Characterization of Growth Rate and Interfacial Roughness of Multilayer Optical X-ray Coatings

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Abstract

Optical parameters of thin multilayer coatings of tungsten and boron carbide produced by sputtering deposition were investigated in this project using x-ray reflectivity techniques. Observations were made of the influence of changes in power and gas-mixture percentages on the ion bombardment causing interfacial roughness and fluctuations in growth rate analyzed using the X-ray reflectometry and IMD. Growth rate seems to have a proportional relationship with power and interfacial roughness is observed to be minimized at 5% N_2 for 80 W, 160 W and 320 W cathode powers.

I. Introduction

For over three decades, vacuum-deposited optical coatings have been available for modern precision optics. It is important for thin-film engineers to understand the parameters which affect the film thickness and interfacial roughness of the bilayers deposited by a magnetron source. Argonne National Lab produces some of the brightest X-ray beams in the Americas. We are now moving towards coherent light sources thus high quality optics are essential to preserve the coherence and brightness of beams for experiments in the storage rings. Understanding the optimized parameters of a deposition system in crucial in creating high precision optics. In this project thin film deposition and analysis of multilayer optical x-ray coatings are performed at Advance Photon Source at Argonne National Lab.

The purpose of this work is to investigate the sputtering parameters resulting in characterization of the new profile coating system deposition system. The material systems explored in this work are tungsten and boron carbide (B_4C), of which mono and multilayers will be deposited with changing parameters including deposition powers and gas-mixture percentages. The main goal is to characterize interfacial roughness by deposition of multilayer optical x-ray coatings comprised of very similar d-spacings and gamma ratios and entails an exploration of growth environment parameter space of the independent variables.

In order to systematically record how material growth rate is affected by an independent variable, multilayers of 20 and 40 bilayer independent stacks will be deposited on the same silicon substrate as rate test. Multilayers with 100 period will also be deposited on 2mm silicon substrate for the XRR measurement.

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II. Deposition

The work was mainly split into three sections:

- the deposition of the multilayers using the new deposition system
- the X-Ray Reflectivity (XRR) measurements
- and the simulation of the Bragg peaks using IMD

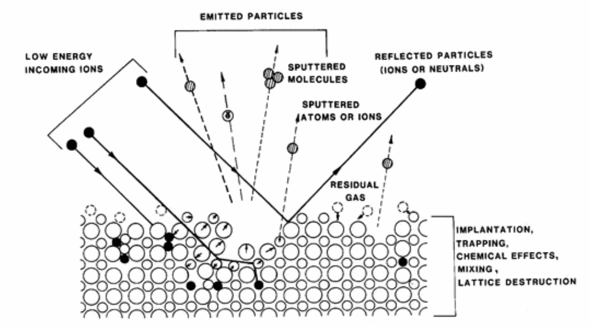

Figure 1: *General ion-surface interaction processes.*

The sputtering system works by creating gaseous plasma of Ar and N_2 in a vacuum condition of 1 mT. Ions are accelerated from this plasma into the negatively charged cathode where the target is located. The targets are tungsten and the $B4C$ in our case. Energy transfers and the target material is ejected in the form of mainly neutral particles and the Silicon substrate will be coated by a thin film of the source material; see Fig. 1. This process is repeated for both tungsten and B_4C targets to create the alternating bilayer thin film on the substrate. The sputtering rate or the deposition growth rate depends mainly on the voltage across the cathode, the ratio of nitrogen ions versus argon ions and distance between the substrate and target. These were the independent variables of the experiment.

The tungsten target is shown in Fig. 2 and the one to the left of it is the B_4C target. Silicon substrates shown in the photo were deposited with mono and multilayers. A rate test was first conducted for all samples to find the growth rate at each condition.

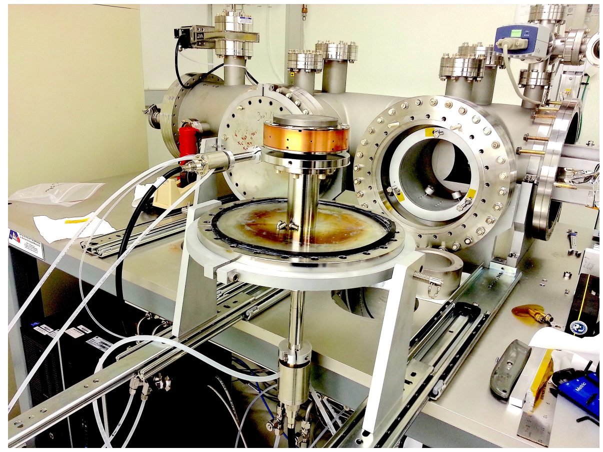

Figure 2: *Vented magnetron sputtering system showing boron carbide target and horizontal substrate transfer motor.*

The rate test involved bottom bilayer composed of 40 cycles of 4 loops of tungsten at 18mm/s and 5 loops of B_4C at 20mm/s. The top bilayer composed of 20 cycles of 2 loops of tungsten at 18mm/s and 5 loops of B_4C at 20mm/s. Based on this knowledge of the growth rates, the real samples can be deposited at different conditions with roughly similar d-spacing for a comparison of reflectivity at different parameters. The final samples consisted of 100 bilayers of the two target materials with a d-spacing of 25 Å.

$$d = \frac{growthrate \times w}{v} \tag{1}$$

Most magnetron sputtering systems have the substrate fixed in the center of the system with allowed rotations to face the target. The new deposition system at Argonne is designed so that the substrate is attached to a high performance servo drive that can move freely in the z-axis to pass in front of the target. The growth rate of the system (1) depends on d which stands for the layer thickness deposited per loop, w the width of the mask in front of the target and v for the motor velocity.

III. X-Ray Reflectometry Measurements

X-ray reflecting multilayer coatings are synthetic structures consisting of alternating layers of two materials with significant differences in scattering power. The structure of these multilayers can be regarded as a one dimensional crystal where a period is equivalent to the thickness of two adjacent layers, analogous to the distance between the atomic planes in crystals. The d-spacing we are looking at in this project ranges from 30 to 100 Å for the rate test and 25 Å for the real samples.

Most technological applications of thin films require films of definite thickness as it influences the reflectivity of the thin film. The thickness of one bilayer of tungsten and B_4C is known as the d-spacing or the period of the multilayer shown in Fig. 3.

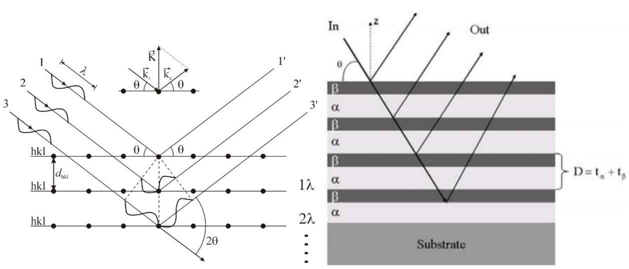

Figure 3: *Illustration of crystal x-ray diffraction structure (left) and multilayer mirror structure (right).*

$$d = \frac{n\lambda}{2sin\theta} \tag{2}$$

When x-rays are scattered from a crystal lattice, peaks of scattered intensity are observed at different angles of scattering. The condition for maximum intensity is contained in Bragg's law (2). This equation allows for calculation of the d-spacing of the optics after determining the wavelength of the incident x-rays and the angle of the maximum constructive interference.

X-ray reflection by multilayer coating is based on the principle of interference. These alternating layers of the two materials, with significant differences in scattering power, create a synthetic structure that behaves like a one-dimensional crystal. XRR method involves monitoring the intensity of the x-ray beam reflected by the sample at grazing angles from -0.1°to 5°. The x-ray reflectometer produces a monochromatic x-ray beam at an energy level of 8.048 keV (wavelength of 1.54 Å) that irradiates the sample at a grazing angle ω and the reflected intensity at an angle 2θ is recorded. Using Bragg?s law, we can find the d-spacing from the angle of the Bragg?s Peak. We can infer the interfacial roughness by simulating the reflectivity of the multilayers.

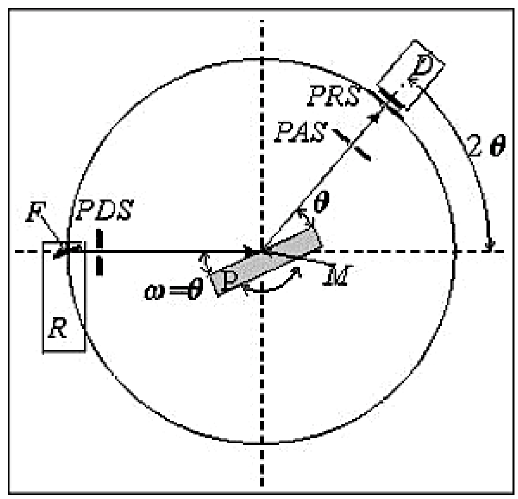

Figure 4: *Theta angle measurements on the XRR system. : $\theta/2\theta$ Scan: The condition of incident angle $\omega = (2\theta)/2 = \theta =$ outgoing angle is satisfied. The detector D rotates at twice the speed of the sample P.*

Investigations of the optical coatings of W and B_4C have shown that changes in power of the cathode and nitrogen ratio indeed led to changes in the coating characteristics. The results of the x-ray refractivity measurements are presented in Table 1. The top graph in Fig. 7 shows the XRR and simulation for one sample where reflectivity was recorded.

Table 1: *Table of Deposition Samples*

Parameters			
Power (W)	N_2 level (%)	Reflectivity	D-spacing
320	0	0.01072	19.6566
320	5	0.43811	27.0698
320	10	0.35557	23.1632
320	20	0.33538	23.3470
320	40	0.38748	25.0708
160	0	0.00001	19.6566
160	5	0.52219	23.8516
160	10	0.34245	21.7147
160	20	0.31669	22.5134
160	40	0.30490	25.7285
80	0	0.04027	20.6688
80	5	0.41066	23.2241
80	10	0.25776	20.6688
80	20	0.06317	18.8590
80	40	0.15673	21.2662

The results from Table 1 can be presented on a 2-D and 3-D plots shown in Fig. 5

In both plots, we can see there is an obvious trend in the reflectivity of the samples with a change in nitrogen values. There seems to be a consistent peak in reflectivity at 5% with a trough at 20%. At the maximum reflectivity there is minimal interfacial roughness. The optimized condition for reflectivity and interfacial roughness would be at 5% N_2 ratio and 160 W cathode power.

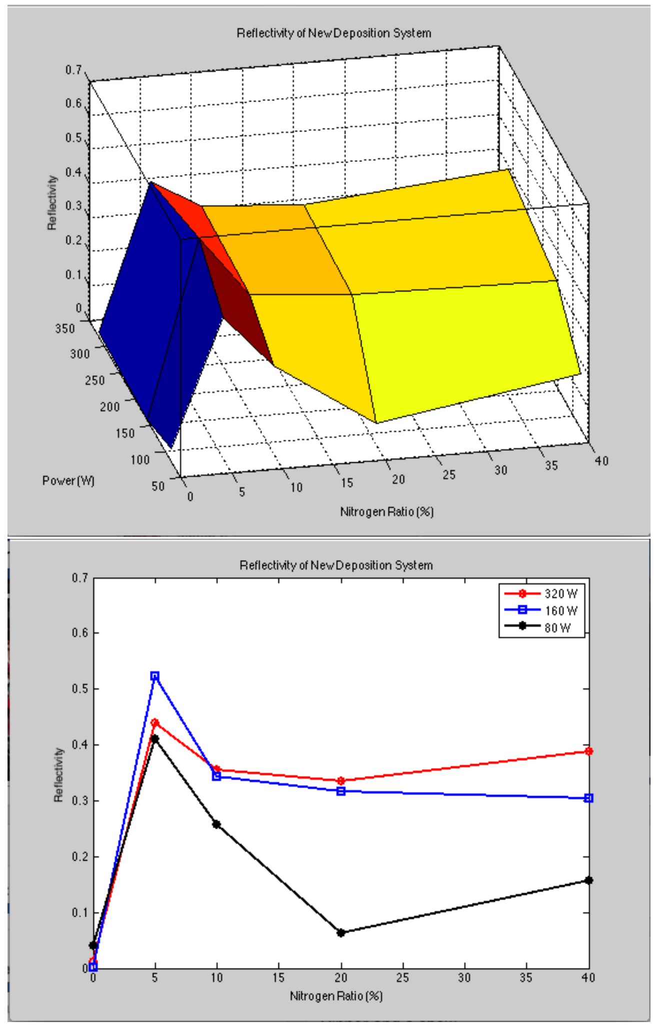

Figure 5: *Reflectivity of the new deposition system at powers with different Nitrogen ratios.*

IV. Simulations

The final part of the project was bringing the deposition and XRR stages together to form an overall comprehension on the properties of the multilayers. The reflectivity data collected by XRR is imported into the IMD program to produce simulations on the same graph produced can determine the d-spacing and the gamma ratio of the two substrates with precision; see Fig. 7

The growth rates of the depositions also seem to be greatly influenced by the parameters as shown in Fig. 6. We see the highest growth rate at a cathode power of 160 W at 10% N_2 which is in close proximity to the optimized condition looking at reflectivity.

V. Conclusion

The present work have shown that indeed changes in power level seems to have a dramatic influence on the performance of the new profile coating system. Optimization of the reflectivity of the multilayered thin films occurs at 5% N_2 ratio along with 160 W cathode sputtering power for the system. Future work should be conducted to expand the number of sputtering parameters considered including target-substrate distances.

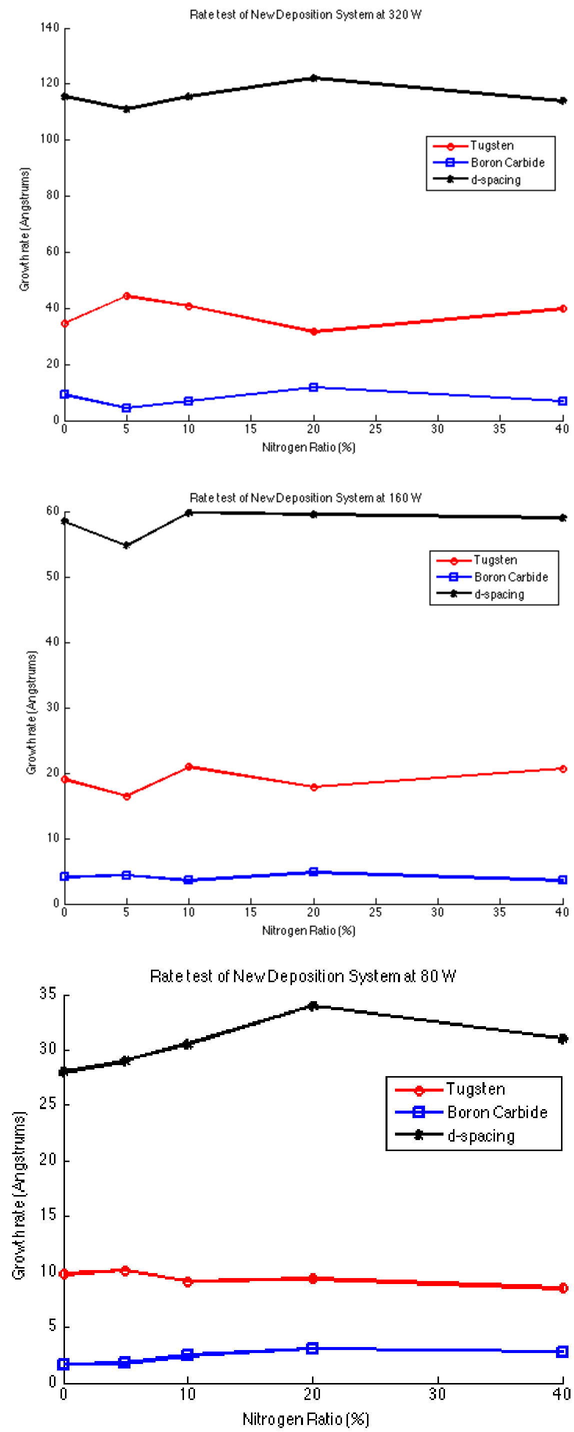

Figure 6: *Growth rates*

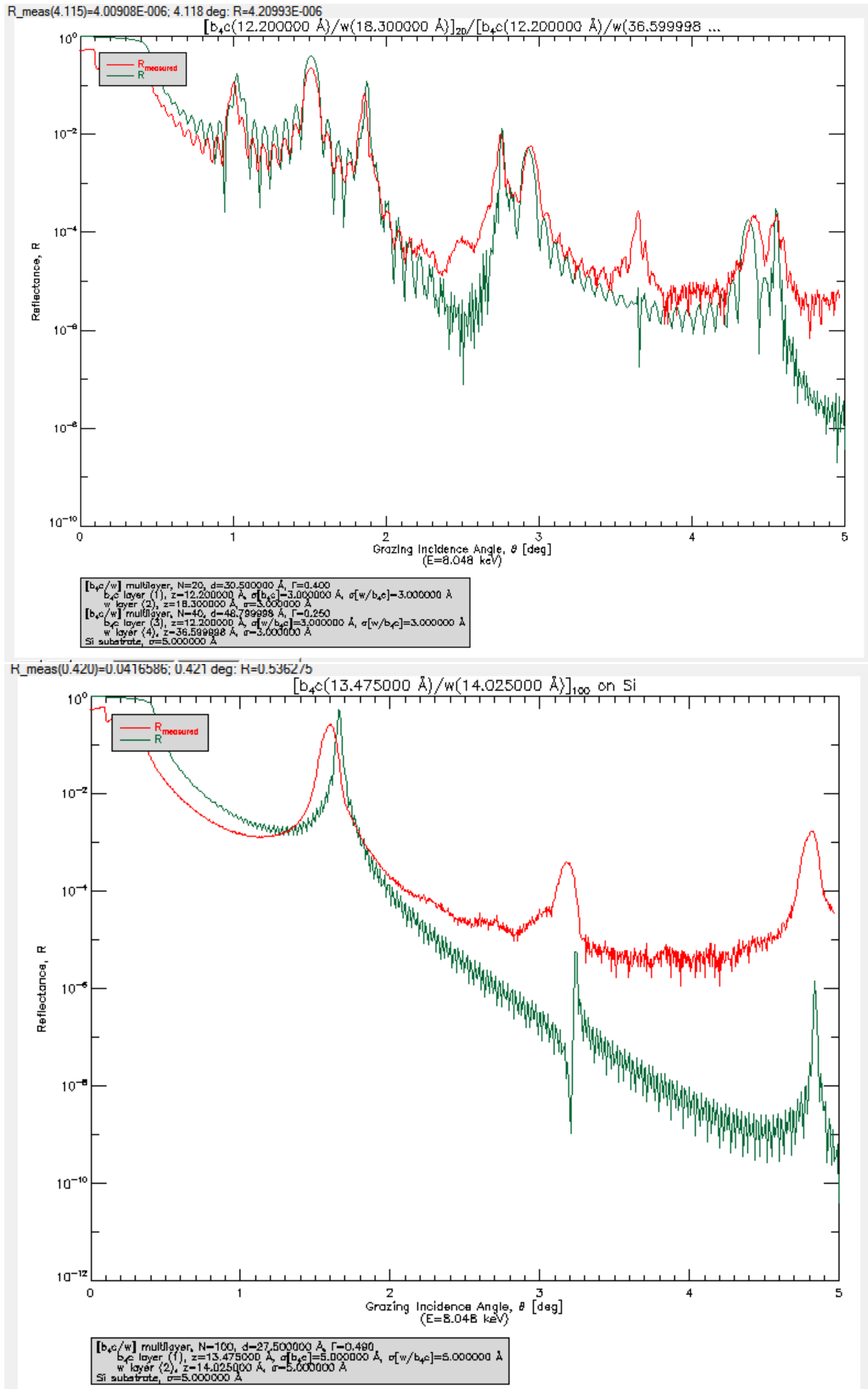

Figure 7: *Recorded reflectivity data and IMD simulations of rate test (top) and new samples (bottom) used to determine d-spacing and reflectivity.*

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