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LETTER TO THE EDITOR

Enhanced magnetic diffraction from ferromagnetic iron

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Abstract. By controlling the polarization of synchrotron radiation with a linear polarizer and a phase plate, the magnetic effect in non-resonant x-ray magnetic diffraction from a ferromagnet was enhanced for the first time. A silicon double-crystal monochromator, the Bragg angle of which was close to 45° , was used as the linear polarizer and a diamond single-crystal was used as the phase plate. The magnetic effect was increased to 2% for the 220 reflection of an iron single-crystal due to the large extinction ratio of the linear polarizer. The method can be applied to magnetic form factor measurements on ferromagnets in general.

Non-resonant x-ray magnetic diffraction is a unique tool used to separate a magnetic form factor into its orbital and the spin parts [1, 2]. For ferromagnets we can utilize the interference term of the magnetic and charge-scattering amplitudes in the cross section using circularly polarized x-rays, but the magnetic scattering peak always coincides with a charge scattering peak in reciprocal space. Some pioneering studies were made on non-resonant x-ray magnetic diffraction from ferromagnets with circularly polarized synchrotron radiation, which showed that the magnetic effect in the diffraction intensities is measurable [3], and magnetic form factors can be obtained [4, 5]. However, the accuracy of these experiments was much lower than that of neutron magnetic diffraction experiments. Early in the 1990s, a white-beam method of non-resonant x-ray magnetic form factors with an accuracy comparable to that of neutron-diffraction experiments. The white-beam method has been applied to a few ferromagnets of 3d, 4f and 5f electron systems at the Synchrotron Radiation Source (SRS) in Daresbury [7] - [9], the European Synchrotron Radiation Facility (ESRF) in Grenoble [10] and the Photon Factory (PF) in Tsukuba [11].

In any experiment concerning non-resonant x-ray magnetic diffraction from ferromagnets the magnetic effect (flipping ratio) of the diffraction intensity should be maximized. This is accomplished by fixing the scattering angle at a specimen to 90° in the orbital plane of the synchrotron. Under this condition the magnetic effect is proportional to the polarization factor (f_P), which is given by [6, 7]

$$f_P = P_c / (1 - P_l)$$

where P_l and P_c are the degrees of linear and circular polarization, respectively. We can enhance the magnetic effect through maximising f_P . In the white-beam method, which usually uses radiation from a bending magnet, f_P is maximized by optimizing the viewing angle above the electron orbit plane; f_P depends on the vertical angular divergence of the electron beam $(\sigma_{y'})$ of a storage ring. The smaller $\sigma_{y'}$ (that is, the lower the beam emittance), the larger f_P becomes. The only way to enhance f_P is to use a low-emittance ring. However, the electron beam orbit becomes less stable when $\sigma_{y'}$ becomes smaller. An unstable electron beam orbit causes unpredictable changes in the f_P , and makes it difficult to obtain accurate magnetic form factors. Another disadvantage of the white-beam method is that strong fluorescent x-rays from a specimen are apt to mask the diffraction signals. Thus the monochromatic-beam method of non-resonant x-ray magnetic diffraction with a stable polarization is needed to obtain accurate magnetic form factors [12]. In our work this is realized by utilizing a phase plate which modifies the horizontal linear polarization of synchrotron radiation.

X-ray phase plates make use of diffractive birefringence in a nearly perfect crystal, such as silicon, germanium or diamond [13, 14]. The characteristic feature of x-ray phase plates is that a phase shift produced between the σ - and π -polarization components is tunable through an offset angle ($\Delta\theta$) from the diffraction condition. We can thus control the polarization of the synchrotron radiation using the phase plate. When the phase plate is combined with a linear polarizer, which increases the degree of linear polarization, a precise control of the polarization and an enhancement of f_P are realized. Here, we report on the first successful result which shows an enhancement of f_P with the linear polarizer and the phase plate.

This experiment was performed on beamline $BL3C_1$ of the Photon Factory at the National Laboratory for High Energy Physics (KEK), where white x-rays from a bending magnet are available. The height of the electron beam orbit in the storage ring was monitored by a beam-position monitor with an accuracy of 10 μ m. The vertical angular divergence of the electron beam ($\sigma_{y'}$) was estimated to be 13 μ rad from previous experiments [15, 16].

The experimental configuration is shown in figure 1. The white x-rays were monochromatized at 8.65 keV by a Si(111) double-crystal monochromator. The incident x-ray energy was chosen so that the Bragg angle of the 220 reflection of an iron specimen was 45°. We utilized either 111 reflection (the Bragg angle $\theta_B = 13.2^\circ$) or 333 reflection ($\theta_B = 43.3^\circ$) in the vertical plane. The incident beam size was 0.1 mm (vertical) × 1 mm (horizontal) for the 111 reflection and 1.5 mm (vertical) × 1 mm (horizontal) for the 333 reflection the double-crystal monochromator functioned as a linear



Figure 1. Experimental configuration of the non-resonant x-ray magnetic diffraction with a Si doublecrystal monochromator (M) functioning as a linear polarizer, and an x-ray phase plate (P). S, specimen of the Fe single-crystal; A, pole piece of the electromagnet; D, intrinsic germanium solid-state detector. The upper part shows a side view and the lower part shows a top view of the configuration.

polarizer, because the Bragg angle was close to 45°. The extinction ratio was 520 for the 333 reflection and 1.14 for the 111 reflection. We estimated the degree of linear polarization to be 0.9920 after the 111 reflection and 0.9998 after the 333 reflection using the calculated degree of linear polarization of the incident radiation according to the program SPECTRA [17] and the extinction ratio of the monochromator.

The polarization of the x-rays after the monochromator was transformed by the phase plate of a 0.5 mm thick (001)-oriented diamond crystal slab. The scattering plane of the phase plate was tilted by 45° with respect to the horizontal plane in order to excite coherently both the σ - and π -polarization components with equal electric-field amplitudes. The phase plate was adjusted to be close to the 111 diffraction condition of the asymmetric Laue case. The degrees of linear and circular polarization (P_l and P_c) of the transmitted x-rays were controlled by the offset angle ($\Delta\theta$) from the 111 diffraction condition.

Monochromatized and polarized x-rays were incident on the specimen of a (110)-oriented iron single-crystal, which was used in previous experiments using the white-beam method [15, 16]. The specimen was 25 mm × 18 mm × 0.3 mm in size, and the longest edge was along the [001] axis. The specimen was set so that the 220 reflection ($\theta_B = 45^\circ$) would take place in the horizontal plane, and the [001] axis would be parallel to the horizontal plane. We measured the intensity of the 220 reflection with an intrinsic germanium solidstate detector, the energy resolution of which was sufficiently good to separate the reflected x-rays (8.65 keV) from the fluorescent x-rays of FeK α (6.4 keV) and FeK β (7.0 keV). When the reflection intensity exceeded 10⁴ cps, we placed an Al-foil absorber in front of the detector to ensure a linear response.

The specimen was magnetized along the [001] axis by a C-type electromagnet. The magnetization direction was reversed every 10 sec. The photon counts for one magnetization direction (I_+) , and those for the reversed magnetization direction (I_-) , were measured alternately and accumulated for 30 min. We obtained the flipping ratio $R = (I_+ - I_-)/(I_+ + I_-)/($

 I_{-}) for various offset angles of the phase plate. For a weak reflection intensity we repeated a 30-min run of the measurement several times to improve the statistical accuracy.



Figure 2. Flipping ratio (R) and the corresponding polarization factor (f_P) of the Fe 220 reflection intensity for the 111 reflection at the monochromator $(\theta_B = 13.2^\circ)$. The *R* is plotted as a function of the offset angle $(\Delta \theta)$ of the phase plate. The open circles show the observation and the solid line shows the calculation.

Figure 2 shows the observed flipping ratios at various values of $\Delta\theta$ s of the phase plate for the 111 reflection at the monochromator. The solid line is a calculated curve of $R(\Delta\theta) = \{(\hbar\omega/mc^2)\mu(220)/n(220)\}f_P(\Delta\theta)$, where $\hbar\omega$ and mc^2 are the energy of the incident x-rays and the electron rest mass, respectively, and $\mu(220)$ and n(220) are the magnetic and charge form factors of Fe at the 220 reciprocal lattice point, respectively [18, 19]. The $f_P(\Delta\theta)$ was calculated assuming that the degree of linear polarization after the monochromator was 0.9920. In figure 2 the experimental data and the calculated curve agree well. The maximum value of R was 3×10^{-3} , which corresponds to $f_P = 8$. This is the first result of non-resonant x-ray magnetic diffraction from a ferromagnet using monochromatic x-rays and a phase plate.

Figure 3 shows the observed *R* for the 333 reflection at the monochromator. In figure 3 the solid line is the calculated curve of *R*, where the degree of linear polarization after the monochromator, 0.9998, is taken into account. In figure 3 the maximum value of *R* amounts to 2×10^{-2} , which is an order of magnitude larger than that in figure 2, and the corresponding f_P is 55. This enhancement of f_P is due to the increased degree of linear polarization due to the double-crystal polarizer. This result shows for the first time that the polarization factor (f_P) can be enhanced by using an x-ray linear polarizer and a phase plate. The slight discrepancies between the experimental and calculated curves are probably due to distortion of the phase plate crystal.

The present new method of non-resonant x-ray magnetic diffraction with a linear polarizer and a phase plate does not necessarily require a low-emittance ring, and is not sensitive to the movement of the electron beam orbit in a storage ring. Therefore, this method can be applied to almost any x-ray beamline of synchrotron-radiation facilities. It is expected that



Figure 3. Flipping ratio (*R*) and the corresponding polarization factor (f_P) of the Fe 220 reflection intensity for the 333 reflection at the monochromator ($\theta_B = 43.3^\circ$). The *R* is plotted as a function of the offset angle ($\Delta \theta$) of the phase plate. The open circles show the observation and the solid line shows the calculation.

 f_P will be much more enhanced if we use a linear polarizer with a larger extinction ratio, such as the Hart - Rodrigues type polarizer [20]. We will be able to achieve an extremely large polarization factor (f_P) by optimizing this method to an undulator beamline of the third generation synchrotron radiation facilities, such as ESRF, APS and SPring-8.

In conclusion, we have developed a new method of non-resonant x-ray magnetic diffraction using a linear polarizer and a phase plate. We could observe the polarization dependence of non-resonant x-ray magnetic diffraction from the Fe crystal by this method. Further, we could enhance the polarization factor up to 55 by increasing the degree of linear polarization with a linear polarizer and modifying the polarization with a phase plate. This new method will prompt magnetic form factor measurements, especially at the third generation synchrotron radiation facilities, such as ESRF, APS and SPring-8.

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