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HAL Id: hal-01702269

<https://hal.science/hal-01702269>

Submitted on 2 Feb 2023

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To cite this article: Fadi Choueikani *et al* 2013 *J. Phys.: Conf. Ser.* **425** 152007

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Preparation for B₄C/Mo₂C multilayer deposition of alternate multilayer gratings with high efficiency in the 0.5-2.5 keV energy range

Fadi Choueikani^{1, 2}, Franck Delmotte¹, Françoise Bridou¹, Bruno Lagarde², Pascal Mercere², Edwige Otero², Philippe Ohresser², François Polack²

¹Laboratoire Charles Fabry, Institut d'Optique, CNRS, Univ Paris Sud
2 Avenue Augustin Fresnel, 91127 Palaiseau Cedex, France

² Synchrotron SOLEIL, L'Orme des Merisiers, Saint-Aubin, BP48, 91192 Gif-sur-Yvette, France

Email : choueikani@synchrotron-soleil.fr

Abstract. This paper presents a study of B₄C/Mo₂C multilayers mirrors with the aim of using it in the achievement of Alternate MultiLayer (AML) grating. Such component allows a high efficiency in the 500-2500 eV energy range for the DEIMOS beamline. Multilayers were deposited on silicon substrate. They are characterized by reflectometry under grazing incidence. Numerical adjustments were performed with a model of two layers in the period without any interfacial. A prototype of AML grating was fabricated and characterized. The efficiency of the first order of diffraction was worth 15% at 1700 eV.

1. Introduction

DEIMOS (Dichroim Experimental Intallation for Magneto-Optical Spectroscopy) is a beamline at the French Synchrotron SOLEIL dedicated to the XMCD (X-ray Magnetic Circular Dichroism) measurements, using soft polarized x-rays. The x-ray source generates photons in the energy range 350-2500eV with variable polarization supplied by HU52 undulator (for more details see ref [1] submitted to this conference). The beam is afterwards monochromatized (resolving power >5000) and then refocalized onto the sample. The energy range covers the principal chemical elements linked to materials showing magnetic properties, namely the first row transition metals $L_{3,2}$ -edges, the rare earth $M_{4,5}$ -edges, but also the K -edges of light elements (C, N, O and S). The end station consists of a superconductive magnet that allows applying a tunable magnetic field of ± 7 T along the photons beam direction and ± 2 T perpendicular to the beam. The sample temperature can be controlled from 1.5K to 350K.

The Variable Groove Depth (VGD) grating which is installed into the monochromator allows an energy working range up to 1500eV. A solution, able to fill the technological break between 1500 and 2500eV, consists in using a two dimensional structure called Alternate MultiLayer grating (AML grating). The AML gratings present a double periodicity at nanometer scale in the plane of the surface and in the perpendicular plane. Therefore, they have properties similar to crystals, with the advantage of the freedom of choosing the periodicities. However, the performance of AML gratings is subordinated to a very definite agreement between the thicknesses of each stacked layer, the optical

index of each material and the etching depth. It is notably affected by the presence of an eventual interdiffusion layer on the interface between two consecutive layers. In this context, this paper is devoted to an experimental study on B_4C/Mo_2C multilayer mirrors for the achievement of AML gratings. The purpose of this study is the control, at sub-nanometer scale, of each layer thickness. Furthermore, it aims at the test of the existence of an eventual interfacial layer in the system that would harm its performance.

2. Alternate Multilayer Grating

The AML grating was fabricated by sputtering deposition of a multilayer coating on a laminar etched grating with a well-defined ratio between depth and lines spacing [1]. The figure 1 presents a cross section of such component. It shows a horizontal periodicity along the x axis which is directly dependent of the period “d” of the laminar grating as well as another periodicity following vertical axis z. The final device will be used in the PGM monochromator of DEIMOS beamline. It will provide a high efficiency in the first diffraction order from 500 to 2500 eV, provided it is set at the corresponding Bragg order of the biperiodic structure. A matched multilayer mirror will compensate its variable deviation angle [2,3]..

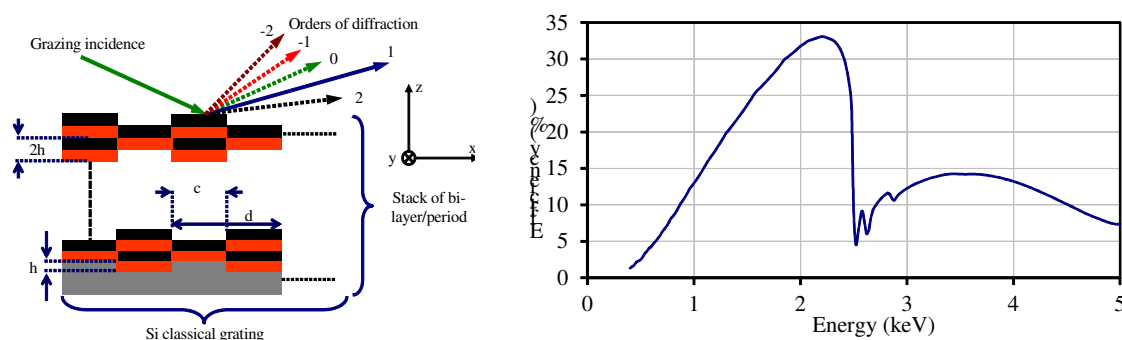


Figure 1. A transversal cross section of AML grating which is constituted of B_4C/Mo multilayer deposited on a laminar grating (figure at left). An example of simulation using the following grating features: 2400 lines/mm, etched depth $h=2.5$ nm, periods along z axis =30, cyclic ratio $c/d=0.5$ (where c is the valley width).

An example of simulation, for a theoretical and perfect model (rectangular profile), with the software “Calcul de Réseaux par Propagation ElectroMagnétique” (CARPEM) [4] is illustrated on the figure 1. It concerns a B_4C/Mo multilayer coated on laminar grating that presents the following features: 2400 lines/mm, 2.5 nm of depth. The figure shows an efficiency of 30 % around 2200eV for the first diffraction order. The efficiency of the other orders is between 10^{-2} and 10^{-1} %. However, the performance of the AML grating is directly dependent on the quality of the laminar grating as well as on the control of the multilayer deposition. In fact, the condition of matching, allowing a strong selectivity and a high efficiency for the selected order, requires the achievement of checkerboards with a strong symmetry.

3. Experiments

A magnetron sputtering deposition system, described elsewhere [5], was used to fabricate B_4C/Mo_2C multilayers. During the process, the chamber of preparation has a nominal vacuum of 5×10^{-8} Torr (mbar). The sputtering was performed with argon gas at a pressure of 2 mTorr. The targets size was 200×80 mm. The radio-frequency power was 150W for B_4C target and the direct current was 0.07 A for Mo_2C targets. In order to obtain a high homogeneity, the samples were rotated, with a velocity of 1°/s, while passing above the targets. The number and the speed of scans determine the thickness of material deposited. Multilayers with different expected thicknesses of B_4C (0.63, 1.25, 2.5 and 5nm) and an expected thickness of Mo_2C layers fixed at 2.5nm, were deposited on Silicon polished

substrates. At the same conditions, samples with an expected thickness of B_4C fixed at 2.5nm and different thicknesses of Mo_2C were also fabricated. The number of periods was 15.

The characterization of multilayers was performed with reflectivity measurements under X-ray grazing incidence. The experimental reflectivity curves were obtained at the Cu K_α radiation of 0.154 nm (~ 8000 eV) using BRUKER-DISCOVER 8 goniometer. In the geometry of the bench, the sample stays immovable. Thus, the measurements were obtained by varying the grazing incidence angle, by moving the arm of source, while hacking the reflected beam (θ - θ scan configuration). The arm of source consists of X-ray tube, a third generation Göbel Mirrors providing the X-ray highest flux density, a divergent slit and a primary Soller slits. The diameter of focalization was around 1mm. The arm of detection consists of secondary Soller slits placed between to divergent slits and the detector.

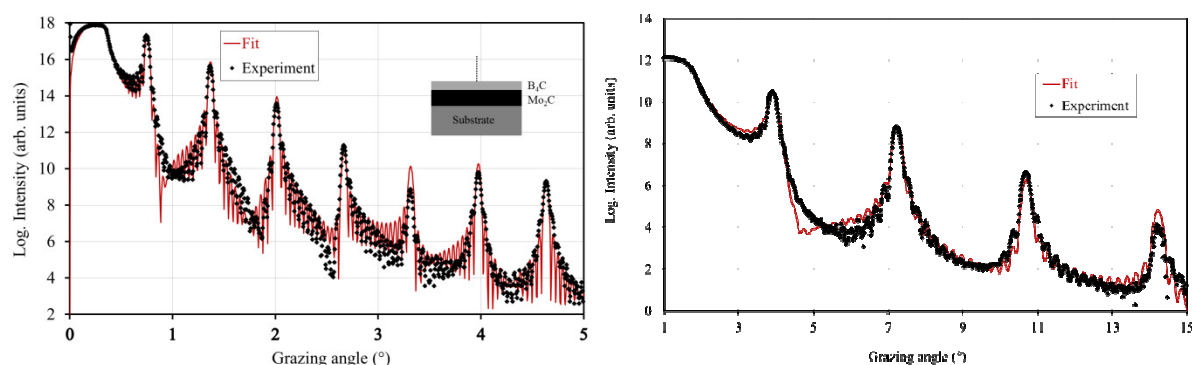


Figure 2. Grazing incidence x-ray reflectivity at 8000 eV (left) and at 1500 eV (right) : experimental measurements (dots) and numerical adjustment (continuous) with a model of two layers in the period. The expected thickness of B_4C and Mo_2C was respectively 5 and 2.5nm. The figure in the inset presents the model of two layers in the period: B_4C/Mo_2C .

The soft X-ray branch of the METROLOGY Beamline of Synchrotron Soleil was also performed to characterize the multilayers [6]. The measurements were obtained by θ - 2θ scan configuration in which the arm of source stays immovable while the sample and the arm of detection are movable. The monochromator used to cover efficiently the entire spectral range is a grating with groove densities of 1200 lines/mm. The spectral range is set to be 400-1700eV. The monochromatic light was then refocused vertically onto the sample using several mirrors. The spectral resolution varied from 4000 (at 400 eV) to 500 (at 1700 eV). The flux was around 4×10^{10} photons/s and the spectral purity was 10^{-3} .

By fitting the reflectivity curve using a trial and error method, the grazing X-ray reflectometry allows the determination of thickness and the interfacial roughness, and, the complex index ($n=1-\delta-i\beta$ where δ is the unit decrement of the refractive index and β is the extinction coefficient) of each of the successive films deposited on the substrate [5].

4. Results and Discuss

Results are summarized in the figure 3. The graph at left presents the evolution of the fitted thicknesses of B_4C and Mo_2C as function of the B_4C expected thickness, for an expected thickness of Mo_2C fixed at 2.5 nm. It shows that the fitted value of Mo_2C thickness (squares) is 2.6 nm which is slightly higher than the expected value (2.5 nm). This graph shows also a linearly variation of the fitted thickness of B_4C (lozenges) as a function of the expected ones. We can also remarks that the fitted thickness of B_4C layer is reduced around 0.5 nm compared to its expected value. The decrement of the refractive index is respectively 2.5×10^{-5} and 0.72×10^{-5} for Mo_2C and B_4C layers.

This behavior was confirmed in the graph at the right of figure 3. This graph presents the variation of the fitted thickness of Mo_2C (lozenges) and B_4C (squares) versus the expected thickness of Mo_2C . The expected thickness of B_4C was fixed at 2.5 nm.

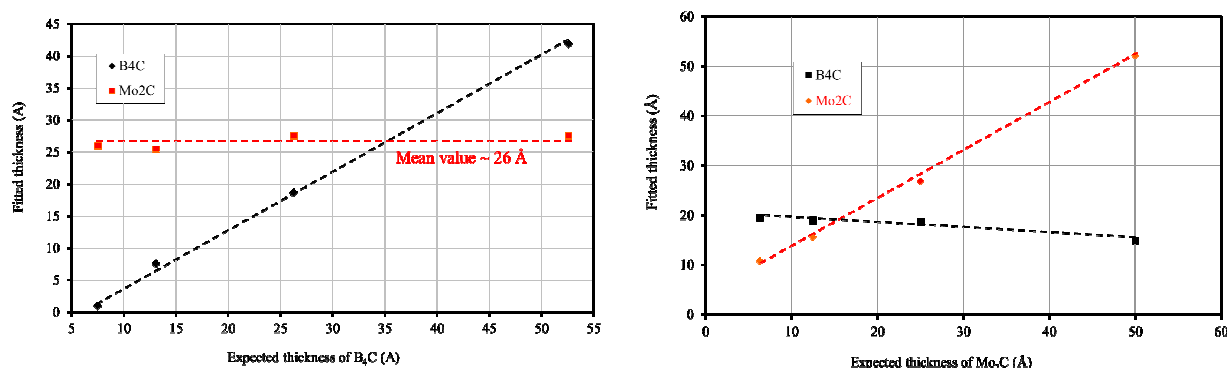


Figure3. Variation of B₄C and Mo₂C fitted thicknesses as function of B₄C expected thickness (left), for a Mo₂C thickness fixed at 2.5 nm. Variation of B₄C and Mo₂C fitted thicknesses versus Mo₂C expected thickness (right), for a B₄C thickness fixed at 2.5 nm.

A prototype of AML grating was fabricated by deposition of B₄C/Mo₂C multilayers on silicon grating. It presents the following features: 2400 lines/mm, $c/d=0.36$, thickness of each layer=2.5 nm. Measurements were performed using the soft x-rays bench of Metrology beamline and the result is illustrated in figure 4. This figure shows efficiency around 15% at 1700 eV. The experimental efficiency value is worth 73% of the theoretical one which was obtained via simulations. This difference is probably due to the real profile of grating which is not rectangular but trapezoidal.

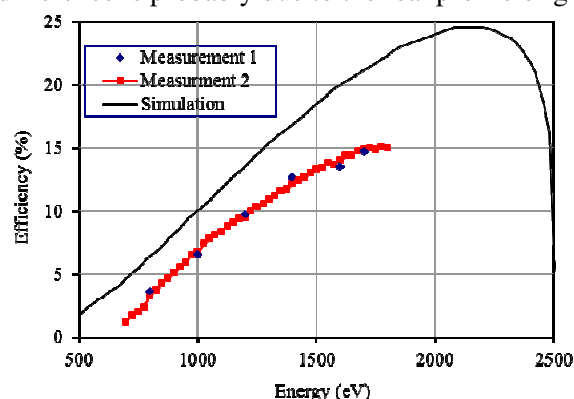


Figure4. Efficiency curve versus photon energy for AML grating prototype that presents the following feature: 2400 lines/mm, $c/d=0.36$, thickness of each layer=2.5 nm.

5. Acknowledgment

This work was supported by a grant from the RTRA (*réseaux thématiques de recherche avancée*) program "Triangle de la Physique"

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