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Fluorescence Microscopy and Other Applications

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X-RAY OPTICS FOR SCANNING FLUORESCENCE MICROSCOPY
AND OTHER APPLICATIONS.

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ABSTRACT

Scanning x-ray fluorescence microscopy is analogous to scanning electron microscopy. Maps of the distribution of chemical elements are produced by scanning the specimen with a very small x-ray beam while collecting the XRF spectrum. Our goal is to perform such scanning microscopy with resolution in the range of <1 to $10\text{ }\mu\text{m}$, using standard laboratory x-ray tubes. In order to increase the radiation flux on the specimen, we are investigating mirror optics in the Kirkpatrick-Baez (K-B) configuration. K-B optics uses two curved mirrors mounted orthogonally along the optical axis. The first mirror provides vertical focus, the second mirror provides horizontal focus. We have used two types of mirrors: synthetic multilayers and crystals. Multilayer mirrors are used with lower energy radiation such as $\text{Cu K}\alpha$. At higher energies such as $\text{Ag K}\alpha$, silicon wafers are used in order to increase the incidence angles and thereby the photon collection efficiency. In order to increase the surface area of multilayers which reflects x-rays at the Bragg angle, we have designed mirrors with the spacing between layers graded along the optic axis in order to compensate for the changing angle of incidence. Likewise, to achieve a large reflecting surface with silicon, the wafers are placed on a specially designed lever arm which is bent into a log spiral by applying force at one end. In this way, the same diffracting angle is maintained over the entire surface of the wafer, providing a large solid angle for photon collection.

INTRODUCTION

Radiography is a widely used technique which readily shows flaws such as cracks, voids, inclusions, or density variations in materials and fabricated devices. In many of our applications, it is desirable to augment radiographic information with knowledge about the distribution of chemical elements such as that provided by x-ray fluorescence. Images of the distribution of elements can be obtained by scanning the specimen with a beam of electrons or other particles, or with x-rays. X-ray beams have advantages compared to particle beams such as: 1) low thermal loading of the specimen; 2) the specimen can be non-conducting; 3) the specimen can be examined in air to avoid loss of volatile components; 4) subsurface details can be imaged because x-rays penetrate some distance below the surface; and 5) minor and trace elements can be imaged because the signal to noise ratio is better. A simple technique is to collimate the x-rays to provide the resolution required. However, if the resolution needed is below 10's of micrometers, the time required becomes many hours for only moderately sized specimens when x-ray tubes rather than synchrotrons are used for excitation. Therefore, something more efficient than simple collimation is required for routine high resolution chemical imaging.

In order to increase resolution and imaging speed, we need to collect x-rays with the largest possible solid angles and focus them onto the smallest possible spots. Besides the primary aim of performing fluorescence imaging, related techniques such as transmission, backscatter, and even acoustic imaging (with a modulated beam) may be feasible. Additionally, fan beams (e.g., from a single mirror) can be used in radiography to reduce out of plane scatter. Fan beams and parallel beams can be used in tomography. We are also aware that the ability to magnify and demagnify objects through the use of x-ray optics can impact related fields such as projection imaging and lithography.

Scanning microscopies can be accomplished by a variety of means. Coded irradiation followed by image reconstruction can be used.¹ If the more direct method of raster scanning is employed, several choices can be made. A simple collimated beam can be used.² A more efficient means is to guide the x-rays to the specimen using hollow fibers^{3,4} or fiber bundles.⁵ Fresnel transmission optics have been used to advantage to focus x-rays,⁶ even at energies up to 20 keV.⁷ We have chosen to concentrate on Kirkpatrick-Baez (K-B) mirror optics^{8,9} because this system is relatively easy to fabricate, it may have high efficiency, and it has desirable optical features.

DISCUSSION

A schematic diagram of K-B optics is shown in Figure 1, with an actual device shown in Figure 2. With the K-B arrangement, we can minimize astigmatism by separately adjusting the focus in the vertical and horizontal planes. (Some astigmatism remains when spherical rather than elliptical mirrors are used.) There is no theoretical limit to the size of the mirrors, so efficiency can be increased with only practical considerations in mind. The mirrors themselves can use

either total specular reflection at longer wavelengths, Bragg reflection from multilayers at intermediate wavelengths, and Bragg reflection from crystals at shorter wavelengths. By careful design of multilayers on silicon wafers, all three ranges of wavelengths can be accommodated with a single mirror system. At longer wavelengths, the mirror behaves only as a smooth surface and total reflection results. At intermediate wavelengths, the multilayer is operative. At yet shorter wavelengths, the x-rays penetrate the multilayer and are diffracted by the silicon interatomic planes.

The focusing of rays is described by the Coddington equations⁹. The focus for tangential rays (in the plane of incidence), is

$$\frac{1}{f_t} = \frac{1}{u} + \frac{1}{v_t} = \frac{2}{R_t \sin \theta}$$

while the focus for sagittal rays (perpendicular to the plane of incidence) is

$$\frac{1}{f_s} = \frac{1}{u} + \frac{1}{v_s} = \frac{2 \sin \theta}{R_s},$$

where θ is the glancing angle of incidence (given by the Bragg equation $n\lambda = 2d \sin \theta$ for diffracting media), f is the focal length, u is the x-ray source to mirror distance, v is the mirror to focus distance, R is the radius of curvature of the mirror, and the subscripts t and s designate tangential and sagittal rays, respectively. The magnification factor for the focal spot M is equal to v/u . As an example, consider $\text{CuK}\alpha_1$ ($\lambda = 1.5406 \text{ \AA}$), a multilayer mirror with a period d of $12 \text{ \AA}$,

Figure 1. Schematic diagram of Kirkpatrick-Baez optics, showing the central ray. Concave mirrors produce images of extended objects at small incidence angles⁸.

$R_t=1000\text{mm}$, $u=353\text{mm}$,
 $v_t=35.3$. There is a 10-
 fold demagnification
 for the tangential
 rays. For the sagittal
 rays to focus at the
 same point, a radius of
 curvature of $R_s = 4.1$
 mm would be required.
 For a spherical mirror
 where R_s and R_t are the
 same, the sagittal rays
 are unfocused, and a
 line image is formed by
 a single mirror. If a
 second mirror is placed
 orthogonally to the
 first and the reflected
 rays intercept it, the
 tangential rays from
 the first mirror become
 sagittal at the second mirror, and the sagittal rays from the
 first mirror become tangential. By appropriate selection of R
 (or θ via the d spacing) for the two mirrors, the beams
 collapse to a point focus.

Figure 2. Kirkpatrick-Baez mirror
 system with benders for silicon
 wafers. The x-ray source is mounted
 on the end of the tube on the left.

In order to design multilayers for the mirrors, we use a
 Fortran program based upon the Fresnel equations which runs
 on a desktop computer.¹⁰ Synthetic multilayers are produced by
 coating an atomically smooth substrate with alternating
 layers of high and low electron density materials with
 interlayer periods as low as 1 nm. We can graphically see the
 reflectivity as a function of angle or wavelength. We can
 study the effects of such parameters as composition, number
 of layers, and the ratio of heavy to light layer thicknesses
 on reflectivity and resolution. Judgements about design
 trade-offs can be made prior to fabrication. In Figure 3, we
 see the effect of multilayer pair composition on resolution.

Another computational aid for mirror design is the ray
 tracing code "Shadow" which was developed at the University
 of Wisconsin Center for X-Ray Lithography.¹¹ A typical pair of
 output graphics is shown in Figure 5, where the improvement
 in focus using optimized geometric figure for the reflecting
 surface is shown. At present, we use spherical substrates for
 our multilayers, because they are readily available. The
 spherical aberration is apparent in Figure 5. Elliptical
 mirrors have been designed but not yet fabricated.

Given the narrow rocking curves shown in Figure 3 for
 multilayer mirrors, diffraction occurs over only a small band

Figure 3. Rocking curves for two multilayer compositions (MoSi₂/Si pair on the left, W/C pair on the right)

Figure 4. D-spacing required to diffract over the entire mirror surface. Designed to produce a parallel beam with Cu K α radiation.

of the mirror surface when the source is at typical distances of 200 to 500 mm. This is because the curvature does not fully compensate for the change of incidence angle along the optic axis. One means to overcome this problem with multilayer mirrors is to vary the spacing between layers so that the Bragg condition for diffraction is satisfied at all points on the surface. A set of curves for spherical optics which satisfy both the Bragg condition and the tangential focusing condition is shown in Figure 4.

Roughly speaking, the interlayer spacing needs to vary by a few tenths of a percent per mm along the optic axis. Such variations are within the capabilities of multilayer fabricators.

For the even narrower rocking curves of crystalline reflectors, another technique must be used to compensate for the angular variation since the interatomic spacing

is not a parameter that can be varied at will. The technique we have selected is to make all the incidence angles identical. The condition of invariant incidence angle is fulfilled for a log-spiral surface, as shown in Figure 6. The log-spiral figure provides 1-to-1 focusing, so we are no longer able to demagnify the anode focal spot as can be done with other figures used with multilayer mirrors. Without demagnification, a smaller focus (lower power) x-ray tube must be used. This drawback of a lower power tube is compensated by both the higher brilliance (i.e., higher heat load per unit area on the anode) and by the greater geometric efficiency due to a higher angle of incidence when using crystal mirrors.

CONCLUSION

We anticipate growing applications for efficient x-ray mirror optics. These applications include scanning x-ray fluorescence imaging (microscopy), analysis of very small objects, fan beam radiography and tomography, and parallel ("pencil") beam tomography. With a parallel beam, long distance alignment problems can also be approached using x-rays.

Figure 6. Side and top schematic views of the bendable wafer support which makes up one mirror of a Kirkpatrick-Baez focusing system.

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