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Monochromators and Analyzers**

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High Resolution Bragg Focusing Optics for Synchrotron Monochromators and Analyzers

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ABSTRACT

A number of different applications for high resolution Bragg Focusing Optics are reviewed. Applications include Sagittal Focusing, Energy Dispersive optics for x-ray absorption and diffraction, a curved analyzer-multichannel detector method for efficient acquisition of powder and small angle scattering data, the use of Backscattering Analyzers for very high resolution inelastic scattering, and curved crystals for high energy applications.

Keywords: X-ray, Bragg Focusing, Energy Dispersive, High Energy, Sagittal Focusing

1. Introduction

High resolution, Bragg focusing optics are playing an increasingly important role in synchrotron research. Probably their most important application is for sagittal focusing in double crystal monochromators. Here the challenge is to produce a sagittal bend on the order of two meters while keeping the crystal flat in the meridional direction to well within the Darwin width. Single bounce, horizontally focusing polychromators and monochromators are used for energy dispersive and for high energy scattering beamlines. Both one and two dimensional focusing analyzer crystal systems have been developed. Elliptically bent focusing crystals have been used in combination with position sensitive detectors to greatly increase the effective counting rate for powder diffraction and small angle scattering. What all these applications have in common is that the figure of the bent crystal must be very exact, so that slope errors are small, on the order of a fraction of the Darwin width (sub arc second accuracy is required). The crystals can be bent either by mechanically applying the appropriate couples to a the crystal or the crystal (or crystals) can be bonded to a substrate of the proper shape. The latter type has achieved a resolution of 0.65 meV. The non bonded types of crystals have the advantage that the bending radius can be changed so that the same crystal can be used over a range of energies. In this paper we will review some of the main applications of high resolution Bragg focusing optics.

2. Sagittal Focusing vs. Mirror Focusing

Bending magnet and wiggler synchrotron sources produce beams which are extended in the horizontal direction (for a typical beam, 30-100 mm at the monochromator). In order to make maximum use of these beams it is necessary to focus. In figure 1 we show a typical bending magnet or wiggler beamline with several different optics schemes. In the side view we show the typical double crystal monochromator arrangement which allows the beam to be monochromatized over a wide range of energies while keeping the beam fixed in position at the sample. In (b) we show the top view of an unfocused beam illustrating that most of the beam will miss the sample (S). At Argonne National Laboratories Advanced Photon Source (APS), the beamlines are 50-60 m long and the wiggler or bending magnet sources are 2-6 mrad wide. The beam at the sample would be at least 10 cm wide and a typical sample is a few mm wide so most of the beam would not be useful. In (c) we show a sagittal focusing arrangement¹⁻⁵. The second crystal is bent so that the plane of its arc is perpendicular to the direction of the beam. The equation governing the focus is:

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$$\frac{2\sin(\theta)}{R} = \frac{1}{p} + \frac{1}{q} \quad (1)$$

Here θ is the Bragg angle of the crystal, R the radius of the crystal and p is the distance from the source and q the distance to the sample. Since the Bragg angles in a typical double crystal monochromator go from 4° to 45° and p and q are about 30 m at APS, R needs to vary from about 2 to 20 m. Let us contrast this to the case of mirror focusing, the other common way of providing focusing. The same equation governs the horizontal focusing but here the θ would refer to the angle of the mirror which is typically less than 5 mrad so that the radius of the mirror is small, of order 10 cm. This means that a mirror can only focus a much narrower beam than a sagittal crystal since the beams footprint on the mirror must be much smaller than its radius. In an article of this length we can not go into the details of the design and construction of sagittal crystals and benders. The basic difficulty is that the crystal must be kept very flat in the meridional direction (it tends to bend in this direction due to the anticlastic effect) and the bender mechanism must be able to remove twist.

Figure 1. In (a), a side view of a synchrotron beam line where RS is the radiation source which could be a bending magnet, wiggler or undulator, DCM is a double crystal monochromator, and S is the focal point. In (b) a top view is shown, in (c) the second crystal is sagittal focusing and in (d) a mirror is used for focusing.

There are many detailed papers on this subject so below we will only list the advantages and disadvantages of sagittal crystal versus mirror focusing for a synchrotron like APS:

- Mirror can focus about 1 mrad, sagittal crystal about 3 mrad
- Sagittal crystal needs to be adjusted with energy, mirror works at fixed radius
- Sagittal crystal system is mechanically quite complex with several adjustments necessary to focus the beam at each energy.
- Mirror can provide both vertical as well as horizontal focusing
- Mirror provides harmonic rejection
- Mirror system 4-6 times more expensive

3. Energy Dispersive Curved Crystals

For over a decade, the curved crystal monochromator (polychromator crystal) has been employed to select a band of X-ray energies for X-ray absorption spectroscopy studies⁶⁻¹⁰. In figure two we show the usual experimental arrangement.

Figure two: Typical Energy dispersive beam line for x-ray absorption fine structure measurements.

The energy bandpass from the polychromator is given by ⁶

$$\Delta E = E_C \times l \times \cot(\theta_C) \times \left(\frac{1}{R} - \frac{\sin(\theta_C)}{p} \right), \quad (1)$$

where l is the length of the X-ray footprint on the curved crystal, p is the crystal-to-source distance, E_C is the energy at the center of the bent crystal, θ_C is the Bragg angle at the center of the crystal and R is the radius of a bent crystal which can be obtained from $\frac{1}{R} = \frac{\sin(\theta_C)}{2} \times \left(\frac{1}{p} + \frac{1}{q} \right)$, where q is the focusing distance.

At beamline X6A at NSLS the polychromator can focus a range of x-ray energies (~1 keV) to a spot of about 100 microns. This arrangement allows one to take data simultaneously, so it is useful for time resolved experiments. Another use of this technique is in circular magnetic dichroism experiments. Here a magnetic sample is placed in a magnetic field which can be reversed and the beam that is used is taken from above or below the plane of the ring so the beam is circularly polarized. By reversing the magnetic field and taking the difference channel by channel the dichroism can be measured. Since nothing needs to be moved, the ability to take all the data at the same time allows the very small dichroism of transition metals to be observed. In figure 3 we show the data of Gofron et al.^{12,13} Note that dichroism shows that features in the data that are of order 10^{-4} can be resolved.

Figure 3: The x-ray near edge and magnetic circular dichroism at the Fe edge in several Fe-Ni alloys

Another application of this technique which is not so widely known is multiwavelength diffraction. This method has been pioneered by Lee et. al.¹⁴ and it promises to be a powerful technique for anomalous diffraction. In this technique the polychromator is coupled with an energy selecting grid plate. This experiment demonstrates the ability to measure reflections at six different wavelengths and their Bijvoet pairs at the same time. On one diffraction image, it is possible to record both the dispersive and Bijvoet

information from a myoglobin crystal for Multi-wavelength Anomalous Diffraction (MAD) phasing. Here the beam from the polychromator is passed through a grid to select six different energies (figure 4).

Figure 4: The general diffraction geometry and the components for the MAD phasing experiment are shown. The XY plane is the diffraction plane of the polychromator.

The single crystal is rotated and instead of one reflection at a time being recorded, many reflections at six different energies are simultaneously recorded. This can greatly reduce systematic errors. As an example the data from a myoglobin crystal is shown in figure 4

Figure 4. The oscillation diffraction images were taken from a myoglobin single crystal aligned about the b^* , c^* plane with b^* as rotation axis. The pattern was taken with a 4° oscillation and 2 minutes exposure at X6A from six different wavelengths.

4. Curved Analyzer crystals for small angle scattering and powder diffraction

In figure 5A we show a schematic drawing of the optics of a high resolution single channel synchrotron based system.¹⁵ The method uses an unfocused monochromatic beam and a flat diffracted beam monochromator. The method is somewhat limited in flux and is not very good for small samples but has extremely high resolution. Figure 5b illustrates how high resolution data can be taken at much higher count rates¹⁶. The beam from the synchrotron is focused (using a mirror) in order to bring the flux within the field of view of the diffracted beam focusing crystal. It seems possible to bring most of the flux to within

the field of view and with focusing in both the vertical and horizontal directions the count rate will improve by about a factor of 150 as compared to the standard method with only a slight loss of resolution.

Fig. 5 Schematic drawings of synchrotron powder diffraction techniques; A. unfocused x-ray beam and flat analyzer, B. Focused synchrotron beam and focusing analyzer crystal. Note the match between the field-of-view and illuminated area.

The technique is also useful for small angle scattering with high countrate and low background (see Figure 6). We also show data taken on a material with a large unit cell , Silver Behenate in figure 7. Both sets of data were taken at Beamline X6B at NSLS ¹⁷

Figure 6: Data taken on a Standard material, for small angle scattering , Chick collagen. These data were taken in 40 seconds.

Figure 7: Data taken on a Standard material, Silver Behenate illustrating the low background and efficiency of the focusing analyzer method. These data were taken in less than one hour of beam time.

5. Backscattering Analyzers for very high resolution inelastic scattering

The studies of electronic and lattice excitations require very high resolution for both monochromators and analyzers. The problem of an efficient monochromator is not as difficult as the analyzer since the beam from the synchrotron (usually an undulator) is so well collimated that either multiple flat very asymmetric crystals¹⁸ or a focusing backscattering analyzer can be used. The scattering from the sample is not well collimated so for the analyzer to be efficient it must accept a large solid angle. In order to get high resolution (meV) one must use high orders of reflection of very perfect crystals and one must use a crystal at very near backscattering. The only material currently available to make crystals from is float zone Si. The resolution of a perfect crystal is given by

$$\frac{\Delta E}{E} = \sqrt{\frac{\cos(\theta - \chi)}{\cos(\theta + \chi)}} (4r_e d^2 / \pi V_c) CF$$

Here χ is the angle between the planes and the surface normal, $r_e = e^2 / mc^2$, the classical electron radius, d the spacing of the planes, V_c the volume of the unit cell, C the polarization factor and F the real part of the structure factor. Empirically for odd index planes, $\frac{\Delta E}{E}$ varies approximately as d to the 2.5 power. The angular resolution required of the optic is;

$$\Delta\theta = \tan\theta \frac{\Delta E}{E}$$

so for θ near 90° , $\tan\theta$ is very large so $\Delta\theta$ can be reasonably large even when $\frac{\Delta E}{E}$ is very small. The

highest resolution work has been done by the group at ESRF¹⁹⁻²⁰ where they constructed a system capable of resolutions of 1-5 meV depending on the crystal orientation. These resolutions are good enough to study photon physics. In figure 7 we show a diagram of this type of beamline.

Figure 8: A high resolution beamline using a backscattering monochromator and a backscattering focusing optic.

The key component of this type of beamline is the focusing optic. In a backscattering optic the strain is the principle problem. Bending the crystal produces strain so for the highest resolution the crystal (or crystals) must be mounted strain free. The Sette group at ESRF has made focusing optics with approximately 12000 small squares of strain free crystal all aligned to within the rocking curve width. There is, of course, a trade

off between resolution and flux, and this group has achieved resolutions of 5.0 meV with Si(9,9,9), 3.2 meV with Si (11,11,11) and 0.65 meV with Si (13,13,13) with reasonably large solid angles. In the lower resolution regime but with much higher flux, Macrander *et al.*²¹ has used spherically bent Ge (4,4,4) crystals and have achieved a resolution of 94 meV. This resolution is very useful to investigate low energy electronic excitations.

6. High Energy Applications

With the advent of the new third generation sources large fluxes of high energy radiation have become available. Important applications include Compton scattering and high energy diffraction. Since at high energy the Darwin widths of crystals are very small even a small amount of strain or bending makes the crystal diffract kinematically which means that the energy and angular band width can be tailored to the application by introducing a controlled amount of strain. P. Suortti and his coworkers²³ have written a series of papers discussing this subject so we will just show how the energy resolution depends on bending and thickness of the crystal. Since Si becomes quite transparent at high energies bent Laue crystals can have reflectivities near unity, so depending on the application, either a Laue or Bragg optic should be considered. For the Bragg case at high energies only the term which comes from the change in Bragg angle as the x-rays pass through the crystal is important and the total energy width is given by;

$$\frac{\Delta E}{E} = \cot \theta (T/R) \tan(\chi + \theta)$$

Here T is the thickness of the crystal and R is radius bend. At low Bragg angles and small or no asymmetry, $\chi \approx \pi/2$ we have;

$$\frac{\Delta E}{E} \approx \frac{T}{L\theta};$$

so for T of order 1 mm, and L about 30 m, about 3° , $\frac{\Delta E}{E}$ is about 0.05%. This is a very reasonable resolution for many experiments and this relatively large band width means that the flux can be quite high. As an example we show a schematic drawing of a high energy beam line that could be used for Compton scattering.

Figure 9 A Bragg scattering high energy beamline for Compton scattering.

SUMMARY

The above applications show the importance of focusing Bragg optics to efficiently utilize synchrotron radiation. The availability of large perfect Si and the relative ease of manufacture should make applications even more wide spread in the future.

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