

In-Situ Monitoring the Thermal Forming of Glass and Si Foils for Space X-Ray Telescopes

L. Pina, M. Mika, R. Havlikova, M. Landova, L. Sveda, R. Hudec, V. Marsikova, A. Inneman

Abstract—We developed a non-contact method for the in-situ monitoring of the thermal forming of glass and Si foils to optimize the manufacture of mirrors for high-resolution space x-ray telescopes. Their construction requires precise and light-weight segmented optics with angular resolution better than 5 arcsec. We used 75x25 mm Desag D263 glass foils 0.75 mm thick and 0.6 mm thick Si foils. The glass foils were shaped by free slumping on a frame at viscosities in the range of $10^{9.3}$ - 10^{12} dPa·s, the Si foils by forced slumping above 1000°C. Using a Nikon D80 digital camera, we took snapshots of a foil's shape every 5 min during its isothermal heat treatment. The obtained results we can use for computer simulations. By comparing the measured and simulated data, we can more precisely define material properties of the foils and optimize the forming technology.

Keywords—Glass, in-situ monitoring, silicone, thermal forming, x-ray telescope

I. INTRODUCTION

NEW astronomical projects on space x-ray telescopes with higher resolutions require novel substrates and cost-effective technologies for the construction of their x-ray reflecting mirrors. The mirror is the most crucial part of the International X-Ray Observatory IXO developed by ESA, NASA, and JAXA. The mirrors must be light and precisely shaped to allow multiple nesting with angular resolution better than 5 arcsec. Promising materials for the mirrors are thin glass sheets [1, 2] or Si wafers [3-7]. They can be precisely shaped by thermal forming to fit the mostly used Wolter type-I geometry [8] while keeping their surface micro-roughness sufficiently low [9]. To achieve these demanding parameters, the forming technology requires further development and optimization. Therefore, we need very detailed information about material properties and their temperature dependence as well as about the dynamics of the thermal forming process. Such information can be collected by the in-situ monitoring of the forming process. The obtained data we can use for computer simulations and further optimization of the forming.

Authors thank for the support of the Grant agency of the Academy of Sciences of the Czech Republic "Material and X-Ray Optical Properties of Formed Silicon Monocrystals," IAXX01220701; and of the research program MSM 6046137302 "Preparation and research of functional materials and material technologies using micro- and nanoscopic methods".

L. Pina is with the Czech Technical University in Prague, Brehova 7, Prague 1, 115 19, Czech Republic (phone: +420 603 253 864; fax: +420 284 684 818; e-mail: ladislav.pina@fjfi.cvut.cz).

M. Mika is with the Institute of Chemical Technology, Technicka 5, Prague 6, 166 28, Czech Republic (e-mail: martin.mika@vscht.cz).

R. Havlikova is with the Czech Technical University in Prague, Brehova 7, Prague 1, 115 19, Czech Republic (e-mail: radka.havlikova@fjfi.cvut.cz).

II. EXPERIMENTAL

A. Glass Thermal Forming

To monitor free slumping of glass, we used borosilicate glass Desag D263 that is produced by Schott company. The micro-roughness of this glass is only few 0.1 nm, density is $2.51 \text{ g}\cdot\text{cm}^{-3}$, and the glass exhibits very good chemical durability. We monitored the slumping of glass foils with rectangular shape 75x25 mm that were 0.75 mm thick. Each foil was placed on two parallel corundum rods supporting it at edges. During the experiment, the foils were heated in an electric furnace from the room temperature up to soaking temperatures slightly above the glass transition temperature. The corresponding viscosities η were from $10^{12.0}$ to $10^{9.3}$ dPa·s. Figure 1 shows a glass foil placed in the oven and its shape development during heat treatment at $\eta = 10^{9.9}$ dPa·s after 20, 40, and 60 min.

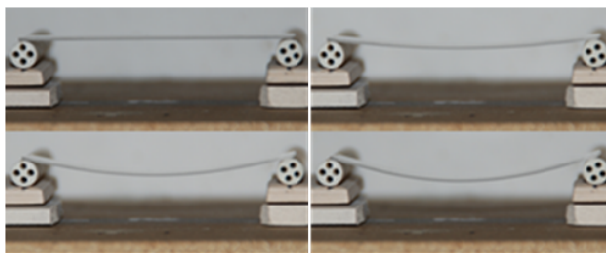


Fig. 1 Slumping of a glass foil at $\eta = 10^{9.9}$ dPa·s by heat treatment for 20, 40, and 60 min

B. Silicon Thermal Forming

For silicon thermal forming, we chose 0.6 mm thick Si monocrystal wafers doped with B. Their orientation was $\langle 100 \rangle$ and the micro-roughness of their surface was below 0.2 nm. The wafers were cut to 75x25 mm samples. In our experiments, we thermally shaped these Si foils by forced hot plastic deformation [10]. For achieving plastic deformations, we put our samples into an electric furnace and heated them up to temperatures above 1000°C. Simultaneously, we applied additional vertical force in the range from 1 to 5 N. The forming process was relatively slow, and to achieve sufficient bending in the range of 0.4-3 mm, it was necessary to keep the samples at constant heat-treatment temperatures from 60 to 120 min. These experiments will be continued with the aim of studying the thermal forming of Si monocrystals in detail, and to measure and verify all relevant material constants at high temperatures.

C. In-Situ Monitoring

For in-situ monitoring the changing shape of foils during thermal forming, we developed a non-contact method. During the heat treatment, we took snapshots of a foil inside the oven every 5 min. We used a Nikon D80 digital camera with an aspherical objective Tamron XR Di II AF 18-200mm. To detect and measure shape changes, we developed new software and automatically or semi-automatically processed the series of images with a computer including precise calibration. The final shape of the samples was measured with the Taylor Hobson PGI PLUS contact profilometer at a room temperature in order to obtain accurate data for fitting the shape and calibration. For each sample, we measured three parallel lines of orthogonal directions; in the center and close to the edges. Recorded data were processed with Taylor Hobson and Matlab software.

III. RESULTS

Taking snapshots of the foils during their isothermal forming enabled us to detailed monitoring the of their shape development. This non-contact optical method gave us possibility to study in-situ the dynamics of the thermal forming process. The obtained shapes were fitted by parabola, polynoms, or circle, and the differences between the theoretical and real shapes were calculated. After the forming, the final shape was measured by the profilometer. In Figure 2, the output from the profilometer shows the differences

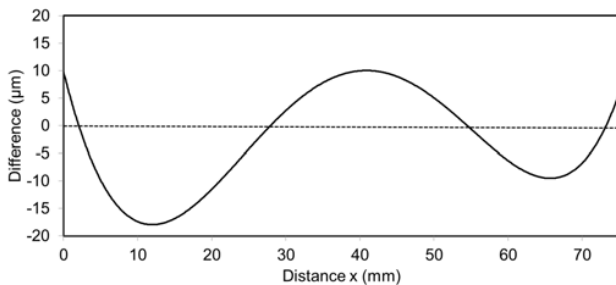


Fig. 2 Glass shape deviation from circle with radius 2033 mm

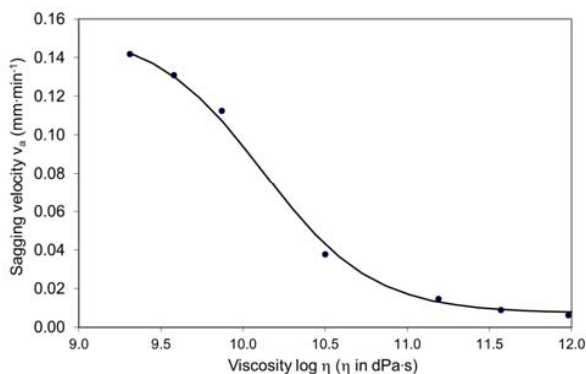


Fig. 3 Average sagging velocity as a function of glass viscosity

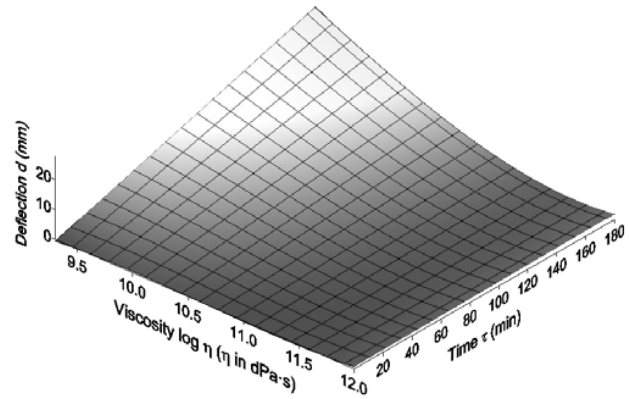


Fig. 4 Deflection as a function of viscosity and heat-treatment time

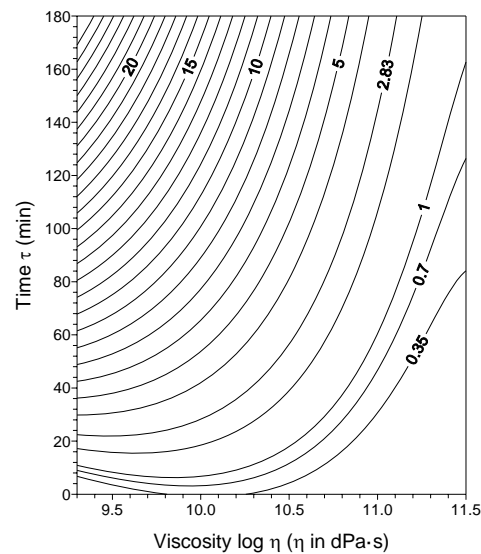


Fig. 5 Map of deflection as a function of viscosity and time

between the sample shape and the fitting circle of radius 2033 mm with peak-to-valley value $PV = 27 \mu\text{m}$.

For calculating the glass viscosity from temperature [11], we used (1):

$$\eta = 3.43 \cdot 10^{-2} \exp\left(\frac{1 \cdot 10^4}{t - 256.9}\right) \quad (1)$$

where η is the viscosity in dPa·s and t is the temperature in °C. We characterized the sagging process by using the value of deflection d in the middle part of the sample and the average sagging velocity v_a for the time interval 0-180 min. In Figure 3, the plot of v_a versus $\log \eta$ indicates a strong effect of the viscosity on sagging. To illustrate both the effect of η and heat-treatment time τ on d , we calculated a 3-D plot and a map shown in Figures 4 and 5. The map contains isolines of a constant d and where values 0.35, 0.70, and 2.83 represent deflections for radius 2, 1 and 0.25 m, respectively.

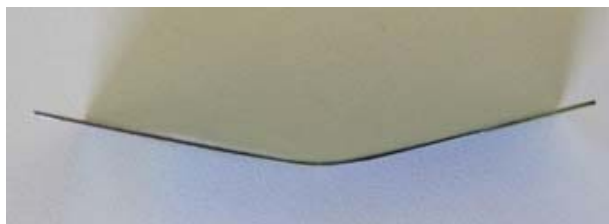


Fig. 6 Silicon foil shaped with a pressing mold of radius 10 mm



Fig. 7 Silicon foil shaped with a pressing mold of radius 75 mm

Successful shaping of silicon foils by hot plastic deformation confirmed again the feasibility of this process. To enable the deformation, our selected thin Si foils contained sufficient concentration of suitable dislocations. At temperatures above 1000°C the mobility of dislocations significantly increased and the application of vertical force facilitated plastic deformation of the samples. In Figures 6 and 7 you can compare two examples of silicon foils shaped with a pressing mold of radius 10 or 75 mm.

IV. DISCUSSION

By using the developed method for in-situ observations, we were able to study the dynamics of thermal forming processes. The free slumping of borosilicate glass foils Desag D263 was possible in the viscosity range from $10^{9.3}$ to $10^{12.0}$ dPa·s. The shapes of glass foils were parabolic but it was also possible to fit them with circles with peak peak-to-valley values typically between 25 to 35 μm . We still continue in our effort to decrease these values to the micron region by optimizing the forming process. The plastic deformation of the glass foils was strongly dependent on viscosity, which is evident from the dependence of the average sagging velocity. When the viscosity decreased from $10^{12.0}$ to $10^{9.3}$ dPa·s, the velocity increased about 10 times, and this increase was exponential for the viscosities from $10^{11.0}$ to $10^{9.3}$ dPa·s. For the viscosities below $10^{9.9}$ dPa·s the velocity increase became less steep, which could be caused by a higher friction and sticking of glass to the supporting corundum rods. The effects of both viscosity and heat-treatment time on glass sagging is clearly illustrated in the 3-D plot, where you can see that the deflection increased exponentially with increasing viscosity and linearly with increasing time. For $\eta < 10^{11.0}$ dPa·s, the relatively flat region steeply increases. This trend is also clearly illustrated in the map with the isolines of constant deflections. Using this map, we can easily determine the parameters of the heat-treatment to manufacture samples with required deflections. For example, to prepare samples of radius 0.25, 1, or 2 m, we need to achieve the deflections

$d = 0.35$, $d = 0.7$, or $d = 2.83$, respectively. These isolines can be represented by equations. For example, by using (2), we can calculate the time required to form samples of radius 1m as a function of viscosity:

$$\tau_1 = 22.927 \log \eta^3 - 664.53 \log \eta^2 + 6411.3 \log \eta - 20583 \quad (2)$$

where τ_1 represents the heat-treatment time in minutes needed to form a glass foil with radius 1 m. To effectively control and speed up the forming process, we consider the optimal heat-treatment time to be between 10 and 30 min. Hence, to form samples of radius from 0.25 to 2 m, the optimal viscosity must be between $10^{9.3}$ and $10^{11.0}$ dPa·s.

Our experiments demonstrated that thin silicon foils can be thermally formed when we reach the region of plastic deformation. It was much more difficult to achieve this type of deformation in silicon than in glass. Since the hot plastic deformation is controlled by dislocations, we need to use foils with sufficient concentrations of properly oriented dislocations. The dislocations move through the silicon foils in a viscous manner; therefore, we need to operate at sufficiently high temperatures to facilitate their transport through the substrate that is under additional vertical pressure. In our experiments, we had to heat the foils above 1000°C for more than 60 min. The radius of pressing mold significantly influenced the shape of silicon foil. The mold with bigger radius formed more circular shapes. Precise forming of silicon foils at such conditions turned to be a highly demanding process that was even more complicated, when we bent the foils not only in one direction but in two perpendicular directions.

A large number of data collected during our experiments was processed and has been used for the development of a numerical model to simulate the thermal forming process. The model has been developed in Comsol Multiphysics software with CFD module [12]. Comparing the measured and simulated data has helped us to more precisely determine material properties of the foils and their temperature dependence. This deeper insight has helped us to improve and optimize the forming; for example, by adding more thermocouples, by adjusting the geometry of the supporting frame, and by modifying heat-treatment conditions. Thus, we have been able to develop a more realistic numeric model for the computer simulation of the thermal forming process.

V. CONCLUSION

The developed non-contact optical method was suitable for in-situ monitoring of the thermal forming process. By using this method, we were able to study the process dynamics. The shaping of the glass and silicon foils was possible in the region of hot plastic deformation. Free slumping of the glass foils was strongly influenced by viscosity, and its shape could be fitted by parabola or circle with peak-to-valley values from 25 to 35 μm . We still work on improvements to make this thermal shaping of free-standing foils competitive to mandrel based thermal shaping. We calculated and plotted the relations between glass shape, viscosity, and heat-treatment time. To

form shapes with radius from 0.25 to 2 m, we consider the optimal heat-treatment time to be between 10 to 30 min, than the corresponding viscosity should be in the range of $10^{9.3}$ - $10^{11.0}$ dPa.s. Forced thermal slumping can be used for the shaping of thin silicon foils. The foils must contain sufficient concentration of properly oriented dislocations to undergo hot plastic deformation at temperatures above 1000°C. The experimental data obtained from the in-situ monitoring has been used for developing a numerical model that has been applied in the simulation of the slumping process. The monitoring of slumping gave us detailed information about the material properties of the foils helping us to better understand and control the thermal forming process.

REFERENCES

- [1] B. Luo, F. E. Bauer, W. N. Brandt, D. M. Alexander, B. D. Lehmer, D. P. Schneider, M. Brusa, A. Comastri, A. C. Fabian, A. Finoguenov, R. Gilli, G. Hasinger, A. E. Hornschemeier, A. Koekemoer, V. Mainieri, M. Paolillo, P. Rosati, O. Shemmer, J. D. Silverman, I. Smail, A. T. Steffen, and C. Vignali, "The Chandra Deep Field-South survey: 2 Ms source catalogs," *ApJS*, vol. 179, pp. 19-36, 2008.
- [2] W. W. Zhang, M. Atanassova, P. Augustin, P. N. Blake, G. Byron, T. Carnahan, K. W. Chan, C. Fleetwood, C. He, M. D. Hill, M. Hong, L. Kolos, J. P. Lehan, J. R. Mazzarella, R. McClelland, L. Olsen, R. Petre, D. Robinson, R. Russell, T. T. Saha, M. Sharpe, M. V. Gubarev, W. D. Jones, S. L. O'Dell, W. Davis, D. R. Caldwell, M. Freeman, W. A. Podgorski, and P. B. Reid, "Mirror technology development for the International X-ray Observatory mission," *Proc. SPIE*, vol. 7437, 0Q:1-12, 2009.
- [3] M. Ghigo, S. Basso, M. Bavdaz, P. Conconi, O. Citterio, M. Civitani, P. Friedrich, D. Gallieni, B. Guldemann, F. Martelli, R. Negri, G. Pagano, G. Pareschi, G. Parodi, L. Proserpio, B. Salmaso, F. Scaglione, D. Spiga, G. Tagliaferri, L. Terzi, M. Tintori, M. Vongehr, E. Wille, A. Winter and A. Zambra, "Hot slumping glass technology for the grazing incidence optics of future missions with particular reference to IXO," *Proc. SPIE*, vol. 7732, pp.0C:1-12, 2010.
- [4] Y. Ezoe, T. Shirata, T. Ohashi, M. Ishida, K. Mitsuda, K. Fujiwara, K. Morishita and K. Nakajima, "Development of high-resolution and light-weight x-ray optics with deformed silicon wafers," *Proc. SPIE*, vol. 7360, pp. 0B:1-8, 2009.
- [5] M. J. Collon, R. Günther, M. Ackermann, R. Partapsing, G. Vacanti, M. W. Beijersbergen, M. Bavdaz, E. Wille, K. Wallace, M. Olde Riekerink, B. Lansdorp, L. de Vrede, L., C. van Baren, P. Müller, M. Krumrey, and M. Freyberg, "Silicon-pore x-ray optics for IXO," *Proc. SPIE*, vol. 7732, pp. 1F:1-9, 2010.
- [6] R. Hudec, L. Pina, V. Semencova, A. Inneman, M. Skulinova, L. Sveda, M. Mika, V. Brozek, R. Kacerovsky, J. Prokop, J. Sik, "Si and glass - novel technologies for space," *Nuclear Physics B*, vol. 166, pp. 258-261, 2007.
- [7] R. Hudec, V. Marsikova, M. Mika, J. Sik, M. Lorenc, L. Pina, A. Inneman, M. Skulinova, "Advanced x-ray optics with Si wafers and slumped glass," *Proc. SPIE*, vol. 7437, pp. 0S:1-12, 2009.
- [8] H. Wolter, "Spiegelsysteme streifenden Einfalls als abbildende Optiken für Röntgenstrahlen (Grazing incidence mirror systems as imaging optics for x rays)," *AnP*, vol. 445, pp. 94-114, 1952.
- [9] L. Proserpio, M. Civitani, M. Ghigo, and G. Pareschi, "Thermal shaping of thin glass substrates for segmented grazing incidence active optics," *Proc. SPIE*, vol. 7803, pp. 0K:1-11, 2010.
- [10] K. Nakajima, K. Fujiwara, and K. Morishita, "Systematic studies of Si and Ge hemispherical concave wafers prepared by plastic deformation," *J. Cryst. Growth*, vol. 311, pp. 4587-4592, 2009.
- [11] www.us.schott.com/special_applications/english/products/thinglass/d263/index.html
- [12] www.comsol.com

Ladislav Pina was born in Kolin, Czech Republic, on September 14, 1946. He received the M.S. and PhD degrees in physics at Faculty of Nuclear Sciences and Physical Engineering, Czech Technical University in Prague (FNSPE CTU) in 1972 and 1978, respectively. He defended his habilitation thesis on Photonics in SXR region for hot plasma physics and new X-ray sources with high brightness at CTU in 1999. He had been visiting scientist at the Queen's University of Belfast from 1980 to 1981. Since 1983 he teaches courses of experimental physics, quantum electronics and X-ray photonics and applications in the Department of Physical Electronics, FNSPE CTU. Since 1985 he holds the position of the head of laboratory for laser interaction with matter, later on laboratory of X-ray photonics. Since 1999 he occupies a position of associated professor at FNSPE CTU. His research interests include diagnostics of laser and capillary discharge plasma, X-ray sources, X-ray spectroscopy, X-ray detectors and X-ray imaging in the EUV and soft X-ray region.

Dr. Pina is an expert with the European Commission and the SPIE optical conferences program committee member.