

# Coating Thin Mirror Segments for Lightweight X-ray Optics

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## ABSTRACT

Next generation's lightweight, high resolution, high throughput optics for x-ray astronomy requires integration of very thin mirror segments into a lightweight telescope housing without distortion. Thin glass substrates with linear dimension of 200 mm and thickness as small as 0.4 mm can now be fabricated to a precision of a few arc-seconds for grazing incidence optics. Subsequent implementation requires a distortion-free deposition of metals such as iridium or platinum. These depositions, however, generally have high coating stresses that cause mirror distortion. In this paper, we discuss the coating stress on these thin glass mirrors and the effort to eliminate their induced distortion. It is shown that balancing the coating distortion either by coating films with tensile and compressive stresses, or on both sides of the mirrors is not sufficient. Heating the mirror in a moderately high temperature turns out to relax the coated films reasonably well to a precision of about a second of arc and therefore provide a practical solution to the coating problem.

**Keywords:** Metallic thin-film deposition, coating stress, x-ray optics, lightweight mirrors, segmented mirrors

## 1. INTRODUCTION

### 1.1 Segmented Lightweight X-ray Optics

Next generation's high resolution, high throughput optics for x-ray astronomy requires integration of a large number of very thin mirror segments into a very lightweight telescope assembly with negligible distortion<sup>1,2,3,4</sup>. The basic grazing incidence reflective x-ray optics of Wolter-I design (or other, such as Kirkpatrick-Baez design) requires fabrication of large but thin mirrors with high precision. Thin, lightweight (areal density of 1.0 kg/m<sup>2</sup>) glass substrates having sizes of 200 mm can now be thermally formed, by slumping on precise mandrels, to achieve a resolution of ~ 3 arc-seconds (half-power diameter, HPD). For example, at NASA/Goddard, glass substrates were routinely made to produce 5" HPD mirror pairs for a telescope design at 8.4 m (Wolter Type I, double reflection.)<sup>2,5</sup> A metallic film that is subsequently deposited onto the substrate's surface for x-ray reflection, however, generally has high stress that distorts the mirror. In this paper, we will report the work to mitigate this problem and to achieve coating with negligible distortion.

It is now generally accepted that the next high throughput telescope will be an assembly of modules of segmented mirrors. Making large, full-shell grazing incidence optics is simply not practical, both in terms of technology and cost. Our group at Goddard has been actively pursuing technology for making lightweight x-ray optics, and has developed a full suite of capabilities to that end. These include substrate fabrication, coating, mirror alignment and mounting, module design and fabrication, in addition to metrology and x-ray testing<sup>2</sup>. To understand our requirement on the mirror, and therefore on mirror coating, it is important to understand our approach to building x-ray telescopes. Our approach is to fabricate mirror segments as precisely as they meet the telescope requirement, and mount them into telescope housings with negligible distortion. This is the approach taken by missions such as Astro-H<sup>6</sup>, and the mission concept for a future large X-ray observatory AXSIO<sup>7</sup>. Other approaches are possible. For example, one can force a mirror segment into an externally pre-determined shape. This was done, for example, for the x-ray telescopes for the NuSTAR<sup>8</sup> mission and is the basis of ESA's Silicon Pore Optics<sup>9</sup>. In such approach, the precision of the pre-determined structure that the mirror goes into is naturally of paramount importance. Another possibility is active optics. Techniques of actively adjusting

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many thin mirrors' figures, (with piezoelectric devices, for example) are being considered by some groups<sup>10</sup>. Our present approach is to satisfy a twofold goal. The first aims at realizing an x-ray telescope with a resolution of 5''-10'' immediately, as all the basic ingredients to achieve that are available. We are going through many tests and qualifications to raise the Technology Readiness Level. Secondly, the advance in mirror fabrication and mounting will serve as a basis for systems with arc-second resolution. Here, we focus on addressing the development of mirror coating. The important issue of mirror alignment and mounting is discussed in a separate contribution<sup>11</sup>.

We have studied coating stress with different methods of deposition, and of different metals<sup>12</sup>. Our choices for high density metal for x-ray reflections include gold (Au), platinum (Pt) and Iridium (Ir). Among them, Ir has the highest density and reflectivity at the soft x-ray band, and is preferred for our application. Fundamentally, metal such as Ir has high enough film stress to cause low order distortion.

Metallic deposition for lightweight mirror has to meet two requirements. First, the deposition on the originally smooth substrate must produce a smooth surface with sub-nm roughness. This is more important for reflection of harder x-ray (a few to 10 keV.) Reflection of x-rays with  $E > 10$  keV is not effective with a single film but can be attained with multi-layer coating. Secondly, the coating must be sufficiently relaxed so that coating stress produce only negligible distortion to the thin mirror. In our present pursuit of an arc-second telescope, the coating distortion is to be limited to an arc-second or less. Coating stress can impart on a mirror low order axial curvature, such as axial sag. For such distortion, the resolution requirement translates to about 0.1  $\mu\text{m}$  for a mirror we typically work with (200 mm).

## 1.2 Coating Stress

We have used magnetron sputtering and atomic layer deposition to provide efficient coating of mirrors. Sputtering is especially suitable because of its ease of deposition for metals with high melting point. The efficiency in coating single layer, typically about 20 nm thick to provide sufficient reflectivity, is an important advantage. However, sputtering at room temperature produces thin film with high stress<sup>12,13,14</sup>. Magnetron-deposited Ir (or other metal such as Cr or Pt) films of about 10 nm in thickness can have stress of a few GPa. Such high integrated stress (stress x thickness), of tens of Pascal.meter (Pa.m), distorts the figure of the 0.4 mm thick mirror significantly. For iridium, the coating distortion is about 3  $\mu\text{m}$  PV for every 10 nm deposited. Figure 1 shows the measurement, with interferometer coupled with a cylindrical null optics<sup>18</sup>, of a typical distortion exhibiting high convex axial profiles at the two azimuthal edges.

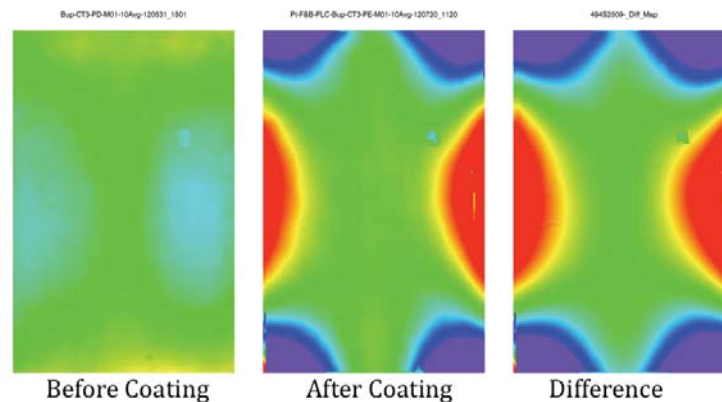


Figure 1. Coating distortion of thin glass mirror. The surface maps show the surface profile of approximately conical shaped mirror segment before and after coating. The substrate is thermally formed from Schott D263 glass. It is 200 mm long axially (vertical) and 0.4 mm thick. The deposited material is iridium. The film is magnetron-sputtered with a thickness of 20 nm. The distortion is very significant at 6  $\mu\text{m}$  (PV). The axial profiles at the azimuthal edges are convex (color coding: red: out of the surface; blue: into the surface.)

We have also used atomic layer deposition for coating. Atomic layer deposition potentially offers another film growth mechanism that may be low-stress and the film may be more uniform. In addition, atomic layer deposition is intrinsically simultaneous on all surfaces. Simultaneous coating on the front side and backside of the mirror can provide a natural cancellation of stress-induced distortion. However, extensive studies using atomic layer deposition did not produce undistorted mirrors as expected. We will discuss the result from atomic layer deposition below.

The mirror distortion can be modeled by applying a given stress on the surface of a mirror. Its dependence on the film's and substrate's thicknesses, as well as film stress and substrate stiffness, can easily be understood by considering a flat. For a flat, a uniform coating stress generates additional curvature  $K = 6(1-\nu)\sigma\tau_f/E\tau_s^2$ , where  $\nu$ ,  $E$  are the Poisson ratio and the Young's modulus of the substrate,  $\tau_s$  and  $\tau_f$  are the thickness of the substrate and film, respectively. It can be seen that the curvature generated by the film is proportional to the integrated stress ( $\sigma\tau_f$ ) of the film. For a 20 nm film with 3 GPa stress on 0.4 mm glass substrate, the curvature is  $2.5 \times 10^{-4} \text{ m}^{-1}$ , which is significant distortion if impart on our mirrors. Since our substrate is not flat, but is nearly cylindrical in shape, the stiffness along the axial direction is much stronger than in the azimuthal direction. The sag of the mirror does not have a single value illustrated in the example above, but rather depends on the mirror's azimuth.

Figure 2 shows the result from a finite-element model, with 5 GPa stress, 20 nm thick film deposited on a curved glass mirror. In the model, the thickness distribution along the axial direction (vertical) is not uniform but instead matches that measured from our magnetron sputtering experiment (that is about 20% thinner at the ends than at the middle.) This distribution matches that from actual deposition.

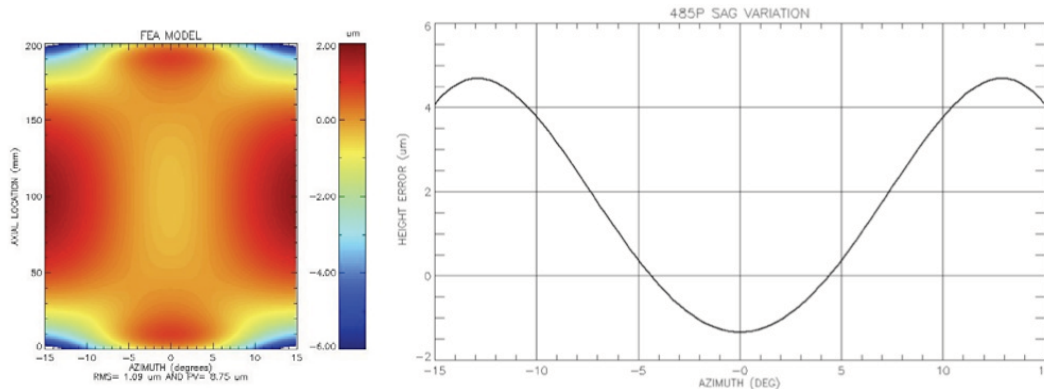


Figure 2. Surface figure distortion from finite-element model of a thin film with a stress of 5 GPa and a thickness of 20 nm. The substrate is a 0.4 mm thick glass 200 mm long (vertical axis) spanning 30° azimuthally with a diameter of about 485 mm. (Left) The radial deviation of surface. Blue color coding is into the surface (radially larger). (Right) Axial 2<sup>nd</sup> order sag as a function of azimuth. The turning around of sag at the azimuthal ends is due to non-uniformity of coating in the axial direction.

## 2. EXPERIMENTAL APPROACHES

To eliminate mirror distortion, we took three general approaches. Fundamentally, it is ideal to reduce the internal stress of the film. Alternatively, mirror distortion can be minimized by balancing distortions resulted from stresses of different films. For the second approach, we combined films with opposite stresses, and we also deposited film on the front side and backside of the mirror. Some of these results are reported previously in these conferences and will only be summarized briefly here. Finally, one can consider correcting the figure error with other controllably produced stresses, such as a differential sputtering<sup>15</sup>, magneto-strictive deposition<sup>16</sup>, or ion implantation of the substrate<sup>17</sup>.

### 2.1 Cr/Ir Bi-layer Deposition

The iridium film deposited with magnetron sputtering has a compressive stress of about 4 GPa. We took advantage of the fact that chromium deposition produces a film of tensile stress. By combining an Ir and a Cr layer with a proper thickness ratio, an effective zero stress bi-layer film may be obtained. To achieve negligible mirror distortion, a Cr film can be deposited as an under-layer with the Ir film on top. Such a combination of deposition was made by Reflective X-ray Optics LLC (RXO), New York. Individual stresses of magnetron-sputtered Ir and Cr films were first measured. The combined stress was then calibrated for an Ir film with a fixed thickness on wafers by depositing Cr films of different thicknesses. The correct combination is obtained when the bi-layer reaches a total zero combined stress. For 20 nm Ir, 15 nm of Cr is needed. To confirm, 22 nm of Cr was found to balance 30 nm of Ir.

Ir/Cr films with the correct thickness ratio were then deposited on curved substrates. The resulting mirrors, however, were still significantly distorted. This fact was also confirmed with deposition of 3 combinations of films with different targeted total stresses for about 200 MPa, 400 MPa, and 800 MPa. Ir thickness was fixed at 16 nm. The result, however,

did not show the expected trend of increasing distortion with the correct magnitude. The residual stresses on the mirrors still produced a 1-2  $\mu\text{m}$  of sag variation. We concluded that the balancing of film stresses did not work probably due to the different spatial distribution of the depositions of the two metals (the calibration was done with small wafers), and the stresses are not sufficiently reproducible to generate a perfect balance of stresses.

### 2.2 Coating on the Front and Back Sides

Another way to balance the effect of mirror distortion is to deposit films on both sides of the substrate. The films from the front side and back side act to deflect the substrate in opposite directions. Coating the backside of the mirrors is also advantageous in providing a better radiative coupling between highly packed mirrors and therefore giving a smaller thermal gradient for the telescope in space. For this to work, a mirror has to be coated on both sides with films with the same thickness and stress distributions (strictly, only the same distribution of the integrated stress,  $\sigma \tau_f$ , is needed), then the effect from coating stress can be balanced. Precise coating at a few percents is required, as we estimated a precision of  $\sim 1 \text{ Pa}\cdot\text{m}$  effective integrated stress out of a  $\sim 40 \text{ Pa}\cdot\text{m}$  original films.

In our experiments, Ir was magnetron-sputtered onto full-size glass substrates in separate depositions at the front and back surfaces. Nominal 20 nm films were deposited. Surface measurements were made before the front side deposition, after the front-side deposition and after the backside deposition. We found that the axial sag changes were as expected after Ir films were deposited on the front side of the mirrors. The changes from the backside depositions are similar but the magnitude was different at the middle azimuth of the mirrors. We concluded that the difference resulted from the different geometry of mirrors oriented facing towards and opposite the target in the coating chamber. The middle azimuths of the mirrors simply are at different distances to the target when the concave and convex sides of the mirrors are facing the target. In principle, the turntable in the sputtering chamber can be configured so that the film's angular distribution due to different substrate-target distance may be compensated. However, like balancing mirror distortion with bi-layer deposition, the reproducibility of the coating in such balancing act was considered not sufficient. We opted to pursue a more precise coating balance with atomic layer deposition.

### 2.3 Atomic Layer Deposition

Atomic layer deposition (ALD) is an attractive coating method that coats the front and back sides of a mirror under the same condition simultaneously. The chemical deposition allows conformal deposition simultaneously on all surfaces. Films were grown on all sides, including the edges, of the mirrors. In this way, the front and back sides of the mirror is expected to achieve a much more precise balance. ALD coating potentially can also produce films with lower stress.

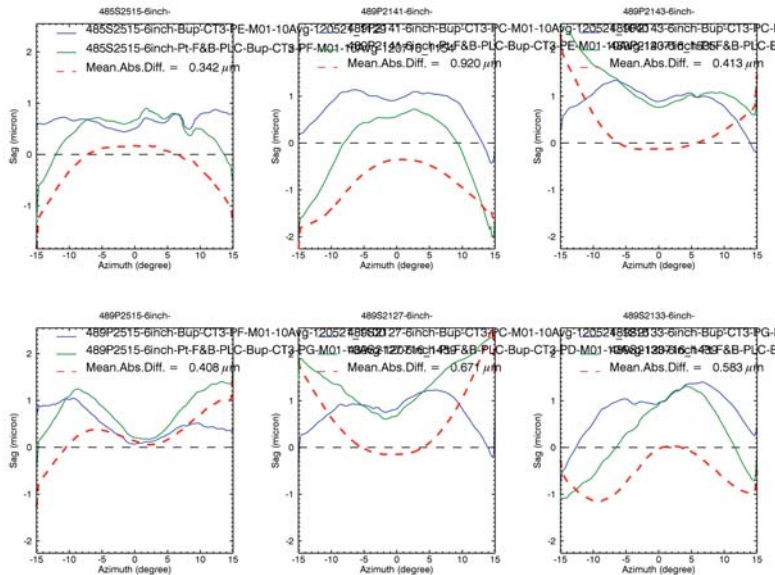


Figure 3. Axial sag variations after deposition of platinum on curved mirrors with atomic layer deposition by Arradience, Inc. Six mirrors are shown. The axial sags as functions of azimuth before and after coating are compared (blue: before coating, green: after coating, red dash: difference.)

ALD deposition typically operates at temperatures of several hundred Celsius, and at pressures of hundreds of mTorr to 1 Torr. At such pressure and temperature, the mean free path is sub-mm and the gas is transported as a fluid continuum. This ability of the gas to get into all surfaces even within a small space is useful for our application. Besides coating on both sides of the mirror's surface, many mirrors can be coated simultaneously while closed packed, improving coating efficiency. Each ALD coating run takes much longer time, however, as mono-layers of materials have to be deposited in repetitive cycles. Hundreds of cycles are typically needed for  $\sim 20$  nm of deposition (at a rate of  $\sim 1.0 \text{ \AA} / \text{cycle}$  or less.) For our experimentation, we used several service providers for our studies of ALD coating: Beneq (Vantaa, Finland) did the coating of iridium, while Cambridge NanoTech (Cambridge, Massachusetts, USA), and Arradance (Cambridge, Massachusetts, USA) coated platinum. All were done on typical curved glass substrates with a Wolter-I design. Figures 3-5 show several examples of the resulting changes in axial sag variation from the depositions carried out by each of the vendors.

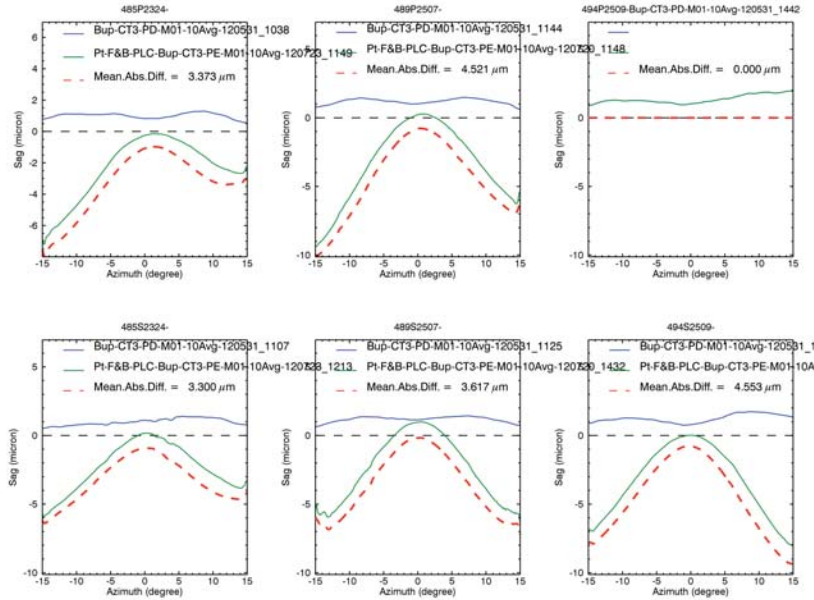


Figure 4. Axial sag variations after deposition of platinum on curved mirrors with atomic layer deposition by Cambridge NanoTech. Five mirrors are shown in a set of 6 depositions (one mirror, listed also in the upper right plot for completeness, was damaged in the process and no data could be obtained from it.) The axial sags as functions of azimuth before and after coating are compared. The color codes are the same as in Figure 3.

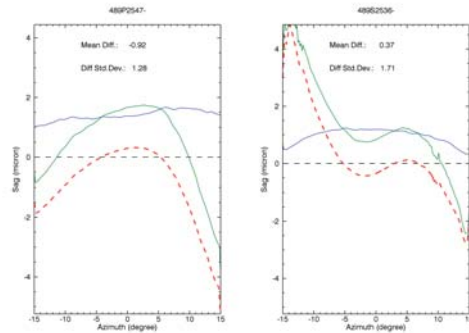


Figure 5. Axial sag variations of iridium on curved mirrors with atomic layer deposition by Beneq. Two mirrors are shown. The axial sags as functions of azimuth before and after coating are compared. The color codes are the same as in Figure 3.



As can be seen, the atomic layer deposition did not automatically return a mirror without distortion, as hoped for from a true front-and-back compensation of film stresses. It can also be concluded that a straight application of ALD coating did not have low enough stress that meet our needs for coating lightweight thin mirrors.

For the ALD coating of Pt by Arradiance, the full range of axial sag variation due to coating is about 2  $\mu\text{m}$  (red curve in Figure 3), and their dependence on azimuth varies, from second order to third order, and with different signs. The smaller (about 30%) magnitude in sag variation, compared to magnetron sputtered film with the same ( $\sim 20$  nm) thickness, implies that perhaps some compensation between the front and the back surfaces were achieved, but the film stress is just as large. We did not pursue a calibration of the film stress due to this unsatisfactory result. This non-repeatability from coating to coating also indicates that the deposition is not steady spatially, perhaps due to sensitivity in nucleation of Pt atoms on the glass surface.

The coating done at Cambridge NanoTech exhibited rather different characteristics. The magnitude of sag variation is large. In fact, it is approximately the same as those obtained from magnetron sputtering, at about 6  $\mu\text{m}$  for the  $\sim 20$  nm thick Pt film. The change in axial sag variation due the coating, however, was very consistent not only in magnitude, but also in azimuthal dependence (quadratic). It is unclear what the difference between the two ALD coatings done by the two vendors are (as detailed coating information is proprietary and is not known to us.) Nevertheless, it can be concluded that the Pt films deposited by this vendor has consistently high film stress, perhaps as high as 5 GPa (higher if some compensation between front and back surfaces were achieved.)

Figure 5 shows a pair of mirrors coated with Ir at Beneq. This company is the only vendor that we used which provided atomic layer deposition of iridium (a preferred metal for better x-ray reflectivity.) It is also the only company that has a sufficiently large chamber to accommodate mirrors that can be oriented vertically or horizontally. Indeed, we fabricated mirror holders for coating for the particular purpose of testing the ALD coating in vertical and horizontal directions, as the flow of precursor and gravity sag are different for mirrors in different orientations. Result from coating from Beneq is similar to those from Arradiance in that the change in axial sag variation is not repeatable. The magnitude of impact from film stress is also generally larger, but it can be due to the fact that Ir, rather than Pt, was used instead. It should also be noted that the Ir process requires a high operating temperatures, at about 350°C. The thermal effect from the cooling down of the film on a mirror may have significant impact on the mirror's distortion. We will address this further in the following section on thermal annealing.

### 3. ANNEALING OF METALLIC FILM

Since neither our approaches to balance the film stresses nor to use atomic layer deposition worked to our satisfaction, we went back to visit the possibility of reducing the internal stress of the film. The coating stress can be minimized by modifying the condition of deposition. In the case of sputtering, dependence on gas type, pressure, potential difference at the cathode, substrate to target distance, etc., can be optimized. Post-coat annealing can also be used to improve the mirror's figure. Thermal annealing, however, is very limited since the substrates were thermally slumped at  $\sim 600^\circ\text{C}$ , and prolonged heating above 400°C will begin to soften the mirror and change its figure. Nevertheless, we began with thermal annealing of magnetron-sputtered films to explore the boundary of the annealing parameter space.

In addition to the requirement that any thermal treatment must not distort the glass substrate itself at the temperature that can relax the deposited film, the annealing must not degrade the micro-roughness of the film. For magnetron-sputtered films, we confirmed that these requirements are satisfied at a temperature of 350°C, for a soaking time of 8 hours (plus a few hours of temperature ramp-up and cool-down). Surface roughness was measured with an optical surface profiler (Zygo NewView). The rms roughness was spot-checked to be 0.3 – 0.4 nm, at a field of view of 100 – 300  $\mu\text{m}$ , for a number of mirrors before and after thermal annealing. Further confirmation will be done with x-ray reflectivity measurements.

#### 3.1 Annealing of Magnetron-Sputtered and Atomic Layer Deposited Films

We also confirmed that the substrates were stable after heating them up to 400°C (for a soak time of 8 hours). Some examples are shown in Figure 9. At 350°C, annealing of both magnetron sputtered films (Figure 6) and atomic layer deposited iridium films (Figure 7) were demonstrated. The film relaxation was not perfect as there was residual distortion. The magnitudes of axial sag distortion, however, were significantly reduced. In the case of magnetron-sputtered film, the factor of reduction is about 5 for the 20 nm films. In the case of ALD deposited film, it is about a factor of 4.

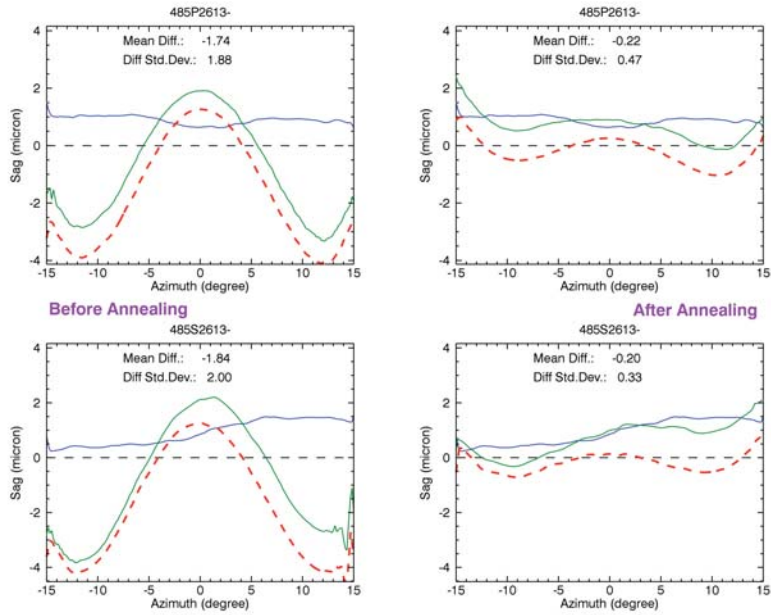


Figure 6. Effect of thermal annealing of magnetron sputtered iridium coating at 350°C, for a soak time of 8 hours. The coating thickness is 20 nm. Two examples are shown here, with mirror ID 489P2613 on the top row and 489S2613 on the bottom row. The left column shows the change in axial sag variation after the deposition (blue: original substrates, green: after deposition, red: difference). The column on the right shows the change after thermal annealing (blue: original substrates; green: after thermal annealing.) The film relaxation from the thermal treatment is not perfect, but the magnitude of variation was changed, by a factor of 4 to 6.

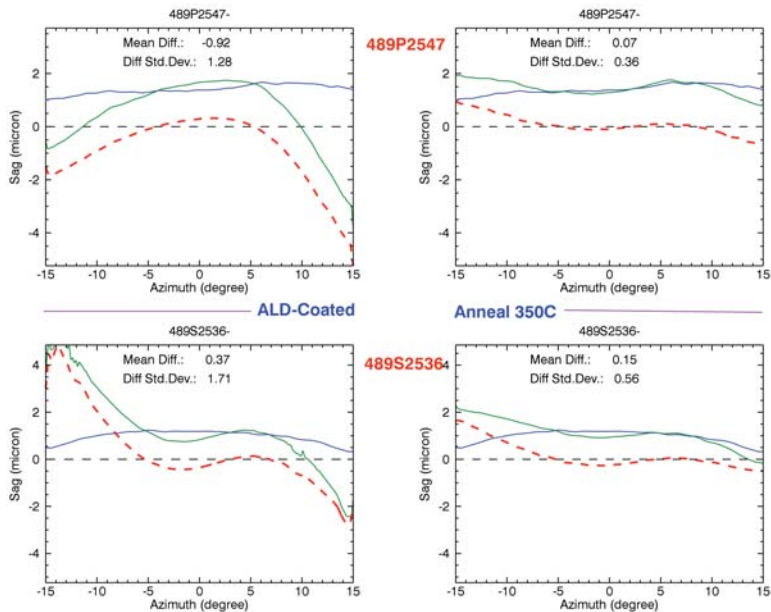


Figure 7. Relaxation of atomic layer deposited iridium film annealed at the same condition as in Figure 5 (350°C, for a soak time of 8 hours). Two mirrors are shown on the top and bottom rows. The left column shows the change in axial sag variation after the deposition (blue: original substrates, green: after deposition, red: difference). The column on the right shows the change after thermal annealing (blue: original substrates; green: after thermal annealing.) Like the annealing of magnetron sputtered films, relaxation of the coating stress is not complete, but the magnitude of variation was reduced, approximately by a factor of 4.

### 3.2 Dependence on Film Thickness

To further reduce the residual distortion, we studied the dependence on film thickness. A set of 6 mirrors were prepared, with magnetron-sputtered iridium coating in 3 different thicknesses: 5, 10, and 20 nm. There were 2 mirrors with identical conditions for consistency check. The substrates are thermally slumped glass cut to size. To prepare for consistent coatings, the substrates were cleaned with a controlled washing procedure and contaminants were further removed in an ultrasonic cleaner. A thorough process turned out to be important to eliminate any patchy defects after annealing. As described before, interferometric surface measurements were made for the substrates, the coated mirrors and the mirrors after annealing. Substrate annealing process was the same (350°C, with an 8 hour soak).

The resulting axial sag variation, as a function of film thickness, is shown in Figure 8. For the coated mirrors without annealing, the distortion varies linearly with thickness, as expected. The change in sag variation is generally negative (see Figure 6). Both the average and rms sag variation depend on film thickness with a rate of 0.1 μm for each nm of deposition. With annealing, the dependence is no longer linear, but with an optimum at around 10 nm. The mean sag variation goes through zero at that thickness while the rms attains a minimum. It is interesting that the annealing is not effective for thicker films. 350°C is probably too low an annealing temperature for the high melting point metal. Another possible cause is the thermal distortion from CTE mismatch between the iridium film and the glass substrate, as the mirror was cooled down from high temperatures. It is also possible that interface effect at the boundary between the iridium layer and the glass affects the thinner films. We will discuss this further in a later section.

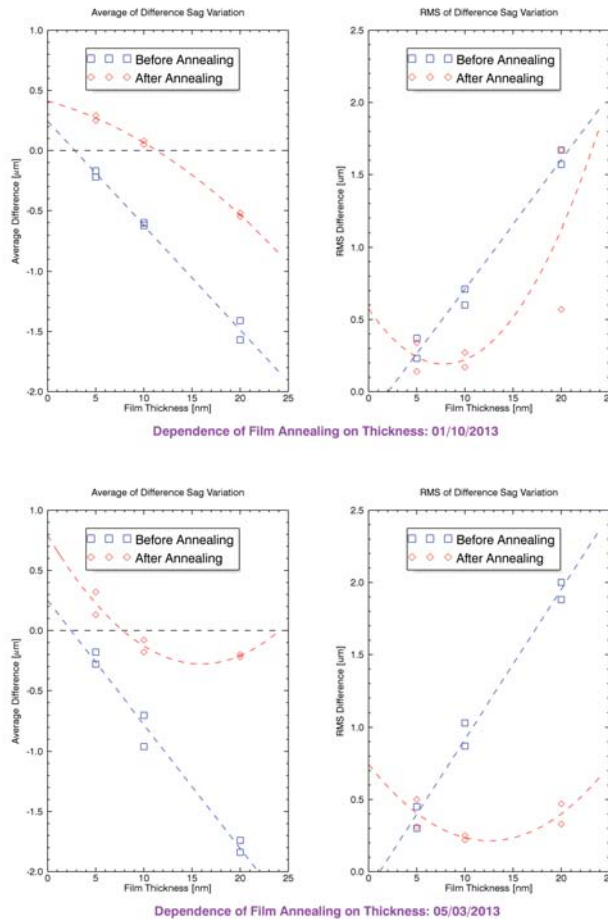


Figure 8. Dependence of change in axial sag variation on film thickness in annealing. The left panels show the trend of average change in sag variations. The right panels show the trend of rms change of sag variation. The bottom row shows the result of a repeated experiment, for consistency check. The sag variation exhibits a linear dependence on thickness for un-annealed film, as expected. The sag variation reaches an optimum after annealing, at a film thickness of about 10 nm.



We note that, for our application, there is a requirement on the thickness of the coating, in order to obtain sufficient x-ray reflectivity. For application in the soft x-ray band up to 10 keV, 15- 20 nm of iridium is required. The fact that the optimum is not lower than 10 nm is fortunate for us. From the experimental result, a film of 20 nm is not unacceptable, due to the slow changing behavior of the thickness-sag curve in the 10 – 20 nm region. We summarize the observation in the following table.

Table 1. Summary of experimental result from annealing iridium films deposited with different thickness.

	After Coating	After Thermal Annealing
Change in Mean Sag	-The deposited film’s impact on mean sag is generally negative. -The change is linear. The rate is about 0.1 $\mu\text{m}/\text{nm}$ of Ir deposition.	-The mean sag at larger film thickness remains negative while that for smaller thickness is reversed -Near-zero mean change at thickness $\sim 10$ nm
RMS sag variation	-The rms difference is linear with Ir thickness. -The rate is also 0.1 $\mu\text{m}/\text{nm}$ . PV is 0.3 $\mu\text{m}/\text{nm}$ .	-The rms difference is not monotonic -Optimal at about 10-15 nm of Ir (for the given annealing condition.)

### 3.3 Dependence on Annealing Temperature

The residual mirror distortion from annealing was further optimized with respect to annealing temperature. In the experiment, a set of mirrors were prepared with the same film thickness (10 nm), and were soaked at different temperatures. The test temperature range covers  $T = 260^\circ\text{C}$ ,  $290^\circ\text{C}$ ,  $320^\circ\text{C}$ ,  $350^\circ\text{C}$  (from previous tests),  $380^\circ\text{C}$ , and  $410^\circ\text{C}$ . The thermal soak time is fixed at 8 hours. The preparation and cleaning of the glass substrates were the same as described before. The coating on the mirrors was uniformly 10 nm, based on the previous tests. There were two mirrors for each testing condition, again, for consistency check. To make sure that the substrate were not distorted at higher temperature, an extra substrate was placed in oven to go through the same thermal cycle, for the mirrors soaked in the higher temperature than  $320^\circ\text{C}$ . The result in that range is shown in Figure 9 (result at  $350^\circ\text{C}$  is not repeated.)

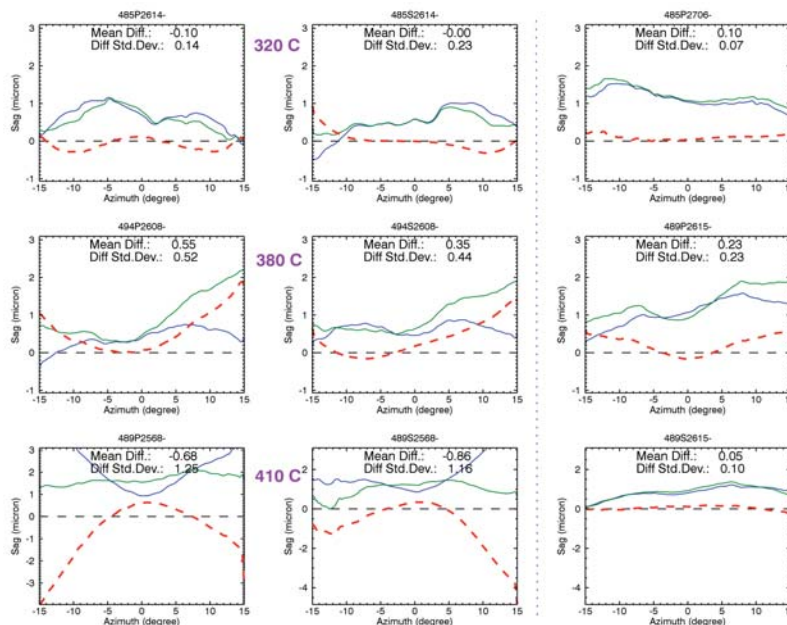


Figure 9. Sag Variation from Annealing at different temperatures ( $320^\circ\text{C}$ ,  $380^\circ\text{C}$ ,  $410^\circ\text{C}$ ). The color coding is the same as in Figures 6 and 7. The right column shows the change in sag variation of bare substrates heated in the same thermal cycle as the annealed segments. It demonstrated the stability of substrates against heating. (The anomaly in the  $380^\circ\text{C}$  may be due issues in measurement. However, its actual cause is unclear.)

The average and rms sag variation change is plotted in Figure 10. As for the dependence on film thickness, there is a minimum distortion, in terms of rms sag variation, corresponding to a temperature of 313°C. The poor film relaxation at lower temperature is, naturally, normal. The rapid worsening of residual mirror distortion at higher temperatures demands a special explanation. A possibility is that, at higher temperature, even though the films are better relaxed, the thermal stress arising from CTE mismatch between the iridium films and the glass substrates might have put additional distortion on the mirrors, as they were cooled down from high temperatures. The result is that the CTE effect completely dominates as the thermal relaxation saturates at high temperature. The same effect may explain the dependence on film thickness, as the thermal integrated stress is generally proportional to the temperature difference and film thickness.

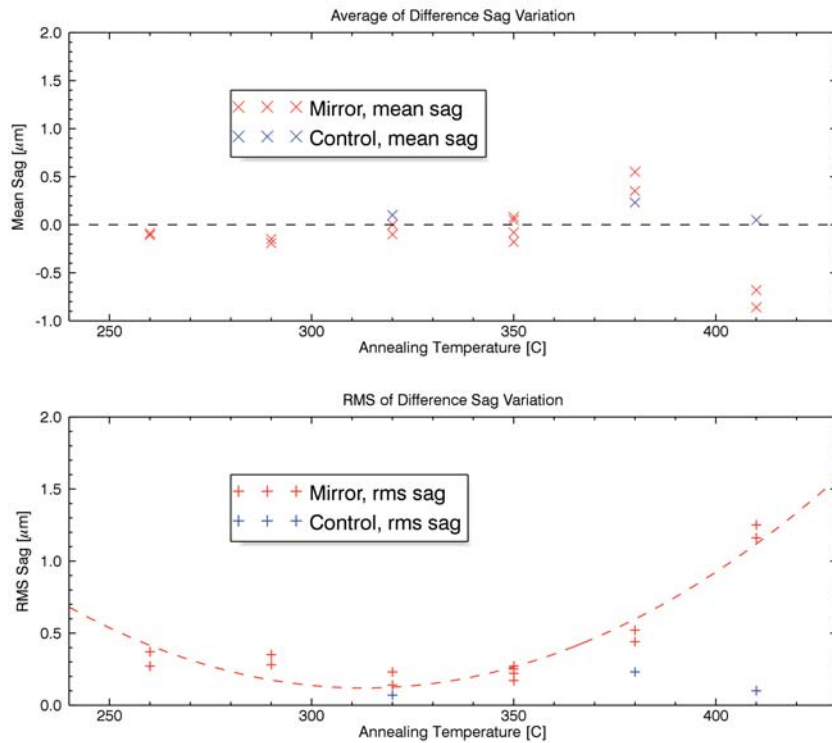


Figure 10. Dependence of change in axial sag variation on annealing temperature. The top graph shows the dependence of average change in sag variations. The bottom panel shows the trend of rms change of sag variation. At higher temperatures, the sag variation increases again beyond about 350°C. The larger distortion may come from thermal stress arising from CTE mismatch between the iridium films and the glass substrates.

We attempt to understand this larger thermal stressing at higher annealing temperature or for thicker films by modeling the effect. A simple model, using temperature-independent CTE and elastic modulus, is shown in Figure 11. The constancy of CTE and modulus parameters were justified on the basis that thermal expansion and elastic strain of the Schott glass were measured to be linear in the temperature range up to about 400°C. Those for iridium are expected to be linear, too, for the high melting point metal and the small strain. The model, however, only produces distortion at least an order of magnitude of what was observed. A finer model may be needed to establish conclusively this as the main cause of the observed effect.

Heating the mirror in the moderately high temperature therefore provide a simple and practical solution to the coating problem. The optimized temperature at about 310°C is sufficient low to not distort the substrate in any way. The residual distortion, at only 0.2 µm rms and essentially negligible change in axial sag, over 200 mm long mirrors, represents about 1-2" of error in mirror figure. It is considered compatible with other errors in our present state of mirror fabrication. Detail modeling of thermal stresses from the annealing is on-going to understand the cause of the remaining distortion.

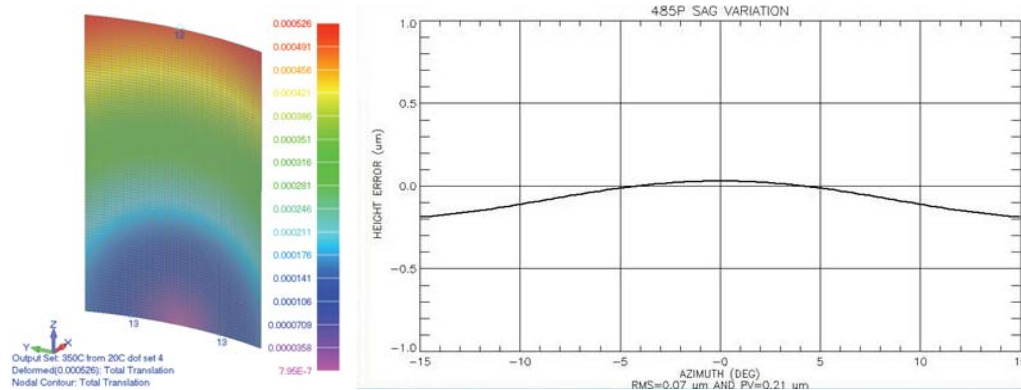


Figure 11. Finite element modeling of the mirror distortion from thermal stress arising from bi-layer effect between an iridium layer and the glass substrate. The Ir film is 20 nm thick, and the glass (Schott D263) substrate is 0.4 mm. The simple model uses temperature-independent material properties which is a reasonably justified assumption for the temperature range considered, from 20°C to 350°C. This analysis only gives a small fraction of the distortion observed.

#### 4. CONCLUSION

Coating distortion of thin glass mirror was studied in order to produce x-ray reflecting mirrors with arc-second resolution. Coating stress compensation by depositing bi-layers with opposite coating stress was studied before and was shown not to be adequate. The attempt to balance the mirror distortion by coating on both sides of the mirrors with either magnetron sputtering and atomic layer deposition was not successful. Heating the mirrors after coating turns out to be an effective way to relax the film stress. At the temperature range we investigated, the heat treatment did not alter the glass substrate. The surface roughness was not degraded either. There is a small residual mirror distortion remaining, however. The small error represents about 1-2'' contribution to the overall mirror HPD.

The residual mirror distortion was found to depend on film thickness. Before annealing, the deposited film produces a mirror distortion that is linear with thickness, as expected. With annealing at a fixed temperature, 350°C in our case, the stress relaxation is not effective both for thick and thin films. The optimal thickness is around 10 nm. The residual distortion was found also to depend on the heat soak temperature, non-monotonically. It is interesting that, at higher temperature, the annealing did not continue to improve the figure recovery from coating stress. The film relaxation is likely to be actually better but the associated thermal stress may have caused a thermal distortion that grows with temperature difference. The optimal temperature for 10 nm thick iridium films were found to be about 310°C. At this optimized condition, the residual distortion, in terms of axial sag variation, is only about 0.2 μm (rms), which is close to measurement uncertainty.

In summary, post-deposition heat treatment of the mirrors produces mirrors with quality compatible with our requirement, only adding about 1''-2'' of error. The heat treatment therefore provides a practically useful solution to our coating problem. It is conceivable that further reduction can be achieved from partial cancellation of the residual distortion from thermal stress by coating both sides of the mirror. Coating on both sides is desired also for reason of better radiative coupling between closely packed mirrors in a telescope.

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