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OPTICAL DESIGN FOR THE NSLS CRYSTALLOGRAPHY BEAM LINE*

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The design goal of this beam line is to produce a photon beam well suited to accurate single-crystal diffraction measurements, with particular attention to the possibilities of the use of anomalous dispersion. These goals lead to the following criteria: (1) A beam uniform in intensity to 1% over an area $0.3 \times 0.3 \text{ mm}^2$, (2) $\Delta E/E \approx 5 \times 10^{-4}$, and (3) Tunable over the energy range 5-21 keV accessible from a NSLS bending magnet.

These criteria lead to a number of design decisions that have been applied to this and other NSLS beam lines: (1) A mirror is placed as the first optical element to better match the vertical opening angle of the photon beam to the typical rocking curves of low-index silicon Bragg peaks, reducing $\Delta E/E$ in a nondispersive, two-crystal monochromator. (2) With this mirror in place a two-crystal parallel mode monochromator with constant deviation is chosen. (3) A post-mirror to condense the radiation is placed after the monochromator. The entire optical system has been designed to operate in an ultra-high vacuum environment. The details of the mirror designs, coatings and grazing angles are presented as well as other possible choices for these elements. Ray tracing results are given for the crystallography beam line for comparison with the design goals.

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I. INTRODUCTION

The basic objective for the crystallography beam line is to be able to perform highly accurate Bragg intensity measurements over as large a range of incident x-ray wavelength as practicable. The different types of experiments planned for this beam line may be classified in a number of categories. Structural studies are anticipated on very small (diameter $< 50 \mu\text{m}$) single crystals of technologically important materials such as zeolites. Very accurate x-ray data from the synchrotron will be used to analyze charge-density distributions, for example in transition metal cluster compounds. Anomalous scattering will be utilized for solving structures, and the tunability of the beam will also be exploited in angle-resolved single crystal extended x-ray absorption fine structure (EXAFS) studies. Finally, diffraction data will be obtained for macromolecules, with emphasis on systems such as viruses that have very large unit cells.

In the sections that follow, the design parameters dictated by these experimental goals and the optical solutions that will accomplish them for the NSLS crystallography beam line will be presented. The aim in our design is to provide an instrument that is easily operated without sacrificing performance.

II. DESIGN PARAMETERS

The scientific goals of this beam line demand several particular parameters for the incident beam impinging on the sample. First, the accurate structure factor studies require beam intensity uniformity of 1% or better over the sample area, typically $0.3 \times 0.3 \text{ mm}^2$. Second, the anomalous scattering experiments and angle-resolved EXAFS need monochromaticity comparable to the core hole lifetime which is typically $1-3 \times 10^{-4} \text{ } \Delta E/E$. Finally, these studies need optics that cover the wavelength range from $\sim 0.6 \text{ \AA}$, which is the cutoff of the arc sources at NSLS, to $2.5 \text{ \AA}$ with relatively simple adjustments.

III. MONOCHROMATOR

There are a number of ways to achieve the required energy resolution. The various choices are best evaluated by first looking at the resolution from a single, flat crystal scattering photons with the diffraction plane vertical. All discussion will assume this geometry since the synchrotron radiation is strongly polarized and collimated in the plane of the storage ring. The energy resolution $\Delta E/E$ for this flat crystal is:

$$\frac{\Delta E}{E} = (\cot \theta_B) \Delta \theta .$$

θ_B is the nominal Bragg angle and $\Delta \theta$ is the angular variation of the incident photons, σ_v , convoluted with the Darwin width of the Bragg reflection, ω_D . For two Gaussians of width ω_D and σ_v :

$$\Delta \theta = \sqrt{\omega_D^2 + \sigma_v^2} .$$

For most perfect crystals in symmetric Bragg reflection $\sigma_v \gg \omega_D$. Further, σ_v is a function of photon energy. This functional dependence for a Gaussian approximation¹ to the actual vertical angular distribution of the photons is given by:

$$N(\psi_v) \propto e^{-\psi_v^2 / 2\sigma_v^2}$$

where

$$\gamma \sigma_v = -0.565 \left(\frac{E_c}{E} \right)^{0.425}$$

γ is the ratio of the electron energy to its rest energy and E_c is the critical energy of the storage ring. The $2\sigma_v$ values of $N(\psi_v)$ as a function of photon energy for 2.5 GeV electrons at the NSLS are plotted in Fig. 1a, and the corresponding resolution, assuming $\omega_D = 0$, for a number of silicon crystal reflections is shown in Fig. 1b. It is clear from Fig. 1b that the desired resolution of $\sim 3 \times 10^{-4}$ cannot be achieved simply with a single flat crystal or two crystals in the nondispersive or parallel mode.

The question is how to achieve resolutions of $\sim 3 \times 10^{-4}$ or something close to this. If the contribution from the photon opening angle can be eliminated the resolution is given by:

$$\Delta E/E = (\cot \theta_B) \omega_D$$

where, neglecting absorption:

$$\omega_D = \frac{4d^2}{\pi} \left(\frac{e^2}{mc^2} \right) \frac{\tan \theta_B}{V} |F_{\bar{H}}| .$$

Here d is the interplanar spacing, θ_B the Bragg angle, (e^2/mc^2) the classical electron radius, V the volume of the unit cell and $F_{\bar{H}}$ the structure factor for the reflection with reciprocal lattice vector $\bar{H}$. The width ω_D depends only upon the tangent of the Bragg angle so that the resolution is independent of energy and is dependent only upon the choice of reflection. Values for some typical reflections from Si and Ge are given in Table I. These values of the resolution can be achieved using the crystals in the dispersive or anti-parallel mode, where only the Darwin width contributes. Beaumont and Hart² devised a clever way of combining two sets of parallel crystals with the middle two crystals arranged in an anti-parallel way. This scheme keeps the incident and exit beams fixed as the two crystal pairs are rotated to change energy (Bragg angle). However, there is a

significant loss in intensity for the anti-parallel spectrometer matched to the storage ring because $\sigma_v > \omega_D$ and only those photons making an angle within the Darwin width for the chosen reflection are transmitted. For example with 8 keV photons the Si(111) has $\omega_D = 7$ arc sec while $2\sigma_v = 39$ arc sec at 2.5 GeV in the NSLS X-ray ring.

IV. PREMIRROR

The performance of any of the two monochromators discussed above would be improved with a better match of the synchrotron source to the crystal monochromator, i.e. if σ_v were reduced with an optical element before the monochromator. To obtain parallel radiation in one direction from a line source one would use a parabolic mirror. The electron beam in the storage ring, however, has a finite vertical size. Therefore, a single point on the mirror, seeing the whole source, reflects a range of angles given by S_v/F_1 where S_v is the source height and F_1 the distance between source and mirror. For a Gaussian distribution of electrons with $4\sigma_{\text{vert.}} = 0.5$ mm, a parabolic mirror at 5 m from the source gives a Gaussian angular spread of $2\sigma_v = 10$ arc sec, and the corresponding value of $\Delta\theta$ gives $\Delta E/E \leq 3 \times 10^{-4}$ over the entire wavelength range for the parallel mode two-crystal monochromator.

The parabolic mirror is particularly appealing because, as was shown by Kirkpatrick and Baez³ a sphere is a very good approximation to a parabola for our choice of grazing angle and focal length. In particular, the spherical mirror can accept the full vertical emittance of the storage ring with aberrations that are small compared to 10 arc sec.

The remaining problem to be solved concerns how best to focus the monochromatic radiation onto the sample. In this hypothetical beam line, the parabolic mirror would be followed by a crystal monochromator, and then a condensing mirror to focus the radiation. Keeping in mind the low cost of the spherical mirror, the choice for the second mirror of a bent cylinder focusing in both directions was investigated using a ray tracing program developed at Brookhaven,⁴ as will be discussed in the next section.

Another possibility for matching the source to the monochromator and focusing the beam onto the sample would be to use two paraboloidal mirrors which could be fabricated most economically by diamond turning techniques. The first mirror to collimate the source would be followed in turn by a crystal monochromator and then the second mirror to focus the radiation onto the sample. The first question to be answered is how well the paraboloid collimates the radiation to provide the angular spread needed to give the desired energy resolution. Again, assuming the 4σ vertical size of the beam to be 0.5 mm, the simple S_v/F_1 result will be the same as for the parabola considered before, $2\sigma_v = 10$ arc sec at 5 m. There will be an additional contribution from the horizontal source size because the mirror is now a surface of revolution rather than being flat in the transverse direction. This contribution has been evaluated and its functional dependence on source to mirror distance F_1 , horizontal source size S_h , horizontal angular acceptance ω_h , and grazing angle θ determined to be

$$\Delta\theta_p \propto \frac{\omega_h S_h}{F_1 \theta}.$$

Figure 2 shows ray tracing results for this functional form. This contribution is added in quadrature with the spread due to vertical source size to give

$$\sigma_v = \sqrt{\left(\frac{S_v}{F_1}\right)^2 + \left(\frac{C_o \omega_h S_h}{F_1 \theta}\right)^2}.$$

V. CONDENSING ELEMENT

The second mirror is used to focus the radiation onto the sample with the constraint of 1% intensity uniformity over a $0.3 \times 0.3 \text{ mm}^2$ area. As was mentioned in the previous section, a paraboloid as a premirror followed by a paraboloid as a condensing element is a desirable choice, except for the high cost. The results of ray tracing for such a system with a source to premirror distance of 5.2 m, providing a σ_v of 13 arc sec at the

monochromator, and post mirror to sample distance of 13.3 m is shown in Fig. 3. This configuration gives a magnification of the source by a factor of 2.6, which provides the proper uniformity ($2\sigma \geq 2.1$ mm for a Gaussian distribution) in the horizontal plane, but does not provide this uniformity in the vertical. The vertical uniformity could be enhanced by an asymmetric crystal monochromator with a magnification of 2 or so.

An alternative to the double paraboloid configuration is a parabola followed by a bent cylinder. This geometry has been studied with ray tracing in both the coma cancelling arrangement (both mirrors deflecting in the same direction) and for the two deflections in the opposite directions which makes the final exit beam horizontal. The results of the ray tracing are shown in Fig. 4a and b for these two cases, respectively. It can be seen that there appears to be no advantage to the coma cancelling geometry at these grazing angles. In both cases the angular spread incident on the monochromator is 10 arc sec and the beam uniformity is within 1% over a $0.3 \times 0.3 \text{ mm}^2$ illuminated area. It should be noted that the cylinder radius in the vertical plane is calculated neglecting the presence of the parabolic pre-mirror (sphere in the present case) which provides an additional factor of two or so to the vertical beam size at the sample. That is enough to give the uniformity as required.

FINAL DESIGN

Considering the results of the ray tracing and the requirements of the beam line the final design configuration consists of a spherical collimating mirror, followed by a two-crystal nondispersive monochromator and finally by a bent cylindrical mirror. The two mirrors are arranged so as to produce a horizontal exit beam and this geometry should not provide any adverse effects on the optical performance. The two mirrors will be coated with Rh and set at a 3.0 mrad grazing angle, which is equal to the critical angle of total external reflection at $\lambda = 0.6 \text{ \AA}$. This provides two important features: (1) Only 2% of the incident x-ray power is absorbed by the first mirror, and the second mirror is only illuminated with monochromatic light and (2) The rhodium L edges are at $\lambda > 3 \text{ \AA}$ and the K edge is at $\lambda < 0.6 \text{ \AA}$ so the entire usable spectrum is covered without crossing any edges in the reflecting optics. The beam line will

collect typically 3 mrad of horizontal divergence and provide an output convergence of roughly 40 arc sec vertical at the sample. This design configuration should provide a simple beam line from an operational viewpoint because only the monochromator will have to be scanned to tune over the entire spectral range required.

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FIGURE CAPTIONS

- Fig. 1a. The $2\sigma_v$ values of the photon opening angle vs. photon energy for the NSLS x-ray ring at 2.5 GeV electron energy.
- 1b. Energy resolution vs. photon energy for several Si reflections from the spread in opening angle only.
- Fig. 2. Variation of vertical collimation $\Delta\theta_p$ as a function of horizontal acceptance, ω_h , horizontal source size, S_h , focal length, F_1 , and grazing angle θ for a paraboloidal mirror.
- Fig. 3. Equi-intensity contours at $z = 22$ m for a beam line with a paraboloidal mirror M1, $F_1 = 5.2$ m and paraboloidal mirror M2, $F_2 = 13.3$ m arranged in the coma cancelling geometry. Both mirrors are set with a grazing angle of 3.0 mrad and accept 3.0 mrad of horizontal divergence.
- Fig. 4a. Equi-intensity contours for the coma cancelling arrangement of parabolic (sphere) M1, $F_1 = 5.2$ m and bent cylinder M2, $F_1 = 3.7$ m, $F_2 = 13.3$ m set at a grazing angle of 3 mrad for both mirrors, and accepting 3.0 mrad of horizontal divergence.
- 4b. Same mirrors as in (a) arranged in the opposite sense to provide a horizontal exit beam.

Table I. Intrinsic Resolution for Several Bragg Reflections from
Silicon and Germanium

<u>Reflection</u>	<u>$\Delta E/E$</u>
Si(111)	1.33×10^{-4}
Si(220)	5.6×10^{-5}
Si(440)	8.9×10^{-6}
Si(880)	6.6×10^{-7}
Ge(111)	3.2×10^{-4}
Ge(220)	1.5×10^{-4}

Fig. 1

Fig. 2

Fig. 3

Fig. 4a

Fig. 4b