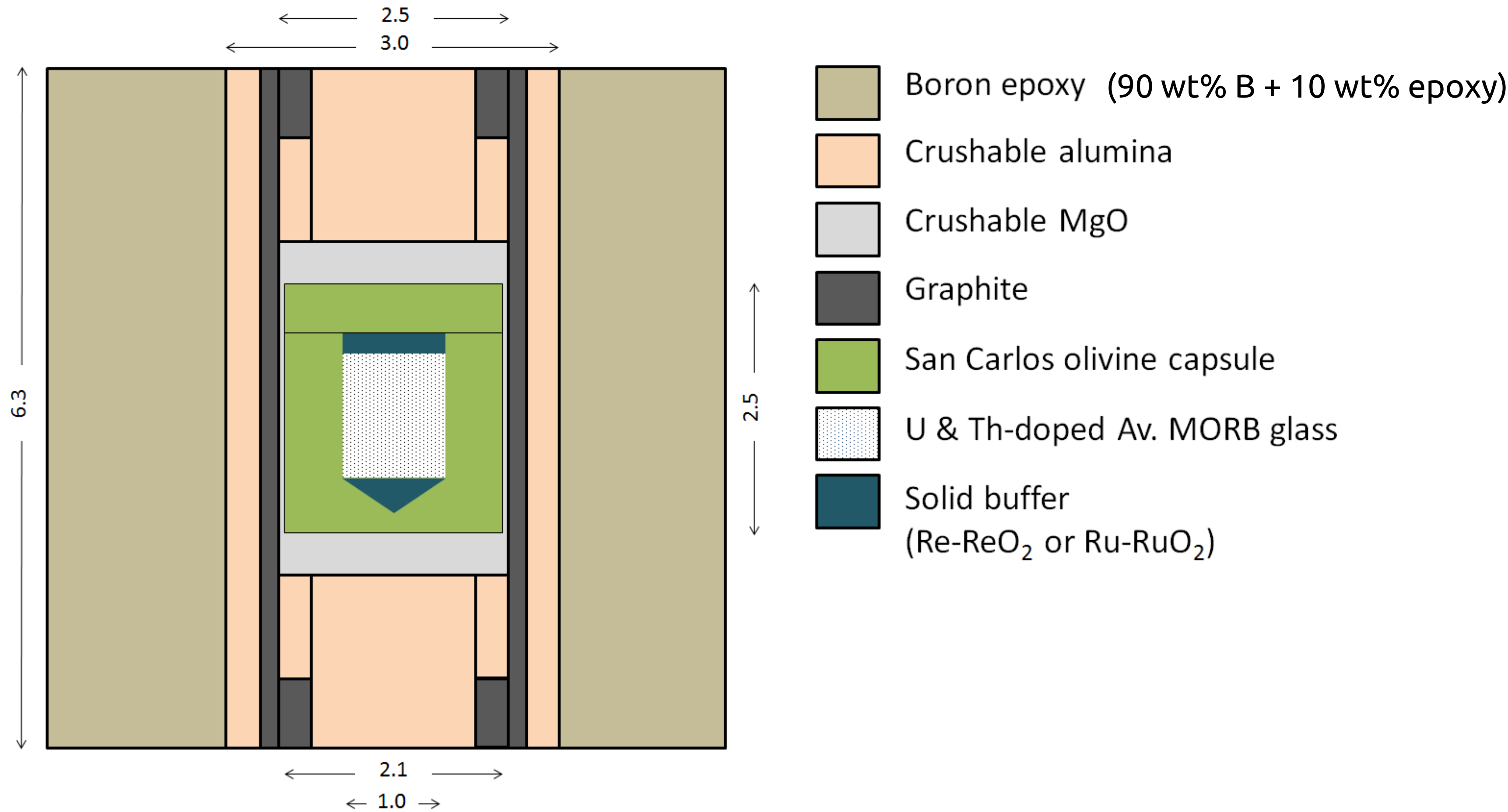

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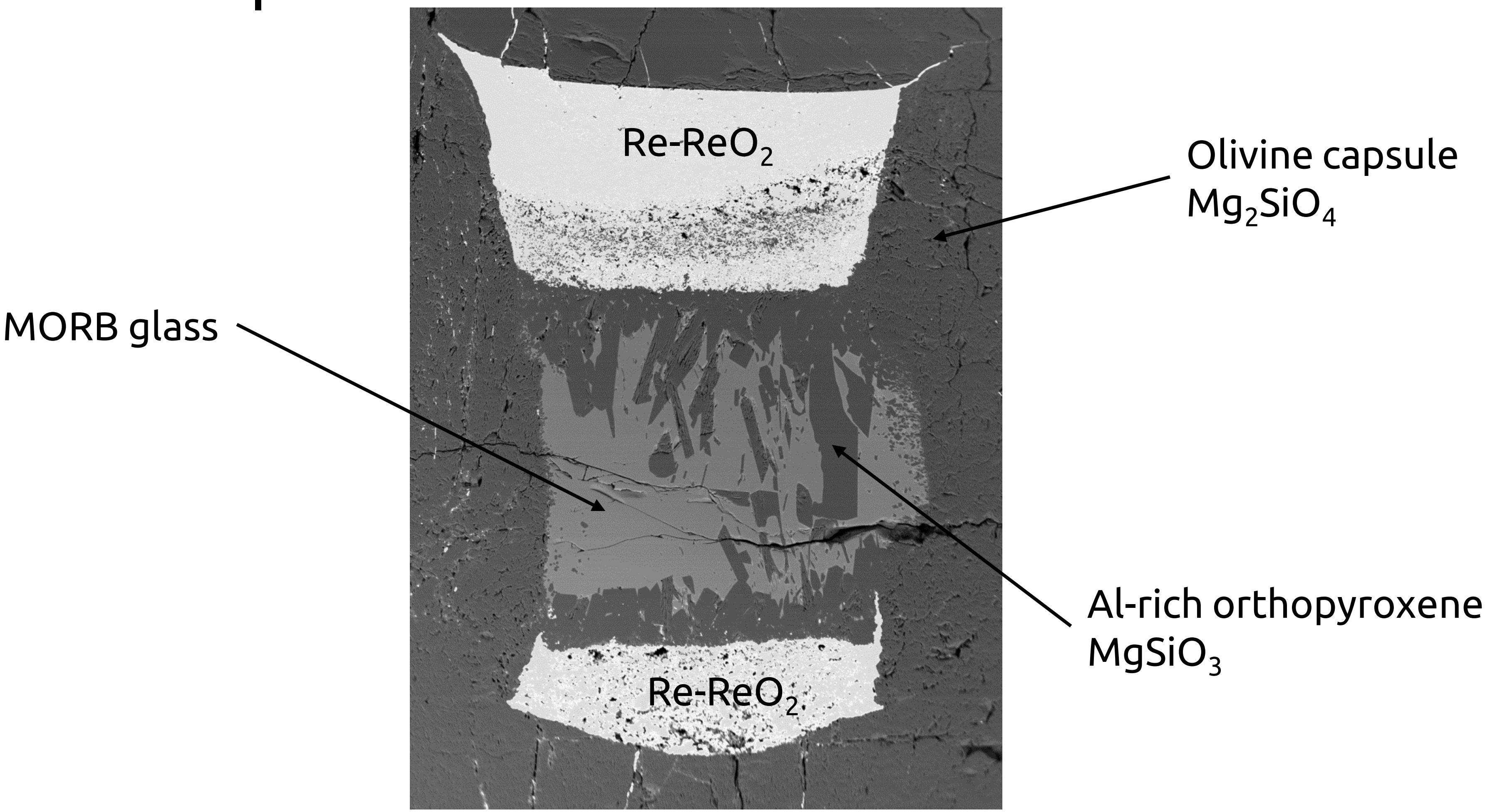

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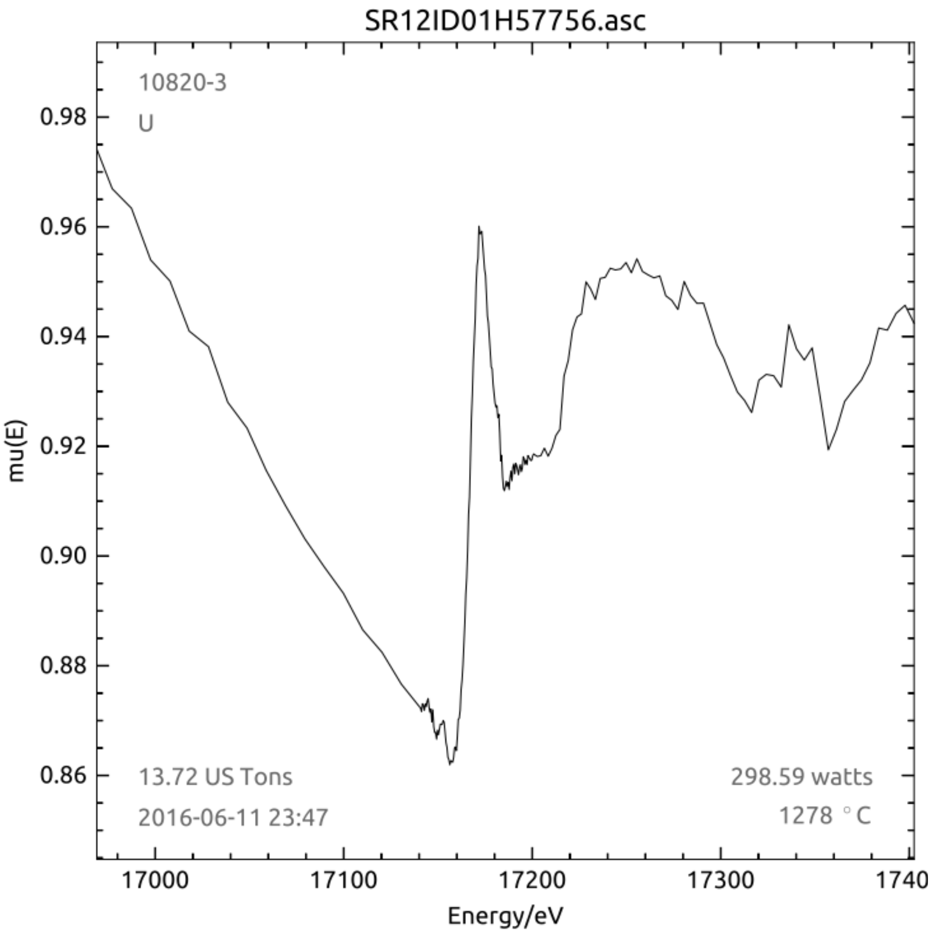


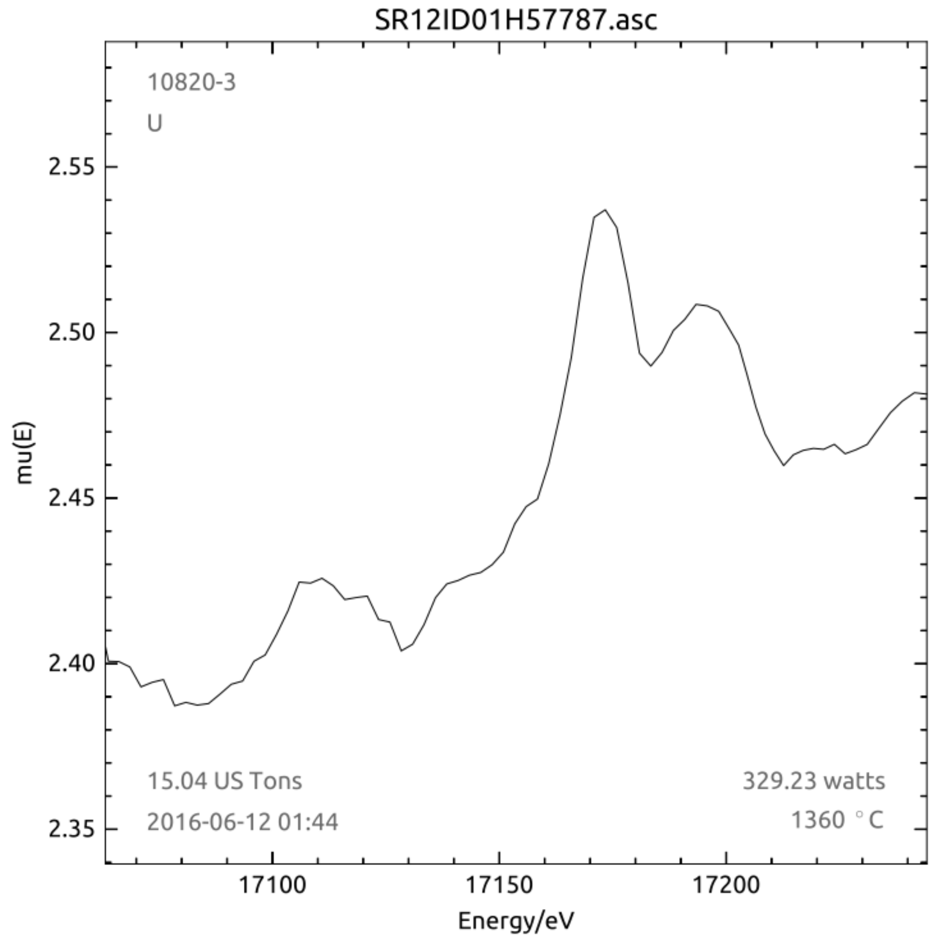

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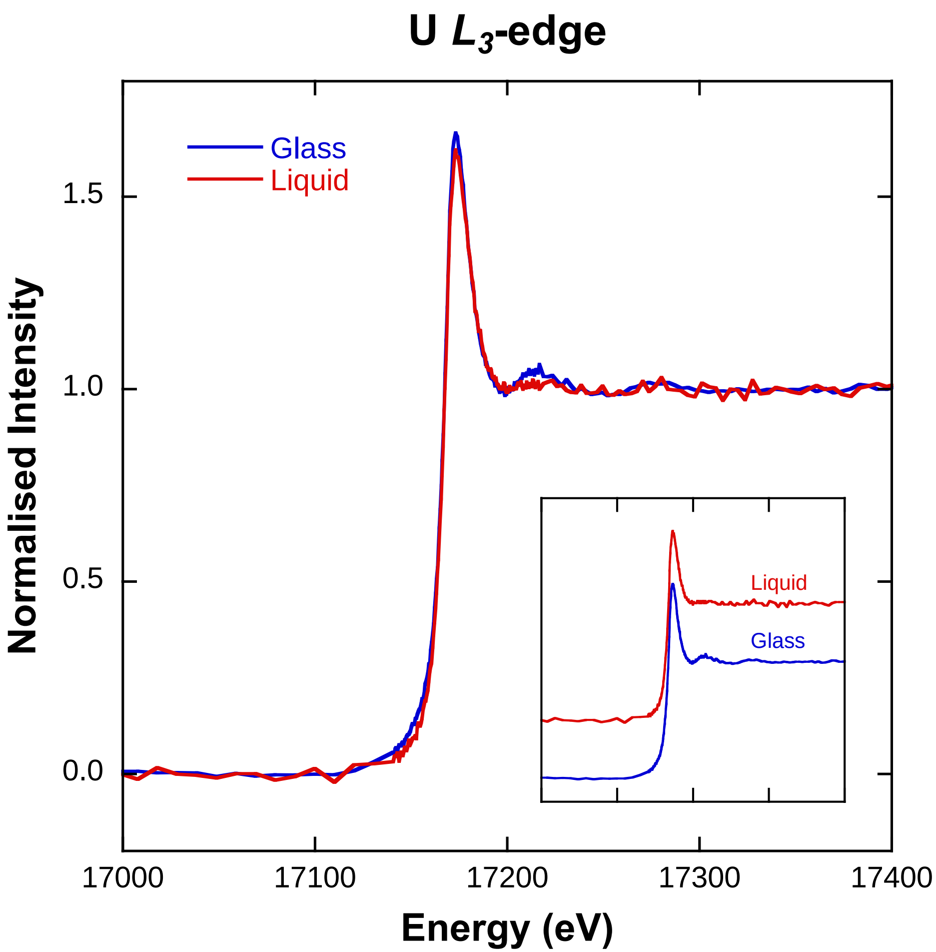


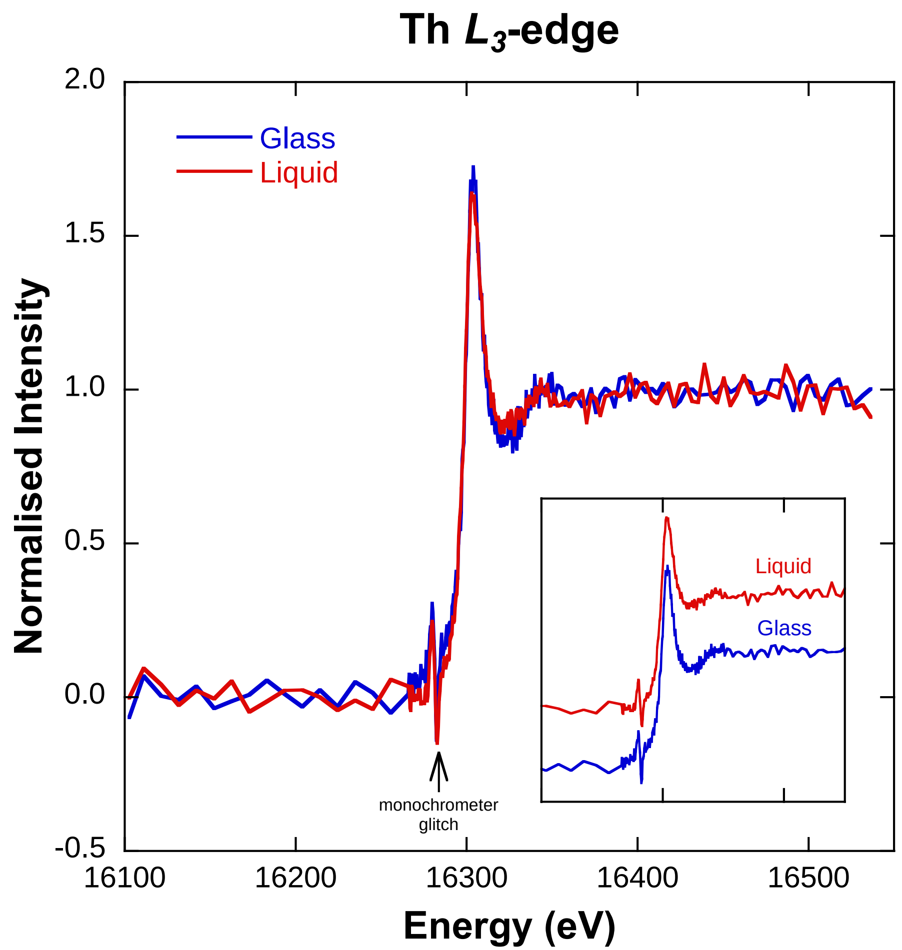

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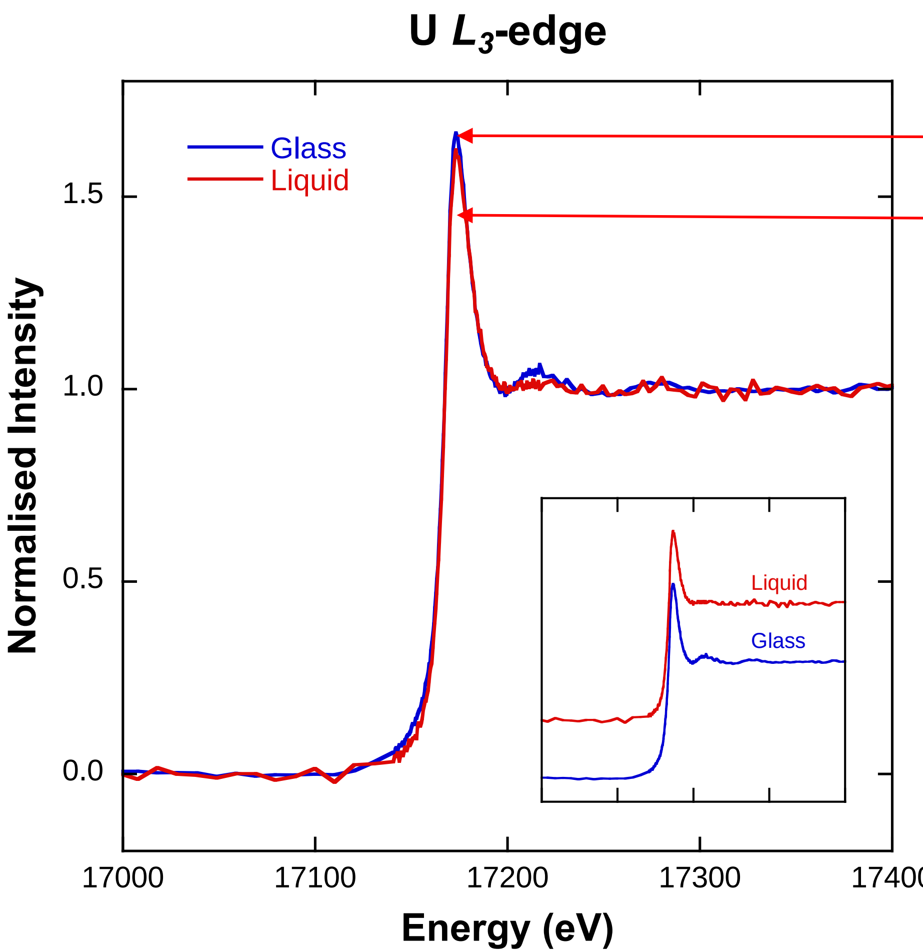
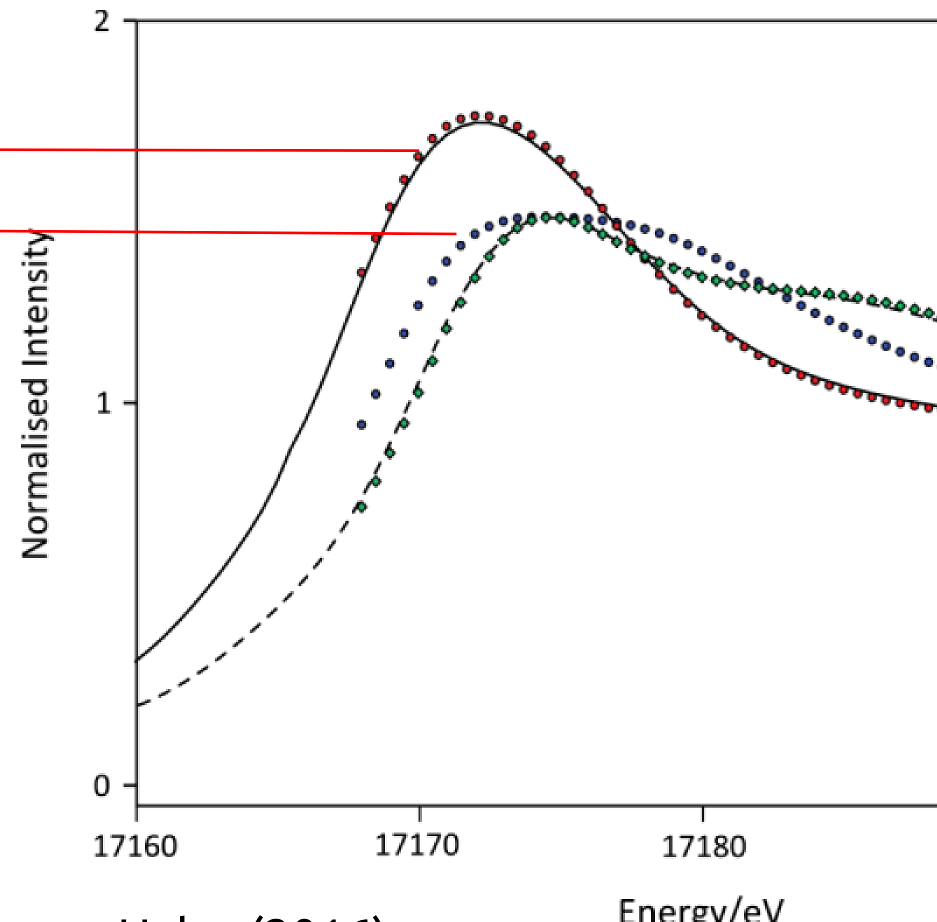
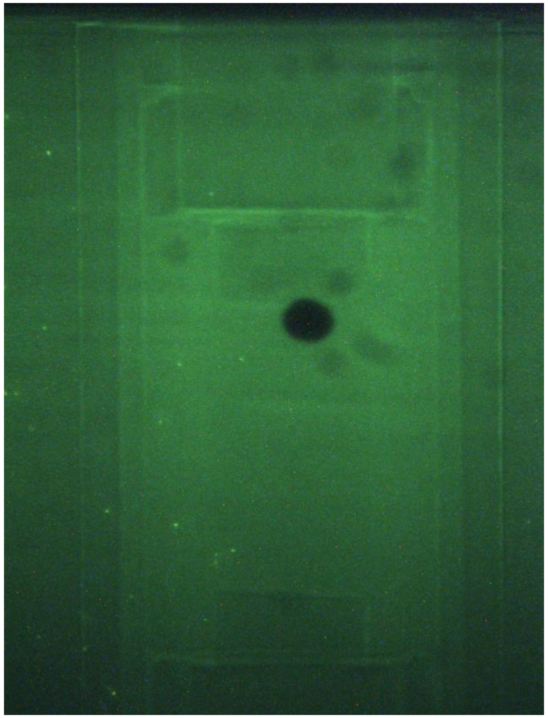



Halse (2016)

# Second user experiment

- **Proof of concept**: Falling sphere viscometry



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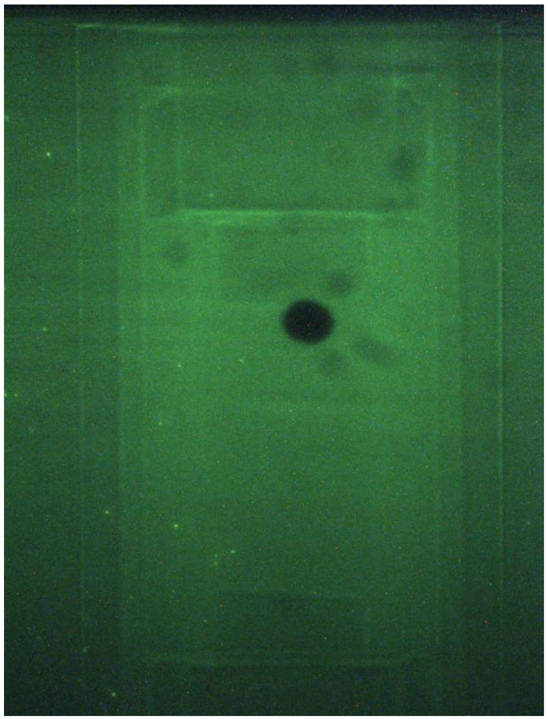
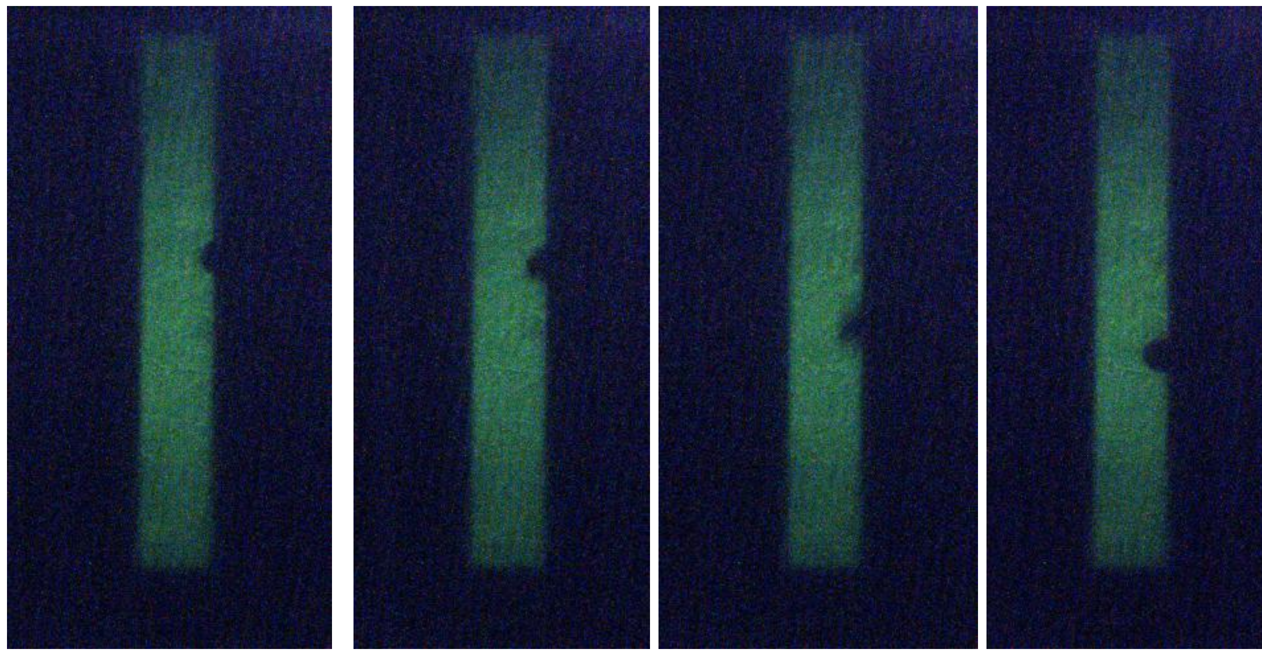
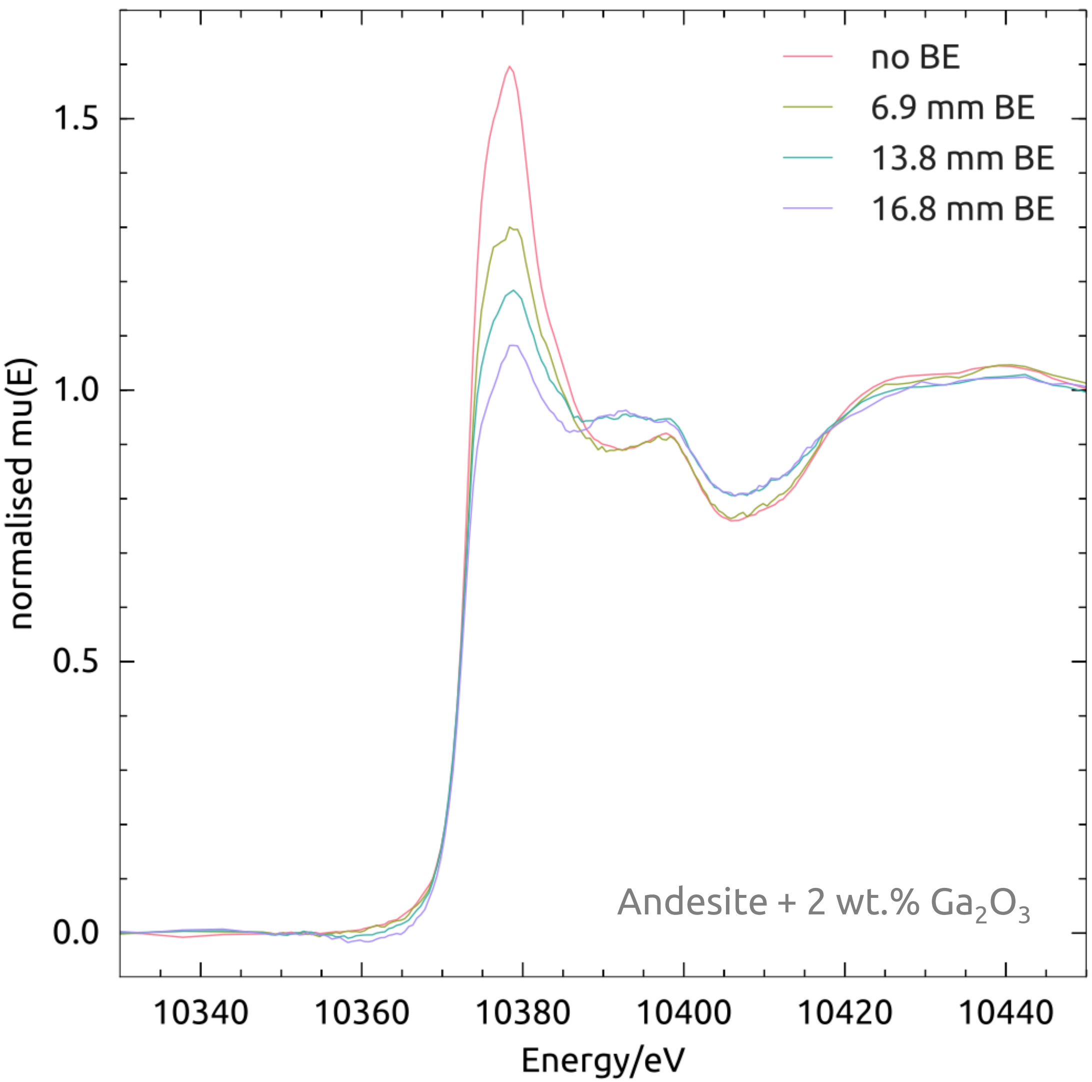




Soda-lime glass sample, ~300 µm Pt ‘sphere’, ~1.1 GPa, ~1500 °C, 38 keV incident energy

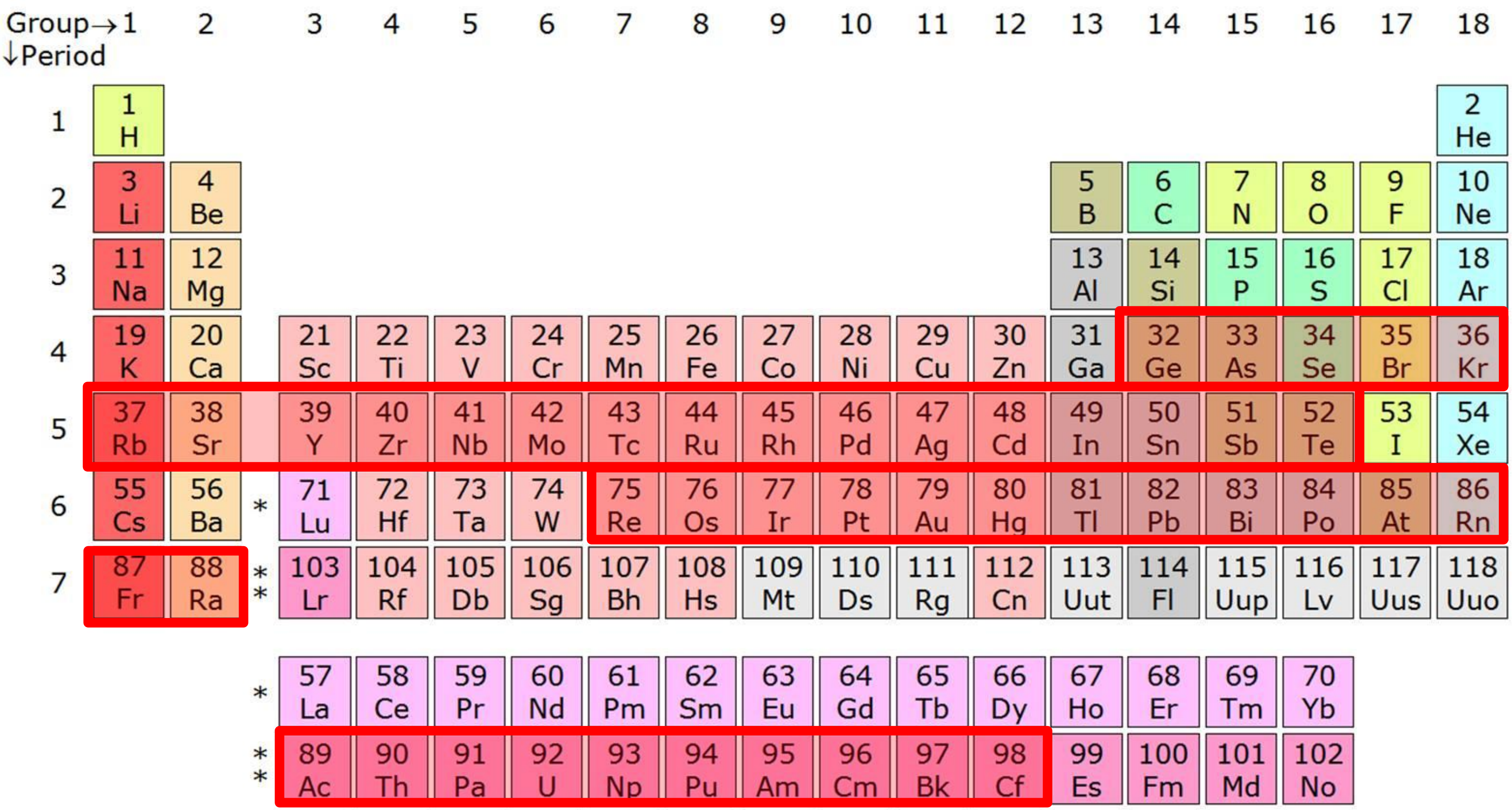
# How low can we go?

- Lowest accessible energy for XANES



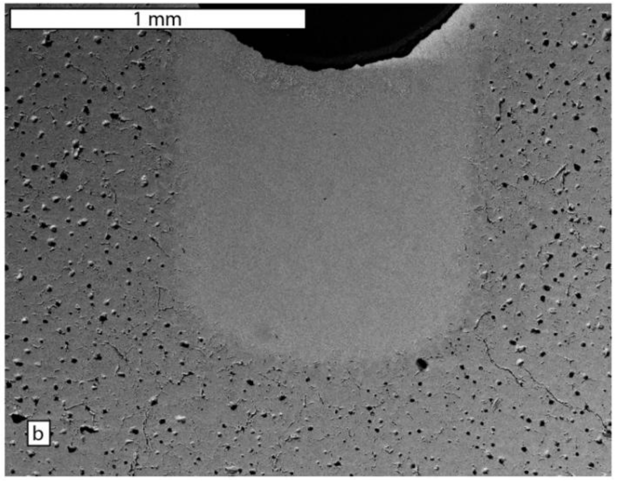
# Accessible elements for XANES

## 10 → 34 keV (with focussing mirror)



# The future

- New monochromator for XAS will permit faster XANES scans
- Improved capsule methods
    - Polycrystalline capsule
    - Hot-isostatic pressing
- New assembly design
    - More robust heaters
- Improvements to hydraulics



Nash, Smythe & Wood (2016)

# Other techniques at XAS

- *In situ* density measurements

*American Mineralogist, Volume 95, pages 144–147, 2010*

**Density of dry peridotite magma at high pressure using an X-ray absorption method**

TATSUYA SAKAMAKI,[1,*] EIJI OHTANI,[1] SATORU URAKAWA,[2] AKIO SUZUKI,[1]
AND YOSHINORI KATAYAMA[3]

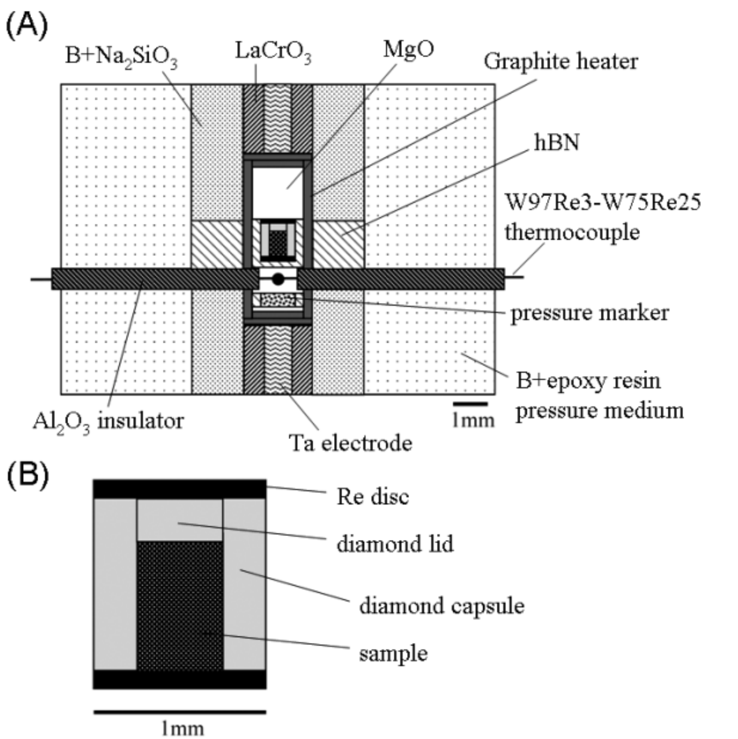



**FIGURE 2.** (**a**) Cell assembly used in the experiments. (**b**) A schematic diagram of the sample assembly.

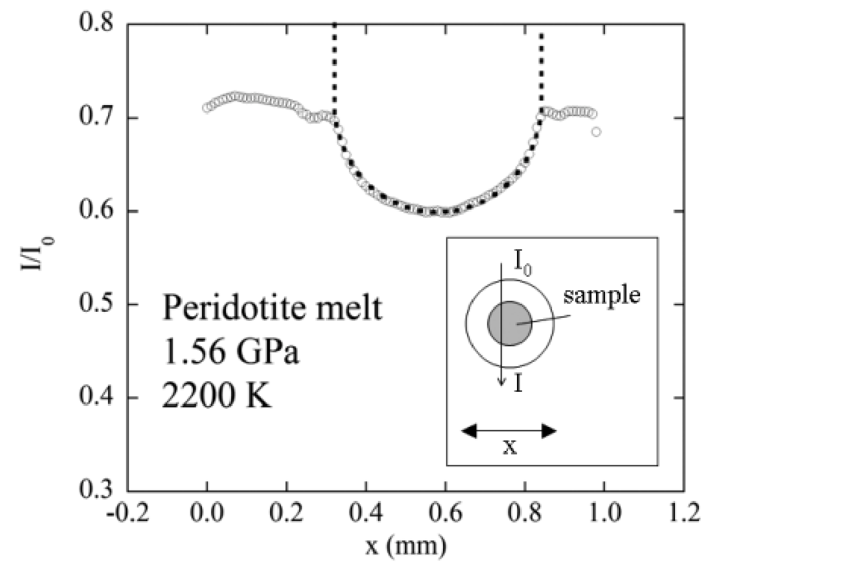



**FIGURE 3.** X-ray absorption of a peridotite melt at 1.56 GPa and 2200 K as a function of the position of the sample. The inset shows the configuration of the X-ray optics used for the absorption measurements. Key: $I_0$ = intensity before absorption; $I$ = intensity after absorption; and $x$ = the position of the sample.

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- *In situ* density measurements

Earth and Planetary Science Letters 287 (2009) 293–297

Measurement of hydrous peridotite magma density at high pressure using the X-ray absorption method

Tatsuya Sakamaki [a,*], Eiji Ohtani [a], Satoru Urakawa [b], Akio Suzuki [a], Yoshinori Katayama [c]

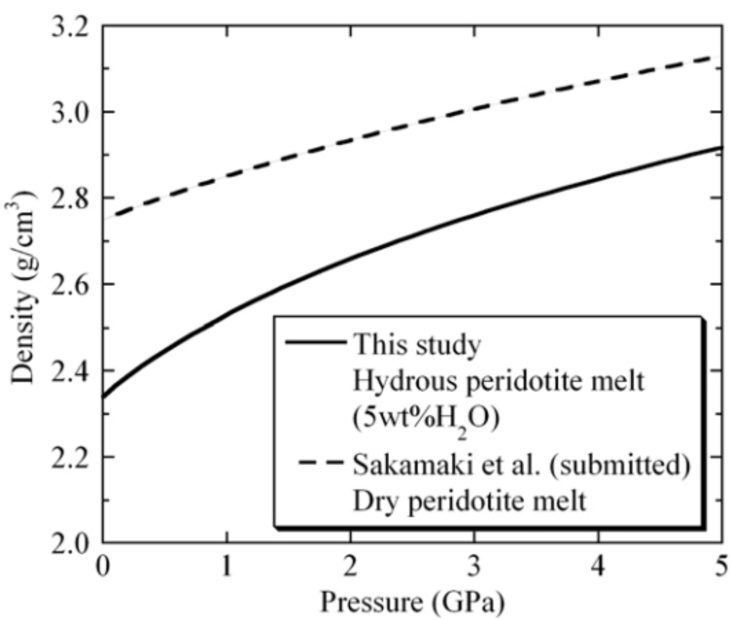



**Fig. 5.** Isothermal compression curves of dry and hydrous peridotite melts at 1973 K.

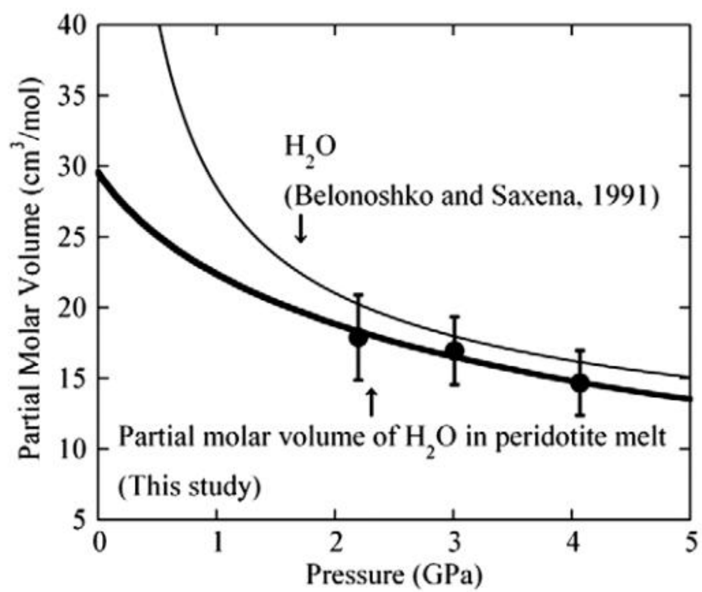
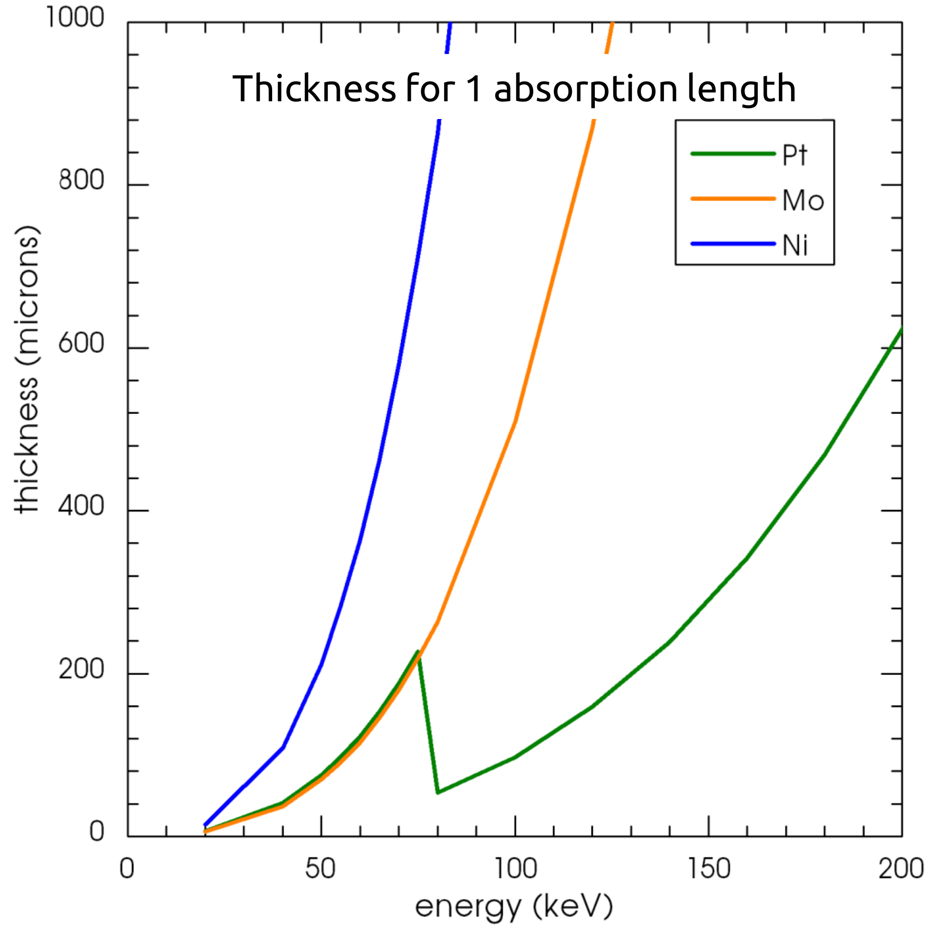


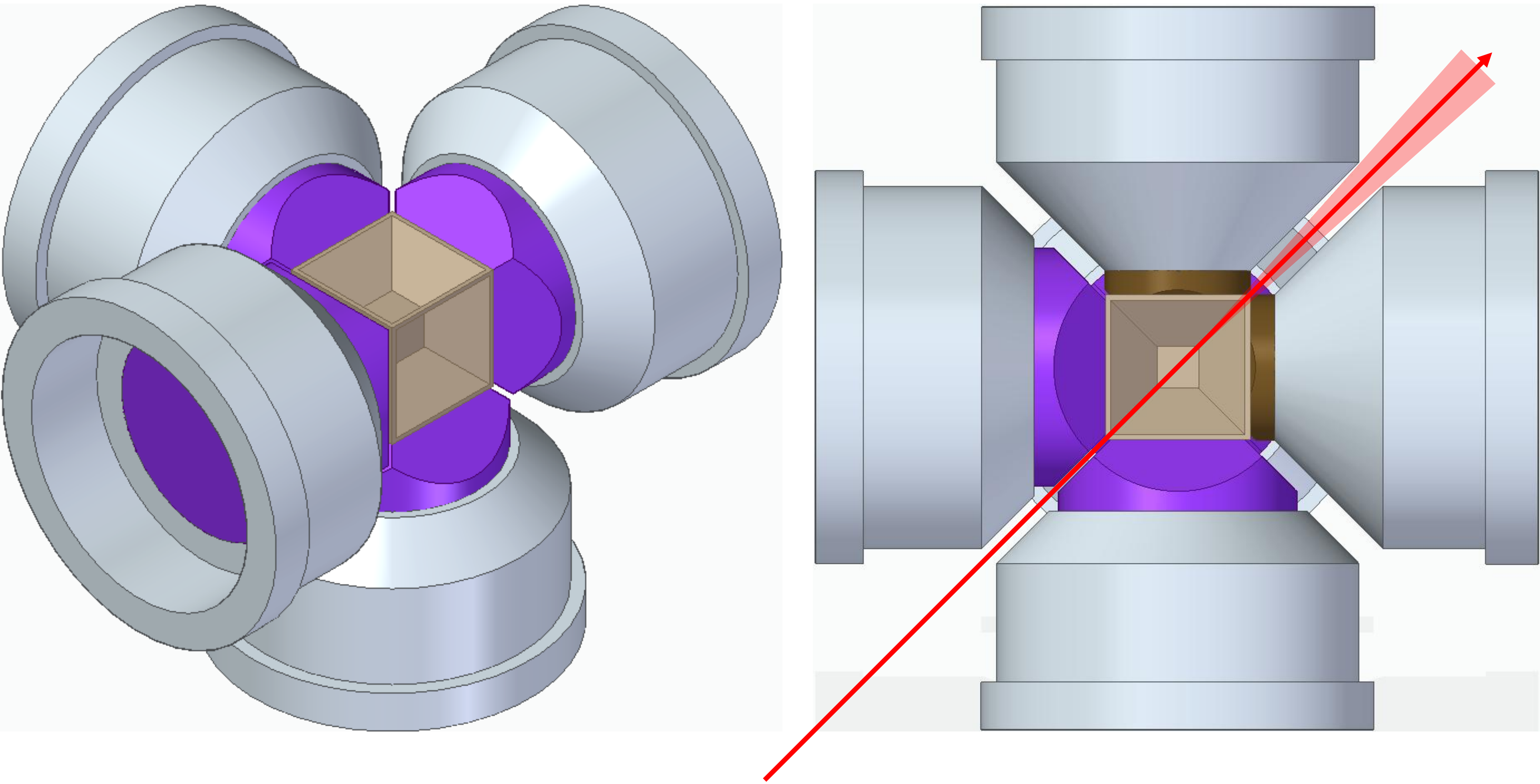

**Fig. 6.** Compression curves of the partial molar volume of $H_2O$ in magma and $H_2O$ at 1973 K.

# The future

- Install the D-DIA apparatus on IMBL
  - Imaging
  - *In situ* diffraction
  - Deformation




1 absorption length = length for 36.8% absorption



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