PROCEEDINGS OF SPIE

SPIEDigitalLibrary.org/conference-proceedings-of-spie

Development of iridium coated x-ray mirrors for astronomical applications

Thorsten Döhring, Anne-Catherine Probst, Florian Emmerich, Manfred Stollenwerk, Veronika Stehlíková, et al.

Thorsten Döhring, Anne-Catherine Probst, Florian Emmerich, Manfred Stollenwerk, Veronika Stehlíková, Peter Friedrich, Christine Damm, "Development of iridium coated x-ray mirrors for astronomical applications," Proc. SPIE 10399, Optics for EUV, X-Ray, and Gamma-Ray Astronomy VIII, 103991C (29 August 2017); doi: 10.1117/12.2273988

Event: SPIE Optical Engineering + Applications, 2017, San Diego, California, United States

SPIE.

Downloaded From: https://www.spiedigitallibrary.org/conference-proceedings-of-spie on 2/28/2019 Terms of Use: https://www.spiedigitallibrary.org/terms-of-use

Development of iridium coated X-ray mirrors for astronomical applications

Thorsten Döhring*[a], Anne-Catherine Probst[a], Florian Emmerich[a], Manfred Stollenwerk[a], Veronika Stehlíková[b,c], Peter Friedrich[c], Christine Damm[d]

[a] Aschaffenburg University of Applied Sciences, D-63743 Aschaffenburg, Germany;
[b] Czech Technical University in Prague, CZ-16627 Prague 6, Czech Republic;
[c] Max-Planck-Institute for Extraterrestrial Physics, D-85748 Garching, Germany
[d] IFW Dresden e.V., Helmholtzstraße 20, D-01069 Dresden, Germany

ABSTRACT

Future space-based X-ray observatories need to be very lightweight for launcher mass constraints. Therefore they will use a reduced mirror thickness, which results in the additional requirement of low coating stress to avoid deformation of the initial precisely shaped mirror substrates. Due to their excellent reflection properties iridium coatings are sometimes applied for grazing incidence mirrors in astronomical X-ray telescopes. At Aschaffenburg University of Applied Sciences the coating of thin iridium films by an RF-magnetron sputtering technique is under development. The work is embedded in collaborations with the Max-Planck-Institute for Extraterrestrial Physics in Germany, the Czech Technical University in Prague, the Osservatorio Astronomico di Brera in Italy, the German Leibniz Institute for Solid State and Materials Research in Dresden, and the French Institute Fresnel. Sputtering with different parameters leads to iridium films with different properties. The current work is focused on the microstructure of the iridium coatings to study the influence of the substrate and of the argon gas pressure on the thin film growing process. Correlations between coating density, surface micro-roughness, the crystalline structure of the iridium layers, and the expected reflectivity of the X-ray mirror as well as coating stress effects are presented and discussed. The final goal of the project is to integrate the produced prototype mirrors into an X-ray telescope module. On a longer timescale measurements of the mirror modules optical performance are planned at the X-ray test facility PANTER.

Keywords: X-ray, telescope, mirrors, lightweight optics, Lobster eye, iridium, coating, sputtering

1. INTRODUCTION

Previously used mirror technologies are not able to fulfil the challenging requirements of future X-ray telescopes [1]. Consequently new technical approaches for the X-ray mirror production are under development. In Europe the technical baseline for the planned X-ray observatory ATHENA is the innovative approach of silicon pore optics [2]. The NuSTAR mission of NASA uses segmented mirrors shells made from thin slumped glasses, successfully demonstrating the feasibility of the glass forming technology for lightweighted X-ray telescopes [3]. Also in Europe the hot forming of thin glasses is being developed as an alternative technology for lightweight X-ray mirrors [4]. Another design approach is to use coated silicon wafer as substrates for Lobster Eye type wide-field optics [5, 6].

Grazing incidence X-ray mirrors are usually coated with thin layers of high-Z materials to provide highest reflectivity for X-rays. Thereby the technical requirements for mirrors coatings in the soft X-ray range are [7]:

- high X-ray reflectivity, also in double reflection
- no mirror shape deformation introduced by the coating, corresponding to low coating stress
- a coating process suitable also for serial production
- no degradation of the mirrors during storage and in space.

For previous X-ray telescopes often gold (Au) was selected for the mirrors reflective layer [8]. Calculations based on the optical constants of high-Z materials showed that iridium coatings promise highest reflectivity for X-ray mirrors in the intended photon energy range [9]. Based on this result it was decided to develop a mirror coating based on this alternative material.

* Contact: thorsten.doehring@h-ab.de; phone +49 6021 4206782; fax +49 6021 4206801; www.h-ab.de

Optics for EUV, X-Ray, and Gamma-Ray Astronomy VIII, edited by Stephen L. O'Dell, Giovanni Pareschi, Proc. of SPIE Vol. 10399, 103991C · © 2017 SPIE · CCC code: 0277-786X/17/$18 · doi: 10.1117/12.2273988

Downloaded From: https://www.spiedigitallibrary.org/conference-proceedings-of-spie on 2/28/2019
Terms of Use: https://www.spiedigitallibrary.org/terms-of-use

Aschaffenburg University purchased an iridium sputter target (dia. 150 mm x 3 mm) in September 2015, which provides a quite unique possibility for the development of X-ray mirror coatings. Thereby Aschaffenburg University and its partners are following two different optic design approaches, both using iridium coatings developed by Aschaffenburg University.

Commonly astronomical X-ray telescopes are based on the Wolter I optical design, which consists of two consecutive reflections at grazing incidence on a primary paraboloid mirror and a secondary hyperboloid mirror, as depicted in figure 1. The three main parameters describing an X-ray telescope are the collecting area, the angular resolution and the weight per unit area. To enhance the collecting area of the telescope for a given geometrical area (i.e. the filling factor of the optical system), a number of mirrors are integrated inside each other, taking care not to experience mutual obscuration. The thinner the mirrors, the tighter they can be nested [10]. By using segmented mirrors and not monolithic mirror shells, the production process is more adequate for a serial production.

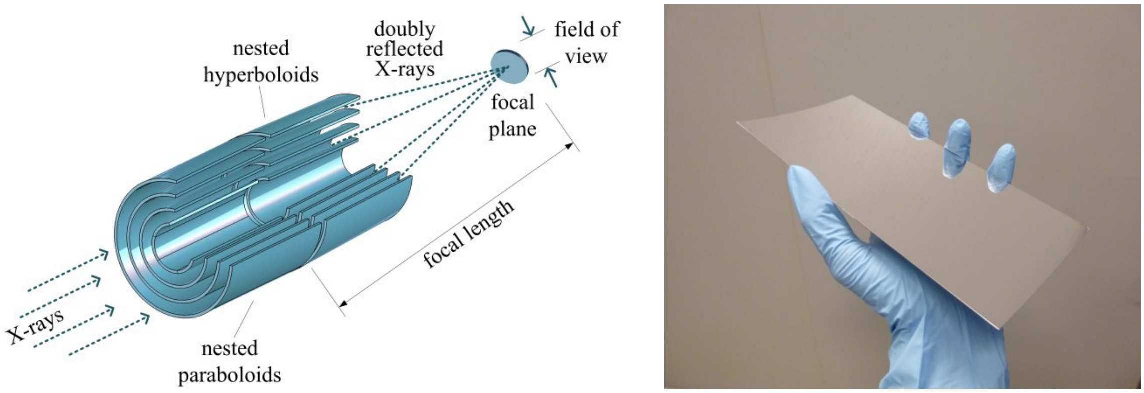


Fig. 1: Principle of a segmented Wolter I type X-ray telescope and a corresponding slumped glass mirror segment.

Historically also other designs of X-ray optics have been developed. Already in 1979, Roger Angel presented an imaging system for an X-ray telescope based on the eye of a Lobster [11]. Based on such bionic approach the Czech Technical University in Prague had chosen a special Lobster eye X-ray optics design in Schmidt's arrangement, which uses dual reflections to increase the collecting area of the astronomical telescope [6, 12]. As shown in figure 2, the individual mirrors of this wide-field telescope are made of flat silicon wafers.

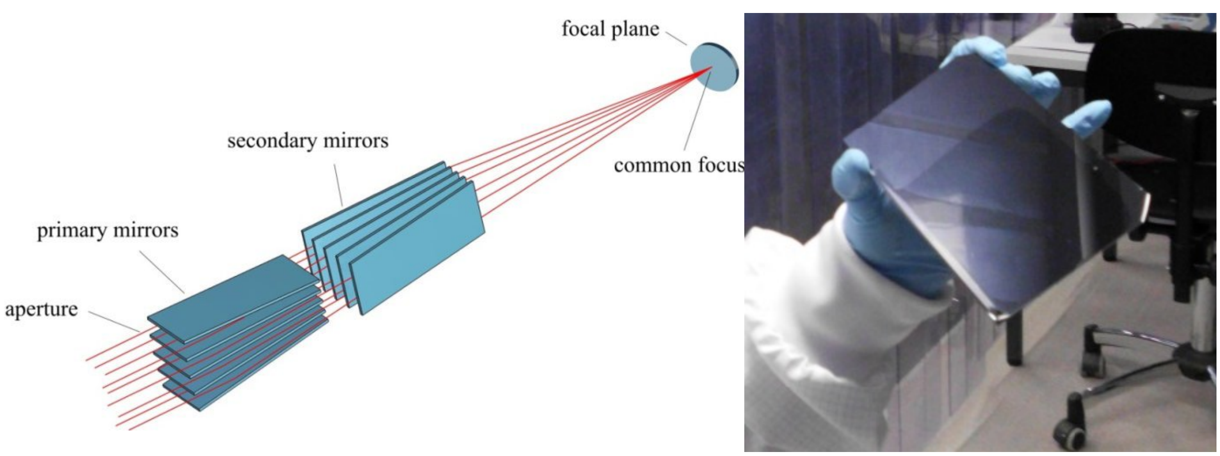


Figure 2: Principle of the modified Lobster Eye X-ray optics and a corresponding coated silicon wafer.

Downloaded From: https://www.spiedigitallibrary.org/conference-proceedings-of-spie on 2/28/2019
Terms of Use: https://www.spiedigitallibrary.org/terms-of-use

2. PROGRAMMATIC CONTEXT

As reflected in Figure 3, the presented work is embedded in collaborations of Aschaffenburg University with the Max-Planck-Institute for Extraterrestrial Physics in Garching / Germany, the Czech Technical University in Prague / Czech Republic, the Osservatorio Astronomico di Brera in Italy, the Leibniz Institute for Solid State and Materials Research in Dresden / Germany, and the French Institute Fresnel in Marseille. Thereby financial support from different funding agencies has been granted, allowing the project work to be executed.

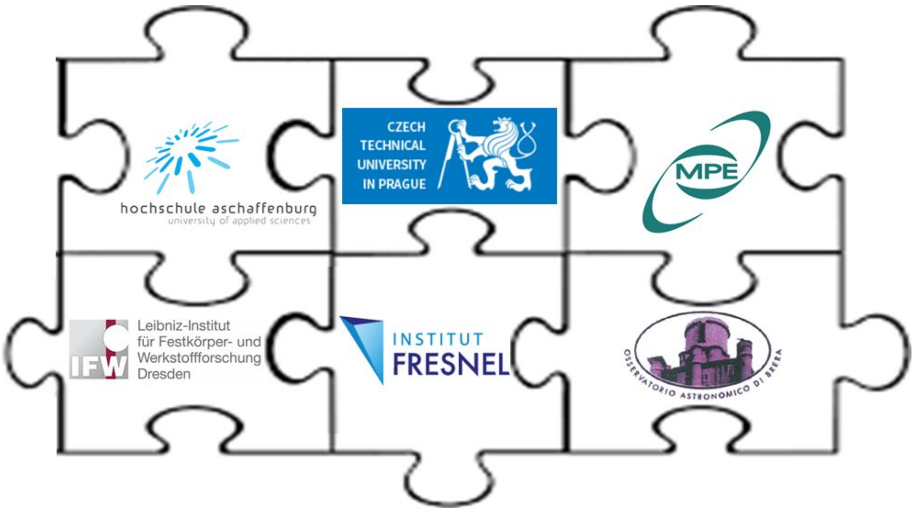


Fig. 3: Logos of Aschaffenburg University and its international project partners.

Technical and economic aspects of an X-ray mirror production based on slumped glass optics are studied within the interdisciplinary project INTRAAST, a German acronym for "industry transfer of astronomical mirror technologies" [13]. This project, funded for the years 2015 to 2017, is embedded in the cooperation between the Max-Planck-Institute for Extraterrestrial Physics and the Aschaffenburg University of Applied Sciences. The INTRAAST project is gratefully supported by funding from the Bavarian State Ministry for Education and Culture, Sciences and Art.

Scientist from the Aschaffenburg University of Applied Sciences and the Czech Technical University in Prague started the project JEUMICO (an acronym for "Joint European Mirror Competence") to develop mirrors for X-ray telescopes based on a modified Lobster Eye type optical design. The aim of the project is to combine experience, expertise and instrumentation of the Bavarian and the Czech partners by the application of thin film mirror coatings, especially for astrophysical applications. The projects goal is also to enhance the scientific cooperation between both research groups, based on a living cooperation between both universities on an organizational level. The JEUMICO project was funded for the years 2016 to 2017 via the funding agency BAYHOST, the Bavarian Academic Center for Central, Eastern and Southeastern Europe [12].

The cooperation of Aschaffenburg University with the Osservatorio Astronomico di Brera has been made possible by two travelling grants, funded by the AHEAD-project as part of Horizon 2020 program. In addition the Bavarian-French Academic Centre (BFHZ) supported the cooperation between Aschaffenburg University and the Institute Fresnel also by travelling grants.

3. EXPERIMENTAL SET-UP FOR THE COATING DEVELOPMENT

Radio frequency (rf) magnetron sputtering technology is applied at Aschaffenburg University of Applied Sciences for the iridium coating process, using a sputtering equipment system supplied by Aurion Anlagentechnik GmbH (Germany), as shown in figure 4 [8]. The principle of the corresponding sputtering process is schematically depicted in figure 5. The iridium sputtering target is inclined by an angle of 40° with regards to the normal of the substrate´s plane and the distance between target and substrate is at about 120 mm along the normal. To prevent the coating from contamination, the background pressure in the vacuum chamber was set to $5 \cdot 10^{-5}$ mbar. At the iridium target the electrical power was adjusted to 300 W. The sputtering process occurs with an argon gas plasma (purity: 99.999 %), which is ionized by the

Downloaded From: https://www.spiedigitallibrary.org/conference-proceedings-of-spie on 2/28/2019
Terms of Use: https://www.spiedigitallibrary.org/terms-of-use

Figure 4: Sputtering chamber at Aschaffenburg University.

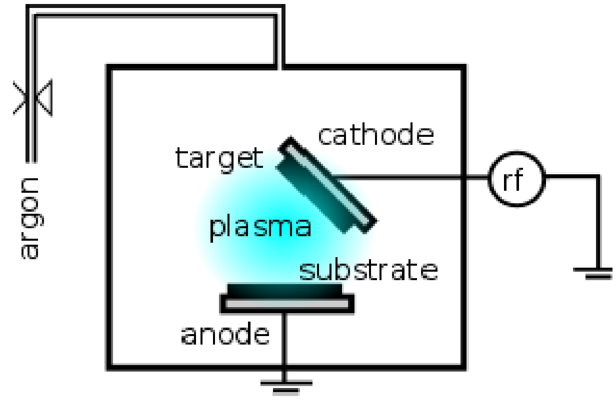
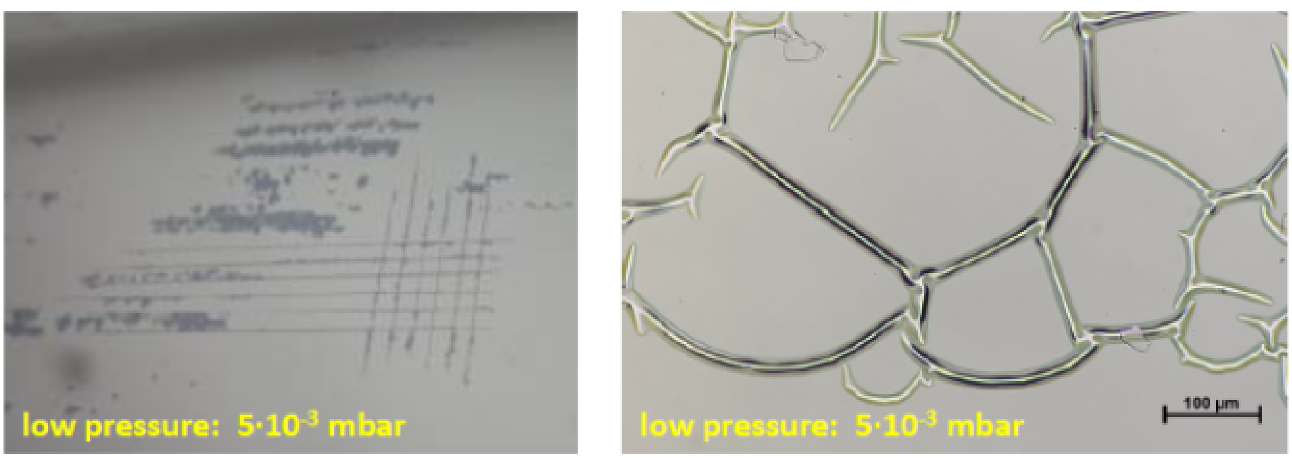


Figure 5: Schematic set-up for the sputtering process.

electrical field and confined close to the target by the magnetic field. The argon ions are bombarding the surface of the target from which iridium atoms are ejected. During the sputtering process the argon gas flow was set to 50 sccm. The main parameter during coating development was the variation of the argon sputtering pressure. By adjusting the throughput of the valve connecting the sputtering chamber to the pump system, the total pressure was varied between $5.6 \cdot 10^{-4}$ mbar and $8 \cdot 10^{-2}$ mbar to investigate the influence of the sputtering pressure on the coating properties. The iridium atoms sputtered from the target by the argon ions condense on the substrate, building up an iridium film which thickness is adjusted by the sputtering time. Typically film thicknesses between 30 nm and 100 nm were produced. No additional active heating was applied on the substrate during the sputtering process. The substrate itself is rotating with a rotation speed of 8 rpm to improve the homogeneity of the film deposition. The homogeneity in the film thickness was evaluated to ± 2% on a diameter of 150 mm. The iridium coatings were deposited on silicon wafers, on rectangular and circular flat glass samples as well as on slumped glass samples. During the development process investigations of the coating properties were also performed using smaller glass pieces.

4. THE CHALLENGE OF COATING STRESS



a) Cross-cut adhesion test **b) Micrograph of stress induced cracks**

Figure 6 a&b. Cross-cut adhesion test (left) and a micrograph of stress induced cracks in an iridium coated mirror.

Iridium coatings promise high reflectivity for grazing incidence X-ray mirrors [9]. This material is therefore often preferred for the coating of soft X-ray telescopes. However, the applied iridium layers may suffer from high coating stress [14]. Previous coating campaigns showed that iridium coatings with high reflectivity are unfortunately able to deform thin mirror glass substrates (0.4 mm thick D263 glass) quite strongly, due to their high intrinsic coating stress [7]. In extreme cases, the high stress level can induce cracks within the coated layer. In some cases even delamination of coated iridium

Downloaded From: https://www.spiedigitallibrary.org/conference-proceedings-of-spie on 2/28/2019
Terms of Use: https://www.spiedigitallibrary.org/terms-of-use

layers has been observed. To test the long-term stability of the iridium coatings we performed a cross-cut adhesion tests according to the norm ISO2409. These adhesion tests were realized in cooperation with the Czech company HVM Plasma s.r.o.. An example of such test is illustrated in figure 6a, were insufficient adhesion was found for an iridium coating directly sputtered on a silicon wafer at a low sputtering pressure of $5 \cdot 10^{-3}$ mbar. Another example is shown in figure 6b, where the micrograph shows severe cracks in a 50 nm thick iridium coating sputtered on a silicon wafer, also sputtered at a low sputtering pressure of $5 \cdot 10^{-3}$ mbar. These disturbing effects are violating the requirement of long-term stability of the mirrors respectively their coatings. Sometimes the coatings are even not stable in short times.

5. MICROROUGHNESS AND LAYER MICROSTRUCTURE

However, coating stress and long-term stability are not the only important requirements for the coatings. For the discussed request of high reflectivity a low micro-roughness and a high density are required in addition for the coated X-ray mirrors. In a first approach a series of iridium coatings were produced by variation of the argon sputtering pressure. The coatings have been characterized by help of an atomic force microscope (AFM). It can be concluded from figure 7 that the variation of the argon sputtering pressure results in contradicting sputtering parameters [7]. A low pressure resulted in low micro-roughness of the iridium layers and in a high coating density, but these coatings suffer from a high coating stress [8]. On the other hand, a high argon pressure during processing leads to a low coating stress, but the iridium layers have a high roughness and a low coating density [8]. For a deeper understanding of the process we evaluated the layer microstructure of the coatings in more detail.

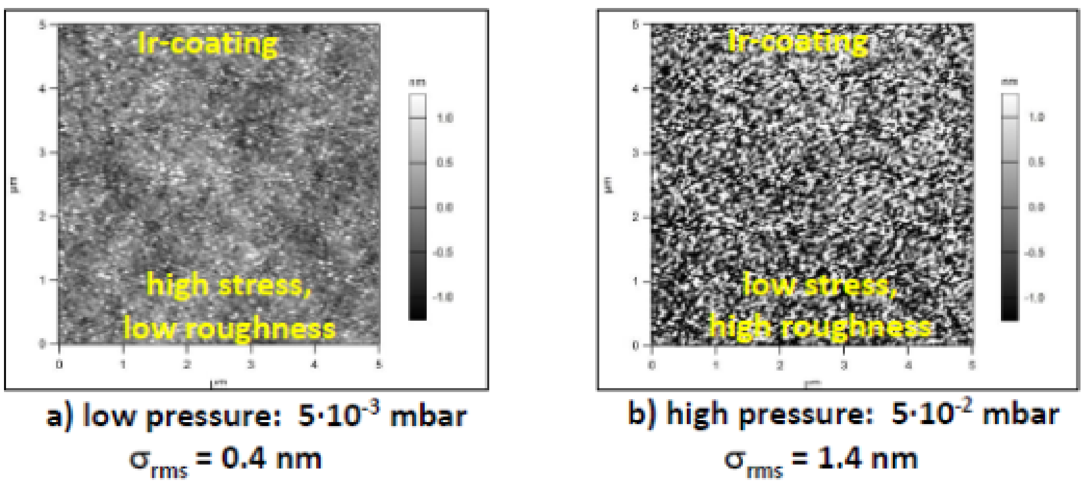
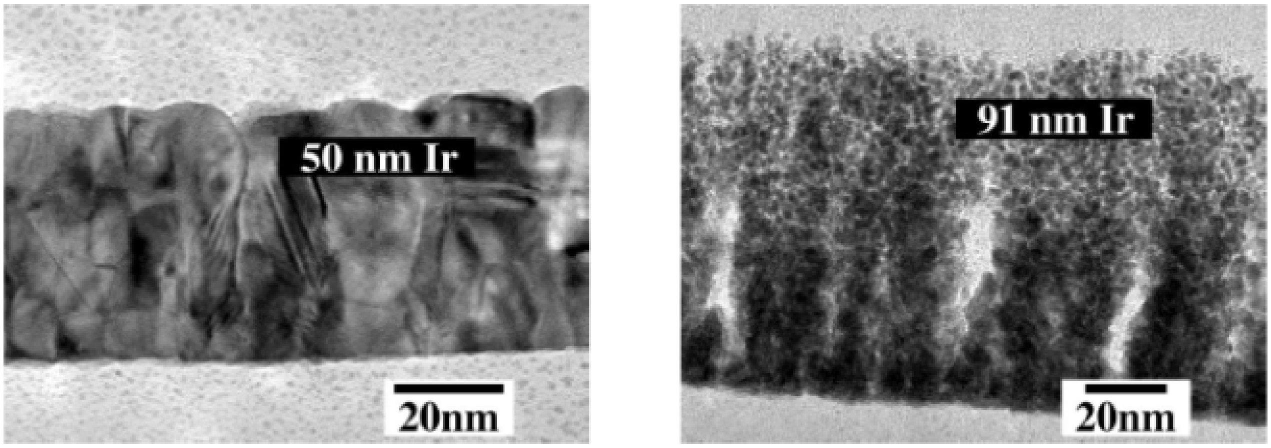


Figure 7 a&b. AFM micrographs of iridium coatings, produced at a low (a) and at a high argon sputtering pressure (b).

It was already described that optimum coating process parameters seems to be contrarious, as for low coating stress high pressure is required and for good roughness and high iridium density low pressure is needed [7]. Therefore high resolution transmission electron microscope (TEM) measurements were performed to study nanostructure of the layers and the growing process. Two examples are shown in figure 8. The influence of argon sputtering pressure on iridium layer growing was found as follows: Low argon pressure during coating resulted in iridium layers of densely packed coarse grains. The surface micro-roughness is following the shape of the individual grains (figure 8a). High argon pressure during sputtering leads to a fine porous structure. The layer is made of small separated grains; resulting in a corresponding high micro-roughness and a low coating density (figure 8b). Both layers are crystalline and combined of face centered cubic (fcc) iridium crystals [8].

From these images it was concluded that the pure variation of argon pressure will not solve the problem of the contradicting coating parameters to achieve good reflectivity and low coating stress simultaneously. So other methods were taken into account.

Downloaded From: https://www.spiedigitallibrary.org/conference-proceedings-of-spie on 2/28/2019
Terms of Use: https://www.spiedigitallibrary.org/terms-of-use



a) low pressure: $5 \cdot 10^{-3}$ mbar   b) high pressure: $8 \cdot 10^{-2}$ mbar

Figure 8 a&b. TEM micrographs of iridium coatings, produced at a low (a) and at a high argon sputtering pressure (b).

6. APPLICATION OF A CHROMIUM ADHESION LAYER

To overcome the challenge of a simultaneous achievement of low coating stress and good reflectivity, in a second approach the application of an intermediate chromium adhesion layer between substrate and iridium coating was studied. Similar approaches are discussed in literature [15, 16]. As shown in figure 9, the cross-cut adhesion tests at mirrors and the corresponding micrograph with a Cr/Ir-bilayer demonstrated that the problems of cracks or delamination caused by high coating stress could be overcome.

a) Cross-cut adhesion test of a Cr/Ir - bilayer   b) Micrograph of a Cr/Ir – bilayer (without cracks)

Figure 9 a&b. Cross-cut test (left) and micrograph of an iridium coated mirror without cracks (right) after application of an intermediate chromium adhesion layer.

Sputtering at a low argon pressure of $5 \cdot 10^{-3}$ mbar should keep the high density of the iridium layer, but we had concerns that the intermediate chromium layer could result in a higher micro-roughness of the reflecting surface.

Fortunately it was found that the application of an intermediate thin Cr adhesion layer (see figure 10b) does not affect the micro-roughness of the uncoated glass (see figure 10a). In conclusion: By applying an intermediate chromium adhesion layer we achieved a suitable mirror coating. Figure 10c depicts the surface of a 50 nm thick iridium coating with an intermediate chromium adhesion layer of 10 nm. In this case also no cracks and no coating delamination were observed.

Downloaded From: https://www.spiedigitallibrary.org/conference-proceedings-of-spie on 2/28/2019
Terms of Use: https://www.spiedigitallibrary.org/terms-of-use

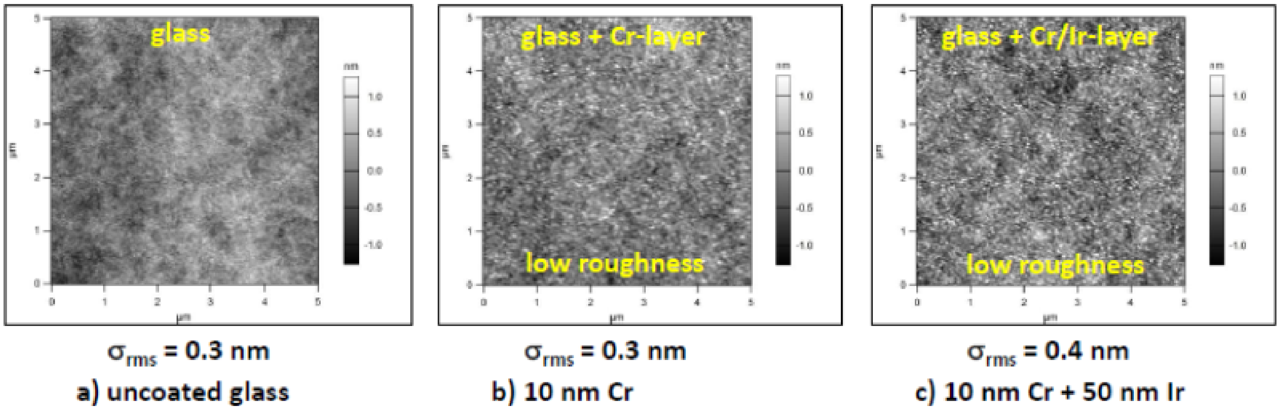


Figure 10 a&b&c. AFM micrographs of a bare glass substrate, a chromium coating, and a Cr/Ir-bilayer coating, both produced at a low argon sputtering pressure of $5\cdot10^{-3}$ mbar.

7. SUMMARY AND OUTLOOK

Previously used mirror technologies are not able to fulfil the challenging requirements of future X-ray telescopes. The need for a new mirror technology consequently triggers the development of new technical approaches for X-ray mirror production. There are different requirements for the X-ray mirrors and their coatings. They have to provide high X-ray reflectivity, low coating stress should avoid mirror shape deformations introduced by the coatings, the coating process need to be suitable also for serial production, and the mirrors should show no degradation of the mirrors during storage and in space.

A first study by variation of argon sputtering pressure resulted in disadvantageous coating properties [7]. Low pressure resulted in low micro-roughness of the iridium layers and in a high coating density, but these coatings suffer from high coating stress. On the other hand, high argon pressure during processing leads to low coating stress, but the iridium layers have a high roughness and a low coating density. For a deeper understanding of the process we evaluated the layer microstructure of the coatings in more detail. It was concluded that the variation of argon pressure alone will not solve the problem of the contradicting sputtering parameters. However, an intermediate chromium adhesion layer can overcome this problem. Further stress compensation methods, especially the optimization of the chromium layer thickness, an annealing of the coating, and backside stress compensation coatings are under development. On a longer timescale measurements of the mirror modules optical performance are planned at the X-ray test facility PANTER.

ACKNOWLEDGEMENT

The presented work was done in collaboration between the Aschaffenburg University of Applied Sciences and its international partners. The political intention to enhance the scientific cooperation between the countries in Europe – and a corresponding generous binational funding allowed this project to be procured. Just recently, in April 2017, funding for the follow-up project TRILAMICO (an acronym for: Trilateral Mirror Collaboration) was granted by the Bavarian-Czech University Agency BTHA and by the Bavarian-French University Centre BFHZ. So the successfully started joint work will continue.

We want to thank Sebastian Zeising from Aschaffenburg University of Applied Sciences for his illustrating drawings. The presented work was done in the frame of the JEUMICO project, which is gratefully funded by a Joint Call of the Bavarian State Ministry for Education and Culture, Sciences and Art and the Ministry of Education, Youth and Sports of the Czech Republic. Additionally parts of the work are performed within the context of the INTRAAST project, which is financed by the Bavarian State Ministry for Education and Culture, Sciences and Art.

Downloaded From: https://www.spiedigitallibrary.org/conference-proceedings-of-spie on 2/28/2019
Terms of Use: https://www.spiedigitallibrary.org/terms-of-use

REFERENCES

[1] L. Proserpio et al.: " Industrialization Scenario for X-ray Telescope Production based on Glass Slumping", Proc. SPIE 9144, 914448 (2014)

[2] M. Bavdaz et al.: "X-Ray Pore Optics Technology and Their Application in Space Telescopes", in: "X-Ray Optics and Instrumentation", Hindawi Publishing Corporation, Volume 2010, Article ID 295095 (2010)

[3] W. W. Zhang: "Manufacture of mirror glass substrates for the NuSTAR mission", Proc. SPIE 7437, 74370N (2009)

[4] A. Winter et al.: "Indirect glass slumping for future x-ray missions: overview, status and progress", Proc. SPIE 9603, 96030S (2015)

[5] R. Hudec et al.: "Applications of Lobster eye optics", Proc. SPIE 9510, 95100A (2015)

[6] V. Stehlíková et al.: "Study of Lobster eye optics with iridium coated X-ray mirrors for a rocket experiment", Proc. SPIE 10235, 1023505 (2017)

[7] T. Döhring et al.: "Development of low-stress iridium coatings for astronomical x-ray mirrors", Proc. SPIE 9905, 99056S (2016)

[8] A.-C. Probst et al.: "Iridium coatings for space based X-ray optics", Proceedings of the International Conference on Space Optics, Biarritz, France, 18 - 21 October 2016, Paper FP_IC_177 (2016)

[9] T. Döhring et al.: "The challenge of developing thin mirror shells for future X-ray telescopes", Proc. SPIE 9628, 962809 (2015)

[10] P. Gorenstein: "Focusing X-Ray Optics for Astronomy" in: S. L. O'Dell (Ed.): "X-Ray Optics and Instrumentation", Hindawi Publishing Corporation (2010)

[11] J. R. P. Angel: "Lobster eyes as X-ray telescopes", Astrophysical Journal, vol. 233, pp. 364–373 (1979)

[12] T. Döhring et al.: „Prototyping iridium coated mirrors for X-ray astronomy", Proc. SPIE 10235, 1023504 (2017)

[13] T. Döhring et al.: "Transfer of astronomical mirror technologies – Project INTRAAST", Proceedings of EOSMTOC conference, ISBN 978-952-93-5069-8 (2015)

[14] D. M. Broadway et al., "Achieving zero stress in iridium, chromium, and nickel thin films", Proc. SPIE 9510, 95100E, 2015

[15] K.-W. Chan, et al., "Coating thin mirror segments for lightweight X-ray optics", Proc. SPIE 8861, 88610X (2013)

[16] K.-W. Chan et al.: "Preserving accurate figures in coating and bonding mirrors for lightweight x-ray telescopes", Proc. SPIE 9144, 914440 (2014)

Downloaded From: https://www.spiedigitallibrary.org/conference-proceedings-of-spie on 2/28/2019
Terms of Use: https://www.spiedigitallibrary.org/terms-of-use