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The effect of deposition process parameters on thin film coatings for the Athena X-ray optics

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ABSTRACT

The thin film coating technology for the European Space Agency mission, Advanced Telescope for High-Energy Astrophysics (Athena) has been established. The X-ray optics of the Athena telescope is based on Silicon Pore Optics (SPO) technology which is enhanced by the thin film coatings deposited on the reflective surface of the SPO plates.

In this work, we present a literature study of the coating process parameter space and provide an overview of the thin film properties with a focus on micro roughness, chemical composition and wear resistance when deposited under various process conditions. We determined, that the thin film density depends strongly on the mobility of the adatoms on the substrate surface. Some coating process parameters, which have a significant impact on the adatom mobility are the discharge voltage, the working gas pressure and the substrate temperature.

1. INTRODUCTION

The largest space-borne X-ray focusing telescope, Athena (Advanced telescope for high-energy astrophysics), to be built, is selected for flight in the early 2030s¹. The novel Athena optics is currently in phase B with a technology readiness level of 5-6 and foreseen to move into phase C after mission adoption planned for end 2022². The mission enabling technology is based on silicon pore optics, which combined with a state-of-the-art thin film coating, enables the required throughput and performance to achieve the science goals³.

The thin film coating development for the Athena mission is progressing rapidly. Materials, such as, iridium, chromium, boron carbide, silicon carbide and carbon are comprehensively investigated⁴⁻⁶. The thin film coating development strategy, we developed for the Athena mission, emphasises a de-risking of the thin film chemical stability, thin film reproducibility and thin film compatibility with the SPO process technology, while achieving an excellent throughput performance. By investigating the coating process parameter space, we will identify the best coating conditions, in the BS1500S made by VON ARDENNE, for producing more than a 100,000 mirror

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plates with ideally identical thin film coatings.

The dedicated Athena coating chamber (BS1500S) is installed at cosine and was commissioned by DTU Space⁷. This led to a thin film coating development transfer from the coating chamber (DTU475) at DTU Space to the BS1500S, which now serves as the main coating chamber of the Athena thin film coatings. Both coating chambers produce excellent thin film coatings and their features, such as, chamber geometry and chamber components are compared in Figure 1. The coating chamber has recently been upgraded with an additional turbo pump, an additional magnetron and a bake-out system. This allows for a higher productivity and coating of tri-layers.

In this paper, We present the direct current (DC) magnetron sputtering deposition process and a literature study of the deposition process parameter impact on the thin film growth. Based on this, we provide an overview of the process parameters which can be investigated further in the thin film coating development for the Athena mission. Lastly, we discuss the foreseen experimental coating developments planned in the near future.

Figure 1: Comparison of the geometry, the capability and the features of DTU475 and BS1500S coating chambers.

2. THIN FILM GROWTH BASED ON DC MAGNETRON SPUTTERING

2.1 DC magnetron deposition process

Thin film growth, utilizing the DC magnetron sputtering technique, is dependent on several process parameters in the deposition process. We describe the deposition process by dividing it into five regimes (I–V), which is

illustrated in Figure 2.

(I) Initially, the coating chamber is evacuated to high vacuum which decreases the number of potential contaminants. In a "good vacuum" which is defined by Mattox⁸ as a pressure of 10^{-6} mbar, the number of molecules/cm³ is on the order of 10^{10} . A process gas, typically argon, is injected into the chamber creating an argon atmosphere also known as the working gas pressure. (II) The plasma environment consists of an electrical field, a static magnetic field, free electrons, argon atoms and ionized argon. The plasma environment impacts the sputter yield (deposition rate) and the kinetic energy of the sputtered atoms. (III) The target material and the plasma environment are strongly dependent on one another. Target materials have different electrical and chemical properties influencing the geometry of the electrical field. (IV) The sputtered atoms are travelling through region (II) towards the substrate, which can cause interactions between particles. Parameters mentioned in region (I) can impact the flight direction and kinetic energy of the sputtered atoms. (V) The sputtered atoms arrive at the substrate surface where they begin to form a thin film.

Figure 2: Illustration of the thin film deposition process in a DC magnetron sputtering system.

2.2 Process parameters impacting the film growth

The growth of the thin film is mainly driven by the kinetic energy of the adatoms (single atoms on a crystal surface), the substrate morphology and topography, and the sputtered material. A simulation by Müller⁹ indicates that adatoms with a low kinetic energy results in a porous film whereas adatoms impinging the substrate surface with a higher kinetic energy leads to a densification of the grown film. The kinetic energy of the adatoms is related to the kinetic energy of the argon ion energy and the average recoil energy of the sputtered atoms. This is expressed by Depla¹⁰ with the simplified equation given below:

$$E_{avg} = U_s \times \ln(\gamma \times E_{ion}/U_s)$$

, where U_s is the surface energy barrier, γ is the energy transfer factor and E_{ion} is the kinetic energy of the impinging working gas ion.

Another process parameter that impacts the adatom mobility is the substrate temperature. A simulation by Müller¹¹ suggests that the adatom migration rate is higher when the substrate temperature is higher. This is also illustrated by Thornton's¹² thin film Structure Zone Model. In which, a qualitative description of the thin film growth and morphology with respect to the working gas pressure and the ratio between the substrate temperature and the melting temperature of the sputtered material.

Applying a substrate bias to the substrate, is a technique to decrease the porosity of specific thin films. Hu,¹³ presents a study in which an increasing substrate bias results in an increase in density of a boron carbide film. By applying the substrate voltage it increases the kinetic energy of the adatoms.

Huang¹⁴ suggests that the compactness of the grown film is also dependent on the deposition rate. In Huang's simulations a higher deposition rate results in a higher island density due to the diffusion sufficiency of the adatoms. The deposition rate depends on the sputter yield which is material dependent. The sputter yield is given by the following equation:

$$S(E_{ion}) = \frac{3}{4\pi^2} \alpha \frac{\gamma \times E_{ion}}{U_s}$$

, where α is a dimensionless mass ratio function for an elastic collision. The sputter yield is linearly proportional with the kinetic energy of the impinging ions below 1 keV. Simulations by Depla¹⁰ show that low-Z materials in general have a lower sputter yield than high-Z materials when using an argon working gas.

3. PROCESS PARAMETER SPACE FOR THE ATHENA THIN FILM COATINGS

In this section, we discuss the deposition process parameter, which have been investigated and are still under investigation, in order to produce the thin film coatings for the Athena mission. The deposition process parameters and their impact on the thin film properties are shown in Figure 3 and elaborated upon in the following subsections.

3.1 Discharge power and discharge voltage

The discharge power density applied to the materials installed in the BS1500S is based on the research and development performed in the DTU475. We apply a discharge power density of 3.1 W/cm² on the iridium target and 5.17 W/cm² on the boron carbide target. Due to the geometry being different between the two systems, mainly, the target surface area being 3.2 times larger in the BS1500S, we observe a higher discharge power and discharge voltage. This is shown in Figure 4 and Figure 5 for iridium and boron carbide, respectively. The graphs indicate the required electrical settings and time to deposit ~10 nm single layer iridium and ~8 nm single layer boron carbide. We observe, that the deposition rate is about two-three times higher in the BS1500S for both materials and that no arcing is observed during the depositions. The small variation in discharge voltage is caused by the a slight working gas pressure variation, which is likely caused by the mechanical movement of the carousel.

The increase in discharge voltage is directly related to the kinetic energy of the sputtered atoms as described in subsection 2.2. This partly explains why the boron carbide films deposited in the BS1500S system is more resistance to oxygen incorporation than the boron carbide films deposited under the shown circumstances in the

DTU475 system¹⁵. An increase in kinetic energy of the sputtered atoms, allows the adatoms to populate more densely on the surface of the substrate.

The increase in discharge voltage lead to an increase in the sputter yield which along with the larger geometry of the target in the BS1500S system results in a higher deposition rate.

The limit of the applied discharge voltage is when it increases rapidly with time. If the discharge voltage is unstable and increases significantly it can result in arcing (short between the cathode and the anode) which eliminates the plasma.

Figure 3: Process parameters which can be modified/investigated for the Athena thin film coatings.

Figure 4: Comparison of electrical properties during deposition of a 10 nm Ir film.

Figure 5: Comparison of electrical properties during deposition of a 8 nm B₄C film.

3.2 Working gas pressure and target-to-substrate distance

The number of collisions between the sputtered atoms and the atoms in the working gas atmosphere impacts the kinetic energy of the adatoms. However, reducing the working gas pressure may impact the stability of the plasma as a certain density of the gas atoms are required to sustain a stable plasma. It is therefore favorable to have a low working gas pressure and a low target-to-substrate distance (TSD) while maintaining a stable plasma. The working gas pressure (argon gas) utilized in the BS1500S is based on the research and development performed in the DTU475 system. Currently, the operational working gas pressure in the BS1500S system is 3.4×10^{-3} mbar compared to 4.0×10^{-3} mbar in the DTU475 system. During the commissioning phase of the BS1500S, we investigated the impact on the coating uniformity across the vertical direction of the carrier. It turned out

that the shortest available TSD of a 105 mm resulted in the best coating uniformity. The TSD used in the deposition of the iridium and boron carbide coatings in the DTU475 system was 155 mm. This is 50 mm longer throwing distance which introduces more collisions which is likely another factor impacting the stability of the boron carbide films.

3.3 Base pressure, plasma cleaning and pre-sputtering

Contamination in thin film coatings will influence the film morphology, film composition, film roughness and film stress. We reduce the contamination in the Athena thin film coatings by evacuating the vacuum chamber to a pressure below 2×10^{-6} mbar to remove most of the water and gas residuals. When the chamber is evacuated, we plasma clean the substrate surface in an argon/oxygen atmosphere to remove organic contamination on the substrate surface¹⁶. Prior to the thin film deposition, a pre-sputtering is performed to remove organic contamination and potential oxidized material on the target surface. Pre-sputtering is also important for stabilizing the plasma conditions prior to deposition on the substrate. The pre-sputter time depend on the working gas pressure and is necessary to perform until the discharge voltage is stable within a few volts.

3.4 Reactive sputtering

A reactive sputtering study was performed in the DTU475 coating chamber and the results indicate that the film composition of boron carbide was modified. However, the reactively sputtered boron carbide films exhibited a chemical instability when stored in atmospheric conditions. The reactively sputtered film evaporated with time which was also the case for the non-reactively sputtered boron carbide and the study was therefore inconclusive. Literature suggest that the reactively sputtered boron carbide films exceed a lower film stress and a lower film roughness¹⁷.

In order to perform a reactive sputtering study in the BS1500S, the coating chamber would require some upgrades.

3.5 Substrate heating and substrate bias voltage

A dense growth of the thin film requires energy. We discussed the correlation between the kinetic energy of the adatoms and their mobility on the substrate surface. However, additional energy can be brought into the system by applying substrate heating or substrate bias voltage.

At DTU Space, we measured the temperature profile during deposition of a Ni/C multilayer with a deposition duration of 4 hours (Figure 6). The average temperature on the substrate surface during deposition was approximately 40° C due to the kinetic energy of the impacting atoms. The oscillations observed in the graph originates from the substrate being exposed to the nickel and carbon sputtered atoms, respectively.

Substrate heating and substrate bias voltage are process parameters which can be explored for the Athena thin film coatings, however the photoresist patterning on the substrate surface may limit the maximum substrate temperature to $\sim 70^\circ$. With the new bake-out feature, we may be able to heat the substrates during deposition to 40–60° C, however this requires further research. Another process parameter, we will investigate, is post coating plasma exposure.

Figure 6: Temperature profile during a deposition of a 50 bilayer Ni/C coating. The coating was performed in the DTU475 system. Credit: *Studies of Multilayer Coatings for Soft Gamma Ray Focusing*¹⁸.

3.6 Post coating plasma exposure

There are numerous techniques known using the plasma assistance during layer deposition to densify the coated material^{19,20}. Mainly, this technique is used for evaporated and ion sputtered layers. The magnetron sputtering technology shows a high intrinsic energy level enabling depositing dense thin films. Here, due to the usage of collimators, most of sputtered particles (approximately 70%) are filtered by the collimator and the energy on the substrate surface is significantly decreased. So, beside the voltage biasing and substrate heating it could be a good option to increase the adatom energy and increase the layer density. The advantage comparing to the heating is, that the energy is delivered directly in the growing layer and the thermal load to the substrate can be reduced.

The technology of plasma assisted deposition, usually, utilizes a simultaneously deposition and ion/plasma treatment. An upgrade allowing this approach is too challenging to be realized in the existing production machine BS1500S. So a different approach will be tested, a sequence of coating and ion treatment steps. Pre-investigations in laboratory scale showed promising results and we will implement the technology in the production scale. Following points are to be optimized during this work:

- Number of the steps in a sequence to achieve a homogeneous energy utilization in the depth of the layer
- Balancing of the deposition rate and the etch effect causing by the ion source
- Developing technology sequences, which allow for thin film production utilizing this technique without significant losses in the productivity

4. FUTURE EXPERIMENTAL WORK

Based on the literature study and the current coating development performed, we will explore the parameter space by depositing thin film coatings under numerous conditions. We plan to investigate the effect of varying the discharge voltage together with the working gas pressure to obtain the lowest working gas pressure along with the highest discharge voltage. We will determine the optimal pre-sputter time based on the different working gas pressures and the different materials.

We foresee, that a post coating plasma exposure will introduce an amount of energy into the thin film which may allow the atoms/molecules in the film composition to position themselves more densely.

5. SUMMARY

We presented a comparison of the research and development coating chamber, DTU475, with the industrial scale coating chamber, BS1500S. Two chambers, which are used for the development of the thin film coatings for Athena and which produce excellent thin film coatings. We discussed the utilized thin film deposition process and defined five different regions, wherein, each of the regions have numerous process parameters impacting the thin film growth. We determined several process parameters which has an impact on thin film growth with a main focus on film density and stability. The discharge power and voltage as well as the argon pressure will be investigated further in the BS1500S along with the post process plasma exposure.

We determined that the deposition process parameters are crucial to control and monitor closely during the flight production phase. Minor changes can modify the thin film uniformity, morphology, composition, roughness and stress.

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