Multilayers and crystal for a multi-bandpass monochromator

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ABSTRACT

VESPERS beamline is a hard X-ray microprobe beamline dedicated to micro-diffraction and micro-fluorescence analysis at the Canadian Light Source; it requires multi-bandpass X-ray beams for different types of samples and experiments. A specially designed double crystal/multilayer monochromator was built for this purpose with three different bandpasses: 0.01%, 1.6% and 10%. The diffraction elements used for the monochromator have a triple-stripe design using Si(111) crystal as a substrate with two differing stripes of Mo/B_4C multilayers deposited on. The uncovered Si(111) section provides a 0.01% bandpass, while the periodic and depth-graded Mo/B_4C multilyers provide 1.6% and 10% bandpasses, respectively. This paper outlines the requirements and specifications of the diffracting elements as well as the design, deposition and optimization of the multilayers. The performance of the deposited multilayer structures has been tested using Cu- Kα radiation line with a Huber diffractometer.

Keywords: Synchrotron radiation, beamline, X-ray, microprobe, monochromator, multilayer, crystal, bandpass, reflectivity, diffraction

1. INTRODUCTION

VESPERS (VEry Sensitive Elemental and Structural Probe Employing Radiation from a Synchrotron) beamline is one of the Phase II beamlines at the Canadian Light Source, whose construction is completed and has now moved into the commissioning phase. It is a hard X-ray microprobe beamline with a photon energy range of 7-30 keV. The beamline is dedicated to micro-diffraction and micro-fluorescence analysis for materials as different as metals and minerals [1,2]. Fig. 1 shows a schematic layout of this beamline.

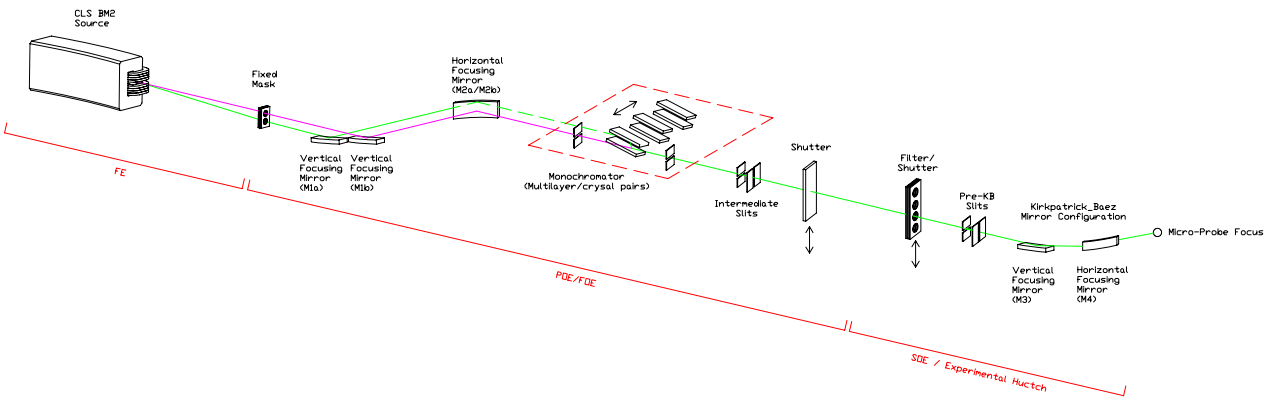

Fig. 1. Beamline layout of the VESPERS beamline at the Canadian Light Source. A multi-bandpass monochromator is indicated by a dashed lined box.

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Laue diffraction and X-ray fluorescence (XRF) spectroscopy are the principal techniques employed on this beamline for structural and elemental analysis, respectively [1,2]. To simplify the Laue diffraction analysis of crystals of varying degrees of symmetry and to provide optimum XRF excitation for many matrices [2], X-ray beams of varying bandpass are desirable. The beamline is designed to have a capability to produce X-rays with several widely differing bandwidths, 0.01%, 1.6%, 10%, as well as a fully polychromatic beam. The availability of 0.01% bandpass also allows the absorption edge of a particular element to be scanned to provide X-ray absorption spectroscopic (XAS) information on the sample [1,2].

A double crystal/multilayer monochromator (see Fig. 1) was specially designed and built to provide multi-bandpass X-rays with the bandwidths of 0.01%. 1.6% and 10%. The present work describes the optics, i.e., diffraction elements, used for this monochromator. The design and specifications will be discussed. Deposition and optimization of the multilayers will be presented together with the performance test using Cu- Kα radiation line with a Huber diffractometer.

2. CRYSTAL AND MULTILAYERS

2.1 General design and specification

To achieve a multiple bandpass over entire energy range, Si(111) crystal, a periodic structure multilayer, and a depth-graded multilayer have been selected to provide 0.01%, 1.6% and 10% bandpass conditions. To simplify the mechanical structure of the monochromator, we introduced a *three-in-one* diffracting element design as illustrated in Fig. 2. Two multilayers: one periodic structure and one depth-graded structure were deposited as narrow stripes onto a super-polished Si(111) crystal. The uncovered Si(111) portion of the crystal provides 0.01% bandpass, while the periodic and depth-graded multilayers provide 1.6% and 10% bandpasses, respectively. The bandpass is changed from one setting to another by lateral translation of the monochromator across the path of the incident X-ray beam.

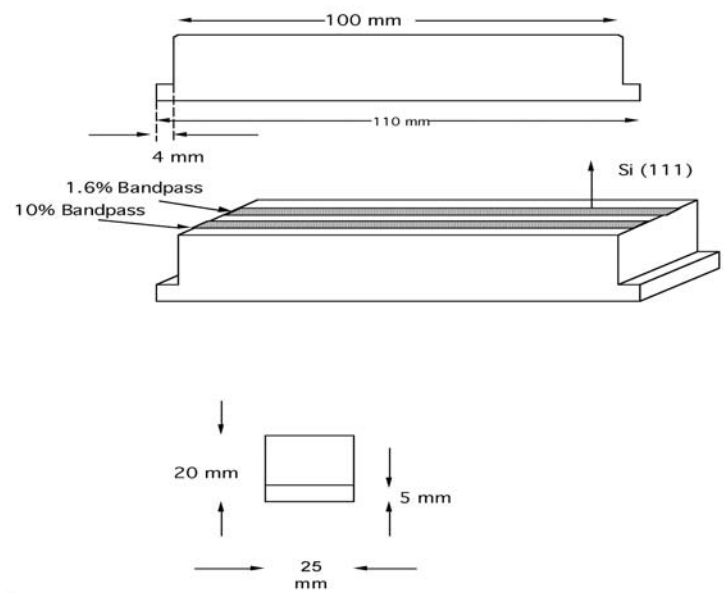

Fig. 2. Si(111) crystal substrate with two multilayer stripes deposited on. The uncovered Si(111) section provides 0.01% bandpass, while the periodic and depth-graded multilayers provide 1.6% and 10% bandpasses, respectively.

The energy range of the beamline is ~7-30 keV and the corresponding angular range of the monochromator is 0.3-17˚; it is controlled by a sine bar drive. To cover the entire energy range, the multilayers have to able to be illuminated at a very small incident angle (as small as 0.3˚) because of a relatively large d-spacing of available multilayers. By considering different pairs of materials and their d-spacing, Mo/B_4C bi-layers were chosen with average d-spacing of 3.2 nm (for

1.6% bandpass) and 3.4 nm (for 10% bandpass). Both structures used 150 bi-layers. Design of the depth-graded structure was optimized by using a numerical algorithm [3] that determines the required individual layer thickness in a multilayer stack to produce the desired reflectivity curve at a particular photon energy and incident angle. The depth-graded structure was optimized to yield a Π-shape reflection at 10 keV with an average d-spacing of ~3.4nm. The shape of the reflectivity curve changes with photon energy because of the optical properties of the materials. This can be seen in Fig. 3, where the calculated reflectivity of the depth graded Mo/B_4C structure is determined as a function of photon energy. Thickness of the individual layers can be seen in Fig. 4. Expected performance of the periodic Mo/B_4C multilayer is shown in Fig. 5. It can be seen from these figures that the peak reflectivity in the range of 5 keV to 30 keV is 30%-40% for the depth-graded structure and 50%-90% for the periodic multilayer.

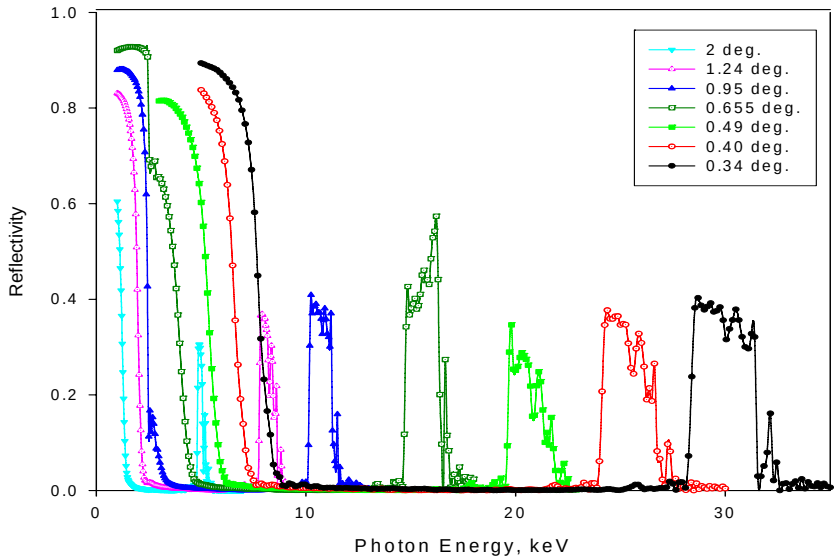

Fig. 3. Calculated reflectivity of the depth-graded Mo/B_4C multilayer for energies ranging from 6-30 keV. The average d-spacing is 3.4 nm.

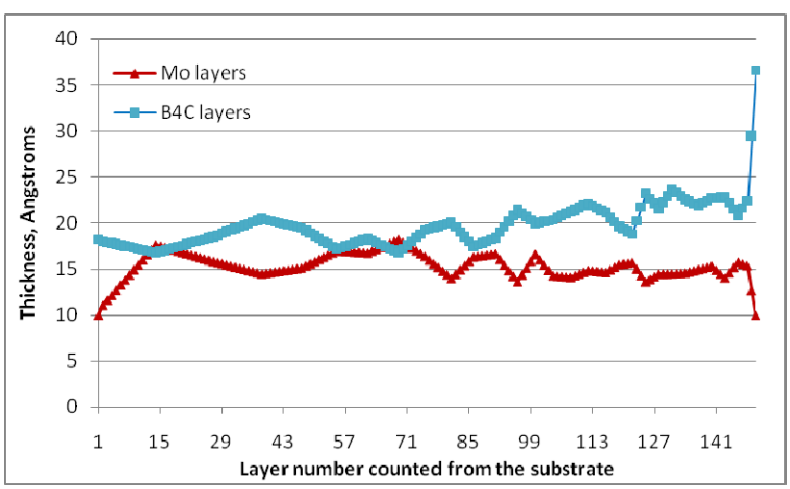

Fig. 4. Thickness of individual Mo and B_4C layers in the depth-graded Mo/B_4C structure.

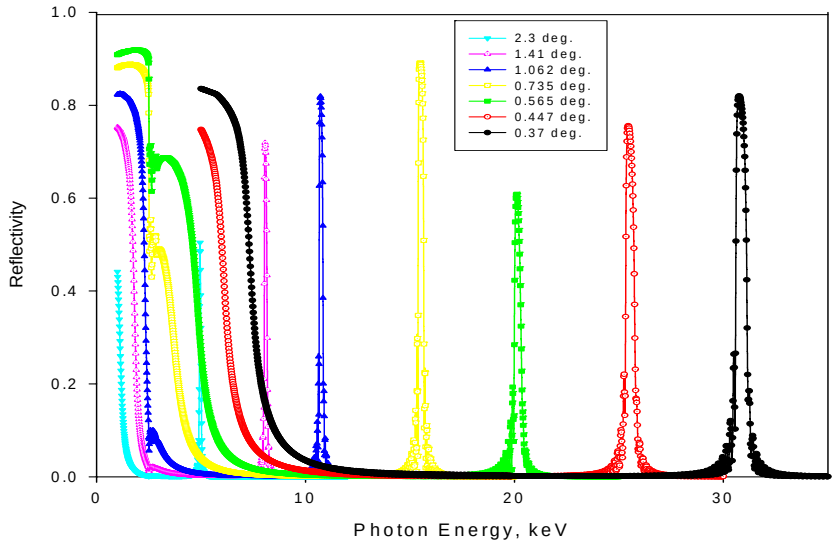

Fig. 5. Calculated reflectivity of the periodic Mo/B_4C multilayer between 6-30 keV. The average d-spacing is 3.2 nm.

2.2 Si(111) crystal substrate

The crystal substrates (see Fig. 2) were manufactured from float zone single-crystal Si. The Si(111) Bragg planes are parallel to the substrate surface within 0.1˚ [4]. The substrate surfaces were super-polished to achieve both low High Spatial Frequency Roughness (HSFR) and small figure errors (FE). Performance of the Si(111) crystals was tested by measuring the crystal rocking curve at the Cu-Kα (8.05keV) radiation line using a double axis diffractometer with the symmetric Si(111)-Si(111) setting. The FWHM widths of the rocking curve measured are 8.0±0.2 arc seconds, which meet the specifications of less than 10% greater than the theoretical width 7.6 arc seconds.

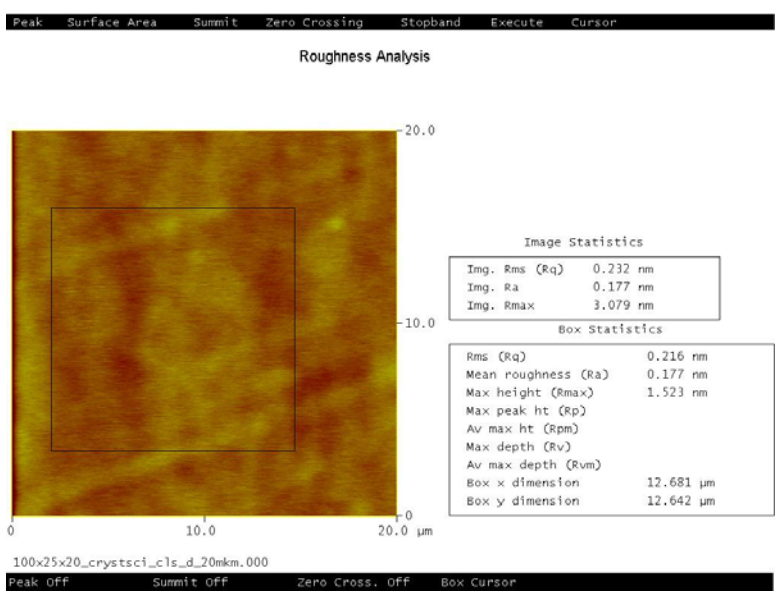

Fig. 6. A typical AFM image of the Si(111) crystal substrate surface in a field of 20µm×20µm.

Surface roughness of the crystal substrates was measured by MicroXAM. The measurements were made with 20× objective in a field of 412μm × 312μm and were taken over five different positions uniformly distributed over the entire surface. The surface roughness determined were all below 0.16 nm rms. HSFR of the Si(111) surface was measured by atomic force microscopy (AFM) in 1μm × 1μm and 20μm × 20μm areas. A typical AFM image and average roughness values are shown in Fig. 6 and Table 1, respectively. Surface figure error was tested by a Long Trace Profiler (LTP) with 2mm diameter beam. Scan length was 95 mm which is 5 mm less than the entire clear aperture (CA) of the optics. The measured substrate radii of curvature and slope errors are also presented in Table 1.

Table 1. Roughness of Si(111) substrates measured by AFM and surface figure errors measured by LTP.

Substrate	HSFR (1μm × 1μm) rms, nm	HSFR (20μm × 20μm) rms, nm	Radius of curvature, m	Slope error, μrad rms
1	0.10	0.17	23028	1.48
2	0.17	0.22	16745	1.40
3	0.10	0.22	72294	1.94
4	0.16	0.24	52163	2.30

2.3 Deposition of multilayers

A schematic top view of the deposition system is shown elsewhere [5]. The substrate is mounted on the facet of a rotating carousel which passes in front of the magnetron sputtering sources to form an alternating structure. The desired thickness h of the coating of each material is obtained through the control of the rotational period T of the carousel. The rotational period changes with layer index with a set of rotation times for each material.

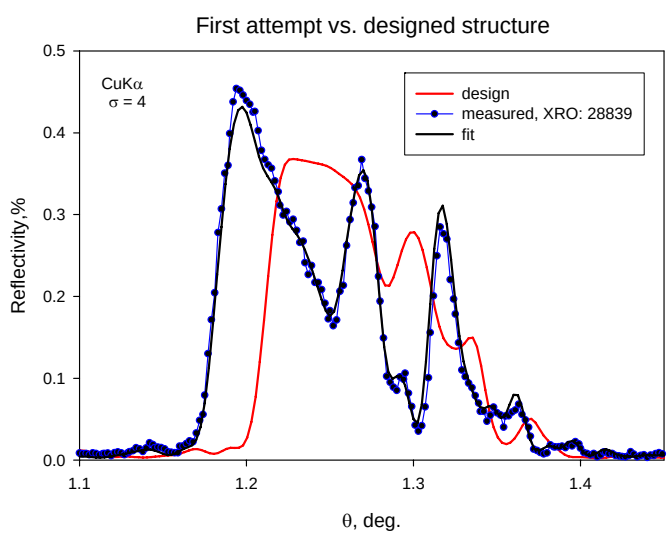

Fig. 7. Measured and fitted reflectivity at Cu-Kα after the first deposition attempt as compared with the design.

During calibration, the dependence of individual layer thickness was determined with respect to the rotation period for each material. Typically, this dependence is linear and can be described as,

$$h(T) = \alpha T + \beta$$

From the linear parameters found experimentally, along with knowledge of the desired thicknesses (Fig. 4), a set of provisional rotational periods were generated for each material. The first deposition attempt was then carried out. In the event that the desired design is not obtained, the actual thicknesses of the layers are determined using a method

described in Ref. [6]. Fig. 7 shows measured Cu-Kα reflectivity obtained from the first attempt of depth-graded multilayer deposition. After applying the method to solve the inverse problem, a new set of α and β parameters were determined then applied for another attempt of depth-graded structure deposition. This cycle can be repeated depending on complexity of the structure. Comparison of the initial and optimized values of α and β for each material is presented in Fig. 8.

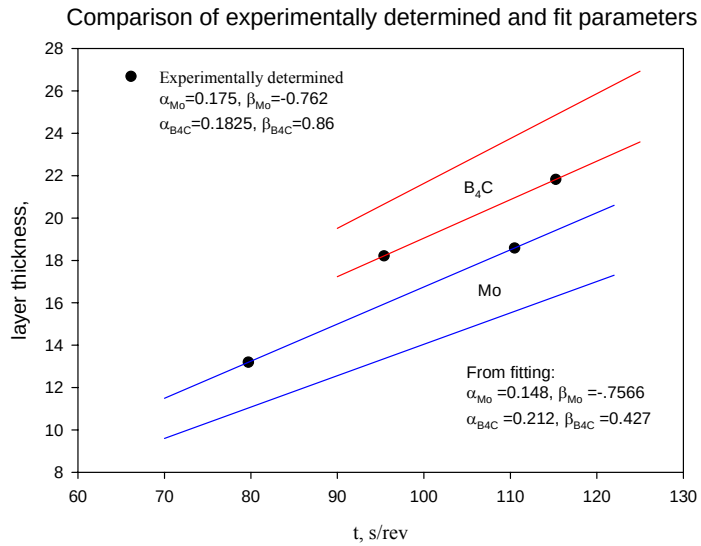

Fig. 8. Comparison of the initial and optimized values of α and β for Mo and B_4C layers.

2.4 Test of the deposited multilayer structures

Two sets of the multilayers were deposited. Each set contained two identical multilayer mirrors. A picture of one of two sets is presented as Fig. 9.

Fig. 9. A picture of one set of three-stripe diffracting elements. Each includes the crystal surface and two stripes of multilayers: one periodic, one depth-graded structure.

X-ray performance of the deposited multilayer structures was tested at the Cu-Kα (8.05 keV) radiation line by a Huber diffractometer using θ-2θ scans with a beam divergence of ~11arcsec. The measurements were performed at three different locations, 15mm, 50mm, and 85mm, along the length of the optics. Figs. 10 and 11 represent the θ-2θ scans for one of each type of multilayers.

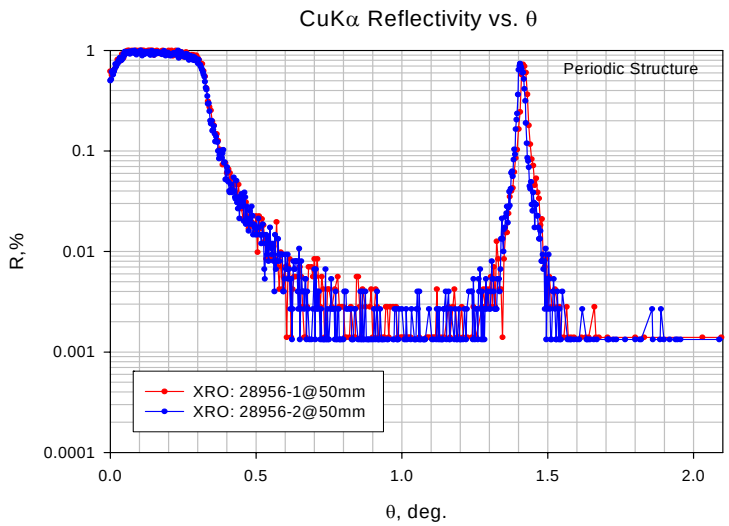

Fig. 10. Cu-Kα reflectivity measurements using θ-2θ scans from the periodic structure Mo/B_4C multilayer.

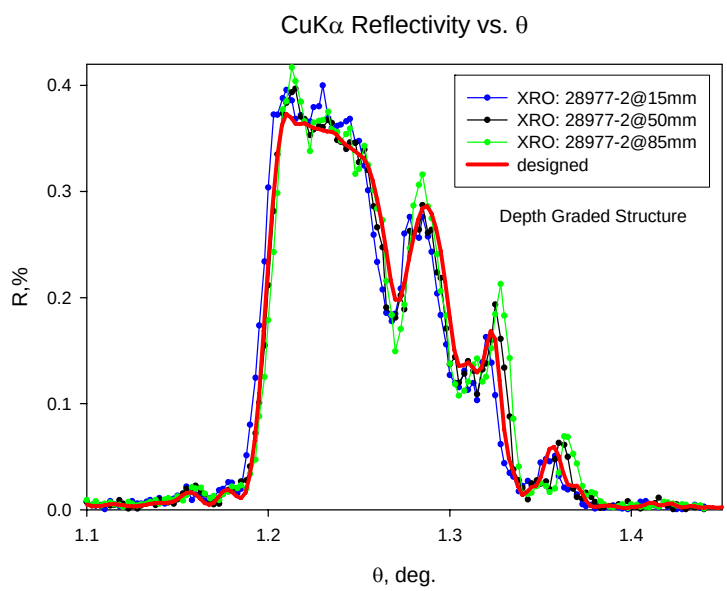

Fig. 11. Cu-Kα reflectivity measurements using θ-2θ scans from the depth-graded Mo/B_4C multilayer.

The reflectivity curves at different points along the mirrors look very similar, indicating good coating uniformity. X-ray performance of both periodic structures and depth-graded structures fit very well with the designed reflectivity curves. This close fit shows that errors in thicknesses of the individual layers are relatively small.

However, an angular shift was observed between designed and experimental curves in another set of the depth-graded structures as shown in Fig. 12. The shift is ~0.01˚. Such shift indicates that the average d-spacing of the depth-graded structure is ~0.03nm less than the designed structure. The mismatch corresponds to ~0.8% of the average d-spacing. Instability of the deposition rate from run-to-run is responsible for this shift in d-spacing. The shape of the experimental curve matches that of the designed curve very well. This indicates that the relative thicknesses of the layers are close enough to the designed structure. In service, this set will work identically to the other set with a small adjustment in theta-to-energy correspondence.

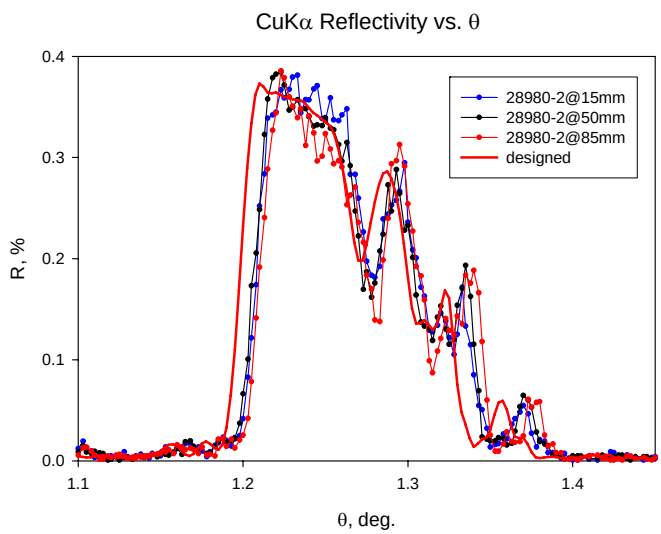

Fig. 12. Cu-Kα reflectivity measurements using θ-2θ scans from one depth-graded Mo/B_4C multilayer, which show an angular shift between designed and experimental curves.

3. CONCLUSIONS

We have developed a triple-stripe diffracting element using Si(111) crystal substrate with two deposited stripes of Mo/B_4C multilayers for a multi-bandpass monochromator on the VESPERS beamline at the Canadian Light Source. The uncovered Si(111) section provides 0.01% bandpass; the periodic and depth-graded Mo/B_4C multilayers provide 1.6% and 10% bandpasses, respectively. The crystal substrates were manufactured and super-polished to meet both crystal and multilayer-substrate specifications. The multilayers were optimized for the integrated reflectivity over the energy range of 10-20 keV with reasonably good efficiency from 6 to 30 keV. The performance of the deposited multilayer structures has been tested using Cu-Kα radiation line with a Huber diffractometer. The test results show a very good agreement to the designed performance.

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