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IRIDIUM COATINGS FOR SPACE BASED X-RAY OPTICS

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Future investigations of astronomical X-ray sources require light weight telescope systems with large collecting areas and good angular resolution. The Wolter I type telescope design offers a suitable possibility for obtaining performant X-ray mirrors with high collecting areas. The technology based on replicated slumped glass optics using thin glasses thereby provides the opportunity to fulfil the light weight and mass production requirements. In NASA's telescope NuSTAR this technology has been proven as advantageous compared to previous systems. Coating thin glasses with iridium, gold or platinum enhances the reflectivity of X-ray mirrors. Low coating stress, high density and a low surface micro-roughness are thereby necessary to provide a good X-ray reflectivity performance. Owing to the high reflectivity of iridium for photon energies up to 5 keV, the presented work is focusing on the process development of iridium coatings (film thickness ≤ 100 nm) on thin glass substrates. We consider thin glasses formed into the parabolic and hyperbolic shapes of the Wolter I design by thermally treating the glasses on concave moulds using the indirect glass slumping technique. With this technique the glass surface to be coated keeps its original roughness since it is not in contact with the mould during the thermal treatment. The developed deposition process of the iridium thin films on glass substrates is based on magnetron sputtering. Sputtering with different parameters (for example by variation of argon gas pressure) leads to thin iridium films with different properties. Investigations on the dependency of the density of the thin film, its crystal structure, the surface roughness and the coating stress on the sputtering parameters are considered in the context of the influence of the iridium film´s properties on the reflectivity of the coated mirrors.

I. INTRODUCTION

An optical mirror reflecting visible light under nearly normal incidence mainly absorbs or transmits X-rays, but is not reflecting them. By changing the angle of incidence to grazing incidence, X-ray reflectivity is increased. Grazing incidence mirrors provide sufficient reflectivity of X-rays at a grazing incidence angle typically below 3°. The Wolter I type design for X-ray telescopes combines two types of grazing incidence mirrors with different geometries (see Fig. 1): a primary paraboloidal mirror and a secondary hyperboloidal one [1, 2]. The advantage of a double mirror system is a reduction of optical aberrations such as coma effects. To augment the collecting area of the X-ray telescope several nested mirror shells are combined together [3].
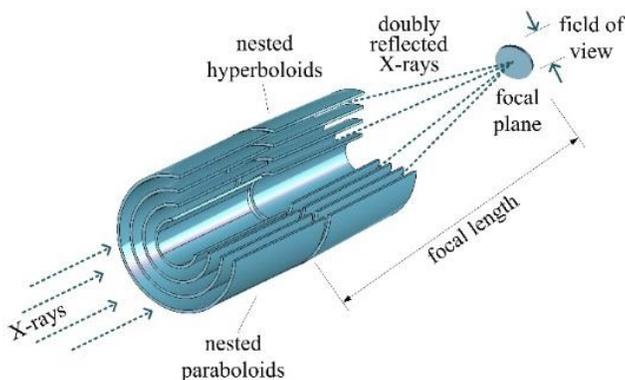

Fig. 1. Schematical representation of the principle of a segmented Wolter I type X-ray telescope [4].

The Wolter I type telescope design is widely applied in space missions as depicted in the overview list of table 1. Slightly variations from this design are also used: The conical approximation in which the paraboloids and hyperboloids are replaced by two conical mirrors and the Wolter-Schwarzschild configuration [5].
The best angular resolution of X-ray telescopes is so far obtained using the technology of directly polished mirror shells. An angular resolution of 0.5 arcsec was realized for the Chandra mission, launched in 1999, which consists of four thick walled polished Zerodur shells. This X-ray telescope however depicts a low effective area of only 0.08 m² at X-ray energies of 1-1.5 keV and a high mass to area ratio of 12500 kg/m². Improved effective area and reduced weight together with good angular resolution are aimed for X-ray telescopes of future space missions such as ATHENA (Advanced Telescope for High ENergy Astrophysics) for the European Space Agency (ESA)

[6] or X-ray-Surveyor for the National Aeronautics and Space Administration (NASA) [7, 8]. Enhancement of the effective area should be realized for ATHENA by increasing the shells number to 250. To ensure both, reasonable telescopes volume and weight, reducing the thickness of the mirrors is required. A thickness of 0.17 mm is proposed for the ATHENA mirror thickness (i.e. more than 100 times lower than that of the Chandra mission). The angular resolution of the telescope is driven both by the rms roughness of the mirror and by the aberrations of the mirror system.

Table 1. Overview of the main characteristics of realized and planned X-ray satellites.
*: substrate rms roughness: ≤ 0.5 nm, ++: balloon experiment, n.: information not available.

Mission	Launch	Energy range in keV	Ang. Res. HEW in arcsec	Eff. Area @ 1-1.5 keV in m²	Mass/Area in kg/m²	Mirror substrate* material	Mirror substrate* thickness in mm	Mirror coating material	Mirror coating thickness in nm	Mirror coating rms roughness in nm	Ref.
Wolter I configuration											
Directly polished shells mirror technology											
Einstein	1978	0.1 – 4	~ 10	0.04	-	fused quartz glass	~ 20	Ni	60	n.	[9] [10]
ROSAT	1990	0.1 – 2	< 5	0.10	6000	Zerodur	16 – 25	Au	≤ 250	n.	[9] [10] [11]
Chandra AXAF	1999	0.1 – 10	0.5	0.1	12500	Zerodur	~ 25	Ir	≤ 250	n.	[9] [10] [12] [13]
Replicated Ni shells mirror technology											
XMM-Newton	1999	0.1 – 12	15	0.15 (×3)	2834	Ni	0.5 – 1.1	Au	≤ 250	n.	[9] [10]
JET-X/ Swift	2004	0.2 – 10	18	0.011	6090	Ni	0.6 – 1.1	n.	n.	n.	[9] [10]
eROSITA on RST	2017	0.3 – 10	15	0.0350 (×7)	1171	Ni	0.2 – 0.4	Au	≤ 250	n.	[9] [10]
Conical approximation of Wolter I configuration											
Replicated Ni shells mirror technology											
BeppoSAX	1996	0.1 – 10	60	0.0123 (×4)	7317	Ni	0.2 – 0.4	Au	≤ 250	n.	[9] [10]
Thin Al foil segments mirror technology											
ASCA	1993	≤ 12	200	0.041 (×4)	975	Al	0.125	Au	n.	0.35	[9] [10]
Suzaku ASTRO-E	2005	0.2 – 12	114	0.04 (×5)	300	Al	0.155	Au (XRT-I) / Pt (XRT-S)	n. / n.	n. / n.	[9] [10]
Hitomi ASTRO-H HXT	2016	5 – 80	102	≥ 0.03 (×2) @ 30 keV	2660	Al	0.2	Pt/C	n.	n.	[9] [10]
Hitomi ASTRO-H SXT	2016	0.3 – 12	102	0.0562 (×2)	800	Al	0.15 – 0.31	Au	n.	n.	[9] [10]
Slumped thin glass foils mirror technology											
HEFT++	2005	20 – 70	60	0.025	3600	glass Schott-D263	0.3	W/aSiC multilayers (500 layer pairs)	3600	n.	[9] [10] [14] [15] [16]
NuSTAR	2012	6 – 79	58	0.05 (×2)	500	glass Schott-D263	0.2	Pt/C multilayers (inner foils) / W/Si multilayers (outer foils)	n. / n.	0.3	[9] [10]
Wolter-Schwarzschild configuration											
Silicon pore optics mirror technology											
ATHENA	2028	0.3 – 12	5	2.00	< 500	Si	0.17	Ir/B₄C based bi- or multi-layer	11nm/8nm or ratio: 0.6	< 0.5	[6] [9] [10] [17] [18]
Technology of actively or passively adjustable segments											
X-ray Surveyor	2030	0.2 – 10	0.5	3	500	will depend on the applied technology					[7] [8]

Coating of glass mirrors enhances their X-ray reflectivity. The coating material needs to be selected depending on its reflectivity in the energy range of the X-rays to be investigated. Fig. 2 shows the calculated reflectivity of coating materials used for different space-based missions for photon energies below 10 keV. An rms surface roughness of 0 nm, a density of the bulk material and a grazing incidence angle of 0.8° are assumed for the calculations. Among the high-Z materials gold (Au), platinum (Pt), tungsten (W) and iridium (Ir), Ir depicts the highest reflectivity over the whole energy range up to 10 keV. The combination of Ir and a boron carbide (B_4C) overlay as a bilayer coating or as the basis of a multilayer coating – as it is intended by the ESA for the future ATHENA telescope – should depict a quite high reflectivity for photon energies below 5 keV. B_4C indeed overwhelms the sharp drop in reflectivity of Ir at photon energies close to 2 keV with a quite high reflectivity of about 0.9. Coatings based on Ir/B_4C are developed at DTU Space, Technical University of Denmark [18-21].
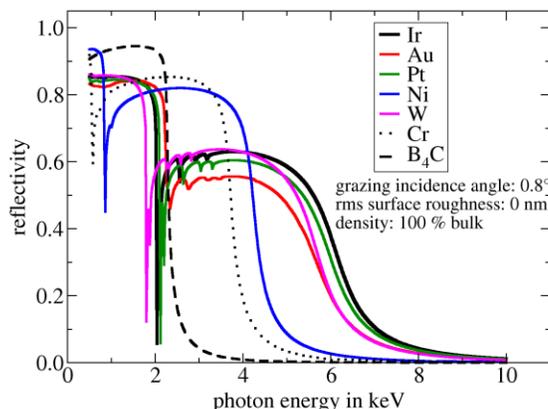

Fig. 2. Computed X-ray reflectivity behavior of different coating materials for a grazing incidence angle of 0.8° and assuming a bulk density and no surface roughness. Calculations obtained from [22, 23].

The presented work is related to the development of iridium coatings on thin glasses at the Aschaffenburg University of Applied Sciences. The mirror substrate production technology selected in this case is based on slumping thin glass foils, applying an indirect slumping method which is developed and investigated at the Max-Planck-Institute for Extraterrestrial Physics (MPE).

II. X-RAY MIRROR CONCEPTION

A. Basic requirements for space-based optics

Among the performance requirements with regards to the angular resolution and the effective area in the requested photon energy range, X-ray telescopes have to fulfil some basic exigences related to the materials employed to build them up. To ensure a lightweight telescope system a low density of the materials is necessary. Chemically inert materials are preferred to reduce interactions with the environment. A further aspect is stability against temperature and time during storage and at launch. No stability tests under different environmental conditions were done so far within this project.

B. X-ray mirror production

The principle of the mirror production of X-ray telescopes proposed by MPE should be suitable for a serial production process [9, 24, 25]. Initial flat glass substrates are supported by a partially hyperbola and partially parabola shaped mould. By heating up the glass segments at temperatures above the glass transition temperature, the glass deforms and adopts under gravitation's influence the shape of the mould yielding to hyperbola and parabola glass segments. Applying reflective coatings on the mirror surface will improve the reflectivity of the mirrors. Several coated glass segments are aligned and integrated into single modules, which will be assembled in a last step to obtain the whole X-ray mirror system. The advantage of the indirect glass slumping technique is that the further coated glass surface is not in contact with the mould, so that no damage of this surface should occur ensuring no significant modification of the rms surface roughness of the mirror substrate.

C. Mirror substrates

D263Teco borosilicate glass of Co. Schott (Germany) of dimensions 100 mm x 200 mm and circular wafers with 150 mm diameter, both with a thickness of 0.4 mm, were selected for the coating development. This glass type was already used for the HEFT (High Energy Focusing Telescope) balloon experiment and for the NuSTAR (Nuclear spectroscopic telescope area) mission, both using the slumped thin glass foils mirror technology. With

an rms roughness below 1 nm [26] this glass material depicts a good surface quality. According to its coefficient of thermal expansion of 7.2 ppm/K within the temperature range from 20°C to 300°C [26], it should also be compatible with Ir, which depicts a coefficient of thermal expansion of 7.33 ppm/K in the temperature range from 30°C to 865°C [27]. The glass transition temperature of 557°C [26] is advantageous for the slumping process insofar that relatively low temperatures (maximum 620°C [9]) have hence to be reached. The glass density of 2.51 g/cm^3 [26] is relevant for the mass to area ratio of the telescope system.

D. Mirror coating

Due to the high reflectivity of bulk Ir when compared to others like Au or Pt, Ir has been selected for the mirror coating. A further advantage is the chemical stability of Ir [28]. Challenges in the development of an Ir-coating for X-ray mirrors lie in producing a coating which simultaneously should depict a good reflectivity for X-rays for photon energies below 10 keV, a good stability in time and should not induce any mechanical deformation of the optical system. The reflectivity is influenced by both the density of the material and the surface roughness. The combination of a high density and low surface roughness (below 1 nm) will provide the best reflectivity. Intrinsic stress of the coating would modify the initial shape of the mirror substrate and therefore generate aberrations. Furthermore such stress could affect the stability of the system with respect to time while on a microscopic scale the coating will tend to rearrange itself in order to reduce the stress.

III. EXPERIMENTAL METHODS

A. Coating method

Thin film deposition of Ir on the mirror substrate occurs in a radio frequency (rf) magnetron sputtering process using argon (Ar) with a purity of 99.999 % as process gas. The physical vapor deposition system used is a sputtering equipment type VPA 21 from the company Aurion Anlagentechnik GmbH (Germany). Previous to the sputtering process the vacuum chamber was evacuated to a pressure of $5 \cdot 10^{-5}$ mbar to avoid contamination of the coating with air. The iridium target (purity: 99.9 %, diameter: 150 mm) is electrically connected to an rf generator supplying a power of 300 W. The inclination angle between the target and the normal of the substrate is 40° and the target to substrate distance amounts to 120 mm along the normal. The Ar gas flow and the pumping power were adjusted for each sputtering run to set a definite sputtering pressure which was remained constant during the sputtering process. The substrate is not actively heated. Thin Ir films were produced with thicknesses of 30 nm or 100 nm with an homogeneity of 2 % on a substrate diameter of 150 mm. This homogeneity is mainly achieved by rotation of the substrate during sputtering.

According to Broadway et al. [29] the sputtering pressure is a relevant parameter influencing the intrinsic stress of coating. Coating experiments were therefore performed using different sputtering pressures to investigate the influence of this parameter on the film properties. The sputtering pressure was varied between $5.6 \cdot 10^{-4}$ mbar and $6.0 \cdot 10^{-2}$ mbar in different sputtering runs (corresponding to Ar gas flows varying between 10 sccm and 50 sccm). Investigations of the coating properties presented here were performed using circular flat glass samples with a diameter of 150 mm as mirror substrates as well as smaller glass pieces.

B. Optical metrology

Deformation of the glass after applying a coating is an indicator for the coating stress. The coating stress therefore should be evaluated from the differences in the shape of the glass after and before coating. Measurements of the shape of the glass were performed by using a white-light interferometer TMS-500 TopMap from Polytec GmbH (Germany). For the measurements the circular glass wafer was supported at the edges over a width of 2 mm by a radial support. In this configuration the wafer is deformed due to gravitation. To evaluate the coating stress from the comparison of the measurements taken before and after coating we have to ensure that the deformation due to gravity effects is identical in both cases. This requests a good repeatability in positioning the glass samples on the support and relative to the measurement instrument.

C. X-ray metrology

Coating properties were investigated using X-ray metrology with Cu-Kα-radiation with 8048 eV photon energy and two different experimental set-ups. The crystal structure of the Ir coatings was characterized by performing X-ray diffraction (XRD) measurements at the University of Applied Sciences Saarbrücken (Germany) with a D8 focus X-ray Diffraction instrument supplied by Bruker AXS GmbH (Germany). The measurements occured by grazing incidence diffraction with a grazing incidence angle of 0.8° and varying the angle 2θ between the surface of the coated sample and the detector from 20° to 110° in 0.02° steps. In addition the density and the surface micro-roughness of the Ir coatings were investigated by means of grazing incidence X-ray reflectometry (GIXRR) measurements at the Tongji University (China) with a D1 X-ray Reflectometry equipment from Bede Scientific

Inc. (USA). The grazing incidence angle was in this case varied from 0° to 2.0° in 0.01° steps using the θ-2θ scanning mode.

IV. PROPERTIES OF IR-COATED MIRRORS

A. Glass deformation due to coating

Optical measurements of the shape of the surface of circular glasses D263Teco after and before Ir-coating reveal that at low sputtering pressures the glass is significantly deformed due to the coating (at $2.3 \cdot 10^{-3}$ mbar several µm deformation were noted). In contrast, the observed glass deformation was lower when increasing the sputtering pressure. This observation indicates that the coating stress can be influenced by variation of the sputtering pressure, it decreases when increasing the sputtering pressure. A quantitative analysis however is not available yet since the repeatability in positioning the circular glass samples on the measurement set-up was not sufficient to get confident values for the deformation. The qualitative tendency of the dependency of the coating stress on the sputtering pressure is roughly depicted by the arrow in the upper sketch of Fig. 4.

B. Properties of Ir-coatings

The sharp intensity peaks of diffracted X-rays obtained from XRD measurements on two Ir-coated glass samples sputtered at a pressure of $5.0 \cdot 10^{-3}$ mbar and of $4.5 \cdot 10^{-2}$ mbar respectively revealed that both Ir thin films are crystalline (see the green and black curves of the left diagram of Fig. 3). The comparison of the experimental data with the calculated X-ray diffraction peaks positions of Owen et al. [30] for an Ir cubic face centered (fcc) structure with a lattice parameter of 3.86 Å shows a quite good concordance (see the red lines corresponding to the data from the Powder Diffraction File 03-065-1686 [31] in the left diagram of Fig. 3). We can conclude from these measurements that the crystal structure of both Ir thin films is a fcc structure with a lattice parameter close to 3.86 Å. However, differences are visible in the full width at half maximum (FWHM) of the main peak at $2\theta \approx 40.9°$ corresponding to the X-ray diffraction peak of the (111) lattice plane. The FWHM lies by about 0.76° for the Ir film sputtered at $5.0 \cdot 10^{-3}$ mbar and by 1.17° for the Ir film obtained from sputtering at a pressure of $4.5 \cdot 10^{-2}$ mbar (see the lower diagram of Fig. 4). According to Scherrer´s formula [32, 33] this difference is an indication for a finer-grained crystalline structure at sputtering pressures of $4.5 \cdot 10^{-2}$ mbar than at $5.0 \cdot 10^{-3}$ mbar.

The right diagram of Fig. 3 depicts the intensity measured on a 30 nm thick Ir film obtained from X-ray reflectometry measurements as a function of grazing incidence angle. In this case the Ir film was obtained from sputtering at $4.5 \cdot 10^{-2}$ mbar. With a grazing incidence angle of ~ 0.7° the intensity reflected by the Ir film is roughly reduced by two orders of magnitude of the intensity reflected when the incidence angle is 0.1°. For a given material, the dependency of the reflected X-ray intensity on the grazing incidence is dependent on the thickness, the density and the surface roughness. The best fit of the experimental data with an one-layer model [22, 23] taking into account these dependencies therefore yields informations about the film thickness, the density and the surface roughness.
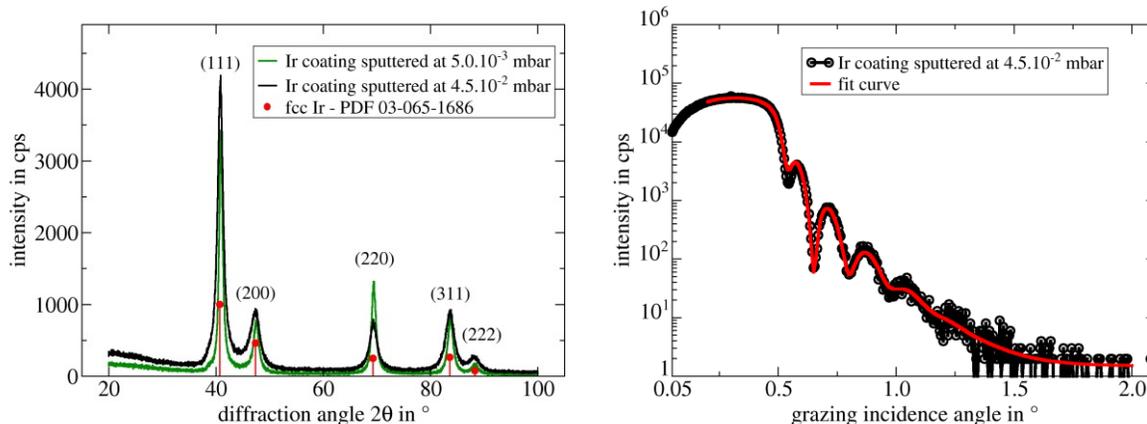

Fig. 3. Left: X-ray diffractogram of two 100 nm thick Ir films sputtered at $5.0 \cdot 10^{-3}$ mbar (green) and respectively at $4.5 \cdot 10^{-2}$ mbar (black), and calculated X-ray diffraction peaks of cubic face centered (fcc) Ir from the Powder Diffraction File 03-065-1686 [31, 30] (red). Right: Grazing incidence X-ray reflectometry measurement data (circles) of a X-ray mirror with a 30 nm thick Ir film coated at $4.5 \cdot 10^{-2}$ mbar and corresponding fit curve (red line).

It was observed that the reflectivity of Ir thin films sputtered at different sputtering pressures but with same thickness, have different dependencies on the grazing incidence angle, indicating different densities and different rms surface roughnesses. The results on the evaluated density and rms surface roughness of the different Ir thin

films are graphically represented in Fig. 4 as a function of the total sputtering pressure. When sputtering at total pressures below $3.0 \cdot 10^{-2}$ mbar, both the density and the rms surface roughness of the Ir films are strongly sensitive to the pressure, whereas, above $3.0 \cdot 10^{-2}$ mbar, the density and the rms surface roughness seem not to vary significantly with the sputtering pressure. Sputtering at a total pressure of $5.0 \cdot 10^{-3}$ mbar results in an Ir thin film with a density close to 90 % of bulk density and an rms surface roughness of 1 nm. When increasing the sputtering pressure to and above $3.0 \cdot 10^{-2}$ mbar the film density is lowered to 62 % of bulk density whereas the rms surface roughness is increased to roughly 2 nm [32].
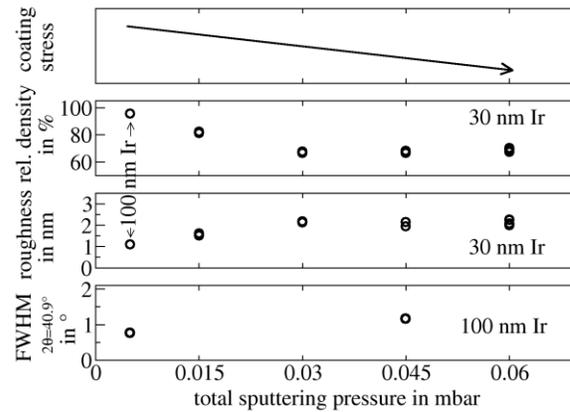
Fig. 4. Properties of the iridium thin films as a function of the total sputtering pressure [34].

C. X-ray reflectivity of Ir-coated mirrors

The results obtained from the X-ray metrology on Ir thin films show that the properties of Ir coatings are strongly correlated to the sputtering pressure. The reflectivity arising out of a reduced density and an rms surface roughness up to 2 nm is evaluated for a grazing incidence angle of 0.8° and for photon energies below 10 keV [22, 23]. Fig. 5 depicts the calculated reflectivity of Ir with the density and rms surface roughness values from Fig. 4, i.e. corresponding to the properties of Ir coatings obtained when sputtering at pressures between $5.0 \cdot 10^{-3}$ mbar and $6.0 \cdot 10^{-2}$ mbar. In the left diagram of Fig. 5 X-ray reflectivity of bulk Ir, Au and Pt are also drawn (dashed lines) to compare the expected reflectivity of the different Ir coatings to that of the high-Z bulk materials. We can observe that the reflectivity of Ir coatings sputtered at pressures higher than $5.0 \cdot 10^{-3}$ mbar drops significantly with increasing sputtering pressure. This can also be seen from the right diagram of Fig. 5, which shows the expected reflectivities of Ir coatings for a photon energy of 5 keV as a function of the total sputtering pressure. At lower density and higher rms roughness the reflectivity is reduced. However the Ir coating sputtered at a pressure of $5.0 \cdot 10^{-3}$ mbar (density: 90% bulk, rms surface roughness: 1 nm) should depict reflectivity similar to bulk Au for photon energies up to 10 keV, but lower than bulk Pt with no roughness.
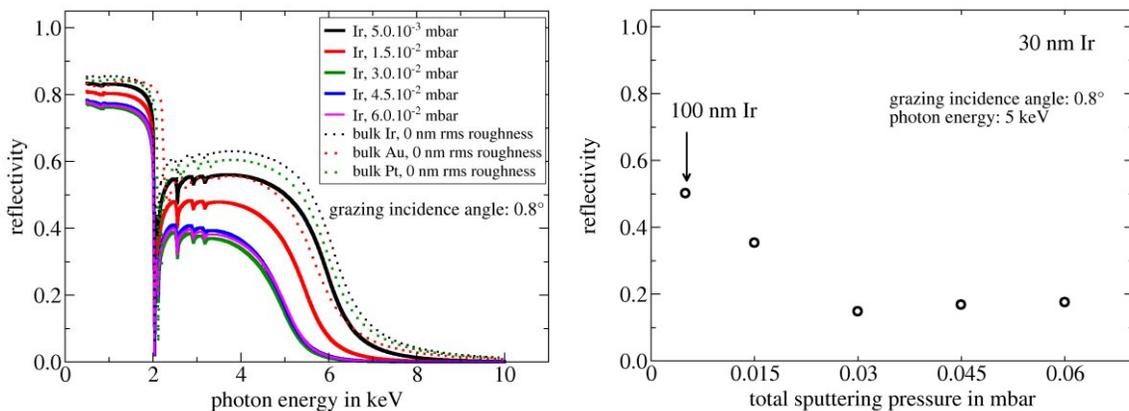

Fig. 5. Calculated X-ray reflectivity of the Ir thin films with density and rms surface roughness of Fig. 4. Calculations obtained from [22, 23] using a grazing incidence angle of 0.8°. Left: Plot as a function of the photon energy including a comparison to the X-ray reflectivity of Ir, Au and Pt bulk materials with assumed rms surface roughness of 0 nm. Right: Plot as a function of the total sputtering pressure at a photon energy of 5 keV.

V. SUMMARY AND OUTLOOK

It is proposed to realize Wolter I type telescopes with an assembly of segmented X-ray mirrors to enhance the angular resolution and the effective area aimed for future lightweight X-ray telescopes, each X-ray mirror consisting of a thin glass coated with a reflective material. Against this background, the Max-Planck-Institute for Extraterrestrial Physics develops an indirect slumping glass technology to shape the mirror glass substrates to the required parabola and hyperbola shapes, whereas Aschaffenburg University of Applied Sciences is investigating a coating process for the glass segments based on the high Z-material Ir.

For this purpose low coating stress is required to minimize aberrations of X-ray mirrors. According to our preliminary qualitative results on glass deformation due to coating, low stress Ir thin films are obtained by sputter deposition at high pressures ($> 3.0 \cdot 10^{-2}$ mbar). However, these films depict a fine-grained crystalline fcc structure with reduced density (at least 60% of bulk density) and higher rms surface roughness (about 2 nm), leading to reduced reflectivity for photon energies up to 10 keV. Iridium thin films sputtered at lower total pressures ($5.0 \cdot 10^{-3}$ mbar) depict in contrast high density and low surface roughness, but high intrinsic stress. Annealing those Ir films should involve a rearrangement of the Ir atoms, which should reduce the coating stress [35]. A further way is to compensate the deformation by applying the same coating on both sides of the mirror or by deposition of an additional layer with the opposite stress between the mirror substrate and the Ir coating. These possibilities will be taken into account for the next steps of developing Ir coated X-ray mirrors. Further efforts to improve the shape measurements will be performed to provide the quantitative evaluation of the glass deformation due to the coating.

ACKNOWLEDGEMENT

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