Reflective Coating for Lightweight X-ray Optics

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ABSTRACT

X-ray reflective coating for next generation's lightweight, high resolution, optics for astronomy requires thin-film deposition that is precisely fine-tuned so that it will not distort the thin sub-mm substrates. Film of very low stress is required. Alternatively, mirror distortion can be cancelled by precisely balancing the deformation from multiple films. We will present results on metallic film deposition for the lightweight optics under development. These efforts include: low-stress deposition by magnetron sputtering and atomic layer deposition of the metals, balancing of gross deformation with two-layer depositions of opposite stresses and with depositions on both sides of the thin mirrors.

Keywords: Times Roman, image area, acronyms, references

1. INTRODUCTION

Lightweight segmented grazing incidence x-ray telescopes have been built at NASA/Goddard Space Flight Center since the early 1990's. Similar to their full-shell counterparts, these telescopes consist of two stages of concentric shells which focus x-ray in two reflections. The basic designs follow the Wolter Type I prescription or its approximate double conical form. Segmentation, as oppose to full 360° shells, allows such telescope to be integrated from smaller units to full telescopes with simplified fabrication procedure and lower cost. Many x-ray astronomical missions with such segmented lightweight optics were flown. The X-Ray Telescopes onboard the Advanced Satellite for Cosmology and Astrophysics (ASCA) and Suzaku missions were the first of this kind built at NASA/Goddard in collaboration with Japan's JAXA. This tradition is to be followed with the upcoming mission Astro-H. The recently launched hard x-ray mission NuSTAR also carries segmented lightweight optics made with glass substrates.

The segmented approach, allowing telescopes of essentially any size to be built from small mirror modules, therefore remove much of the challenge of making shells of very large diameters. For such optics, in addition, the use of very thin substrates fits naturally with segmentation, therefore allowing very lightweight and compact telescopes to be realized. However, if large effective area is its strength, the angular resolution is currently its limitation. So far, such telescopes generally have lower angular resolution, even though there is no fundamental reason why it should necessarily be so. Typical contrasts of these aspects can be seen in optics in the Chandra vs. Suzaku missions. Further development of such segmented lightweight optics would necessarily be to improve its resolution, and indeed, much improvement has been made in recent years.

Part of the reason for the generally lower resolution for these thin mirrors is the lack of structural rigidity due to the mirrors' thinness---they are typically a few hundred micrometers thick. The lack of constraint from a full 360° shell does not help either. Procedures from cutting the substrates, depositing metallic film for x-ray reflection, mounting the mirrors to integrating the modules all can be potential source of distortion. Gravity sag/relief and thermal stress are also contributing factors in ground tests and in space.

In this paper, we will concentrate on the contribution of coating stress on the distortion of typical glass substrates and our effort to limit it. Glass substrates are used in the aforementioned NuSTAR mission, and were developed for the optics of the mission concepts Constellation-X and International X-ray Observatory. The coating is generally a single layer of high-Z materials (e.g., Ir, Pt, Au, etc.) for good x-ray reflectivity in the soft x-ray band around a few keV. For harder x-

ray multi-layer coating, such as those used for the optics in NuSTAR and the Hard X-ray Telescopes on Astro-H, are needed.

Even though, in principle, a mirror substrate can sustain a certain level of distortion, if subsequent alignment/mounting processes is able to correct the mirror figure. However, such corrective mounting method has to be developed. Presently, at the level of angular resolution that we are aiming at, namely, ~ arc-second, no such correctly method is experimentally demonstrated. Our basic working principle is therefore to maintain the mirror's figure as much as possible in the coating process (as a matter of fact, also in all subsequent processes). That this is a good working principle is supported by the fact that glass substrates produced at NASA/Goddard have attained good (about a few arc-seconds) figures and the resolution has been improving; and that precise (also at the level of a few arc-seconds) bonding techniques has been developed. It is our belief that this working principle---to produce substrates at the highest resolution and limit deterioration of all subsequent processes---is feasible and is currently most likely to allow building of an x-ray telescope at 1 arc-second resolution or better.

1.1 Metallic Overcoat

Glazing incidence Wolter Type I optics or their approximate forms are the most common design of astronomical telescopes. In fact, all x-ray astronomical telescopes to date use for focusing x-ray the high reflectivity, at low grazing angle, of x-ray from smooth surfaces of high-Z materials (metal with high atomic numbers such as Au, Pt, and Ir.) There are at least two requirements for such application. First, the substrates and the deposition of metallic material must produce a smooth, sub-nm roughness, surface. This is more important for reflection of harder x-ray (longer wavelengths), up to 10 keV. Reflection of x-ray with $E > 10$ keV usually requires multi-layer coating. Secondly, the coating must be sufficiently relaxed so that any remaining coating stress must not distort the mirror beyond the telescope's requirement. In our present pursuit of an arc-second telescope, that means the coating distortion is to be limited to an arc-second or less.

Magnetron sputtering has been used to provide efficient coating of mirrors. This method of deposition is especially suitable for coating of the thin shell grazing mirrors because of its ease of deposition and high speed. Unlike normal incidence optics, a grazing incidence telescope may have actual mirror coating area 2 orders of magnitude larger than the aperture. The physical mirror surface area is larger for telescopes of larger focal length and grazing angle. Nevertheless, magnetron can produce thin film with high stress. In our experimentation, magnetron deposited Ir (or Cr, Pt) films of about 10 nm in thickness can have stress of GPa. Such high stress distorts the figure of the 0.4 mm thick glass mirrors we currently use beyond the arc-second that we require.

1.2 Approaches

To eliminate such distortion, we take two separate approaches: The most obvious is to improve the sputtered film's quality by reducing its internal stress, or use an alternate coating method that is intrinsically more relaxed. Another approach is to cancel the film's distortional action by applying it to both the front side and the backside of the substrate.

To obtain a low stress film, we approach this with a deposition of Ir-Cr bi-layer, aiming to achieve a zero effective stress of the combined film. Ir and Cr films, as magnetron-sputtered in room temperatures, have rather large stresses of more than 1 GPa, but with opposite signs (Ir is compressive; Cr is tensile). By choosing their thicknesses properly, we may get an effect stress that is sufficiently low for our purpose. We will discuss the result of such experimentation in Section 3.

Another way to balance the effect of mirror stress is to deposit films on both sides of the substrate. The films from the front and back sides will act to stress the substrate in opposite senses. There are also other advantages for coating the back side of the mirrors, for instance, in thermal considerations of these closely packed mirror segments. For structural consideration, the key is that the substrates are not flat and some adjustment may be made in order to deposit the right amount of materials so that the effect from their stresses will cancel. We will address these issues in Section 4.1.

In magnetron sputtering, the sputtered atoms come approximately from the direction of the target. Any backside film will have to be deposit separately. For the purposing of cancelling effect of film stress with a backside coating, a better deposition method will be an isotropic deposition. For this purpose, we investigated atomic layer deposition (ALD) which is an isotropic deposition and coat the both sides (and the edges) of the mirrors simultaneously. We will report preliminary results from such tests in Section 4.2.

2. COATING AND MIRROR DISTORTION

For the purpose of this paper, we will discuss coating stress and distortion in the context of glass substrates we have developed originally for the Constellation-X and International X-ray Observatory mission concepts. Both of these missions required optics of over 1 m in diameter, to provide over 10,000 cm^2 of effective area at 1 keV soft x-ray. The mass limits require mirrors' areal density of about 1 kg/m^2, or about 0.4 mm thickness for the substrate, if glass of density 2.5 g/cm^3 is used. A baseline 0.4 mm thick borosilicate glass substrate (Schott D263 glass) was chosen for these projects. It should be noted that, the telescopes on board NuSTAR, used the same glass with 0.2 mm thickness.

For the segmentation, we have chosen to use 200 mm long segments. The angular span depends on radial position of the mirror shell, with smaller angular segmentation at larger radius, in order to maintain a reasonable aspect ratio of the mirror and also better stiffness, which are important in the precision alignment and bonding process. For mirror radius around 250 mm, 30° angular span has been chosen. (See reference [1] for various considerations and optimization.) We will base our experiment on mirrors with these dimensions. It should be noted that other mission specification will demand different requirement for coating, and not all substrates are made of glass. In fact, the missions that started this all, the ASCA and Suzaku missions, had optics that made of aluminum substrates, and they are also thinner (about 0.15 mm thick). The mission concept Athena uses silicon as substrates. However, these optics have rather different mounting schemes and resolution specification, and therefore coating requirements.

To have high reflectivity for soft x-ray (from < 1 keV to 10 keV), even at grazing incidence, coating of high-Z metal is needed. In general, for sufficiently thick films, the reflectivity for x-ray drops significantly when the grazing angle is large. The 'critical angle' depends on the incident x-ray energy and is lower for higher energy. Roughly, critical angle θ_c ≈ √(2δ), where δ is the decrement of the real part of reflective index: $n_R = 1 - δ$. The decrement δ depends on incident x-ray energy E as ~ E^{-2}, therefore, θ_c ~ $1/E$. The dependence of x-ray reflectivity on incidence angle for a few x-ray energies is illustrated in Figure 1.
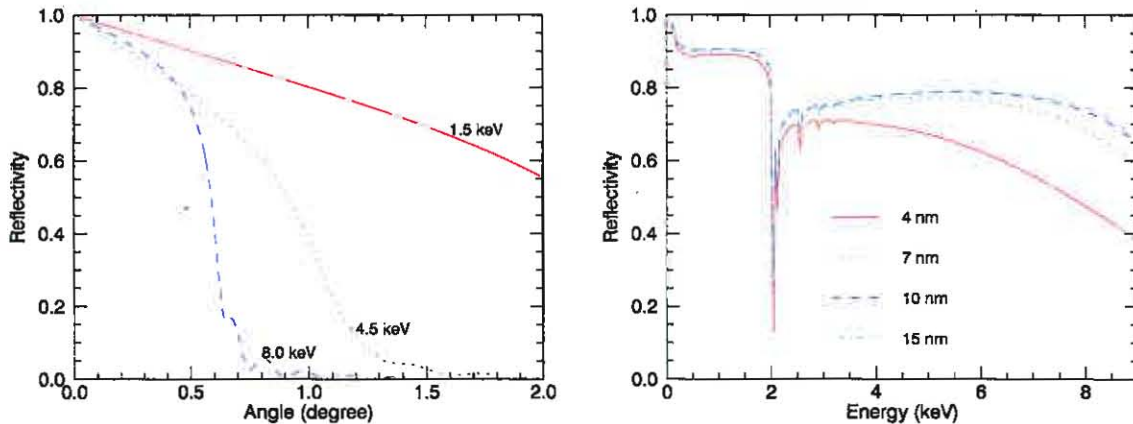

Figure 1. Reflectivity of x-ray from Ir surface. (Left) Dependence of reflectivity on incidence angle is shown for 3 different x-ray energies. The film is 20 nm of Ir with a nominal 0.4 nm roughness. Critical angle for 8 keV x-ray, for example, is about 0.5°, at grazing angle above which, the reflectivity drops precipitously. (Right) Dependence of reflectivity on energy is shown for different film thickness. The film is Ir on glass (SiO$_2$) substrate, with a nominal roughness of 0.4 nm. The incidence angle is 0.4°. It can be seen that for the 4 nm film, there is significant penetration of the film by the x-ray, especially at higher energy. Having a film thicker than 15 nm or so will not improve its reflectivity under these conditions.

Film with small thickness will be penetrated by x-ray radiation at higher energies. As can be seen in Figure 1, a film thinner than 4 nm will not reflect x-ray fully under the specified or similar conditions; while film thicker 15 nm or so gets no further improvement in reflectivity. We choose 20 nm as our nominal film thickness (for Ir) for maximum reflectivity.

2.1 Coating Stress

High coating stress will distort the mirror and degrade its resolution. For a flat, a uniform coating stress generates additional curvature given by:

$$K = \frac{6(1-\upsilon)\sigma\tau_f}{E\tau_s^2},$$ (1)

where υ, E are the Poisson ratio and the Young's modulus of the substrate, τ_s and τ_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 stress of the film, σ. For a 20 nm film with 3 GPa stress on 0.4 mm glass substrate, the curvature is 2.5×10^{-4} m^{-1}, which is equivalent to a sag of 1.25 μm over the length of the substrate, 200 mm. Since the substrate is not actually 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 in fact does not have a single value used in the example above, but rather depends on azimuth. The example above nevertheless provides a general dependence on the relevant quantity (note the dependence on $E\tau_s^2$) and gives an indication of the orders of magnitude of the relevant quantities.

Surface distortion can be modeled by applying a given strain/stress on the surface of a mirror. Figure 2 shows the result from a finite-element model of a case with 5 GPa stress in a 20 nm thick film deposited on a glass mirror with the aforementioned dimension. In the model, the thickness distribution along the axial direction (along the vertical axis) matches that measured from our magnetron sputtering experiment: one that is about 20% thin at the ends than at the middle. Because of its shape, the glass substrate responds in a more complicated way than a simple bow. In fact, the axial profiles at different azimuth are different. The sag derived for each azimuth is shown on the right panel. This distribution matches that from actual deposition.
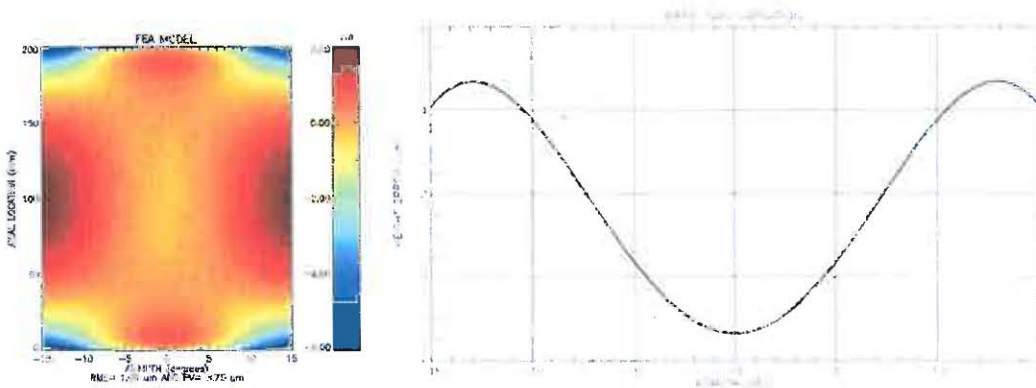

Figure 2. Change in surface figure from finite-element model of a glass substrate stressed from a film. The input strain film is from a thin film with a constant stress of 5 GPa and a thickness of 20 nm. The substrate is a 0.4 mm thick glass 200 mm long (axial direction is vertical in the graphic), spanning 30° azimuthally with a diameter of about 485 mm. (Left) The radial deviation of surface. (Right) The sag parameter at each azimuth. The turning around of sag at the azimuthal ends is due to non-uniformity of coating in the axial direction. See section 3.1.

2.2 Experimental result from magnetron sputtering

It has been known that magnetron-sputtered films of heavy metal such as Ir and Pt have high stresses. To evaluate the relevance of stress in our process of making arc-second mirror, we deposited films on small glass samples as well as 10 cm Si wafers. The curvatures of these samples were measured before and after coating. By using equation (1) above, the stress can then be derived. For the small samples (glass slides, about 2.5 cm in size), accurate curvatures can be measured with interferometric surface profiler such as Zgyo's NewView profiler, which can cover field of view of a few mm with a depth resolution better than 0.1 nm. Care must be taken to ensure that no additional phase change occurs between the surface before and after the coating, as they have different materials then. This can be accomplished by pre-coating the sample with a thin preparatory coat. For large Si wafers, laser scanning technique can be used to obtain linear profiles. We used film stress measuring FSM from Frontier Semiconductor to obtain the curvature, and hence stresses.

In a series of experiments, we sputtered Ir or Cr on the glass or Si substrates, using a DC planar magnetron. Typical parameters for the deposition are as follows. We used a base pressure of 5-9 μTorr, and a sputter gas (Ar) pressure of 2 mTorr. Voltage was set at 350V and current was maintained at about 0.85A. The thickness is controlled by time of deposition. On the Si wafers, several runs were made over a period of time. The result is summarized in Table 3 below.

Several observations can be made. First, the stresses are quite high, at a few GPa. With such a high stress, a film of 20 nm thickness, will produce significant distortion for arc-second mirrors, as shown above in Section 2.1. Secondly, it was

confirmed that Ir and Cr do have opposite stress. It should be noted that the second series shown in Table 3 showed higher stresses probably because of an underestimate of film thickness. There, film thickness was not measured but nominal numbers from the coater were used. There were indication that these numbers may be underestimated and the actual stress may be more in line with ~ -4 GPa.

A bi-layer with Ir and Cr (the Cr layer at the bottom can also serve as a binding layer) can therefore be used to obtain a zero net effective stress and distortion on substrates. Result from this effort will be discussed in Section 3.1.

Table 1. Summary of film stresses on Si and glass substrates. Ir and Cr films were magnetron-sputtered on different substrates. Different method were used to obtain the stresses (see text.) The stresses were in general very high, ~ 4 GPa for Ir films and 1.5 GPa for Cr films. The signs of stresses for Ir and Cr are also opposite: compressive for Ir and tensile for Cr.

Film	Sample/Method	Thickness (nm)	Stress (GPa)
Ir	Si wafer	20 – 40	-3.5 ± 0.2
Ir	Si wafer	15 – 30	-5.0 ± 0.14
Ir	glass	10 – 30	-3.5 ± 0.9
Cr	glass	10 – 30	+1.6 ± 1.1

We proceeded to deposit film on full-size substrates in order to properly assess the effect of coating stress on making precision x-ray mirrors. The substrates are approximate cylindrical in shape with a 250 mm radius. The height is 200 mm and the width is about 125 mm. Ideally, film thickness and stress should be uniform over the complete surface. Due to the limited length of the target (< 250 mm), the thickness was not uniform in the axial direction. In the azimuthal direction, uniform coating was maintained by rotating the substrate in a turntable inside the sputtering chamber. Figure 3 shows the axial distribution of thickness at 5 azimuthal positions. The thickness were made on specified spots on a mirror with a step created by masking during the coating. Each mask zone (a region of a few mm wide along a full masked line) yields 2 step heights. The steps heights were measured with an optical profiler with 0.1 nm vertical resolution. The measured non-uniformity shown is indirectly confirmed in finite element modeling, shown in Figure 2. With an otherwise completely uniform coating, the sag variation would more resemble a simple quadratic curve. The actual 4th order dependence observed comes out of the model when the axial thickness profile in Figure 3 was used.
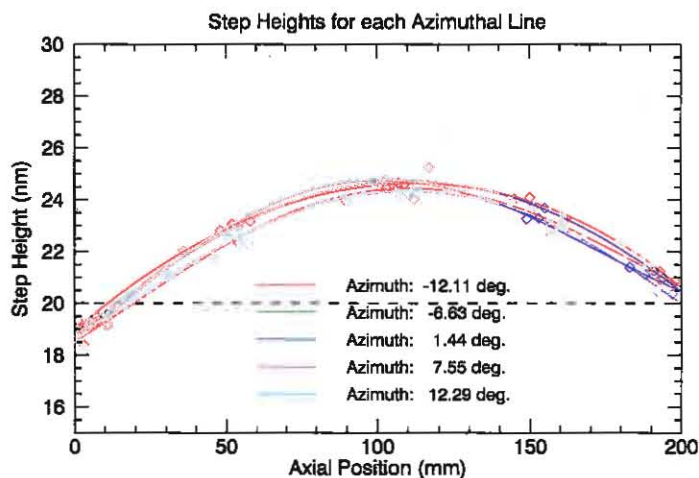

Figure 3. Thickness distribution of magnetron-sputtered film in the axial direction, at 5 different azimuths. The nominal thickness for the deposition is 20 nm. It is seen that two ends of the mirror did not received sufficient coating.

That film with thickness of about 20 nm and with stress as high as 4 GPa would have a detrimental effect making precision mirrors is clear, both from simple analytical argument, finite element modeling, as well as actual measurements. To show that experimentally, Ir films of different thickness was deposited on the mirrors, and the distortion of mirrors is obtained by comparing the their shapes before and after coating. Full surface of mirrors can be measured with an interferometer with a null lens providing a cylindrical wavefront. Alternatively, deposition can be

made incrementally on a mirror to show the effect of total stress from accumulated material. In Figure 4, the sag parameter, as a function of the azimuth, is shown for a mirror (55° in angular span) with successive deposition of 10 nm of Ir. To ensure the first layer also have attached to the substrate in the same way, a thin base coat was made first to ensure a good reference. It can be seen that the sag variation increases steadily with increasing film thickness, from 10 to 40 nm. The variance can be used as a measure of such effect. The rms value progresses with a rate of $(0.184 \pm 0.003)\,\tau_f$ + (0.49 ± 0.05) μm, where τ_f is the film thickness in nm. A 1 μm sag additional sag has a detrimental effect on the resolution of these mirrors. After all, the nominal axial sag of these mirrors, to achieve optimal focus, is just about 1.1 μm by design.
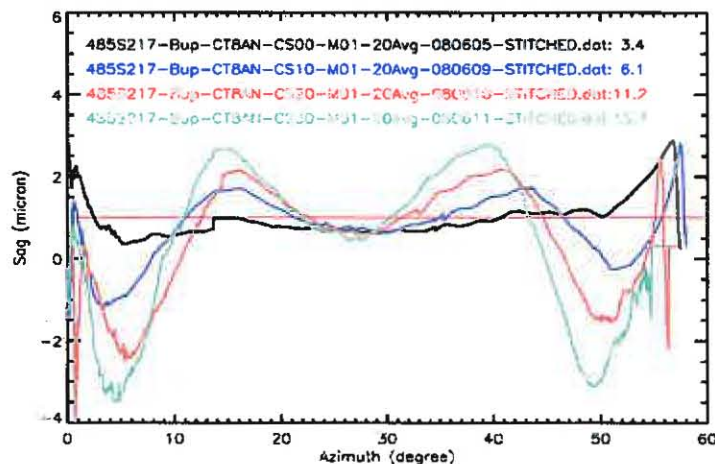

Figure 4. Dependence of sag variation on film thickness. Ir films were successively deposited on a mirror in increments of 10 nm. As can be seen, the sag variation increases steadily as the film thickness increases.

3. LOW STRESS FILM DEPOSITION

To make more precise mirrors, film stress will have to be reduced. Without raising the substrate temperature substantially, the coating stress may be modified by adjusting the sputtering rate and distance. (There are good reason not to raise the substrate temperature. These glass substrates were thermally formed at ~ 500°C and therefore will soften and lose its figure when heated substantially. The difficulty and additional investment in baking is not encouraging, either.) However, these adjustments are generally quite limited by the nature of the material being deposited: the heavy metals being coated, e.g., Ir, usually has a very high melting point to allow a low stress film to be deposited.

We attempt to take advantage of the opposite senses of Ir and Cr stresses, in order to achieve a low stress film. By combining an Ir and a Cr layer with a proper thickness ratio, an effective zero stress bi-layer film may be obtained. Such deposition were made at the laboratory at Reflective X-ray Optics LLC (RXO), New York.

Stress-balanced Ir/Cr bilayer coatings were deposited by magnetron sputtering in argon, using a deposition system that has been developed at RXO. The planar, rectangular magnetron cathodes use targets measuring 50 x 9 cm^2 by 0.6 cm thickness. The Ir cathode was operated at 400W power, and the Cr cathode at 100W power, with the Ar gas pressure held at 1.6 mTorr. Film stress was measured using the wafer curvature technique, and layer thicknesses were determined from X-ray reflectance measurements at 8 keV.

3.1 Calibration of Ir /Cr stress

Combined stress was first calibrated on wafer before deposition on curved glass substrates was made. In the process, a fixed Ir thickness was set and a series of depositions were made with different thickness of the underlying Cr layer. The total stress was measured. Results from these calibration runs are shown in Figure 5. At zero Cr thickness, the stress for Ir film is about -4 GPa, as we discuss in Section 2.2. As expected, the effective film stress went down as the Cr thickness increased. As shown in Figure 5, the calibrations were repeated and confirmed again in 2012.
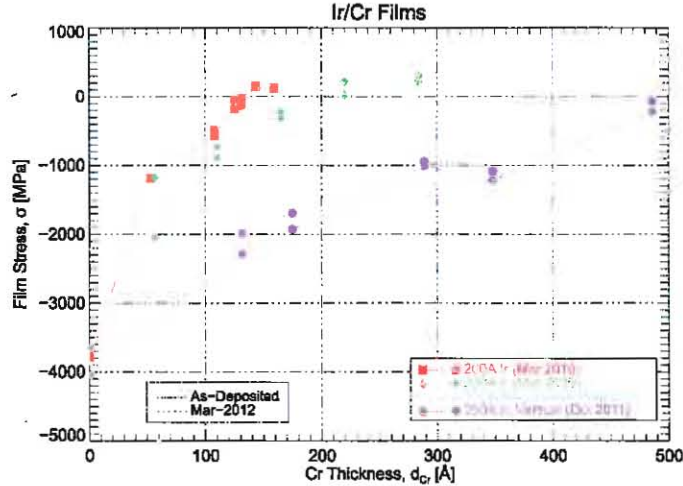
Figure 5. Effective stress of the Ir/Cr bi-layer, for fixed Ir thicknesses. Ir film stress is about -4 GPa without Cr. Effective stress can be reduced to zero, within +/- 50 MPa, as Cr thickness is increased. Calibration measurements on wafer were made again in 2012 and stresses were shown to be repeatedly reproduced. (Also shown are Ir with B_4C and Ir deposition in other configuration.)

3.2 Experiments and Results

With the Ir/Cr bi-layer thicknesses calibrated, low stress films were deposited on actual mirrors. To assess their effect, Ir/Cr films were deposited on 3 pairs of mirrors (dimensions in Section 2) with 3 sets of targeted total stress for each pair: +221 MPa, -426 MPa, and -887 MPa. Ir thickness is nominally fixed at 16 nm. Full surface interferometric measurements were made before and after deposition for each mirror.
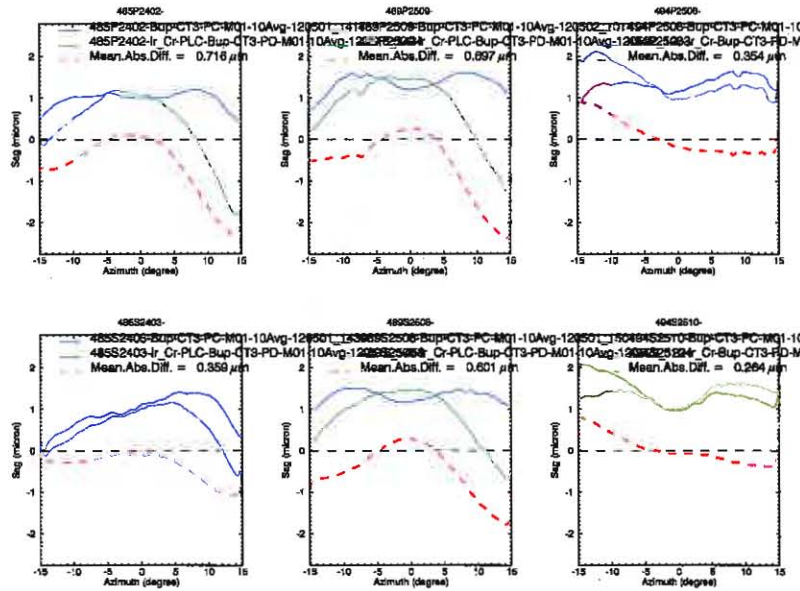

Figure 6. Sag changes of mirrors coated with Ir/Cr bi-layer, with 3 sets of targeted effective stresses. +221 MPa (Left), -426 MPa (Middle), -887 MPa (Right). Each targeted stress was received on 2 mirrors. The dashed lines show the difference in sag variation between mirrors with and without coating.

It is seen that the pairs of mirrors that received the same targeted stress have very similar change in sag, showing the repeatability of the coating. However, the change is not proportional to the applied stresses. We would expect the change for the mirrors shown in the left column in Figure 6 be opposite to the others and smaller in magnitude, and the change for the mirrors shown in the right column be 2 times than that in the middle column. More disturbingly, the change for

the mirrors with the smallest nominal stress ~ 0.2 GPa still produce a sag variation that is too large practically---more than 1 μm at one side of the mirrors, as well as not being consistent with models of uniform stress. The result is also peculiar in the way that the distortion is not symmetrical with respect of the left and right side of the mirrors.

The reason for this result is presently not clear and the experiment is still under further investigation. It should be noted, however, that the precision of sag measurement is sufficient, at about 0.15 μm. Part of the uncertainty for the non-proportionality of distortion with stress may come from the fact that the total thicknesses were not fixed for all mirrors. As the thickness of the Cr layer was varied to produce different stresses, the total thickness was larger for smaller for high (negative) stress films. Since it is the product of film thickness and stress that produce the curvature of the substrates (see equation 1), the effect from the high (negative) stress film are scaled back somewhat. Nevertheless, even with this consideration, the films did not produce a reasonable scaling between cases with different stresses.

Non-uniformity of film stress (or thickness) over the mirrors' surface may be a reasonable argument for the result. Such non-uniformity with respect to the mirrors' left and right sides certainly can produce a left-right asymmetry. In particular, if the uniformity of Ir and Cr layer were not the same, complex distortion pattern may result.

4. BALANCING MIRROR DISTORTION

Another approach to achieving no net distortion on the mirror is to balance the strain with a front and a back side films. If a mirror can be coated on both sides with films with the same thickness and stress distributions (or the same distribution of the product thickness x stress), then the effect from coating stress can be balanced. Precise coating, at a few percent, is required, though, if ~ 100 MPa effective stress needs to come out from a ~ 4 GPa original film.

4.1 2-sided Coating with Magnetron-sputtering

We carried out experiments with 2-sided coating. Ir was magnetron-sputtered onto full-size glass substrates in separate depositions at the front and back surfaces. Four substrates were used for this experiment. Nominal 20 nm films were deposited first in the front surface and subsequently in the back surface. Surface measurements were made before front-side deposition, after the front-side deposition and after the backside deposition. Sag parameters were derived from these measurements at each azimuth and the result is shown in Figure 7.
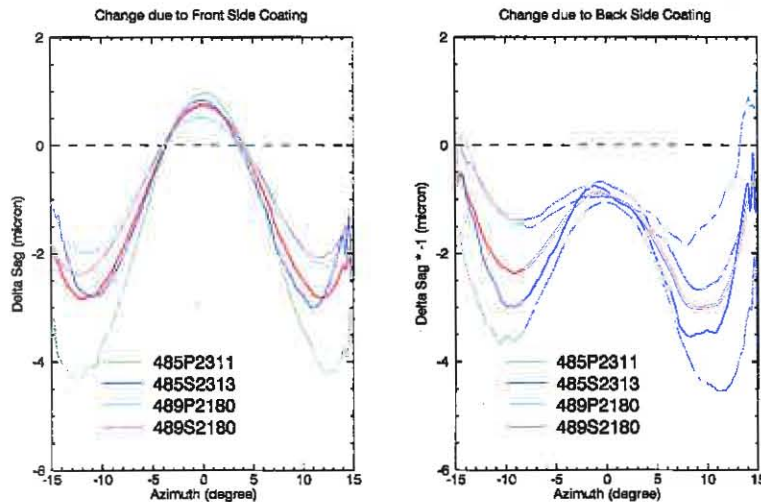

Figure 7. Sag change after 2-sided coating. Four mirrors were used with the same deposition parameters. (Left) Changes in sag variation after the front-side depositions were made. (Right) Further changes in sag variation after the backside depositions were made. The change is plotted with the sign reverse for visual comparison.

From Figure 7, it is seen that the sag changes are as expected after Ir films were deposited on the front side of the mirrors. (See finite element model shown in Figure 2. The model output there is the change in radial height, and therefore is opposite in sign when compared to change in mirror surface height.) The changes from the backside

depositions are similar but the magnitude is not the same, especially at the middle azimuth of the mirrors. The difference can come from the geometry of the set up of mirrors in the coating chamber. In the normal, front-sided, deposition, mirrors were mounted on a turntable that rotated in front of the sputtering target. The concave side of the mirror received sputtered Ir at an even distance for different azimuths. However, this is not the case when the mirrors were turned with their back facing the target. The substrate-target distance is no the same for the two orientations, especially for the middle azimuth. Presently, we are in the process of improving the setup and planning to deposit with a variable angular speed for the turntable so that the film's angular distribution due to different substrate-target distance may be compensated.

4.2 2-sided Coating with Atomic Layer Deposition

An attractive alternative is to deposit the front and back sides of a mirror with the same process simultaneously. For this purpose, sputtering in vacuum is not suitable. Recently, we began to investigate atomic layer deposition (ALD) as a practical means of deposition for this approach.
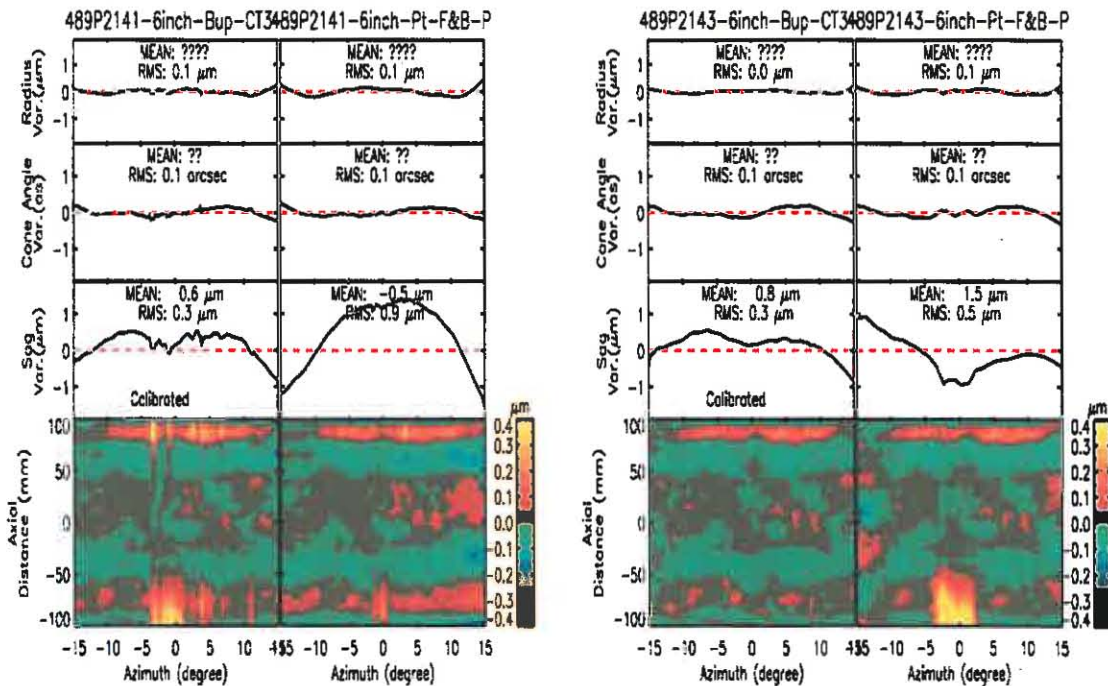

Figure 8. Sag change after ALD coating. Two mirrors were coated. Mirror 489P-2141 was run exactly the same way as the first mirror, however, upon inspection it was apparent that the film was indeterminately thinner. This occurred because the Pt source had been completely depleted at some point during the deposition.

In atomic layer deposition, thin films were grown on both sides of the mirrors (in fact, on all sides, including the edges) in a relatively high pressure reactor. The chemical deposition in a non-vacuum environment allows conformal deposition simultaneously on all surfaces. Deposition is controlled through precise cycling of two or more chemical vapors. ALD coating potentially can also produce films with much lower stress. The advantage of ALD for our purpose is therefore many-fold: (1) It produces conformal coating on all surfaces, regardless of mirror shape and sizes, (2) The coating is highly uniform, (3) The coating potentially has lower stress, (4) ALD films are dense and smooth (metal density and smoothness is important for x-ray reflectivity).

To experiment with ALD coating, we used ALD local facilities and commercial vendors. At present, we have work done or planned at the University of Maryland, College Park (Maryland, USA), and several thin film equipment providers including: Benec (Vantaa, Finland), Cambridge NanoTech (Cambridge, Massachusetts, USA), and Arradiance (Cambridge, Massachusetts, USA).

Preliminary results are encouraging. We shall know more as more results are coming in. Below, we described a preliminary result of ALD coating with Pt, done on 2 mirrors by Arradiance.

In this coating, Pt thin films were grown from TMMCP ((Trimethyl)methylcyclopentadienylplatinum(IV)) and O_2 in an Arradiance GEMStar-6 system at 250°C. The operating pressure is 200-400 mTorr. Films were grown using a novel, proprietary, high exposure Pt nucleation method that insures uniform Pt films with zero initial growth inhibition. The deposition was made at about 1.0 Å/cycle. A total of 325 cycles was used, yielding ~30-32nm of Pt on both sides of the mirrors. Two mirrors were coated. For this preliminary investigation, only mirrors of reduced dimension (150 mm in axial length instead of the standard 200 mm) could fit in the existing coating system. Comparison of the mirrors' surface before and after coating is shown in Figure 8.

The surface change after ALD coating is quite good, as shown in the left panel of Figure 8. This is even more encouraging considering the fact that this is a first rough trial, and the mirrors did not exactly fit properly in the coater. The coating on the second mirror was not successful, as the deposition was not completed due to the depletion of the Pt source during that deposition. More trials and coating with Ir are planned.

5. CONCLUSION

The frontier for next generation's lightweight, high-resolution x-ray optics for astronomy is to improve the angular resolution thin shell optics. Low stress coating for such thin-substrates is critical to preserve the high performance of these substrates. At NASA/Goddard, we have been developing thermally formed glass substrates performing at a few arc-second level, and the resolution is improving. In parallel, low stress coating techniques is sought to match that development. Magnetron-sputtering provides an efficient means to coat very large surface areas for these grazing incidence optics and has been use for lightweight x-ray optics on missions such as ASCA, Suzaku and NuSTAR. However, sputtering of high-Z metal such as Ir produces films with high stress, of the order of GPa. Such high stress distorts the thin mirrors beyond the arc-second resolution that is required.

In the search of a low-distortion coating scheme, we currently are pursuing two approaches. One is to achieve low effective stress by depositing film with 2 layers of materials, such as Ir and Cr, which have compressive and tensile stresses respectively. By properly adjusting the thickness ratio of these two materials, zero (< 50 MPa, practically, for our purpose) stress can be achieved. Calibration with wafer has demonstrated the feasibility of the concept. However, deposition of such bi-layer on full-size mirror did not yield undistorted mirrors yet. The reason may have to do with the uniformity of the coating (stress or thickness) over the whole surface of the mirror.

Another approach to figure preservation is to compensation any deflection deposition on both sides of a mirror. The concept was demonstrated to a large extent when front-and-back deposition with magnetron-sputtering show very similar (but with opposite sign) change. The change in curvature is not exact, though, with incomplete cancellation especially at the middle azimuth. The reason for this is that the near cylindrical mirror curve oppositely in the coating chamber for front and back coating. This geometric difference can be solved with a proper configuration of the mount of the substrate. Such modification is still in the work.

Along the same line of front-and-back coating, atomic layer deposition offers a more suitable solution than sputtering. ALD coating is naturally simultaneous for all surfaces, it is uniform and is low stress. Preliminary test showed that this approach is encouraging. Presently, we are engaging local facilities and commercial vendors to carry out more tests.

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