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# DESIGN, FABRICATION AND TESTING OF MULTILAYER COATED X-RAY OPTICS FOR THE WATER WINDOW IMAGING X-RAY MICROSCOPE


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## INTRODUCTION

Hoover *et al* [1] built and tested two imaging Schwarzschild multilayer microscopes. These instruments were constructed as prototypes for the "Water Window Imaging X-Ray Microscope," which is a doubly reflecting, multilayer x-ray microscope configured to operate within the "water window." The "water window" is the narrow region of the x-ray spectrum between the K absorption edges of oxygen ( $\lambda = 23.3$ Å) and of carbon ( $\lambda = 43.62$ Å ), where water is relatively highly transmissive and carbon is highly absorptive. This property of these materials, thus permits the use of high resolution multilayer x-ray microscopes for producing high contrast images of carbon-based structures within the aqueous physiological environments of living cells. We report the design, fabrication and testing of multilayer optics that operate in this regime.

## MULTILAYER DESIGN

Normal - incidence x-ray optical instruments have mirrors coated with multiple thin atomic layers ("multilayers"), also known as LSM ("Layered Synthetic Microstructures") or ("thin film multilayers"). The concept of multilayer and multilayer interference coated optics is deceptively simple; materials with contrasting optical properties are stacked with Angstrom scale percision, on a suitably figured substrate so that refelections from successive interfaces are coherently added to form a strong reflected beam[7].

Normal-incidence multilayer x-ray optics are made by coating (in a vacuum) ultrasmooth surfaces with a series of alternating layers of two different materials, one a strong x-ray scatterer with high atomic number, and the second a weak x-ray scatterer or "spacer" with low atomic number. This synthetic structure acts as an x-ray Bragg diffractor. X-ray reflectivity is maximized when the condition for constructive interference is satisfied as indicated by the Bragg equation $n\lambda = 2d \sin \theta$ , where n is the order of diffraction, $\theta$ is the angle of incidence with respect to the surface, d is the layer-pair thickness, and $\lambda$ is the wavelength of the incident radiation [8]. For mirrors operating at normal incidence ($\theta = 90^o$), the equation becomes; $\lambda = 2d$, where $\lambda$ is the wavelength of peak reflectivity of the first order Bragg diffracted light and d is the sum of the two layer thicknesses. Therefore, to make a multilayer x-ray optic with maximized reflecitivity at a wavelength $\lambda = 35$ Å the layer pair thickness needs to be 17.5 Å.

Although the reflectivity at each interface is small, when tens to hundreds of layer pairs are used, excellent reflectivities (more than 50% near normal incidence) can be achieved[8]. The properties of the reflected light such as intensity, bandpass, and image quality are, however, strongly dependent on a number of factors, including the relative thichness and absorption coefficients of the materials, the uniformity of the layers and the nature of the interfaces, the stability of the multilayer, and the smoothness of the substrate. Furthermore,the performance of a multilayer optical system is strongly dependent on the use of filters to exclude long wavelength light which is specularly reflected by the multilayer, and high resolution detectors to record the image[7].

The prototype mirror systems for the Water Window Imaging X-Ray Microscope were coated with Molybdenum/Silicon (Mo/Si) multilayers for operation near 135 Å [9]. Coatings using Nb/Si multilayers optimized for this wavelength have also been fabricated at MSFC and have exhibited excellent reflectivity in test carried out at NIST [9].

OSMC [9], successfully applied 200 layer pair Tungsten/Boron Carbide ($W/B_4C$) multilayer coatings with a d spacing of 17.35 Å to the 30X hemlite grade sapphire optics for the Water Window Imaging X-Ray Microscope. They achieved, with the normal incidence reflectivity of the finished mirrors, about 2% at 35 Å; about 25% of theoretical. They also demonstrated 3% reflectivity at $\lambda = 42.8$ Å using $W/B_4C$ fabricated with a d spacing of 20.6 Å.

MSFC [10], in an effort to develop multilayer coatings with higher reflectivity in the water window used Nickel/Titanium (Ni/Ti) multilayers because theoretical studies revealed that very high reflectivity might be achieved in the 35 - 43 Å regime [9]. Coatings with 200 layer pair Ni/Ti multilayers with a d spacing of 17.6 Å have been fabricated on float glass, silicon wafers, and ultra smooth concave spherical substrates of fused silica[9]. They did not achieve admirable results.

Theoretical studies show that 200 layer pair Nickel/Boron Nitride (Ni/BN) multilayer coatings with a d spacing of 17.7Å might achieve a maximum reflectivity of $\approx$ 21% in the 34 - 36 Å regime, (Figure 1). Due to the design of the MSFC sputtering setup it was decided, as a first attempt, to use only 75 layer pair Ni/BN.

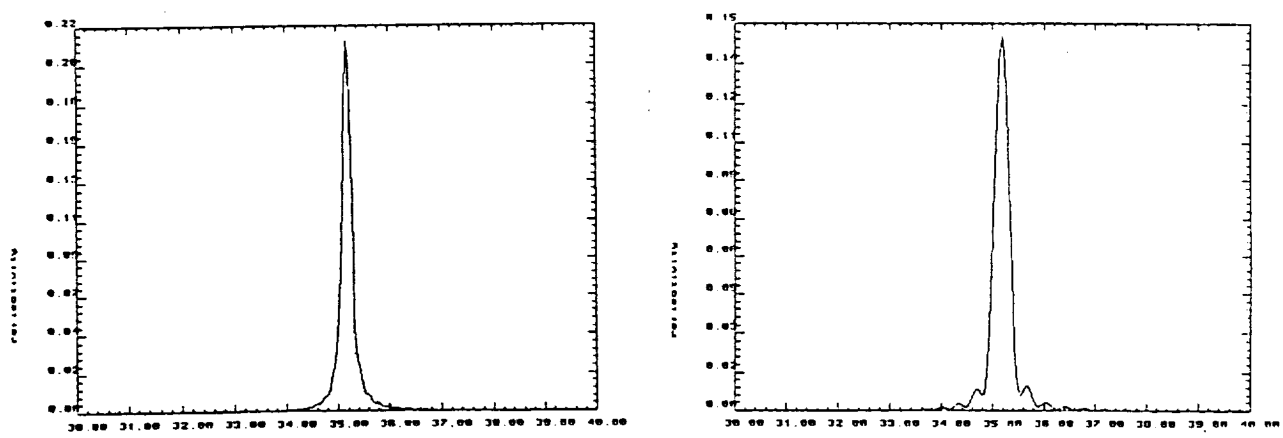

Figure 1. Theoretical x-ray reflectivity for 200-L and 75-R layer pair Nickel/Boron Nitride

## MULTILAYER FABRICATION AND TESTING

There are many different methods for depositing thin films. However, for the purpose of depositing thin, uniform layers nanometers thick "sputtering", with deposition rates from 5 - 50 nm/minute, is slow enough to be relatively well controlled. Sputtering is the process by which atoms from a target are knocked off via collision by another species[3].

In the setup at MSFC's Space Sciences Laboratory[4], a planar diode radio frequency - sputtering system modified to triode operation by addition of a ring filament surrounding the anode in an ordinary laboratory environment has been used to produce several two - component multilayer optics (Figure 2)[3].

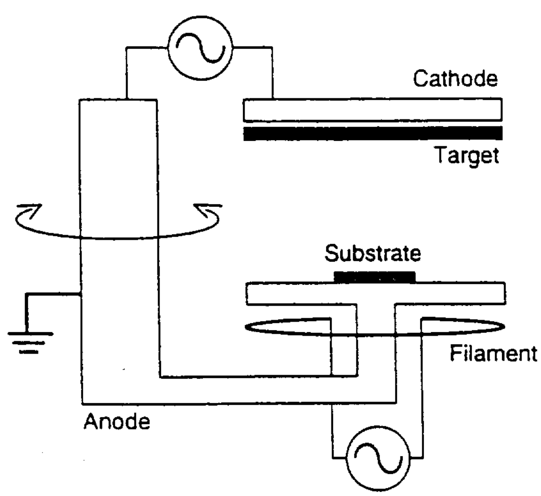


Figure 2. MSFC R.F. Diode Set-Up

High purity argon is introduced into the evacuated sputtering chamber. A forward power at 1300 KHz is applied while the filament ejects free electrons into the chamber. These electrons, having high mobility, collide with argon gas and produce + argon ions with much lower mobility. During the production of the argon plasma the water cooled anode carring the substrate to be coated, floats to a DC potential higher than the cathode containing the target. This accelerates the ions toward the target knocking off atoms which float to the substrate[3].

Depostion rates can be determined by depositing a substance onto float glass (or similar smooth surface) and using a stylus profilometer to determine the thickness of a film deposited for a known time. It has been found that only the central 5 cm of the 15.2 cm target produces fairly uniform deposition rate[3,4].

We decided, as a first trial, to deposit 75 layer pairs of Ni/BN on float glass and silicon wafers. Our first task was to change to the Nickel and Boron Nitride targets in the sputtering chamber and determine the rate of deposition of each target. We placed four (4) squares of 7059 float glass on the anode, one with a razor blade mask. The chamber was then evacuated to $10^{-6}$ torr. After introducing 10 μm argon at $10^{-2}$ torr., we presputtered the targets onto the shutter for 20 minutes each. We partially covering two of the squares with the shutter, then we sputtered BN on the substrates at 75 watts forward power at - 290 volts bias for 20 minutes. After moving the razor blade mask over slightly we then sputtered Ni for 20 minutes at 75 watts forward and - 744 volts bias. These samples were then removed to determine the rate of deposition of each of the materials on the substrate

It was determined from stylus profilometer traces and densitometer transmittance that the Ni film was about 450 Å thick and the total film was about 1350 Å thick, therefore BN was about 900 Å thick. This gives Nickel a deposition rate of .375 Å/s and Boron Nitride a deposition rate of .75 Å/s. Nickel, then, should be sputtered twice as long as Boron Nitride. Since we want reflectivity at $\lambda$ = 35 Å, the d spacing needs to be 17.5 Å, therefore, Boron Nitride should be sputtered 12 seconds.

We placed two (2) squares of 7059 float glass and two (2) pieces of silicon wafer on the anode, then evacuated the chamber to $10^{-6}$ torr. After presputtering the Ni target for 15 minutes at 75 watts forward, - 750 volts bias and the BN target for 7.5 minutes at 75 watts forward, - 245 volts bias in 10 μm argon at $10^{-2}$ torr., onto the shutter, we deposited 75 layer pairs of Ni/BN onto 2 samples of float glass and 2 samples of silicon wafer, sputtering Ni 2 X 12 seconds at 75 watts forward, - 713 volts bias and BN 1 X 12 seconds at 75 watts forward, - 239 volts bias.

Stylus profilometer traces of the coatings indicated a total thickness of the 75 layer pairs of Ni/BN to be 1400 Å, giving a d spacing of 18.7 Å. Theoretical reflectivity for normal incidence x-rays $\lambda$ = 37 Å (since $\lambda$ = 2d) should be ≈ 10%. Reflectivity tests with the Rigaku Bede Diffractometer using a Cu K$\alpha$ source at $\lambda$ = 1.54 Å should indicate a reflectance of ≈ 33% at 2.4° grazing angle. The actual test, however, did not indicate any significant reflectance on the float glass or the silicon wafers.

Our second trial was to produce a 30 layer pair Ni/BN mulyilayer optic at double the sputtering time, 24 seconds BN, 48 seconds Ni , under the same conditions as our first try using four (4) squares of 7059 float glass. Stylus profilometer traces of this coating measured a total thickness of ≈ 1400Å /30 layer pairs, giving a d spacing of 46.7 Å; $\lambda$ = 93.4 Å. Diffractometer tests, using 1.54 Å x-rays revealed good reflectivity on all four squares. The Bragg equation suggest that at a grazing incidence angle of $2\theta$ = 1.9° for first order maximum:

$$\sin\theta = n\lambda/2d = 1\,(1.54\text{ Å})/\,2\,(46.7\text{ Å}) = .0165$$
$$\theta = .945^{\circ}\,;\ 2\theta = 1.9^{\circ}$$

The best results were from sample 7/17/96 - 2, where $2\theta$ at approximately 1.90° for first order peak had over 150,000 counts ( Figure 3) giving a $\lambda$ = 93.1 Å.

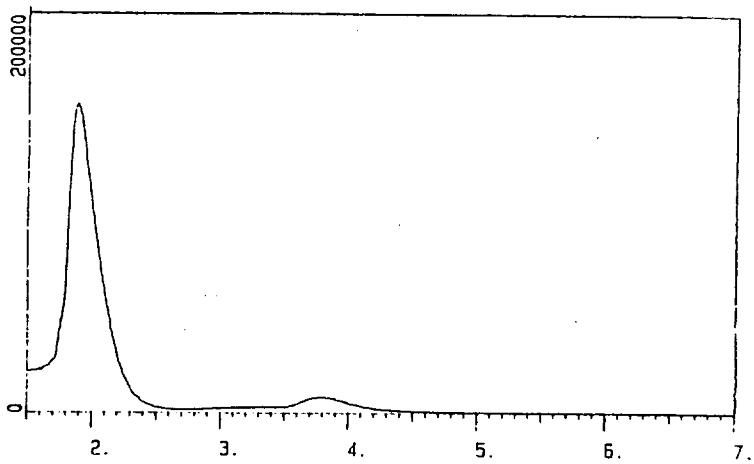


Figure 3: Reflectivity sample 7/17/96 -2 Ni/BN

Considering the success of the second trial, as a third trial we decided to fabricated 40 layer pairs of Ni/BN on two squares of 7059 float glass, reducing the time, to try to fabricate an x-ray optic that would operate in the water window. Using the same conditions as our previous two trials, but shortning the sputtering time to 14 seconds for BN and 28 seconds for Ni.

Stylus profilometer traces of this coating measured a total thickness of approximately 840 Å/40 layer pairs, giving a d spacing of 21 Å; $\lambda$ = 42 Å. Diffractometer tests exhibited reflectivity on sample 7/18/96 - 2, but at half the expected angle. The Bragg equation suggest that maximum reflectivity should occur at $2\theta = 4.2^{o}$, but diffractometer test show maximum reflectivity at $2\theta = 2.2^{o}$ (Figure 4). This may be harmonic, second order diffraction or from two pairs.

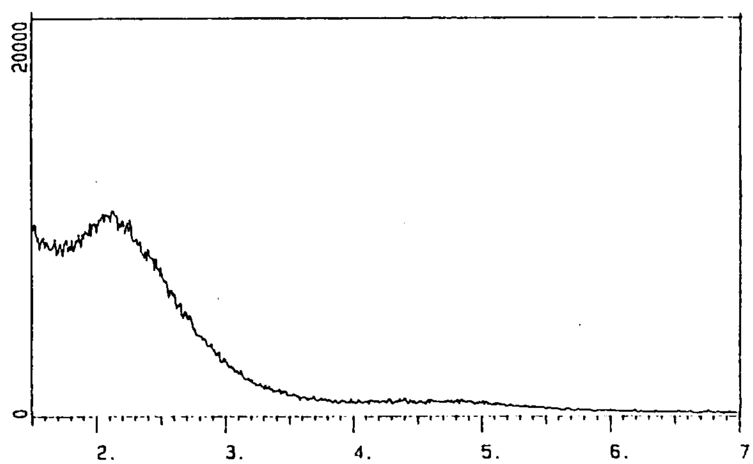


Figure 4: Reflectivity sample 7/18/96 -2 Ni/BN

## CONCLUSION

Three multilayer coated x-ray optics were designed, fabricated and tested. We have demonstrated that Ni/BN multilayer x-ray optics do produce predictible reflectivity at wavelengths in the 93 Å regime and reflectivity in the 42/84 Å regime. Theoretical evidence indicates that they should also produce reflectivity at wavelengths well within the water window, however, careful attention must be given to depositing layers an integral number of atomic layers thick to achieve a smooth layer interface.

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