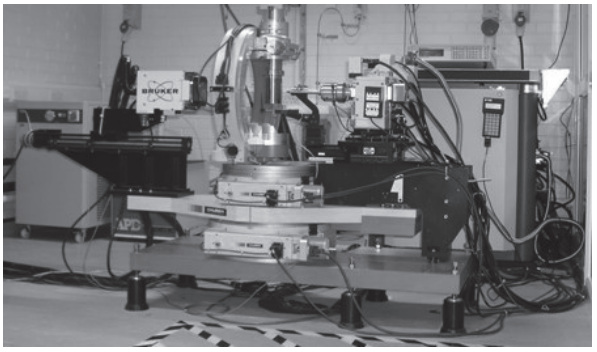
[1] has been designed for structural studies under extreme sample environments, combining equipment from several manufacturers along with custom designed components. The unique end product provides unparalleled access to 'in house' diffraction data allowing data collections to be performed at temperatures as low as 2.0 K. Operation and monitoring of the system, as well as data processing, requires novel approaches. In particular, the method of cooling means that several objects along with the sample are present in the primary X-ray beam and these features need to be accounted for. However, temperature is not the only thermodynamic variable to be explored through single crystal diffraction and there is growing interest in the field of structural studies under high pressure [2] and light irradiation [3]. Hence, the development of XIPHOS includes the controllable use of a combined range of temperatures, high pressures and/or irradiation wavelengths which expands the functionality of 'in-house' systems for the study of solid state phenomena.



[1] M.R. Probert, C.M. Robertson, J.A. Coome, J.A.K. Howard, B.C. Michell, A.E. Goeta *J. Appl. Cryst.* **2010**, *43*, 1415-1418. [2] E.V. Boldyreva *Acta Cryst.* **2008**, *A64*, 218-231. [3] P. Gütlich, H.A. Goodwin *Top. Curr. Chem.* **2004**, *234*.

Keywords: instrumentation, cryocrystallography, photocrystallography

MS48.P01

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

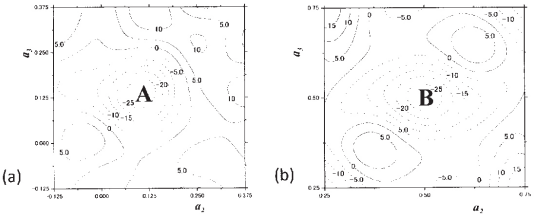
X-Ray resonant scattering - origin of Fe *K* Pre-Edge peak of magnetite

Satoshi Sasaki, Takuya Yasue, Maki Okube, *Materials and structures laboratory, Tokyo Institute of Technology, Yokohama (Japan).* E-mail: sasaki@n.cc.titech.ac.jp

The difference-Fourier synthesis between two sets of observed data was applied to X-ray resonant scattering (XRS). The XRS effect measured at a pre-edge of Fe *K* absorption edge makes it possible to give the information on a location of electrons resonantly scattered and therefore, of targeted atoms. The electron density can be estimated to subtract the resonant intensity at the pre-edge from off-edge intensity. Using a difference in the XRS pre-edge intensity measured at energy E_{on} and E_{off}, a difference in electron-density is given by $\Delta\rho(\boldsymbol{r}) = V^{-1} \Sigma\Sigma\Sigma\{F(hkl)_{on}\text{-}F(hkl)_{off}\}\exp(-2\pi i\boldsymbol{k}\cdot\boldsymbol{r})$, where $F(hkl)$ and $\boldsymbol{k}$ are the crystal structure factor and scattering vector, respectively, and then the termination effect of Fourier series is automatically corrected.

It is interesting to pinpoint specific atoms by extracting some electrons resonantly scattered. For example, the position of 1*s* electrons can be estimated from the *shell structure factors* to be the balance between X-ray resonant scattering (XRS) and total intensity [1]. Recently, the electron-density analysis of magnetite has revealed the overlapping of magnetic electron orbitals among neighboring atoms, by making use of the intensity difference in resonant X-ray magnetic scattering between left- and right-circular polarizations [2]. Magnetite has the crystal structure of inverse-spinel type of $[Fe^{3+}]^A[Fe^{2+}Fe^{3+}]^BO_4$, where only Fe^{3+} occupies the tetrahedral A sites and Fe^{2+} and Fe^{3+} equally occupy the octahedral B sites. It is known that in X-ray absorption experiments magnetite has a pre-edge structure at the Fe *K* edge. Although the pre-edge peak is considered as a dipole-transition mechanism of Fe^{3+} in the tetrahedral sites, the site symmetry of the octahedral sites is .-3*m* and gives another possibility on the origin with super-exchange interaction of A-O- B sites.

Magnetite used in this study has a cell dimension of a = 8.4000(3) Å with the space group of *Fd*-3*m*. Synchrotron experiments were performed at BL-6C of the Photon Factory using a conventional Rigaku AFC5 four-circle diffractometer. Si(111) double-crystal monochromator and diamond(001) phase retarder were used to produce circularly polarized X-rays at the Fe *K* edge. Intensity data for 354 reflections were collected in the range $2\theta \leq 90°$ with an ω-2θ step scan mode. Figure1 shows the electron-density maps of magnetite on the planes passing through (a) A and (b) B sites, respectively. Contours are at intervals of 0.5 e/Å^3, where the numbers in maps are magnified by 10 and solid lines are zero and positive and broken lines are negative. negative peaks appear around A and B sites in heights of -2.7 and -2.9 e/Å^3, respectively. It is suggested that the pre-edge peak of magnetite originates in both A and B sites.



[1] S. Sasaki , K. Tsukimura, *J. Phys. Soc. Jpn.* **1987**, *56*, 437-440. [2] Y. Kaneko, M. Okube, S. Sasaki, *Am. Inst. Phys. Conf. Proc.* **2010**, *1234*, 871-874.

Keywords: resonant_scattering, electron_density, synchrotron

MS48.P02

Acta Cryst. (2011) A**67,** C529-C530

Electron-density distribution of Fe_3O_4 on resonant X-Ray magnetic scattering

Yusuke Abe,[a] Maki Okube,[b] Satoshi Sasaki,[b] [a]*Interdisciplinary Graduate School of Science and Engineering, Tokyo Institute of Technology, Yokohama (Japan).* [b]*Materials and Structures Laboratory, Tokyo Institute of Technology, Yokohama (Japan)*. E-mail: abe.y.am@m.titech.ac.jp

A combination of resonant X-ray magnetic scattering (RXMS) [1-3] and difference-Fourier synthesis makes it possible to visualize the distribution of magnetic electrons. RXMS is a useful tool to examine the magnetic structure associated with specific electronic states such as 3*d*-4*p* interactions. The electron-density maps can be synthesized on the basis of a difference in the RXMS intensity between left- and right-circular polarizations.

The crystal structure of magnetite Fe_3O_4 is of inverse-spinel type, where there are tetrahedral A site occupied by Fe^{3+} and octahedral B site occupied equally by Fe^{2+} and Fe^{3+}. The magnetic and electronic properties are influenced by the cation distribution of Fe atoms in two kinds of the sites. X-ray magnetic circular dichroism (XMCD) of magnetite gives a dispersive signal at a pre-edge of Fe *K* absorption edge, which is observable by site-specific magnetic electrons.