physicochemical properties through the application of pressure and temperature as a result of changes in crystal packing and distortion/ compression of coordination bonds. Measuring the structural changes of these complexes with relation to pressure and temperature allows for a greater insight into how these properties arise. Typically however it is difficult in current crystallographic studies to simultaneously alter the pressure and temperature of the experiment, due to the inherent difficulties associated with heating or cooling a diamond anvil cell, as well as difficulties in accurate and precise recording of the internal cell temperature.

Successfully merging the fields of low-temperature and high-pressure crystallography would however provide huge benefits to the study of structure-behaviour relationships. Low-temperature and high-pressure studies could be performed simultaneously or consecutively using the same crystal and experimental setup, allowing for improved modelling of complicated multi-variable phase changes. Significant improvements in data quality would be observed for high pressure studies conducted at low temperatures due to reduced thermal and vibrational motion. Secondary radiation damage to crystals is also temperature dependant thus lower temperature acquisition will prolong the lifespan of crystals under high-pressure study [1]. The desire for this combined approach has thus necessitated the design of a novel diamond anvil cell which can be successfully implemented for high-pressure low-temperature crystallographic studies.

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Keywords: high-pressure, low-temperature, X-Ray

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Novel microfocus x-ray sources for high-pressure crystallography

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Diamond anvil cells (DAC's) are widely used for examining the crystal structure of materials under high pressure. The area of reciprocal space accessible in a high-pressure X-ray diffraction experiment is primarily restricted by the geometry of the DAC. For a typical high-pressure experiment using Mo radiation, only a small fraction of all reflections can be collected. This can be as low as 30% for triclinic crystal structures. Using radiation with a shorter wavelength, such as Ag-K_α, a larger portion of the reciprocal space is accessible, thus increasing the number of observations and the resolution of the data. However, because of the low intensity of conventional Ag sealed tubes, Ag sources are rarely used for high-pressure studies in the home lab.

Microfocus sealed tube sources have proven to deliver flux densities beyond that of traditional X-ray sources when combined with 2D focusing multilayer mirrors [1, 2]. The sharp beam profile of these sources produces a high flux density at the sample position, thus leading to strong diffracted intensities. Furthermore, the small beam cross-section significantly reduces the background that usually results from scattering at the gasket of the DAC. Therefore, this type of source presents a promising alternative to classical sealed tube sources currently being used in high-pressure crystallography.

We will be reporting on the latest developments on microfocus X-ray sources (Ag and Mo anodes) which enable a clear increase in intensity compared to other sealed tube sources. Selected results on the use of these sources in high-pressure crystallography will be presented.

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Silver the new gold standard for high pressure single crystal X-ray diffraction

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The advances in X-ray focusing optics have reignited the interest in alternative home laboratory source wavelengths. Ag X-radiation has several advantages to longer wavelengths, particularly for use with diamond anvil cells (DAC's), for high pressure diffraction experiments. The compression of the reciprocal lattice, from the shorter wavelength, allows significantly more data to be collected under restricted experimental conditions. Additionally the lower absorption and hence attenuation by the diamond anvils increases the usefulness of this wavelength for these experiments.

Previous attempts to utilize Ag radiation, from sealed tube X-ray sources, have been thwarted by the low incident flux levels achievable. The new Ag IμS system [1] combats this problem by employing multilayer focusing optics which allow a high flux density to be achieved at the sample position even at low power levels (30W). The focused nature of the source also reduces unwanted interference in the X-ray diffraction pattern originating from the body of the DAC and the metal gasket forming the sample chamber.

The XIPHOS diffraction facility [2] at Durham University has been expanded with the construction of a 'sister' diffractometer coupling a 4-circle Huber goniometer with a Bruker APEXII detector and a Ag IμS source. This has allowed the exploration of a number of samples under high pressure to a greater resolution than previously accessible in the home laboratory. Of particular interest are compounds that exhibit different solid state phases which are dependent on the crystallization method employed (high pressure or low temperature).

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The XIPHOS diffraction facility for extreme sample environments

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Pushing the boundaries of experimental single crystal diffraction, particularly in the home laboratory, requires significant deviation from off-the-shelf instrumentation. The XIPHOS diffraction facility