



<b>Publication Year</b>	2015
<b>Acceptance in OA</b>	2020-04-16T11:52:27Z
<b>Title</b>	Using iridium films to compensate for piezo-electric materials processing stresses in adjustable x-ray optics
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<b>Publisher's version (DOI)</b>	10.1117/12.2191404
<b>Handle</b>	<a href="http://hdl.handle.net/20.500.12386/24066">http://hdl.handle.net/20.500.12386/24066</a>
<b>Serie</b>	PROCEEDINGS OF SPIE
<b>Volume</b>	9603

# PROCEEDINGS OF SPIE

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**SPIE.**

Event: SPIE Optical Engineering + Applications, 2015, San Diego, California, United States

# Using Iridium films to compensate for piezo-electric materials processing stresses in adjustable X-ray optics

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## ABSTRACT

Adjustable X-ray optics represent a potential enabling technology for simultaneously achieving large effective area and high angular resolution for future X-ray Astronomy missions. The adjustable optics employ a bimorph mirror composed of a thin (1.5  $\mu\text{m}$ ) film of piezoelectric material deposited on the back of a 0.4 mm thick conical mirror segment. The application of localized electric fields in the piezoelectric material, normal to the mirror surface, result in localized deformations in mirror shape. Thus, mirror fabrication and mounting induced figure errors can be corrected, without the need for a massive reaction structure. With this approach, though, film stresses in the piezoelectric layer, resulting from deposition, crystallization, and differences in coefficient of thermal expansion, can distort the mirror. The large relative thickness of the piezoelectric material compared to the glass means that even 100MPa stresses can result in significant distortions.

We have examined compensating for the piezoelectric processing related distortions by the deposition of controlled stress chromium/iridium films on the front surface of the mirror. We describe our experiments with tuning the product of the chromium/iridium film stress and film thickness to balance that resulting from the piezoelectric layer. We also evaluated the repeatability of this deposition process, and the robustness of the iridium coating.

**Keywords:** X-ray optics, adjustable X-ray optics, piezo-electric materials, film stress

## INTRODUCTION

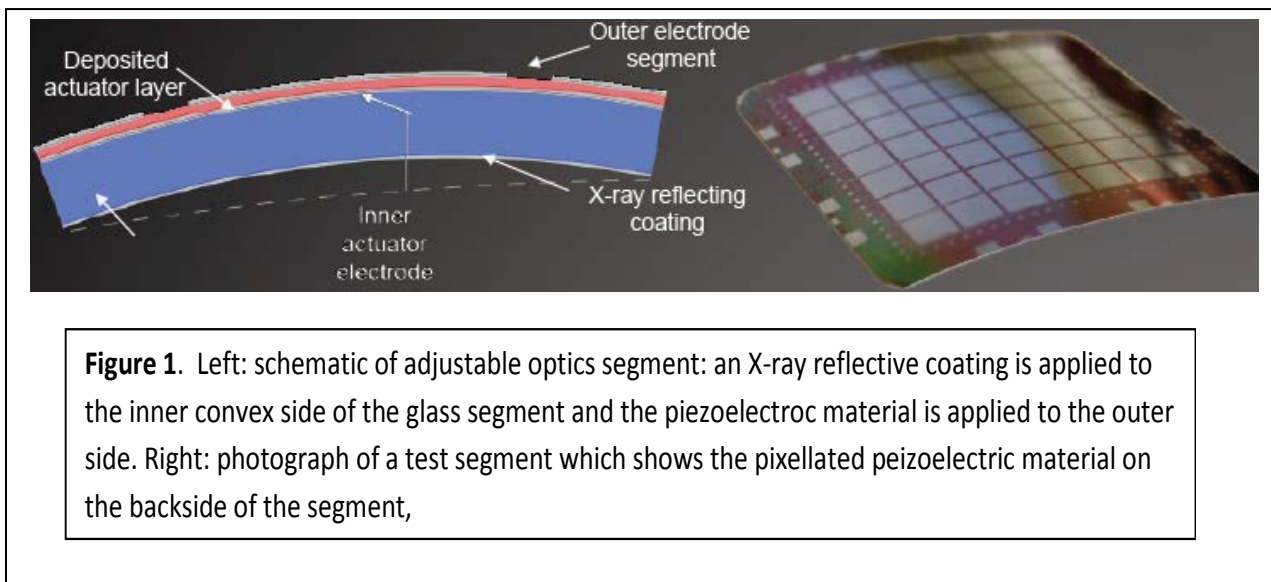
The science requirements for the X-ray Surveyor mission concept<sup>[1]</sup>, a successor to the *Chandra X-ray Observatory*, require high throughput and 0.5 arcsecond resolution optics. The angular resolution will be at least as good as Chandra and it will have  $\sim 30$  times the effective area. Such a large effective area necessarily implies light weight optics to meet the stringent mass requirements of the spacecraft. To meet these mass requirements a thin, segmented optics approach is being studied by several groups. The various approaches include: adjustable optics<sup>[2,3]</sup>, differential deposition figuring<sup>[4]</sup>, polished single crystal silicon<sup>[5]</sup>, magneto-strictive films<sup>[6,7]</sup> and direct polishing<sup>[8]</sup>. We present results from a recent study which uses a bilayer film to balance stresses due to the piezoelectric film which is part of in the adjustable optics approach.

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## Adjustable Optics Approach

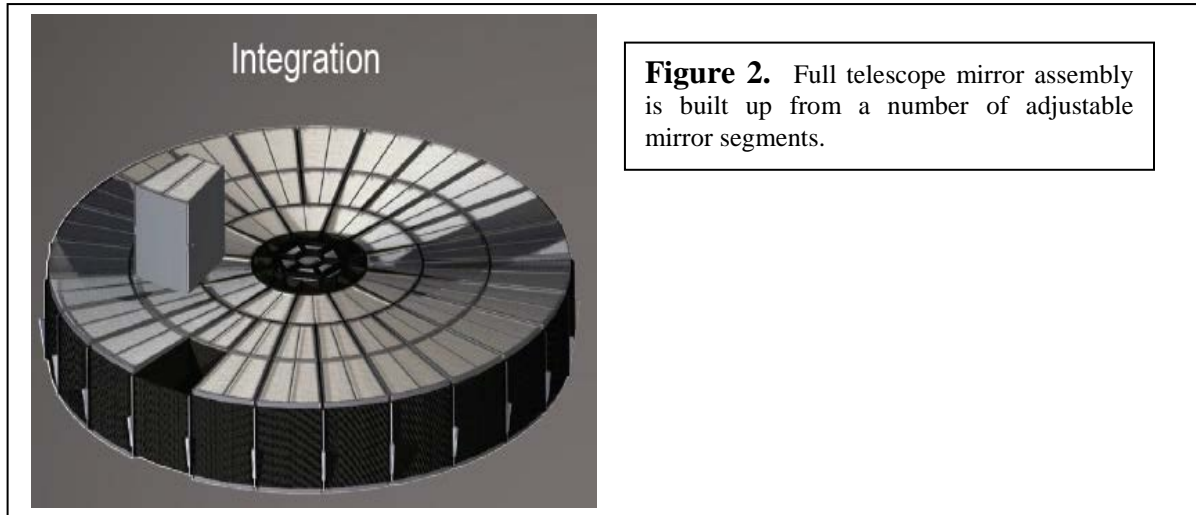
Adjustable X-ray optics represent one approach to large-area sub-arcsec imaging that eases constraints on the mirror mounting system and thermal environment. Our adjustable mirror approach employs a thin film of piezoelectric material deposited on the back of extremely thin mirrors<sup>[9,10]</sup>. The concept is shown in Figure 1. A uniform platinum ground electrode is first deposited on the back surface of the 0.4-mm thick mirror, followed by ~1.5- $\mu\text{m}$  layer of the piezoelectric material lead zirconate titanate (PZT actuator layer), as shown in figure 1, left. A pattern of discrete, independently addressable, platinum electrodes are lithographically deposited on top of the PZT layer (shown in Fig. 1, right). Applying a voltage across the top and bottom platinum electrodes produces a local electric field normal to the mirror surface, resulting in a strain in the PZT material parallel to the mirror surface. This produces a local deformation (in the vicinity of the energized pixel, or cell), called an “influence function”. The amplitude as a function of applied voltage and shape of the influence function for each piezo cell can be calibrated. A least-squares fit or deconvolution is used to determine the individual voltage required for each piezo cell to minimize the mirror figure error thus optimizing optical performance. Thermally formed 0.4-mm thick Corning Eagle<sup>TM</sup> glass is used as the substrate for the adjustable optic.



A full telescope mirror assembly is built up from a number of these adjustable mirrors, as shown in figure 2. Several mirror segments are co-aligned to form, e.g., a Wolter-I shell of a paraboloid of revolution and a hyperboloid of revolution. Many shells are nested together to produce the full collecting area.

We are developing X-ray optics using this approach to simultaneously achieve the requirements of large collecting area and high resolution for potential future missions. Several challenges remain before we can successfully demonstrate that this technology meets the necessary requirements.

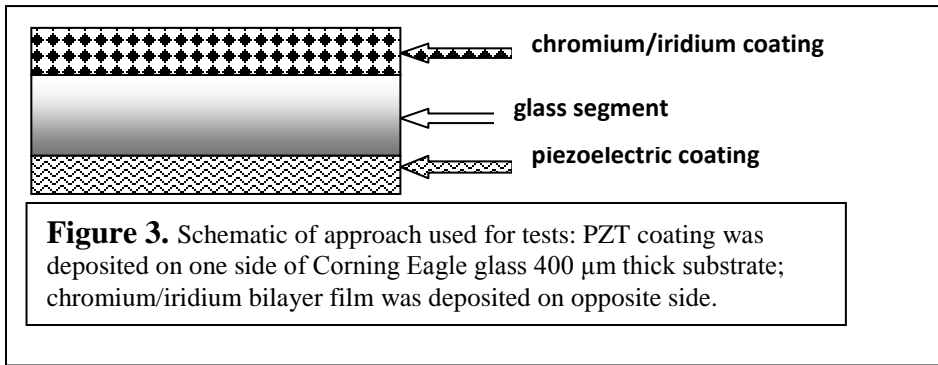
Stress in the deposited piezoelectric film is one challenge that is addressed in this paper. We report on a recent study to deposit Cr/Ir coatings on thin glass substrates with stress times film thickness levels similar to those of the deposited piezoelectric films. This could effectively allow us to compensate for, or balance, the stress due to the deposited piezoelectric film.



### Compensating for Stress

As stated above, a voltage is applied across the piezoelectric material to create the desired deformation in each cell to correct the figure of the segment. However, stress is introduced in the deposition of the piezoelectric material and electrodes. In addition to the deposition stresses due to the thick film piezoelectric, the high annealing temperature of 550 C coupled with the different coefficient of thermal expansion of the piezo versus the glass, and dimensional changes from amorphous to crystalline PZT during the anneal, all add to produce the stress on a 400 micron thin glass segment. It is necessary to remove or balance this stress if we are to reach the required sub arcsecond figure necessary for the science requirements of future X-ray missions.

To eliminate the mirror distortion created by the deposition of the PZT, we are studying the approach of depositing a film on the opposite side of the substrate to balance the stress. Figure 3 presents a schematic of this approach. To produce the desired deformations in the mirror segments of the telescope, the thickness of the PZT layer must be 1.5  $\mu\text{m}$ . Therefore two inch diameter 400 micron thick Eagle glass substrates were used to measure the stress of deposited piezoelectric films. A 1.5 micron thick piezoelectric film was deposited on several substrates and a profilometer was used to measure curvature before and after coating. The measured stress was  $\sim 100\text{MPa}$  tensile, yielding an integrated stress (i.e. stress times film thickness) of  $\sim 150\text{MPa}\cdot\mu\text{m}$ . To balance this effect, the film deposited on the opposite side must also produce  $150\text{MPa}\cdot\mu\text{m}$  of tensile stress.



For this approach to work, in addition to the magnitude and direction (tensile vs. compressive) of the stress, one must also duplicate the functional form, or spatial distribution, of the stress across the substrate. If the stress produced by the PZT is uniform across the substrate, then applying a uniform compensating stress of the same magnitude and direction on the opposing side should balance this stress. If the stress produced by the PZT has a complicated spatial distribution, it may not be possible to balance such a stress well enough to achieve the necessary figure requirements. However, the first question to answer is whether or not we can achieve the required magnitude and direction of the stress and whether or not this is reproducible.

The film we chose to use for this demonstration is a chromium/iridium bilayer film. Iridium was chosen for its high electron density and superior X-ray reflectivity in the soft X-ray band. However, magnetron deposited iridium has a compressive stress, which would deform the substrate in the wrong direction. Chromium, which is often used as a binder layer when depositing iridium on glass, can be deposited with either compressive or tensile stress depending on the deposition parameters. Therefore the idea was to combine these two materials to try to achieve the required 150 MPa- $\mu\text{m}$  of tensile stress to balance the PZT.

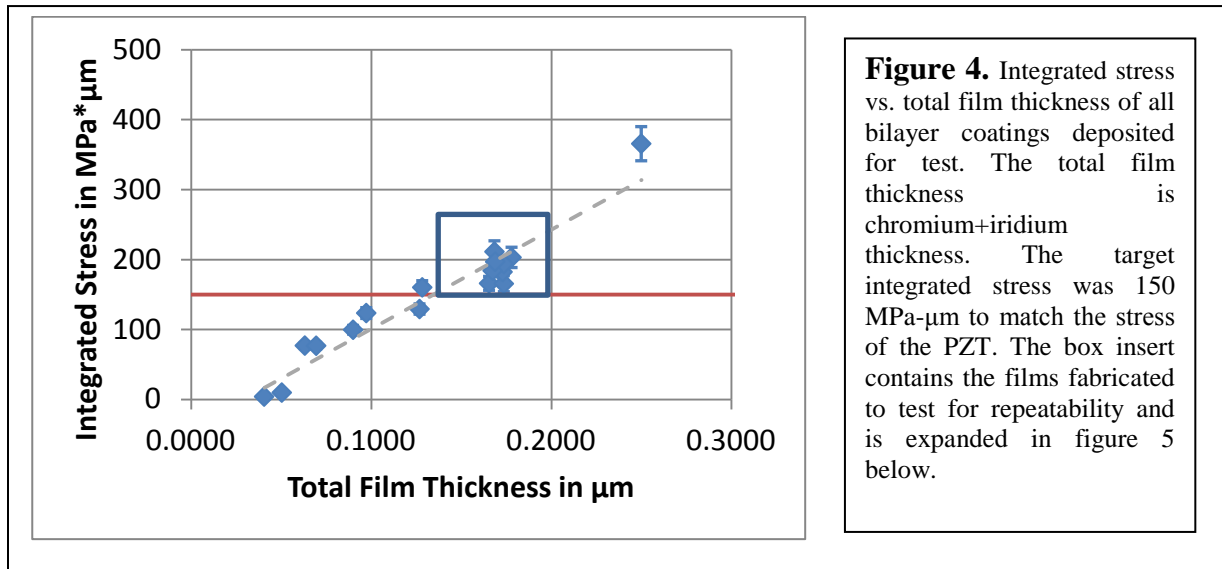
### Chromium/Iridium Bilayer

The micro-roughness and stress in thin films can be tuned by varying the deposition parameters such as: argon pressure, target-substrate distance, current (i.e. deposition rate), thickness etc. Since the iridium film will be used as the reflector layer of the telescope it must be deposited with low surface micro-roughness to yield high reflectivity. A 150 MPa- $\mu\text{m}$  tensile stress is needed to compensate for the PZT, therefore this must be the combined stress of the chromium/iridium film.

DC magnetron sputtering was used to fabricate the bilayer films. For these tests the film thickness of the iridium film was kept constant at 100  $\text{\AA}$ , using a current of 50mA and argon pressure of 2 millitorr to provide a film with < 5  $\text{\AA}$  micro-roughness. The thickness of the chromium film was varied along with the pressure and current to produce films of varying tensile stress.

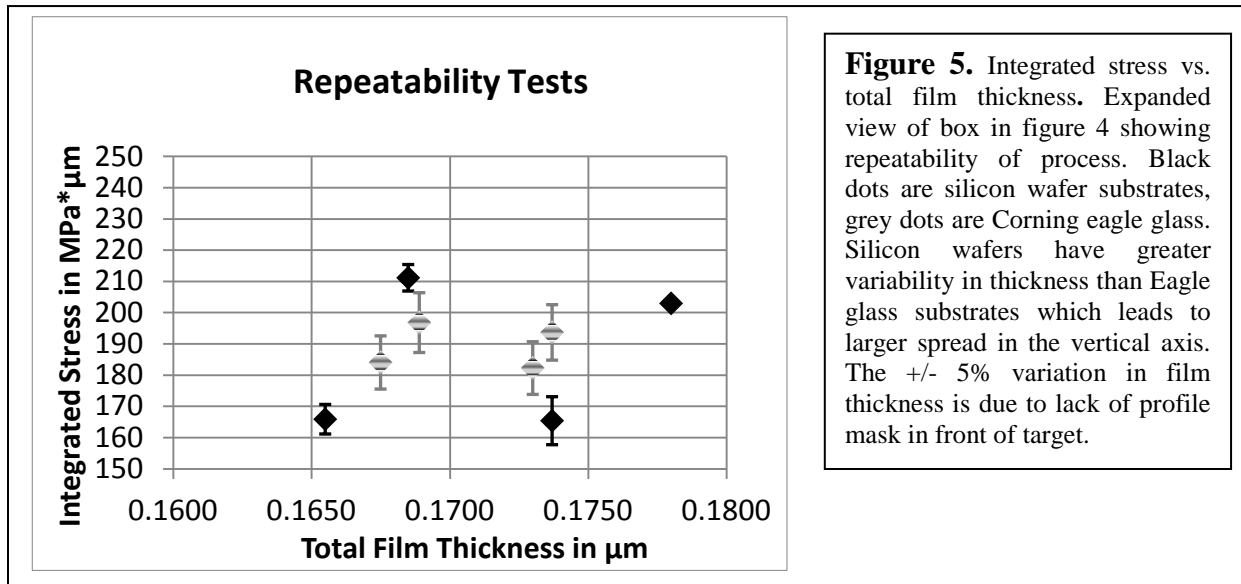
The argon pressure for different films was varied from from 2 to 5 millitorr and the current from 50 mA to 150 mA. The thickness of the chromium films varied from 300  $\text{\AA}$  to 2400  $\text{\AA}$ .

The several bilayer films were deposited on two-inch diameter silicon wafers to achieve the desired integrated stress. Chromium was deposited as the first layer, followed by an iridium film on top. The radius of curvature of the substrate was measured before and after film deposition in two perpendicular directions. Stoney's equation<sup>[11]</sup> was then used to compute the magnitude and direction of the stress applied by the film to the substrate. Figure 4 presents integrated-stress vs. total thickness plot showing all of the trial wafers. The target integrated stress we wanted to achieve was 150 MPa- $\mu\text{m}$  of tensile stress to match the stress of the PZT. The deposition parameters for chromium which yielded a total integrated stress of 150 MPa- $\mu\text{m}$  (including top layer of iridium) were: 100mA, 2mT, 1600 Å thick.



**Figure 4.** Integrated stress vs. total film thickness of all bilayer coatings deposited for test. The total film thickness is chromium+iridium thickness. The target integrated stress was 150 MPa- $\mu\text{m}$  to match the stress of the PZT. The box insert contains the films fabricated to test for repeatability and is expanded in figure 5 below.

To test for repeatability, these parameters were then used to fabricate chromium/iridium bilayer films on eight substrates: four silicon wafers and four two inch diameter 400 micron thick Corning Eagle™ glass substrates. The results of these repeatability tests are shown in figure 5. The four grey data points represent the results for the eagle glass; the four black data points for the silicon wafers. The +/- 5% variation in film thickness is due to the sputter process variability because no uniformity mask was used during deposition. The variation in thickness of the silicon substrates is greater than that of the eagle glass and this accounts for the greater spread in integrated stress for the silicon wafers.



**Figure 5.** Integrated stress vs. total film thickness. Expanded view of box in figure 4 showing repeatability of process. Black dots are silicon wafer substrates, grey dots are Corning eagle glass. Silicon wafers have greater variability in thickness than Eagle glass substrates which leads to larger spread in the vertical axis. The +/- 5% variation in film thickness is due to lack of profile mask in front of target.

**Micro-roughness**

A Zygo New View optical profiler was used to measure surface roughness on two of the coated Corning Eagle™ glass substrates from figure 5. The data was taken at five locations on each sample: the center and approximately 0.5 inch from center at 0,90,180,270 degrees. Data was taken using a 5x magnification objective, with a 640x480 pixel camera of 4.4 micron/pixel resolution.

Data analysis was performed in MATLAB. Each data set was flattened by fitting and subtracting a parabolic surface. For each data set, power spectral density plots were produced along each trace in the x and y directions using a Hamming window, via Matlab’s built-in periodogram function. These were averaged to produce average PSDs in the x and y directions for each data set. Each PSD was normalized using Parseval’s equality. Table 1 shows the data for each of these samples along with the RMS microroughness for each which is < 3 Å. These roughness levels are well suited for high reflectance and relatively low scatter of X-rays at grazing incidence up to energies of ~ 10 keV, the bandwidth of the X-ray Surveyor mission concept.

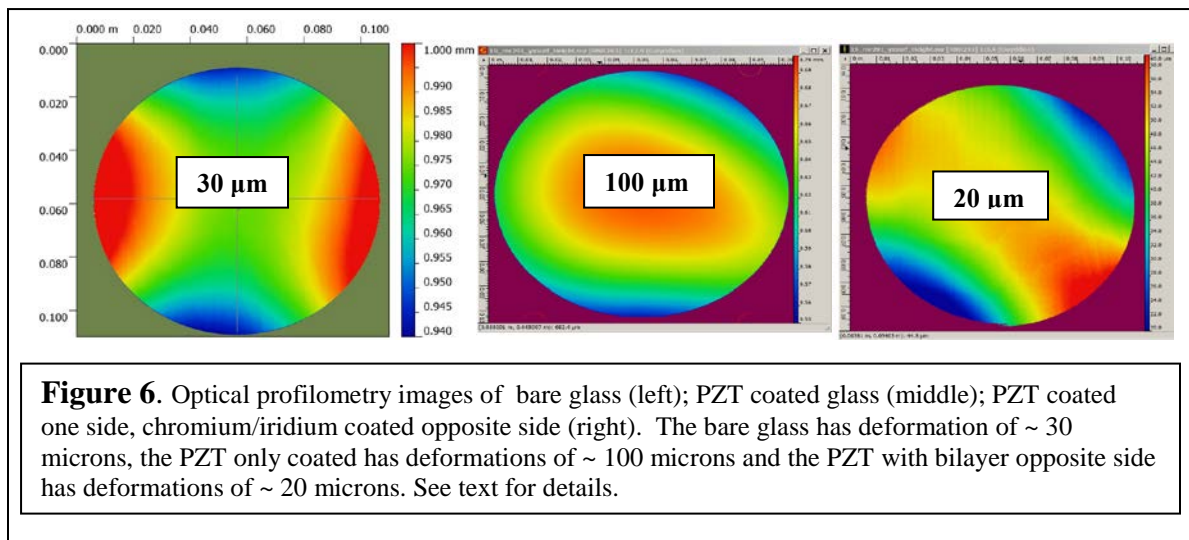
**Table 1 microroughness data from two bilayer coated 400 micron thick Corning eagle glass substrates. Five points were measured on each substrate. RMS microroughness of each was < 3Å.**

	Micro-roughness (Å)					RMS
<b>Sample1</b>	<b>2.95</b>	<b>2.6</b>	<b>3.11</b>	<b>2.88</b>	<b>3.18</b>	<b>2.95</b>
<b>Sample2</b>	<b>2.78</b>	<b>2.96</b>	<b>2.51</b>	<b>2.91</b>	<b>2.95</b>	<b>2.83</b>

**Proof-of-Concept**

Having achieved the necessary magnitude and direction of stress in the chromium/iridium films, the next test is to apply the bilayer film to the backside of a PZT coated substrate. For this test two eagle glass substrates were coated with 1.5 μm of PZT. A Cr/Ir bilayer film was deposited on the opposite

side of one of these substrates, using deposition parameters to yield  $\sim 150\text{MPa}\cdot\mu\text{m}$  of tensile stress to balance the effect of the stress due to the piezoelectric film. One bare glass substrate was used as a control for this test. Fig.6 presents the optical profile images of these three substrates along with the measured peak-to-valley deflection for each: control (fig 6a) has maximum peak-to-valley deformation of  $\sim 30$  microns astigmatism; substrate with piezoelectric film (fig 6b) has maximum peak-to-valley deformations of  $\sim 100$  microns, predominantly spherical with some astigmatism; and substrate with piezoelectric and Cr/Ir (fig 6c) has maximum peak-to-valley deformations of  $\sim 20$  microns astigmatism. This results shows that by depositing a chromium/iridium bilayer film with the proper parameters on the frontside of the PZT coated Eagle™ glass one can reduce the maximum deformation of the flat glass to a magnitude similar to that of the bare glass. This study has not investigated any spatial variation that may exist, although the shapes of the “before” and “after” deformations are consistent with uniform stress. We also did not attempt at this time to carefully calibrate the PZT related stresses to attempt to exactly cancel the processing stress. Our intent was to demonstrate that a reasonably controlled front surface X-ray reflecting layer can be deposited under repeatable conditions that introduces a compensation stress to that produced by the PZT processing. Further study is needed to complete these tests on conical glass substrates.



## SUMMARY

Adjustable X-ray optics represent a potential enabling technology for simultaneously achieving large effective area and high angular resolution for future X-ray Astronomy missions.

Controlled stress chromium/iridium bilayer films were deposited on the front surface of flat 2-inch diameter PZT coated substrates to compensate for the stress induced by the PZT. We described our experiments to tune the product of the chromium/iridium film stress and film thickness to balance that resulting from the piezoelectric layer. We also evaluated the repeatability of this deposition process, and the robustness of the iridium coating.

These roughness levels of the iridium films were measured using a ZYGO profiler. Their micro-roughness of  $\sim 3\text{\AA}$  is well suited for high reflectance and relatively low scatter of X-rays at grazing incidence up to energies of  $\sim 10$  keV, the bandwidth of the X-ray Surveyor mission concept.

However, further data using X-ray reflectivity measurements to evaluate microroughness is desirable. We have shown that by depositing a chromium/iridium bilayer film with the proper parameters on the frontside of the PZT coated Eagle™ glass the maximum deformation of the flat glass can be reduced to a magnitude similar to that of the bare glass. Further study is needed to complete these tests on conical glass substrates and to investigate any non-uniformities in stress that may exist across the substrate.

## REFERENCES

- [1] Jessica A. Gaskin et al., "The *x-ray surveyor* mission: a concept study", Proc. SPIE, Vol 9601-18, these proceedings (2015).
- [2] Reid, P. B.; Aldcroft, T. L.; Cotroneo, V.; Davis, W.; Johnson-Wilke, R. L.; McMudroch, S.; Ramsey, B. D.; Schwartz, D. A.; Trolrier-McKinstry, S.; Vikhlinin, A.; Wilke, R. H. T., "Technology development of adjustable grazing incidence x-ray optics for sub-arc second imaging", Proc. SPIE, Vol 8443, id. 84430T, 8 pp. (2012)
- [3] Reid, Paul B.; Aldcroft, Thomas L.; Allured, Ryan; Cotroneo, Vincenzo; Johnson-Wilke, Raegan L.; Marquez, Vanessa; McMudroch, Stuart; O'Dell, Stephen L.; Ramsey, Brian D.; Schwartz, Daniel A.; Trolrier-McKinstry, Susan E.; Vikhlinin, Alexey A.; Wilke, Rudeger H. T.; Zhao, Rui, "Development status of adjustable grazing incidence optics for 0.5 arcsecond x-ray imaging", Proc. SPIE, Vol 9208, id. 920807 9 pp. (2014)
- [4] Kilaru, Kiranmayee; Ramsey, Brian D.; Gubarev, Mikhail V.; Gregory, Don A., "Differential deposition technique for figure corrections in grazing-incidence x-ray optics", Optical Engineering, Volume 50, Issue 10, pp. 106501-106501-6 (2011).
- [5] Riveros, Raul E.; Bly, Vincent T.; Kolos, Linette D.; McKeon, Kevin P.; Mazzarella, James R.; Miller, Timothy M.; Zhang, William W., "Fabrication of single crystal silicon mirror substrates for X-ray astronomical missions", Proc. SPIE, Volume 9144, id. 914445 6 pp. (2014).
- [6] Ulmer, Melville P.; Wang, Xiaoli; Cao, Jian; Graham, Michael E.; Vaynman, Semyon, "Update to an application using magnetic smart materials to modify the shape of an x-ray telescope mirror", Proc. SPIE, Volume 8861, id. 88611R 8 pp. (2013).
- [7] Ulmer, M. P.; Graham, M. E.; Vaynman, S.; Cao, J.; Takacs, P. Z., "Deformable mirrors for x-ray astronomy and beyond", Proc. SPIE, Volume 8076, id. 807605 (2011).
- [8] M. Gubarev, B. Ramsey, J. K. Kolodziejczak, W. S. Smith, J. Roche, W. Jones, C. Griffith, T. Kester, C. Atkins, W. Arnold, "Direct fabrication of full-shell x-ray optics", Proc. SPIE, Vol 9603 these proceedings (2015).
- [9] Reid, P., et al., "Development of adjustable grazing incidence optics for Generation-X," SPIE Proc. 7011, 70110V (2008).
- [10] Wilke, Rudeger H. T.; Johnson-Wilke, Raegan L.; Cotroneo, Vincenzo; Davis, William N.; Reid, Paul B.; Schwartz, Daniel A.; Trolrier-McKinstry, Susan, "Sputter deposition of PZT piezoelectric films on thin glass substrates for adjustable x-ray optics", Applied Optics, vol. 52, issue 14, p. 3412 (2013)
- [11] G. Stoney, Proc. R. Soc. London Ser. A, Vol. 82, (1909)