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Total number of authors:
17

Published in:
Proceedings of SPIE

Link to article, DOI:
[10.1117/12.2594559](https://doi.org/10.1117/12.2594559)

Publication date:
2021

Document Version
Publisher's PDF, also known as Version of record

[Link back to DTU Orbit](#)

Citation (APA):

Henriksen, P. L., Ferreira, D. D. M., Massahi, S., Svendsen, S., Gellert, N., Christensen, F. E., Jegers, A. S., Landgraf, B., Thete, A., Collon, M., Handick, E., Cibik, L., Krumrey, M., Gollwitzer, C., Ferreira, I., Shortt, B., & Bavdaz, M. (2021). Impact of annealing on performance of X-ray mirror coatings for Athena. In S. L. O'Dell, J. A. Gaskin, & G. Pareschi (Eds.), *Proceedings of SPIE: Optics for EUV, X-Ray, and Gamma-Ray Astronomy X* [118220D] SPIE - International Society for Optical Engineering. Proceedings of SPIE - The International Society for Optical Engineering Vol. 11822 <https://doi.org/10.1117/12.2594559>

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Impact of annealing on performance of X-ray mirror coatings for Athena

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ABSTRACT

As part of the manufacturing process of mirror modules for the Athena X-ray telescope, Silicon Pore Optics plates are assembled into mirror module stacks. The plates that form each stack are held together by direct bonding, relying on van der Waals forces and covalent bonds for adhesion. One way to increase the strength of the covalent bonds is through annealing of the mirror stacks. It is of critical importance to the mission to ensure compatibility between the reflective coating and any post-coating processing of the plates. We present our findings of the impact of annealing on the X-ray reflectance of coated mirrors relevant for the Athena mission. These are Ir single layers, as well as Ir/B₄C, Ir/SiC, and Ir/C bilayers. We investigate the effect on the performance of the coatings after annealing at atmospheric pressure and at a low vacuum using X-ray reflectometry. B₄C is found to suffer degradation from annealing under atmospheric conditions but not when annealed in vacuum. All other materials investigated are robust to atmospheric annealing.

Keywords: Athena, X-ray mirrors, reflective coatings, annealing, X-ray reflectometry

1. INTRODUCTION

The Advanced Telescope for High-Energy Astrophysics (Athena) is a large-class ESA selected mission aimed at studying the hot and energetic Universe.¹ The X-ray optics will be based on Silicon Pore Optics (SPO) technology and consist of more than 100,000 plates individually coated and stacked to form the complete mirror assembly. The stacking of the SPO plates must ensure that the plates are bonded with a strength sufficient to withstand the forces involved with the launch. Prior to deposition of the X-ray reflective coating, stripes of photoresist are deposited on the plates that, upon removal, expose part of the plates to allow for direct stacking. After coating deposition, the photoresist stripes are chemically lifted using a dimethyl sulfoxide (DMSO) bath.² The surfaces are cleaned using an SC-1 recipe,³ allowing for direct hydrophilic bonding.

While direct silicon-to-silicon bonding can occur at room temperature, the surfaces are initially held together by relatively weak van der Waals forces and hydrogen bonding. They can, however, form stronger covalent bonds through annealing at elevated temperatures, where the bonding strength can be gradually increased through thermal annealing until the bulk fracture strength of Si is reached above 1000 °C.⁴ Since stacking occurs after the coating deposition, it is crucial that any subsequent processing step such as annealing does not compromise the performance of the coating. There are methods to improve bonding at more moderate temperatures. For example the surfaces can be put under ultra-high-vacuum to ensure they are free of adsorbate layers and thus

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form a stronger bond. Alternatively, chemical surface activation by oxygen plasma exposure can help to achieve higher bonding strengths, just as annealing at moderate temperatures (200–500 °C) has been shown to increase the bonding strength between oxidized silicon wafers.⁵

Studies of thermal cycles simulating low-Earth orbit temperature fluctuations on W/B₄C multilayers found, however, that the enhanced temperatures have a slight negative influence on mirror reflectance at 1.5 keV, corresponding to an increase in interface roughness.⁶ In this paper the compatibility of the Athena candidate coatings (Ir, Ir/SiC, Ir/B₄C, and Ir/C) with a thermal annealing procedure aiming to improve SPO plate bonding strength is investigated.

2. EXPERIMENTAL PROCEDURE

For this study samples were prepared with reflective coatings with each of the Athena optics candidate material combinations. Sets of mirror samples were created where one was kept as reference and its partner sample underwent annealing. The pairs of samples were measured using X-ray reflectometry (XRR) at 1.487 keV and 8.048 keV prior to annealing and at regular time intervals after annealing to study the temporal stability of the coatings and identify any long-term effects caused by the annealing procedure.

2.1 Sample preparation

The substrates used were rectangular plates of 70 × 10 mm² cut out of 200 mm double side super polished Si wafers. The sample sets were mounted in close proximity to each other on the carriers to ensure maximal coating uniformity. They were plasma cleaned in-situ and coated at a pressure of 3.4×10^{-3} mbar using DC magnetron sputtering at the Silicon Pore Optics coating facility for the Athena mission.⁷

2.1.1 Sample annealing

Annealing was carried out at DTU Space using a Lab Companion OV-11 vacuum-compatible oven following specifications defined by cosine Research BV. The annealing procedure calls for annealing carried out in a constant atmosphere at ambient pressure. The samples are brought to a temperature of 200 °C over a two-hour period and kept there for 50 hours. The samples are allowed to slowly cool off for 8 hours before the oven is opened and the annealed sample can be characterized.

Following the results observed on some samples annealed under atmospheric conditions, low-vacuum annealing on additional samples was carried out to determine the effect of atmospheric annealing to that in a low-pressure environment. For vacuum annealing a Varian SH-100 dry oil-free pump with a pumping speed of 83 lpm and an ultimate pressure of 6.6×10^{-2} mbar was used.⁸ The reachable pressure is dependent on the vacuum chamber and the vacuum hose but a reasonable estimate is that the pressure reached was around 10^{-1} mbar. The vapor pressure of water at room temperature is on the order of 20 mbar. Table 1 gives an overview of the samples used for this study.

Table 1: List of samples and associated coatings used in this work.

Reflector Overcoat	10 nm Ir	30 nm Ir	30 nm Ir 12 nm B ₄ C	20 nm Ir 8 nm B ₄ C	30 nm Ir 4 nm SiC	10 nm Ir 8 nm C
Reference sample	cs00096	cs00108	cs00148	cs00144	cs00135	cs00190
Atmospheric annealing	cs00098	cs00110	cs00149	-	cs00134	cs00193
Vacuum annealing	-	-	-	cs00145	-	cs00192

2.2 Sample characterization

Characterization of the treated and reference samples was carried out using X-ray reflectometry (XRR) before annealing and immediately after annealing, as well as after 1 week, 1 month, 3 months, and 6 months. Although measurements were taken at regular intervals following annealing, the effect of annealing on mirror reflectance was immediate with no temporal evolution, so for clarity, only one measurement post-annealing at +6 months will be shown in the results section.

2.2.1 X-ray reflectometer facilities

Specular θ - 2θ measurements were carried out using the Low-Energy X-ray Reflectometer (LEXR) operating at 1.487 keV⁹ and a Rigaku XRD Smartlab operating at 8.048 keV. The low-energy measurements are crucial for identifying any changes to the low-density overcoat and at the same time represents a data point close to the 1 keV effective area requirement of Athena. A step size of 50 mdeg and a range in θ of 0–15 deg was used for the specular measurements and the system has a beam divergence < 12.5 mdeg. LEXR was also used to obtain measurements of the non-specular scattering. Scatter measurements were carried out around specular peaks at about 4 deg using a detector slit of 1 mm, corresponding to an angular acceptance of 0.14 deg. LEXR uses a 2D CCD detector with a pixel pitch of 26 μm . The error bars on LEXR data represent only statistical uncertainties and thus underestimate the total errors.

Measurements at 8.048 keV easily penetrate the overcoat and primarily yield information about the high-density reflector underneath. For these measurements a step size of 15 mdeg and a range in θ of 0–3 deg was used. The Smartlab uses a D/teX Ultra 1D detector and an angular resolution of 7 mdeg was assumed for measurements with this system.

In addition to the fixed-energy measurements carried out at 1.487 keV and 8.048 keV, samples were measured at incidence angles of 0.6 deg and 1 deg in the energy range 3.4–10 keV at the Four-Crystal-Monochromator (FCM) beamline of the PTB laboratory at the BESSY II synchrotron radiation facility. The energy resolution is < 1 eV and measurements were performed using the 4.5×4.5 mm² GaP diode detector.¹⁰

2.2.2 Fitting of XRR data

Data was analyzed using the IMD software¹¹ with optical constants from The Center for X-Ray Optics at Lawrence Berkeley National Laboratory.¹² The models used consist of an Si substrate with a 2.5 nm layer of native SiO₂¹³ where, to reduce model complexity, the roughness of the SiO₂ layer is coupled to that of the substrate-oxide interface. On the Ir single layers, a hydrocarbon overlayer of density 1 g/cm³ and surface roughness coupled to that of the overlayer-Ir interface is included in the model to account for the naturally occurring contamination layer. For all samples with a deposited low-Z overcoating, thickness z , density ρ , and roughness σ are fitted independently of the Ir undercoat.

3. RESULTS AND DISCUSSION

The results of the annealing study are presented in this section. First the temporal stability of the coatings is demonstrated by comparing XRR measurements of the reference samples taken shortly after coating and 6 months after annealing of their sample pairs. Secondly, the impact of annealing on both specular and non-specular XRR measurements is shown. The effect of an overlayer degradation on the specular reflectance at 1.487 keV is simulated, and finally, we investigate the reflectance of the samples in the energy range 3.4–10 keV.

3.1 Temporal stability of mirror coatings

The untreated reference samples were monitored over a period ranging from prior to annealing of the pairs to 6 months after the thermal treatment. Figure 1 shows XRR measurements at 1.487 keV and 8.048 keV for each of the six reference samples over the six month period of this study.

Figure 1: Specular reflectivity measurements of reference samples before annealing and 6 months after annealing of treatment samples plotted in reciprocal space. 8.048 keV measurements of the 20/8 nm Ir/B₄C samples are not available.

Note the presence of a double critical angle on the 1.487 keV measurements due to the low-density overlayers, particularly evident on the sample with a 12 nm B₄C overcoat. The best-fit parameters are listed in Table 2. Since no time-dependent evolution was observed on the reference samples, any change on the annealed pairs must be an effect of the annealing itself rather than temporal.

3.2 Impact of annealing on sample reflectance

Upon removal of sample cs00149 from the oven, it was immediately apparent that the B₄C was altered by the treatment. Figure 2 shows an optical image of the sample and its reference. As no change had been observed on the annealed Ir/SiC, visually or in XRR measurements, annealing under low-vacuum was performed only on another Ir/B₄C sample as well as on an Ir/C sample.

Figure 3 shows the effect of annealing on the X-ray reflectance of the samples. The best-fit parameters are listed in Table 2 along with those for the reference samples. Note that the overcoat parameters are difficult to constrain at 8.048 keV due to their limited reflectance at this energy and the strong signal from the Ir coating.

Both the 10 nm and 30 nm Ir coatings were unaffected by the annealing procedure. Iridium has, in fact, been found to be unaffected by the full stacking procedure, including chemical processing and annealing.¹⁴ The Ir/SiC bilayer was also found to be robust to the thermal treatment with a possible positive effect in terms of a reduction of the SiC roughness, which is in agreement with previous results obtained from coatings deposited at

Figure 2: Optical image showing absence of the distinct yellow tint of the B_4C on the annealed sample.

Figure 3: Specular reflectivity measurements of annealing samples before annealing and 6 months after annealing plotted in reciprocal space. AA denotes atmospheric annealing and VA denotes vacuum annealing. 8.048 keV measurements of the 20/8 nm Ir/ B_4C samples are not available.

DTU Space.¹⁵ The Ir/ B_4C bilayer annealed under atmospheric conditions underwent drastic changes, similar to previous observations.¹⁶ The XRR measurements post-annealing resembles those of an Ir single layer without an overcoat and the best-fit parameters indicate a B_4C layer with significantly reduced thickness and density. Drawing further conclusions on the characteristics of the annealed B_4C is not possible from XRR fitting alone, as we are modelling a layer that might be patchy or not present at all. After vacuum annealing of the Ir/ B_4C it was not possible by eye to see a difference in the coating as was the case for atmospheric annealing. Post-annealing XRR measurements show a larger fringe amplitude at large incidence angles than before annealing. It is not clear from the XRR fits what causes this difference but other characterization methods such as X-ray photoelectron spectroscopy could be used to gain further insight into the effect of both atmospheric and vacuum annealing B_4C . Atmospheric or vacuum annealing of the Ir/C had no effect on performance compared to the reference sample.

Table 2: Best-fit parameters to reference and annealed samples. The overcoat (OC) on Ir single layers at 1.487 keV are the naturally occurring hydrocarbons rather than an intentionally deposited coating. *Indicates a fixed parameter.

Overcoat (OC)	Sample	XRR Energy	Measurement time	OC z (nm)	OC ρ (g/cm ³)	OC σ (nm)	Ir z (nm)	Ir σ (nm)	Sub σ (nm)
None	cs00096	1.487 keV	Pre-annealing + 6 months	1.8	1.00*	Ir σ	9.9	0.29	0.33
				1.8	1.00*	Ir σ	9.9	0.26	0.29
	Reference	8.048 keV	Pre-annealing + 6 months	-	-	-	9.8	0.37	0.16
				-	-	-	9.9	0.37	0.16
	cs00098	1.487 keV	Pre-annealing + 6 months	1.6	1.00*	Ir σ	9.9	0.27	0.32
				1.5	1.00*	Ir σ	10.0	0.30	0.28
None	Atmospheric annealed	8.048 keV	Pre-annealing + 6 months	-	-	-	9.9	0.37	0.17
				-	-	-	9.9	0.37	0.21
	cs00108	1.487 keV	Pre-annealing + 6 months	1.8	1.00*	Ir σ	30.2	0.36	0.29
				2.1	1.00*	Ir σ	30.2	0.36	0.25
	Reference	8.048 keV	Pre-annealing + 6 months	-	-	-	30.1	0.41	0.17
				-	-	-	30.1	0.40	0.17
None	cs00110	1.487 keV	Pre-annealing + 6 months	1.7	1.00*	Ir σ	30.4	0.34	0.29
				1.8	1.00*	Ir σ	30.5	0.37	0.24
	Atmospheric annealed	8.048 keV	Pre-annealing + 6 months	-	-	-	30.4	0.41	0.16
				-	-	-	30.4	0.42	0.19
SiC	cs00135	1.487 keV	Pre-annealing + 6 months	4.2	3.18	0.88	29.9	0.24	0.34
				4.3	3.07	0.96	29.9	0.26	0.33
	Reference	8.048 keV	Pre-annealing + 6 months	4.2	3.19	0.86	29.9	0.29	0.30
				4.3	3.17	1.03	29.9	0.29	0.31
	cs00134	1.487 keV	Pre-annealing + 6 months	3.9	3.20	1.13	29.9	0.27	0.35
				4.0	2.84	0.71	30.0	0.31	0.34
SiC	Atmospheric annealed	8.048 keV	Pre-annealing + 6 months	4.1	3.21	1.05	30.0	0.31	0.34
				4.0	3.12	0.89	30.1	0.40	0.23
B ₄ C	cs00144	1.487 keV	Pre-annealing + 6 months	8.3	2.27	1.09	19.6	0.60	0.10
				8.7	2.87	1.42	19.6	0.38	0.41
	cs00145	1.487 keV	Pre-annealing + 6 months	8.2	2.25	0.93	19.7	0.37	0.41
				8.8	2.70	1.77	19.8	0.29	0.33
	cs00148	1.487 keV	Pre-annealing + 6 months	12.9	2.42	0.82	29.9	0.23	0.30
				12.9	2.43	1.03	29.8	0.23	0.29
	Reference	8.048 keV	Pre-annealing + 6 months	12.9	2.59	0.76	29.9	0.25	0.31
				13.1	2.68	0.74	29.9	0.25	0.31
	cs00149	1.487 keV	Pre-annealing + 6 months	12.6	2.56	1.04	29.9	0.25	0.32
				7.6	1.08	0.37	30.1	0.34	0.24
B ₄ C	Atmospheric annealed	8.048 keV	Pre-annealing + 6 months	12.5	2.68	0.99	29.9	0.27	0.32
				7.6	1.96	0.52	30.0	0.29	0.30
C	cs00190	1.487 keV	Pre-annealing + 6 months	8.3	2.28	0.82	9.9	0.23	0.34
				8.7	2.32	1.11	9.9	0.21	0.36
	Reference	8.048 keV	Pre-annealing + 6 months	8.3	2.50	0.79	9.9	0.25	0.33
				8.3	2.55	0.94	9.9	0.25	0.34
	cs00193	1.487 keV	Pre-annealing + 6 months	8.4	2.10	0.76	9.9	0.23	0.31
				9.6	2.36	1.83	9.9	0.19	0.33
	Atmospheric annealed	8.048 keV	Pre-annealing + 6 months	8.4	2.52	0.84	9.9	0.25	0.33
				10.6	2.11	0.59	10.0	0.25	0.32
	cs00192	1.487 keV	Pre-annealing + 6 months	8.6	2.24	0.90	10.0	0.25	0.32
				9.2	2.30	1.41	10.0	0.16	0.34
C	Vacuum annealed	8.048 keV	Pre-annealing + 6 months	8.5	2.50	0.86	10.0	0.25	0.32
				9.2	2.71	1.8	10.0	0.22	0.32

3.3 Impact of annealing on non-specular scattering

Measurements of the distribution of non-specular scattering can reveal information about interface morphology. In addition certain features, such as the Yoneda peak,¹⁷ give an indication of the angles of incidence and exit at which point the scattering volume is maximal. Figure 4 shows measurements of the non-specular scattering from the samples before annealing and at annealing + 6 months.

Figure 4: Non-specular scatter measurements at 1.487 keV. For the single layer Ir reference samples and the vacuum annealed Ir/B₄C no scatter measurements are available pre-annealing so the data has been overplotted directly. On the other samples a scaling has been used for clarity in comparing each individual sample before/after treatment.

No difference is observed in the amount of non-specular scattering from either the Ir single layers or the Ir/SiC after annealing. The small variations on the scattering from the Ir/C is most likely due to systematic variations in the measurement. The atmospherically annealed Ir/B₄C shows a clear change in the Yoneda peak after annealing, at which point the shape of the scatter function is similar to that of Ir without a low-Z overcoating, confirming the partial removal of B₄C as a scattering medium on this sample. Generally, the change in fitted roughness from the specular measurements is not reflected on the scatter measurements in terms of a difference in scattering intensity compared to before annealing. This indicates that the evolution of the roughness is more related to diffuseness of the interfaces (compositional grading normal to the surface) rather than genuine roughness in the conventional sense.

3.4 Simulated effect of overlayer degradation on specular reflectance at 1.487 keV

The effect of a removal of the overcoating layer is primarily evident at low energies and more so for the case of thick overcoats. Figure 5 shows a simulation of the effect of a reduction of the B₄C, SiC and C layers equivalent to the effect observed on atmospherically annealed B₄C (i.e a reduction in thickness, density, and roughness of 40%, 58%, and 64% respectively) at 1.487 keV.

Figure 5: 1.487 keV reflectometry data of vacuum annealed Ir/B₄C and atmospherically annealed Ir/SiC and Ir/C coated samples along with best fit models (from Table 2) and a simulation showing the effect of overlayer degradation on the sample reflectance.

The curves simulating an overlayer degradation of the same magnitude as that of the atmospherically annealed B₄C are distinctly different from the measured data and fits to the vacuum annealed B₄C and atmospherically annealed Ir/SiC and Ir/C samples. Particularly the entire Ir/C curve is different but also on the Ir/B₄C can the difference be seen on the double critical angle and around 5 deg. The effect is smallest on the Ir/SiC due to the small thickness of the layer but the region around 4 deg and 9 deg indicates that no such degradation on the SiC has taken place.

3.5 Characterization at intermediate energies

In addition to θ - 2θ measurements at 1.487 keV and 8.048 keV, the samples were measured at the FCM beamline at the PTB laboratory at BESSY II after annealing. Fits to the reference sample data at 0.6 deg yielded models that can reproduce the data of both reference and annealed samples at 0.6 deg and 1 deg, provided small offsets in the grazing incidence angle are accounted for, as shown in Figure 6. Only the atmospherically annealed Ir/B₄C requires two different models to reproduce the measured data from reference and annealed samples. We see a drastic change in the shape of the reflectance curve from the removal of the B₄C. This effect, particularly the bumps from the B₄C below 7 keV at 0.6 deg, can not be accounted for simply by an offset in incidence angle. All other samples with an overcoat have a characteristic shape at 5–6 keV at 0.6 deg and below 4 keV at 1 deg.

The reflectance data in the range 3.4–10 keV is thus in agreement with the results from single-energy characterization, that only B₄C annealed under ambient conditions suffers degradation.

Figure 6: Reflectance as function of energy for incidence angles of 0.6 and 1 deg. The simulated reflectance shown is from models with parameters listed in the table. In all cases except for atmospheric annealed Ir/B₄C, the same model fits both reference and annealed data sets with an offset in grazing incidence angle of up to 13 mdeg. *Indicates a fixed parameter.

4. SUMMARY

A study was carried out on the effect that annealing at 200 °C for 50 hours has on reflectance of witness samples coated with the Athena coating material candidates. We observed a drastic degradation of the B₄C when annealed under atmospheric conditions. No such degradation was observed on SiC, C, or B₄C annealed under low vacuum. Simulations of the effect on reflectance curves of a degradation similar to that observed on atmospheric annealed B₄C indicates that such an effect would have been identifiable on the 1.487 keV measurements. The nature of the degradation and any compositional or structural changes in the film from thermal treatment could be evaluated by other means such as atomic force microscopy or X-ray photoelectron spectroscopy.

Based on the results of this work, subsequent studies will be carried out with the relevant coating recipes deposited on SPO plates representative for substrates to be used for Athena.

ACKNOWLEDGMENTS

This activity has been funded by the European Space Agency under contract 4000102248/10/NL/PM.

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