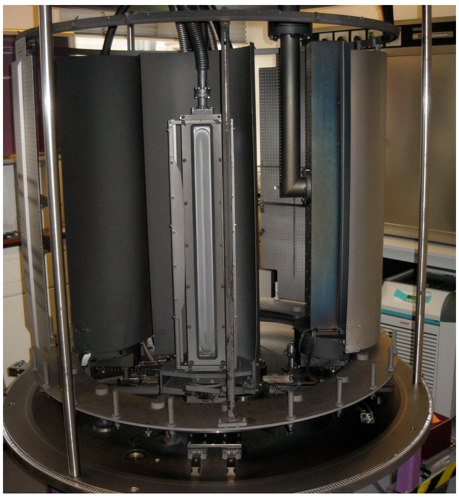
MULTILAYER COATINGS FOR ATHENA
TECHNICAL NOTES 4 - METHODOLOGY FOR COATING AND TESTING

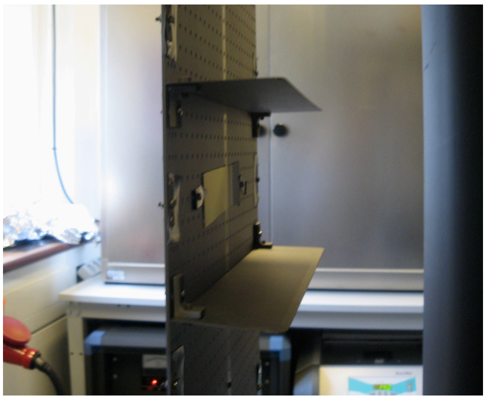
Anders Clemen Jakobsen[1], Desiree Della Monica Ferreira[1], Finn Christensen[1]
1. Technical University of Denmark, DTU Space
Juliane Maries Vej 30, Copenhagen Ø
2100 Denmark

1. INTRODUCTION

The coating facility at DTU Space is arranged with vertical sputtering cathodes pointing outwards in a circular vacuum chamber. Substrates pass in front of each cathode at a speed determined by the desired layer thickness since the sputtering rate for a cathode is approximately constant (within <1 %).
Substrates are mounted on vertical mounting plates that are placed on a rotating ring in the sputtering chamber.
After chamber is closed, a roughing pump and two turbo molecular pumps evacuates the chamber to a pressure of maximum $2*10^{-6}$ Torr before pure Ar gas is let into the chamber at a constant flow rate. The desired total pressure with Ar gas is $2.8*10^{-3}$ Torr, to ensure the fraction of other gasses to be less than 0.1%.
A voltage of ~ 400 V is applied to each cathode and the current set individually depending on sputtering material and desired coating rate. For Ir/B4C coatings, two target materials are used: 99.95 % pure cold-pressed Iridium and 99.99 % pure cold-pressed B4C.
A picture of the open sputter chamber can be seen below:



To decrease the amount of sputtered atoms hitting the substrate at an angle, which increases roughness, one of two methods are usually applied. The first one involves separator plates placed above and below the substrate on the mounting plate, that will collimate sputtered atoms in the vertical direction only. The distance between the plates can be changed to both give high collimation, but still avoid shadowing on the outer parts of a substrate which gives a non-uniform coating. A side-view of a mounting plate with separator plates can be seen in the picture below:

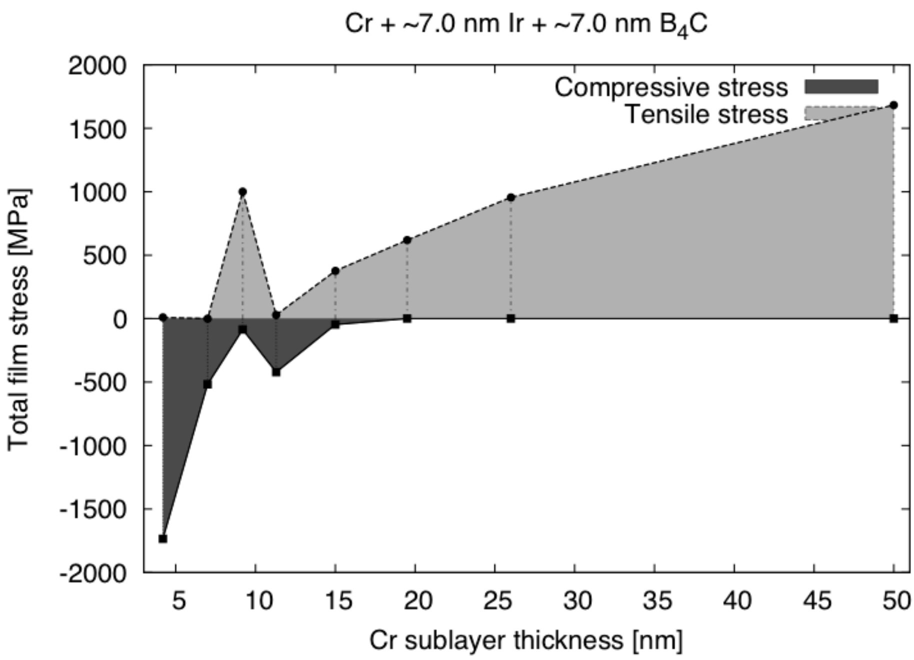


Another usable method of collimation is to place an aluminum honeycomb in front of the cathode. This will collimate in both horizontal and vertical directions, but will also decrease the coating rate by a factor of 3.
The coating rates are very different for low density and high density materials. To get approximately similar coating rates for e.g. Ir and B4C, the power applied to the Ir cathode has to be 150 W when the power applied to the B4C cathode is 1000 W. Coating with plain carbon is even slower, as e.g. two carbon cathodes running at 1100 W is required to keep up with a Pt cathode running 150 W.
The coating rate for Si is approximately half of the coating rate of W when running at the same power and a full NuSTAR multilayer of 1 μm total thickness takes about 6 hours with two Si cathodes and one W cathode running at ~900 W. A full multilayer of Pt/C of 0.8 μm thickness takes almost 12 hours with two C cathodes running at 1100 W and one Pt cathode running at 150 W.
Ir/B4C coatings using only one cathode of each can coat approximately 0.3-0.4 μm per hour and twice that with an extra cathode for B4C and double the power on the Ir cathode.
All these coating rates are with separator plates only.

2. Mirror plate bonding

To join two Silicon Pore Optics plates together, a direct Si-Si contact is required between the ribs of the upper plate and the wedged silicon oxide surface of the lower plate. Therefore a coating configuration is required where either the plate is masked during coating, or the coating is removable in the areas requiring Si-Si bonding after the coating using a resist layer.
The removal of a resist layer with acetone after coating will require the coating to have both a large surface binding efficiency and a low stress in the film, so as to come off cleanly. Especially for the regions of the film deposited at the edge between resist layer and regular surface, it is important to get smooth and vertical separation lines; otherwise some areas of

the coating close to the ribs in the final mirror configuration might become unusable, resulting in a lowering of total reflective area.
The thickness and stress of a coating can both be a factor when removing the resist layer, i.e. thinner, low stress films will possibly yield the best results and give the smooth vertical separation lines.
As a baseline, it was proposed to use an Ir/B4C bilayer for all mirrors of ATHENA, but lab tests at DTU Space have shown the films to have stress of around -4 GPa and removal of resist layer underneath these coatings at Cosine has shown extensive flaking. Preliminary tests with Cr/Ir/B4C trilayers have shown that the stress can be lowered to -400 MPa using a sublayer of Cr of around 20 nm thickness, with room for further improvements.
Further tests are required to show the optimum coating configuration of Cr/Ir/B4C trilayer coatings, including stress and roughness.
Stress measurements of preliminary Cr/Ir/B4C trilayer coatings can be seen in the plot below. It can be seen that low stress Ir/B4C films are possible using a Cr sublayer.
Further tests for Cr sublayers involve optimizing the Cr sublayer thickness after changing Ir/B4C bilayer dimensions.



The possibility of using other materials should also be explored, such as a B4C sublayer, which would reduce the complexity of a final coating system since fewer sputter cathodes would be required. To explore this possibility, samples will be made with B4C coatings of varying thicknesses. On top of these will be coated an Ir/B4C bilayer of fixed dimensions. The stress will be measured in those samples and compared to Ir/B4C bilayer films without B4C sublayer.
The feasibility of a resist layer with simple multilayer coatings should be further investigated. It is unknown whether simple multilayers can be removed cleanly from areas with resist using acetone without flaking and whether stress is a factor.
Stress measurements of ATHENA multilayer coating candidates will be carried out and resist removal test will be done on SPO substrates either at DTU Space or Cosine.
The possibility of reducing the stress in multilayers using sublayers or reactive gas should be further examined.

The alternate method for depositing striped coatings onto a substrate is to mask the substrate with a ribbed steel plate as has been tested in previous work[1]. The mask completely shadows the areas that should not be coated and therefore will make a clean Si surface for Si-Si bonding completely independent of the coating.

Experiments with a B4C sublayer was not done as it was later discovered that B4C itself will give a tensile stress on a coating. Adding a B4C sublayer would only worsen film stress. Additionally, resist removal was not carried out.

3. Substrate configuration

As the SPO substrates will be bonded together in a bent configuration, it has been proposed to coat the substrates while bent to reduce stress in the films in their final configuration. A few problems arise when coating bent substrates using magnetron sputter deposition:

- The distance from substrate to sputter target is highly influential in the deposition rate of atoms onto the substrate surface. Even a difference in distance of a few millimeters will result in a non-uniform film thickness. That could change the d-spacing in multilayers over area of a single SPO substrate and make it extremely difficult to model the ATHENA optic in ray tracing applications.

- Coating the substrate bent will require the substrate to remain bent from coating to resist removal and finally bonding with another substrate. Unbending and bending the substrate as would be done when unmounting and finally stacking the substrate might cause additional stress to the coating. That would require a massive amount of substrate holders with a specific curvature capable of going both into a sputtering chamber, through an acetone bath and still hold the substrate in the correct bended position while being bonded to another substrate.

It should also be noted that when the substrates are bent into the final configuration, the resist has been removed, leaving only strips of coating with a maximum width of 0.83 mm. So the total stress applied by bending the substrate is divided over the strips that each has room to expand to both sides due to the narrow bands between the strips without coating.

Finally, the preliminary trilayer Cr/Ir/B4C coating tests have shown that the stress might be 'tailored' by changing the thickness of the Cr sublayer, so if the bending exerts a compressive force on the film, the coating can be designed to be tensile to even out the total stress.
Further tests in this subject are required, but we propose to only follow this if problems with the coating in the flat configuration are discovered.

4. Coating roughness and stress

To reduce stress and roughness in the coatings, a possible solution could be the introduction of nitrogen gas during sputter deposition. Earlier results [2] have shown that small amounts of nitrogen gas can reduce the interface roughness of some materials.
Preliminary test coatings at DTU Space using N2 gas during deposition of a Ir/B4C bilayer

have shown a moderate reduction of stress (-4 GPa to –2 GPa), but no clear reduction of roughness. The parameters for improvement are limited in the case of reactive gas sputter deposition in DC magnetron sputtering chambers as only the N2 concentration can be changed and only between 0% and 30%. At higher concentrations, the N2 molecules will adsorb to the target surface and create insulating regions, so-called "target poisoning", which induces arcing that can cause the cathodes to shorten.
For simple multilayers, the roughness reducing abilities of N2 gas are possibly more evident, and we propose to further investigate that combination. A lower roughness in the simple multilayers might increase the final effective area of the ATHENA optic. ATHENA multilayer coating candidates will be produced using various concentrations of N2 gas. The samples will be measured for stress in a point stylus device at DTU Space and for interfacial roughness using 8 keV reflectometry.
The stress induced to a coated SPO substrate by bending it should be tested and in some way quantified to give an idea of the fragility of coatings in their final configuration.
We propose to use mechanical stress calculations to give an indication of the stress induced to a coating on bended SPO plates for ATHENA.

External stress induced on the coating due to bending of the substrate was not carried out, as the result would be arbitrary without an approximate stress value at which the coating breaks down.

5. MASKED DEPOSITION

Earlier work [1] has shown the feasibility of using a mask during deposition to develop striped coatings on SPO substrates. A ribbed mask is placed on top of a substrate using an infrared camera in a microscope and an infrared diode. Since infrared light shines through Si, it is possible to see the ribs of the SPO substrate from the mirror side using the infrared microscope. The mask is then moved until the ribs of the SPO substrate is aligned with the ribs of the mask. Four small springs are then clamped to the surface to secure the mask and the entire mounting jig is moved to the sputtering chamber for deposition. The four springs are totally ~ 10 mm2 and resulting in a loss of coated surface area of ~0.3 %. The longitudinal ribs of the mask are also held in place by two transversal ribs that results in an additional ~0.4 % loss of coated surface.
Also to consider is that the masks are made of stainless steel, and contact with the Si surface of the SPO substrate can results in scratches. A method to overcome this was proposed in [1] to be a coating of 30 nm Pt on the underside of the mask, as the softer metal would be less likely to scratch the surface.
For the full set of substrates for ATHENA, an automatic mask placing solution would have to be developed, as it is unfeasible to place it by hand in large numbers. A robotic auto-alignment setup could be a solution, although it might be too complex.
Depending on the setup of the final SPO coating facility, the SPO plates could be put in precisely machined substrate holders. For the ATHENA baseline, 12 different sizes of SPO substrates are needed, so only 12 different substrate holders would have to be manufactured. These substrate holders could include a precise mechanical mounting for a mask that could cover all the substrates in the substrate holder. The necessary machined precision of the substrate holders would probably have to be at maximum of 5-10 μm, so both the substrate and mask are hold tightly in the same place. Ideally the mask would just be clipped on or held in place by screws, but requiring a minimum of precision alignment by technicians.

It was agreed with ESA to focus on deposition on substrates with resist, as masked deposition would create too many challenges during production.

6. COATING VERIFICATION AND TEST METHODOLOGY

Coating verification of each of the optimized coatings must be performed at wavelength at DTU-space and the ESA BESSY facility. This should include both reflectometry and scatter measurements as well as AFM and cross-sectional TEM. When basic function is demonstrated and quantified the following test program should be conducted:
Adhesion test according to ISO9211-4(AD5). This test must be performed on multiple repeated coating runs for each coating type. It should be performed both before and after mask removal. We anticipate that eventually 10 test runs should be performed. It should also eventually be performed on a witness sample for every production coating run. The test should ensure that all possible positions/variations in coating chamber/process is represented. It should not be performed before a fixed set of cleaning/handling procedures of substrates have been agreed upon.

Adhesions tests for all possible sample positions in the chamber were not carried out. The results would not be representable for deposition setup of full optic coating.

After removal of resist, an additional adhesion test is required to determine the possible degradation of adhesion after contact with acetone.
Abrasion as per ISO9211-4(AD5). Again this test needs to be done for multiple runs and can be combined with the above tests. After successful testing it should not be necessary to do in production coating runs. This test should include before and after X-ray testing.

Humidity test. 48 h. at a relative humidity of 95 % according to ISO9022(AD6). This test can be performed at a relatively low number of coating runs. The test will include before and after X-ray testing.
Thermal cycling including qualification margin according to ECSS standard(AD4). 8 cycles are proposed. In contrast to what is specified in the RFQ we propose to do an X-ray test after 1st, 2nd, 4th and 8th cycle.

Humidity and thermal tests were carried out at an external tester, but it became clear that the company did not have experience with clean/delicate test objects. The tests were not done to a satisfactory degree. The tests were not redone as the new lab at DTU Space could not be completed in time.

In addition to the above tests we propose four additional tests:
A thermal destructive test. For each coating type a set of samples is heated in 50 degrees C increments and tested with X-rays after each cycle until breakdown of the structure and if possible the nature of the breakdown is analyzed.

As DTU Space lab was not ready, the thermal destructive tests were not done.

Long term stability test. Although the above can be likened to an accelerated long term stability test we propose that we, as soon as possible, dedicate a set of wedged plates for

each coating type as the long term stability test samples and X-ray test these at regular intervals (6 month to one year) until launch.
Radiation test using more than 100 MeV protons with a flux similar to what is expected for several years in space. This test needs to be supported by ESA.
For every optimized coating we populate the coating chamber with stress measuring samples covering every foreseeable geometry and map out the stress distribution for multiple coating runs. This does not require wedge plates but can be done on standard stress Si samples.

Radiation tests were not carried out at ESA.

During production coating we propose:
Two dedicated test substrate are used for adhesion tests, before and after acetone bath.
A stress sample included in every coating cycle and variations of the stress is continuously tracked.
An X-ray witness sample measured after each coating cycle and stored for later tests.

Alternatively, as fast automated X-ray system to measure all plates before and after coating could be included. It would give reflectivity data of every surface of the telescope to build into a final model of ATHENA performance.

References

[1] C.P. Jensen, M. Ackermann, F.E. Christensen, M. J. Collon, M. Krumrey, 'Coating of silicon pore optics', SPIE Vol. 7437 (2009)
[2] D.L. Windt, 'Reduction of stress and roughness by reactive sputtering in W/B4C X-ray multilayer films', SPIE 6688 (2007)