

coefficient) and a precise evaluation of the effective incident spectrum have to be addressed.

We determined a transferable effective flux curve by comparing the measured Laue intensities of a well characterized standard crystal (calcite) with the corresponding calculated structure factors [1]. The thus derived normalized flux curve proved to be transferable between different mineral structures. First applications involving the unambiguous indexation of primitive trigonal unit cells as well as work to enable routine structure solution and refinement based on intensities derived from Laue diffraction experiments will be discussed.

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**Keywords:** laue microdiffraction, structure solution, inorganic material

## MS.47.4

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### High pressure freezing of protein crystals

Anja Burkhardt,<sup>a</sup> Martin Warmer,<sup>b</sup> Armin Wagner,<sup>c</sup> Rudolph Reimer,<sup>b</sup> Heinrich Hohenberg,<sup>b</sup> Alke Meents,<sup>a</sup> <sup>a</sup>*DESY-HASYLAB, Hamburg, (Germany)*. <sup>b</sup>*Heinrich-Pette-Institute for Experimental Virology and Immunology, Hamburg, (Germany)*. <sup>c</sup>*Diamond Light Source, Didcot, (United Kingdom)*. E-mail: anja.burkhardt@desy.de

The standard method to reduce radiation damage on biological samples is cryo cooling to cryogenic temperatures by immersing them in liquid nitrogen or a cold nitrogen gas stream [1]. Protein crystals typically contain up to 90% of solvent. In order to suppress the formation of crystalline ice upon cooling, which destroys the crystal lattice, cryoprotectants such as ethylene glycol or glycerol have to be applied. Finding a suitable cryoprotectant for a specific crystal is a very time and crystal consuming trial and error process. Moreover, the crystal quality is often degraded upon flash-cooling even if adequate cryoprotectants have been found. Such degradation manifests itself in an increase of the crystal mosaicity and a decrease in observable diffraction resolution which finally limits the ability to phase the structure.

High pressure freezing (HPF) allows cryogenic cooling of macromolecular samples by application of high pressures and low temperatures without formation of hexagonal ice and avoiding penetrative cryoprotectants [2-6].

A HPF protocol for several test proteins, e.g. thaumatin, hen egg-white lysozyme and porcine insulin, has been developed and established. For that purpose protein crystals are grown in cellulose carbonate microtubes *via* dialysis or in glass capillaries using counter diffusion techniques [7] to facilitate sample handling during the freezing procedure. Subsequently, the samples are frozen at 210 MPa while being cooled to liquid-nitrogen temperatures using a Bal-Tec HPM 010 instrument.

First X-ray diffraction experiments revealed a superior quality of the high pressure frozen samples. Due to the formation of high-density amorphous (HDA) ice, HPF crystals were diffracting to higher resolution and showed better R values compared to normally flash-cooled samples.

For the first time data sets of freeze substituted protein crystals have been successfully collected. Freeze substitution was performed on high pressure frozen lysozyme crystals at 183 K using ethanol as solvent. These crystals showed a different crystal symmetry and an increased cell volume compared to unsubstituted samples.

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**Keywords:** protein crystallography, high pressure, cryo cooling

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### Synchrotron X-ray diffraction tomography technique using diamond anvil cell

Haozhe Liu, Luhong Wang, Zhanhai Yu, Lingping Kong, Jinggeng Zhao, Dawei Dong, Chunyu Li, Zhiguo Liu, *Natural Science Research Center, Harbin Institute of Technology, Harbin 150080, (China)*. Email: Haozhe@hit.edu.cn

The joint effort from multiple user groups and beamlines in Advanced Photon Source, Argonne National Laboratory will be introduced for the development of synchrotron x-ray diffraction tomographic method combined with high pressure diamond anvil cell (DAC) technique, which offers great opportunity for the deeper understanding on phase transition behaviors of materials under high pressure extreme conditions. These studies will broaden our horizons and perspectives of the states of materials upon compression. The procedure of the pressure-induced amorphous state to amorphous state transitions, crystallization from amorphous state under pressure, as well as the phase transition and equation of state studies for simple metal powder samples, such as iron, silver and titanium, was probed using the novel techniques. The structural evolution of amorphous materials, as well as powder samples under high pressure conditions up to couple ten GPa pressure range were studied by synchrotron x-ray diffraction and imaging tomography methods in 3-D space domain in the DAC. These novel researches will provide new insight on the nature of phase transition, highlight the relationship of deviatoric stress and anisotropic elastic strain in 3-D space between the old and new phases during these phase transition processes, provide new invitation for the electronic theoretical studies for the phase stability, and improve our understanding three dimensionally of the kinetic process of the crystallization, phase transition path and mechanism in various type materials at high pressure extreme conditions.

**Keywords:** high pressure, diffraction tomography, phase transition

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### Probing the electronic structure of correlated electron systems with synchrotron light

Stephen B Dugdale,<sup>a</sup> Jude Laverock,<sup>a</sup> Claudia Utfeld,<sup>a</sup> Thomas D Haynes,<sup>a</sup> Jonathan A. Duffy,<sup>b</sup> Matthew W. Butchers,<sup>b</sup> Jonathan W Taylor<sup>c</sup> and Sean Giblin,<sup>c</sup> <sup>a</sup>*H.H. Wills Physics Laboratory, University of Bristol, Tyndall Avenue, Bristol BS8 1TL (United Kingdom)*. <sup>b</sup>*Department of Physics, University of Warwick, Coventry CV4 7AL (United Kingdom)*. <sup>c</sup>*ISIS Facility, Rutherford Appleton Laboratory, Chilton, Oxfordshire OX11 0QX (United Kingdom)*. E-mail: s.b.dugdale@bristol.ac.uk

When other methods for mapping the Fermi surface are excluded (for example, owing to sample quality, concerns about the surface, substitutional disorder, or simply the temperature at which the phase of interest exists), then the Fermi surface can be accessed through