Production and performance of multilayer coated conic sections

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ABSTRACT

Previously we have reported on our work on coating a truncated-cone-shaped "engineering" (not high quality in terms of smoothness) mandrel and have removed the layers, intact, on the inside of an electroform with a cylindrical, truncated-cone geometry. We have advanced to using a high quality (about 0.5 nm) smooth truncated cone. We report our latest advances in refining our fabrication techniques and the results of X-ray measurements. The X-ray measurements made at the Argonne APS SRI-CAT 2-BM-B beam-line were at 10 and 30 keV. The results showed that we had produced excellent Si/W multilayers on the inside of a 10 cm long by about 10 cm diameter truncated cone shaped mirror. We estimate the reflectivity of the layers at the primary Bragg peak to be well above 10%. We also show that the multilayers were uniform around the mirror.

Keywords: X-ray Optics, Multilayers, Electroforming, X-ray Reflectivity

1. INTRODUCTION

In previous work, we have proved that the basic concept of the "intact electroforming multilayer process" (IEMP) replica mirror fabrication is valid.¹ The crux of the matter is, however, perfecting the details of the process so that high quality (greater than 20%) reflectivity multilayers can be produced. One of our major achievements was to make two good mirrors in a row, whereas previously, after making one good one mirror, it took several attempts to produce another good mirror. This has taken significant effort in the form of quality control and care in exactly how each step is performed. Below in section 5, we describe the results of X-ray reflectivity measurements at the Argonne National Laboratory (ANL) Advanced Photon Source (APS). First, however, we review the basic concept and the astrophysical motivation behind using this technique.

2. THE PROCESS

The IEMP involves these basic steps: (1) A multilayer is sputter deposited on a mandrel (master) that is the complement of the figure of the optic that is $desired^{2,1,3}$; (2) A nickel mirror is electro-deposited on the coated mandrel; (3) The electroformed mirror is removed with the multilayers intact as a coating on the reflective inside mirror walls.

Our group uses a CN_x sputter coating technology^{4,5} to both smooth and to protect the mandrel surface. We also place a release layer on the mandrel to assure release of the coating from the mandrel. After removal of the mirror from the mandrel, the release layer is dissolved without harming the underlying multilayer coating.

3. MOTIVATION

Both the scientific and technical motivation for using the electroforming approach has been nicely described elsewhere,⁶ but besides giving a section here on science for completeness, we also point out the advantages of coating the mandrel rather than the inside of an electroformed mirror.

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3.1. Technical Advantages of Our Approach

Our approach allows the mandrel to be placed at a nearly arbitrary distance away from the sputtering targets which facilitates uniform coating deposition. The CN_x coating smoothes the mandrel down to the 0.2-0.3 nm level which means the electroformed replica can be made as smooth as 0.4-0.5 nm and is less costly and more robust than directly smoothing the surface to 0.2nm by polishing techniques. Furthermore, as shown by the power of the Chandra Observatory with images of fields such as the Galactic center: the higher the angular resolution of the optics, the better. The performance of electroformed optics has always exceeded that of the conical approximation approach, and is most likely to continue that advantage into the foreseeable future. Finally, the ability to coat the mandrel means that relatively short focal length optics (1-2 meter) can be produced, which gives a distinct advantage over other methods which typically require 6-8 meter focal lengths. In summary, our approach is worth perfecting, and as we show in section 5, we are rapidly approaching the ability to be able to promise making high quality multilayer coated optics on demand.

3.2. Astrophysical Motivation

Hard X-ray astronomy is still in its infancy because it has yet to achieve the power of focusing that allows both imaging and greatly reduced background (and hence greatly improved sensitivity). Here we provide a summary of just some of the issues that can be addressed with better than or equal to 30" angular resolution with X-ray optics that are coupled with position sensitive detectors that match the requisite positional accuracy for the angular resolution. In the general cases we discuss below, imaging, and greatly improved sensitivity over the Oriented Scintillation Spectrometer Experiment (OSSE⁷; ≥ 100) are necessary for these studies to be carried out. Such capabilities (over 100 times better than OSSE; $\leq 20\mu$ Crabs) are within the grasp of hard X-ray mirror systems.

The active galactic nuclei (AGN) paradigm is a topic of great interest not only because of the inherently interesting physics surrounding massive black holes, but also because the study of AGNs couples to the origin and evolution of galaxies. How the black hole at the center of the AGN evolves and accretes material is not well understood. Measurements in the ~ 40 to 100 keV range allow us to distinguish between more thermal-like spectra such as NGC4151⁸ and those dominated by power law components, e.g. 3C273 and EGRET blazars in general (cf. ref. 9 and references therein). Detecting and classifying such systems will take us a step closer to determining viable physical models of these objects. Furthermore, the contribution of AGNs to the diffuse X-ray background is also important to determine, and more data are necessary to confirm or deny the tentative conclusion of ref. 10 where it was concluded that AGNs do indeed produce the diffuse X-ray background in the 40 keV to 100 keV region. Our own galactic center is related to AGNs. There is probably a massive black hole there. High sensitivity and the ability to image the region will allow us to test advection-dominated accretion flow¹¹ models that have been proposed to explain the much less than Eddington limit X-ray flux that is observed.

Another area of interest is supernovae (SN), and the understanding of SN-light curves has taken on increased importance as they are now being used as cosmic distance indicators to measure the cosmological constant, Λ . ⁴⁴*Ti* should be detectable from supernova remnants that have been produced in the past few hundred years, if current models are correct (e.g. refs. 12,13). Even though ⁴⁴*Ti* is not a major source of energy for type SN Ia light curves (cf. ref 12), it always helps to compare data with models as much as possible (cf. ref. 14). This includes SN ⁴⁴*Ti* production. Also, SN are though to produce cosmic rays (cf. refs. 15–17) and measuring the non-thermal radiation from SNRs will allow us to measure the cosmic ray population in young SNRs. SN produce pulsars which also produce hard X-ray emission. Pulsars may explain the origin of the diffuse ~ 50 keV to 100 keV emission seen from the Galactic plane (cf. ref. 18).

Many more pulsars than have currently been detected, however, must necessarily be producing hard X-rays.^{19,20} When a focusing system is flown and many more pulsars are detected, we will be able to develop useful models of the pulsar hard X-ray emission versus magnetic field, period, and orientation with respect to the Earth. These new data will allow us to derive a phenomenological model of the hard X-ray emission from pulsars, and confirm or deny the proposition that pulsars produce the diffuse emission from the Galactic plane. It will also allow us to use pulsar birth rates to derive the origins of the properties of pulsars we see today by estimating the spin and magnetic field of pulsars at birth. The best direct evidence for the strength of pulsar magnetic fields comes from the measurement of cyclotron absorption features near 30 keV in X-ray binary systems. About 10 such systems are known (cf. ref. 21 and references therein), but with a new sensitive instrument, this number should be increased dramatically, so that we will have a large enough sample to relate the magnetic fields to models of the accretion flow and X-ray

emission. Finally, other pulsars have fields as large as $\sim 10^{14}$ G, i.e. soft gamma-ray repeaters (SGRs²²⁻²⁴). These SGRs are extremely interesting to study since they have such extremely strong fields. Measuring the X-ray emission from SGRs in their quiescent state will give us a better view of the dependence of the hard X-ray emission on the magnetic field and more data towards understanding the physics of these bizarre objects.

In conclusion, hard X-ray astronomy is an exceedingly rich field of study that will benefit greatly from the ability to focus the hard X-ray sky.

4. MIRROR FABRICATION

We have given a description of the process we used in previous work.¹ The mirror fabrication process starts with the mandrel, which in this case was purchased (by Allen S. Krieger) from Hyperfine Inc in Boulder CO. This mandrel was made of Al and coated with electroless nickel and smoothed to approximately 0.5 nm. The mandrel was then coated with CN_x at Northwestern University. The mandrel was then coated with a release layer, sputter-coated with 60 layers of Si/W, evaporatively coated with Cr and then Au to assure adhesion to the electro-plating and then the piece was placed in an electroforming bath and electroplated with ~ 0.6 mm of nickel. There was just enough tensile stress in the electro-plated mirror so that it released without the need to cool the mandrel more than from the about 45C of the bath to room temperature (about 27C).

A schematic of the mandrel is shown in Figure 1 and in Figure 2 we see an image of the mandrel and the mirror after the release layer has been removed.



Figure 1. A schematic of the mandrel we used to make our mirrors. The base is about 10 cm in diameter and the draft angle is about 0.5 degrees.

5. MIRROR CHARACTERIZATION VIA X-RAYS

Just by visual inspection we could determine that the multilayers were transfered intact from the mandrel to the mirror, as the mirror surface is shiny. We produced 2 multilayer coated replica mirrors and cut several pieces from the first for use in our own X-ray diffractometer and to examine with a Nomarski microscope. From this work we determined that multilayers were present, the d-spacing is about 5.3 nm, and that the surface roughness is about 0.6

nm. We then took the complete second mirror to the APS at Argonne and measured the X-ray reflectivity at 10 and 30 keV. We used a Huber stage on the SRI-CAT 2-BM-B line. The mirror was about 0.66 meters from the NaI X-ray detector. The detector slit was set to 1 mm in the vertical direction (in the scattering plane) to accept most of the reflected beam. The detector slit width was 10 mm in the horizontal direction. The slit that defined the beam was 100 microns in the vertical direction and 2 mm in the horizontal direction. A platinum mirror was used as a "low-pass energy filter" to prevent X rays with higher order harmonic energies above 30 keV from reaching the sample. The sample surface was mounted at the center of the diffractometer by bisecting the direct beam when its surface was parallel to the beam. Specular X-ray reflectivity measurements were made by maintaining the momentum transfer wave-vector, q, normal to the surface. We did this by scanning two theta, the detector arm, at twice the rate of theta, the tilt of the sample with respect to the incident beam in the scattering plane. This is, therefore, called a theta-two theta scan. Before each theta-two theta scan, the curvature of the mirror was aligned to the beam by rotating the sample using the chi and phi motion of the diffractometer, so that the optical axis of the conical mirror was in the scattering plane.



Figure 2. The nickel mirror with multilayers on the inside. This is after removal from the mandrel and dissolution of release layer. The wall thickness is about 0.6 mm, the diameter of the mirror is about 10 cm.

In order to check the homogeneity of the deposition, we rotated the mirror about its optical axis and made another measurement. For each rotation angle, we measured the X-ray reflectivity at two energies, 10 and 30 keV. We did this in order to be able to distinguish between geometrical effects and energy dependent effects such as scattering and the angles of the Bragg peaks. We chose 10 and 30 keV because of the simplicity of the experimental procedure. At 10 keV, we used the Si(111) reflection from a Si double crystal monochromator. The X-ray reflectivity measurements at 30 keV were made by simply using the Si(333) reflection and by adding an extra X-ray attenuator to stop the 10 keV X-rays but does not require a change in the monochromator setting. The NaI detector also allowed us to distinguish between 10 and 30 keV X rays so that the attenuator did not need to completely block the 10 keV flux.

Also, the 30 keV measurement demonstrates that the multilayers perform in the desired (for astronomical applications) energy range. We had hoped to obtain a direct measure of the absolute reflectivity of the mirror, but due to



Figure 3. From left to right, these are referred to as Figures 3 a, b, and c in the text. These are the results of 3 different so called "theta-two theta scans", see text. Figures a and c are from the same orientation (about the optical/long axis of the mirror), and Figure b is for a rotation of about 90 degrees about the optical axis. The solid lines show the measured reflectivity and the dashed lines the modeled reflectivity, see text.

geometrical distortions of the mirror caused by the shape of the mandrel, we can only infer an absolute reflectivity. In Figures 3 a, b, and c we show the reflectivity at two different rotations about the optical axis at 10 keV and prior to the rotation about the optical axis rotation, we show the reflection at 30 keV. These are results are by far and away the best we've ever achieved. The dashed curves are were generated by IMD^{25} and are all the exact same model of 60 Si/W layers with the Si layer being 4.229 nm and the W being 1.062 nm. In order to explain the successive suppression of second and third order multilayer peaks, we used 0.6 nm of inter-diffusion/roughness in the multilayers, which agrees well with our estimates of the surface roughness from our examination of samples with a Nomarski microscope. Although the IMD model presented in Figure 3 exhibits excellent agreement with the data, we do not claim that this model is unique, as there are many parameters in the fitting procedure plus there could be geometrical effects that could also have caused the suppression of the second and third order peaks. Therefore, it is possible that the inter-diffusion/roughness was actually less than the fitted value of 0.6 nm.

5.1. Interpretation

We conclude that these multilayer d-spacings are uniform to the 1% level around the cylindrical azimuth of the mirror and that the d-spacing of the multilayers is also constant as a function of depth at about the 1% level. We base these conclusions on the comparison of the data with the IMD model which was based on just on a single d-spacing. First, we used exactly the same d-spacing to fit the two measurements at 10 keV made at different rotations about the optical axis of the mirror. Second, we were able to derive exactly the same d-spacing for the reflectivity of the much more penetrating 30 keV X-ray results as we derived for the 10 keV results. Furthermore, the detection of 3 peaks at 10 keV and the narrowness of the peaks at 10 and 30 keV plus that we needed only one d-spacing for our excellent fit to the data indicates that we have a well defined coherent set of multilayers.

The absolute reflectivity is difficult to calculate, however, since geometrical effects clearly affected the reflectivity measurements. For example, in Figure 3a below 0.5 degrees, the reflectivity raises more rapidly than the theoretical curve. In Figure 3b, the reflectivity at angles below 0.5 degrees first raises more rapidly and then falls below the theoretical curve. Therefore, a comparison between the direct beam and the reflected beam could not be used to produce a viable absolute reflectivity number. Rather, we use the results of our model fitting and conclude that the absolute reflectivity of the primary (first) multilayer peaks is well over 10%. Furthermore, only a 20% increase in signal was detected when the detector slit was opened to 2 mm in the vertical direction, which indicated we did not observe a significant amount of diffuse scattering.

5.2. Followup Work

When time and funding permit, we will scale up to making about 12 cm diameter and 30-40 cm long high quality Wolter I mirrors. In the mean time, we plan to make new measurements with pieces cut from our existing mirrors which will be small enough so that the geometrical effects of the gross mirror shape can be ignored. These measurements will then be used to make a more accurate determination of the absolute reflectivity. Second, we have in hand another even higher quality mandrel which we can use to make more and even better quality multilayer coated mirrors, and we will proceed to make graded d-spacing multilayer coated mirrors from this mandrel.

6. SUMMARY AND CONCLUSIONS

In summary we have shown that there are technical reasons as well a strong astrophysical motivation for developing the technology to make mirrors that are coated with multilayers using the IEMP. We have shown that the IEMP is viable by measuring the reflectivity of a mirror made by this process at both 10 keV and 30 keV. The results indicate highly efficient reflectivity at the primary Bragg peaks (well above 10%) from 60 Si/W multilayers. Although we have not yet made full fledged high quality Wolter I mirrors with graded d-spacing, we have provided enough evidence to proceed, and we are now confident we will succeed in making mirrors that will be used for imaging in the 40 keV range and beyond.

ACKNOWLEDGMENTS

This work was supported in part by NASA grant NAG5-5094. We thank Prof. Yip-Wah Chung for advice, and Dr. Derrick C. Mancini and the rest of the Argonne National Lab APS SRI-CAT for support and providing us access to the 2-BM-B beam-line. The use of APS was supported by the U.S. Department of Energy (DOE), Basic Energy Sciences (BES), Office of Science, under Contract No. W-31-109-Eng-30. We thank Drs. Allen S. Krieger and Daniel Parsignault for providing the mandrel. We thank Mr. Matthew Steele for fitting the X-ray reflectivity data with the IMD code.

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