Progress of coating stress compensation of silicon mirrors for Lynx x-ray telescope mission concept using thermal oxide patterning method

Youwei Yao
Brandon D. Chalifoux
Ralf K. Heilmann
Kai-Wing Chan
Hideyuki Mori
Takashi Okajima
William W. Zhang
Mark L. Schattenburg
Progress of coating stress compensation of silicon mirrors for Lynx x-ray telescope mission concept using thermal oxide patterning method

Youwei Yao, a,* Brandon D. Chalifoux, a,b Ralf K. Heilmann, a Kai-Wing Chan, c,d Hideyuki Morii, c,d Takashi Okajima, e William W. Zhang, e and Mark L. Schattenburg a

aMIT Kavli Institute for Astrophysics and Space Research, Space Nanotechnology Laboratory, Cambridge, Massachusetts, United States
bMIT, Department of Mechanical Engineering, Cambridge, Massachusetts, United States
cNASA Goddard Space Flight Center, Greenbelt, Maryland, United States
dUniversity of Maryland-Baltimore County, Baltimore, Maryland, United States

Abstract. A thermal oxide patterning method has proven to be effective for correcting coating-stress-induced distortion on flat silicon wafers. We report progress on developing this method for correcting curved silicon mirrors distorted by front-side iridium coatings. Owing to the difference in geometry, a finite element model has been established to calculate the appropriate duty cycle maps in thermal oxide hexagon patterns used for compensation. In addition, a photolithographic process, along with three-dimensional printed equipment, has been developed for creating patterns precisely on the back side of curved mirrors. The developed method has been used to recover the original surface shape of two silicon mirrors which are 100-mm long, 0.5-mm thick, having 312-mm radius of curvature, and 30 deg in azimuthal span (Wolter-I geometry). These mirrors’ front sides are sputter-coated by 20-nm iridium layers with ~70 N/m integrated stress. Measurement results show that the developed method can mitigate coating-induced distortion by a factor of ~5 in RMS height and ~4 in RMS slope error, corresponding to ~0.5 arc sec RMS slope error. Residual errors after correction are dominated by mid-frequency ripples created during the annealing process, which will be resolved in the future. The presented method is precise and inexpensive and a potential candidate for resolving the coating stress issue for Lynx optics in the future.

Keywords: x-ray; silicon mirror; thermal oxide pattern; figure correction; coating stress; stability.

1 Introduction

NASA’s next-generation flagship x-ray telescope concept, called Lynx, is currently under study for consideration by the 2020 Astrophysics Decadal Review. The mission concept calls for a similar angular resolution as Chandra (0.5 arc sec half-power diameter), but with a much higher effective area (30x) and wider field of view. These ambitious goals automatically lead to a lightweight mirror strategy, as has been demonstrated by NuSTAR’s hot slumping mirrors, but requiring ~100x better surface figure quality. In the past several years, a group at the NASA Goddard Space Flight Center (GSFC) has developed a technology for producing low-cost silicon metal-shell mirrors with ~0.5 arc sec RMS slope error. Owing to the intrinsic properties of monocrystalline silicon, which has very low internal stress, these mirrors can be thinned to 0.5 mm without figure degradation on the polished side. Mirror surface quality is still undergoing rapid improvements toward meeting Lynx requirements.

The effective area of mirrors in the x-ray band (0.1 to 10 keV) relies on the performance of reflective coatings. A high-quality coating utilizing high density and low surface roughness increases the critical angle at a fixed photon energy or increases the critical energy at a fixed grazing angle, thereby benefiting the telescope’s throughput. Sputtered iridium coatings had been preferred for Chandra’s thick full-shell mirrors due to their high performance and good stability. Unfortunately, these coatings have high compressive stress, which will distort thin segmented silicon mirrors far beyond Lynx tolerances.

A number of solutions for mitigating coating-induced distortion in thin x-ray mirrors have been investigated by several groups. Technologies can be summarized and divided into two kinds. The first kind minimized the coating distortion by reducing the front-side coating stress. A group at NASA Marshall Space Flight Center, by measuring the wafer curvature in situ and controlling the working gas pressure, was able to deposit a 15-nm-thick iridium layer on a 2-inch diameter flat wafer with stress around 3 MPa (0.05 N/m), which may be low enough for Lynx. However, the coating stress uniformity, stability, and reflectivity are critical and have not yet been demonstrated. Another group at GSFC tried to balance the compressive stress in iridium films by depositing a chromium layer with tensile stress underneath the iridium. They also attempted to reduce the compressive stress in iridium layers by annealing at 350°C. Neither of these methods alleviated the mirror sag error to better than 1 to 2 arc sec.

The second kind of technology compensated iridium stress by coating a layer with compressive stress on the back side of the mirrors. Methods using atomic layer deposition, ion implantation, sputtering iridium layers with optimized thickness, chromium layers with manipulated bias voltage, and PZT active correction were attempted by multiple groups and have achieved limited success, but a general robust method has not yet been demonstrated.
The thermal oxide patterning method\textsuperscript{14,15} developed by our group has demonstrated a capability of compensating coating stress distortion on flat silicon wafers with high precision and good stability, which could potentially benefit x-ray mirrors with high coating quality and excellent surface shape. Since the size of the oxide patterns on the back side of the mirror is relatively large (0.5 mm), the method has potential for low-cost mass production. In this paper, we report progress of extending this method to GSFC’s curved silicon mirrors.

2 Process

In this paper we demonstrate the thermal oxide patterning method for compensating coating stress on two pieces of curved silicon mirrors. The mirror geometry is as follows: 100-mm long, 0.5-mm thick, 312-mm radius of curvature, and 30 deg in azimuthal span (Wolter-I geometry). Mirror front sides are coated with 20-nm-thick iridium layers with $\sim$70 N/m integrated stress. Chromium layers of 3-nm thickness were first sputtered before the iridium to (1) improve adhesion and (2) reduce the overall stress after coating. The mirrors were fabricated, coated, and measured at GSFC. The thermal oxide patterns were designed and produced on mirror back sides at MIT.

The process flow is shown in Fig. 1. In step 1, the fabricated mirrors are oxidized at 1050°C for 3 h to grow $\sim$200 nm thermal SiO$_2$ layers on both sides of the mirrors. This process produces $\sim$70 N/m integrated stress in the thermal oxide, which can be used in later steps to compensate for iridium coating stress. The thickness of the oxide could be adjusted for different coating stress. After oxidation, in step 2, the mirror front side is measured by a metrology tool at GSFC, which is an interferometer with a cylindrical null.\textsuperscript{16} We have found that the measured surface figure before and after oxidation is unchanged (within 10 nm in RMS height), indicating the integrated stress field in the back side oxide is the same as that on the front side. The

![Fig. 1 Process flow of coating stress compensation for curved silicon mirrors.](https://ebooks.spiedigitallibrary.org/journals/Journal-of-Astronomical-Telescopes,-Instruments,-and-Systems)
surface topology map $M_1$ measured in step 2 is used as the mirror’s initial figure.

In step 3, the mirror back side is spin-coated with Dow SPR-700 photoresist (PR) by using our curved mirror spin tool (CMST) (see Sec. 2.4) and baked on a hotplate at 110°C for 3 min. Then the mirror is dipped into 1:7 buffered oxide etch (BOE) for 5 min to remove the front-side oxide. The back-side PR layer is subsequently removed in piranha etch solution.

In step 4, the surface topology map of the mirror back-side oxide, $M_2$, is measured by the metrology tool. The stress map of the oxide layer is calculated from the deformation map $M_2 - M_1$. The stress map calculation is based on our finite element (FE) model (see Sec. 2.1).

In step 5, the mirror front side is coated first with a 3-nm chromium layer to promote adhesion and a 20-nm iridium layer for x-ray reflection. The average integrated stress in the coating is $\sim$70 N/m. This step is accomplished by a DC magnetron sputtering system at GSFC. After coating, the surface topology of the mirror $M_3$ is measured in step 6 to calculate the coating-induced distortion, $M_3 - M_2$, thereby deriving a coating stress map as a reference for the following annealing process.

In step 7, the coated mirror is annealed in a tube furnace for 2 h (Sec. 2.2). The annealing temperature is determined carefully to ensure that (1) the coating stress after annealing is slightly lower than the oxide stress and (2) the surface roughness does not degrade. After annealing, in step 8, the mirror topology is again measured as $M_4$ to determine the mirror distortion induced by annealed coating, which is $M_4 - M_2$. The stress map of the annealed coating is thus derived.

Based on the calculated stress maps of the annealed coating and thermal oxide layers, in step 9, a hexagon pattern in the thermal oxide layer is designed to precisely balance the coating stress (Sec. 2.3). The pattern is an array of hexagon voids with fixed pitch (0.5 mm) and variable duty cycle. The pattern geometry and the design process are the same as we applied on flat silicon wafers.\cite{14,15}

In step 10, the designed patterns are fabricated in the thermal oxide layer on curved surfaces by using a customized photolithographic process (see Sec. 2.3). In the last step 11, the front surface topology of the back-side patterned mirrors are again measured to confirm the quality of coating stress compensation.

### 2.1 Metrology and Stress Calculation

The design of the hexagon pattern is derived from the measured stress maps of the annealed iridium coating and thermal oxide layers, which are calculated from the measured surface topologies. Therefore, surface metrology is of crucial importance. In this work, the measurements of the mirror surfaces are accomplished by an interferometer with a cylindrical null at GSFC. The 100 mm × 30 deg mirror surfaces are covered by $\sim$420 × 175 pixels which corresponds to $\sim$0.25 mm × $\sim$0.17 deg per pixel. Each column of the measured data (along axial direction) is fitted by 31 terms of Legendre polynomials. The zeroth and first Legendre orders, which represent piston and tilt of the axial profiles, are removed to eliminate defocusing errors in measurements. The fits without those two terms are used as the surface topography. Figure 2(a) shows an example, which is the measured surface distortion induced by back-side oxide on mirror 312S1024.

By using measured distortions, stress maps in the thermal oxide and iridium layers are calculated by using an FE model established in the commercial software ADINA. The calculation is based on a pseudoinverse method, which is similar to the one we established for flat wafers.\cite{14,15} Based on the assumption that the stress maps are dominated by low-frequency components, in our model the stress is represented by a two-dimensional Legendre polynomial which has nine terms (the first three orders for two directions produce $3^2$ terms). Figure 2(b) shows a calculated stress map in the thermal oxide layer based on the measured distortion.

The calculated stress distribution in oxide varies by $\sim$50%, due perhaps to multiple reasons: (1) compared with a flat
substrate, the deformation of a curved mirror is less sensitive to
the coating stress (at least in the axial direction), therefore, small
measurement errors in surface topology may lead to a high
variation in calculated stress and (2) nonuniformity of the thick-
ness or crystal orientation in curved silicon mirrors may result in
deviation in stress calculation.

2.2 Annealing Process for Reflective Coatings

The photolithographic process for creating PR patterns requires
baking the coated mirror at ∼110°C, which can potentially relax
the stress in the chromium and iridium films and cause large
compensation errors. Therefore, the mirrors are annealed after
sputtering at a higher temperature to relax and stabilize the
stress. Since the thermal oxide patterning method compensates
the coating stress by means of the stress in the oxide, and the
crystallographic orientation in curved silicon mirrors can vary
due to the sputtering conditions, sometimes the coating stress may be higher than the oxide stress,
which will jeopardize the stress compensation. An annealing
process with appropriate temperature after deposition can reduce
the coating stress to within the capture range.

To demonstrate effective annealing, we coated nine flat sil-
icon wafers with 20-nm iridium, annealed them under different
temperatures, and tracked the stress relaxation for multiple ther-
mal cycles, as shown in Fig. 3. Since we did not find adhesion
problems on those wafers, there was no adhesive interlayer
between iridium coatings and silicon wafers. This methodology
for chromium-coated flat wafers has already been demonstrated
in Refs. 14 and 15.

For these annealing cycles most of the stress is relaxed and
stabilized in the first cycle, which indicates that an annealing
time of 2 h is appropriate. The remaining residual stresses after
the first cycle for 200°C and 300°C are ∼75% and ∼45% of the
stress just after coating, respectively. When the temperature is
400°C, the relaxed stress values are scattered, which may be due
to random crystallization in the iridium film, suggesting that
the annealing temperature is preferred to be lower than 300°C.
In addition, the surface roughness of iridium coating does not
degrade under 350°C, as has been indicated in Ref. 9. These
results help in determining the optimum annealing temperature
based on the stress in iridium and thermal oxide. Figure 4 shows
measured results for mirror 312S1024, which is the calculated
stress map of the iridium coating before and after annealing at
250°C.

Since, for mirror 312S1024, the iridium coating stress is near
the same level of the thermal oxide stress, as shown in Fig. 2(b),
the annealing temperature is determined to be 250°C. The
stress relaxation is ∼40% as predicted in Fig. 3. The stress dis-
tribution in the coating before and after annealing is similar to
the distribution in thermal oxide, which may be due to system-
atic measurement or stress calculation artifacts that should can-
cel in the duty cycle calculation, as is demonstrated in the next
section.
2.3 Pattern Design

As has been demonstrated in our previous work, the thermal oxide pattern is an array of hexagon-shaped voids with adjustable size in each cell.14,15 Since the pitch between the hexagons is fixed at 0.5 mm, the area fraction of the hexagon void relative to its unit cell is defined as a duty cycle, which can be calculated by the following equation:

\[
\text{Duty cycle}(\theta, Y) = \frac{S_{\text{oxide}}(\theta, Y) - S_{\text{annealed}}(\theta, Y)}{S_{\text{oxide}}(\theta, Y)},
\]

where \(\theta\) is the coordinate in the azimuthal direction and \(Y\) is the axial location. Here, \(S_{\text{oxide}}(\theta, Y)\) is the measured stress in thermal oxide, corresponding to Fig. 2(b), and \(S_{\text{annealed}}(\theta, Y)\) is the annealed coating stress, corresponding to Fig. 4(b). The calculated duty cycle map based on those two figures is shown in Fig. 5.

The derived duty cycle map is converted to a hexagon plot and saved as an AutoCAD R12 file for the photomask vendor. A plastic film mask is selected for producing the hexagon pattern, which will be discussed in next section.

2.4 Photo Lithography Process on Curved Mirrors

A great challenge of this work is to produce the designed pattern on a rough-and-curved silicon surface with good precision. In addition, the roughness of iridium coating on the front side should not degrade through the process. After much trial and error, the fabrication process was eventually matured and is described as follows:

1. Manually paint MicroChemicals AZ-5214 PR on the front side of the mirror and bake the mirror on a hotplate under 110°C for 5 min. Since the iridium coating is covered by a harden PR layer, it is prevented from the following mechanical and chemical exposure to avoid roughness degradation.

2. Place the mirror (facing down) on the CMST (Fig. 6) and carefully align it to the center. Apply vacuum to the mirror to constrain it tightly on the CMST. Spin-coat Dow SPR-700 PR on the back side of the mirror. The RPM is set at 3000, which produces a uniform \(\sim 1 \mu\text{m}\) PR layer on the back side. The spin-coated mirror is baked on a hotplate for 5 min to harden the PR.

3. Place the mirror on a curved mirror exposure tool (CMET, Fig. 7). Press a plastic film mask with the designed hexagon pattern over the mirror, carefully align the mask with the mirror, and fix it with tape. Then cover the mask and the mirror with a plastic vacuum bag. Seal the bag with sticky rubber at the edge of the CMET. Pull a vacuum on the CMET; due to the designed channels, the vacuum bag can press the mask.
tightly on the back side of the mirror to ensure high precision of pattern transfer.

4. Exposure with UV light (365- and 406-nm wavelengths) for seven cycles. The exposure time for each cycle is 60 s, which leads to 420-s exposure time in total, corresponding to a dose of \(3780 \text{ mJ/cm}^2\).

5. Remove the mirror and immerse in developer solution (Microposit MF CD-26) for 90 s. Rinse it in deionized water and blow dry in \(\text{N}_2\). This results in a PR layer with hexagon voids patterned on the top of the thermal oxide.

6. Dip the mirror in a BOE solution for 5 min so the thermal oxide within the hexagon voids is thoroughly removed.

7. Soak the mirror in acetone and isopropyl alcohol to strip the PR from both sides of the mirror. Blow dry it in \(\text{N}_2\).

After these steps, a thermal oxide pattern remains, as shown in Fig. 8.

3 Results

The process was applied to two mirrors, 312S1024 and 312P1052, to compensate for the surface distortion induced by 20-nm iridium coatings. Figure 9 shows the measured deformation from the iridium coating (left) and after compensation (right). Table 1 lists the calculated RMS heights and slope errors from these results. The thermal oxide patterning method successfully reduced the coating stress-induced distortion by a
factor of $\sim 5$ in RMS height and $\sim 4$ in RMS slope error on those mirrors, which is less than we achieved on flat wafers ($\sim 68$ in RMS height and $\sim 50$ in RMS slope error).

After a close look at the data, we discovered that the residual errors on these mirrors after compensation are dominated by centimeter-scale ripples. Figure 10 shows the measured surface deformation of mirror 312S1024 in steps 2, 6, 8, and 11 relative to the initial shape before oxidation (step 0), where the data are fit by Legendre polynomials, and low frequency terms from zeroth to fourth order are excluded from the plots. The results clearly show that the mid-frequency ripples appeared after the step 7 annealing process and remained after correction in Step 11.

**Table 1** RMS heights and slope errors of coating distortions calculated from Fig. 9.

<table>
<thead>
<tr>
<th>Coating distort.</th>
<th>After Comp.</th>
<th>Coating distort.</th>
<th>After Comp.</th>
</tr>
</thead>
<tbody>
<tr>
<td>RMS height (nm)</td>
<td>113.7</td>
<td>24.6</td>
<td>1.895</td>
</tr>
<tr>
<td>312S1024</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>312P1052</td>
<td>101.2</td>
<td>19.3</td>
<td>1.801</td>
</tr>
</tbody>
</table>

Fig. 9 Measured surface distortion induced by coating (a) after annealing and (b) after stress compensation. For mirror 312S1024, the initial oxide stress is shown in Fig. 2, the coating stress is shown in Fig. 4, the calculated duty cycle is shown in Fig. 5, and the created pattern is shown in Fig. 8.
Possible explanations for this phenomenon are still under investigation. Multiple solutions will be followed up to eliminate the mid-frequency errors, including improvements of the coating stress uniformity in annealing process and stress calculations with higher-order Legendre polynomials. Progress will be reported in future work.

4 Conclusions

We have developed a thermal oxide patterning method for compensating silicon mirror distortion induced by coating stress. This method has been demonstrated by compensating the coating stresses in 20-nm-thick iridium films on two pieces of GSFC-fabricated silicon mirrors which are 100-mm long, 0.5-mm thick, having 312-mm radius of curvature, and 30 deg in azimuthal span (Wolter-I geometry). As subtasks for this effort, we have developed (1) a process to design the thermal oxide hexagon pattern, (2) an annealing process to stabilize and adjust the coating stress, and (3) a photolithographic process to create hexagon patterns on the back side surface of the mirror, which are curved and rough. Experimental results show that we have successfully reduced the coating-induced distortion by a factor of \( \sim 5 \) in RMS height errors and \( \sim 4 \) in RMS slope errors, which corresponds to the 0.5 arc sec level in RMS slope errors. The residual errors are dominated by mid-frequency stress ripples which appear to be generated by the annealing process.

For future work, we plan to improve the stress compensation precision by eliminating the mid-frequency residual errors. Annealing process parameters will be optimized, and higher-frequency stress functions will be used to design oxide patterns for better compensation.

Acknowledgments

This work was supported by NASA Grant Nos. NNX14AE76G and NNX17AE47G.
Yao et al.: Progress of coating stress compensation of silicon mirrors for Lynx x-ray

References


Biographies of the authors are not available.