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# Further Support of the Development of Advanced Multilayer Optics Coatings for X-Ray Imaging

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Further Support of the Development of Advanced Multilayer Optics  
Coatings for X-Ray Imaging

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Final Report

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## Introduction

SAO is working with LLNL to develop Wolter-I optics for x-ray imaging applications at energies of 15-50 keV and above at NNSA facilities. Our work includes R&D on advanced multilayer coatings as well as fabrication and testing of Wolter-I multilayer coated optics. It also includes delivery of custom 3.05-meter focal length Wolter optics with response at energies of interest to Sandia National Laboratories and LLNL. The mandrels for the custom optics are fabricated at MSFC. In previous contracts, SAO delivered several optics to LLNL with response at various energies.

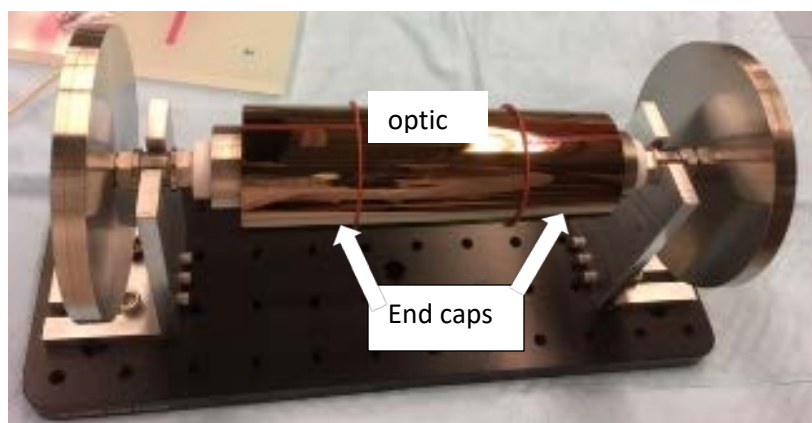
During this present contract period two tasks were carried out. Task1: the coating, replication and characterization of a custom optic with peak response at 22.8 keV, using the custom-designed SANZ mandrels. Task2: carrying out modifications to the coating chamber at SAO to accommodate new, longer, custom-designed mandrels (SANZlong) that will be fabricated at NASA MSFC in FY2020. The longer mandrels are necessary to mitigate end effects observed in the replication process for the small optics currently being fabricated.

## TASK1

Task1 was to fabricate an optic with spectral response peaked at 22.8 keV. Two optics, Ag4 and Ag5 were fabricated, characterized and delivered to LLNL for this task. Ag4 optic was fabricated using SANZ1a mandrel and Ag5 optic was fabricated using SANZ1b mandrel. Both mandrels were fabricated at MSFC using the same specifications as described below.

## Mandrel

Two SANZ mandrels (SANZ1a and SANZ1b) with identical specifications were delivered to SAO during a previous subcontract and were used to fabricate the optics for this contract. They were designed by LLNL and fabricated at MSFC. One of the mandrels is shown mounted on an inspection station in **Figure 1**. The mandrel is an ellipse-hyperbola (point-to-point focus) Wolter-I design with focal length 3.05 m and source optic distance of 677.78 mm. The mirror lengths of the hyperbolic and elliptic sections are  $LH = 30.0$  mm and  $LE = 30.97$  mm, respectively, with an inflection point radius of  $r_{int} = 23.3$  mm. The graze angles vary from  $0.6524 - 0.6327^\circ$  along the length of the hyperbola and from  $0.63311 - 0.6321^\circ$  for the ellipse. The mandrel can be used for Mo  $K\alpha$  (16.9 – 18.6 keV) and Ag  $K\alpha$  energies (21.5 – 23.4 keV) when using appropriate multilayer coatings.



**Figure 1.** SANZ1 mandrel shown on inspection station fixture. Mandrel is 139 mm long with optic length 60.97 mm. Optic is central part with end cap on either side. End caps are necessary to provide uniform fields during fabrication but are not part of the final optic.

## Replicated Optics

The optics are fabricated using an electroform nickel replication technique. One of the advantages of this technique is that a given mandrel can be replicated many times to fabricate several optics. After separating the shell from the mandrel, the mandrel is cleaned and replicated again to fabricate another optic. These mandrels have been replicated several times to fabricate optics with different energy responses for this collaboration.

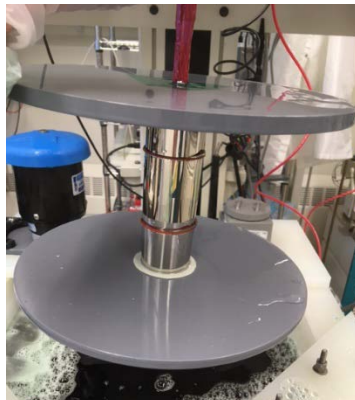
## Coating

Task 1 of this contract required fabricating an optic with peak energy response of 22.8 keV with FWHM  $\sim 1$  keV, therefore it was necessary to coat the optic with an appropriate multilayer. The materials chosen for the multilayer coating were tungsten and silicon. The coating design was based on a constant layer thickness to reflect x-rays at 22.8 keV, allowing for a total spread of  $\pm 1$  keV in the energy response of the optic. The energy window of  $\pm 1$  keV allows for small deviations in the thickness of the individual layers that are deposited yet is still a small enough window to allow for discrimination between K-shell emission and continuum emission of materials of interest.

Model #	Coating thickness (Å)		Max R (%)
	E end	H end	
1	25.8	25.8	32
2	25.6	26.1	16
3*	26.0 (N=30)	26.5 (N=30)	28
	24.9 (N=30)	25.4 (N=30)	

**Figure 2.** Table shows theoretical maximum reflectivity for 3 multilayer coating models which yield response at 22.8 keV. All models use 60 layer pairs. 1: bi-layers with the same constant d for all layers; 2: assumes slight variation in coating uniformity along the axis - bilayer thickness varies by 0.5 Å; 3: uses 2 constant-d block coating. Model 3, the 2 block constant-d was chosen for these optics (see text).

**Figure 2** compares the theoretical single bounce reflectivity for a few of the models considered for the multilayer coating. Model 1 with a constant-d stack of 25.8 Å yields the highest theoretical single bounce reflectivity. However the deposition time for this multilayer coating is  $> 12$  hours. The software corrects for an expected change in deposition rate over time, but the correction is not perfect, therefore model 3 with a double block stack design was chosen which allows for a small variation in coating thickness with depth.



**Figure 3.** Mandrel fixtured for plating bath



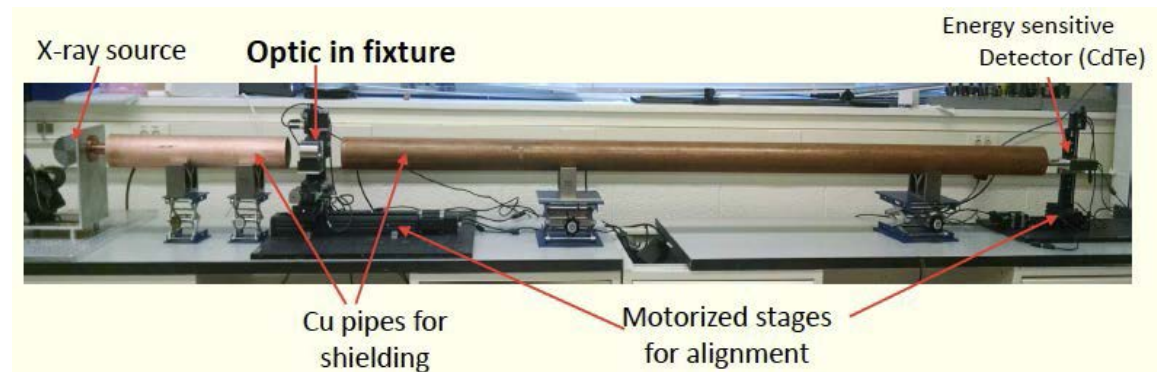
**Figure 4.** Mandrel (left) and replicated multilayer coated optic (right) after separation; W/Si multilayer coating is on inside of optic; NiCo optic is 300  $\mu\text{m}$  thick.

## Optic

After coating the mandrel was fixtured as shown in **Figure 3** and placed in the electroforming bath for ~ 15 hours to grow a 300 microns thick nickel substrate over the multilayer. The mandrel was then removed and placed in a cold water bath to separate the multilayer coated optic from the mandrel (see **Figure 4**).

## X-Ray testing

X-ray testing of the optic was carried out using the beamline shown in **Figure 5** which includes: an x-ray source, an Amptek detector, an X/Y tip/tilt stage and linear stage to position the optic and a 3 m long pipe for shielding. The source is an Oxford tungsten x-ray source with 50 micron nominal spot size; the Amptek detector is a 5 mm x 5 mm single pixel silicon drift detector.

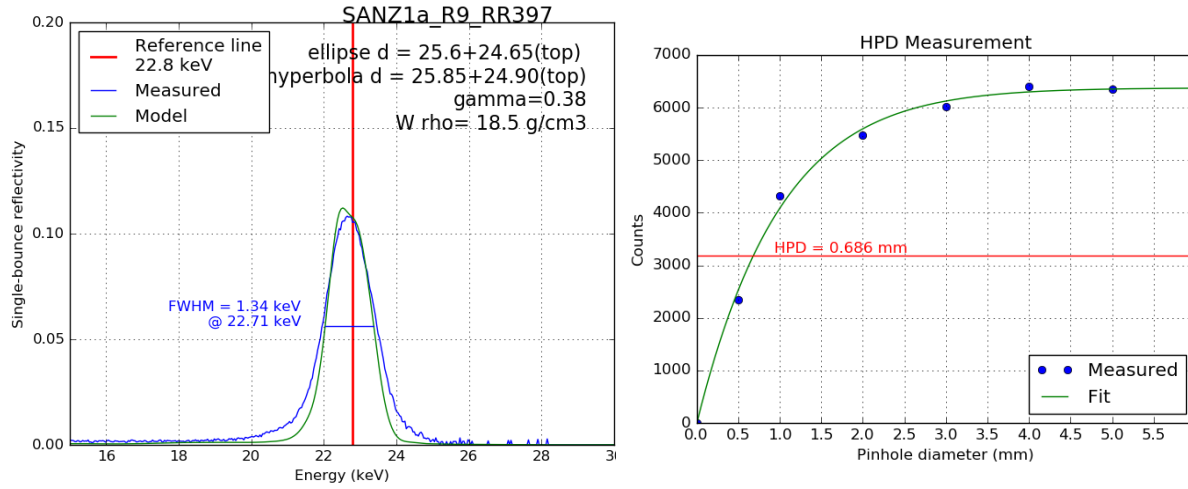


**Figure 5.** X-ray beamline used to characterize response of multilayer coated Wolter X-ray optics. Two detectors are used for this configuration: CdTe detector (shown) and also silicon drift detector which was used for characterizing SANZ optics.

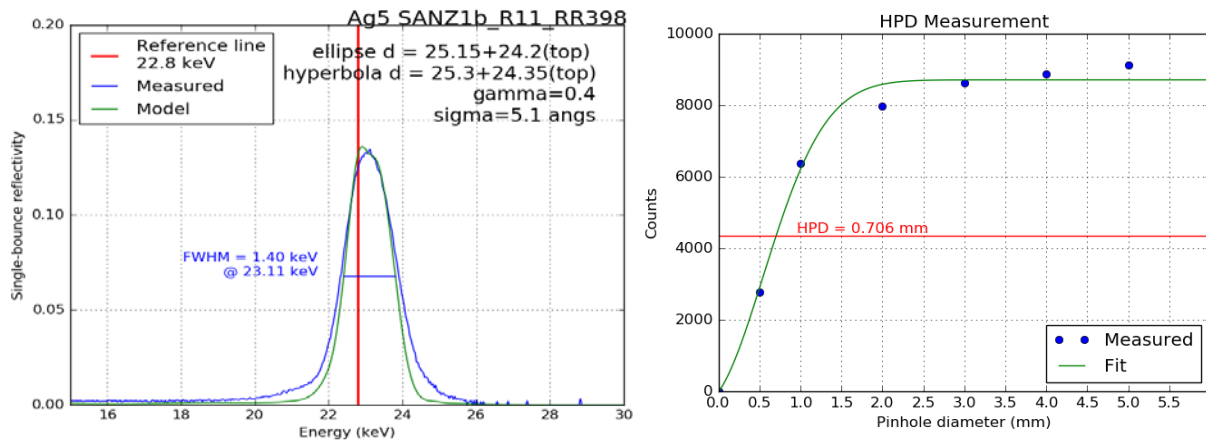
Two optics, Ag4 and Ag5, were fabricated, tested and delivered to LLNL. The x-ray data are presented in **Figure 6 and 7** below which present single bounce reflectivity vs. energy plots to show the spectral response; also shown are HPD measurements for each optic. The measured peak reflectivity for Ag4 was 22.7 keV and for Ag5 was 23.1 keV; the goal was 22.8 keV. The difference in peak energies between the two optics is due to slight difference of ~ 0.5 Å in d-spacing of the multilayer stack, as shown in the figure. The FWHM is 1.34 and 1.4 respectively for Ag4 and Ag5. The measured HPD for Ag4 was 56 arcsec and was 60 arcsec for Ag5. We note that Ag4 was replicated from SANZ1a mandrel and Ag5 was replicated from SANZ1b mandrel. Although the design specification for both mandrels was the same, it's not surprising that there are small differences in the final mandrels and SANZ1a seemed to be slightly better in terms of angular resolution. Table 1 below presents some of the relevant measured data for each replicated optic.

**Table 1**

Replication Run	Purpose	Peak	HPD	Mandrel
R9_RR397	Ag 4	22.7 keV (target: 22.8 keV)	HPD: 56 arcsec - FWHM: 1.3 keV	Sandia 1A
R11_RR398	Ag 5	23.11 keV (target: 22.8 keV)	HPD: 60 arcsec - FWHM: 1.4 keV	Sandia 1B



**Figure 6.** Characterization of optic **Ag4**. **Left:** Plot shows data (blue) and model (green) for single bounce reflectivity vs. energy. 60 W/Si bi-layers were deposited. Ellipse end used 30 bilayers with d-spacings of 25.6 Å on bottom followed by d-spacings of 24.65 Å on top; hyperbola used 30 bilayers with d-spacings of 25.85 Å on bottom followed by d-spacings of 24.90 Å on top to achieve a peak energy of 22.7 keV, as shown. Reference line shown at 22.8 keV was target peak energy. **Right:** plot of measured HPD shows 686 microns (56 arcsec).



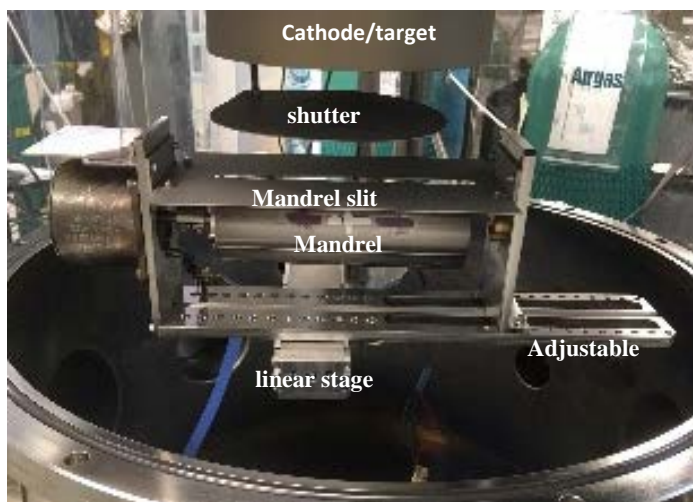
**Figure 7.** Characterization of optic **Ag5**. **Left:** Plot shows data (blue) and model (green) for single bounce reflectivity vs. energy. 60 W/Si bi-layers were deposited. Ellipse end was 30 bilayers with d-spacings of 25.1 Å on bottom followed by d-spacings of 24.2 Å on top; hyperbola was 30 bilayers with d-spacings of 25.3 Å on bottom followed by d-spacings of 24.3 Å on top to achieve a peak energy of 23.1 keV, as shown. Reference line shown at 22.8 keV was target peak energy. **Right:** plot of measured HPD shows 706 microns (60 arcsec).



## Task 2

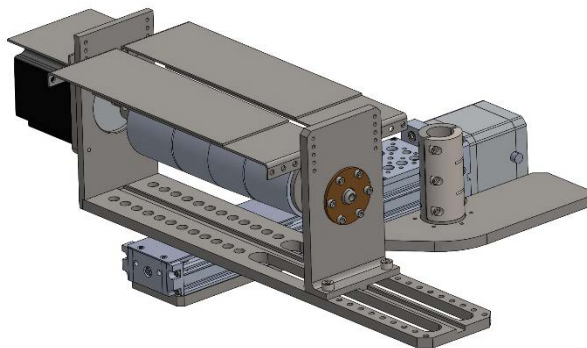
### Modifications to coating chamber

The separation layer vacuum coating chamber is equipped with two 4-inch diameter cathodes which down-sputter onto the mandrel. The mandrel rotates about its axis while it passes underneath the target during the coating cycle. The goal of task 2 was to modify this chamber to accommodate new longer mandrels. To maintain a uniform coating thickness with the longer mandrels it was necessary to introduce a computer controlled linear stage to keep the mandrel centered under the cathode as it sweeps across the target area. The new mandrels will be up to 1.5 times the length of the present SANZ mandrel and are being designed to mitigate end effects observed in the replication process for the small optics currently being fabricated. The modified coating chamber is shown in **Figure 8**.



**Figure 8.** Photo of inside of vacuum coating chamber shows cathode (top) along with target shutter. Mandrel is mounted in new adjustable length fixture with mandrel slit; also shown is the added linear stage, necessary to keep the mandrel centered under the target as it rotates.

**Figure 9** presents the engineering drawing for the mandrel fixture housing and shows how it integrates with the linear stage and adapter plate for the rotation/linear stage interface. 2<sup>nd</sup> motor used for linear stage control is also visible.



**Figure 9.** Engineering drawing showing new design for mandrel fixture housing, linear stage, mandrel slit plate and adapter plate for rotation/linear stage interface.

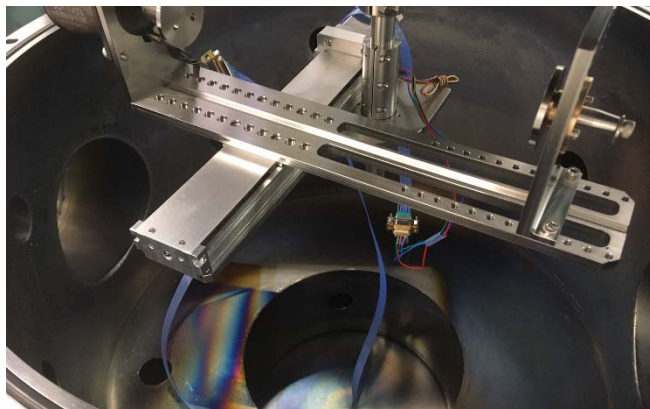
**Figure 10** is an engineering CAD model which shows the SANZ mandrel mounted in the modified mandrel fixture housing. The additional fixture length designed for the SANZlong mandrel can be seen in the model. Also visible above the mandrel is the mandrel slit which is



needed to constrain the deposition angle. The motor used for mandrel rotation is also shown in this model. The detail drawing in **Figure 10** (right) shows the bushing and end plate used to support the mandrel. This end plate is adjustable to accommodate mandrels as long as 1.5 x SANZ mandrel length. **Figure 11** is close-up detail of completed fixture plus linear stage in vacuum chamber.



**Figure 10. Left:** Engineering design showing modified mandrel fixture housing with mandrel slit plate. **Right:** Detail design of bushing and end plate to support mandrel while rotating.



**Figure11.** Photo shows finished modifications (in vacuum chamber) which include: programmable 150mm linear stage, support bracket for longer mandrels, fixturing to secure mandrels during rotation and adapters to fixture to vacuum motors.

## Testing

In addition to hardware modifications software modifications were also necessary to control the additional linear stage motion and integrate it with the motion of the existing two motors. The SANZ mandrel was used to carry out initial testing/shake-down of the hardware modifications.

## Summary

Two optics, Ag4 and Ag5, with peak response at 22.7 and 23.1 keV, respectively, were fabricated and tested during this phase and the optics were delivered to LLNL. In addition both hardware and software modifications were carried out for the coating chamber to accommodate new, longer, custom-designed mandrels (SANZlong) that will be fabricated at NASA MSFC in FY2020. During the next phase (FY2020) a longer test mandrel will be fabricated and uniformity coating tests will be carried out to test the new software algorithms introduced to integrate the three motors.