CDT-CMP

Hot Science Under Pressure

Lawrence Gammond

Department of Physics, University of Bath, Bath, BA2 7AY, UK

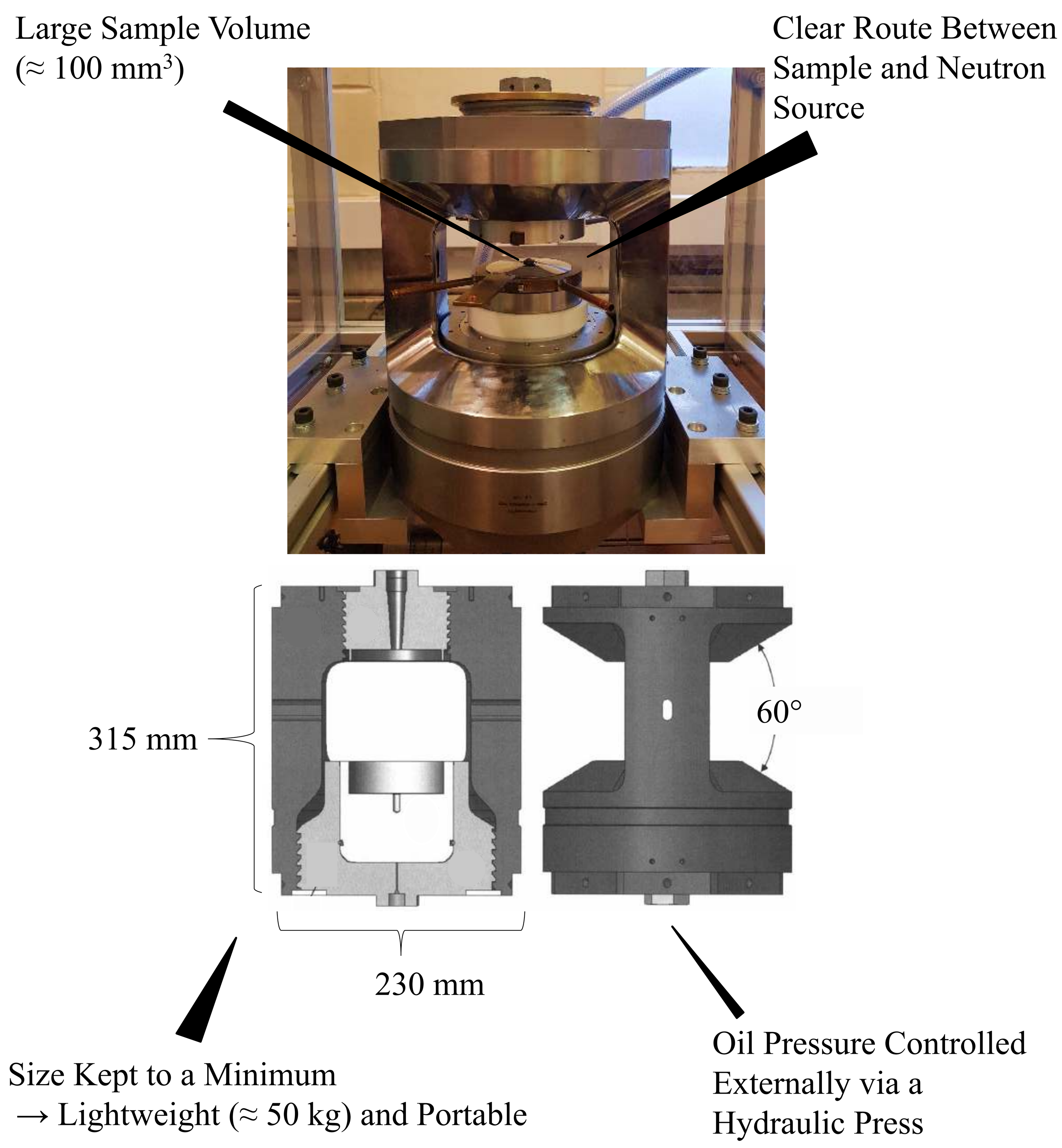
UNIVERSITY OF BATH

Abstract

Exposure of a material to extreme high pressure and temperature conditions will have an effect on the material's atomic structure, and hence its physical and chemical properties. To understand the structural transformations that may occur, it is necessary to perform *in situ* diffraction experiments, where neutron diffraction offers complementary information to x-ray diffraction. To eliminate the contribution to a diffraction pattern from a pressure/temperature calibrant, it is necessary to produce calibration curves. This poster outlines current methods of achieving high pressures and high temperatures for neutron diffraction, and gives the results from initial calibration experiments performed at the University of Bath.

The Paris-Edinburgh Press: Designed for Neutron Scattering

Shown below are pictures of the VX4 Paris-Edinburgh press. Key features that make the Paris-Edinburgh press ideal for neutron diffraction experiments are highlighted [1,2].



Anvils

Interchangeable anvils allows the user to tailor the press to their experimental requirements. Shown below are examples of commonly used anvil profiles [3].

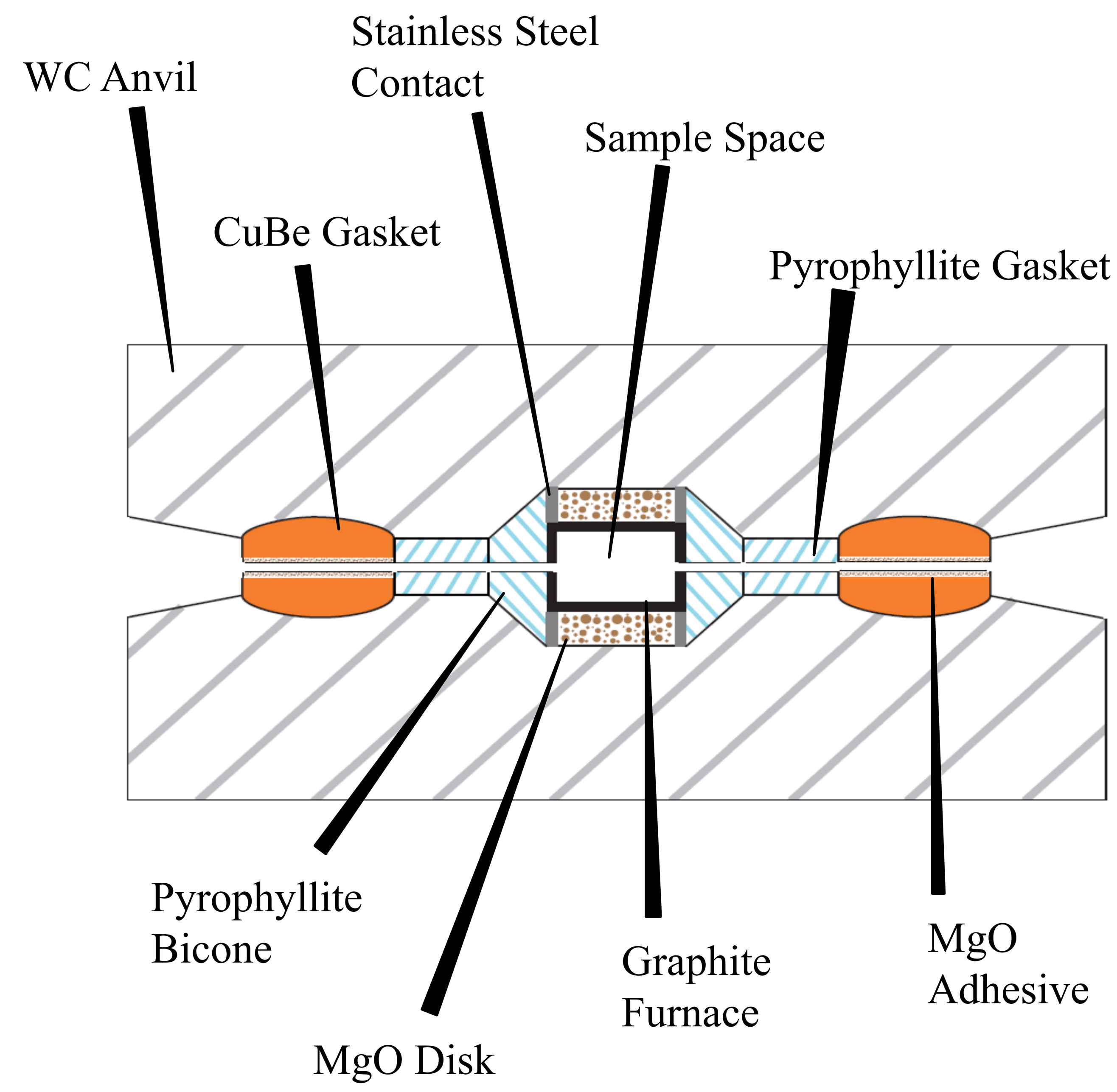
Single Toroid
- Routine experiments performed to 10 GPa for WC anvils
- Can be used to encapsulate liquid samples

Double Toroid
- Routine experiments performed to 14 GPa for WC anvils and 20 GPa for Sintered Diamond anvils
- Smaller sample volume
- Can be used to encapsulate liquid samples

Pseudo-Conoidal
- Adapted for internal heater
- Routine experiments performed to 7 GPa for WC anvils
- Minimises background scattering from gaskets

High Temperature

Sample temperatures up to 1700 °C can be achieved using an internal heating system [4]. The high pressure-high temperature set-up being developed at the University of Bath is based on the design of Klotz et al. [5]. A cross section of the set-up is shown below. Current is supplied to the anvils and runs through the graphite heater, leading to resistive heating. For electrically conductive samples, an MgO sample chamber can be used to isolate the sample from the furnace.

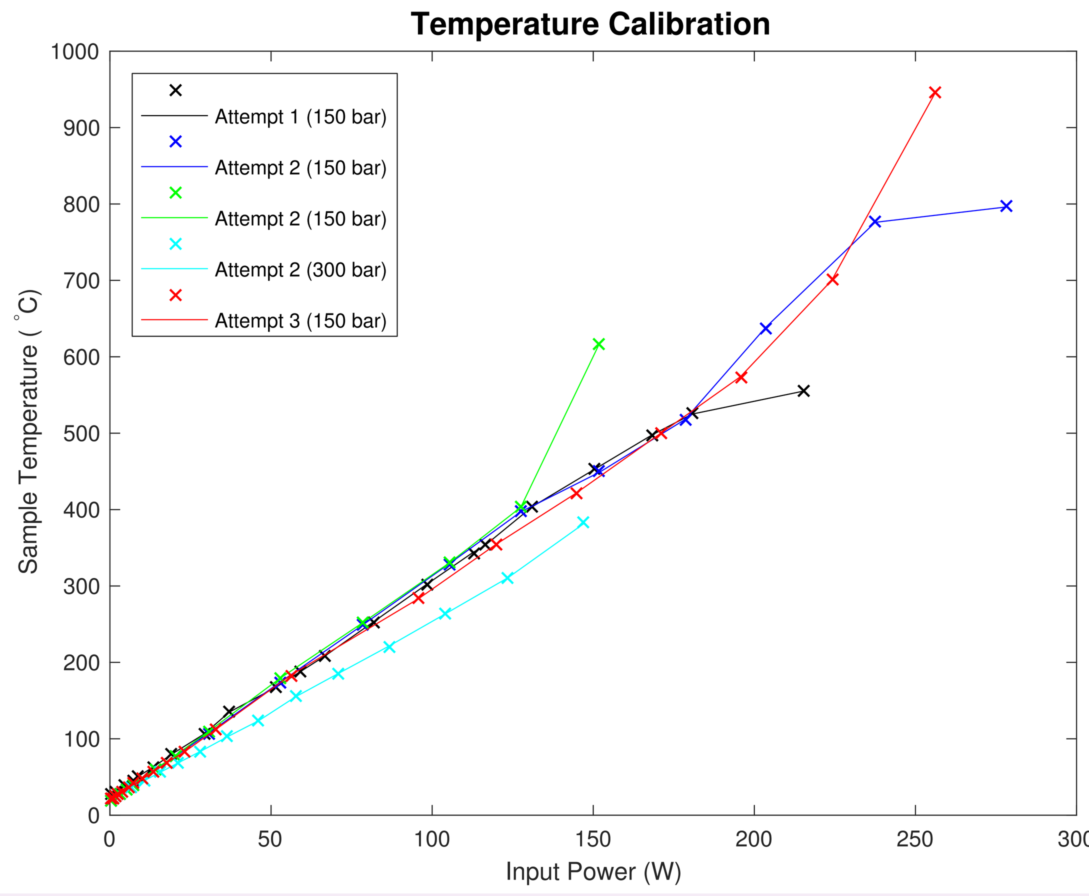


Calibration

In a neutron scattering experiments it is possible to determine the pressure and temperature at the sample position by including a calibrant. However, when neutrons are inaccessible, or when data contamination must be minimized, it is necessary to estimate the pressure and temperature by using pre-determined calibration curves.

Drilling through the gaskets of the internal heating set-up (above) allows electrodes and thermocouples to be inserted into the sample space. By measuring the pressure (using e.g. the resistance of manganin) for a given oil pressure, and the temperature for a given input power, calibration curves can be produced.

Initial temperature calibrations (right) have shown excellent agreement up to ≈500 °C at a load of 150 bar.



Future Work

- Pressure calibrations using the pressure dependent resistance of manganin and the phase transitions of bismuth are currently underway. Once the temperature and pressure calibration methods have been refined, the results will be used to map the oil pressure and input power to the sample pressure and temperature, respectively.
- New furnace materials are being tested to access higher temperatures.

References

[1] J. M. Besson et al., "*Neutron Powder Diffraction Above 10 GPa*", Physica B **180** 907-910 (1992)
[2] S. Klotz et al., "*A New Type of Compact Large-Capacity Press for Neutron and X-ray Scattering*", High Pres. Res., **24**, 1, 219-223 (2004)
[3] S. Klotz, "*Techniques in High Neutron Scattering*", U.S., Taylor and Francis (2013)
[4] Y. Le Godec et al., "*Neutron Diffraction at Simultaneous High Temperatures and Pressures, with Measurement of Temperature by Neutron Radiography*", Min. Mag., **65**, 6, 737-748 (2001)
[5] S. Klotz et al., "*The α-γ-ε Triple Point of Iron Investigated by High Pressure-High Temperature Neutron Scattering*", App. Phys. Lett., **93**, 91904, (2008)

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