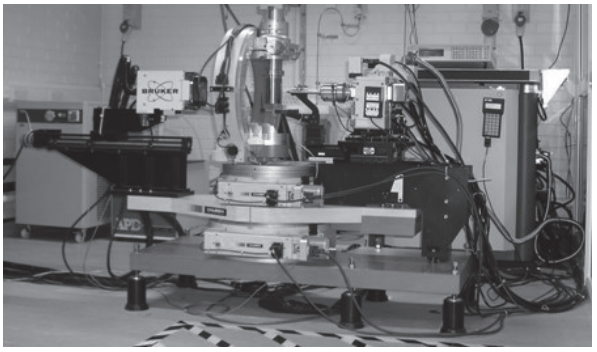
[1] has been designed for structural studies under extreme sample environments, combining equipment from several manufacturers along with custom designed components. The unique end product provides unparalleled access to 'in house' diffraction data allowing data collections to be performed at temperatures 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.



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**Keywords: instrumentation, cryocrystallography, photocrystallography**

## MS48.P01

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

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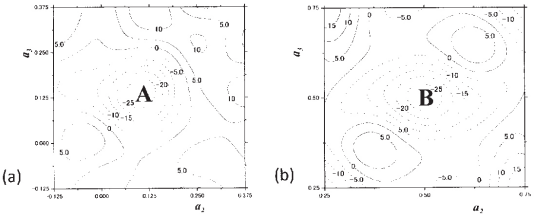
The difference-Fourier synthesis between two sets of observed data was applied to X-ray resonant scattering (XRS). The XRS effect measured at a pre-edge of Fe *K* absorption edge makes it possible to give the information on a location of electrons resonantly scattered and therefore, of targeted atoms. 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.

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**Keywords: resonant_scattering, electron_density, synchrotron**

## MS48.P02

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

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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.

Therefore, our energy-selective intensity measurements were made to choose a positive-peak position of the dispersive XMCD, which locates in a wavelength of λ = 1.7442 Å (*E* = 7.1082 keV).

The synchrotron experiments on RXMS of magnetite were performed with a Rigaku AFC5 four-circle diffractometer in Photon Factory BL-6C, where monochromatized X-rays are reproduced with the circularly polarization by a diamond (001) phase retarder. A spherical crystal of 0.13 mm in diameter was mounted along the $a_3$ axis on the glass fiber on a rare-earth magnet. The crystal was grown from $Fe_3O_4$ powder in Pt-10 % Rh crucible by the Bridgman method in the CO-$CO_2$ atmosphere and provided by Drs. S. Todo and H. Kawata. The cell dimension is $a$ = 8.4000(3) Å (*s.g. Fd*-3*m*). At temperatures of $T$ = 125, 200 and 300 K, integrated intensity data were collected at a scan speed of 0.5 °/min in ω. A total of 425 reflections was collected within the range of $2\theta \leq 100°$ and $-7 \leq h, k, l \leq 7$ and corrected for the angle-dependent polarization effect. Intensity difference ($I^+ - I^-$) between left- and right-circular polarizations extracts the RXMS effect and is roughly proportional to the real part of $F^*_{charge}F_{spin}$ in complex conjugation of crystal structure factors.

Difference-Fourier maps on targeted magnetic electrons were sybthesized from the $F$ difference between left- and right-circular polarizations. With some replacements of calculated $F_{calc}$ for observed one, the usual difference-Fourier formalism can be used for the difference in the electron density of $[\Delta\rho_{obs}(\boldsymbol{r})^{left} - \Delta\rho_{obs}(\boldsymbol{r})^{right}]$ [4]. Nonessential effects such as charge scattering and experimental errors can be cancelled out in the difference-Fourier synthesis. Thus, in this study difference-Fourier maps of magnetite were obtained as a function of temperature and will be discussed for the magnetic electron density at the electronic transition energy so far examined. Our results show that the appearance of positive and negative peaks are caused by magnetic unpaired 3*d* electrons around Fe atoms associated with neighboring oxygen and the other Fe atoms. It suggests the existence of the A-O-B super exchange interaction.

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**Keywords: resonant_magnetic_scattering, electron_density, iron_oxide**

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### Magnetic structures of $BaTiMFe_{10}O_{19}$ (M = Mn, Co) by resonant magnetic scattering

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The magnetic structures of *M*-type $BaTiMFe_{10}O_{19}$ (M = Mn, Co) have been determined by the resonant X-ray magnetic scattering (RXMS) method [1-4], where the spin-orbit coupling gives magnetic resonance at the *K* edge through the superexchange interaction between 4*p* and 3*d* sates with 2*p* of oxygen atoms. Although barium hexaferrite $BaFe_{12}O_{19}$ has the strong uniaxial anisotropy in magnetization along *c* axis, the substitution of $Fe^{3+}$ by $Ti^{4+}/M^{2+}$ results in a weakening of the magnetic interactions. The structure analyses based on neutron diffraction [5] and RXMS [4] measurements have suggested that $Ti^{4+}/Co^{2+}$-substituted crystals have ferrimagnetic structures with the canting of the magnetic moments. It is considered that the array of magnetic moments among five cation sites is different between $Ti^{4+}/Mn^{2+}$ and $Ti^{4+}/Co^{2+}$ substitutions.

Synchrotron X-ray intensity measurements were made for single crystals of ferrimagnetic ferrites at BL-6C of the Photon Factory. X-ray magnetic circular dichroism (XMCD) and RXMS effects were examined with intensity differences between the right- and left-circular polarizations, produced by a transmission-type phase retarder of diamond (001). The XMCD measurements are important to pinpoint the photon energy required for RXMS, where a negative XMCD signal around $E$ = 7.123 keV has a chemical shift between $Ti^{4+}/Mn^{2+}$ and $Ti^{4+}/Co^{2+}$ ferrites. By using a Rigaku AFC5 four-circle diffractometer, intensity measurements of RXMS for $BaTiMnFe_{10}O_{19}$ and $BaTiCoFe_{10}O_{19}$ were made in an ω–2θ scan mode at wavelengths of λ = 1.7402 Å (*E* = 7.1245 keV) and 1.7406 Å (7.1228 keV) at the Fe *K* edge, respectively.

Single crystals of ferrites were grown by a flux method. The crystal symmetry is hexagonal with the space group $P6_3/mmc$ and cell dimensions are $a$ = 5.9039(2) and $c$ = 23.2047(8) Å for Ti-Mn and $a$ = 5.8955(3) and $c$ = 23.205(2) Å for Ti-Co. The crystal structure can be built up with a sequence of spinel *fcc* blocks of $(Fe_6O_8)^{2+}$ and *hcp* blocks of $(BaFe_6O_{11})^{2-}$. Five independent Fe sites exist as tetrahedral $4f_1$, bipyramidal 2*b* and three octahedral sites of 2*a*, $4f_2$ and 12*k*. The cation distributions of the barium ferrites have been estimated from single-crystal X-ray diffraction data [6]. Spin orientations were estimated in the least-squares method based on an asymmetrical ratio $\Delta R = (I^+ - I^-)/(I^+ + I^-)$, where $I^+$ and $I^-$ are left- and right-circular polarized intensities, respectively. The degree of the spin canting for $BaTiMnFe_{10}O_{19}$ was determined in the least-squares calculations with resonant magnetic scattering factors, which was compared with that of $BaTiCoFe_{10}O_{19}$ in the relation between magnetic helices and cation substitution.

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**Keywords: resonant_magnetic_scattering, magnetic_structure, barium_ferrite**

## MS48.P04

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### X-ray magnetic diffraction and magnetic compton scattering of Pd-Co and Pt-Fe

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X-ray magnetic diffraction (XMD) experiment has been performed for $Cu_3Au$-type single crystal alloys of disordered $Pd_3Co$ and ordered $Fe_3Pt$, and magnetic Compton scattering (MCS) experiment has been performed for $Pd_3Co$. The aim of this study is to estimate spin and orbital magnetic moments of the alloys. Electron probe micro analysis (EPMA) has shown that precise chemical composition of $Pd_3Co$ is $Pd_{3.2}Co_{0.8}$. The XMD and MCS experiment were made on BL3C of