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Performance and Stability of Mirror Coatings for the ATHENA Mission

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ABSTRACT

We present the expected coating performance based on design and simulations, tested coating performance evaluated by means of X-ray reflectometry and short and long term stability of several materials considered as coating options for the X-ray mirrors of the ATHENA mission. As part of this study we also report on the compatibility of the X-ray reflecting coatings to the industrial processes involved in the assembly of mirror modules using Silicon Pore Optics technology.

Keywords: ATHENA, SPO, coating design, X-ray mirrors, XRR, multilayer, X-ray optics, Ir/B₄C, Ir/SiC, Ir, effective area

1. INTRODUCTION

ATHENA (Advanced Telescope for High Energy Astrophysics)¹ is the second Large class mission selected by ESA with planned launch in the early 2030s. ATHENA will operate in the X-ray range between 0.1 keV and 10 keV to address the Hot and Energetic Universe scientific theme.

As the technology behind the X-ray optics for ATHENA evolves from its original concept,² so does the developments of the mirror coatings for the many Silicon Pore Optics (SPO) X-ray mirrors that will be on-board the observatory.³⁻⁹ The latest update on the geometry of the optics sets new requirements for the telescope on-axis effective area. Furthermore, instability of the B₄C coating can challenge the performance of the telescope at 1 keV.¹⁰

Single metal coatings were used in previous X-ray observatories to achieve a high reflectivity in the energy range 0.1 keV to 10 keV. The critical angle for total external reflection is maximized with the goal of achieving maximum overall throughput for the telescope.

The use of a light material, such as B₄C, on top of a metal coating, such as Ir, enhances the throughput at low energies and it is particularly relevant in the energy range between 0.1 keV and 5 keV keV.^{3,4} The light material coating can be optimized so it does not jeopardize the throughput at higher energies.¹⁰ In the case of a bilayer made of a heavy, high-Z material, with a top layer of a light, low-Z material, the reflectance of the low-Z material is higher than of the high-Z material for grazing incident angles smaller than the critical angle because of the smaller extinction coefficient. That also allows for higher energy photons to penetrate the low-Z material and be reflected by the high-Z material without significant loss. To improve throughput in the low energy range between 0.1 keV and 5.0 keV and at the same time enhance performance beyond 5 keV, the use a linear graded multilayer underneath an optimized heavy/light bilayer is a suitable alternative.¹⁰

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Ring	Width (mm)	Length (mm)	Mid Radius (m)
1	37.096	101.504	0.286
2	50.158	83.388	0.348
3	49.838	70.762	0.411
4	49.613	61.460	0.473
5	89.363	54.321	0.535
6	82.476	48.671	0.597
7	77.571	44.087	0.659
8	86.892	40.294	0.722
9	82.053	37.104	0.784
10	90.205	34.383	0.846
11	85.538	32.036	0.908
12	92.782	29.990	0.970
13	88.326	28.191	1.032
14	94.845	26.597	1.095
15	90.608	25.175	1.157

Table 1. Geometry of the X-ray mirrors for each SPO mirror module ring.¹²

Optimization of the coating design along with investigation of the performance and stability of the possible materials presenting performance close to that expected for B₄C can improve the throughput at lower energies and allow for the requirements on the effective area to be met.

The newest concept adopted for the ATHENA optics considered for the results presented here is of a fixed focal length of 12 m single telescope consisting of 15 rings of SPO mirror modules, each module is made of two sets of 68 reflecting X-ray mirrors. The entire optics containing 678 mirror modules. Because of the reduction in the geometry of the optics from 20 mirror module rings to 15 mirrors module rings, the new requirement for ATHENA's on-axis effective area is 1.4 m² at 1 keV and 0.25 m² at 6 keV.

In this study we present both expected coating performance based on theoretical models and experimental results on Ir, B₄C and SiC coatings.

2. COATING DESIGN AND EXPECTED PERFORMANCE

The effective area of the telescope depends on the collecting area of the optics and the capacity of mirrors in reflecting X-rays. The low energy photons will be successfully reflected throughout the optics while the higher energy photons are mostly reflected in the inner rings of mirror modules.¹⁰ By removing the outermost five mirror module rings, the effect will be larger at energies below 4 keV without affecting the telescope performance beyond 5 keV.

In this study we compute the on-axis effective area based on the updated geometry of the ATHENA optics and consider the performance of different materials. The computation makes use of the most recent coating optimization designs¹⁰ obtained by evaluating the coating performance over the integrated energy range of ATHENA. The on-axis effective areas computed are illustrated in figure 1. The configuration of the SPO mirror modules considered is listed in table 1. The coating models considered for computation of effective areas were generated using the IMD software.¹¹ The same coating recipe is considered for all mirror modules with an average surface roughness of 0.45 nm.

The computed on-axis effective at 1 keV and 6 keV based on the expected performance of the coating designs is listed in table 2. As expected, the reduction of the five outermost mirror module rings affects the telescope performance significantly at 1 keV. Based on the theoretical models considered here, the Ir/B₄C bilayer coating can still provide an effective area above the required at 1 keV. At 6 keV the performance is just below the requirement. As reported previously, the use of multilayer coatings can improve the performance at 6 keV.¹⁰

In terms of performance, the Ir/B₄C bilayer coating is closely followed by the Ir/SiC bilayer, motivating the consideration of SiC as replacement for B₄C. The recipe considered for Ir/SiC is 10 nm of Ir with a top layer of

Figure 1. On-axis effective area curves of the ATHENA telescope considering the previous 20 mirror module rings geometry compared to the 15 mirror module rings geometry and coating designs for different materials.

Coating	Geometry	Aeff 1 keV (m ²)	Aeff 6 keV (m ²)
Ir/B ₄ C bilayer	20 rings	2.3093	0.2469
Ir/B ₄ C bilayer	15 rings	1.5067	0.2468
Ir/SiC bilayer	15 rings	1.4344	0.2424
Ir/Si bilayer	15 rings	1.3612	0.2428
Ir single layer	15 rings	1.2271	0.2394
no coating	15 rings	1.2008	0.0003

Table 2. On-axis effective area (Aeff) at 1 keV and 6 keV of the ATHENA telescope considering the previous 20 mirror module rings geometry compared to the 15 mirror module rings geometry and coating designs for different materials.

SiC of 4 nm. Experimental considerations on stability of both Ir/B₄C and Ir/SiC bilayers are presented in this study.

3. STABILITY OF IRIIDIUM AND BORON CARBIDE COATINGS

3.1 Short and long term stability

The first coated SPO samples representative of the present ATHENA optics were produced back in 2011 at DTU Space. Since then, coating characterization by using X-ray reflectometry (XRR) has been performed by DTU Space both using the Cu K α 8 keV source at the X-ray facility at DTU Space and with synchrotron radiation at the four-crystal monochromator (FCM) beamline in the laboratory of PTB at BESSY II in Berlin.¹³ This beamline provides monochromatic radiation of high spectral purity in the photon energy range from 1.75 keV to 10 keV. It is equipped with a UHV-reflectometer providing all 6 degrees of freedom for sample positioning.¹⁴ The samples are loaded via a vacuum load-lock system. During the last years, the set-up has been further improved. Now, typically 8 witness samples or up to 4 SPO samples can be loaded. After finding the alignment settings for each sample, the alignment is verified using a CCD-camera system at about 1.5 m from the sample. One pixel on the CCD corresponds to a change of the grazing incidence angle of about 2 arcsec. All angle scans at fixed photon energy or energy scans at fixed angle are started simultaneously so that the real measurements can run automatically over night. A further improvement is an additional slit system directly in front of the samples and a thin photodiode after this beam defining slit which is used for normalization of the incoming beam. With this transmission photodiode in the beam, the photon energy range from 3 keV to 10 keV is accessible. The results of these measurements have been analyzed individually,^{5-7,10} for a few selected samples recurrent measurements were performed between 2012 and 2017 allowing for an evaluation of the stability of the Ir/B₄C coatings, until recently considered as baseline for the ATHENA mission.

In this study we present the measurements of two SPO samples, one coated with a Ir/B₄C bilayer with a Cr undercoat layer to balance stress and another with a Ir/B₄C linear graded multilayer. Both recipes considered are optimized coating designs considered to enhance the performance of the ATHENA X-ray mirrors.⁴

The sample SPO 05-03-07 is coated with a Cr/Ir/B₄C trilayer. Energy scans in the range between 3 keV and 10 keV of the sample have been performed at the laboratory of PTB at BESSY II in 2012, 2013, 2014 and 2017 at 0.6° grazing incidence angle.

The data measured at the different times are presented in figure 2. From the plot it is clear that the reflective performance of the coating has changed significantly between the measurements taken just after coating in 2012 and after one year in 2013. However, from the data obtained in 2014 and 2017, the coating appears to have stabilized since the initial change.

The theoretical models for each coating were fit to the XRR data using the IMD software.¹¹ Models fitted to energy scans from 2012 and 2013 are presented with the respective data sets in figure 3. The drastic change in the shape of the reflectivity curve over time can be attributed to oxidation and/or reduced density of the material in the top layer. Thus, instead of B₄C the model fitted to the data has a B₂O₃ top layer whose density has been used as a fit parameter. The use of B₂O₃ is motivated by a independent study of B₄C chemical stability performed at DTU Space.¹⁵ The densities of Ir and Cr were kept fixed at their nominal value.

The resulting best-fit model was able to give a reasonable fit to the data at the lower energies, although the fitted thickness of the top layer material is larger in comparison to the previous Cr/Ir/B₄C model based on 2012 data. Further studies on the oxidization of the B₄C are being carried out. The best-fit parameters obtained are listed in table 3.

Figure 2. Energy scan at 0.6° (left) measured at the laboratory of PTB at BESSY II and angle scan at 8 keV (right) measured at DTU Space of SPO 05-03-07 coated with a Cr/Ir/B₄C trilayer measured between 2012 and 2017.

Figure 3. Energy scan measurements at 0.6° of SPO 05-03-07 coated with a Cr/Ir/B₄C trilayer along with the best-fit theoretical IMD models.

Scan	Year	Model	z_{Cr} (nm)	z_{Ir} (nm)	z_{top} (nm)	ρ_{top} (g/cm ³)	$\langle\sigma\rangle$ (nm)
E (0.6°)	2012	Cr/Ir/B ₄ C	11.78	8.07	8.67	2.52	0.94
E (0.6°)	2013	Cr/Ir/B ₂ O ₃	11.67	8.9	12.32	2.02	0.71

Table 3. Best-fit results of energy scan measurements of SPO 05-03-07 Cr/Ir/B₄C trilayer.

The second sample considered is SPO 123-03-30, an Ir/B₄C linear graded multilayer coated SPO sample consisting of five linear graded bilayers with an optimized Ir/B₄C on top of the graded bilayer. XRR measurements of both energy scans at incident grazing angles of 0.6° in the energy range 3 keV to 10 keV performed at the FCM beamline of PTB at BESSY II and 8 keV angle scans were performed at DTU Space in 2014 shortly after coating and again in 2017. The measurements are presented in figure 4.

Comparing the energy scan curves, it is possible to observe a decrease in reflectivity at the lower energies. This effect is consistent to partial or total removal of the top B₄C layer. The change observed at higher energies indicates change in the multilayer structure. The change in performance observed at the energy scan is not observed at the angle scan at 8 keV. The best-fit parameters obtained using IMD models are listed in table 4.

Figure 4. Energy scan measurements at grazing angle of 0.6° measured in 2014 and 2017 at the FCM beamline of PTB at BESSY II (left) and 8 keV angle scans performed at DTU Space (right) of SPO 123-03-30 coated with Ir/B₄C linear graded multilayer coating along with their best-fit IMD models.

	June 2014, E (0.6°)			April 2017, E (0.6°)		
Layer	z (nm)	Γ	$\langle\sigma\rangle$ (nm)	z (nm)	Γ	$\langle\sigma\rangle$ (nm)
Top	9.9	-	0.45	0.01	-	0.45
Cap	11.4	-	1.0	10.7	-	1.3
d_5	11.0	0.32	0.61	11.5	0.32	0.63
d_4	9.6	0.30	0.59	9.7	0.25	0.39
d_3	8.2	0.29	0.57	7.5	0.24	0.77
d_2	6.8	0.26	0.63	6.5	0.32	0.63
d_1	5.4	0.22	0.45	5.7	0.29	0.45

Table 4. Best-fit results of XRR measurements of SPO 123-03-30 coated with an Ir/B₄C linear graded multilayer.

3.2 Imaging multilayers with TEM

In order to image the individual layers in the coated multilayer and evaluate their composition, we make use of Scanning Transmission Electron Microscopy (STEM) and Electron Energy Loss Spectroscopy (EELS) measurements.

The SPO sample 64-01-25 coated with an Ir/B₄C multilayer in 2014 was prepared for STEM via the focused ion beam (FIB) technique using the Helios dual-beam SEM and FIB instrument at DTU CEN. This technique is a reliable and rapid way, with full visual control of the etching procedure.^{16,17} In addition, most of the procedure is performed under high vacuum.

The sample preparation is a crucial step and affects the quality of STEM results. Herein, we explain briefly how the sample is prepared using FIB. Detailed information on the FIB method and procedures is presented elsewhere.^{18–20}

STEM and EELS measurement were performed with the FEI Titan instrument operated in high-angle annular dark field (HAADF) mode at an acceleration voltage of 120 kV with probe size of ~ 0.7 nm.

Figure 5. HAADF-STEM image (left) of the Ir/B₄C ML sample. The Ir and B₄C rich layers are light-gray and dark-gray bands in the image, respectively. EELS spectrum (right) revealing the K edges of boron and carbon.

Figure 5 shows a STEM image of the cross-section of the Ir/B₄C multilayer along with an example of the EELS results for B and C analysis. The image reveals 11 distinct layers between the substrate and the very top protective Pt layer, deposited under sample preparation. The contrast between layers in the STEM image arises from differences in the atomic number of heavy Ir and light elements B and C. The original coating design is made of five linear graded bilayers of Ir/B₄C followed by a bilayer of Ir/B₄C. The six bright layers observed are Ir rich, the 5 darker layers are B and C rich layers based on the preliminary EELS analysis, which is consistent with the expected B₄C layer. The very top layer of B₄C is not visible in the STEM image and we believe it is removed during the sample preparation. A major disadvantage of the FIB method is working with the heavy Ga ions which cause damages on the surface of materials,¹⁶ especially for samples with top layer composed of light elements. Although a protective pt layer was used, it is not impossible to consider the destructive nature of the Ga ion beam as a potential culprit.

Considering the instability of the B₄C layer observed and the technique involved in the sample preparation, it is not possible to evaluate the very top layer of B₄C with this study. With the exception of the very top layers, the Ir and B₄C rich layers are consistent with the desired coating design. A combined study comparing results from XRR, STEM, EELS and X-ray photoelectron spectroscopy (XPS) is ongoing at DTU Space and will soon be reported.

3.3 Thermal stability

The SPO mirror modules are stacked upon each other and bonded together with Van-der-Waals bonds, generating rigid and self-supporting mirror modules, without adhesives being needed.²¹ The Van-der-Waals bonds can be turned into even stronger covalent bonds by means of thermal annealing.²² Whether or not annealing is required, and what the exact recipe will be, is part of current investigations carried out by ESA and cosine BV. As part of this study, we evaluate the effects of annealing on the coating types under study for ATHENA.

To validate the compatibility of the mirror coatings to the annealing procedure, silicon witness samples (WS) were coated and submitted to the present annealing procedure performed by cosine BV to enhance bonding. The annealing procedure was performed at DTU Space mimicking the same conditions at cosine BV and consists of heating the substrates to a temperature of 200 °C for 50 hours, added to two hours of pre-heating.

Samples	Coating	Annealing	Ir (nm)	B ₄ C (nm)	B ₂ O ₃ (nm)
si6381	Ir/B ₄ C	-	9.2	2.4	8.2
si6375	Ir/B ₄ C	200 °C	9.2	2.6	4.2
si6518	Ir	-	9.2	-	-
si6523	Ir	200 °C	9.2	-	-

Table 5. The best-fit values for layer thickness obtained by fitting the theoretical models for each coating the XRR data using the IMD software.¹¹

The coating deposition was performed using the DC magnetron sputtering facility at DTU Space and characterization performed by means of XRR. The measurements were performed at the 8 keV X-ray facility at DTU Space and at the FCM beamline of PTB at BESSY II in Berlin.¹³

Figure 6. Energy scan measurements for reference and heated Ir/B₄C and Ir only samples

The preliminary results on Ir/B₄C bilayer coatings were previously presented,¹⁰ here we revisit the data and add the results obtained for a single layer of Ir after annealing. The XRR results for both Ir/B₄C and Ir alone are shown in figure 6. The theoretical models for each coating were fit to the XRR data using the IMD software.¹¹ The best-fit results for the layers thicknesses are reported in table 5.

Motivated by other studies on properties of B₄C ongoing at DTU Space,¹⁵ the B₂O₃ layer was added to the coating model to account for oxidization of the B₄C layer. The addition of the oxide layer appears to represent well the data.

The difference in performance of the Ir/B₄C coating after the annealing procedure is clear. The shape of the reflectivity curve changes dramatically after annealing. There is also a significant variation in the best-fit parameter for layer thickness for the B₂O₃, which seems to be vanishing. The best-fit roughness of the B₄C layer for sample si6375 is 1.6 nm, that is an increase of 1.3 nm compared to the obtained for the reference, unheated sample, si6381. The best fit roughness for the substrate and Ir interfaces remained as the unheated samples.

For the Ir single layer, the performance of the coating after annealing remains the same, we observed a small degradation of the reflectivity curve due to an increase in the substrate roughness but no change in the Ir layer thickness. The increase in roughness is caused by hydrocarbon contamination of the witness sample and not by changing of the properties of the Ir coating.

The study of thermal stability of coating will be extended to SPO substrates, representative of the ATHENA optics.

Sample ID	Type	Coating	Coating date
217-08	SPO	Ir single layer	Feb/2018
312-02	SPO	Ir/SiC bilayer	Feb/2018

Table 6. List of silicon pore optics (SPO) samples considered for Ir and Ir/SiC coating characterization.

4. IRIDIUM AND SILICON CARBIDE COATINGS

Considering the challenges the B_4C coatings have been presenting, in order to achieve the ambition for the telescope effective area at 1 keV, the Ir/SiC bilayer design has been proposed where the SiC top layer substitutes B_4C to improve the reflectivity in the low energy range. Ir single layer coatings are investigated as well and are considered as an essential reference coating. Assuring that a single simple layer of Ir is compatible with the SPO technology adopted by the ATHENA mission is crucial for further coating development.

The purpose of this study is to determine the stability over time of Ir and Ir/SiC coated samples stored at atmospheric conditions to provide the first results on the suitability of these coatings for the ATHENA mission.

4.1 Sample preparation and characterization

The samples considered for Ir and Ir/SiC are listed in table 6. Both the SPO samples and witness samples were coated at the DC magnetron sputtering facility at DTU Space according to the optimized coating design. The aimed coating thickness for Ir single-layer samples was $z_{Ir} = 10.0$ nm while the Ir/SiC bilayers were optimized to $z_{Ir} = 10.0$ nm with a top layer of $z_{SiC} = 4.0$ nm.

The measurements were performed at the 8 keV X-ray facility at DTU Space and at the FCM beamline of PTB at BESSY II. Measurements of the coatings at the laboratory of PTB were performed in March 2018 and May 2018 and include angle scans measured at a fixed energy of 8 keV, and energy scans between 3.4 keV and 10.0 keV at fixed grazing incidence angles of 0.6° and 1.5° . The data was taken on three different positions on each sample (center ± 1 cm). The energy ranges and the angles of incidence were selected to be representative of the ATHENA telescope.

4.2 Ir single-layer coatings

XRR measurements of Ir single-layer coatings are shown in figure 7 which compares BESSY measurements from March 2018 and May 2018 for three positions on SPO 217-08. We detect no significant evolution over time in the energy scan at grazing incident angle of 0.6° in figure 7a. The same result is observed for the angle scans at 8 keV, shown in figure 7.

Figure 7. XRR of Ir coated SPO 217-08. The 8 keV angle scan at DTU was measured just after coating, while BESSY measurements were performed 1 month and 3 months after coating.

4.3 Ir/SiC bilayer coatings

XRR measurements of Ir/SiC bilayer coatings are shown in figure 8. Figure 8 shows a comparison between BESSY energy scans for three separate positions on Ir/SiC coated samples measured in March and May 2018. The reflectivity in the energy range between 3.4 keV and 7.0 keV which is particularly sensitive to the thickness and composition of the top layer appears unchanged, and hence indicates stability of the SiC film within the two months between measurements. The angle scans presented in figure 8 shows no change from date of coating to measurements taken one month and three months after coating.

Figure 8. XRR of Ir/SiC coated SPO 312-02. The DTU Space 8 keV angle scan was measured immediately after coating, while BESSY measurements were performed after 1 and 3 months.

5. SUMMARY

We present calculations of the telescope effective area for different optics geometries and coating materials. According to the coating expected performance, the B₄C top layer provides the highest effective area at 1 keV followed by SiC.

The experimental results on coating development show instability of the B₄C top layer over time where oxidation appears to heavily affect the coating performance. The degradation of the top B₄C layer is observed on both simple tri-layer and multilayer coatings. Thermal stability studies presented here have also shown that the Ir/B₄C bilayer coating is not stable over the annealing at 200 °C. Ir single layer coatings have also been exposed to the annealing procedure. No change in coating performance of Ir was observed and we regard Ir single layer as robust over long time annealing. STEM and EELS techniques are being used to investigate the coating structure and composition and will allow for new insight on the coating stability.

Motivated by the expected performance of coating designs, we further investigate Ir/SiC bilayer and Ir single layer coatings experimental performance. The designed coatings were deposited on SPO substrates representative of the ATHENA optics and characterized with X-ray reflectometry. Both Ir single layer and Ir/SiC bilayer showed no degradation in performance over the period of measurements. The results presented here indicate that SiC is a suitable alternative to B₄C and seems to be stable over time. The optimized Ir/SiC bilayer coating design consists of 10 nm Ir with 4 nm SiC.

The coating development and characterization is an ongoing activity at DTU Space and part of the efforts for achieving the requirements for performance of the ATHENA optics prior to the Mission Adoption Review expected in 2021. The ongoing activities to be reported soon include understanding of coating composition and stability by a combined study comparing results from XRR, STEM, EELS and XPS, evaluation of thermal stability of Ir/SiC bilayer coatings, robustness of coatings to the industrial processes involving the production of SPO mirror modules including stability over photolithography and stacking procedures, and systematic characterization of dedicated samples for assessment of short and long term stability of the mirror coatings considered for the ATHENA mission.

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