Compensating film stress in silicon substrates for the Lynx X-ray telescope mission concept using ion implantation

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ABSTRACT

Ion implantation is used to correct figure errors resulting from film stress in thin silicon mirror substrates. The Lynx mission concept requires mirrors with extremely small figure errors and excellent X-ray reflectivity, and only a small portion of the mirror error budget may be allocated to distortion from film stress. While reducing film stress in itself is ideal, compensation of film stress may be required. In addition, compensation, in combination with other film stress reduction techniques, may allow freedom in making coatings with optimal x-ray performance while minimizing distortion. Ion implantation offers a rapid method of applying a precise stress distribution to the backside of a mirror, which may be used to compensate for a uniform or non-uniform film stress. In this paper, we demonstrate the use of ion implantation to achieve a roughly 10x reduction in deformation from film stress, and that the stress from ion implantation is stable over at least five months.

Keywords: X-ray, Lynx, telescope, optics, figure correction, ion implantation, film stress, stability

1. INTRODUCTION

The Lynx X-ray telescope mission concept[1] calls for a 0.5 arcsecond half-power diameter (HPD) angular resolution with effective area > 2 m^2. This telescope would require several hundred square meters of extremely accurate mirror surface area. In order to densely nest the mirror shells and keep mass low, the mirrors must be very thin. Three approaches are currently under consideration[1] for the telescope mirror assembly: full-shell fused silica mirrors[2], segmented single-crystal silicon mirrors using a meta-shell concept[3], and segmented glass mirrors with active figure correction[4]. The latter two concepts use thin mirror segments (as opposed to full mirror shells), and significant progress has been made in developing excellent mirror figure rapidly approaching the requirements for Lynx. However, stress in the reflective X-ray coating presents substantial difficulty, since thin mirror segments are very compliant and deform due to intrinsic stress in the film. The goal of the present work is to compensate for the deformation caused by thin film deposition, and we focus specifically on compensating for film stress in metal films used as a reflective layer, in single-crystal silicon substrates, using ion implantation.

A thin reflective film, typically high-Z metals such as iridium, is required for efficient grazing-incidence X-ray reflection. These films typically have very large intrinsic stress, which can cause delamination of the film from the substrate, and also results in deformation of the substrate, which degrades the angular resolution of the telescope. Deposition parameters, such as plasma power and gas pressure, strongly affect the film stress, roughness and density. It may be possible to choose parameters that maintain high density and low roughness while minimizing stress[5], but decoupling the film optical properties from the stress properties may allow a much simpler coating process and would enable more design freedom for optical coating experts.

Typical film stress for sputtered iridium is -1 to -3 GPa (negative stress indicates compressive), and the typical thickness should be about 20 nm for soft X-rays (~1 keV)[6]. The film stress multiplied by the film thickness, called the integrated stress, determines the deformation of the substrate. Even assuming that the typical -20 to -60 N/m integrated stress could be reduced to -1 N/m, the deformation from this film stress is significant for a mission with requirements like Lynx. Figure 1 shows the deformation of a 0.5 mm-thick silicon mirror due to a -1 N/m integrated stress (equivalent to -50 MPa stress and 20 nm thickness), calculated using ADINA finite element software. The mirror radius is 250 mm, the length is 100 mm, the chord (width) is 100 mm, and the cone angle is 0.36° (i.e., a primary mirror for a roughly 10 m focal length telescope). The root mean-squared slope error in the axial direction is 0.09 arcsec. Since the primary and secondary mirror errors are correlated, and since reflections double the angular deviation of the reflected rays, this will likely represent a significant error compared to a 0.5 arcsec HPD angular resolution requirement. It is important to emphasize that 1 N/m is a very small stress tolerance.

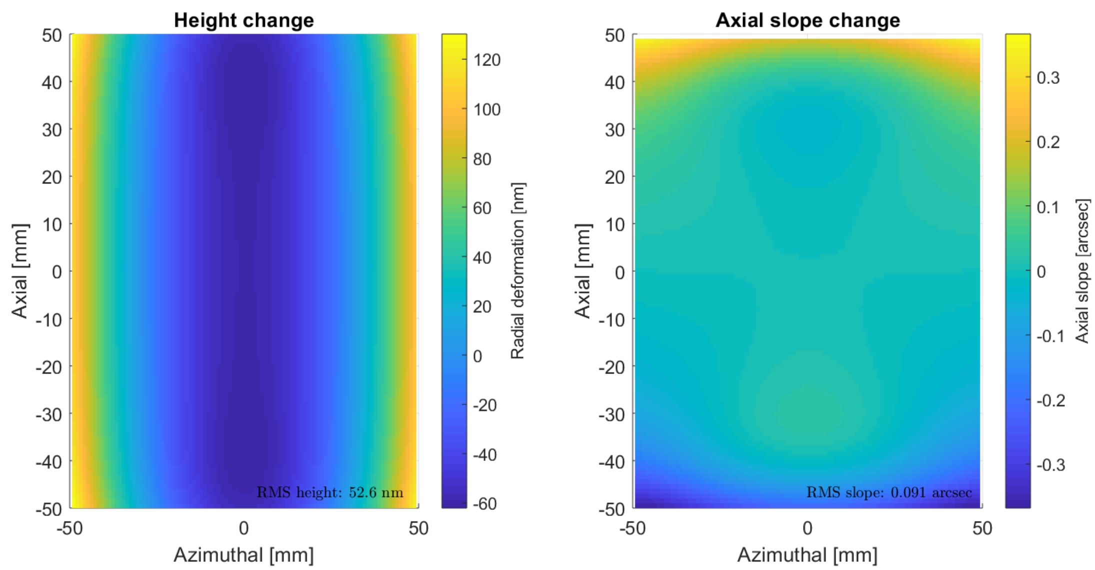

Figure 1. Modeled deformation of a conical mirror, resulting from -1 N/m integrated stress applied to the concave surface. The mirror dimensions are: front radius 250 mm, length 100 mm, width 100 mm, cone angle 0.36°. The RMS axial slope change is 0.09 arcsec.

Ion implantation is a method of generating a stable stress in silicon (as well as glass), which we have reported on previously[7]. Other methods of applying stress to silicon, which are currently under study, include: using patterned thermal oxide[8], depositing a magneto-strictive film[9], depositing piezoelectric film[4], or depositing a chromium film with varying substrate bias[10].

Ion implantation is an attractive process for compensating for film stress since it does not require deposition of any films. The film stress compensation process has only two steps (implanting and annealing), compared to >10 for patterned thermal oxide[8]. Ion implantation could be very quick, currently requiring about 22 minutes per 100 mm silicon wafer with a research-grade ion accelerator. With a commercial ion implanter, this could be much faster due to higher achievable ion beam current.

With any method of compensating for film stress, stability and accuracy are absolutely critical. Ion implantation allows precise control of the stress by controlling the number of ions implanted, allowing us to routinely reduce the deformation from film stress by a factor of 10. We will also show that with post-implant annealing, the implanted stress is stable over at least five months, and robust to heating up to 70°C.

2. PROCEDURE

In this section, we detail the process we developed for compensating for film stress using ion implantation. Since ion implantation does not require any film deposition, the process requires fewer steps than for thermal oxide patterning[8]. We will demonstrate this process on flat silicon wafers, with results in Section 3. This process could, in principle, easily be adapted to curved mirrors. The process, from a raw uncoated silicon substrate to a coated and compensated silicon substrate, consists of seven steps, three of which are measurements. The process is summarized in Table 1 and detailed in the following subsections.

Table 1. Process outline for compensating film stress using ion implantation.

Process step		Details
1	Measure	Shack-Hartmann metrology tool
2	Coat	RF sputter 30 nm Cr with substrate bias
3	Anneal	200-300 °C in N_2 for 4 hours
4	Measure	Shack-Hartmann metrology tool
5	Implant	2 MeV Si^{++} ions, dose depends on stress
6	Anneal	120 °C in N_2 for 4 hours
7	Measure	Shack-Hartmann metrology tool

2.1 Surface measurements and stress calculations

Surface measurements were performed using a Shack-Hartmann metrology tool[11] and a low-stress wafer mounting structure[12]. Since a Shack-Hartmann tool measures surface slopes, surface height maps must be constructed either by zonal (integration) methods or fitting to Zernike polynomials (or other functions). In this work, all surface height maps have been fit to the first 15 Zernike polynomials. After several improvements in software, mounting procedures, and building an enclosure to reduce air turbulence, we have observed about 15 nm RMS repeatability over the entire surface of 100 mm silicon wafers using this metrology tool. The noise spectrum of this tool is shown in Figure 2. This spectrum is the standard deviation of each Zernike term in repeated measurements of a bare, unprocessed silicon wafer. The 17 measurements that compose this sample took place over 35 days, and each measurement was taken on a different day. The root-sum-of-squares of the Zernike terms shown here, and the standard deviation of the surface height, is 15.5 nm.

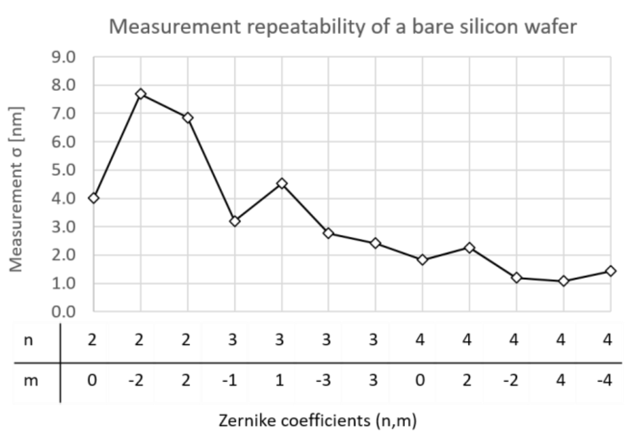

Figure 2. Measurement repeatability of Shack-Hartmann metrology tool, represented as a spectrum of Zernike polynomials with radial degree n and azimuthal order m. Over several months, a bare, unprocessed double-side polished wafer was measured. The repeatability plotted above is the standard deviation of each Zernike term over a 30-day period, and is composed of 17 measurements taken on different days. The RMS surface variation is 15.5 nm.

When a stressed film is applied to the surface of a substrate, it causes deformation. We calculate the stress in the film by evaluating the change in shape of the substrate (as measured by the Shack-Hartmann metrology tool). The stress in a metal film is typically equibiaxial (i.e., isotropic), but non-uniform over the surface. In this work, we calculate the stress using a pseudo-inverse, a method that we have applied previously[13]. We apply a set of N stress distribution functions $s_i(r,\theta)$ to a finite element (FE) model, where i is an index indicating which stress distribution function is applied. We then compile the resulting surface height changes $w_i(r,\theta)$ into a matrix $\boldsymbol{A}$,

$$\boldsymbol{A} = \overbrace{\begin{bmatrix} w_1(r_1,\theta_1) & \dots & w_N(r_2,\theta_2) \\ w_1(r_2,\theta_2) & \ddots & w_N(r_2,\theta_2) \\ \vdots & \dots & \vdots \end{bmatrix}}^{stress\ function\ s_i(r,\theta) \rightarrow deformation\ w_i(r,\theta)}$$

The measured displacement at each node in the FE model is $\vec{w}_{meas}$. The stress at each node is then $\vec{s}_{meas} = \boldsymbol{A}^{+}\vec{w}_{meas}$, where $\boldsymbol{A}^{+}$ is the pseudo-inverse of $\boldsymbol{A}$. An example of a measured change in surface height and the corresponding stress measurement is shown in Figure 3.

We chose to use the pseudo-inverse method is instead of an analytical approach to calculating the equibiaxial stress distribution. With perfect metrology and a deformation that was caused by equibiaxial stress, the analytical solution and the pseudo-inverse solution would be identical since there is exactly one equibiaxial stress distribution that produces a particular deformation, in this case. However, metrology noise adds additional deformation components that cannot be exactly generated or corrected using equibiaxial stress only. In this case, many slightly different stress fields could explain the measured deformation with error comparable to the measurement noise of 15 nm RMS. The pseudo-inverse method simply minimizes the RMS difference between the measured deformation and the simulated deformation (typically about 5 nm RMS), whereas an analytical approach requires one to decide which deformation components are real and which are noise. We chose to use the pseudo-inverse method because it is not subject to any choice.

2.2 Metal film deposition and annealing

In this work, we coated flat wafers with chromium and compensated for the resulting film stress. While most x-ray mirrors are coated with iridium, an iridium sputtering target is expensive, and for process development using chromium should be an acceptable substitute as long as the stress is similar in sign and magnitude to iridium. We are only concerned about the deformation caused by the film stress, which has a very weak dependence on the film mechanical properties when the film is so thin compared to the substrate (e.g., 20 nm film thickness compared to 500,000 nm substrate thickness).

Iridium, under sputtering process conditions that result in excellent film roughness and density, typically has a film stress around -1 GPa[5,6] (compressive). For a thickness of 20 nm, this results in an integrated stress of -30 N/m. Chromium, under typical sputtering conditions (e.g., 3 mTorr Ar pressure), results in a tensile stress. However, substrate bias can be used to ensure sputtered chromium films have a compressive stress[10]. The substrate bias accelerates the Ar ions in the plasma, bombarding the surface and providing an atomic peening effect that compresses the film. Biasing the substrate, we were able to consistently achieve compressive integrated film stresses of -10 to -30 N/m (i.e., -0.3 to -1 GPa in a 30 nm film), after annealing.

The films were RF sputtered using a tool from AJA International. The wafer is located on a rotating stage about 300 mm away from the sputtering gun. The deposition was conducted with 3 mTorr Ar, and 150 W RF power applied to the sputtering target. Prior to deposition, the wafer was back-sputtered to remove any organic contaminants, by biasing the wafer to 25 W and 190 V using an RF bias supply. During deposition, the wafer was biased at 10 W and 95 V. The thickness of the film was estimated using a quartz crystal monitor immediately prior to the deposition.

After deposition of the film, the film stress must be stabilized. Yao, et al.[8] found that the Cr stress degrades over time after the deposition, by about 5-10%. Annealing the film at 200-300 °C for 2 hours is sufficient to stabilize the film stress. Annealing also reduces the film stress by 50-70%.

We did not measure the film thickness directly, since we are only interested in the integrated film stress (stress times film thickness), which we can calculate from the deformation measurement (see Section 2.1) without knowledge of the film thickness. The average integrated stress for the five coated wafers used in this work is summarized in Table 2. The deformation resulting from the Cr film, and the corresponding integrated stress, is shown in Figure 3.

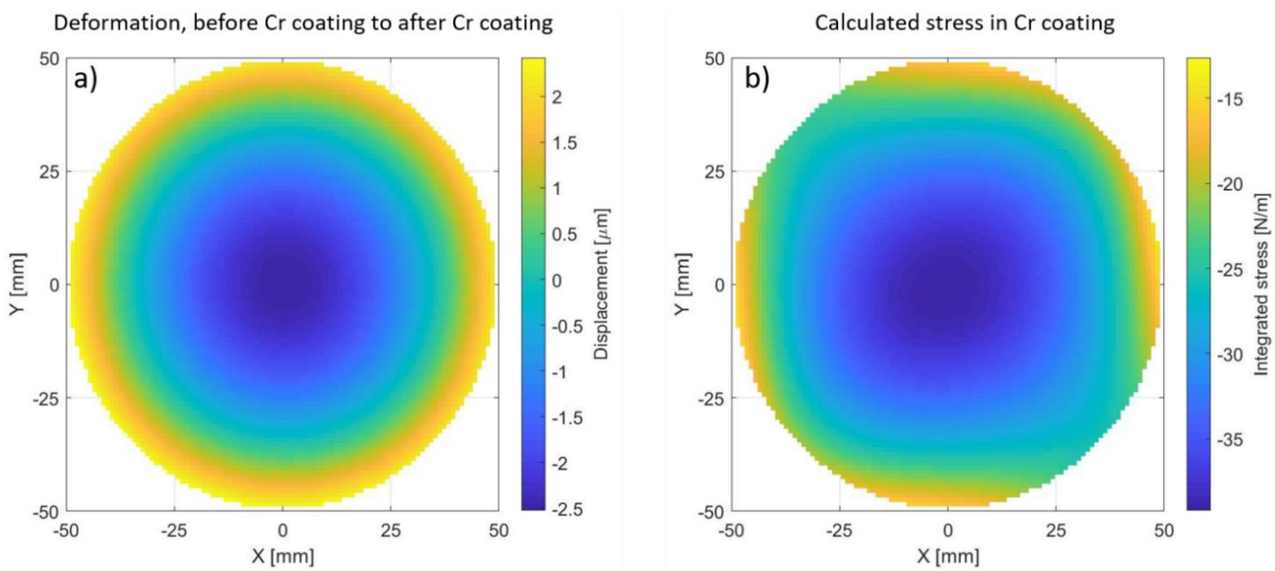

Figure 3. Measured deformation and calculated stress in a chromium film. The deformation (a) was measured using the Shack-Hartmann metrology tool and fit to Zernike polynomials. The integrated stress (b) was calculated using the pseudo-inverse method, and is non-uniform due to non-spherical terms in the deformation map.

Table 2. Measured values for the five wafers coated for this study.

Wafer	**RMS deformation [nm]**		**Mean integrated stress [N/m]**	
	After deposition	**After annealing**	**After deposition**	**After annealing**
1	3,953	820	-85.9	-15.7
2	4,028	1,415	-88.2	-28.6
3	2,842	1,508	-60.1	-30.4
4	2,107	1,028	-47.0	-21.9
5	2,048	1,037	-46.6	-22.7

As shown in Figure 3, the film stress is not uniform. The Zernike spectrum of the deformation from the film stress indicates that there are several terms other than spherical curvature that are significantly above the metrology noise floor, which leads to the calculated non-uniform equibiaxial stress. This type of stress map is seen in every coated wafer that we measured.

2.3 Ion implantation and annealing

Implanting high-energy ions (in this work, 2 MeV) into crystalline silicon results in a compressive stress near the surface that is dependent on the ion dose, or the number of implanted ions per unit area. This effect has been observed for low-energy ions down to tens of keV, which can be achieved using commercial ion implanters. The ion implanter available at MIT is a General Ionex Tandetron ion accelerator, which most effectively generates a >1 MeV Si ion beam. Based on this machine's capabilities, for this work we have used 2 MeV Si^{++} ions, with an ion beam current around 1.5 μA.

The mechanism driving the stress generation is crystal lattice damage, as shown by Rutherford backscattering spectroscopy[14] and Raman scattering spectroscopy[15]. Amorphous silicon has a ~3% lower density than crystalline silicon, so compressive stress develops in the damaged silicon. The fraction of additional material added by the ion implantation is ~0.01% by volume, which is far smaller than the density change. For 2 MeV Si^{++} ions implanted into crystalline silicon, the ions stop within the first ~2 μm of the surface, and this is where the crystal damage is contained.

We are interested in controlling the integrated stress, since this is the parameter related to deformation of the substrate, as with the film stress discussed in Section 2.2. The primary variable that affects the integrated stress is the ion dose, which is the number of ions implanted per unit area. However, it is well-known that heating allows some defects generated by the ion bombardment move back to their lattice positions. We also find that substrate temperature during implantation has a small but important effect on the integrated stress.

The integrated stress as a function of ion dose is shown in Figure 4. This stress is always compressive due to the expansion of the damaged silicon. Each data point represents one wafer, and for each wafer, we measured the change in shape and calculated the integrated stress map as described in Section 2.1. Each wafer, after ion implantation, was annealed for 4 hours at 120 °C as discussed below. The integrated stress plotted in Figure 4 is the mean integrated stress for each wafer after annealing. For a dose of 8×10^{13} ions/cm^2 (or equivalently, 30 N/m integrated stress) and a beam current of 1.5 μA, the implant time is about 22 minutes.

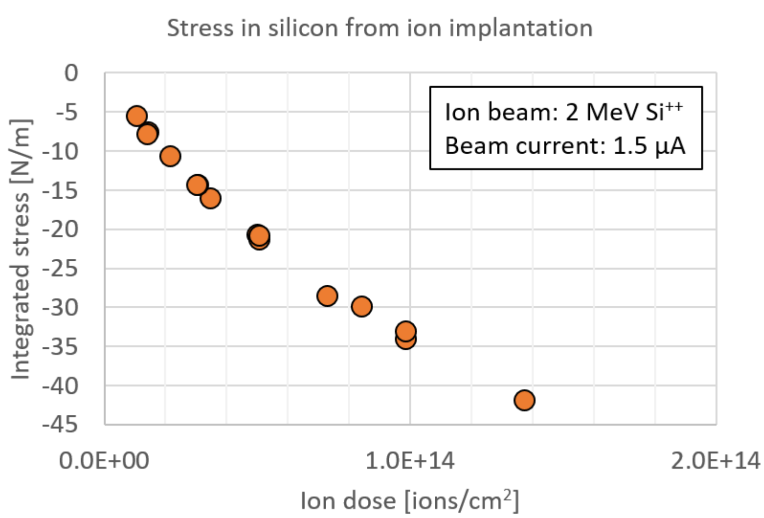

Figure 4. Integrated stress induced in silicon due to ion implantation.

After ion implantation, the damage to the crystal lattice begins to heal over time, reducing the stress in the wafer. Annealing the wafer at a temperature above 100 °C stabilizes the stress. Figure 5 shows the integrated stress in 11 wafers that were implanted to a high integrated stress (~-100 N/m) and monitored over time after annealing at various temperatures. The change in stress over time in wafers that were not annealed is large. Annealing the wafers at 80 °C reduces the change in stress, but does not eliminate it. Annealing above 100 °C appears to eliminate the change in stress over time, so for additional safety, we choose to anneal all wafers at 120 °C. The negative effect of annealing at a higher temperature is that the integrated stress is reduced further. However, since the implant time is typically <30 minutes, this is a minor drawback at this point.

Stability is critical for any space telescope. We measured three wafers that were annealed at 120 °C over a period of five months, and found that there is no measureable change in shape of the implanted wafers, as shown in Figure 6a. The three wafers, S2018122114, S2018122115, and S2018122120 were initially implanted to an integrated stress of -21.2, -20.8, and -28.5 N/m. None of these three wafers show any measurable relaxation. Two additional wafers were baked three times each for 4 hours at 60-70 °C in air to test whether thermal cycling at temperatures that could be encountered during launch will change the implanted stress. There is no significant change in the shape of these two wafers, as shown in Figure 6b. These results suggest that after 4 hours of post-implant annealing at 120 °C, the implant-induced stress is stable over time and under repeated thermal cycles. The changes measured for these five wafers are consistent with measurement noise.

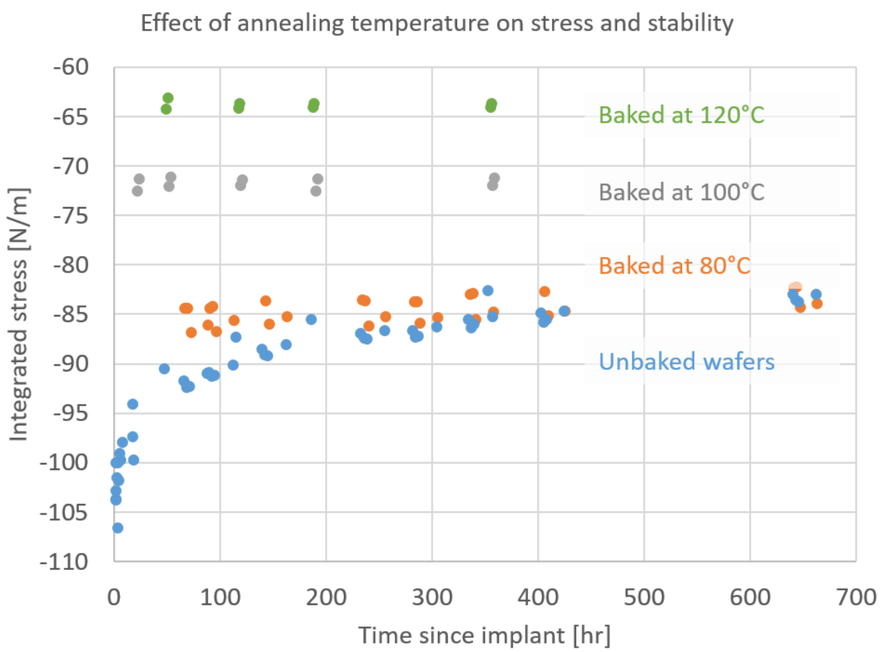

Figure 5. Effects of annealing on integrated stress magnitude and stability. The annealed wafers were baked for 4 hours at the indicated temperatures in a box furnace in air.

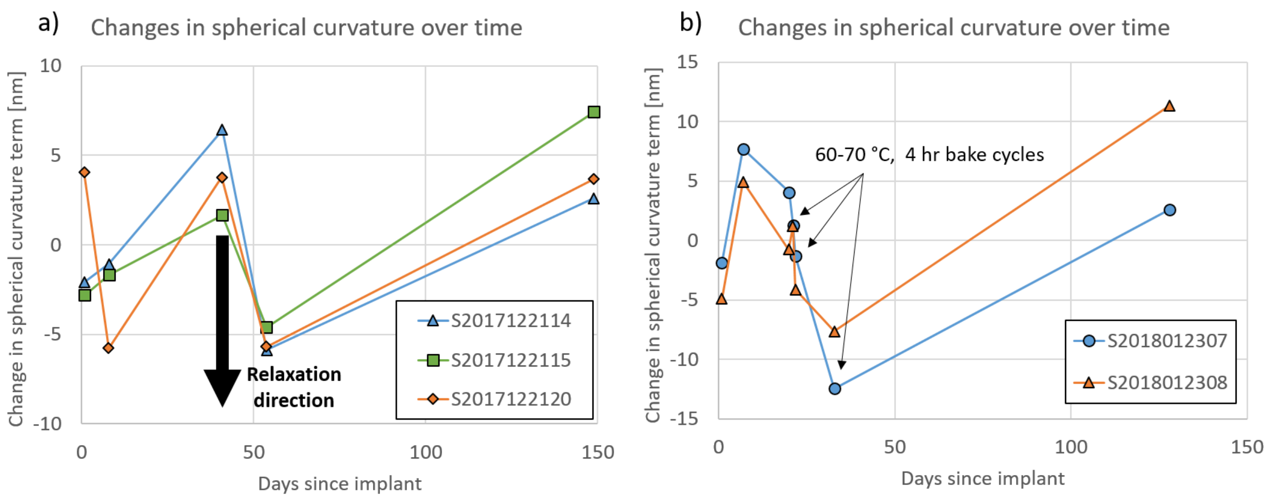

Figure 6. Variation of spherical curvature over time for five implanted wafers: a) changes in three wafers kept at room temperature for five months, showing changes consistent with measurement noise (± 4 nm RMS), and b) two implanted wafers monitored over time but also baked three times at 60-70 °C for 4 hours. The changes here are also consistent with measurement noise.

3. FILM STRESS COMPENSATION RESULTS

Five silicon wafers were implanted to compensate for the deformation from a 30 nm Cr film. The results are summarized in Table 3. Figure 7 shows the deformation caused by the Cr film (after annealing) and the net deformation caused by the Cr film plus ion implantation. The target deformation shown in Figure 7b is to be zero, which would indicate that the wafer has exactly the same shape after film deposition and ion implantation as before film deposition. If this were to be applied to a curved mirror, this would mean that the mirror figure would be unaffected by the film deposition. We find that the residual error is consistently around 100 nm RMS.

Table 3. Measured wafer height and slope values before and after ion implant correction

	RMS deformation from Cr film		**Final RMS values**		**Relative improvement**	
Wafer	**Height [nm]**	**Slope [arc-sec]**	**Height [nm]**	**Slope [arc-sec]**	**Height**	**Slope**
1	820.3	14.8	34.0	0.9	24.1	16.0
2	1415.2	25.5	93.7	2.1	15.1	12.3
3	1507.6	27.2	126.1	2.5	12.0	11.0
4	1028.4	18.6	115.9	2.2	8.9	8.4
5	1036.9	18.7	119.9	2.2	8.6	8.4

Comparing the residual error to the deformation caused by the film stress, we find that the RMS height and slopes are all consistently reduced by about a factor of 10. The RMS heights are calculated from the Zernike fit of the slopes measured from the Shack-Hartmann metrology system, and the RMS slopes are calculated directly from the slopes measured from the Shack-Hartmann metrology system. The fact that the improvement in RMS height and slope is very similar suggests that higher-frequency errors are not introduced into the mirror by the ion implantation.

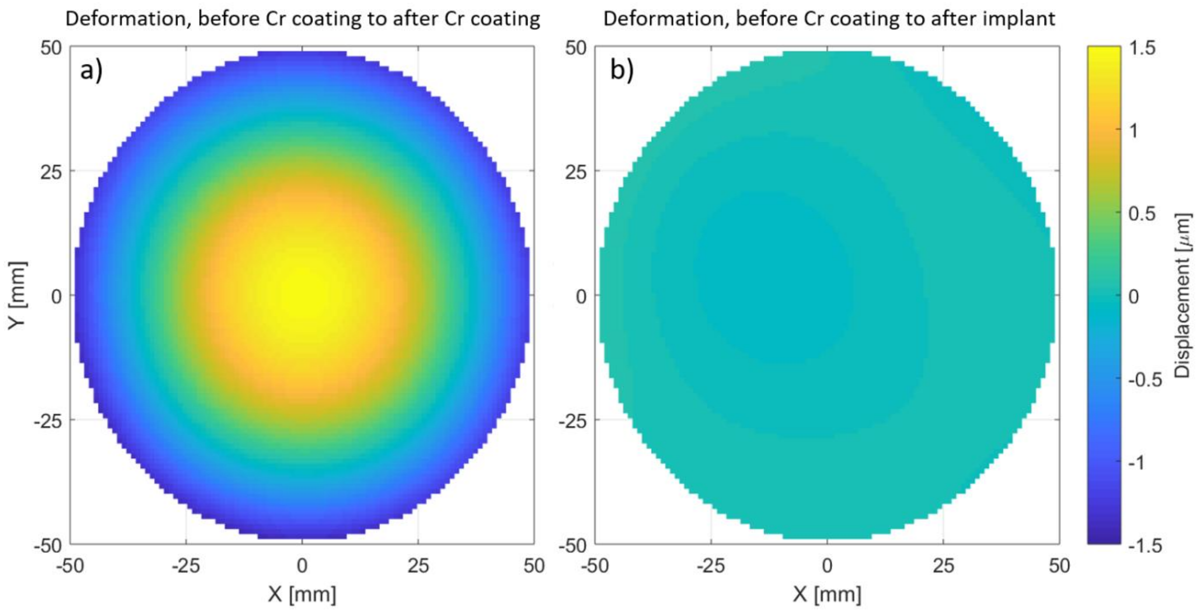

Figure 7. Comparison of deformation from: a) chromium coating stress, and b) entire process. These images illustrate that the deformation due to coating stress is mostly eliminated using the ion implantation process.

Figure 8 shows the Zernike spectrum of the residual error for each of the five wafers. The residual error is dominated by spherical curvature (n=2, m=0), indicating that the average stress from the ion implantation is not perfectly controlled. Aside from the spherical curvature error, astigmatism (n=2, m=±2) and coma (n=3, m=±1) are often most significant. We are currently investigating possible causes of these terms. The coma terms may come from temperature gradients during the post-implant annealing step, since a linear variation in stress leads to a coma deformation.

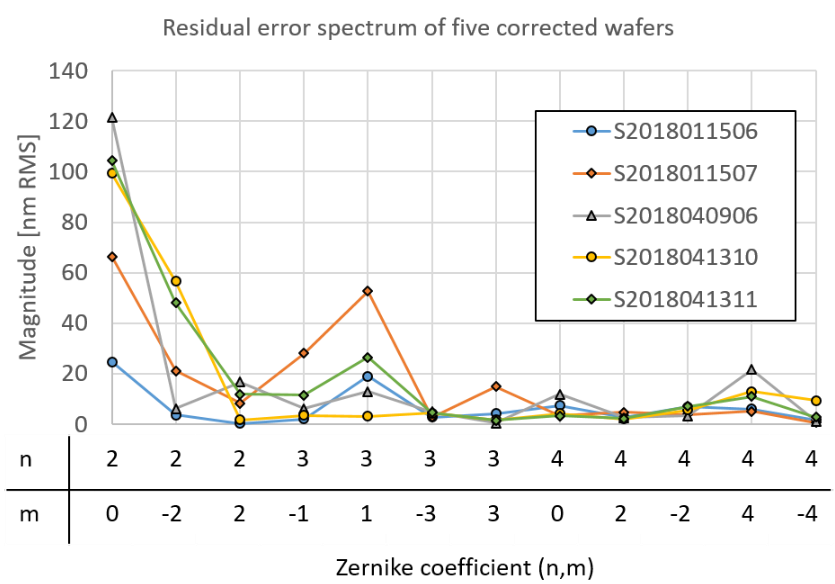

Figure 8. Residual error Zernike spectrum for the five wafers used in this study. The absolute value of each Zernike component magnitude is plotted, where n is the radial degree and m is the azimuthal order of the Zernike polynomial.

4. CONCLUSIONS AND FUTURE WORK

We have demonstrated that ion implantation is a viable method of compensating for compressive film stress in silicon substrates. We have shown that a factor of 10 improvement in surface figure can be routinely achieved, and that the implanted stress is stable. The time required to implant mirrors is only about 22 minutes, using a research-grade accelerator that has much lower ion beam current than commercial ion implanter machines.

While the relative improvement is currently not as large as for the thermal oxide patterning method presented by Yao, et al.[8], it may be sufficient for the Lynx X-ray observatory. We must apply this process to curved mirrors to test whether the current process can adequately compensate for coating stress. If further improvement is required, it might be possible to develop a two-pass implant process. The primary challenge with such a process would be to develop a set of annealing cycles that would make the two implant cycles independent.

Stability is absolutely critical for any process used to fabricate space telescope mirrors, and film stress compensation is no exception. We have shown that the stress from ion implantation is very stable in silicon, as measured over five months, if the wafers are annealed at 120 °C for 4 hours. We have also shown that implanted and annealed wafers are not affected by heating to 60-70 °C for 12 hours. With the annealing process we developed, the changes in wafer shape are below the noise floor of our metrology system. Determining whether the stress is stable to a level required by Lynx would require a more sensitive and stable metrology system. However, if long-term stress relaxation were to be discovered with a more sensitive metrology system, then we expect annealing hotter and/or longer would stabilize the stress. The only downside, which is not necessarily a problem, to this is that the implantation time will increase because annealing hotter further reduces the implant stress.

Lynx requires production of tens of thousands of mirror segments. As with any technology used to produce the Lynx mirror assembly, ion implantation would need to be industrialized. It may be possible to process tens of thousands of mirror segments at MIT in just a couple of years, even with the low ion beam current available with this accelerator. A commercial ion implanter, many of which are readily available and would require minimal modification, could process the mirrors in a much shorter time period since the ion beam currents are much larger. We have previously reported[13] on implanting 150 keV Si^{+} ions into silicon wafers using a commercial ion implanter, and the stress is quite similar to the stress from the 2 MeV Si^{++} ions used in the present work. Based on that work, the implant time using a commercial implanter could be just a couple of minutes.

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