

# Particulate Contamination Effects on Silicon Pore Optics for X-ray missions

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### Introduction



Athena (Advanced Telescope for High-Energy Astrophysics) is ESA's next generation X-ray observatory. Using a movable and tilting mirror assembly, the telescope will regularly change focus between its two instruments:

- the X-ray Integral Field Unit (X-IFU) which consists of a cryogenic X-ray spectrometer, based on a large array of Transition Edge Sensors (TES) and will provide spatially-resolved highresolution spectroscopy.
- the Wide Field Imager (WFI), a Silicon-based detector using DEPFET Active Pixel Sensor (APS) technology. WFI will provide a large field of view and both high count-rate and moderate spectroscopic resolution capability.

The ATHENA mission requires the largest X-ray optics ever built. In order to provide the effective area required to achieve the demanding scientific goals, the ATHENA telescope optics needs to cover a circular area of about 2.5 m diameter. This mirror assembly consists several hundred mirror modules, which have to be accurately coaligned on a stiff and stable optical bench, within a stringent mass budget.

The technology behind such mirror modules is Silicon Pore Optics (SPO).





### **Silicon Pore Optics**



Silicon Pore Optics (SPO) is a lightweight high performance X-ray optics technology, under development by ESA and Cosine Measurement Systems. Several hundreds of SPO mirror modules will be integrated and co-aligned onto the ATHENA (Advanced Telescope for High-ENergy Astrophysics) Mirror Assembly Module (MAM).

SPO uses commercially available Si wafers, which have surface figure and roughness quality ideally suited to X-ray optics application and consist of stacks of thin silicon mirrors, which together provide a large effective area wit a relatively low mass.



Massahi, S. (2019). Industrialization of the mirror plate coatings for the ATHENA mission. Technical University of Denmark.



Marcos Bavdaz, Max Collon, Marco Beijersbergen, Kotska Wallace and Eric Wille

X-Ray Optics and Instrumentation Volume 2010, Article ID 295095, 15 pages doi:10.1155/2010/295095

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## **Contamination Effects and Requirements**



Contamination effects may adversely affect the performance of high resolution X-ray grazing incidence optics of telescopes (X-ray scattering and absorption), leading to a direct decrease of the X-ray mirror effective area.

The ESA ATHENA study team has expressed the Mirror Assembly Module (MAM) budget in effective area loss, in which the losses due to contamination effects are included. The effective area of a telescope mirror is an important characteristic for its performance as it reflects the ability of the mirror to collect radiation at different photon energies.

To be able to define a cost-effective contamination control programme, one needs to address how much the performance of the optics are affected by contamination. The current study addressed specifically particulate contamination on the MAM sensitive units – the mirror modules (SPO technology).

The characterization of the sensitiveness to particle contamination, via experimental methods will also lead to the derivation of a meaningful and verified contamination requirement at EOL, for the ATHENA optics.

# **Test objectives and approach**



Models exist to assess the impact of particulate contamination of the effective area. However, these are mostly based on geometric effects. Moreover, there is lack of experimental data which provides a good understanding of the contamination effects at ATHENA representative energy ranges.

In the framework of ATHENA, a study on the effects of particulate contamination onto the ATHENA mirror surface was initiated, comprising the following steps/work tasks:



Experimental work covered in this presentation

#### **Test Methods: Task 1**



Automated particle detection, via optical microscopy, was employed on SPO samples of 80 mm x 40 mm (type A) and 110 mm x 40 mm (type B).

PAC levels and distribution determination was carried both at single plate level and at stack level, where 3 SPO plates were stacked together in order to simulate a simplified MM.

In total 6 stacks were exposed (2 stacks per different environment).



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SPO plates
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SPO plates stacked (simplified MM geometry)



## **Test Methods: Task 1**



The test campaign consisted, briefly, in exposing the SPO samples in a controlled environment, with respect to particulate fallout and measure the PAC levels **<u>directly</u>** on the SPO plates. Measurements allowed to determine the correlation between the fallout onto SPO geometries and the deposition rates of the environments, as well as the relation with respect to horizontally placed witness samples. The test setup consisted of:

- 6 SPO stacks (3 type A stacks and 3 type B each stack consists of 3 Si plates) test samples
- Silicon wafers monitor witnesses, not test samples (for environment deposition rates determination);
- **PFO plates-** monitor witnesses (for environment deposition rates determination);
- Optical Microscope (LEICA DM6000): to characterize PAC on both the test samples and the witnesses;
- **Mk5 Photometer:** to measure the obscuration factor of the PFO plates.







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# Particulate Distribution on SPO Geometries



An example of the test results of 1 out of 3 exposure tests (2 stacks) are shown in the table below and in the next slides.

| PAC levels (Obscuration Factors over time)                      |        |  |                   |                   |  |                   |                  |
|---|--------|--|-------------------|-------------------|--|-------------------|------------------|
|   | PAC    | Vertical Configuration (∆PAC (ppm)=OF <sub>IPi</sub> -OF <sub>BOT</sub> )<br>(Ratio in SPO Stacks / Witness Substrate) |                   |                   | Horizontal Configuration (∆PAC=OF <sub>IPi</sub> -OF <sub>IP4</sub> )<br>(Ratio in SPO Stacks / Witness Substrate) |                   |                  |
| Sample  | ВОТ    | IP 1<br>(7 days)   | IP 2<br>(18 days) | IP 3<br>(28 days) | IP4<br>(41 days)   | IP 5<br>(10 days) | IP6<br>(22 days) |
| Witness Substrates  |        | 560 ppm  | 1253 ppm          | 2463 ppm          | 3097 ppm   | 907 ppm           | 6102 ppm         |
|   |        |  |                   |                   |  |                   |                  |
| SPO Stack 2A<br>(ratio to horizontal deposition rate)           | 7 ppm  | 47 ppm<br>8.4%   | 106 ppm<br>8.4%   | 99 ppm<br>4 %     | 137 ppm<br>4.4%  | 13 ppm<br>1.4 %   | 44 ppm<br>0.7 %  |
| SPO Stack 2B<br>(ratio to horizontal deposition rate)           | 30 ppm | 0  | 14 ppm<br>1.1%    | 45 ppm<br>1.8%    | 57 ppm<br>1.8%   | 21 ppm<br>2.3 %   | 40 ppm<br>0.7%   |
|   |        |  |                   |                   |  |                   |                  |
| PFOs Vertical*<br>(PFOs Vertical/PFOs Horizontal)               |        | 37 ppm<br>8.3%   | 81 ppm<br>7.0%    | 131 ppm<br>5.8%   | 192 ppm<br>6.2%  | 66 ppm<br>5.5%    | 121 ppm<br>3.8%  |
| Si Wafer Vertical**<br>(Si Wafer Vertical/Si Wafers Horizontal) |        | 14 ppm<br>2.3%   | 26 ppm<br>2.1%    | 93 ppm<br>3.6%    | 176 ppm<br>5.6%  | 14 ppm<br>1.7%    | 107 ppm<br>1.9%  |

\* PFO plates Vertical/Horizontal ratios (in %) are a result of the exclusive comparison between PFO plates measurements – respective obscuration factors (OF) not presented in table.

\*\* Si Wafer plates Vertical/Horizontal ratios (in %) are a result of the exclusive comparison between Si Wafers measurements – respective obscuration factors (OF) not presented in table

#### **Particulate Distribution on SPO Geometries**





Correlation between PAC fallout deposition on horizontal exposed witness surfaces and vertically exposed surfaces (both SPO plates and Si witness wafer).

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#### **Particulate Distribution on SPO Geometries**





Ratios of the SPO geometries both exposed vertically and horizontally.



- A correlation between particulate fallout contamination, monitored with witness plates and the actual deposited particulate contamination, within the optical active surfaces of the ATHENA MMs (internal pores) was obtained.
- It should be noted that a significant number of factors will influence the deposition rates and the particulate distribution inside the SPO pores. However, it is believed that the current activity provides a good estimation of the reduction factor, which represent the particle fallout deposition on the optical surface of the pores as a function of witness/horizontal deposition;
- Based on the obtained results and assuming a single reduction factor, independent of the 2 types of MMs tested, it is proposed to consider a 5% to 10% (conservative approach) ratio between the internal pores of the MMs to horizontally exposed witness samples;

### Task 2: X-ray Characterization of PAC in SPO

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The goal of task 2 is to assess how the levels and distribution of particulate contamination on the SPO plates influence their respective X-ray performance (X-ray reflectivity - effective area).

The X-ray measurements were performed in the DTU Space Low-Energy X-Ray Reflectometer (LEXR) facility which is equipped with an Al source for measurements at 1.487 keV.



Side-view drawing of LEXR. Measurements given in mm. A: X-ray source, B: Kirkpatrick-Baez mirrors, C: Slits 1 and 2, D: Monochromator, E: Slit 3, F: Sample stage, G: Slit 4, H: Detector.

Henriksen, P. L.; Christensen, F. E.; Massahi, S.; Ferreira, D. D. M.; Svendsen, S.; Jafari, A.; Shortt, B. Published in: Proceedings of SPIE 11119, Optics for EUV, X-Ray, and Gamma-Ray Astronomy IX



Typical beam path in reflectometer. The beam enters the vacuum chamber from the source assembly (top right), passes through a series of slits and is monochromatized before reflecting off a sample and impinging upon the detector (top left). The detector is here illustrated in the maximum position  $2\theta = 35^{\circ}$ .

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#### **Test Plan**









- The test campaign was performed by the X-ray optics group at DTU Space in collaboration with ESA.
- The PAC levels on each sample were measured via optical microscopy, directly in each sample (in 3 zones of the sample), both before and after vacuum exposure. A total of 3 SPO plates were assessed;
- Each SPO sample was exposed to the environment for a different duration (to provide different PAC deposition levels per test sample).

#### **Visual Inspection**









Photographic records of both the monitoring samples and the test samples are shown.

The test samples visual inspection presented refers to after the vacuum exposure, showing that a considerable number of particles are still present after vacuum exposure (pump down and re-pressurization).

# **PAC Levels Test Results: BOT vs EOT**



After vacuum exposure, particulate contamination redistribution and release from the surface has occurred. Test results show that the loss of the PAC levels after the vacuum exposure is, consistently, comprised around **25% to 30%**.



Samples (zone bottom) in LEXR Facilty

### **X-ray Reflectivity of samples**





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## X-ray Reflectivity of samples: Trend Analysis





NOTE: PAC levels consider correspond to an average of the three zones per sample and considering both the values before and after vacuum exposure.

| Sample | PAC<br>Levels | Reflectivity<br>at 0.4° | Reflectivity<br>at 0.5° | Reflectivity<br>at 0.6° |
|--------|---------------|-------------------------|-------------------------|-------------------------|
| S0     | -             | 0.94                    | 0.93                    | 0.91                    |
|        |               |                         |                         |                         |
| S1     | 478 ppm       | 0.93                    | 0.92                    | 0.90                    |
| S2     | 731 ppm       | 0.91                    | 0.89                    | 0.88                    |
| S3     | 1181 ppm      | 0.87                    | 0.87                    | 0.85                    |

| Sample | PAC      | Loss of Reflectivity (wrt S0) |      |              |  |
|--------|----------|-------------------------------|------|--------------|--|
|        | Levels   | <b>0.4</b> °                  | 0.5° | <b>0.6</b> ° |  |
| S1     | 478 ppm  | 1.1%                          | 1.1% | 1.1%         |  |
| S2     | 731 ppm  | 3.2%                          | 4.3% | 3.3%         |  |
| S3     | 1181 ppm | 7.4%                          | 6.5% | 6.6%         |  |

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### X-ray Reflectivity of samples: Trend Analysis





NOTE: In both cases, values before and after vacuum exposure are considered.

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# **Summary and Conclusions**



- Establishing and understanding suitable cleanliness and contamination requirements is a challenging task. However, it is important to work on their derivation and/or justification as early as possible. While this carry some costs to execute the respective tasks, knowing the system limitation may end up in significant savings at a later stage of any space programme;
- The current work presented the approach established to assess the sensitivity of the ATHENA optics SPO technology, towards particulate contamination. Focus was given in the experimental determination of the X-ray reflection degradation of the optics, due to particulate contamination;
- The methodology employed was based in numerous direct measurements of particulate contamination deposition on the optical surfaces of interest. It allowed to better understand on particles deposit in the SPO geometries and along with X-ray measurements at grazing angles how it affects performance;
- A trend on the loss of X-ray reflectivity, at 1.54 keV, for angles below the critical angle of silicon was observed, with most contaminated samples giving rise to a slight loss in reflectance, as expected. Losses up to 7% were observed for PAC levels as high as around 1000 ppm;
- While a significant number of experimental uncertainties not may be neglected, it is believed that the carried work can improve optical performance simulations of the ATHENA telescope mirror. Moreover, the current work will support the cleanliness and contamination end of life requirements for the ATHENA optics.

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#### **Future Work**



- It is highlighted that the (chemical) nature of the particulate contamination was outside the scope of this study. Particulate contamination was assumed to be mostly organic and coming from a static environment (absence of common AIT activities) – in future testing this can be addressed;
- Kick-off further test campaigns in order to complete the assessment of the sensitiveness of the ATHENA optics to contamination (both scattering and effective area), e.g. the impact of different levels and nature of molecular contamination on the performance of the SPO technology;
- Consolidate the cleanliness requirements of the ATHENA optics for both MOC and PAC;
- Once all test campaigns are completed, the X-ray tracer model that simulates the optical performance of the ATHENA telescope mirror shall be feed with the test data obtained.

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