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

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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 for National Nuclear Security Administration (NNSA) facilities. SAO work includes R&D on advanced multilayer coatings as well as fabrication and testing of Wolter 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 optics are fabricated using an electroform nickel replication technique which has been used to fabricate optics for X-ray astronomy satellites over the past two decades. This technique starts with a figured, polished mandrel from which the optics are replicated. To carry this technique over to fabricate optics for NNSA facilities, several additional barriers had to be overcome to deposit multilayer coatings on integral optics with radii as small as 25 mm. These breakthroughs allow fabrication of optics which meet the spatial and spectral specifications required by NNSA facilities.

During this present contract period SAO delivered four custom optics to LLNL with spectral response at or near the K_α line of silver, 22.16 keV. The optics are referred to below as: Ag0, Ag1, Ag2, Ag3.

Mandrel

Two SANZ mandrels (SANZ1a and SANZ1b) with identical specifications were fabricated by MSFC and delivered to SAO. SANZ1b, shown **in Figure 1,** was used to fabricate the optics for this contract. 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 rint = 23.3 mm. The graze angles vary from $0.6524 - 0.6327^\circ$ along the length of the hyperbola and from $0.63311 - 0.6321^\circ$ along the ellipse.

Fabrication/Coating

The optics were replicated from the SANZ mandrel 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. Appropriate tungsten and silicon constant-d thickness multilayer coatings were used to provide the required narrow bandwidth about the target energies for response at 22.16, 22.5 and 22.8 keV.

The coating design was based on a constant layer thickness to reflect x-rays at the appropriate energy. Over the course of this study, the energy of interest ranged from 22.16 keV to 22.8 keV as more testing was carried out at LLNL and SNL and specifications were refined. The energy window of ±1 keV about the target energy 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.

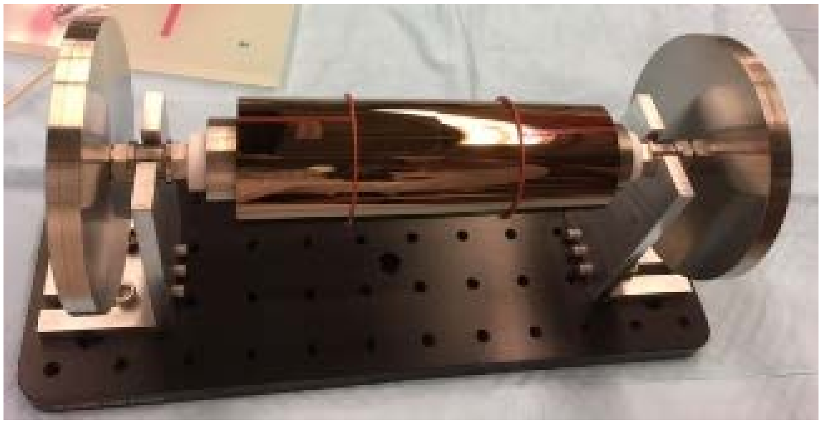
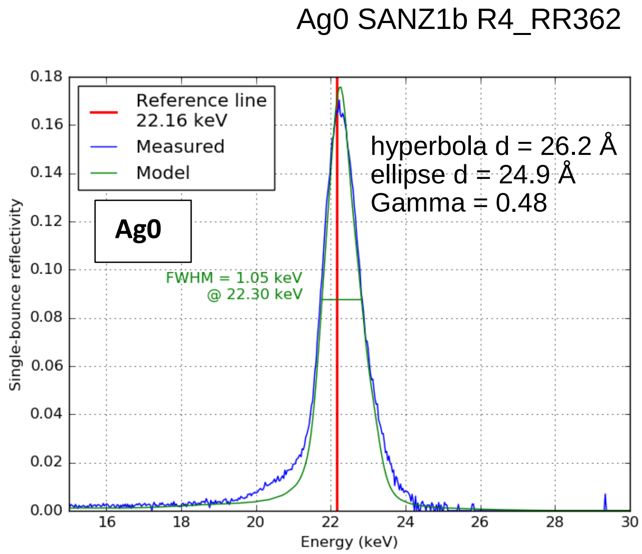


Figure 1. SANZ1b mandrel shown on inspection station fixture. Mandrel is 139 mm long with optic length 60.97 mm. Optic is central part with end cap on either side. End caps are necessary to provide uniform fields during fabrication but are not part of the final optic.

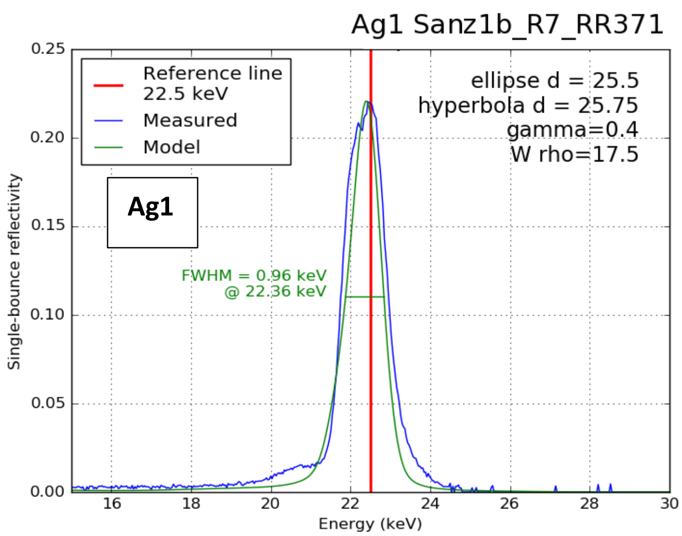
X-Ray testing

Before delivery to LLNL, X-ray testing of all optics was carried out at the SAO beamline 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.

The first two optics, Ag0 and Ag1, were fabricated with target energy peaks specified at 22.16 and 22.5 keV. The coating design for Ag0 and Ag1 was for 60 constant-d bi-layers with target d-spacing of 25.0 Å and 25.4 Å respectively, with the same coating on both the ellipse and hyperbola sides. Because the deposition time for this multilayer coating was > 12 hours a software correction was added for an expected change in deposition rate over time (due to target wear and heating effects). **Figure 2** shows the measured single bounce reflectivity vs. energy for the first two fabricated optics, Ag0 and Ag1. The blue line in the plots is the data and the green line is a model fit to the data. Next to each plot is a table of the d-spacing showing the thickness used to create the model fit vs. the target d-spacing. Although the target d-spacing was the same for both the ellipse and hyperbola sides, the achieved d-spacing differs slightly between the two sides; this effect is a few percent and may be due to a non-uniformity in the plasma produced by the cathodes. Also the measured d-spacing differed from the target by a few percent which may be due to a small error in the software correction that was made to account for heating effects and target wear. The peak reflectivity achieved by Ag0 and Ag1 was considerably lower than the theoretical 32%. This may be associated with the microroughness of the mandrels, but investigations to better understand causes of this are ongoing.



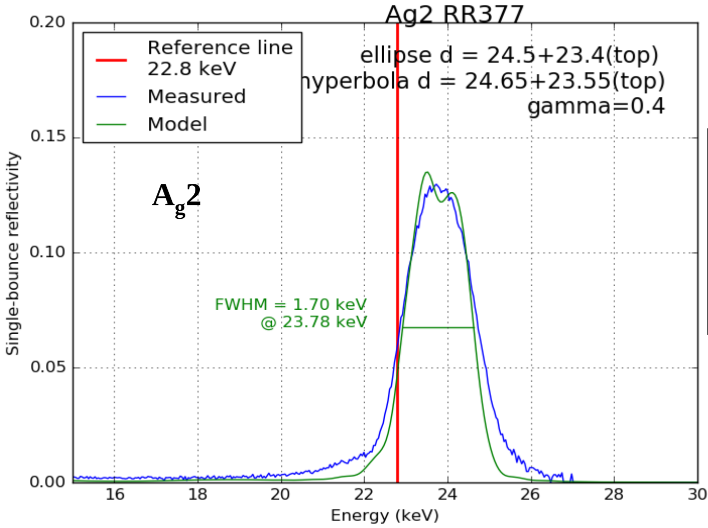
Ag0	d-space(Å)	
	model	target
Hyp	26.2	25.0
Ell	24.9	25.0



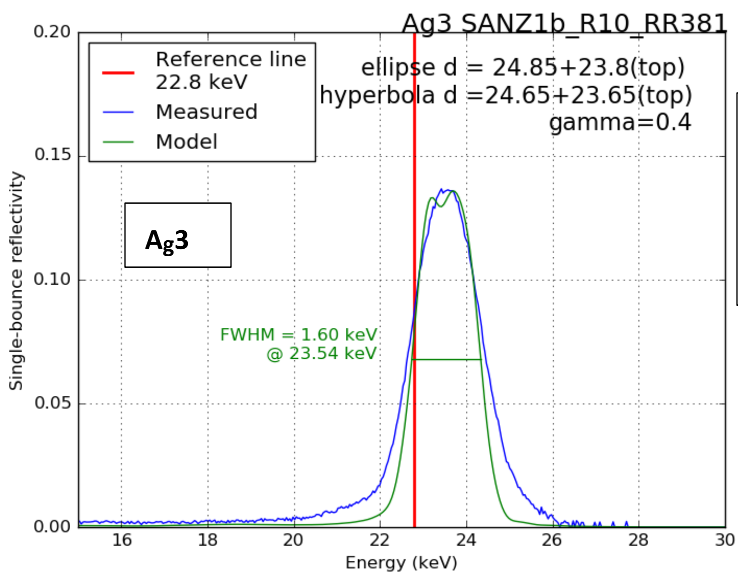
Ag1	d-space(Å)	
	model	target
Hyp	25.75	25.4
Ell	25.5	25.4

Figure 2. Plots show data for single bounce reflectivity vs. energy for Ag0(top) and Ag1(bottom). Blue line is measured data and green line is model fit; the red line indicates the target for the peak energy for each optic. As the plots indicate, the spectral peak achieved for each was within 0.15 keV of target. As more testing was carried out by SNL and LLNL, the specification for peak energy changed from 22.16 keV for Ag0 to 22.5 keV for Ag1. The tables on the right of each plot show the multilayer d-spacing thickness, in angstroms, used to create the model fit along with the d-spacing target we were trying to achieve. The d-spacings which are shown in the tables indicate that the d-spacing of the multilayer met the spec to better that 1.0 angstrom. (See text for more detail of coating.)

The next two optics, Ag2 and Ag3, were designed for a peak energy of 22.8 keV. They were designed with a double stack, i.e. 2 slightly different constant-d spacings with 30 bilayers for each. The double stack design was introduced to broaden the FWHM slightly (from 1keV to 2 keV).



Ag2	d-space(Å)	
	BOTTOM model/target	TOP model/target
Hyp	24.65/25.4	23.55/24.6
Ell	24.5/25.4	23.4/24.6



Ag2	d-space(Å)	
	BOTTOM model/target	TOP model/target
Hyp	24.65/25.4	23.65/24.6
Ell	24.85/25.4	23.8/24.6

Figure 3. Plots show single bounce reflectivity vs. energy data for Ag2 (top) and Ag3 (bottom). Blue line is measured data and green line is model fit; the red line indicates the goal for the peak energy for each optic. The spectral response encompassed the goal energy for these 2 optics, although the peak was higher than the goal – due to d-spacing being ~ 1 angstrom too thin. The goal for the peak energy was changed during the study, therefore d-spacing thickness had to be modified a few times. See text for more detail of coating. The tables below each plot show the multilayer d-spacing thickness, in angstroms, used to create the model fit along with the d-spacing target we were trying to achieve.

The plots for reflectivity vs. energy for Ag2 and Ag3 are shown in **Figure 3**. These were the first two optics fabricated for target energy of 22.8 keV, and the first two fabricated with a double stack. As shown in Figure 3 (top), the peak energy measured for Ag2 was 23.8 keV, 1 keV higher than the target; the peak energy measured for Ag3 was a bit closer to 22.8 at 23.5 keV. It takes a few iterations to produce the right d-spacing along with the proper corrections for wear and heating. In both cases, the d-spacing was off by ~ 1 Å, which indicates the small margin of error available when fabricating these optics.

Table 1 presents a short summary with some of the relevant data of the four optics that were fabricated. The difference in measured peak energies between the optics is due to a difference in d-spacing of the multilayer coating. The specification for the target peak energy was changed over the course of this study. The initial specification was for spectral response peak at 22.16 keV. This specification was changed to 22.5 keV and finally 22.8 keV as the study progressed and as further testing was carried out at LLNL and SNL. Therefore it was necessary to re-tune the d-spacing of the multilayer stack from optic to optic. Heating effects during the deposition and expected erosion of the targets make it difficult to set the exact parameters for a given energy (d-spacing) and it typically takes a couple of coating trials to produce an optic with the specified energy peak.

Table 1		
Optic#	**Spectral peak Measured/target**	**HPD**
A_g0	22.3 keV / 22.16 keV	66 arcsec
A_g1	23.4 keV / 22.5 keV	55 arcsec
A_g2	23.8 keV /22.8 keV	82 arcsec
A_g3	23.5 keV/ 22.8 keV	54 arcsec

The expected HPD for a replicated optic can be extrapolated from the metrology data of the mandrel. The expected HPD for an optic replicated from the current SANZ mandrel is ~ 55 arcsec. **Figure 4** presents plots for HPD measurements for each of the four optics. The HPD measured for Ag1 and Ag3 agrees with the expected. However the measured values for Ag0 and Ag2 are well outside the expected HPD. **Figure 5** is a photograph showing the edge of Ag0. Clearly visible around the edge of the replicated optic are pits, which can develop around the edges of the optic during growth if there is not enough agitation in the bath and/or not enough surfactant (or wetting) in the solution during growth. This pitting effects the final figure of the optic and explains the higher HPD. After observing the pitting on Ag0, the pump was adjusted to provide more agitation in the bath and the HPD of the next optic, Ag1, was as expected. After observing the pitting again on Ag2, the surfactant was adjusted to improve the wetting and again the HPD of the following optic, Ag3, was as expected.

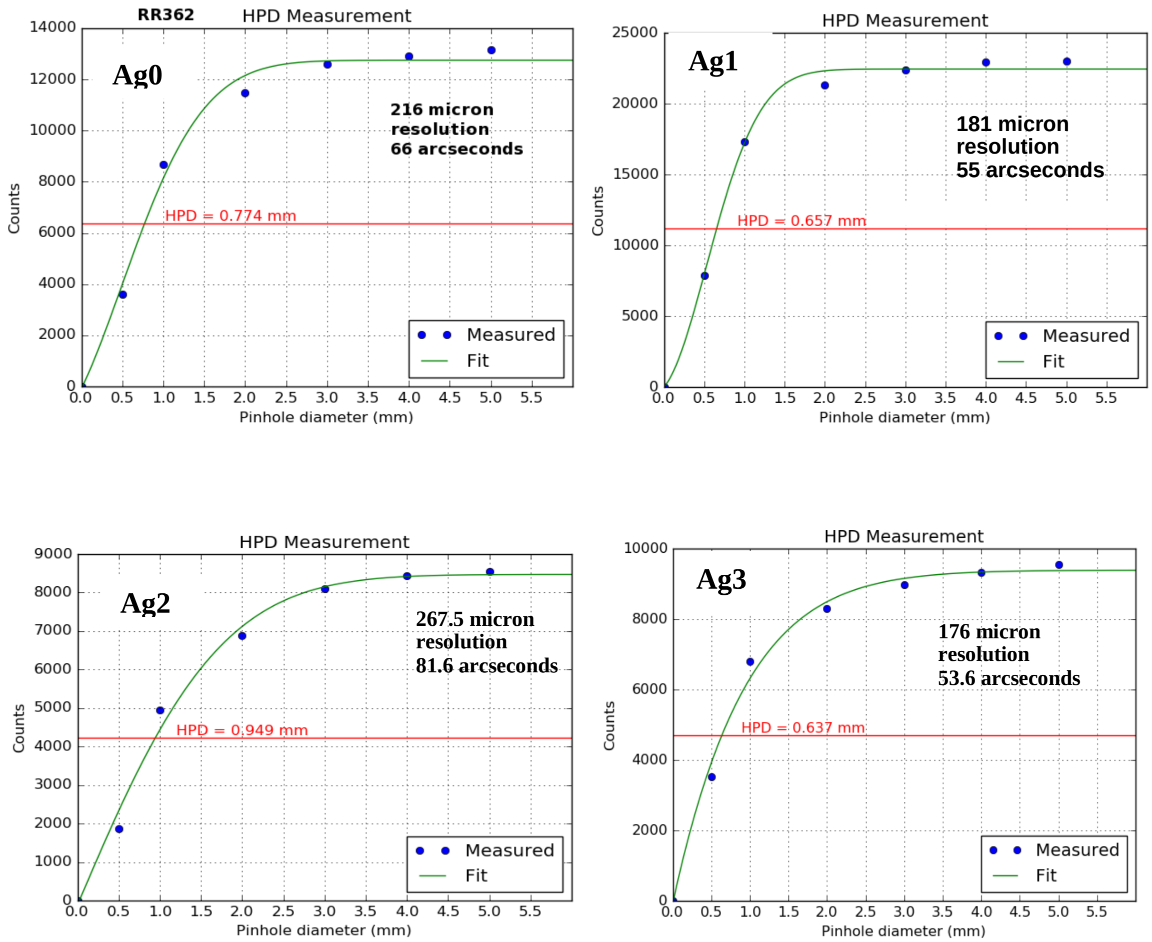


Figure 4. HPD measurements of the four Ag optics delivered to LLNL. Ag1 and Ag3 had measured HPD consistent with what was expected from the mandrel. The higher than expected HPD of the Ag0 and Ag2 optics was due to pits at edge of optic caused by low surfactant in bath and low agitation of bath during plating (see text).

Figure 5. End of optic shows pits which effects final figure of optic. This was result of not enough surfactant in bath.

Summary

Four custom optics were fabricated and delivered to LLNL for this contract period. The optics, referred to as : Ag0, Ag1, Ag2, Ag3 were replicated from the SANZ1b mandrel which 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. All four optics were tested at SAO in the X-ray beamline before delivery. The peak energy for each optic along with measured HPD is shown in the plots in the included figures. The target energy for all optics fell within the measured spectral peaks; Ag0 and Ag1measured peak was at the target energy whereas Ag2 and Ag3 peak was slightly above the target; this was due to a re-calibration which was necessary for the 2-block d-spacing used for Ag2 and Ag3. The measured HPD for Ag1 and Ag3 was as expected; the HPD for Ag0 and Ag2 was higher than expected due to lack of agitation and surfactant in the bath during growth.