# **International Conference on Space Optics—ICSO 2008**

Toulouse, France

14-17 October 2008

Edited by Josiane Costeraste, Errico Armandillo, and Nikos Karafolas



Space optics with silicon wafers and slumped glass

- R. Hudec
- V. Semencova
- A. Inneman
- M. Skulinova
- et al.



International Conference on Space Optics — ICSO 2008, edited by Josiane Costeraste, Errico Armandillo, Nikos Karafolas, Proc. of SPIE Vol. 10566, 105660Q · © 2008 ESA and CNES CCC code: 0277-786X/17/\$18 · doi: 10.1117/12.2308249

# SPACE OPTICS WITH SILICON WAFERS AND SLUMPED GLASS

R. Hudec<sup>(1,2)</sup>, L. Pina<sup>(2,3)</sup>, V. Semencova<sup>(2,5)</sup>, A. Inneman<sup>(2)</sup>, M. Skulinova<sup>(1)</sup>, L. Sveda<sup>(3)</sup>, M. Mika<sup>(5)</sup>, J. Sik<sup>(6)</sup> and M. Lorenc<sup>(6)</sup>

<sup>(1)</sup>Astronomical Institute, Academy of Sciences of the Czech Republic, 251 65 Ondrejov, Czech Republic,

Email:rhudec@asu.cas.cz

<sup>(2)</sup>Rigaku Innovative Technologies Europe, Prague, Czech Republic
<sup>(3)</sup>Czech Technical University, Faculty of Nuclear Sciences, Prague, Czech Republic
<sup>(5)</sup>Institute of Chemical Technology, Prague, Czech Republic
<sup>(6)</sup>ON Semiconductor Czech Republic

#### ABSTRACT

The future space X-ray astronomy imaging missions require very large collecting areas at still fine angular resolution and reasonable weight. The novel substrates for X-ray mirrors such as Silicon wafers and thin thermally formed glass enable wide applications of precise and very light weight (volume densities 2.3 to 2.5 gcm<sup>-3</sup>) optics. The recent status of novel technologies as well as developed test samples with emphasis on precise optical surfaces based on novel materials and their space applications is presented and discussed.

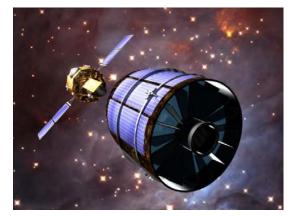
### 1. INTRODUCTION

Based on major discoveries of recent and previous Xray astronomy space missions, all major space agencies consider future missions with imaging X-ray telescopes onboard. Future large space X-ray telescopes (such as IXO/XEUS considered by ESA<sup>13</sup> or IXO/Constellation X by NASA) require precise and light-weight X-ray optics based on numerous thin reflecting shells. Novel approaches and advanced technologies are to be exploited and developed. In this contribution, we refer on results of test X-ray mirror shells produced by glass thermal forming (GTF) and by shaping Si wafers. Both glass foils and Si wafers are commercially available, have excellent surface microroughness of few 0.1 nm, and low weight (the volume density is 2.5 g  $\text{cm}^{-3}$  for glass and 2.3 g cm<sup>-3</sup> for Si). Technologies are to be exploited how to shape these substrates to achieve the required precise X-ray optics geometries without degradations of the fine surface microroughness. Although glass and recently silicon wafers are considered to represent most promising materials for future advanced large aperture space X-ray telescopes, there exist also other alternative materials worth further study such as amorphous metals and glassy carbon<sup>16</sup>. In order to achieve sub-arsec angular resolutions, principles of active optics are to be adopted.

## 2. THE THIN GLASS TECHNOLOGY

The glass technology belongs to one of most promising ones, as the volume density of glass is nearly four times less if compared with electroformed nickel layers. The glass foils may be used either as flats, or alternatively may be shaped or thermally slumped to achieve the required geometry. The thermal forming of glass is not a new technology since it has been used in various regions of glass industry and glass art as well as in the production of Cerenkov mirrors. However, the application of this technology in X-ray optics is related with the need to significantly improve the accuracy and minimize the errors. As the first step, small (various sizes typically less than 100 x 100 mm) glass samples of various types provided by various manufacturers have been used and thermally shaped. The geometry was either flat or curved (cylindrical or parabolic). The project continued with larger samples (up to 300 x 300 mm) and further profiles. The recent efforts focus on optimization of related parameters of both glass material and substrates as well as of the slumping process.

Fig 1. One of the designs of the ESA XEUS X-ray observatory.



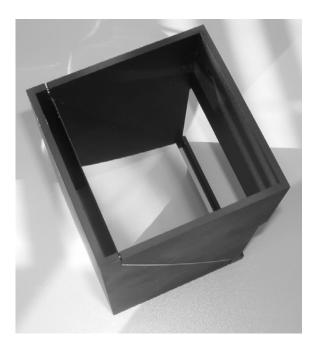


Fig. 2. Thermally formed parabola (gold-coated glass 150 x 100 x 0.75 mm).

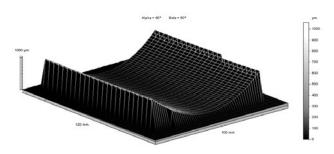


Fig. 3. Thermally formed glass sheet, 100 x 150 x 0.75 mm parabola, Still optical profilometer 3D plot.

The glass samples were thermally formed at Reflex, Prague, as well as at the Institute of Chemical Technology in Prague. For large samples (300 x 300 mm), facilities at Optical Development Workshop in Turnov have been used. Our strategy is to develop technology suitable for mass and inexpensive production of thin X-ray optics shells i.e. to avoid expensive mandrels and techniques not suitable for mass production or being too expensive. Numerous glass samples have been shaped and tested. The shapes and profiles of both mandrels as well as the resulting glass replicas have been carefully measured by metrology devices. The preliminary results show that the quality of the thermal glass replica can be significantly improved by the optimisation of the material and design of the mandrel, by the modification of the thermal forming process, as well as by the optimisation of the temperature. After the (partly significant) modifications and improvements we have obtained the resulting deviation of the thermally formed glass foil from the ideal designed profile less than 1 micrometer (peak to valley value) in the best case. This value is however strongly dependent on the exact temperature, so we believe that a further improvements are still possible. The fine original microroughnes (typically better than 1 nm) of the original float glass foil has found not to be degraded by the thermal forming process. We note that our approach in thermal glass forming is different from those used by another authors<sup>17, 19</sup> aaa. The recent efforts was devoted to the optimization of the whole process, using and comparing different forming strategies etc., as the final goal is to further improve the forming accuracy to less than 0.1 micron values. For near future, we plan continuation of these efforts together with investigations of computer-controlled forming of glass foils (according to the principles of active optics).

# 3. THE SILICON WAFERS TECHNOLOGY

Another alternative recently considered as one of most promising<sup>14, 15</sup>, is the use of X-ray optics based on commercially available silicon wafers manufactured mainly for purposes of semiconductor industry. Silicon is relatively light (volume density 2.3 g cm<sup>-3</sup>) and already during the manufacture process it is lapped and polished (either on one or on both sides) to very fine smoothness (better than few 0.1 nm) and thickness homogeneity (of the order of 1 micrometer).

The main preferences of the application of Si wafers in space X-ray optics are (i) the low volume density (2.3 g cm<sup>-3</sup>) which is more than 4x less than the electroformed nickel used in the past for galvanoplastic replication of multiply nested X-ray mirrors and slightly less than alternative approach of glass foils, (ii) very high thickness homogeneity typically less than 1 micron over 100 mm, and (iii) very small surface microroughness either on one or on both sides (typically of order of few 0.1 nm or even less).

Silicon wafers were expected to be used in the ESA XEUS project and are still considered for the IXO project. The recent baseline optics for the IXO X-ray telescope design is based on X-Ray High precision Pore Optics (X-HPO), a technology currently under development with ESA funding (RD-Opt, RD-HPO), in view of achieving large effective areas with low mass, reduce telescope length, high stiffness, and a monolithic structure, favoured to handle the thermal environment and simplify the alignment process<sup>17</sup>. In

addition, due to the higher packing density and the associated shorter mirrors required, the conical approximation to the Wolter-I geometry becomes possible. The X-HPO optics is based on ribbed Silicon wafers stacked together. The forming of the Si wafers to achieve the conical approximation is achieved by stacking large number of plates together using a mandrel. The typical size of the used Si wafers is  $10 \times 10 \text{ cm}^{17}$ .

There exist also alternative X-ray optics arrangements with use of Si wafers. In this contribution we refer on the development of the alternative design of innovative precise X-ray optics based on Silicon wafers. Our approach is based on two steps, namely (i) on development if dedicated Si wafers with properties optimised for the use in space X-ray telescopes and (ii) on precise shaping the wafers to optical surfaces. The stacking to achieve nested arrays is performed after the wafers have been shaped. This means, that in this approach the Multi Foil Optics (MFO) is created from shaped Si wafers. For more details on MFO see Hudec et al. 2005<sup>16</sup>. This alternative approach does not require ribbed surface of used Si wafers, hence the problems with transferring any deviation, stress, and/or inaccuracy from one wafer to the neighbouring plates or even to whole stacked assembly will be avoided. On the other hand, suitable technologies for precise stacking of optically formed wafers to multiple array has to be developed.

It is important to note that the Si wafers available on the market are designed for the use mainly in the semiconductor industry. It is obvious that the requirements of this industry are not the same as the requirements of precise space X-ray optics. The Si wafers represent a monocrystal (single crystal) with some specifics and this must be also taken into account. Moreover, the Si wafers are fragile and their precise bending and/or shaping is very difficult (for thicknesses required for X-ray telescopes i.e. around 0.3 - 1.0 mm; the exception represent the thinned Si wafers with thickness below 0.1 mm but these can be hardly used in this type of X-ray optics because of diffraction limits). Also, while their thickness homogeneity is mostly perfect, the same is not valid for commercially available wafers for their flatness (note that we mean here the deviation of the upper surface of a free standing Si wafer from an ideal plane, while in the semiconductor community usually flatness is represented by a set of parameters).

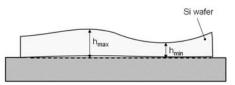
We conclude that in order the achieve the very high accuracy required by future large space X-ray telescope experiments like ESA/NASA/JAXA IXO, the parameters of the Si wafers are to be optimized (for application in X-ray optics) already at the production stage. This is why we have established and developed a multidisciplinary working group including specialists from the development department of Si wafer industry with the goal to design and manufacture Si wafers with improved parameters (mostly flatness) optimized for application in X-ray telescopes. It should be noted that the manufacture of silicon wafers is a complicated process with numerous technological steps and with many free parameters, which can be modified and optimised to achieve the optimal performance. This can be useful also to further improve the quality of the X-HPO optics.

As we deal with high-quality X-ray imaging, the smoothness of the reflecting surface is important. The standard microroughness of commercially available (we have used the products of ON Semiconductor Czech Republic) is of order of 0.1 nm as confirmed by several in dependent measurements by different techniques including the Atomic Force Microscope (AFM). This is related to the method of chemical polishing used during the manufacture of Si wafers. The microroughness of Si wafers exceeds the microroughness of glass foils and most of other alternative mirror materials and substrates.

The flatness (in the sense of the deviation of the upper surface of a free standing Si wafer from a plane) of commercially available Si wafers was however found not to be optimal for use in high-quality (order of arces angular resolutions) X-ray optics. The most of Si wafers show deviations from the plane of order of few tens of microns. After modifying the technology process during the Si wafer manufacture, we were able to reduce this value to less than few microns. Also the thickness homogeneity was improved. In collaboration with the manufacturer, further steps are planned to improve the flatness (deviation from an ideal plane) and the thickness homogeneity of Si wafers. These and planned improvements introduced at the stage of the Si wafers manufacture can be applied also for other design of Si wafer optics including the X-HPO, and can play a crucial role in the IXO project.

The X-ray optics design for IXO is based on Wolter 1 arrangement and hence requires curved surfaces. However, due to the material properties of monocrystalline Si, the Si wafers (except very thin wafers) are extremely difficult to shape. It is obvious that we have to overcome this problem in order to achieve the fine accuracy and stability required by future large X-ray telescopes. The final goal is to provide optically shaped Si wafers with no or little internal stress. Three various alternative technologies to shape Si wafers have been designed and tested to achieve precise optical surfaces. The samples shaped and tested were typically 100 to 150 mm large, typically 0.6 to 1.3 mm thick, and were bent to either cylindrical or parabolic test surfaces. One method (technology I) is the method of plastic deformation of monocrystalline Si at high temperature i.e. thermal shaping in analogy to the thermal shaping (slumping) procedure applied for glass X-ray optics<sup>16</sup>. This

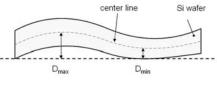
requires high temperature (typically more than 1000°C) as well as special atmosphere during the forming to avoid the surface degradation of the wafer and of the mandrel. The two alternative technologies (technology II and III) rely on physical and chemical processes, at this stage proprietary, and have also lead to test samples shaped to precise optical surfaces.



TIR = h<sub>max</sub> - h<sub>min</sub>



 $TTV = T_{max} - T_{min}$ 



Warp =  $(D_{max} - D_{min}) / 2$ 

Fig. 4: Definition of silicon wafer flatness: TTV –Total Thickness Variation, TIR – Total Indicator Reading, WARP - difference between the maximum and minimum deviations of a wafer's median surface with respect to a reference plane.

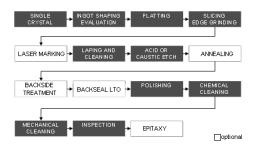


Fig. 5: Basic chart of silicon wafer manufacturing flow.

The development described here is based on a scientific approach and hence the large number of samples formed with different parameters must be precisely measured and in detail investigated. Especially precise metrology and measurements play a crucial role in this type of experiment. The samples of

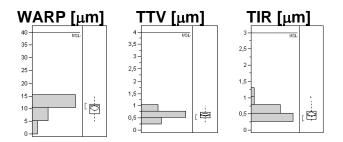


Fig. 6: Measured distributions of flatness parameters for 24 silicon wafers. Upper specification limit (USL) for semiconductor application. Wafers were manufactured with the method for high flatness.

bent wafers with all three technologies have been measured including Taylor-Hobson mechanical and STILL optical profilometry as well as optical interferometry (ZYGO) and AFM analyses. It has been confirmed that all three technologies studied does not degrades the intrinsic fine microroughness of the wafer. While the two physical/chemical technologies exploited give peak to valley deviations (of real surface of the sample compared with ideal optical surface) of less than 1 to 2 microns over 150 mm sample length, as preliminary values, the deviations of first thermally bent sample are larger, of order of 10 microns). Taking into account that the applied temperatures as well as other parameters were not optimised for this first sample, we expect that the PV value can be further reduced down to order of 1 micron and perhaps even below. Fine adjustments of parameters can however further improve the accuracy of the results also for the other two techniques.

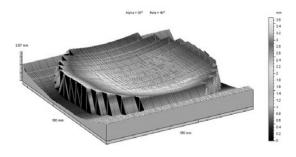


Fig. 7. 3D optical profilometry (STILL) of shaped (by technology II) Si wafer (R=1650 mm,D=150 mm, thickness 1.3 mm). Measured area  $1.4 \times 1.1$  mm, PV 2.8 nm, Rz 2.0 nm, RMS 0.2 nm, Ra 0.2 nm.

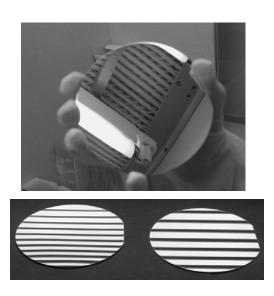


Fig. 8. Up: optically formed (technology II) Si wafer, diameter 100 mm, thickness 0.8 mm. Down: Si wafer (D = 150 mm, 1.3 mm thick) - flat (right) and optically bent (left).

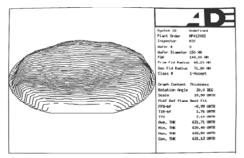


Fig. 9: Example of flatness measurement of standard 150 mm silicon wafer used for technologies with photolithographic detail ~ 5  $\mu$ m. Measured in 1275 points with ADE 7000 Wafercheck. Thickness in the wafer center (Cen. THK) 631.13  $\mu$ m, minimal measured thickness: (Min. THK) 630.40  $\mu$ m, maximal measured thickness (Max. THK) 632.50  $\mu$ m; Total thickness variation: TTV = (Max. THK) – (Min. THK) = 2.10  $\mu$ m; TIR: 1.76  $\mu$ m.

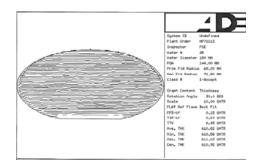


Fig. 10: Example of flatness measurement of 150 mm Si wafer developed for sub-micron technologies in ON Semiconductor. Measured in 1275 points with ADE 7000 Wafercheck. Thickness in the wafer center (Cen. THK) 610.92  $\mu$ m, minimal measured thickness (Min. THK) 610.58  $\mu$ m, maximal measured thickness (Max. THK) 611.03  $\mu$ m. Total thickness variation: TTV = (Max. THK) – (Min. THK) = 0.45  $\mu$ m; TIR: 0.29  $\mu$ m.

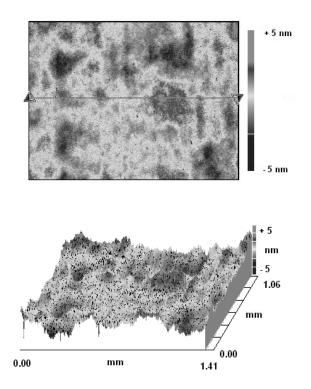


Fig. 11: Optically formed (technology II) Si wafer measurement by Zygo interferometer (2D and 3D images, measured area 1.4 x 1.1 mm, PV = 0.04 microns, RMS= 1.1 nm, Ra=0.9 nm).

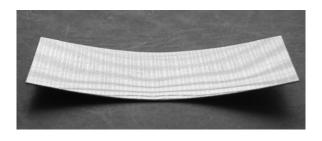


Fig. 12. Thermally formed (technology I) Si wafer to test cylinder (R = 150 mm, 72 x 23 x 0.625 mm).

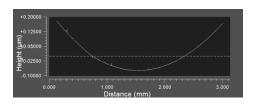


Fig. 13: Parabolized Si wafer, D=100 mm, ZYGO measurement, profile (technology III).



Fig. 14. Test module for tests performance of glass foils vs. shaped Si wafers. Test elliptical Kirkpatrick-Baez optical system, focus 0.5 m,  $58 \times 50 \times 100 \text{ mm}$ , glass foils  $40 \times 40 \times 0.3 \text{ mm}$ , Si wafers  $40 \times 40 \times 0.4 \text{ mm}$ .



Fig. 15: Test parabolic KB X-ray imaging system based on parabolically shaped glass foils with dimensions of  $300 \times 100 \times 0.3$  mm. The complete KB

module has dimensions  $108 \times 320 \times 50$  mm and the focal length is 0.6 m.

### 4. CONCLUSIONS

Two promising technologies suitable for future largeaperture and fine resolution X-ray telescopes were in detail exploited and investigated, namely the Glass Thermal Forming and Si wafer bending, In both cases, promising results have been achieved, with peak to valley deviations of final profiles from the ideal ones being of order of 1 micron in the best cases, with space for further essential improvements and optimization. An interdisciplinary co-operation (team with 10 members) is operated within the Czech Republic with experienced teams including researches from the large company producing Si wafers. Si wafers were

successfully bent to desired geometry by three different techniques. In the best cases, the accuracy achieved for the 150 mm Si wafer is 1 to 2 microns PV for deviation from the ideal optical surface. The experiments continue to further improve the forming accuracy.



Fig. 16: Optical test of experimental KB system (dimension – 108 mm x 320 mm x 50 mm, glass foils – 100 mm x 300 mm, focus 0.6 m).

## ACKNOWLEDGEMENTS

We acknowledge the support provided by the Grant Agency of the Academy of Science of the Czech Republic, grant IAAX01220701, by the Ministry of Education and Youth of the Czech Republic, projects ME918 and ME911 and by Ministry of Industry and Trade of the Czech Republic, FT-TA3/112. The investigations related to the ESA XEUS project are supported by the ESA PECS Project No. 98038. M.S. acknowledges the support by the junior grant by the Grant Agency of the Czech Republic, grant 202/07/P510. This study of radiation resistance was also a part of the research programme MSM 6046137302 Preparation and research of functional materials and material technologies using micro- and nanoscopic methods. We also acknowledge the support from the Institute of Plasma Physics of the Academy of Sciences of the Czech Republic in Prague, and from the Physical institute of the Academy of Sciences in Prague. We also acknowledge collaboration with Centre for Advanced Instrumentation, Netpark Research Institute, Sedgefield, United Kingdom, in metrology and measurements of some of the samples and the Institute of Condensed Matter Physic, Masaryk University Brno, Czech Republic, for AFM measurements of silicon wafers surfaces

## REFERENCES

- 1. Gorenstein, P. et al., SPIE Vol. 2805, 74, 1996.
- 2. Gorenstein, P., SPIE Vol. 3444, 382, 1998.
- 3. Hudec R. et al., SPIE Vol. 1343, 162, 1991.
- 4. Hudec R., Pina L and Inneman A., SPIE Vol. **3766**, 62, 1999.
- Hudec R., Pina L. and Inneman A., SPIE Vol. 4012, 422, 2000.
- Hudec R., Inneman A. and Pina L., "Lobster-Eye: Novel X-ray Telescopes for the 21st Century", New Century of X-ray Astronomy, ASP Conference Proceedings Vol. 251. Edited by H. Inoue and H. Kunieda. ISBN: 1-58381-091-9. San Francisco: Astronomical Society of the Pacific, p.542, 2001.
- 7. Inneman A., Hudec R., Pina L. and Gorenstein P., SPIE Vol. **3766**, 72, 1999.
- Inneman A., Hudec R. and Pina L., SPIE Vol. 4138, 94, 2000.
- 9. Joensen K. et al., SPIE Vol. 2279, 180, 1994.

- 10. Citterio O. et al., SPIE Vol. 4496, 23, 2002.
- Marsch H. et al., Introduction to Carbon Technologies, University of Alicante, ISBN :84-7098-317-4, 1997.
- 12. Ivan, A. et al., SPIE Vol. 4496, 134, 2002.
- 13. Aschenbach B. et al., ESA-SP 1253, 2001.
- 14. Bavdaz, M. et al., SPIE Proc. 5539, 85B, 2004.
- 15. Beijersbergen, M. et al., SPIE Proc. **5539**, 104B, 2004.
- Hudec R. et al. Optics for EUV, X-Ray, and Gamma-Ray Astronomy II. Edited by Citterio, Oberto; O'Dell, Stephen L. Proceedings of the SPIE, Volume **5900**, pp. 276-287, 2005.
- Ghigo, M. et al. Optics for EUV, X-Ray, and Gamma-Ray Astronomy. Edited by Citterio, Oberto; O'Dell, Stephen L. Proceedings of the SPIE, Volume **5168**, pp. 180-195, 2004.
- 18. Parmar A. et al., SCI-A/2006/054/NR.
- Friedrich, P. et al. Optics for EUV, X-Ray, and Gamma-Ray Astronomy II. Edited by Citterio, Oberto; O'Dell, Stephen L. Proceedings of the SPIE, Volume **5900**, pp. 258-265, 2005.