X-ray double phase retarders to compensate for off-axis aberration

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An X-ray double phase retarder system composed of two transmission-type phase retarders is proposed and developed in order to compensate for off-axis aberration (phase-shift inhomogeneity due to angular divergence of incident X-rays). The scattering planes of the two phase retarders are set to be inclined by 45° with respect to the plane of incident polarization, but the two phase retarders give Bragg reflections in opposite directions. By using this X-ray optical system, vertically polarized X-rays with a 0.99 degree of linear polarization were obtained from horizontally polarized synchrotron radiation with a horizontal beam divergence of 20 arcsec (0.1 mrad). This value is favorably compared with the value of 0.87 which was obtained using a conventional single phase retarder of identical total thickness, 627 μm. The comparison was made at the nickel *K*-absorption edge (8333 eV) with the condition that 47% of incident X-rays were transmitted through the two phase retarder crystals. The crystals were (100)-oriented diamond plates giving asymmetric 111 Laue reflections.

Keywords: X-ray diffraction; X-ray phase retarders; X-ray phase plates; polarized X-rays; X-ray polarization; dynamical diffraction theory; diamond.

1. Introduction

Since the suggestion by Skalicky & Malgrange (1972), several X-ray phase-retarder candidates for utilizing polarization-dependent dispersion in Bragg-reflecting perfect crystals have been proposed (Hart, 1978; Annaka *et al.*, 1980; Annaka, 1982; Golovchenko *et al.*, 1986; Mills, 1987). It has come to be recognized nowadays, however, that the transmission-type X-ray phase retarder developed by Hirano *et al.* (1991, 1992, 1993, 1995) is a decisive X-ray optical device for controlling the polarization state of X-rays arbitrarily. Its early applications were reported by Ishikawa *et al.* (1991, 1992) and Giles *et al.* (1994). The transmission-type X-ray phase retarder works on the principle that σ- and π-polarized components of forward-diffracted X-rays propagate in a perfect crystal with different phase velocities which depend on the deviation angle from the exact Bragg condition. The phase shift between σ- and π-polarizations of forward-diffracted X-rays changes with angular deviation from the Bragg condition more gradually compared with that of Bragg-reflected X-rays. Therefore, the transmission-type phase retarder has the advantage that it gives a more homogeneous phase shift for a divergent X-ray beam compared with reflection-type phase retarders (Hart, 1978; Annaka *et al.*, 1980; Annaka, 1982; Golovchenko *et al.*, 1986; Mills, 1987). In spite of this advantage, X-ray beams incident on the transmission-type phase retarder have to be collimated when a strictly defined phase shift is required, *e.g.* when circularly or vertical-linearly polarized X-rays with high degrees of polarization are required (Hirano *et al.*, 1993). In other words, the transmission-type phase retarder still suffers from phase-shift inhomogeneity due to angular divergence of the incident beam (off-axis aberration). However, beam collimation is obtained only at the expense of the incident photon flux.

In this paper we will propose an X-ray double phase retarder system which can compensate for the off-axis aberration. It is composed of two successive transmission-type X-ray phase retarders made of diamond crystals of half thicknesses which give Bragg reflections in opposite directions. It requires a well defined uniform polarization state of the incident X-ray beam which is produced by an X-ray polarizer in order to gain its advantage effectively. Hart & Rodrigues (1979) invented a tunable X-ray polarizer made of a silicon channel-cut crystal equipped with an offset mechanism, which can produce a high degree of linear polarization of incident X-rays with a horizontal-polarization:vertical-polarization intensity ratio as high as 10^6. The principle of the double phase retarder system will be described, and its advantage over the conventional single phase retarder when combined with the Hart–Rodrigues' X-ray polarizer will be shown.

2. Principle of the double phase retarders

Fig. 1(*a*) shows the configuration of double phase retarders arranged so that the off-axis aberration can be compensated. Unit vectors $\mathbf{e}_x$, $\mathbf{e}_y$ and $\mathbf{e}_z$ drawn in the upper part of Fig. 1 construct an orthogonal coordinate system. The scattering planes of the two crystals are in the plane of the drawing and are inclined by 45° with respect to the polarization plane of the incident synchrotron radiation whose polarized direction is $\mathbf{e}_x + \mathbf{e}_y$. In this arrangement the two crystals give Bragg reflections of Laue geometry in opposite directions. Now, let us consider beam paths *A*, *B* and *C* drawn in Fig. 1(*a*). X-rays propagating in beam path *A* are incident on the first phase retarder with a higher angle than in beam path *B*, but incident on the second phase retarder with a lower angle. The situation is contrary in the case of beam path *C*. Figs. 1(*b*) and 1(*c*) show dispersion surfaces for the first and second phase retarders, respectively. Dispersion surfaces for π-(x-)polarization are closer to the asymptotes by a factor of $\cos 2\theta_B$ than those for σ-(y-)polarization, where θ_B is the Bragg reflection angle.

As is deduced from the dynamical diffraction theory, when the incident X-rays excite four tie points [two each for σ-(y-) and π-(x-)polarizations] on the dispersion surfaces at the low-angle side of the Bragg condition in a Laue geometry, a dominant part of the forward-diffracted X-ray amplitude in the crystal is given by the excitation points on the branches outside the Lorentz points E_1 and E_2. The circumstances are contrary at the high-angle side. In Figs. 1(*b*) and 1(*c*), dominant and less dominant parts of dispersion surfaces for forward-diffracted X-rays are drawn by solid and dashed curves, respectively. Therefore, a transmission-type phase retarder gives a phase retardation to σ-(y-)polarization when it works at the low-angle side of the Bragg condition in a Laue geometry. In Figs. 1(*b*) and 1(*c*), when both the first and second phase retarders work to give phase retardation to σ-polarization, tie points on the upper left part of the dispersion surfaces for the first phase retarder and those on the upper right dispersion surfaces for the second phase retarder are mainly excited by the incident radiation. When monochromatic divergent X-rays are transmitted through the first and second phase retarders consecutively with a condition that reflection angles are lower than the Bragg condition, X-rays propagating in directions A, B and C in Fig. 1(*a*) excite tie points on the dispersion surfaces in Fig. 1(*b*) with vectors $\mathbf{a}_1$, $\mathbf{b}_1$ and $\mathbf{c}_1$ and excite tie points in Fig. 1(*c*) with vectors $\mathbf{a}_2$, $\mathbf{b}_2$ and $\mathbf{c}_2$. $\mathbf{a}_1$, $\mathbf{b}_1$ and $\mathbf{c}_1$ are vectors normal to the surface of the first phase retarder, and $\mathbf{a}_2$, $\mathbf{b}_2$ and $\mathbf{c}_2$ are those of the second phase retarder. The positions of those vectors are determined by the angular deviation of incident radiation from the exact Bragg condition. Now, let us introduce the difference of the transmitted wave-number vector between σ- and π-polarizations, $\Delta\mathbf{k}_o = \mathbf{k}_o^{(\sigma)} - \mathbf{k}_o^{(\pi)}$; *e.g.* $\Delta\mathbf{k}_o$ given at tie points excited by vector $\mathbf{a}_1$ is denoted by $\Delta\mathbf{k}_o(\mathbf{a}_1)$. It can be found from Fig. 1(*b*) that $|\Delta\mathbf{k}_o(\mathbf{a}_1)| > |\Delta\mathbf{k}_o(\mathbf{b}_1)| > |\Delta\mathbf{k}_o(\mathbf{c}_1)|$, to the contrary from Fig. 1(*c*) that $|\Delta\mathbf{k}_o(\mathbf{a}_2)| < |\Delta\mathbf{k}_o(\mathbf{b}_2)| < |\Delta\mathbf{k}_o(\mathbf{c}_2)|$. Consequently, phase-shift gradients (off-axis aberration) with the first and the second phase retarders are cancelled out by each other.

This situation is clarified in Fig. 2, in which phase shifts given by the phase retarders are plotted as ordinate against the incident angle of X-rays to phase retarders as abscissa. Dashed curves, $\Delta\varphi_1$ and $\Delta\varphi_2$, are phase shifts given by the first and second phase retarders, respectively. Solid curves, $\Delta\varphi_1 + \Delta\varphi_2$ and $2\Delta\varphi_1$, are those given by the double phase retarders as shown in Fig. 1(*a*) and by a single phase retarder that is twice as thick as the first phase retarder, respectively. Angular positions a, b and c on the abscissa correspond to beam paths A, B and C, respectively, in Fig. 1(*a*). An angular distribution of incident X-ray intensity on the abscissa shown as α in Fig. 2 is projected onto curves $2\Delta\varphi_1$ and $\Delta\varphi_1 + \Delta\varphi_2$, resulting in phase-shift distributions shown as β and γ on the left-hand and right-hand ordinates. The range of phase-shift distribution γ is clearly suppressed compared with β, which reveals the compensation of phase-shift inhomogeneity due to the off-axis aberration.

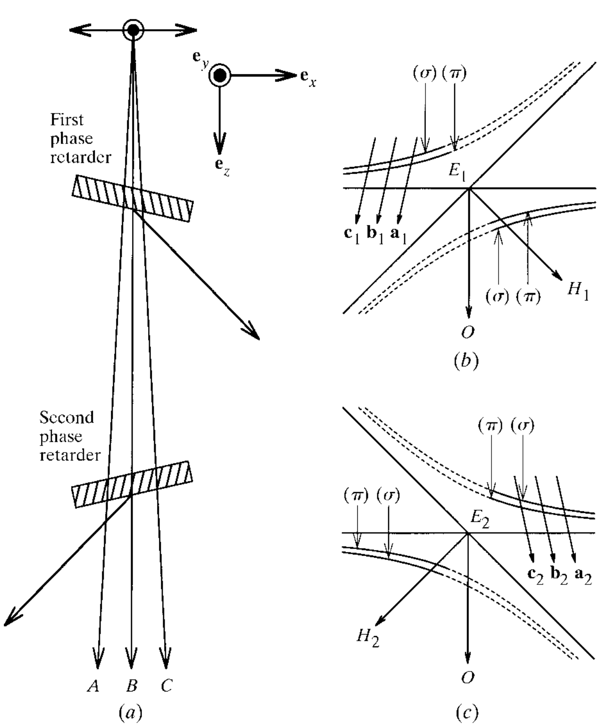

Figure 1
(*a*) Arrangement of the X-ray double phase retarder system to compensate for the off-axis aberration of the transmission-type phase retarder. (*b*), (*c*) Dispersion surfaces in reciprocal space corresponding to the first and second phase retarders, respectively.

3. Experimental

In order to evaluate the effect of the compensation of the off-axis aberration, we have performed experiments at BL-15B$_1$ of the Photon Factory, Institute of Materials Structure Science. The experimental set-up is shown in Fig. 3. The double phase retarder system was placed between channel-cut polarizer- and analyzer-crystals which gave four-bounced silicon 511 reflections in a symmetric Bragg geometry (Hart & Rodrigues, 1979). The polarizer- and analyzer-crystals were arranged in a parallel nicol geometry, *i.e.* the scattering planes of both the crystals were adjusted to be vertical within an error of 0.1°.

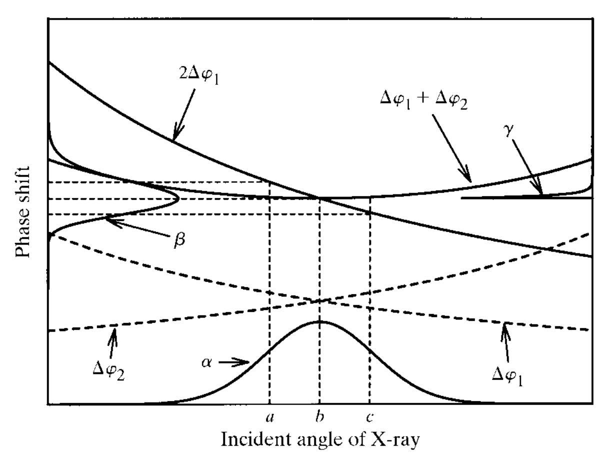

Figure 2
Phase shifts with phase retarders as functions of the incident angle of the X-rays. Phase shifts, $\Delta\varphi_1$ and $\Delta\varphi_2$ given by the first and second phase retarders arranged in the anti-parallel geometry are drawn as dashed curves. Phase shifts given by the double phase retarders (solid curves) arranged in the parallel and anti-parallel geometries are given by $2\Delta\varphi_1$ and by $\Delta\varphi_1 + \Delta\varphi_2$, respectively.

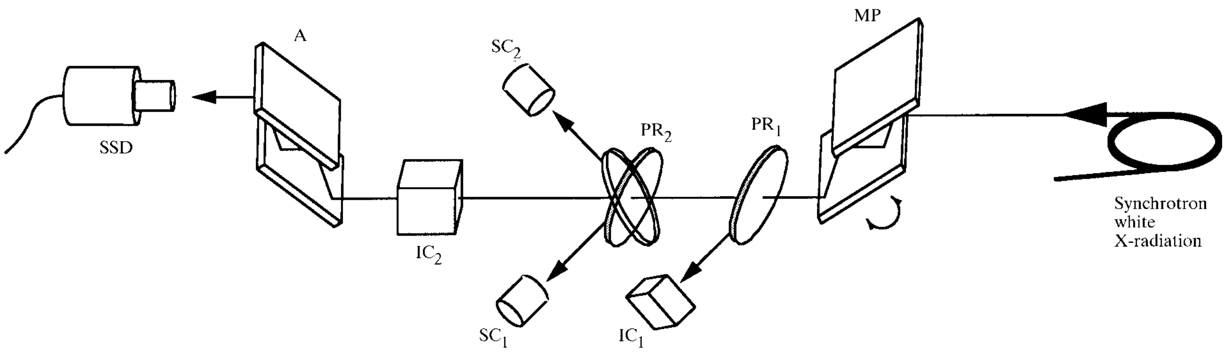

Figure 3
Experimental arrangement for estimating the effect of the compensation of the off-axis aberration. MP: silicon 511 monochromating polarizer; PR_1 and PR_2: first and second diamond 111 phase retarders; IC_1: ionization chamber monitoring the X-rays Bragg-reflected by the first phase retarder; SC_1 and SC_2: scintillation counters monitoring the X-rays Bragg-reflected by the second phase retarder; IC_2: ionization chamber monitoring the X-rays transmitted through the double phase retarders; A: silicon 511 analyzer crystal arranged in a parallel nicol geometry with respect to the polarizer.

The polarizer was water-cooled and worked also as a monochromator. The experiments were performed at a fixed photon energy of the nickel K-absorption edge (8333 eV) with the Bragg angle of the polarizer and analyzer at 45.38°. This allows us to obtain an extremely high extinction ratio (the reflectivity ratio of σ- to π-polarization) of 3×10^9 as calculated from fourfold convolution of the Darwin curves for σ- and π-polarizations based on the dynamical diffraction theory. The X-ray beam was limited by a slit to 2.5 mm (H) × 2 mm (V). The horizontal angular divergence of X-rays outgoing from the polarizer was about 20 arcsec. The first and second phase retarders were (100)-oriented diamond crystals giving 111 reflection in an asymmetric Laue geometry. The two phase retarders were approximately circular-shaped crystals with diameters of about 5 mm, whose thicknesses were 313 µm (first crystal) and 314 µm (second crystal). About 47% of incident photons were transmitted thorough the two phase retarders. Two types of geometry of the double phase retarders were examined and compared with the identical effective thickness of the phase retarder crystals: parallel geometry, in which vector products $\mathbf{k}_o \times \mathbf{k}_{h_1}$ and $\mathbf{k}_o \times \mathbf{k}_{h_2}$ are parallel; and anti-parallel geometry, in which $\mathbf{k}_o \times \mathbf{k}_{h_1}$ and $\mathbf{k}_o \times \mathbf{k}_{h_2}$ are anti-parallel. Here, $\mathbf{k}_o$ is a wave-number vector of the transmitted beam, and $\mathbf{k}_{h_1}$ and $\mathbf{k}_{h_2}$ are wave-number vectors of beams Bragg-reflected by the first and second phase retarders, respectively. The scattering planes of the phase retarders were inclined by 45° with respect to the horizontal plane. The horizontally polarized component of the X-rays was measured with a germanium solid-state detector (SSD) placed behind the analyzer, by changing the deviation angle from the Bragg condition of the two phase retarders simultaneously. In the experiment, the deviation angles of the two phase retarders were kept at the same value so that the first and second phase retarders gave the same value of phase shift between σ- and π-polarizations. Bragg-reflected X-ray intensity from the first phase retarder PR_1 was monitored by an ionization chamber IC_1 and that from the second phase retarder PR_2 was monitored by a scintillation counter SC_1 or SC_2. The intensity of the X-rays transmitted through the first and second phase retarders consecutively was monitored by an ionization chamber IC_2. Signals of IC_1, (SC_1 or SC_2), IC_2 and SSD were measured for 1 s at each angular position of the phase retarders which were rotated with a step of 0.9 arcsec.

4. Results and discussions

Fig. 4 shows the experimental results. The abscissa indicates the deviation angle $\Delta\theta$ of the first and second phase retarders

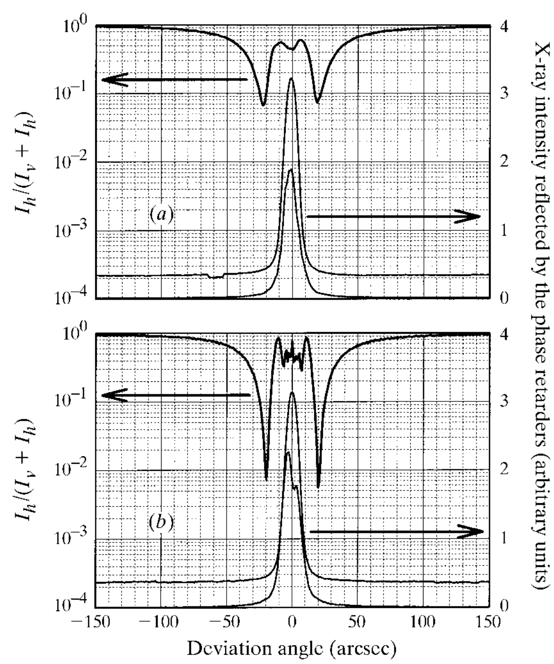

Figure 4
The rate of the horizontally polarized component to the total X-ray intensity (logarithmic left-hand ordinate) as a function of deviation angle from the Bragg condition of the first and second phase retarders. The right-hand ordinate shows intensities of X-rays reflected by the first (upper curve) and the second (lower curve) phase retarders. (a) and (b) correspond to cases of parallel and anti-parallel geometries, respectively.

from the Bragg condition in arcsec. The right-hand ordinate on an arbitrary linear scale shows X-ray intensities reflected by the first and second phase retarders. The left-hand ordinate on a logarithmic scale shows the ratio of the horizontally polarized component to the total X-ray intensity incident on the analyzer, $I_h(\Delta\theta)/[I_v(\Delta\theta)+I_h(\Delta\theta)]$, where $I_h(\Delta\theta)$ and $I_v(\Delta\theta)$ are horizontally and vertically polarized components of X-rays incident on the analyzer; Fig. 4(*a*) for the parallel and Fig. 4(*b*) for the anti-parallel geometries. Values of $I_h(\Delta\theta)/[I_v(\Delta\theta)+I_h(\Delta\theta)]$ were calculated using the following procedure.

The X-ray intensity $I_{\rm total}(\Delta\theta)$ monitored with the ionization chamber IC_2 is proportional to $I_v(\Delta\theta)+I_h(\Delta\theta)$ at any position of $\Delta\theta$. Therefore, $I_v(\Delta\theta)+I_h(\Delta\theta)$ is given by

$$I_v(\Delta\theta)+I_h(\Delta\theta)=cI_{\rm total}(\Delta\theta), \tag{1}$$

where c is a constant determined by the detection efficiency of the ionization chamber IC_2. Here, values of $I_h(\Delta\theta)$ and $I_{\rm total}(\Delta\theta)$ are directly measured as signals of SSD and IC_2, but $I_v(\Delta\theta)$ is not directly known. On the other hand, at $\Delta\theta=-180$ arcsec, far enough from the Bragg condition of phase retarders, a large part of $cI_{\rm total}(-180\,{\rm arcsec})$ in equation (1) is occupied by $I_h(-180\,{\rm arcsec})$ and is expected from the dynamical diffraction theory to be

$$I_h(-180\,{\rm arcsec})=0.978\,c\,I_{\rm total}(-180\,{\rm arcsec}). \tag{2}$$

The value of c can be calculated from the above equation. By comparing (1) and (2) we obtain the following relation,

$$\frac{I_h(\Delta\theta)}{I_v(\Delta\theta)+I_h(\Delta\theta)}=I_h(\Delta\theta)/\left[cI_{total}(\Delta\theta)\right] \tag{3a}$$

$$=\frac{0.978I_{\rm total}(-180\,{\rm arcsec})I_h(\Delta\theta)}{I_h(-180\,{\rm arcsec})I_{\rm total}(\Delta\theta)}. \tag{3b}$$

The degree of vertically linear polarization $P_v(\Delta\theta)$ is defined and is evidently represented as follows,

$$P_v(\Delta\theta)\equiv\left[I_v(\Delta\theta)-I_h(\Delta\theta)\right]/\left[I_v(\Delta\theta)+I_h(\Delta\theta)\right] \tag{4a}$$

$$=1-\left\{2I_h(\Delta\theta)/\left[I_v(\Delta\theta)+I_h(\Delta\theta)\right]\right\}. \tag{4b}$$

Both in Figs. 4(*a*) and 4(*b*) dips are observed at about ±20 arcsec from the Bragg condition, which indicates that vertical polarization is produced with a phase shift of $\pm\pi$ ($\pm\pi/2$ with each phase retarder) given by the double phase retarders. Comparing the depth of the dips in Figs. 4(*a*) and 4(*b*), it is found that a residual horizontally polarized component in vertically polarized X-rays produced with the anti-parallel geometry is about one-tenth of that in the case of the parallel geometry. The parallel geometry corresponds to a single phase retarder of total thickness 627 µm (313 µm + 314 µm). At positions of the dips on the low-angle (left) and the high-angle (right) side of the Bragg condition, maximum degrees of vertical polarization $P_v^{\rm (L.Max)}$ and $P_v^{\rm (R.Max)}$ were calculated by using equations (3*b*) and (4*b*). In the case of the parallel geometry (Fig. 4*a*), $P_v^{\rm (L.Max)}$ = 0.867 and $P_v^{\rm (R.Max)}$ = 0.854, while in the case of the anti-parallel geometry (Fig. 4*b*), $P_v^{\rm (L.Max)}$ = 0.985 and $P_v^{\rm (R.Max)}$ = 0.989. These results reveal an evident advantage in the anti-parallel geometry compared with the parallel geometry which corresponds to the conventional single X-ray phase retarder. Furthermore, a fine oscillatory profile is observed between the dips in Fig. 4(*b*) which is due to a rapid change of the phase shift. However, such oscillation is not found in Fig. 4(*a*) owing to a blurred phase shift due to the off-axis aberration.

This X-ray double phase retarder system in the anti-parallel geometry enables us to create a high degree of arbitrary X-ray polarization even with a divergent X-ray beam available from a bending magnet of a second-generation synchrotron radiation source. By dividing a single phase retarder into two phase retarders of half thickness, a higher degree of X-ray polarization can be obtained compared with a conventional single phase retarder.

Another advantage in the X-ray double phase retarder system compared with the conventional one is concerned with its application to polarized XAFS in which horizontal and vertical polarizations of X-rays have to be quickly switched holding high degrees of polarization. A zero phase shift to generate horizontal polarization is achieved by slightly rotating either crystal of the double phase retarders so as to give a phase shift whose sign is just contrary to that of the other crystal, whereas, in the case of a conventional single phase retarder, residual phase shift remains even if the crystal is rotated far from the Bragg condition. This means that a linear polarization switching with a higher frequency is feasible with the double phase retarders, holding high degrees of both vertical and horizontal polarizations of X-rays. The present method should be compared with an X-ray linear dichroism experiment recently reported by Goulon *et al.* (1998), in which a transmission-type phase retarder giving a phase shift of $\pm\pi/2$ was coupled with a helical undulator synchrotron X-ray source. We consider that our linear-polarization switching method, whose application to X-ray polarization imaging has already been published in part (Sato *et al.*, 2000) and further application to polarized XAFS will be reported in the near future, has an advantage in the degree of linear polarization over the method using a helical undulator coupled with a single phase retarder. The circular polarization state of the circularly polarized synchrotron radiation source is disturbed when reflected by monochromator crystals, whereas a high degree of linear polarization generated by Hart–Rodrigues' monochromating polarizer is held just upstream of the double phase retarder system and then only slightly disturbed being transmitted through the phase retarders by residual off-axis (and partially chromatic) aberration.

In order to take advantage of the double phase retarders in the anti-parallel geometry more effectively, the most suitable reflection index has to be chosen for the polarizer so that the Bragg angle is approximately 45°. By choosing the reflection index of the silicon crystal adequately, an extremely high extinction ratio (R_σ/R_π; R_σ and R_π are reflectivities for σ- and π-polarizations) of $>10^4$ can be achieved using the Hart–

Rodrigues' polarizer equipped with an offset mechanism in the wavelength range 1–3 Å (Hart & Rodrigues, 1979). For example, we have performed polarized XAFS experiments using silicon 422 and 511 reflection polarizers for samples containing cobalt and nickel whose K-absorption edges are 7709 and 8333 eV (1.60 and 1.49 Å).

The double phase retarder can also be applied to measurements of natural and magnetic circular dichroism in which high degrees of circular polarization are required. By using this system, we have successfully measured natural circular dichroism spectra with an α nickel sulfate hexahydrate (chiral crystal) in the vicinity of the nickel K-absorption edge by switching the helicity of circularly polarized X-rays with high degrees of circular polarization (Ueji *et al.*, 2000).

The energy spread of X-rays reflected by the polarizer is estimated to be 0.6 eV from the vertical beam size (2 mm) and distance from synchrotron X-ray source (30 m). However, the energy spread of X-rays reflected by the analyzer is further suppressed since the polarizer and analyzer crystals were arranged in a $(+-+-, -+-+)$ geometry. From a consideration using the Du Mond diagram, the energy spread of X-rays incident on the SSD is estimated to be about 0.07 eV. Therefore, phase-shift inhomogeneity due to the energy spread of X-rays (chromatic aberration) is negligible in the present work, in which we have evaluated the double phase retarders proposed for the compensation of the off-axis aberration.

In the present work, we attempted using phase retarder crystals giving reflections in the Laue geometry. Another candidate is the Bragg geometry (Hirano *et al.*, 1991, 1992, 1993, 1995; Ishikawa *et al.*, 1991, 1992; Giles *et al.*, 1994). We consider that the Laue geometry is more suitable than the Bragg geometry for gaining a homogeneous phase shift and a resultant high degree of polarization owing to the following reason. In the Bragg geometry, wave fields of forward-diffracted and reflected X-rays in the crystal are distributed in a broad region surrounded by front and back surfaces of the phase retarder crystal, one edge of the crystal and a vector from the beam incidence position to the exit position of directly transmitted X-rays. The purity of the polarization state, *i.e.* phase-shift homogeneity of forward-diffracted X-rays downstream of the phase retarder, is contaminated with the complex polarization state of the forward-diffracted wave field whose position is distant from the exit position of directly transmitted X-rays. On the other hand, in the Laue geometry, such contamination as in the Bragg geometry is not expected. However, experimental comparison between the Bragg and Laue geometries used in the double phase retarder (in the anti-parallel geometry) is now under consideration.

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