lower mantle silicates influence the density and the elastic and seismic properties remain an open question.

Here we develop an energy-domain synchrotron radiation (SR) ^{57}Fe-Mössbauer spectrometer using the single-line Mossbauer source at beamline BL10XU of SPring-8 for enabling simultaneous X-ray diffraction (XRD) and Mössbauer spectroscopy (MS) experiments at high pressures and high/low temperatures in a diamond-anvil cell technique. The diffractometer for the energy-domain SR-MS consists of a high-resolution monochromator and a variable-frequency nuclear monochromator. The single-line Mössbauer sources with an energy width of neV can be extracted from SR using pure nuclear Bragg scattering. For MS measurements on iron-bearing materials, the incident X-rays are tuned to 14.4 keV. For XRD the energy of the incident X-ray beam from undulator source plan to be tuned the high-order harmonic of 14.4 keV. Many geological-relevant materials have no large hyperfine interactions, and exhibit the splitting collapses at high pressure. The energy-domain SR-MS method works in any operation mode for the storage ring. Analysis of MS spectra in energy domain is well established and is easier than that in time domain. Therefore, the energy-domain MS using SR would be suitable for high pressure studies of the magnetic and electronic properties of deep Earth materials, compared with nuclear resonance forward scattering of SR that is a time analogue of MS.

The combined system of XRD and MS can potentially offer the essential information for resolving outstanding issues in mineral physics. The MS method can be used with glass and fluid phase as well as crystalline materials. At present, we are at the stage where the device is started up. The device should be fully available in the second stage of 2011. In this talk, we describe this new facility and report on progress.

Keywords: mössbauer spectroscopy, nuclear bragg scattering, high pressure

MS47.P03

Acta Cryst. (2011) A**67,** C527

Setting up a home high pressure laboratory from scratch – when is it worth it?

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Although high pressure crystallography continues to expand in both scope and popularity [1], the specialized equipment and skills required to pursue such studies in an effective manner mean that it is still regarded as largely the preserve of large, well-established and highly-experienced groups. However, our experience of high pressure studies conducted at central facilities [2], where extensive expertise and support may be available, led us (and other researchers) to consider establishing high pressure facilities within our home laboratories. While these will obviously be modest in comparison to powerful central facilities, they offer the advantage of much greater access, as well as more flexibility and convenience to carry out high pressure experiments.

We have recently set up dedicated in-house high pressure facilities in Nottingham, within a crystallographic laboratory where no such facilities had previously been available [3], and we will describe our experience and the factors which had to be considered during the process. These factors include the nature and extent of the scientific program to be undertaken, grant support, other funding, accommodation and expert advice and support: their influence and interaction must be taken into account when deciding whether in-house high-pressure facilities are viable and, if they are, in designing, commissioning and installing them.

This contribution will also explore some of the different options available, taking into account local requirements and circumstances.

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Keywords: high pressure, instrumentation, techniques

MS47.P04

Acta Cryst. (2011) A**67,** C527

High-resolution single-crystal neutron diffraction to 10 GPa

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Single-crystal neutron diffraction is the technique of choice when trying to determine the nature of complex multi-site disorder and anisotropic atomic displacement parameters (ADPs). The absence of form factor effects in the neutron scattering process gives inherent advantages for measurement of disorder and ADPs and the access to three dimensional data provided by a single crystal gives greatly enhanced accuracy on directional dependence.

Now, with the development of new cell, anvil and gasket technologies, a significant breakthrough has been made, allowing us for the first time to take full structure refinement to ~10 GPa on the D9 instrument at ILL, Grenoble, and the Laue TOF instrument (SXD) at the ISIS Facility, UK.

We will describe the results from studies of squaric acid and KDP up to pressures of 10 GPa where we have been studying the proton distributions in nearly centred and centred hydrogen bonds. We will also present results from samples grown in situ at high pressure of phases of ice and ammonia water mixtures. We will describe the technical aspects of performing high-resolution single-crystal studies at high pressure both at neutron spallation and at reactor sources.

Details of analysis techniques will also be described. Finally we will also highlight developments in view to increase the range of both science and pressures that can be achieved.

Keywords: high-pressure, neutron diffraction, single-crystal, hydrogen bond

MS47.P05

Acta Cryst. (2011) A**67,** C527-C528

Low-temperature high-pressure analysis utilizing a novel pressure cell design

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Inorganic compounds can undergo significant changes in their

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

MS47.P06

Acta Cryst. (2011) A**67,** C528

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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Keywords: instrumentation, X-ray source, high-Pressure crystallography

MS47.P07

Acta Cryst. (2011) A**67,** C528

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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Keywords: experimental, pressure, phase

MS47.P08

Acta Cryst. (2011) A**67,** C528-C529

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