SUPER MIRROR FABRICATION VIA ELECTROFORMING

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ABSTRACT

As part of a project to develop methods of placing highly reflective multilayer coatings on the inside of Wolter I mirrors, we have been pursuing a program of measuring flat mirrors. These flats have been produced and examined at various stages of the process we plan to use to fabricate multilayer coated Wolter I mirrors. The flats were measured via optical profiler, AFM, (both done at Brookhaven National Lab) and X-ray reflection (done at the Argonne National Lab (ANL) Advanced Photon Source (APS)). We report for the first time, to our knowledge, the successful placement of multilayers on an electroform by depositing the multilayers on a master and then electroforming onto this master and removing the multilayers, intact, on the electroform. This process is the one we plan to use to place multilayers on the inside of Wolter I optics.

KEY WORDS X rays, Multilayers, AFM, Profiler, Wolter I

1. INTRODUCTION

In a separate paper[1], we have reported a process we are developing to produce Wolter I X-ray mirrors coated with multilayers. As part of this program we have fabricated several test flats. We measured these with a Micromap 512 profiler (simply referred to as Micromap below), an

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atomic force microscope (AFM), and with an X-ray beam at the Argonne National Laboratory Advanced Photon Source. Here we report the results of our work.

The ultimate goal of our research is to perfect the fabrication of Wolter I optics with multilayers for as low a price as possible. This will allow us to produce cost-effective X-ray optics for both astrophysical and laboratory uses. The results we report here, however, are simply to demonstrate a proof of concept of the technology by demonstrating that electroforming can be used to produce the desired optics. *No attempt was made to optimize the performance of the samples.*

2. SAMPLE DESCRIPTION

Because we were only performing the simplest proof of concept study, we used flats that were left over from previous projects. As such this set of sample is not homogenous, but the result was that we explored a range of substrates and surface qualities. We had 2 aluminum substrates: one 2-inch diameter 0.5-inch thick, the other 1-inch diameter 0.25-inch thick. These were coated with about 5 mils of electroless nickel and initially polished to sub 1 nm roughness. These samples had an additional hard protective coating made up of a 200 nm thick, approximately 10 nm period multilayer of ZrN and amorphous CN_x followed by about 20 nm of CN_x. Then they were over coated with Si/W X-ray multilayers. We also used 2 ceramic pieces about 2 mm thick that we over coated with vacuum vapor deposited gold and then coated with X-ray multilayers. Finally we coated a 1.5-inch electroformed flat that is about 1 mm thick with X-ray multilayers. These multilayers were all 100 periods of W/Si with W being the initial layer and Si the final layer. The prescribed thickness of each W layer and each Si layer was 3nm.

The X-ray multilayers described above were deposited by Atkinson Thin Film Systems of Hudson, NH. The deposition technique was DC magnetron sputtering in a 4 mtorr atmosphere of argon. The temperature of the substrates was kept below 100 C and the samples were translated between apertures in front of the Si and W sputtering targets.

Just prior to the submission of this for publication, we were able to make X-ray measurements on one more sample. This sample was made by first depositing about 40 nm of gold on top of a 2 inch diameter, 2 mm thick sapphire disk. Then 40 periods of about 1.3 nm of W and 1.3 nm of C were deposited on top. These were made at ANL, the elements were DC sputtered, the sample (Au plus sapphire) was kept a room temperature and the argon atmosphere was kept at about 2.6 mtorr. Then, an adhesion layer of Cr and Cu was evaporated. Each layer was about 50 nm thick. Finally this sapphire, Au/W/C/Cr/Cu piece was placed in an electroforming tank for about 1 day and Ni was electroformed onto the Cu surface. Then the sample was removed from the bath and the electroform was removed from the sapphire. Visual inspection showed that all the layers successfully released from the sapphire to produce proof of concept of making Wolter I mirrors in this fashion. The X-ray measurements of this sample are reported below.

3. X-ray MEASUREMENTS

3.1. GENERAL DESCRIPTION

We used an upstream mirror to act as low pass filter to prevent energies much higher than about 33 keV from reaching the sample, which is mounted on a Huber diffractometer. The distance between the upstream slit and the Huber stage that held the samples was about 5 m and the distance from the sample on the Huber to the detector on the Huber two-theta arm was about 0.8 m. The detector was NaI. The Si 111 crystals in the double crystal monochromator were deliberately tuned to reflect both lines at 10 keV (first order) and at 30 keV (third order). Two distinct voltage windows were set up to count separately the pulses from the 10 and 30 keV photons. The 10 keV data were collected mainly as a cross check that the correct energy dependence for reflectivity was observed. Once we determined that we understood the response of the mirror and the detectors, we placed a filter in the system that blocked the most of 10 keV flux. We therefore only discuss 30 keV measurements, except for the one sample (see below) that was created and tested just as this article was being completed.

The sample and axis of rotation were centered on the X-ray beam. We verified in each case that this was done correctly as the fall on of specular reflection with angle occurred at the correct absolute angle to within about 0.02 degrees.

We made two types of measurements on the samples: "theta/two-theta" scans that rotate the detector twice as far as the sample rotates relative to the beam. Alternatively we set the mirror angle to reflect at a multilayer peak and then scanned the "two-theta" arm to measure the angular spread of the reflected beam. The height of the X-ray beam was defined by a slit down stream from the monochromator. The height was about 100 microns high and about 1 mm wide. The detector slit was set at 1 mm wide for the theta-two theta scans so as to measure the integrated reflected beam and the detector slit was set to 100 mirrors for the angular spread measurements.

Below we present a plot of a few of the samples that we measured. The most significant results are the measurements made on our most recent sample. This sample was the one made via the intact removal of multilayers onto an electroform.

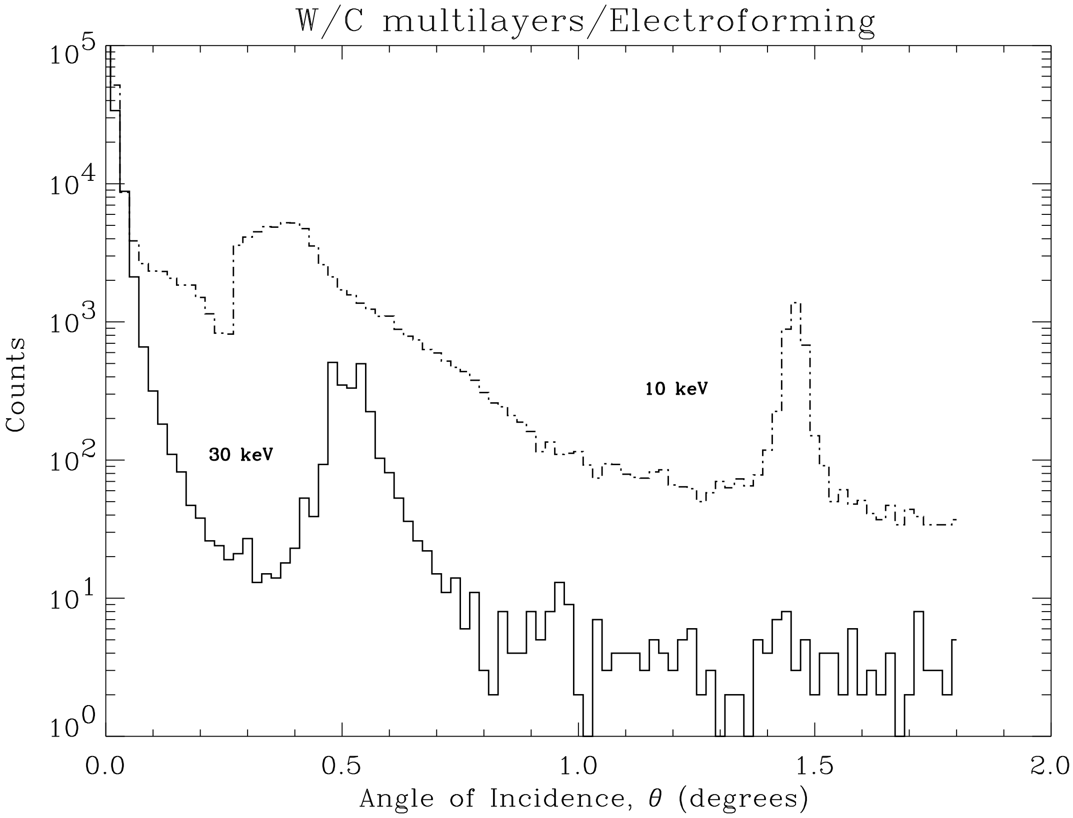

Figure 1: A theta-two theta scan of the sample that we made via electroforming onto multilayers and then removing the multilayer with the electroform.

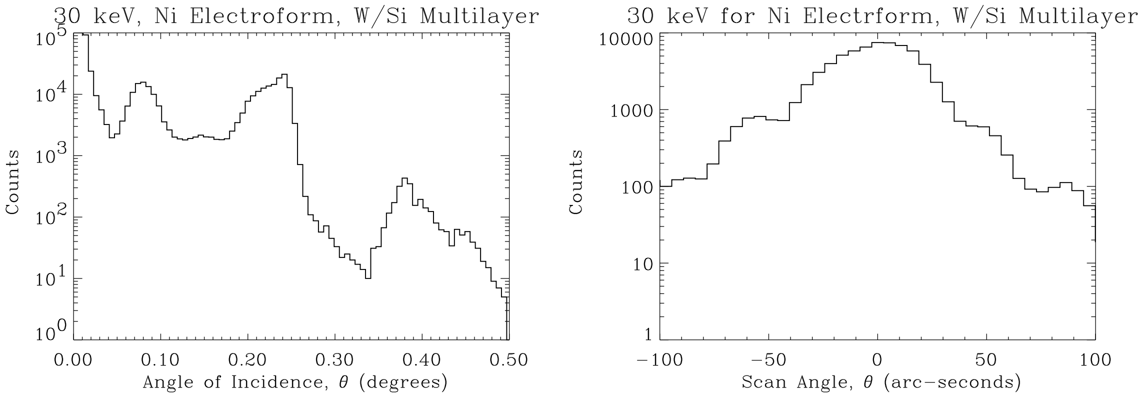

Figure 2: Left: theta/two-theta scan of an electroformed nickel "flat" (nearly flat) upon which multilayers were deposited. Right: A scan of the reflected beam with the mirror set to the angle of the multilayer peak at about 0.25 degrees seen in the figure on the left.

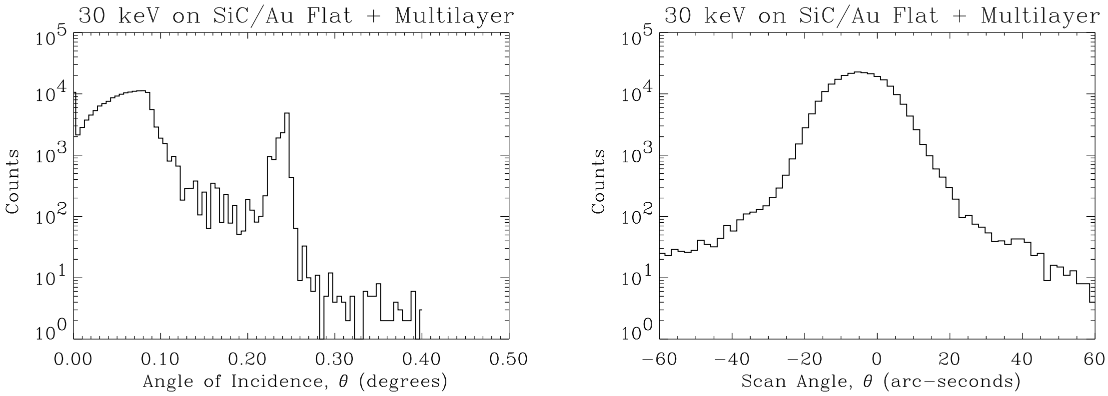

Figure 3: Left: theta/two-theta scan of an SiC "flat" (nearly flat) upon which multilayers were deposited. Right: A scan of the reflected beam with the mirror set to the angle of the multilayer peak about 0.25 degrees seen in the figure on the left.

4. DISCUSSION

4.1. X-ray results

The most important and new aspects of this work are the X-ray results shows in Figure 1 and how they relate to the prospects for producing Wolter I optics with enhanced energy coverage, increased field of view, and reduced focal length by the application of multilayers.

The X-ray results shown in Figure 1 are based on X-ray reflectivity from our sample that was made by the intact removal of multilayers onto an electroform. In Figure 1 we see two multilayer peaks, one at about 0.5 degrees for 30 keV and the other at 10 keV at about 1.5 degrees. These are consistent with a model of 40 layers of W/C with total layer thickness of 2.45 nm and a gamma of 0.4, which is close to the initial request of d = 3 nm and gamma = 0.5.

The peak in the 10 keV scan near 0.35 degrees is not yet completely understood. There are so many different material layers in this sample, however, that it is entirely possible that some combination of these layers is giving rise to the "interference effect" or peak seen near 0.35 degrees. For example, there are several other interfaces: an Au/W interface at the top of the piece; a W/Cr interface at the bottom, a Cr/Cu at the bottom; and, a Cu/Ni interface at the bottom. Some preliminary modeling of the 10 keV reflectivity by different single bi-layer combinations indicates that it is possible to obtain a feature for 10 keV reflectivity similar to that seen near 0.35 degrees in Figure 1. And, it is exciting to notice that there is the prospect for enhancing the reflectivity of mirrors at 10 keV all the way out to 1.5 degrees. Similarly, the 30 keV peak demonstrates the possibility of obtaining an increase in factor of 5 in angle over the putative 0.1 degree critical angle for a bare (no multilayers) Au surface.

In Figure 2 which is the result of multilayers evaporated onto an electroform, we see two multilayer peaks at about 0.25 (primary) and about 0.38 (first harmonic) degrees. For comparison we show results from our best flat piece in Figure 3. Here we see one multilayer peak at about 0.25 degrees. The positions of these peaks are consistent with a model in which there are 100 multilayers, 6 nm deep with a gamma of about 0.45. This is close enough to what was requested for the multilayer deposition (gamma=0.5, total size of one period, W/Si inclusive, of 6 nm) as to be quite reasonable.

Evaporation of multilayers on electroforms was not expected to be too difficult as others[2] have demonstrated that it was possible to deposit multilayers on epoxy coated aluminum foils and multilayers had been deposited once before on one of our electroforms[3]. But a direct deposition technique does not allow the fabrication of multilayer coatings on the inside of Wolter I optics unless the optics are about a meter or more in diameter to allow for the uniform deposition of the multilayers. Or, if smaller mirror diameters are to be combined with direct multilayer deposition, then the mirror must be only a fraction of a complete conic of revolution. Usually about one quarter of the complete 360 degrees of revolution is used[4,5]. Then these "shells" will need to be assembled into a pseudo-Wolter I optic[4,5]. Thus, our demonstration that it is possible to remove multilayers from a master onto a Ni via electroforming is real breakthrough in technique, as this means that it will be possible to produce relatively small and low cost Wolter I (or optics of similar geometry) X-ray super mirrors.

4.2. NON X-RAY EVALUATION

In order to fabricate high performance X-ray optics, quality control is important and related to this aspect of X-ray mirror fabrication we discuss in this section results of the surface quality as measured via the Micromap and AFM, and how they predict the performance of the X-ray results. We first begin with a summary of the Micromap (and in two cases AFM) measurements.

All the substrates were measured prior to being coated with multilayers. Time did not permit us make Micromap measurements after the multilayer deposition. In addition to the Micromap, AFM measurements were made only on the 2 aluminum/electroless nickel substrates. For these two pieces, Micromap results differ significantly, but AFM results do not. Below we discuss the comparison of the X-ray measurements with the Micromap measurements. In Table 1 below we provide a summary of the Micromap measurements on our samples. Micromap measurements were made before and after the CN_xZrN coated Ni/Al samples were over coated with about 50 nm of Ni (to be used as a release agent). Thus the Ni/Al pieces are listed twice in Table 1.

Table 1: Summary of Micromap Measurements. Contour is the root mean square deviation from on a surface contour, and Ra is the RMS of the deviations from a two dimensional profile from the average plane. Both numbers are based on averaging over a 3.25 mm by 0.8 mm area. A 2.5 x objective was used. To describe the samples, we list the elements (or materials), from right to left, starting from the surface and ending with the bottom substrate.

Sample	Contour	Ra
CN_x/ZrN/Ni/Al, $1''$ dia.	0.97 nm	0.71 nm
Ni/CN_x/ZrN/Ni/Al, $1''$ dia.	1.07 nm	0.81 nm
CN_x/ZrN/Ni/Ni/Al, $2''$ dia.	1.07 nm	0.82 nm
Ni/CN_x/ZrN/Ni/Al, $2''$ dia.	1.21 nm	0.84 nm
Au, sapphire, $2''$ dia	0.27 nm	0.28 nm
Au, CVD SiC $1''$ sq.	0.45 nm	0.31 nm
Au, Ni electrofm., $1.5''$ dia.	0.79 nm	0.63 nm

In a previous SPIE proceeding it was suggested[6] that AFM measurements were necessary and sufficient to predict the X-ray reflectivity characteristics of X-ray mirrors. This previous work also reported WYKO (another brand of optical profiler) measurements made with a 20 x objective and the PDS of the WYKO was indeed quite disparate from the AFM PDS (c.f. their Figure 6). In contrast, our Figure 4 (shown below)

Figure 4: Micromap measurements and AFM measurement of sample the 2 in. nickel sample in Table 1.

with a restored PDS shows how the Micromap data give better coverage and comparable amplitudes to those of the AFM in the critical 5×10^{-2} to $5 \times 10^{-1} (m\mu)^{-1}$ frequency domain. We suspect the reason for the disparate AFM and WYKO results in this previous work[2] was because for the WYKO results, they did not use a restored PDS. We suggest, therefore that Micromap measurements are valid for evaluating the surface quality of X-ray mirrors and we provide further evidence for this below.

With multilayers added to the surface, there is an extra requirement on surface smoothness besides the standard grating equation and scattering dependence of surface quality. This is that the quality of the multilayers is generally affected by the surface upon which they are deposited, though at least one group[2] suggests that it is possible to produce effective ion polishing of the substrate by polishing the intial layers of the multilayers with Kr^{+} ions. And another group[7] suggests that certain multilayer combinations are self-planarizing. No special treatment was applied to our multilayers, however, and comparison between X-ray measurements and the Micromap results can be used to judge how the quality of our multilayers was affected by the the substrate smoothness.

Time did not permit detailed calculations based on the Micromap results to compare with our X-ray measurements. We did, however, perform two comparisons between the X-ray results and the Micromap measurements which demonstrates a correlation between the average Micromap measurements (c.f. Table 1) and the X-ray performance of multilayers. Both techniques are susceptible to errors in the macro figure error and alignment with the X-ray beam so that the correlations could be masked by systematic effects. The Micromap measurements of those pieces listed in Table 1 indicate, however, that the radii of curvature of these flats were large. They ranged from kilometers to tens of meters as measured over mm portions of the flats. And, the general trend in Figure 5 is certainly consistent with the hypothesis that the Micromap measurements are sufficient for predicting the behavior of multilayers.

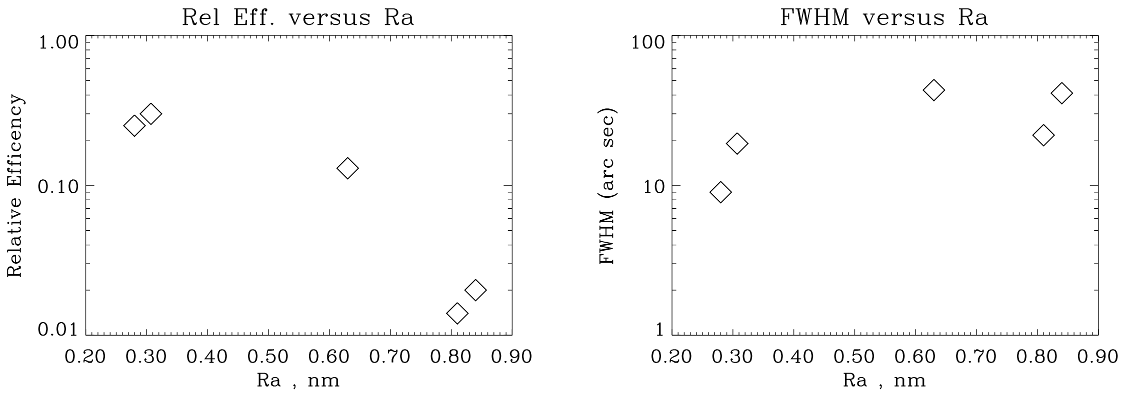

Figure 5: Left: Plot of the relative efficiency of the multilayer peak as defined by the ratio the total counts of the reflected beam at the multilayer peak (c.f. Figures 1 and 2, right hand panels) and the direct beam versus the Ra average values measured for the samples (c.f. Table 1 for Ra values). Right, FWHM of the same multilayer peak versus the Ra of the samples.

5. SUMMARY AND CONCLUSION

We have shown, for the first time, that it is possible to remove multilayers intact from a master onto an electroform and we conclude that the outlook is bright for applying this technique to the fabrication Wolter I super mirrors.

We also find that it is possible to obtain good performance from multilayers deposited on ceramics as well metal surfaces including both originally polished ones and one produced via electroforming replication.

In order to quality control the process, we find that the Micromap profiler measurements on the substrate prior to the deposition of the multilayers are good predictors of the performance of the multilayers. The specific process we used to electroform onto a multilayered master was the simplest one we could devise, as we knew that Au releases from sapphire when we electroform on the Au sapphire combination. We used about 40 nm of gold because we knew that this amount of Au makes smooth surfaces. Much thinner depositions at room temperature (the temperature used here) could lead to the formation of 'islands" which would make the surface too rough for the effective deposition of functional multilayers.

We are now in the process of perfecting the deposition of smoother release layers that are less absorbing. We will include both Au on a heated substrate, Ni, and other materials. We also will advance this technique to metal masters that have been coated with a smoothing and protecting agent such as CN_x[1].

Further improvements to the technique we plan to make are to improve the surface quality upon which the multilayers are deposited and to improve the quality control and handling of the electroformed pieces. When all these improvements are made, we fully expect to achieve angular resolution below 1 arc minute and we do not rule out resolution in the arc second range.

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