Extremely Lightweight X-Ray Optics Based on Thin Substrates

R. Hudec[a,c], J. Sik[f], M. Lorenc[f], L. Pina[g], V. Marsikova[b], M. Mika[e], A. Inneman[b] , and M. Skulinova[a]

[a]Astronomical Institute, Academy of Sciences of the Czech Republic, 251 65 Ondrejov, Czech Republic
[b]Rigaku Innovative Technologies Europe - RITE, Prague, Czech Republic
[c]Czech Technical University, Faculty of Electrical Engineering, Prague, Czech Republic
[e]Institute of Chemical Technology, Prague, Czech Republic
[f]ON Semiconductor Czech Republic
[g]Czech Technical University, Faculty of Nuclear Science, Prague, Czech Republic

ABSTRACT

We report on recent progress with development of astronomical X-ray optics based on bent Si wafers. Recent efforts with Si wafers have been focused on new forming technologies such as method of deposition of thin layers. The role of substrates quality in performance of final mirror arrays, as required by large future space X-ray astronomy experiments was also studied.

1. INTRODUCTION

Future large X-ray telescopes (such as ATHENA, and formerly IXO considered by ESA[3]) require precise and light-weight X-ray optics. Novel approaches and technologies are hence to be exploited. As the weight issue plays an important role and at the same time the large collecting area must be achieved in order to get deep sensitivity limits, the thin X-ray reflecting flats and foils play an increasingly important role in future experiments in X-ray astrophysics. They have opened a new space for various novel approaches and innovative solutions including those never discussed before. The most important use of innovative X-ray reflecting foils and flats is in the future large aperture and high sensitivity X-ray imaging experiments. The Wolter 1 telescope is proposed to be segmented in the ATHENA telescope and analogous space projects, but considerations also exist for large Lobster eye segmented modules, as well as for the segmented Kirkpatrick-Baez systems. The segmentation of the mirror surfaces is extremely important not only for the production of mirror shells, but also for the keeping the weight of large telescopes in a still reasonable limits[2].

In this contribution, we refer on preliminary results of continued efforts to design, to develop, and to test X-ray mirrors produced by precise shaping of silicon wafers. Innovative 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 micro-roughness. It should be noted that although glass and more 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[16].

Silicon wafers are produced mainly for the customers in semiconductor industry by a mass-production. This means that the Si wafers on the market are optimized for the semiconductor industry use, not for the X-ray optics applications. Our investigations have indicated that while the surface micro-roughness is (as a result of chemical polishing) mostly fully adequate for use in X-ray telescopes, there is still chance to modify the manufacturing technology in order to achieve better values of other important parameters such as flatness and thickness homogeneity. In Section 2 of this paper we present some preliminary results of these efforts which can be valuable also for experiments with active X-ray optics.

2. SILICON WAFERS AS X-RAY OPTICS SUBSTRATES

The alternative recently considered as one of most promising[14, 15], is the use of X-ray optics based on commercially available silicon wafers manufactured mainly for purposes of semiconductor industry. Silicon is relatively light and already during the manufacturing process it is lapped and polished (either on one or on both sides) to very fine smoothness and thickness homogeneity (of the order of 1 µm).

The original baseline optics for the IXO (now ATHENA) X-ray telescope design suggested by ESA was based on X-ray High precision Pore Optics (X-HPO), a technology developed 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, favored to handle the thermal environment and simplify the alignment process[17]. 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 chip is 100 mm x 100 mm[17].

In this and previous papers[20, 21] we refer on the development of alternative design of innovative precise X-ray optics based on silicon wafers. Our approach is based on two steps, namely (i) on development of dedicated Si wafers with properties optimized 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)[16].

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 neighboring 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 arrays have to be developed. The recent 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 main preferences of the application of Si wafers in space X-ray optics are (i) the low volume density which is nearly 3.5x 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 µm over 100 mm, and (iii) very small surface micro-roughness either on one or on both sides. On the other hand, the Si wafers represent a single crystal with some specifics and this must be taken into account. Moreover, the 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 represents the thinned Si wafers with thickness below 0.1 mm). 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).

It is obvious that in order the achieve the very high accuracy required by future large space X-ray telescope experiments like ATHENA, 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 a multidisciplinary working group including specialists from the development department of Si wafer industry with the goal to design and manufacture Si wafers optimized for application in X-ray telescopes. The production of silicon wafers is a complex process with numerous technological steps, which can be modified and optimized to achieve the optimal performance. This can be useful also to further improve the quality of the X-HPO optics.

The standard micro-roughness of commercially available Si-wafers (we have used the products of ON Semiconductor Czech Republic) is of order of 0.1 nm as confirmed by several independent measurements by different techniques including the Atomic Force Microscope (AFM). This is related to the method of chemical-mechanical polishing used during the manufacture of Si wafers and meets the requirements of future X-ray telescopes well. In fact, the micro-roughness of Si wafers exceeds significantly the micro-roughness of glass foils and most of other alternative mirror materials and substrates.

However, 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 found not to be optimal for use in X-ray optics. The most of 100 mm and 150 mm Si wafers used for technologies with photolithographic detail ~ 5 µm show deviations from the plane of order of few tens of microns and thickness uniformity of few microns. For more advanced technologies with sub-micron details, flatness parameters have to be improved. ON Semiconductor Czech Republic has developed technological process for higher wafer flatness and further steps are planned to improve the deviation from an ideal plane and the thickness uniformity. As already mentioned, these and planned improvements introduced at the stage of the Si wafers production can be applied also for other design of Si wafer optics including the X-HPO.

2.1 Development of raw Si wafers technology

In addition to the results achieved before [20, 21], further investigation has been performed with emphasis of various aspects of quality improvements necessary for superior quality of X-ray optics based on shaped Si wafers.

As the silicon substrates are used in X-ray optics modules i.e. shaped to X-ray mirrors of Wolter, or alternatively, the Kirkpatrick-Baez geometry, one of important parameters to be improved is the flatness. As the definition of flatness used in the semiconductor industry differs from analogous definition used in physical science, we illustrate the definitions of two basic parameters used in semiconductor industry namely TTV and WARP (Fig. 1).

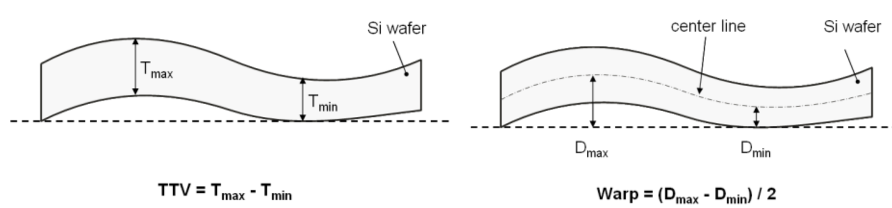

Fig. 1: Definition of silicon wafer flatness: TTV –Total Thickness Variation, WARP - difference between the maximum and minimum deviations of a wafer's median surface with respect to a reference plane.

The geometrical quality of a standard 150 mm Si wafer used for technologies with photolithographic detail ~ 5 µm is illustrated on Fig. 3. For comparison wafer for sub-micron photolithographic detail produced in ON Semiconductor Czech Republic is in Fig. 3. The measurements of 24 silicon wafers flatness, as well as upper specification limit (USL) for semiconductor, flatness not demanding application, are plotted in Fig. 4. All these wafers were manufactured by ON Semiconductor Czech Republic with novel method for high flatness.

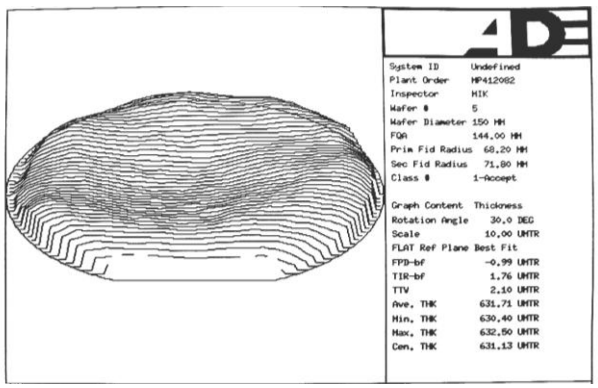

Fig. 2: Example of flatness measurement of standard 150 mm silicon wafer used for technologies with photolithographic detail ~ 5 µm. Measured in 1275 points with ADE 7000 Wafercheck. Thickness in the wafer center (Cen. THK) 631.13 µm, minimal measured thickness: (Min. THK) 630.40 µm, maximal measured thickness (Max. THK) 632.50 µm; Total thickness variation: TTV = (Max. THK) – (Min. THK) = 2.10 µm.

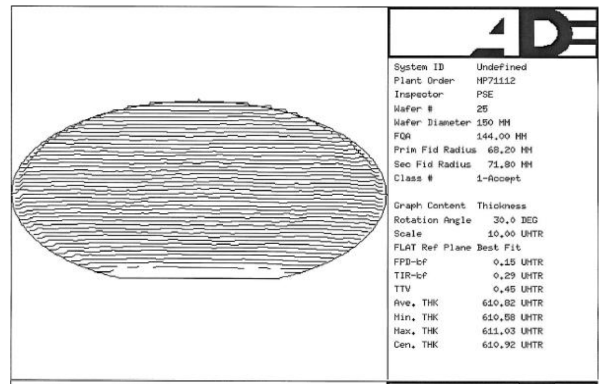

Fig. 3: Example of flatness measurement of 150 mm silicon wafer developed for sub-micron technologies in ON Semiconductor. Measured in 1275 points with ADE 7000 Wafercheck. Thickness in the wafer center (Cen. THK) 610.92 µm, minimal measured thickness (Min. THK) 610.58 µm, maximal measured thickness (Max. THK) 611.03 µm. Total thickness variation: TTV = (Max. THK) – (Min. THK) = 0.45 µm.

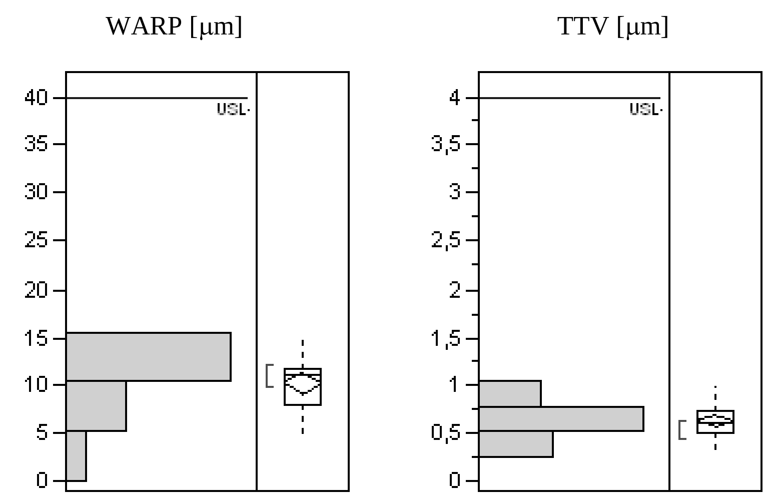

Fig. 4: Measured distributions of flatness parameters for 24 silicon wafers. Upper specification limit (USL) for semiconductor application. Wafers were manufactured with the method for high flatness.

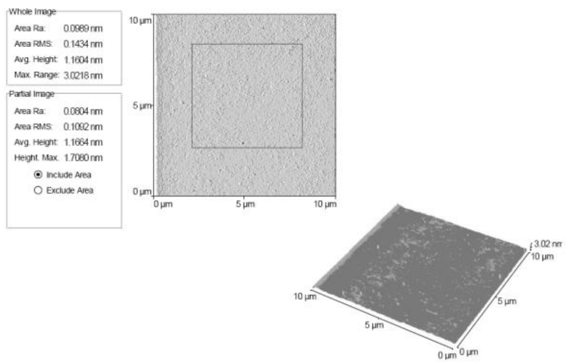

Fig. 5: Surface roughness of polished silicon wafer measured with AFM. Crystallographic orientation (100), FZ wafer slightly doped with boron. Measured area 10 µm x 10 µm, Ra = 0.10 nm, RMS = 0.14 nm.

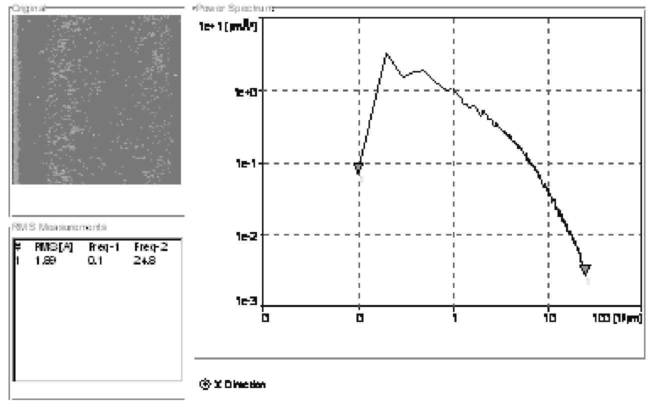

Fig. 6: Power Spectral Density (PSD) function calculated for data from AFM microscopy (

Fig. 5).

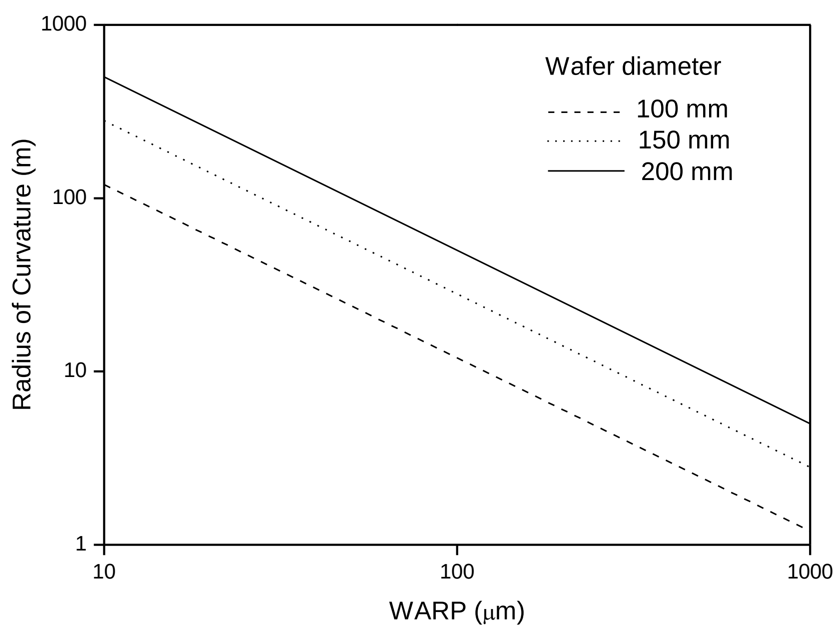

Fig. 7: The dependence of curvature radius on WARP. See Fig. 1 for definition of WARP.

Another important parameter for applications in fine X-ray imaging is the surface micro-roughness. In

Fig. 5, Fig. 6 we show examples of results obtained by AFM microscopy, as well as relevant PSD (Power Spectral Density) plot. These results confirm the superior surface micro-roughness of polished Si wafers meeting the requirements of X-ray optics applications.

Based on our analysis and early investigations, the crystallographic orientation (100) is the optimal solution due to superior surface quality. Values of RMS for polished surface (100) can be as low as ~ 0.1 nm (measured with AFM). There are basically two limitations – (i) defects and particles on the surface and (ii) atomic steps on the surface. Surface with crystallographic orientation (111) is characteristic by high density of atoms on the wafer surface and atomic steps of the high 0.314 nm. The distance between neighboring steps is determined by surface deviation from (111) plane (off orientation). For 1 deg deviation is this distance ~ µm, with decreasing angle of off orientation this distance is increasing. On the single terrace the AFM roughness 0.06 nm was measured[32]. For (100) plane distance between atomic planes is in relation to silicon lattice parameter: 0.543 nm/4 = 0.135 nm. It is also necessary to remind that silicon surface has thin layer of native oxide on the surface. This oxide can be up to few nanometers thick. Deeper study of silicon surface roughness and AFM capability for its measurement is in progress.

Best roughness values were achieved with optimized chemical-mechanical polishing of material with lower surface hardness, e.g., (100) float zone silicon or (100) heavily arsenic doped silicon. For float zone wafer are different mechanical properties given by low oxygen content. High oxygen and boron content in silicon suppress dislocation movement[29] contrary to heavily doped n-type silicon[30, 31].

Single side polished Si wafer represents potential substrate for the mirrors of space X-ray telescopes. Through Si wafers manufacturing technology optimization and for specified Si wafers specification (dopant, orientation, oxygen content) we can achieve thickness variation better than 0.5 µm and surface roughness close to 0.1 nm.

2.2 Shaping of Si wafers

Due to the material properties of monocrystalline Si, the Si wafers are extremely difficult to shape. However 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 superior quality and stability. Mechanical bending of Si wafers at room temperature on mandrel as considered by the X-HPO technique means no negligible internal stress, which can avoid achieving the required very long-term stability and very fine angular accuracy of order of few arcsecs.

Shaping of substrates for mirror geometry is possible, e.g., via methods of optical lithography or direct wafer bonding techniques used in semiconductor industry. We have designed and exploited three various alternative technologies to shape Si wafers to 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[16]. This requires very high temperature (typically more than 1 000°C) as well as special atmosphere during the forming to avoid the surface degradation of the wafer and of the mandrel. The another alternative technology proposed, developed, and tested rely on physical and chemical processes, at this stage proprietary, and have also lead to test samples shaped to precise optical surfaces[20, 21].

In this paper we refer on another proposed and tested technology, namely application of layers with internal stress, see Section 3.

The test samples of optically bent wafers with all three technologies have been carefully measured and tested. Preliminary results were presented and discussed in preceding papers[20,21] and more recent results in this paper. The measurements include Taylor-Hobson mechanical and STILL optical profilometer as well as optical interferometer (ZYGO) and AFM analyses. It has been confirmed that all three studied technologies does not degrade the intrinsic fine micro-roughness of the wafer. While 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 µm over 150 mm sample length, as preliminary values, the deviations of the first thermally bent sample are larger, of order of 10 µm). Taking into account that the applied temperatures as well as other parameters were not optimized for this first sample, we expect that the PV value can be further reduced down to order of 1 µm and perhaps even below. Fine adjustments of parameters can however further improve the accuracy of the results also for the other two techniques. For further details on shaping of Si wafers see our previous papers[20, 21].

3. APPLICATION OF LAYERS WITH INTERNAL STRESS FOR SILICON WAFERS SHAPING

Recently we have investigated the impact of thin film stress on silicon wafer shaping. Layers with internal stress uniformly shape silicon wafer without deterioration of high quality of the polished side. Stress in thin film is supposed to be constant regarding to the film thickness, which is valid for most of dielectric thin films used in microelectronics, except of poly-silicon. Stress in poly silicon layer is reduced with film thickness due to atoms migration into low energy position.

To get low curvature radius R we need to combine layers with high tensile stress on the front side and compressive stress on the back side. The best selection is LPCVD nitride on front side and poly Si on back side. Feasible layer thickness is 1500 nm for poly Si and 300 nm for LPCVD nitride. All process steps have to keep high surface quality of the polished front side.

Furthermore, we have investigated the behaviour of squared Si wafers, required for space X-ray optics modules. We have found that the circular wafer keeps the original axially symmetrical spherical shape after squaring and the solid area can be build from squared segments. Multilayer stack has been designed to decrease the radius of wafer curvature to R ~ 2 m.

We note this technology is suitable for active X-ray optics, as well as of optics with large bending radii such as Kirkpatrick/Baez X-ray optics.

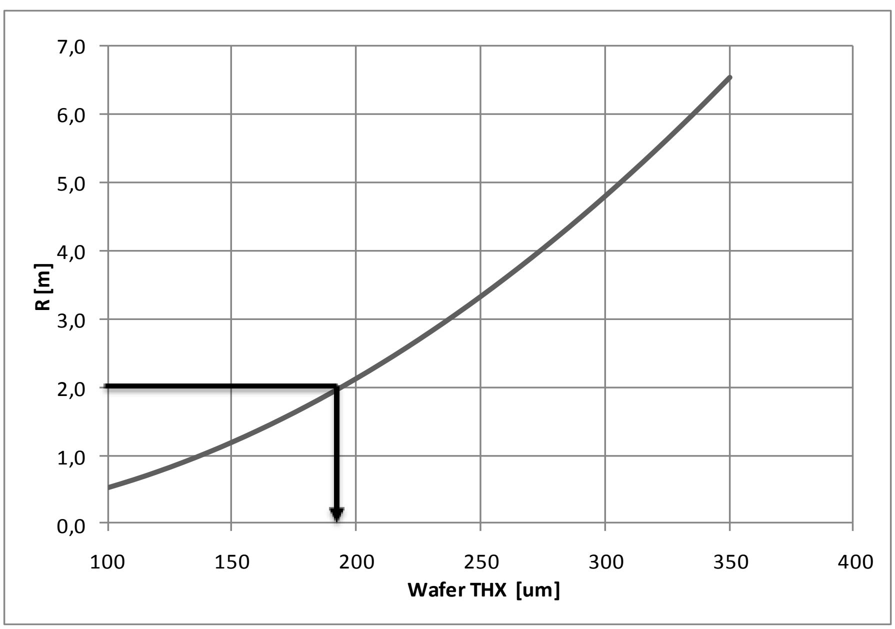

Fig. 8: The dependence of bending radius on wafer thickness.

For designed stack we can calculate the wafer thickness to achieve expected radius of curvature. As we can see in plot in Fig. 8 the wafer thickness 195 µm is needed for R ~ 2 m. For other than spherical shape photolithography has to be used. Suitable technology is available in semiconductor industry

4. CONCLUSIONS

New results in application of Si wafers in future space projects with X-ray optics have been presented and discussed. The Si wafer bending belongs to the most promising technologies for future large space X-ray telescopes such as ATHENA. The results obtained and discussed are valuable also for efforts with active X-ray optics. Development of Si wafers with improved parameters has been initiated, meeting better the requirements of high resolution X-ray optics and X-ray telescopes than the standard wafers.

Impact of thin film stress on wafer shaping has been reviewed. Layers with internal stress uniformly shape silicon wafer without deterioration of high quality of the polished front side. Stress in thin film is supposed to be constant regarding to the film thickness, which is valid for most of dielectric thin films used in microelectronics. The circular wafer keeps the original axially symmetrical spherical shape after squaring. The solid area can be build from squared segments. Multilayer stack has been designed to decrease the radius of wafer curvature to R ~ 2 m. The method is suitable for K-B X-ray optical systems.

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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.
5. Hudec R., Pina L. and Inneman A., SPIE Vol. **4012**, 422, 2000.
6. 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.
8. 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.
11. 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.
16. 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.
17. 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., X-ray Observatory, Study preparation activities status report, ESA SCI-A/2006/054/NR, 2006.

19. 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.
20. Hudec, R. et al., Proceedings of the SPIE, Novel x-ray optics with Si wafers and formed glass, in Space Telescopes and Instrumentation II: Ultraviolet to Gamma Ray. Edited by Turner, Martin J. L.; Hasinger, Günther. Proceedings of the SPIE, Volume 6266, pp. 62661H, 2006.
21. Hudec, R. et al., Novel technologies for x-ray multi-foil optics, in 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.
22. Silicon, Evolution and Future of Technology, eds. P. Siffert, E. Krimmel, Springer-Verlag, 2004.
23. La Pedus, M., Debate rages over 450-mm wafer fabs, 04/28/2006, www.eetimes.com, 2006.
24. Montgomery, J., 450mm by 2012: Between the lines of PR lingo, 05/13/2008, Solid State Technology, sst.pennnet.com, 2008.
25. Kuramoto, M., Super Silicon Initiative and Future Large Wafer Size Diameter, in Semiconductor Silicon, Electrochemical Society Proceedings, p. 163-175, 2002..
26. Watanabe, M., 450 mm Silicon: An Opportunity and Wafer Scaling, The Electrochemical Society Interface, Winter, 28-31, 2006.
27. Fischer, A., Kissinger, G., Load induced stresses and plastic deformation in 450 mm silicon wafers, Appl. Phys. Lett. 91, 111911, 2007.
28. Lammers, D., 'Big Four' Talking 450 With Tool Vendors, Semiconductor International, 10/30/2007, Reed Business Information, 2008.
29. Yonegava, I., Activities of dislocations in heavily impurity-doped Si, J. Phys.: Condens. Matter 12, 10065-10069, 2000.
30. Patel, J. R., and Freeland, P. E., Change of dislocation velocity with Fermi level in silicon, Physical Review Letters 18, No. 20, 833 -835, 1967.
31. Patel, J. R., Testardi, L. R., and Freeland, P. E., Electronic effects on dislocation velocities in heavily doped silicon, Phys. Rev. B 13 No. 8, 3548 – 3557, 1976.
32. www.ntmdt-tips.com/catalog/test_s/products/STEPP.html, 2008.
33. Lanoo, M., J. Bourgoin, J., Point defects in semiconductors, Springer-Verlag Berlin, Heidelberg, New York, 1981.
34. Frank, H. et al., Studium vlivu jaderného záření na elektrické vlastnosti křemíku, research report (in Czech), FJFI Praha, 1977.
35. Frank, H. et al., Výzkum fyzikálních vlastností Si diod pro dozimetrii rychlých neutronů, research report (in Czech), FJFI Praha, 1977.
36. Alam M. S. et al., The Atlas silicon pixel sensor, Nuclear Instruments and Methods in Physics Research A 456, 217 – 232, 2001.
37. Gorelov, I. et al., Electrical characteristics of silicon pixel detectors, Nuclear Instruments and Methods in Physics Research A 489, 202 – 217, 2002.