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

High pressure freezing of protein crystals

Anja Burkhardt,[a] Martin Warmer,[b] Armin Wagner,[c] Rudolph Reimer,[b] Heinrich Hohenberg,[b] Alke Meents,[a] *[a]DESY-HASYLAB, Hamburg, (Germany). [b]Heinrich-Pette-Institute for Experimental Virology and Immunology, Hamburg, (Germany). [c]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 glas 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

MS.47.5

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

MS.48.1

Probing the electronic structure of correlated electron systems with synchrotron light

Stephen B Dugdale,[a] Jude Laverock,[a] Claudia Utfeld,[a] Thomas D Haynes,[a] Jonathan A. Duffy,[b] Matthew W. Butchers,[b] Jonathan W Taylor[c] and Sean Giblin,[c] *[a]H.H. Wills Physics Laboratory, University of Bristol, Tyndall Avenue, Bristol BS8 1TL (United Kingdom). [b]Department of Physics, University of Warwick, Coventry CV4 7AL (United Kingdom). [c]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

measurements of the momentum distribution via X-ray Compton scattering. By measuring the energy distribution of X-ray photons which have been inelastically scattered by the electrons in the sample, it is possible to measure their momentum distribution.

The momentum distribution contains information about the occupied momentum states, and therefore about the Fermi surface. Results are presented of recent Compton scattering measurements [1] performed on optimally Co-doped $BaFe_2As_2$. When compared to *ab initio* calculations which have been adjusted through small shifts of the bands with respect to the Fermi energy, the Compton scattering results can only be understood if those calculations were performed at the LDA-relaxed As position, rather than at the experimental one [2].

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MS.48.2

Resonant X-Ray Magnetic Scattering for transition-metal ferrites

Maki Okube, Satoshi Sasaki, *Materials and Structures Laboratory, Tokyo Institute of Technology, Yokohama (Japan).* E-mail: makisan@lipro.msl.titech.ac.jp

Recent developments in synchrotron radiation research make it possible to use the resonant X-ray magnetic scattering (RXMS) for crystals with complicated structures. Since RXMS has element selectivity and sensitivity to magnetic moments, it is a useful tool to determine the magnetic structures associated with specific electronic states such as 3d-4p interactions. Although various magnetic resonant experiments have been carried out, there are only a few reports on an X-ray crystallographic approach to use Bragg reflections with a wide range of reciprocal space. Taking advantage of single-crystal analysis, three-dimensional magnetic structural analyses have been developed in a combination of RXMS and least-squares refinement or Fourier syntheses. The combined technique allows us to perform element-specific and site-specific analyses on the magnetic structure. The refinement technique has been applied for Ti^{4+}/Co^{2+}-substituted $BaFe_{12}O_{19}$ to determine the canting of magnetic moments in the ferrimagnetic structure [1].

Transition-metal ferrites, having incompletely filled *d*-electron shells of iron, show useful electronic and magnetic interactions related to transition-metal and neighboring oxygen atoms. Magnetite Fe_3O_4 is a typical example to have various physical properties such as metallic behavior, mixed valence and electron hopping material. In the inverse-spinel structure geometrically frustrated, ferric and ferrous ions show a characteristic preference between tetrahedral "A" and octahedral "B" sites occupied by Fe^{3+} and equally by Fe^{2+} and Fe^{3+}, respectively. One of our RXMS studies has been focused on the estimation of magnetic electrons of Fe_3O_4, which pinpoints the electronic transition related to unpaired spins.

Energy-selective intensity measurements were carried out by using four-circle diffractometers at BL-6C of Photon Factory. Circularly-polarized X-rays were produced by a transmitted-type phase retarder of diamond (111). X-ray magnetic circular dichroism (XMCD) at the Fe *K* edge was used to select the X-ray energy of the spectrum, which was connected with the RXMS experiments with left- and right-polarized diffraction data.

The difference in crystal structure factors between left- and right-circular polarized measurements was calculated to synthesize a difference-Fourier map for targeting only magnetic electrons within

energy resolution of 0.5 to 1 eV. The difference-Fourier synthesis is superior in eliminating the other effects such as charge scattering and experimental errors. In the calculation of $[\Delta\rho_{obs}(r)^{left} - \Delta\rho_{obs}(r)^{right}]$ maps, the formalization of electron density to extract individual magnetic effects is given by $\Delta\rho(r) = V^{-1}\Sigma\Sigma\Sigma\{|F_{obs}(hkl)^{left}| - |F_{obs}(hkl)^{right}|\}\exp(-2\pi i k \cdot r)$, where $F(hkl)$ and k are the crystal structure factor and scattering vector, respectively. With some replacements of calculated F_{calc} for observed one, difference-Fourier maps of magnetite were obtained from the usual difference-Fourier formalism for $\Delta\rho(r)$. Positive and negative peaks appeared around the A and B sites, which may be caused by magnetic unpaired 3*d* electrons of Fe atoms associated with neighboring oxygen and the outer Fe atoms.

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MS.48.3

High resolution compton scattering as a new tool for imaging dopants and probing metal-insulator transitions

Arun Bansil, *Physics Department, Northeastern University, Boston (USA).* E-mail: bansil@neu.edu

We discuss two recent applications of high resolution Compton scattering as a new bulk-sensitive tool for probing electronic structure and momentum density of complex materials [1], [2].

Imaging holes in cuprate high-temperature superconductors: We have carried out first-principles electronic structure computations to analyze Compton scattering measurements from a series of $La_{2-x}Sr_xCuO_4$ single crystals in which the hole doping level varies from the underdoped (UD) to the overdoped (OD) regime. Holes in the UD system are found to primarily populate the O-2p orbitals. In contrast, character of holes in the OD system is very different in that these holes mostly enter Cu d orbitals. In this way, we establish that high-resolution Compton scattering can provide a new probe for directly imaging dopants in complex materials. Moreover, our analysis of the momentum density reveals clear existence of a large Fermi surface in the OD system in agreement with band theory predictions [3].

Metal-insulator transition in $La_{2-2x}Sr_{1+2x}Mn_2O_7$: We have studied the [100]-[110] anisotropy of the Compton profile in the bilayer manganite. A quantitative accord is found between band theory predictions and the measured anisotropy in the two metallic phases (i.e., the low temperature ferromagnetic and the colossal magnetoresistant phase under a magnetic field of 7T). Robust signatures of the metal-insulator transition are identified in the momentum density for the paramagnetic phase above the Curie temperature. The number of electrons involved in this transition is estimated. Our study demonstrates the sensitivity of the Compton technique for investigating metal-insulator transitions in complex materials. Work supported by Basic Energy Sciences, United States Department of Energy.

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