Contribution ID : **116** Type : **not specified**

Development of high-pressure single-crystal neutron diffraction on the Laue diffractometer, KOALA, at OPAL

Hydrogen bonds are one of the most important classes of intermolecular interaction, and accurate H-atom positions are critical for analysis of the energy terms which determine the thermodynamic stability of molecular crystals. At ambient pressure and low temperatures, H atoms can often be located by X-ray diffraction, and X-ray data can provide an accurate picture of the intermolecular contacts. High-pressure experiments do not afford this luxury. The high systematic errors introduced by the pressure cell and low completeness mean that H-atom positions are not revealed in X-ray Fourier maps. In some compounds H-atom positions can be inferred from the positions of other atoms, but this is not possible in all cases.

Neutron diffraction data are much more sensitive to H than are X-ray data, and they are essential in cases where accurate H-atom location is important. Neutron powder patterns of complex molecular systems suffer from extensive peak overlap, and single-crystal diffraction therefore has a huge advantage; there is also no need to deuterate. The main disadvantage of neutron diffraction is that a large sample is usually required, which is at odds with the decreasing volumes possible with increasing pressure with existing pressure-cell materials. Modern neutron Laue diffraction and large moissanite anvil cells offer some respite [1], but complementing high-pressure X-ray data with high-pressure neutron data is still fraught with technical challenges to obtain identical conditions.

Initial developmental experiments using a miniature diamond-anvil cell with a single crystal of size typical for X-ray diffraction on the KOALA Laue diffractometer at OPAL have shown the feasibility of the Laue technique for single-crystal neutron studies at high pressure. Remarkably, data completeness is similar to ambient-pressure measurements, despite the presence of the pressure cell. It is now possible to perform joint X-ray and neutron studies on the same sample under identical conditions.

The implications of the high-pressure sample environment for neutron Laue diffraction will be explored and the results from experiments on hexamine presented.

[1] G.J. McIntyre, L. Mélési, M. Guthrie, C.A. Tulk, J. Xu and J.B. Parise, J. Phys.: Condens. Matter 17, S3017 (2006)

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