THE DEVELOPMENT OF A MULTILAYER MIRROR SYSTEM USING CuKα AND CrKα RADIATION FOR USE IN MICRO BEAM X-RAY DIFFRACTOMETRY

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ABSTRACT

A new optic system that produces a monochromatic microbeam for both CuKα and CrKα radiation for X-ray diffraction (XRD) has been developed. Multilayer optics technology is a well-known tool to produce a monochromatic micro X-ray beam for XRD. This new optic was combined with a microfocus rotating anode X-ray generator and a cylindrical imaging plate detector to provide a complete dual wavelength system. This optic system provides more than 300 times greater flux at a focal spot size of 10 µm area compared to a conventional sealed tube pinhole camera system.

INTRODUCTION

X-ray diffraction is used to evaluate properties of materials such as lattice parameters, crystallite size, preferred orientation, residual stress, and pole figures. Several two-dimensional detectors have been developed such as the MWPC (multi-wire proportional counter), CCD (charge coupled device) and IP (imaging plate) detector. These dramatically reduce the measurement time and provide two-dimensional information from specimens [1,2]. The study of new electronic devices requires a microbeam XRD system, but the conventional method of using a pinhole collimator is limited in terms of X-ray intensity. Overnight exposures are typically required to obtain interpretable X-ray diffraction data from areas 10 µm in size or less, and the data is poor compared to that obtained by a multilayer mirror sample.

Recently, multilayer optic technology has been introduced for small area X-ray diffraction experiments, especially for protein crystallography, and current flux levels are similar to a second generation synchrotron bending magnet beam line [3,4]. Current multilayer optics can be used with only one wavelength, typically the characteristic X-ray radiation from a single anode material. Protein crystallography most often utilizes CuKα, but the measurement of the properties of materials often requires both CuKα and CrKα because of material properties such as absorbance and fluorescence and the desired outcome of the measurement. Multilayer optics are suitable for microbeam XRD from the standpoint in that they provide sufficient flux. However, when the anode is changed, the multilayer optic must also be changed to match the radiation. Alignment of a multilayer optic is not trivial, so a new optic, one which works with both CuKα and CrKα, was developed. This new optic confers an advantage of time savings to the experimenter. We chose to use a microfocus rotating X-ray generator with a 70 µm focal spot and a cylindrical image plate detector of radius 127.4 mm to validate the performance of this optic.

The purpose of this study is to demonstrate a multilayer optic system that can be used with both CuKα and CrKα radiation, and to show system performance in combination with a microfocus...
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rotating anode and an IP detector.

INSTRUMENTATION

Dual wavelength (CuKα and CrKα) multilayer optics

The multilayer optic is designed and manufactured by Rigaku Innovative Technologies Inc. (Auburn Hills, Michigan). Depositing alternating layers of light-element and heavy-element-containing materials onto an appropriately curved substrate produces a multilayer optic. The coating used is a proprietary coating. The alternating layers act like the lattice planes in a crystal in that X-rays impinging on a multilayer optic under the conditions of Bragg’s law will produce a monochromatic diffracted X-ray beam. If the layer thickness is varied across a curved substrate, a graded optic can be produced that captures a larger, solid angle of X-rays from the source and produces either a focused or parallel X-ray beam. Figure 1 shows the schematic of a two-wavelength multilayer optic system. It is designed so that the surface reflects CrKα and the deeper layer reflects CuKα radiation. Table I shows design parameters and expected performance comparison between multilayer optics for CrKα and CuKα radiation, and this two-wavelength optics system. For the calculations, we assume the use of a Rigaku MicroMax-007HF operating at 1200 W for Cu and 900 W for Cr radiation, in both cases providing a 70 μm focal spot at a 6° takeoff angle. A description of the models used for the calculations may be found in Verman et al. [5]. The VMHF-Cu and VMHF-Cr optics described in Table I are conventional CuKα and CrKα optics, respectively. The shaded area in the table shows the performance of the dual wavelength optics system. The flux for CuKα, assuming a φ = 0.3 mm collimator for the dual wavelength optic is 2122 Mph/S while the conventional VMHF-Cu optic provides 3432 Mph/S, or 62%. Similarly we predict the dual wavelength optic to provide 79% of the flux for a conventional CrKα optic. The source position and the focus position for both the Cr and Cu surfaces are coincident by design so that the system does not require realignment between wavelength changes.

Table I. Comparison of conventional single wavelength optics for conventional Cu, dual wavelength, and conventional Cr optics, respectively.
Figure 1. Diagram of a single optical system capable of providing two different wavelengths of radiation: Cu*Kα and Cr*Kα.

**System**

Figure 2 shows the optic combined with a microfocus rotating anode X-ray generator, Rigaku RA-Micro7 (MicroMax-007HF), and a cylindrical imaging plate detector, R-AXIS SPIDER. The detector has a large active area providing the simultaneous measurement from -60° to +144° in 2θ. The radius of the cylinder is 127.4 mm and the image plate is 256 × 460 mm. The large aperture of this detector is very helpful for qualitative analysis as will be shown below.
Figure 3 shows the results of data collection using progressively smaller collimators on an Fe powder sample using CrKα radiation with the source operating at 35 kV and 25 mA. Figure 4 displays the results of integration of the Fe(211) Debye ring, clearly demonstrating that a 10 µm sample size is accessible for analysis.

Figure 3. Images of the Fe(211) ring using φ = 100, 50, and 10 µm collimators.

Figure 4. Integrated peak profile of the Fe(211) Debye ring for data collected with the three collimators as shown in Figure 3.
Figures 5 and 6 show the results of the analysis of a sample of bulk aluminum using a 10 \( \mu \text{m} \) collimator. Four experiments were performed: a 5 min exposure with rotating anode and CMF at 40 kV and 30 mA [Figure 5(a)], and three exposures of 5 min [Figure 5(b)], 120 min [Figure 5(c)], and 2400 min [Figure 5(d)] using a fine-focus sealed tube source and monochromator at 40 kV and 36 mA. The Debye rings were integrated and the intensity ratios calculated for the (111), (200), (220), (311), and (222) reflections (Tables II and III). The ratios are consistent between the two comparisons but it should be pointed out that crystallite size does cause some variation in the internal of consistency of the ratios for some reflections.

Figure 5. Diffraction patterns for bulk Al using the rotating anode/CMF system and a fine focus sealed tube with monochromator at various exposure times.

Figure 6. Results of the integration of the Debye rings; black line: CMF 40 kV, 30 mA, exposure 5 min; blue line: sealed tube/monochromator 40 kV, 36 mA, exposure 2,400 min; red line: 120 min; green line: 5 min.
Table II. Comparison of integral intensity between CMF 5 min exposure data and monochromator 120 min exposure data.

<table>
<thead>
<tr>
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<th>A: CMF (5 min)</th>
<th>B: CMF(Calc 120 min) A*24</th>
<th>C: monochromator</th>
<th>D: ratio B/C</th>
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<tr>
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<td>590.8</td>
<td>519.2</td>
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<tr>
<td>(311)</td>
<td>11191.4</td>
<td>268592.6</td>
<td>750.8</td>
<td>357.8</td>
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<tr>
<td>(222)</td>
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<td>32858.6</td>
<td>186.6</td>
<td>176.1</td>
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<tr>
<td>Average</td>
<td>364(180)</td>
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Table III. Comparison of integral intensity between CMF 5 min exposure data and monochromator 2,400 min exposure data.

<table>
<thead>
<tr>
<th></th>
<th>A: CMF (5 min)</th>
<th>B: CMF(Calc 2400 min) A*144</th>
<th>C: monochromator</th>
<th>D: ratio B/C</th>
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<tr>
<td>Average</td>
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</table>

Figure 7. Cu wire bonding analysis using Cu φ = 30 μm microbeam (photograph of specimen and two-dimensional diffraction images).
Photographs of the specimen with measured points and diffracted images at each position are shown in Figure 7. The specimen is a Cu wire bond, and the measurement positions were incremented in 50 µm steps from position #1 to #5. For this experiment, the X-ray source was CuKα operating at 1200 W and collimator size was φ = 30 µm. Using the dual wavelength optic, the exposure time is just 60 s. Figure 8 shows the 2θ intensity data converted from images #1 to #5. In #1, #2, and #3, diffraction from the underlying Cu material was clearly observed, but in #4 and #5 diffraction from Cu was not observed, meaning the X-ray beam was small enough to probe only the desired area.

![Figure 8. 2θ intensity data from Cu wire bonding specimen (position #1 to #5).](image)

**CONCLUSION**

The evaluation of a dual wavelength multilayer optic system for use with CuKα and CrKα radiation was carried out and has been shown to provide a high-flux beam for both anode materials. This new optic, coupled with a microfocus rotating anode generator, reduced exposure times dramatically and permitted the study of areas on the order of 10 µm in minutes rather than hours compared to a conventional fine-focus sealed tube system with a graphite monochromator.

**REFERENCES**