MIRRORS FOR HIGH RESOLUTION X-RAY OPTICS

--- FIGURE PRESERVING Ir/Pt COATING

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29-MAR-2016
Mirror Coating

• Need high density, high-Z material such as Au, Pt, or Ir for effective X-ray reflection in 1 – 10 keV band
• For Ir, will need ~ 15 nm of Ir for maximal reflectivity
Coating Distortion

- A stress of $\sigma \sim 4$ GPa was measured and modeled.
- The distortion can be reproduced with a film of 4-5 GPa stress with measured thickness.

Note: The "W"-shaped azimuthal dependence of $\Delta$Sag is due to the non-uniformity in coating thickness in the axial direction specific to the magnetron used.

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**Basic Experimental Facts**

• **Coating stress of 10 – 20 nm of Ir is sufficiently high** to distort the figure of arc-second thin lightweight mirrors. For iridium:
  – Stress $\sigma \sim 4$ GPa for $\sim 15$ nm film implies 60 N/m integrated stress
  – Need $< 3$ N/m (or stress $< 200$ MPa) for sub-arcsecond optics

• **Basic Approaches for Mitigation**
  A. **Annealing the film**
     – Glass can be heat up to 400°C without distortion. Silicon is even more resistant.
     – It was found that recovery is limited by residual thermal stress from taking the mirror down from high T
  B. **Coating bi-layer films with compressive stress with tensile stress**
  C. **Front-and-back coating with magnetron sputtering or atomic layer deposition**
     – Sputtering involve spanning of substrates. Geometric difference in setup (convexness/concaveness of curved mirrors) does not permit precise front-and-back matching
     – Atomic layer deposition can provide a uniform deposition front and back simultaneously
(1) **Annealing**

- Re-heating anneal the film to relax coating distortion
- **Basic Requirement**
  - Must restore the mirror’s figure / eliminate distortion from coating stress
  - Must not change the substrate’s figure
  - Must not degrade the surface’s micro-roughness
Annealing does not change substrates

Bare glass substrates heated up to 410°C

- Blue: before heat
- Green: after heat
- Red: difference

Substrates are not affected after heating
Coating and Annealing

- Coating: Ir
- Thickness: 5, 10, 20 nm

Example shown: 20 nm

Coating: The distortion is linear: \(<S(\theta)> \approx 0.1 \, \mu m/\, nm \) of Ir deposition; \( S_{PV} \approx 0.3 \, \mu m/\, nm \)

- Heating greatly reduces the figure distortion
- The reduction is not complete

Before Annealing

After Annealing
The non-monotonic behavior may be due to a combination of effects:

- Relatively larger interface effect for thinner film
- Larger distortion induced by CTE-mismatch for thicker film as the mirrors cool after annealing
Dependence on Annealing Temperature

320°C ➔

380°C ➔

410°C ➔

2 mirrors for consistency

Substrate as control

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Temperature Dependence

Non-monotonic trend

- Larger distortion may be induced by CTE-mismatch at higher temperature

Optimal Temperature at ~ 310°C
Annealing

Before Annealing

After Annealing

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(2) Ir/Cr Bi-layer Deposition

- Take advantage of compressive Ir and tensile Cr stresses
- Combined Ir/Cr bi-layer deposition can produce zero integrated stress
- Calibration of Cr film with variable thickness demonstrated the reduction of effective stress of Ir/Cr film to within ± 0.1 GPa

* Work done with D. Windt (2012)
(3) Atomic Layer Deposition

Pt coating

Did not balance mirror distortion

* Coating done at Cambridge NanoTech, Cambridge, MA
Atomic Layer Deposition

Pt coating

Did not achieve the desired effect of balancing mirror distortion

* Coating done at Arradiance, Sudbury, MA
Atomic Layer Deposition

Ir coating

- Did not balance mirror distortion
- Varied among the test pieces
- Different from other ALD test

* ALD coating of Iridium done at Beneq Oy, Finland
(4) Front + Back Deposition: sputtering

- Issue with coating geometrical configuration from coating by just flipping the mirror around
- Fix: using 2 separate magnetrons on the front and back of the rotating mirror
  - Front distortion is not the same as back
- Coating on glass is not the same as on iridium
- That is, front side coating on glass is different from the backside coating (on glass); and coating on the glass surface is also different from that on iridium-coated surface
  - Nature of the surface is critical
  - Larger difference is especially from coating on the surface of front side bare glass
  - Could justify the result of ALD coating
Comparing coating on bare glass (front side) vs. on Iridium-coated surface using Front and Back magnetrons
Comparing coating on bare glass (back side) vs. on Iridium-coated surface using front-and-back magnetrons
Coating on Silicon Wafers

- No change in figure
- Low order bowing < 0.1 μm
- No geometric difference in setup for front and back coating

Original Wafers  |  Front+Back Coating  |  After Annealing
CONCLUSION AND FUTURE DEMONSTRATION

• Coating stress from magnetron sputtering of Iridium can be significantly reduced by annealing
  – Distortion is reduced by a factor of ~ 5
  – Further reduction of distortion can be achieved by front-and-back coating to realized sub-arcsecond coating distortion

• Front-and-Back compensation
  – Difference in geometric set up was fixed but was shown not to be the major contributor
  – Difference in glass front and back surfaces prevent precise compensation even with ALD
  – Difference in the nature of glass surfaces for coating was demonstrated
  – Coating on flat silicon wafers showed that balancing can be achieved

• Coating on silicon mirrors
  – Curved silicon mirrors are coming online to be tested
  – If realized, the annealing and front-and-back coating will produce negligible (<1") distortion for the optics for a 5” mission, removing the coating problem from the list of large risks.