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Characterisation of iridium and low-density bilayer coatings for the Athena optics

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ABSTRACT

The future Athena observatory will feature optics with unprecedented collecting area enabled by Silicon Pore Optics technology. In order to achieve the telescope effective area requirements at 1 keV and 7 keV, thin film coatings of iridium with a low-density overcoat are deposited onto the mirror substrates. Assembling the coated silicon pore optics plates into mirror modules for the Athena optics requires wet chemical processing and thermal annealing. While iridium appears to be compatible with the post-coating processes, previous studies have shown degradation of the low-density material. The overcoat layer is particularly critical for the low-energy telescope performance, so several candidate materials (boron carbide, silicon carbide and carbon) have been studied to identify a compatible thin film design. We present the characterisation of X-ray mirror performance using X-ray reflectometry, as well as the measurements of residual film stress with stylus profilometry. Furthermore, we evaluate the effects of post-coating treatment in order to recommend the most suitable overcoat material for the telescope.

Keywords: Athena, X-ray optics, Silicon Pore Optics, thin film, bilayer, iridium, carbon, boron carbide, silicon carbide

1. INTRODUCTION

The Athena (Advanced Telescope for High ENergy Astrophysics) mission is a future L-class X-ray observatory currently under study and preparation by the European Space Agency (ESA)\textsuperscript{1}. The telescope optics of Athena assumes a Wolter-I configuration with X-ray mirrors based on the Silicon Pore Optics (SPO) technology to provide a high collecting area-to-mass ratio\textsuperscript{2–4}. The optics performance in terms of effective area $A_{\text{eff}}$ is driven by the integrated optics area of the reflector surfaces, and the X-ray reflectance of the mirrors which can be enhanced with the use of optimised thin film coatings on top of the silicon substrate. The Athena X-ray mirror coating development is being carried out by DTU Space\textsuperscript{5–18}.

A bilayer thin film design consisting of an iridium (Ir) layer with a low-density material overlayer has been demonstrated to enhance the reflectance across the 0.1–12 keV spectral range of the Athena telescope\textsuperscript{11}. Importantly for the development of the Athena optics, the coated mirrors must also demonstrate compatibility with

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established wet chemical and annealing procedures required for the stacking of SPO plates. While compatibility with the entire stacking procedure was demonstrated for Ir-only single layer coatings, selecting a suitable low-density overcoat material that is robust to both wet chemical and thermal post-coating processing has proven more challenging.

From design considerations, the best performing bilayer design is achieved with a 10 nm layer of Ir and 8 nm boron carbide (B$_4$C), as the telescope effective area ($A_{\text{eff}}$) computed for different thin film designs in Figure 1 shows. Results from the last few years have however indicated evolution of the B$_4$C layer, in addition to apparent evaporation as a result of annealing.

As an alternative, a 4 nm silicon carbide (SiC) overcoat was suggested as a replacement for B$_4$C despite the comparatively lower performance at 1 keV. Ir/SiC bilayer coatings have appeared robust to annealing, but the thinner layer is susceptible to evolution and its surface may be affected by the wet chemical processing.

An 8 nm layer of carbon (C) was suggested, as a C overcoat layer performs similar to SiC near 1 keV but improves the 2–4 keV $A_{\text{eff}}$. Pre-studies of Ir/C have shown very promising results for the stability of C thin films and compatibility with the SPO stacking procedures.

In preparation for the upscaling in mirror production and stacking, the Athena-dedicated coating facility at cosine Research BV has been upgraded with components to the BS1500S coating machine to reduce loading and pump-down time (improved sample carriers, an additional turbopump, and a bake-out system), as have the post-coating wet chemical treatments been refined after the procurement of a new wetbench for wet chemical processing of SPO plates.

Selecting and finalising the coating design is critical for the validation of bilayer compatibility with the required post-coating processes for the industrial scale production of SPO stacks and mirror modules. This work presents the current most representative thin films produced in the BS1500S coating machine in order to evaluate the compatibility of bilayers of Ir/B$_4$C, Ir/SiC, and Ir/C based on X-ray performance. Additionally, the bilayer thin film stress is measured as a indicator of the stability of the coatings.

Figure 1: Total $A_{\text{eff}}$ for Athena assuming a 15-row optics configuration.
2. SAMPLE PREPARATION

Each of the three bilayer coating design was deposited onto photoresist-patterned middle radius SPO plates with Sacrificial Lateral Extensions (SALEX) measuring 73.7 mm × 40 mm. Super-polished silicon witness samples measuring 70 × 10 × 0.75 mm³ were mounted on a separate carrier at the center position of the SPO plates.

2.1 Thin film deposition

The deposition of iridium thin films with each of the three low-density candidate materials was carried out by direct current (DC) magnetron sputtering in separate coating runs in the BS1500S coating machine. Prior to coating, all samples were plasma cleaned with an Ar/O₂ mix using the in-situ inverse sputter etcher. The coatings were produced using an argon pressure of 3.4 ×10⁻³ mbar, and with discharge power set points of 1860 W for the iridium target, and 3200 W for each of the low-density materials.

2.2 Post-coating treatment

The two-step wet chemistry treatment of the SPO plates, consisting of photoresist lift-off followed by an organic surface cleaning using diluted Standard Clean 1 (SC-1) was performed in the Tetreon wetbench at cosine Research BV.

SPO plates were annealed in air using a Labcompanion OV-11 oven at DTU Space, following the procedure established for heating of SPO stacks at 200°C for 50 hours, including 2 hours of pre-heating.

3. EXPERIMENTAL

The coated SPO plates were measured with X-ray reflectometry at a fixed grazing incidence angle of 0.6 degrees in a range of 4–10 keV at the Four-Crystal Monochromator (FCM) beamline in the Physikalisch-Technische Bundesanstalt (PTB) laboratory at the BESSY II synchrotron radiation facility. Non-specular scatter at 4 keV was measured around the specular peak at 0.6 degrees.

The thin film residual stress of bilayer coatings without exposure to any post-coating treatment was derived by measuring the silicon witness samples using a Dektak 150 stylus profiler at DTU Space before and after coating.

4. RESULTS

Specular and non-specular X-ray measurements were performed for each of the three bilayer candidate designs to evaluate the impact of post-coating wet chemistry treatment and annealing. Measurements of the central spot on each SPO plate are presented.

4.1 X-ray measurements of Ir/B₄C bilayers

Energy scans at 0.6 degrees of the Ir/B₄C coated SPO plates are presented in Figure 2. While previous measurements of the specular reflectance have shown only a minor change in the B₄C layer after wet chemical lift-off and cleaning, these measurements suggest the presence of a surface absorbing layer on the untreated Ir/B₄C sample that appears to be removed with wet chemical lift-off and cleaning. Such an effect on B₄C thin films has not been observed before but it could be an effect of particulate contamination, although its composition is not known.
After annealing in air, oxidation and partial removal of the B$_4$C layer is observed. This is in agreement with previous results which also demonstrated that the B$_4$C film can in fact be preserved by annealing under vacuum\textsuperscript{16}. A simple Ir/B$_4$C bilayer model was not sufficient to fit the data, and instead an Ir/B$_4$C/BCO model was used, with the compound overlayer denoted BCO added to represent both a surface oxide layer and contamination layer. The model assumes a silicon dioxide (SiO$_2$) substrate to represent the thick oxide wedge on top of the silicon. The results are presented in Table 1.

The non-specular scatter measurements at 4 keV shown in Figure 3 indicate minimal change in surface roughness after wet chemistry but suggest that the annealing process changes the surface structure of the bilayer thin film.

Figure 2: Energy scans at a fixed 0.6 degree grazing incidence angle of Ir/B$_4$C bilayer coated SPO plates.

Figure 3: Non-specular scatter at 4 keV measured around a 0.6 degrees grazing incidence angle of Ir/B$_4$C bilayer coated SPO plates.
Table 1: Fit parameters to energy scan data at a fixed 0.6 degree grazing incidence angle of Ir/B₄C bilayer coated samples. Values in bold indicate fixed parameters. The asterisks mark coupled fit parameters.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Post-coating treatment</th>
<th>BCO</th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>z (nm)</td>
<td>σ (nm)</td>
<td>ρ (g/cm³)</td>
<td>z (nm)</td>
<td>σ (nm)</td>
<td>ρ (g/cm³)</td>
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<td>z (nm)</td>
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<td>01632-05</td>
<td>None</td>
<td>2.61</td>
<td>0.99*</td>
<td>1.15</td>
<td>6.26</td>
<td>0.99*</td>
<td>2.20</td>
<td>8.82</td>
<td>0.99*</td>
<td>22.42</td>
<td>0.50</td>
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<tr>
<td>01632-04</td>
<td>Lift-off, cleaning</td>
<td>0.12</td>
<td>0.87*</td>
<td>1.25</td>
<td>6.26</td>
<td>0.87*</td>
<td>2.20</td>
<td>9.17</td>
<td>0.87*</td>
<td>22.42</td>
<td>0.65</td>
</tr>
<tr>
<td>01632-04</td>
<td>Lift-off, cleaning, annealing</td>
<td>4.00</td>
<td>0.72*</td>
<td>1.25</td>
<td>0.18</td>
<td>0.72*</td>
<td>2.20</td>
<td>0.17</td>
<td>0.72*</td>
<td>22.42</td>
<td>0.59</td>
</tr>
</tbody>
</table>

4.2 X-ray measurements of Ir/SiC bilayers

The Ir/SiC bilayer samples measured without any treatment and after wet chemical and thermal post-coating treatment are presented in Figures 4-5. Fit parameters to a simple Ir/SiC bilayer model derived from the energy scan data are presented in Table 2.

![Figure 4](https://www.spiedigitallibrary.org/conference-proceedings-of-spie)  
**Figure 4:** Energy scans at a fixed 0.6 degree grazing incidence angle of Ir/SiC bilayer coated SPO plates.

![Figure 5](https://www.spiedigitallibrary.org/conference-proceedings-of-spie)  
**Figure 5:** Non-specular scatter at 4 keV measured around a 0.6 degrees grazing incidence angle of Ir/SiC bilayer coated SPO plates.
Table 2: Fit parameters to energy scan data at a fixed 0.6 degree grazing incidence angle of Ir/SiC bilayer coated samples. Values in bold indicate fixed parameters. The asterisks mark coupled fit parameters.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Post-coating treatment</th>
<th>SiC</th>
<th>Ir</th>
<th>SiO₂</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>z (nm)</td>
<td>σ (nm)</td>
<td>ρ (g/cm³)</td>
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<tr>
<td>01633-05</td>
<td>None</td>
<td>3.70</td>
<td>0.65*</td>
<td>2.69</td>
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<td>0.58*</td>
<td>2.21</td>
</tr>
<tr>
<td>01607-01</td>
<td>Lift-off, cleaning, annealing</td>
<td>2.53</td>
<td>0.60*</td>
<td>2.26</td>
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</tbody>
</table>

Based on energy scans, the Ir/SiC bilayers overall appear to be robust to wet chemistry and annealing. The fit parameters suggest that the Ir layer is slightly thinner after annealing but this is assumed to be an effect of a small angular shift. Furthermore, the SiC density appears slightly lower after lift-off and cleaning. The apparent decrease in SiC thickness after annealing may be attributed to evaporation of a surface oxide. The non-specular measurements at 4 keV further indicate that the SiC surface roughness is increased after lift-off and cleaning, also suggested by previous experiments\(^{15}\), while annealing appears to also change the characteristic length scales and thereby the surface structure of the SiC layer.

4.3 X-ray measurements of Ir/C bilayers

Figures 6-7 show the energy scans and non-specular scatter for the Ir/C bilayer coated SPO samples, with fit parameters listed in Table 3.

Ir/C bilayers appear to be compatible with both wet chemistry and annealing, as suggested by the derived fit parameters. The non-specular measurements further support that the C surface structure and roughness is unaffected by the SPO post-coating treatments.

![Energy scans at a fixed 0.6 degree grazing incidence angle of Ir/C bilayer coated SPO plates.](Image)

Figure 6: Energy scans at a fixed 0.6 degree grazing incidence angle of Ir/C bilayer coated SPO plates.
Figure 7: Non-specular scatter at 4 keV measured around a 0.6 degrees grazing incidence angle of Ir/C bilayer coated SPO plates.

Table 3: Fit parameters to energy scan data at a fixed 0.6 degree grazing incidence angle of Ir/C bilayer coated samples. Values in bold indicate fixed parameters. The asterisks mark coupled fit parameters.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Post-coating treatment</th>
<th>C</th>
<th></th>
<th>Ir</th>
<th></th>
<th>SiO₂</th>
<th></th>
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</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>z (nm)</td>
<td>σ (nm)</td>
<td>ρ (g/cm³)</td>
<td>z (nm)</td>
<td>σ (nm)</td>
<td>ρ (g/cm³)</td>
</tr>
<tr>
<td>01607-06</td>
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<td>7.31</td>
<td>0.64*</td>
<td>2.09</td>
<td>9.27</td>
<td>0.64*</td>
<td>22.42</td>
</tr>
<tr>
<td>01607-07</td>
<td>Lift-off, cleaning</td>
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<td>2.07</td>
<td>9.35</td>
<td>0.74*</td>
<td>22.42</td>
</tr>
<tr>
<td>01607-07</td>
<td>Lift-off, cleaning, annealing</td>
<td>7.59</td>
<td>0.59*</td>
<td>1.95</td>
<td>9.19</td>
<td>0.59*</td>
<td>22.42</td>
</tr>
</tbody>
</table>

4.4 Thin film stress

The bilayer film stress was derived for each of the three coating designs by measuring the curvature along each silicon strip before and after coating. Each sample was measured three times, and the averaged height was used as input to Stoney’s equation\textsuperscript{23} to fit the data, as shown in Figure 8. The total bilayer thickness \( z_{\text{total}} \) was assumed from the fit parameters derived from the energy scan data for each of the untreated SPO samples.

Table 4: Residual film stress derived for each of the three bilayer designs.

<table>
<thead>
<tr>
<th>Coating design</th>
<th>Bilayer thickness ( z_{\text{total}} ) (nm)</th>
<th>Residual stress (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ir/B₄C</td>
<td>17.69</td>
<td>-2556</td>
</tr>
<tr>
<td>Ir/SiC</td>
<td>13.31</td>
<td>-2346</td>
</tr>
<tr>
<td>Ir/C</td>
<td>16.58</td>
<td>-2004</td>
</tr>
</tbody>
</table>

The \( z_{\text{total}} \) and derived thin film stress are presented in Table 4. The measurements demonstrate compressive stress for all three bilayer designs, with Ir/C having the lowest residual stress of -2004 MPa, and Ir/B₄C having the highest of 2556 MPa.
5. DISCUSSION

X-ray measurements of the three bilayer coating designs demonstrate variations in specular and non-specular performance for the investigated bilayer thin films of Ir/B$_4$C and Ir/SiC after exposure to the current most representative post-coating procedures. Based on the results presented, the optimal coating for Athena is Ir/C, as this bilayer design appears robust to both wet chemical and thermal exposure.

The data from Ir/B$_4$C samples shows that the impact of wet chemical lift-off and cleaning depends on the initial state of both coating and substrate. These results therefore motivate a further systematic study of process parameters of the thin film deposition in addition to the variables of the post-coating treatments, such as chemical concentration, duration, and temperature, in order to define the limits of coating compatibility, and thereby conclude on the mirror coating stability.

The fit parameters overall indicate coating thicknesses that are slightly lower than the design values, as the coating process parameters need to be optimised after the chamber upgrades to compensate for the lower deposition rates. This motivates a re-calibration of the process parameters, in addition to the process parameter study.

Based on the measured thin film stress, the Ir/C bilayer presents the lowest residual stress out of the three coatings investigated. Further studies should consider long-term stability and the effects of wet chemistry and annealing on the residual stress.

A study on trilayer coatings using a chromium underlayer to balance the stress and thereby improve long-term stability is suggested to be carried out with a focus on the effect on non-specular scatter.

6. SUMMARY

We have performed a study of the three bilayer coating designs, including Ir/B$_4$C, Ir/SiC and Ir/C, that have been suggested as baseline coatings for the X-ray reflective mirrors of the Athena telescope. The optics are based on the SPO technology which requires wet chemical photoresist lift-off and SC-1 cleaning for surface activation, as well as annealing in order to facilitate stacking of the coated silicon plates which will be assembled into mirror modules.

Based on X-ray measurements of bilayer coated SPO plates after each process step, the results indicate that both Ir/B$_4$C and Ir/SiC may be affected by the wet chemical and thermal post-coating procedures. Ir/C bilayers appear robust to the current representative SPO stacking procedure while also providing a good mirror
performance, and exhibiting the lowest compressive stress out of the three bilayer designs. Future follow-up studies of the selected coating materials should aim at defining the range of process parameters that establishes compatibility between the thin film and post-coating processing steps in order to minimise potential risks to the production of the Athena mirrors.

7. ACKNOWLEDGEMENT

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REFERENCES


