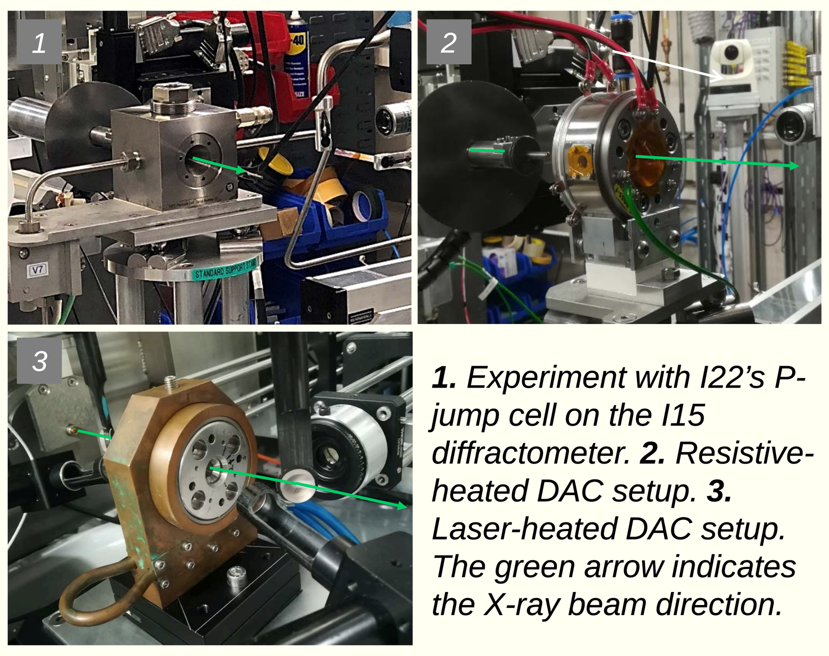
High-Pressure Research Opportunities at Diamond Light Source

Eight beamlines at Diamond Light Source have been designed with high-pressure (P) and variable temperature (T) experiments in mind. Each beamline is optimised for a specific X-ray technique and the P-T ranges covered vary. Here we outline the characteristics of each beamline and present representative research examples.

I15 Extreme Conditions

I15 is dedicated to the study of materials at high-pressures and variable temperature. A double crystal monochromator provides X-rays from 20 to 80 keV for the two endstations. The diffractometer is a multi-purpose station. With beam sizes of 20-70 µm in diameter it serves diamond-anvil cell (DAC) as well as larger volume press experiments. The micro-focus station with a beam size of 5 x 9 µm^2 is optimised for laser-heated DAC experiments.



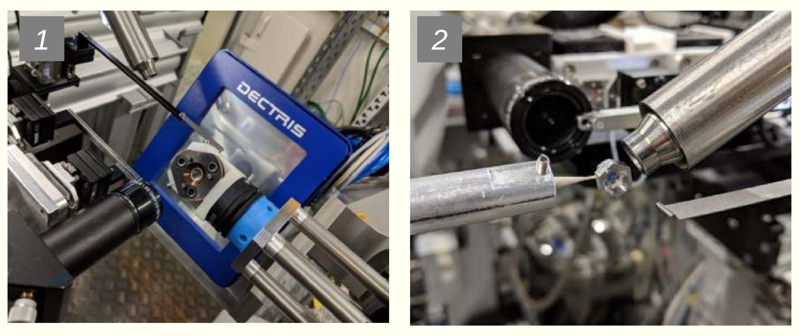
1. Experiment with I22's P-jump cell on the I15 diffractometer. 2. Resistive-heated DAC setup. 3. Laser-heated DAC setup. The green arrow indicates the X-ray beam direction.

Techniques: Powder Diffraction, XPDF

Online capabilities: DACs resistive- and laser-heated, DACs in cryostat, I22's P-jump cell, ruby fluorescence, P controller for DACs, Raman spectroscopy

I19 – Small Molecule Single-Crystal Diffraction

I19 is dedicated to small-molecule single-crystal diffraction. The beamline's high photon flux and low beam divergence allows studying crystal structures of samples that are, due to either their size or complexity, too weakly scattering to be successfully studied with other X-ray sources. The I19 Newport diffractometer in experimental hutch 2, has a 25 kg load capacity at the sample position allowing a variety of high-P devices to be mounted. Equipped with a Dectris Pilatus detector, the setup permits rapid data collection and is ideally suited to high-P single crystal studies with a relatively high throughput.



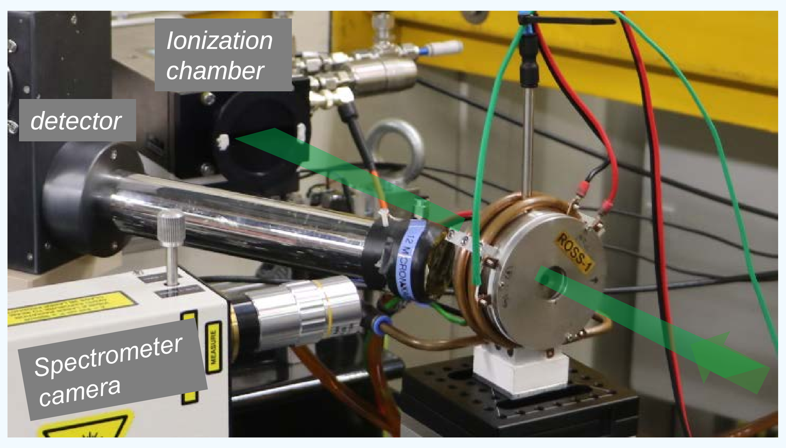
1. Experiment with a Merrill Bassett DAC that is typically used in room-T studies up to 12 GPa.
2. Setup with a turnbuckle cell. This DAC is small enough to be used with the cryostream. The P-T range covered is T=100-500 K and P up to 6 GPa.

Technique: Single-Crystal Diffraction

Online capabilities: Sapphire liquid pressure cell (0-2000 bar, 1-bar P control), DACs, cryostream for DACs, ruby fluorescence

I20 – Versatile X-ray Spectroscopy

I20 is dedicated to X-ray absorption spectroscopy (XAS). It consists of two branches, I20-scanning and I20-EDE, that operate simultaneously and independently. The I20-EDE branch allows, thanks to the dispersive optics, to measure the full absorption spectra in a single shot in less then 1ms. The high time resolution, high flux from the wiggler source, small focal spot and high beam stability make the beamline suitable for XAS measurements at high P-T. Experiments at the Mn, Cu, Zn, Ga and Ge k-edges with a DAC have been performed in transmission mode. Dilute samples can also be measured in fluorescence mode (turbo-XAS).



Turbo-XAS setup on I20-EDE with a hydrothermal DAC from I15 that enables measurements up to 1300 K and 5 GPa. The X-ray path is indicated in green.

Techniques: XAS, EXAFS

Online capabilities: DACs, P controller for DACs, ruby fluorescence, Raman spectroscopy

I18 – Microfocus Spectroscopy

I18 provides an energy-tuneable (2-20.7 keV) X-ray beam with beam sizes down to 2 µm x 2.5 µm. The beamline is optimized to perform X-ray fluorescence (XRF) studies, X-ray absorption spectroscopy (XAS) and X-ray diffraction. I18 can install a range of sample environments including diamond-anvil cells. Please contact I18 staff to discuss possibilities.

Example publication: N.R.C. Corsini et al., *Pressure-induced amorphization and a new high density amorphous metallic phase in matrix-free Ge nanoparticles*, Nano Lett 15(11), 2015. doi 10.1021/acs.nanolett.5b02627

Techniques: XRF, XAS, Powder Diffraction

Online capabilities: DACs

I22 – Small Angle Scattering & Diffraction

I22 offers pressure experiments in two P ranges: up to 5 kbar with the P-jump cell and in the GPa range, up to 60 GPa, with a diamond-anvil cell (DAC). The P-jump cell was developed in collaboration between I22 and Imperial College London. Samples are sealed inside plastic capillaries and water is used to generate P from 1 to 5000 bar and T from 0 to 120 ºC. The system allows static as well as P jumps. P changes take as little as 5 ms and are completely automated.

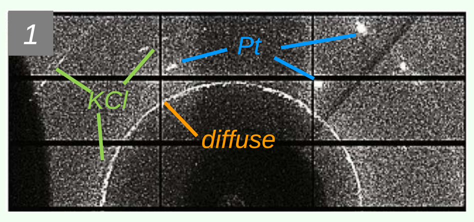
Latest related publication: F. Lehmkühler et al., *Kinetics of pressure-induced nanocrystal superlattice formation*, Phys Chem Chem Phys 47, 2019. doi 10.1039/C9CP04658E

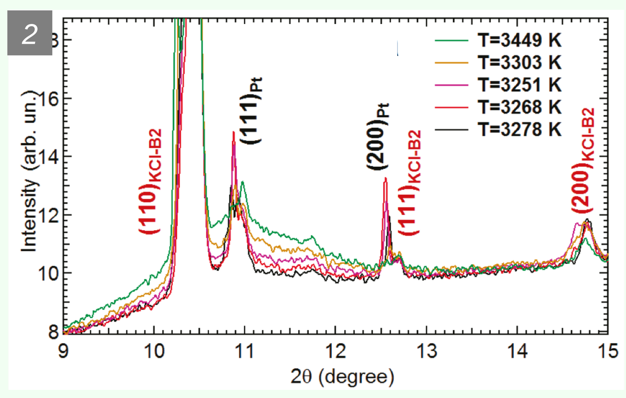
Techniques: SAXS, WAXS, GISAXS

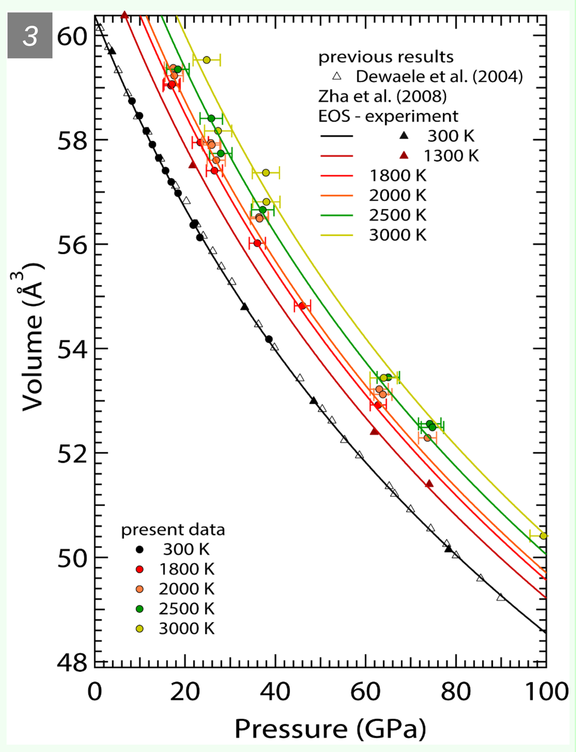
Online capabilities: P-jump cell, DACs

Case Study 1 – *In-situ* Characterization of the High- Pressure High-Temperature Melting Curve of Platinum

In this study on I15 the melting line of platinum was characterized both experimentally, using synchrotron X-ray diffraction combined with the laser-heated diamond-anvil cell, and theoretically, using *ab-initio* simulations. Platinum samples were investigated in the range 10–110 GPa and 300–4800 K. Over the investigated P-T range only the face-centred cubic phase of platinum was observed. The experimentally and theoretically obtained melting points are in good agreement.







1. 2-dimensional diffraction pattern of Pt foil embedded in KCl pressure-transmitted medium measured at ~27 GPa and 3200 K. The combination of single crystal spots and diffuse signal from the liquid sample shows the axial T gradient in the sample.

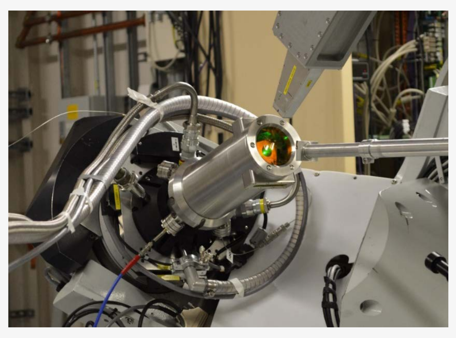
2. Integrated diffraction patterns of Pt foil at 27 GPa at five different temperatures, before and after the onset of the solid-liquid phase transition.

3.Thermal Equation of State of Pt (lines) as reported in Zha et al.(J App Phys 2008) is compared with experimental data (symbols) obtained in the present study and in Dewaele et al. (PRB 2004). The temperature colour code is the same for all the reported studies.

In situ characterization of the high pressure - high temperature melting curve of platinum. Scientific Reports 9, 2019. S Anzellini, V. Monteseguro, E. Bandiello, A. Dewaele, L. Burakovsky, D. Errandonea, 10.1038/s41598-019-49676-y

I16 Materials & Magnetism

I16 has recently developed methodology and tools to measure single-crystal diffraction under pressure at low temperature. Two systems are available, one is based on a miniaturized turnbuckle cell that can be cooled using a LN_2 cryostream. The other prototype is a gas membrane driven DAC that can be used in reflection. It fits the 4K ARS cryocooler. High-pressure experiments are not available through the standard user programme. Please contact beamline staff to discuss possibilities.



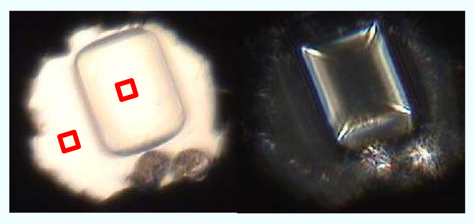
High-pressure low-temperature DAC setup on I16. Data is collected with an in-vacuum Pilatus 100K detector. The X-ray beam direction is from right to left.

Technique: Single-Crystal Diffraction

Online capabilities: DACs, P controller for DACs, cryostream for DACs

B22 - Multimode InfraRed Imaging & Microspectroscopy

B22 provides high brilliance near-IR to far-IR/THz (1.2 eV - 0.6 meV) radiation, enabling high quality spectroscopy of diamond-anvil cell samples with spatial resolution or sample size down to the diffraction limit (3-15 µm in the mid-IR). As well as molecular and lattice vibrational dynamics, electronic band-structure in the 1 eV - 0.6 meV range can be probed to track e.g. pressure induced metal-insulator transitions. IR reflectivity allows determination of the optical dielectric function of the sample.



Xenon Hydride Van der Waals Compound in an IR DAC on B22. Red squares are the sampling areas of 10x10 µm^2.

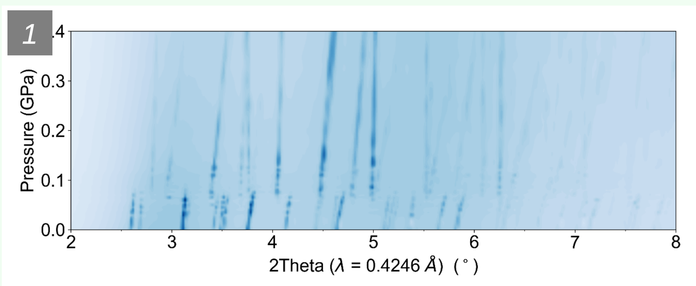
Visible Brightfield Darkfield

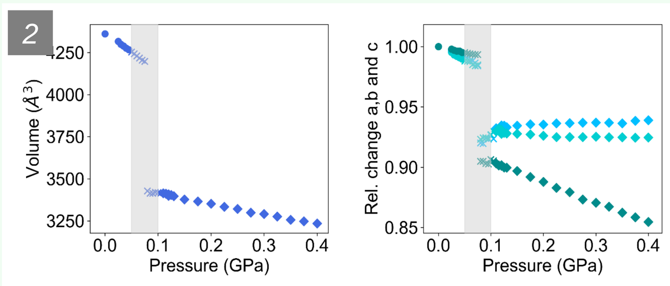
Techniques: IR and THz Micro-spectroscopy & Imaging

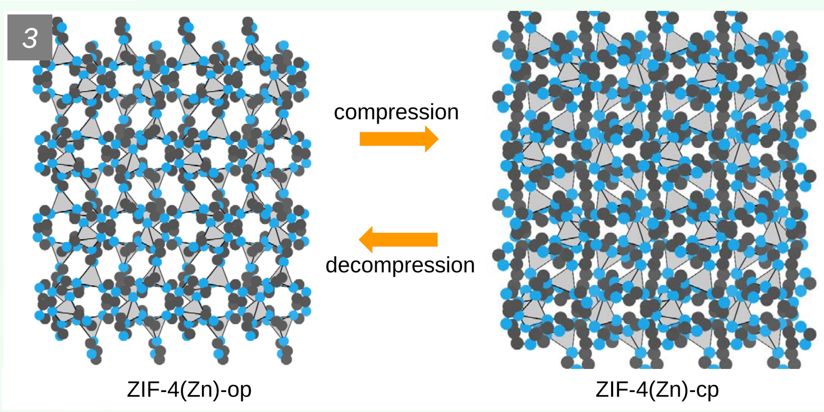
Online capabilities: DACs

Case Study 2 – The Zeolitic Imidazolate Framework ZIF-4 under Low Hydrostatic Pressures

The zeolitic imidazolate framework ZIF-4 exhibits large structural flexibility as a response to hydrostatic pressures, going from an open pore phase (ZIF-4(Zn)-op) to a closed pore phase (ZIF-4(Zn)-cp). A powder X-ray diffraction setup on I15 with the P-jump cell from I22 has allowed tracking the evolution of lattice parameters in P increments as small as Δp = 0.005 Ga (50 bar) from ambient to 0.4 GPa. A reversible phase transition from open to closed pore phase is observed. ZIF-4(Zn) was found to have a bulk modulus of K(ZIF-4(Zn)-op) = 2.01±0.05 GPa and K(ZIF-4(Zn)-cp) = 4.39±0.20 GPa.







1. Contour plot of high-pressure powder patterns. A phase transition is clearly visible at 0.075 GPa.

2. Volume and lattice parameters as a function of P as extracted from Pawley profile fits. Onset of the phase transition is observed at 0.05 GPa. Circles = ZIF-4(Zn)-op, diamonds = ZIF-4(Zn)-cp and crosses = biphasic fit was performed. Colour code: blue = V, light blue = a, light green = b and dark green = c.

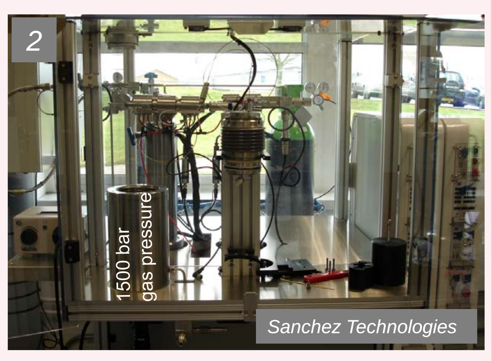
3. Low-pressure structure ZIF-4(Zn)-op and high-pressure structure ZIF-4(Zn)-cp looking down the a axis of the unit cell. Colour code: grey thetrahedra = ZnN_4 units, blue = nitrogen, grey = carbon.

The zeolitic imidazolate framework ZIF-4 under low hydrostatic pressures. Zeitschrift für Anorganische und Allgemeine Chemie 4, 2019. P. Vervoorts, C.L. Hobday, M.G. Ehrenreich, D. Daisenberger, G. Kieslich, 10.1002/zaac.201900046

Available Equipment & Support

- Membrane (m) & screw driven DACs
- Resistive heater sleeves for mDACs
- Internal resistive-heated mDACs
- Micromanipulator for loading DACs
- **Assistance for loading DACs is available.** *Please read I15's DAC Policy on the website and contact I15 staff.*
- Electrical discharge machines for drilling gaskets
- Optical microscopes
- Glovebox with microscope
- Gas loader for DACs (He, Ne, and other gases)
- Pressure controllers for mDACs
- Ruby fluorescence measurement systems
- Raman spectroscopy system

More support facilities are provided by DLS.



1. Membrane DAC from Sanchez Technologies.
2. Gas loader for DACs.

I12 - Joint Engineering, Environmental & Processing

I12 can accommodate a variety of user-owned sample environments, including Paris-Edinburgh (PE) cells. Experiment Hutch 2 has a large sample stage which can take up to 2 tonnes. If you have a PE cell, it may be possible to use it on the beamline, providing the cell is suitably designed to allow passage of incident and diffracted X-ray beams. Please contact the beamline staff to discuss your requirements.

Example publication: J. Philippe et al., *Rotating tomography Paris-Edinburgh cell: a novel portable press for micro-tomographic 4-D imaging at extreme pressure/temperature/stress conditions* , High Pressure Res 36(4), 2016. doi 10.1080/08957959.2016.1221951

Techniques: Imaging & Tomography, Powder Diffraction, Energy-Dispersive X-ray Diffraction

Online capabilities: Paris-Edinburgh cell

Are you having an idea for a high P-T experiment? Do not know who to contact? Get in touch with Annette. Email annette.kleppe@diamond.ac.uk

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