MULTILAYER COATINGS FOR ATHENA
TECHNICAL NOTES 5 - DESCRIPTION OF PRODUCED COATED SAMPLES

Anders Clemen Jakobsen[1], Desiree Della Monica Ferreira[1], Finn Erland Christensen[1]
1. Technical University of Denmark - DTU Space

jakobsen@space.dtu.dk

1 Introduction

This document includes description of the coating process specific to WP3000. It describes the sample qualification process, stress measurements, sample database and coating on bent substrates.

2 Deposition process

The coating process is described in detail in TN4 [1], but with some discrepancies for the coatings done for WP3000 which are described here. The optimum coating recipes for ATHENA described in TN2 [2] are linearly graded multilayers with 5-10 bilayers, so the coating rates for different materials has to be known precisely.

2.1 Coating calibration

For every material combination it is necessary to make calibration coatings to determine the coating rate. A calibration coating is a coating of 10 bilayers of constant d-spacing throughout the stack. This can be done with only one speed on the ring and 10 bilayers make them easier to measure by X-ray Reflectivity (XRR) and determine the exact d-spacing and Γ (ratio between materials in one bilayer). With two calibration coatings it is possible with linear regression to get a coating rate for each of the two materials, with the assumption that the coating rate is constant for any material throughout the coating.

For instance a W/Si multilayer, two calibration coatings were done where the speed of the ring described in stepper motor steps/second were 2000 for the first calibration coating and 3000 for the second calibration coating. For a full revolution of the ring, the stepper motor will have to move 668,000 steps, and for a 10 bilayer coating, 10 revolutions is needed. The thickness of the coating is inversely proportional to the speed of the ring, so the thickest coating should be the one where the speed is 2000 steps/s. The coated samples are measured using the in-house 8 keV XRR facility, and the data is compared to the theoretical model using IMD [3] to determine the d-spacing of the layers, Γ and roughness. See figure 1 for example.

The Γ is similar for each of the two coatings, since the same power settings were used for the cathodes. When plotting the d-spacing in relation to the inverse ring speed,

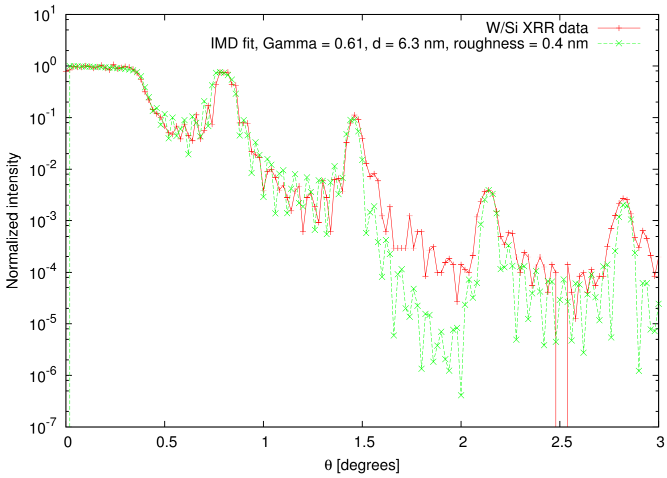

Figure 1: XRR data from a W/Si calibration sample compared to an IMD fit.

we get a data point for each calibration run. Fitting these points using linear regression gives a straight line through (0,0), and the slope of the line can give a ring speed for any desired thickness:

$$v_{speed} = \frac{m_{slope}}{d_{thickness}} \tag{1}$$

When coating the linearly graded multilayers on SPO plates, the ring speed (v_{speed}) for the coating of each layer was calculated using equation (1).

Tests using reactive sputtering with N_2 were also done to test both a possible changes in film stress and interface roughness. For all coatings produced using reactive sputtering, a constant flow rate of 75 sccm[1] Ar and 10 sccm N_2 were supplied to the coating chamber, yielding a total pressure of 2.8 mTorr. In all non-reactive sputter coatings, a flow rate of 87 sccm of Ar were supplied to the chamber, giving the same total pressure, 2.8 mTorr.

The flow rate of 10 sccm N_2 was chosen to give an approximately 10% nitrogen concentration in the chamber. The total pressure of 2.8 mTorr is the lowest Ar pressure possible in the DTU Space sputtering chamber while still avoiding drop-outs on the sputtering cathodes.

2.2 Coating on bent SPO substrates

As described in TN4 [1], it was proposed to coat the SPO plates while bent, so to reduce the stress of the coating in their final configuration. A mandrel was produced with a curvature corresponding to the innermost radius (0.158 m) of an ATHENA optic and long enough to hold the 130 mm SPO plates. For the current ATHENA configuration,

[1] sccm = standard cubic centimeter per minute

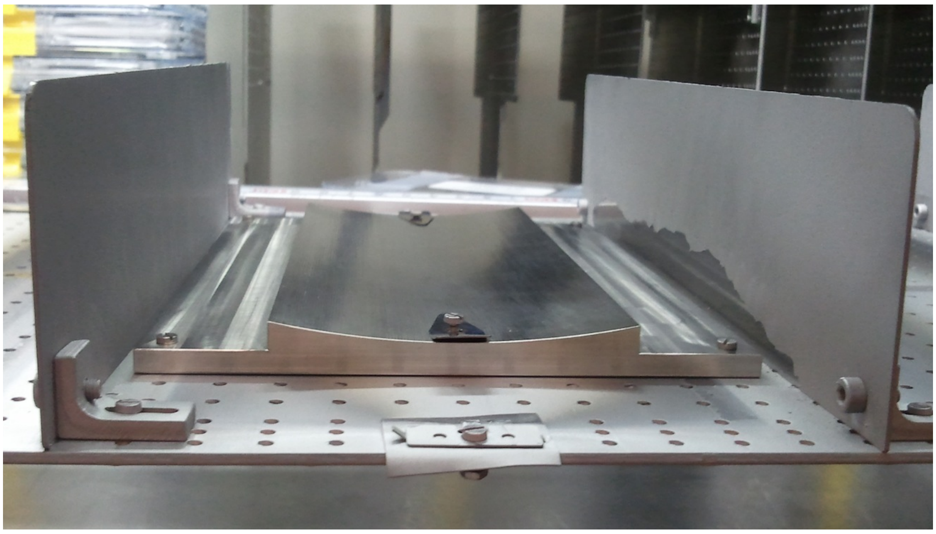

Figure 2: Curved mandrel to hold a 130 mm SPO substrate in a curvature of 0.158 m, corresponding to the innermost radius of ATHENA. A metal clip at either end holds the sample in place during coating.

the innermost radius of each optic will need 150 mm SPO plates. The plate is held down using two metallic clips as seen in picture 2.

Using this method proved unfavorable, since the clips at either end failed to hold down the plate to the mandrel. Instead the plate was bent to a saddle shape before breaking along the middle. Curving the long plates enough to make them fit into the mandrel required careful and uniform pressure on the middle part of the plate, which is not possible to do before a coating is to be applied to the surface.

It should be noted that SPO substrates has never been robotically mounted to a lower radius that 0.3 m, and only using short substrates.

3 Coated samples

For each material combination, three recipes were coated on SPO plates, except for the simple bi/tri-layer coatings which uses the same recipe the entire optic. For each recipe, one SPO plates with resist stripes were coated along with two raw Si wafer pieces and a stress sample. The SPO plate was coated for XRR measurements both at BESSY and the in-house facility. One of the raw Si wafer pieces were for in-house XRR measurements and another for adhesion tests.

One recipe for each material combination also had four extra SPO plates, one for humidity tests, one for temperature tests, one for irradiation tests and one for TEM and AFM measurements. The AFM measurements and results are described in TN6 [4].

3.1 Sample database

Every sample coated is identified with a serial number that is noted in a coating log as well as in a sample database. The database contains information about every sample and coating, so each sample can be looked up in a form and information about pressure, cathode power, position in chamber etc. is easily available. All data is cross correlated, so from looking up one sample it is possible to see which other samples were coated at the same time.

4 Qualification

4.1 Adhesion qualification tests

Adhesion of the coatings was tested according to ISO9211-4(AD5) standard. Three recipes for linearly graded multilayer material combinations and one recipe for single bilayer material combinations were tested using scotch tape. By firmly attaching the tape to the surface and quickly ripping the tape off, a consistent test of the coating adhesion was completed. All the material combinations proved able to withstand the removal of the tape.

4.2 Stress qualification tests

The stress of the coating on the substrate was tested for every material combination and every recipe. A Dektak 150 Stylus profiler was used to twice measure the profile along a sample before a coating. After the coating the sample was measured again to see any change in the deflection of the sample due to the coating. By factoring in the thickness of the coating, a calculation of the compressive and tensile stress of the film was obtained. The samples used were 5 x 80 mm Si wafer pieces. The calculations were done by the control program for the stylus profiler, and descriptions can be find in the pages attached to this technical note. The results of the stress can be seen in figure 3.

Each material combination shows a distinct trend in the amount of compressive stress coming from the coating. The W/B_4C multilayers shows the highest stress values for multilayers, between -1900 and -2800 MPa. Adding N_2 during coating to get reactive sputter deposition, decreases the stress by a small amount (see figure 3). The Pt/B_4C multilayers shows lower stress values, and the same effect can be seen from using N_2 during coating. W/Si multilayers show the lowest stress values of the multilayer material combinations, but using N_2 during coating had the opposite effect, and an increase in stress is seen.

The baseline Ir/B_4C shows the highest stress, around -4000 MPa of compressive stress and using N_2 can lower that value to around -1600 MPa. Using a Cr undercoat on the baseline Ir/B_4C will decrease the stress significantly [5, 6], although there is some variation. In figure 3 is shown the highest value of stress seen in a Cr/Ir/B_4C coating, about -1000 MPa, but values around $\pm$ 100 MPa are common.

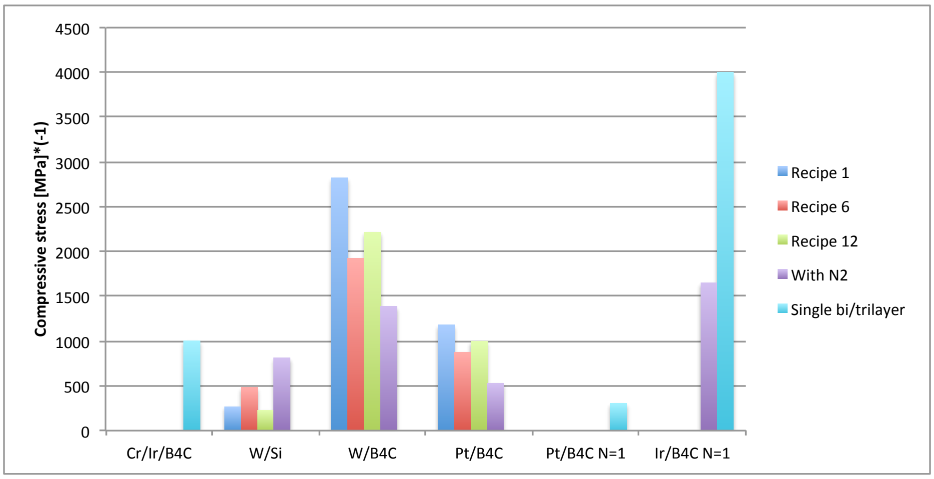

Figure 3: Comparison of measured stress in samples coated with different material combinations and coating recipes.

As mentioned in section 4.1, all the coatings showed good adhesion to the substrate, even in very high stress coatings. The main problems of stress in optical coatings are the tendency of low adhesion and the possibility of bending the substrate, which for X-ray optics would decrease the efficiency of the optic. Since the coatings show good adhesion and since a stack of SPO substrates have little or no tendency to bend, the stress of the coatings produced in this work package will most likely not be an issue for those two reasons.

The long term effects of high stress in multilayer coatings are however not known at this point, so we see it prudent to minimize the stress where possible for ATHENA. For the baseline Ir/B_4C coating, a relatively cheap method of reducing stress is a 100 Å Cr undercoat. W/B_4C and Pt/B_4C multilayer coating stress can be reduced using N_2. W/Si multilayer and Pt/B_4C single layer coatings already show low values of stress, so no measures are needed to lower the stress further.

The coated substrates has been stored and will be measured again at regular intervals to determine any changes in coating geometry, film stress and visual changes from e.g. peeling.

The method of measuring stress from deflection using a stylus profiler can only be used for stress caused by thin film coatings. For measuring the stress induced on a coating by bending a sample, one can use the Stoney equation:

$$\sigma_f = \frac{1}{6R}\frac{E_s d_s^2}{(1-\nu_s)d_f} \tag{2}$$

Where σ_f = stress in the film, R = radius of curvature, E_s = Young's modulus for the substrate material, d_s = substrate thickness, d_f = film thickness, ν_s = Poisson's ratio of substrate.

References

[1] D. D. M. Ferreira, A. C. Jakobsen, and F. E. Christensen. Technical Notes 4, v.2, IXO/DTUS/SYS/TN/0004. Technical report, September 2011.

[2] D. D. M. Ferreira, A. C. Jakobsen, and F. E. Christensen. Technical Notes 2, v.2, IXO/DTUS/SYS/TN/0002. Technical report, September 2011.

[3] David L Windt. IMD - Software for modeling the optical properties of multilayer films. *Computers in Physics*, 12(4):360–370, January 1998.

[4] D. D. M. Ferreira, A. C. Jakobsen, and F. E. Christensen. Technical Notes 6, v.1, IXO/DTUS/SYS/TN/0006. Technical report, May 2012.

[5] Mike Hill, Pete Blake, Ruth Carter, Kai Wing-Chan, and Kay Deere. International X-Ray Observatory (IXO) Segmented Glass Mirror Technology Development Status And Roadmap. September 2010.

[6] Anders C Jakobsen, Desiree Della Monica Ferreira, Finn E Christensen, Brian Shortt, Max Collon, and Marcelo D Ackermann. Preliminary coating design and coating developments for ATHENA. In *Optics for EUV, X-Ray, and Gamma-Ray Astronomy V*. SPIE, September 2011.