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Compatibility of iridium thin films with the Silicon Pore Optics technology for Athena

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ABSTRACT

The development of high-quality thin film coatings for the Athena X-ray optics is progressing, following the commissioning of an industrial scale coating facility. The assembly of silicon pore optics into mirror modules for the Athena telescope requires wet-chemical exposure of coated mirror plates to prepare bonding areas for stacking, as well as an annealing step to improve bond strength. It is therefore critical to evaluate how these post-coating processes could affect the mirror coating performance and stability.

We present X-ray reflectometry characterization of iridium thin films deposited on photoresist patterned Silicon Pore Optics plates to investigate the compatibility with the stacking process steps for the manufacturing of the Athena optics.

Keywords: Athena, X-ray optics, Silicon Pore Optics, thin film, iridium

1. INTRODUCTION

The Athena (Advanced Telescope for High Energy Astrophysics) observatory is a selected L-class mission under preparation by the European Space Agency (ESA)¹. The telescope will feature X-ray optics based on the Silicon Pore Optics (SPO) technology with individual mirror plates coated with X-ray reflective thin films.

As the optics development is scaling up for the industrialized mirror plate production and assembly²⁻⁴, the development of X-ray mirror coatings for Athena is progressing alongside,⁵⁻¹⁴ with several aspects of material stability¹⁴⁻¹⁶ and optimization of performance¹⁷ being investigated.

The thin film designs under study for the Athena X-ray mirrors at present include iridium single-layers, and bilayers of iridium with a low-Z overcoat of either boron carbide, silicon carbide, or carbon.

One of the critical aspects in adopting a coating design is addressing the compatibility of the candidate materials with the wet-chemical, mechanical and thermal post-processing required for the SPO technology.

Previous studies based on preliminary wet-chemical treatment have indicated compatibility of iridium thin films^{14,18}. Since then, further refinement of the wet-chemical processes has been enabled with the installation of a fully automated wet bench at cosine Research BV¹⁹.

In this paper, we present X-ray reflectometry (XRR) characterization of coated, photoresist patterned SPO mirror plates to evaluate the compatibility of iridium thin films with the most representative post-coating processes of the SPO technology.

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2. SAMPLE PREPARATION

The iridium thin films were produced by DC magnetron sputtering in the Von Ardenne BS1500S coating machine installed at cosine Research BV^{20,21}.

The iridium thin films were deposited onto photoresist patterned SPO plates, representative of the Athena middle-radius mirror plates. The plates were exposed to in-situ plasma cleaning prior to thin film deposition in order to remove surface contamination, ensuring a smooth surface and compatibility with the wet-chemical cleaning procedure^{14,22}.

For coating, the argon sputtering pressure was $3.4 \cdot 10^3$ mbar, and a magnetron power of 1800 W was applied. The iridium thin films were coated with a thickness of ~ 20 nm. While the optimized iridium-only design for Athena assumes a thickness of 10 nm¹⁰, these thin films are still representative for characterization of the overall material compatibility with the SPO processing steps.

2.1 Post-coating processing

Figure 1 presents the processing steps at plate and stack level. In this study, the impact of the latter three post-coating treatments, described below, is investigated in terms of thin films quality and stability.

Figure 1: Overview of the processing steps from uncoated SPO plate to X-ray mirror stacks. The reflector surfaces are prepared by plasma cleaning prior to coating. After thin film deposition, the coated SPO plates undergo wet chemistry, stacking, and annealing.

The SPO technology utilizes two wet-chemical processes of coated mirror plates to prepare the surfaces for stacking: *Lift-off and cleaning*. Photoresist lift-off is performed using a DMSO bath to remove the parallel stripes of the thin film, exposing the silicon bonding areas. A diluted Standard Clean 1 (SC-1) is used to remove organic material and activate the bonding surfaces. Both wet-chemical steps were carried out in the new wet bench at cosine Research BV.

After wet-chemical treatment, SPO plates are precisely stacked using a robotic system. The post-stacking annealing is used to increase the bond strength between adjacent plates in the stack. The evaluation of effects of annealing representative of the stacking procedure was carried out using a LabCompanion OV-11 vacuum oven at DTU Space. The stack was heated to 200 °C for 50 hours in air, including 2 hours of pre-heating.

Figure 2: The iridium coated samples. From left to right: SPO plate 01219-12 (no post-coating treatment), SPO plate 01219-09 (lift-off and cleaning), and SPO stack HPO-1722 (before annealing).

3. CHARACTERIZATION AND MODELING

The potential effects of thin film post-processing required for the SPO technology were evaluated through XRR characterization of iridium coated middle-radius SPO plates without any post-coating treatment (ID 01912-12)

and after lift-off and cleaning (ID 01912-09). A 10-plate middle-radius SPO stack (HPO-1722) was characterized before and after annealing.

XRR measurements were carried out at DTU Space using the 8.048 keV reflectometer and the LEXR system operating at 1.487 keV.²³ The angular resolution for both systems is assumed to be 7 mdeg. For the 8.048 keV measurements, the samples were illuminated with a $0.4 \times 0.12 \text{ mm}^2$ beam size, while 1.487 keV measurements were performed with a $0.5 \times 0.4 \text{ mm}^2$ beam. The error bars indicate statistical uncertainties only.

The SPO plates were measured at the center position. For the stack, the top plate was measured in three spots: x0 (center), xm2 (2 mm above center), xp2 (2 mm below center). The top plate was assumed to be representative of the reflective surfaces throughout the stack.

The XRR measurements were fitted in IMD²⁴ using a model with a silicon dioxide substrate and an iridium thin film layer. The material densities were assumed as the nominal values tabulated for the CXRO/LLNL determination of optical constants, as previous studies suggest that the iridium is deposited with the same density within $< 0.1\%$ ¹³.

For the 1.487 keV data, a hydrocarbon overlayer, modeled as a simple COH compound with a fixed density of 1.00 g/cm^3 , was included to account for a surface contamination layer.

3.1 Results

Figures 3-6 present XRR measurements of the iridium coated mirror plates along with the best fits. The best fits parameters are given in Table 1.

Figure 3: XRR measurements at 8.048 keV and 1.487 keV with best fits for iridium coated SPO plate 01219-12 (no post-coating treatment).

Figure 4: XRR measurements at 8.048 keV and 1.487 keV with best fits for iridium coated SPO plate 01219-09 (after wet-chemical lift-off and cleaning).

Figure 5: XRR measurements at 8.048 keV and 1.487 keV with best fits for iridium coated stack HPO-1722 (before annealing). The three measured spots are offset by 10^{-1} and 10^{-2} in reflectance for visibility.

Figure 6: XRR measurements at 8.048 keV and 1.487 keV with best fits for iridium coated stack HPO-1722 (after annealing). The three measured spots are offset by 10^{-1} and 10^{-2} in reflectance for visibility.

Table 1: Fit parameters to XRR measurements performed at 1.487 keV and 8.048 keV of the iridium coated SPO plates and stack. Parameters marked with † were coupled during fitting.

ID	Treatment	Data	Position	Hydrocarbons		Iridium		Substrate
				z (nm)	σ (nm)	z (nm)	σ (nm)	σ (nm)
01219-12	No treatment	1.487 keV	x0	1.6	$0.35^{\dagger}$	19.3	$0.35^{\dagger}$	0.51
		8.048 keV	x0	-	-	19.1	0.33	0.53
01219-09	Lift-off, cleaning	1.487 keV	x0	1.8	$0.36^{\dagger}$	19.3	$0.36^{\dagger}$	0.49
		8.048 keV	x0	-	-	19.0	0.38	0.47
HPO-1722	Before annealing (Lift-off, cleaning, stacking)	1.487 keV	x0	1.9	$0.39^{\dagger}$	19.5	$0.39^{\dagger}$	0.52
			xm2	1.8	$0.37^{\dagger}$	19.5	$0.37^{\dagger}$	0.50
			xp2	1.8	$0.38^{\dagger}$	19.5	$0.38^{\dagger}$	0.52
		8.048 keV	x0	-	-	19.3	0.31	0.47
			xm2	-	-	19.4	0.33	0.46
			xp2	-	-	19.3	0.32	0.49
HPO-1722	After annealing (Lift-off, cleaning, stacking, annealing)	1.487 keV	x0	2.1	$0.38^{\dagger}$	19.5	$0.38^{\dagger}$	0.47
			xm2	2.1	$0.42^{\dagger}$	19.5	$0.42^{\dagger}$	0.50
			xp2	2.1	$0.40^{\dagger}$	19.5	$0.40^{\dagger}$	0.49
		8.048 keV	x0	-	-	19.3	0.35	0.44
			xm2	-	-	19.3	0.34	0.40
			xp2	-	-	19.4	0.35	0.45

The best fit parameters give no indication that the iridium-only thin film is affected by the SPO post-coating process steps. The minor iridium thickness variation is attributed to coating non-uniformity of a few percent.

Figure 7 shows the measured central spots on the SPO plates and the top plate of the stack to compare the XRR performances at 8.048 keV and 1.487 keV. Within the measurement reproducibility, these results support the observation that iridium thin films are not affected by the wet-chemical treatment, mechanical stacking, and annealing.

Figure 7: XRR measurements at 8.048 keV and 1.487 keV of the iridium coated plates after each step in the post-coating sequence.

4. SUMMARY

Thin films of iridium coated on photoresist patterned SPO plates were presented to evaluate the impact of the most representative post-coating treatments required for the industrialized production of SPO mirrors for the Athena telescope. Based on the presented XRR measurements of the iridium-only coating, compatibility with the SPO production processes was demonstrated.

The measured XRR performance at 8.048 keV and 1.487 keV of the coated mirrors and the derived best fit parameters give no indication of change in thickness, roughness or density of the iridium single-layer thin films. The $\sim 2\%$ iridium thickness variation observed across the presented samples is expected from the margin of coating uniformity for the coating machine.

XRR measurements of the SPO stack were carried out on the reflective surface of the top plate only, assuming this to be representative of the overall performance of the coated stack. However, since a temperature gradient through the stack during annealing could have an effect on the coating, in particular for the bilayer designs with a low-Z material, a strategy to best illuminate the pores and measure the thin film performance is being developed.

The results suggest that iridium thin films produced in the Athena-dedicated coating machine are compatible with the entire SPO stacking process. This establishes a baseline for future studies to investigate the stability of performance for the low-Z overcoat candidate materials and to establish their compatibility with the SPO technology.

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