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Abstract: Iridium-based coatings for mirrors of X-ray telescopes are studied. In particular, stress induced deformation is characterized and shown to be compressive and equal to -1786 MPa. Two methods for stress compensation are then studied. One relies on the deposition of silica on the back surface of the substrate and a second one relies on the deposition of a chromium sublayer. Advantages and drawbacks of each of these techniques are presented.

1. Introduction

New technological challenges recently appeared in the field of X-ray optics to simultaneously improve the performance and the cost constraints of the instrumentation used for future astronomical X-ray observatories. Costs diminution for launch can be realized by using lightweight instruments, e.g. ultra-thin X-ray mirrors. In the context of X-ray telescopes, large effective area and good angular resolution are required to augment the performance of the instrumentation. Different telescope designs can fulfill these requirements. They consist of several precisely shaped thin X-ray mirrors arranged in a well-defined geometry. For example, in the case of the Wolter I telescope design, grazing incident X-rays can be focused after being reflected on a primary paraboloid mirror and a secondary hyperboloid mirror (see fig. 1, left) [1]. To increase the collecting area several mirrors with cone shape are nested in each other. Different techniques are proposed to realize future X-ray telescopes with this design. One possibility consists of using thin glass as mirror substrate and to shape the glass into the required paraboloid and hyperboloid shapes by applying the slumping glass technique [2,3]. Another approach which is the baseline in the development of the future ATHENA telescope (Advanced Telescope for High ENergy Astrophysics) of the European Space Agency (ESA) is based on silicon pore optics, which is a pore structure geometry made of thin silicon mirror substrates [4]. Other telescope designs are based on a bionic approach as for example the optic of the Lobster eye. In Schmidt's arrangement grazing incident X-rays are reflected on two consecutive flat mirror sets disposed perpendicular to each other, whereas the mirrors in each set are arranged like in a fan and tilted radially toward a common axis (see fig. 1, right) [5,6].

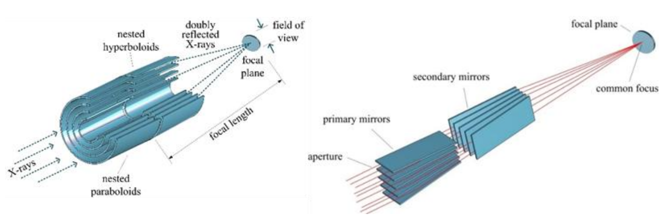

Fig. 1. Scheme of two different types of telescope design for astronomical X-ray observations [7]. Left – Wolter I telescope design, right – Lobster eye design in Schmidt's arrangement.

While using thin mirrors (mirror substrate thickness < 1 mm), coating stress can induce a deformation of the mirrors which would affect the angular resolution of the telescope. Mitigating the stress-induced deformation therefore requires very accurate determination and control of the stress coefficient. The challenge of the coating development for thin X-ray mirrors therefore consists in the combination of two relevant coating properties: high reflectivity and low coating stress. This paper focuses on the problematic of the fabrication of thin flat X-ray mirrors using iridium (Ir) as reflective coating.

The high Z-material Ir is expected to provide a high reflectivity for X-rays with photon energy < 10 keV. It is still used as reflective coating on the X-ray mirrors of the Chandra X-ray Observatory of the National Aeronautics and Space Administration (NASA) [8]. According to preliminary investigations on thin Ir coatings prepared by radio-frequency (rf) magnetron sputtering at a pressure of 5.10^{-3} mbar and under specific conditions the reflectivity of such coatings is expected to up to 86% of the reflectivity of bulk iridium for photon energies of 5 keV [9]. However, high coating stress was observed even provoking a delamination from the substrate. To get rid of this delamination problem a thin adhesive chromium (Cr) coating was applied between the substrate and the Ir coating [7]. However, the high coating stress of Ir still induces a disturbing deformation of the mirror substrate when using glass substrates with a thickness below 1 mm.

This paper presents the study of iridium and chromium coating stress as well as two approaches to mitigate mirror deformations by compensating the coating stress of the Cr/Ir-coating. Applying the same coating on the backside would induce unwanted reflections on the mirror's backside. The application of a non-reflective SiO_2-coating with controlled thickness and well-known counterbalancing coating stress on the backside of the mirror is considered. A second way consists in counterbalancing the stress of iridium coating by applying an under-layer with opposite stress. Both approaches are studied and analyzed.

2. EXPERIMENTAL RESULTS AND ANALYSIS

Investigations of the coating stress were performed using a similar approach than the one that was recently developed for oxyde layers [10]. Two-side polished thin fused silica substrates (diameter: 25 mm, thickness: 1.07 ± 0.02 mm) with a deviation from flatness of $\lambda/4$ at 633 nm were used for the experiments. Using such an approach stress coefficient could be determined with precision of ± 5 MPa.

Thin-film deposition of Cr/Ir-bilayer was performed in a sputtering machine VPA 21 from Aurion Anlagentechnik GmbH. Prior to the depositions, the substrates were treated with a N_2-plasma in the vacuum chamber at a pressure of $5\ 10^{-2}$ mbar. Then, the chamber was pumped down to $3\cdot10^{-5}$ mbar. The sputtering machine is equipped with two targets (diameter: 150 mm, Cr-purity: 99.95%, Ir-purity: 99.9%) inclined by an angle of 45° relative to the surface normal to the substrate. Distance between the center of the target and the center of the substrate's plate was set to 190 mm. To improve the uniformity of the film thickness on the substrate (which is typically 3% on a diameter of 150 mm), the substrate plate is rotating with 8 rpm during deposition. Substrates are neither actively heated nor cooled during deposition. The rf magnetron sputtering method (27.12 MHz) was applied for both depositions with 300 W on the target with a argon gas-flow of 50 sccm (Ar, purity: 99.999 %). Sputtering pressure was $5\cdot10^{-3}$mbar during both depositions and the coating thickness was regulated by the deposition time. Between the application of the Cr- and of the Ir-layer the samples were stored at air.

Thin-film of SiO_2-coatings were carried out using Plasma-Assisted Reactive Magnetron Sputtering (Bühler/Leybold Optics HELIOS machine) in MF-deposition regime. Sputtering was performed with high purity Si target and complete oxidation was obtained through oxygen plasma assistance. For the precise control of the thickness of the layers, optical monitoring was used using a Bühler OMS 5000 system. Precision of about 1-2 nm could be secured on the thickness of every deposited SiO_2 layers.

Evaluation of coating stress was based on surface flatness measurements of the substrates before and after coating and by using the Stoney equation [11]. The shape measurements were performed Zygo New View 7300 white-light-interferometer. Using a calibration based on SiC samples, absolute precision on the flatness of the samples of ± 5 nm could be achieved.

The thickness of each of the deposited Cr- and Ir-layers was measured with a tactile profilometer Dektak XT from Bruker (stylus radius: 12.5 µm). Finally, investigations on the surface micro-roughness of the Cr/Ir-coatings were performed by using an atomic force microscope (AFM) MFP-3D from Asylum Research in AC imaging mode.

2.1 Evaluation of the stress coefficient in Cr and Ir layers

Stress determination of Cr and Ir-layers relied on the deposition of layers with different thicknesses on fused silica substrates and measuring the flatness of the substrates before and after coatings [10]. Let us define the substrate thickness t_S, the radius of curvature of the substrate before deposition R_S, the radius of curvature of the substrate after deposition R_{S+f}, the layer thickness t_f and $\boldsymbol{E_S}$ and $\boldsymbol{v_s}$ the Young modulus and the Poisson coefficient of the substrate, in our case fused silica ($\boldsymbol{E_s}$ = 73 GPa and $\boldsymbol{v_s}$ = 0.16). By applying Stoney equation [11], it is then possible to extract the following equation [10]:

$$\frac{K_S}{R_{Norm}} = \sigma\left(t_f\right)t_f \tag{1}$$

Where

$$K_S = \frac{E_S}{6\left(1-\upsilon_S\right)} \tag{2}$$

is a constant term that depends on the mechanical properties of the substrate, and:

$$R_{Norm} = \frac{R_S R_{S+f}}{{t_S}^2\left(R_S - R_{S+f}\right)} \tag{3}$$

is the radius of curvature, normalized to the substrate thickness which is directly induced by the deposited layer. Let us consider that tensile stress is positive and compressive stress is negative. Equation (1) shows that by plotting the K_S/R_{Norm} dependence on film thickness it is possible to extract the stress coefficient for each of the studied materials. However, in our case, the structure is slightly more complex as not only Ir layers with different thicknesses were deposited but also a Cr layer which is required for adhesion. Therefore, both stress coefficients have to be determined. As chromium layer thickness was constant, we did not first consider the thickness dependence of stress of Cr layers. The samples that we prepared are listed in Table 1. For statistical purpose, we tried, when possible, to use several samples in order to avoid possible errors associated with a specific sample. The surface micro-roughness of these samples was identic to the surface micro-roughness of the substrate and was measured to be around 0.4-0.5 nm.

Table 1. List of prepared samples for Cr and Ir layers stress study

Sample ID	**t_s (mm)**	**t_f Cr (nm)**	**t_f Ir (nm)**	**# of coated samples**
1	1.07	6	0	1
2	1.07	6	19	3
3	1.07	6	30	3

4	1.07	6	49	3
5	1.07	6	97	2

In order to extract the stress coefficient of Ir layers, we plotted in Figure 2, the evolution of K_S/R_{Norm} versus the Ir thickness for every sample.

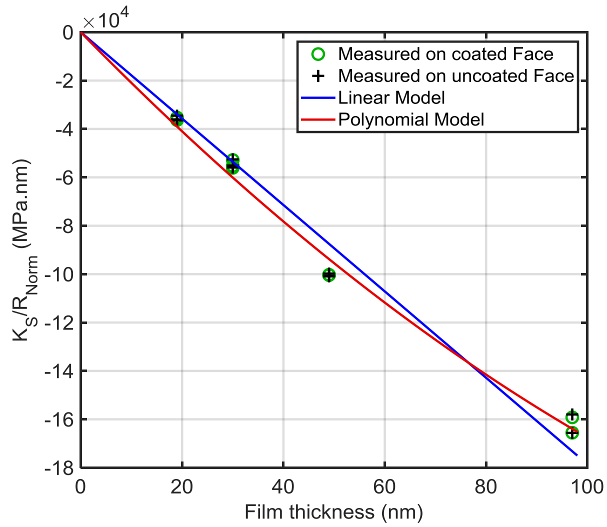

Fig. 2. Evolution of K_S/R_{Norm} versus Ir thickness

The plot in Figure 2 takes into account the stress induced deformation associated with both Cr and Ir layers. In this case, it is possible to show that equation (1) becomes:

$$\frac{\boldsymbol{K_S}}{\boldsymbol{R_{Norm}}} = \boldsymbol{\sigma_{Cr}(t_{Cr})t_{Cr} + \sigma_{Ir}(t_{Ir})t_{Ir}} \quad (4)$$

From measurement on sample #1, the stress coefficient associated with a 6 nm Cr layer can be calculated. In order to extract the parameter of the Ir layer only, we consider as the radius of curvature before deposition value, $\boldsymbol{R_S}$, the calculated radius of curvature of the Cr layer deposited on the substrate. $\boldsymbol{\sigma_{Ir}}$ is then obtained by the slope of the linear or polynomial fit as already implemented with oxyde-based layers, i.e. where:

- linear fit : $\sigma_{Ir} = K_{Ir}$
- 2nd order polynomial fit : $\sigma_{Ir} = A_{Ir} + B_{Ir}\, t_{Ir}$

Table 2 summarizes the obtained results for all materials as well as a figure of merit (MF) defining the average error of the fit.

Table 2. Stress parameters of Ir and Cr layers calculated from the experimental data of Fig. 2

Materials	**Linear Fit**		**2nd order polynomial fit**		
	K (MPa)	MF	A (MPa)	B (MPa.nm^{-1})	MF
Cr	1730	N/A	N/A	N/A	N/A
Ir	-1786	2525	-2143	4.67	1508

Stress in Cr layers is tensile and its coefficient is equal to ~1730 MPa. This value is very large and 5 to 10 times larger than the one that can be encountered with classical dielectric oxyde materials [10]. Regarding Ir, stress is about the same level (-1786 MPa) but compressive. The figure of merit of the 2nd order polynomial fit results in lower figure of merit than the linear fit showing that the stress has some small dependence with the Ir layer thickness. Due to the precision on the thickness of Cr and Ir layers, the precision on the stress coefficient is with ± 50 MPa (i.e. 3%).

2.2 Stress compensation for Ir-based mirrors

High stress level in Ir-based mirrors is highly problematic in the production of X-ray mirrors as they will produce large surface deformation. This problem is even more important as in case of Wolter I telescope design or lobster eye design in Schmidt's arrangement, where each mirror segment is about 150 mm long and less than 1 mm thick. As a consequence, the calculated stress-induced sag will be of several hundreds of microns. To overcome this problem, there are at least three different approaches that only involve coatings developments:

- The first one consists in adapting the deposition parameters to decrease stress level in Ir layers. According to Broadway [12], coating stress of Ir layers is dependent on the sputtering parameters, especially on the sputtering pressure. Lowest coating stress is obtained when sputtering at a pressure of about $3\cdot10^{-2}$ mbar. Based on these results, the influence of sputtering pressure on the X-ray reflectivity of the Ir-layers has recently been studied. However, the expected low-stress Ir layer from sputtering at $3\cdot10^{-2}$ mbar depicts a porous microstructure with a rough surface resulting in low expected X-ray reflectivity [13].
- The second approach consists in depositing coatings on the backside of the substrates in order to compensate the stress induced deformation.
- The third approach is to benefit from the result in Table 2 that Cr-layers stress is tensile while Ir-layers stress is compressive. By adapting the thickness of each type of materials it is possible to generate perfect stress-compensation.

Let us analyze the last two approaches. The first has already been implemented by several other research teams [14, 15]. In their work, back surface coating was implemented in order to both perform stress-compensation but also to generate a specific optical function such as an antireflection or broadband rejection. In our case, the mirrors are supposed to be coated on a single face and back surface reflection is not expected to be a major problem as glass is not transparent at these wavelengths. Indeed, it becomes possible to coat the back surface of each fused silica substrate and coating with silica is a method that will allow compensating stress-induced deformation. The typical stress-coefficient of fused silica as deposited by plasma-assisted reactive magnetron sputtering (Bühler HELIOS) has already been precisely characterized and is equal to [10]:

$$\boldsymbol{\sigma_{SiO_2} = -371.2 - 0.0360 \times t_{SiO_2} \quad MPa} \tag{5}$$

This stress is ~4.3 times smaller than the one of Ir layers. Therefore, the SiO_2 layers thickness required for stress compensation will be ~4.3 times larger. As the thickness of the Ir layers required for achieving high reflection coefficient is pretty small (typically less than 100 nm), the thickness of the silica layers will remain also pretty small (not larger than 500 nm). In addition, the use of a material with low stress coefficient will release some of the precision that is required on the thickness for perfect stress compensation. In order to illustrate the method and its efficiency, we deposited on the back surface of one of the previously fabricated substrates (Sample 5) a silica layer that allows perfect stress compensation. We first calculated the silica layer thickness that is required for stress compensation. As the two different samples 5 did not have perfect flatness and an original sag (before coating), related to surface polishing, we used the value of the sag after deposition on the sample to be coated in order to calculate the required thickness for perfect stress compensation. This resulted in two different thickness values of the silica layer for the same Ir layer thickness. For the considered sample, we coated the back surface with a silica layer of 231 nm thickness. In Table 3 we report various parameters that allow estimating the accuracy of the stress compensation method.

It is clearly visible that using this technique an almost perfect stress-compensation with a residual sag of 17 nm peak-to-valley could be achieved. While original flatness was very good (11 nm), this compensation could also be adapted to account for the original flatness of the glass substrate and partially correct it. One can wonder how such an approach can be adapted to the substrates that will be used in the telescopes, i.e. with 150 mm length and 0.4 mm thickness. Using the determined stress values and supposing that the thickness of the silica layers can be controlled with a precision of ± 1 nm (which is achievable with optical monitoring), the precision of the final flatness is ≈300 nm. Thus, this technique represents an accurate and efficient method for the production of flat Ir-mirrors for X-ray telescopes.

Table 3. Description of the parameters of the Ir mirror that were coated on the back surface for stress compensation.

Coating	**Parameters**	**Calculated values**		**Experimental values**	
		A-face	B-face	A-face	B-face
Uncoated	Radius (m)	N/A	N/A	4374	982
	Sag (nm)	N/A	N/A	11	51
1-side coated	Radius (m)	N/A	N/A	-115	101
	Sag (nm)	N/A	N/A	-434	497
2-side coated	Radius (m)	Infinity	431	5378	1056
	Sag (nm)	0	94	8	38

Let us now analyze the second technique that consists in balancing stress by adjusting the thickness of the Cr and Ir layers. The study that we have performed up to now is not enough to carry out this compensation. It is then mandatory to be able to know the dependence of the stress-induced deformation on the thickness of the Cr-layer. For this purpose, we deposited chromium layers with various thicknesses of 5, 42, 70 and 90 nm on fused silica substrate. Afterwards these samples were coated altogether with an additional Ir layer of 29 nm thickness This simultaneous coating allowed securing that all samples were coated with the same exact Ir layer (apart from uniformity of the coating in the chamber, i.e. less than 3% difference). Finally, the sag of the samples was measured and the evolution of K_S/R_{Norm} versus Cr thickness was plotted in Figure 3. The Cr layer with thickness of about 70 nm allows generating a zero-sag and therefore totally compensates the Ir-layer stress induced deformation. It should be noted that some nonlinear dependence of stress induced deformation on Cr-layer thickness exists. In addition, it can be shown that a further increase of the Cr-layer thickness results in stress relaxation and unpredictable behavior. Large Cr-layer thickness is required for compensation of stress-induced deformation and the required thickness would be even larger for high-reflectivity Ir-based mirrors. In this case the effect on the surface micro-roughness has to be considered as the iridium film growth is influenced by the surface of the under-layer and an increase in the surface micro-roughness of the iridium coating would induce a decrease of its X-ray reflectivity (Table 4). Therefore, such a stress-compensation process is not directly compatible with the production of high reflectivity X-ray mirrors.

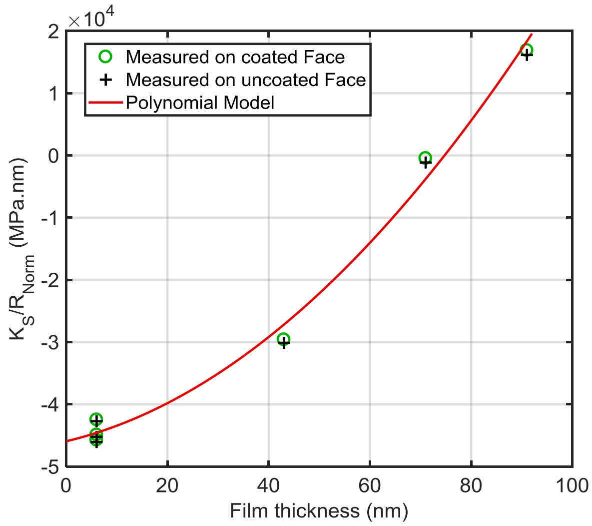

Fig. 3. Evolution of K_S/R_{Norm} versus Cr thickness

It is also worth noting that all the experiments presented in this work were performed by sequential coating of Cr and Ir layers, i.e. by breaking vacuum between each coating run. Such an approach was implemented in order to minimize errors due to coating repeatability. However, it would be important to quantify the effect of this approach especially if the third technique for stress compensation had to be implemented. Also, it exists an additional limitation of both methods: they do not consider thermal induced stress, i.e. the different coefficient of thermal expansions of the different layers and of the substrate would also induce a deformation of the mirror when temperature is changed. It is clear that to be operated in various conditions, such effects would have to be considered.

Table 4. Measured roughness of Cr+IR-coated glass substrates

Sample #	Cr-layer thickness, nm	Ir-layer thickness, nm	Roughness, nm
1	0	0	0.2
2	5	29	0.4
2	90	29	0.7

3. CONCLUSION

We have presented a complete study of stress and stress-compensation in Ir-based X-ray mirrors. Layer stress coefficient of both Cr and Ir layers have been quantified. For the Ir-layers a tensile stress coefficient of 1730 MPa and for the Cr-layers a compressive stress coefficient of -1786 MPa could be calculated. Two methods for stress compensation have been presented and demonstrated. One relies on back surface deposition while the second consists in balancing stress between Cr and Ir layers. Both methods have shown to be efficient from a stress point of view, but the second method induced parasitic effects which could decrease the Ir mirror performance.

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