MIRRORS FOR HIGH RESOLUTION X-RAY OPTICS --- FIGURE PRESERVING IR/PT COATING

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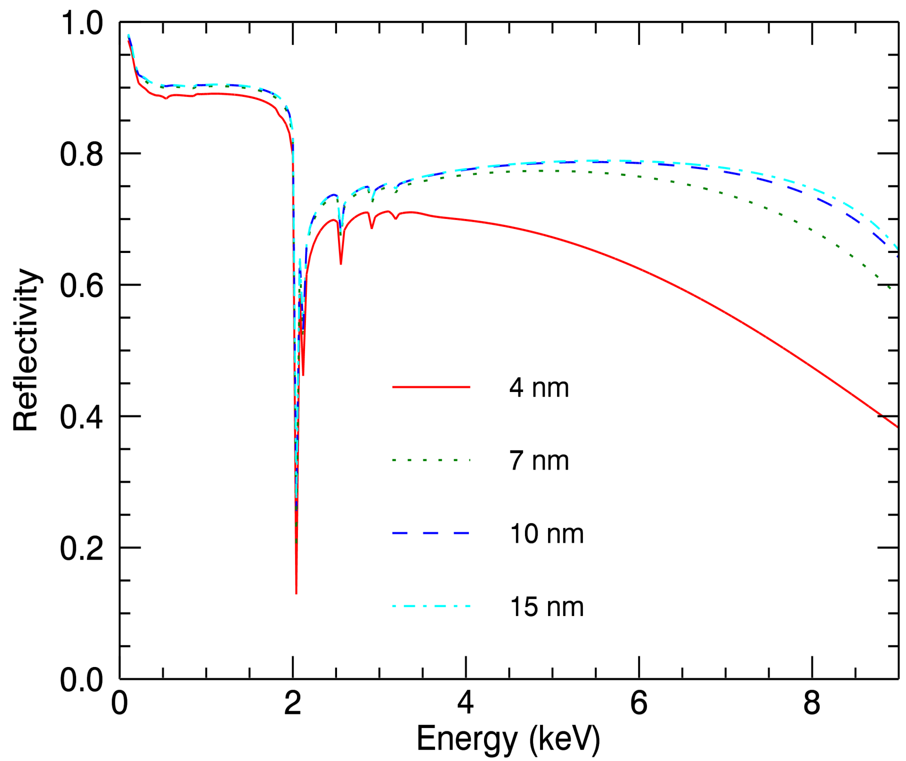
Mirror Coating

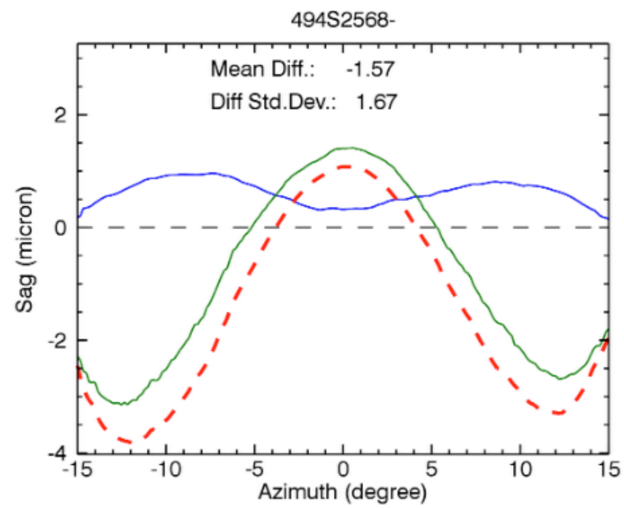
- **Need high density, high-Z material such as Au, Pt, or Ir for effective X-ray reflection in 1 – 10 keV band**
- **For Ir, will need ~ 15 nm of Ir for maximal reflectivity**

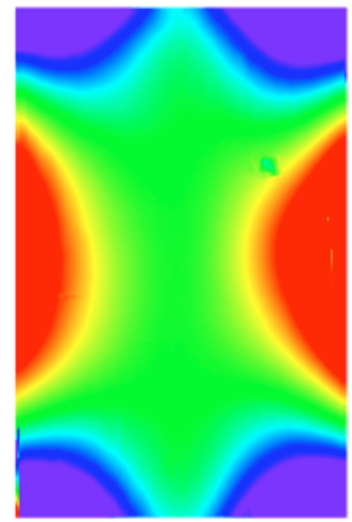


Coating Distortion

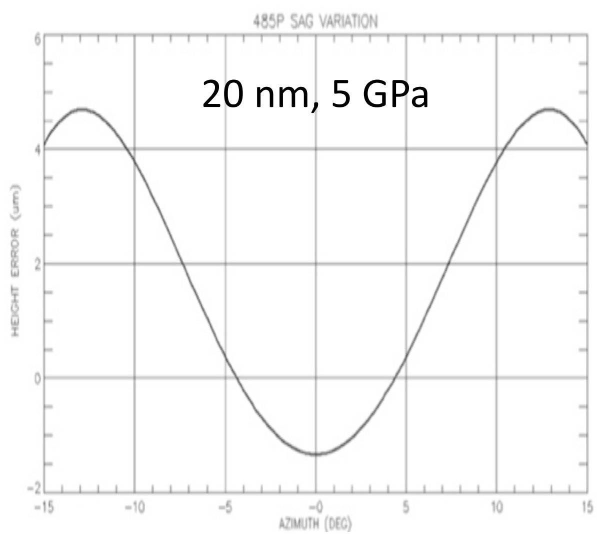
◆ A stress of σ ~ 4 GPa was measured and modeled

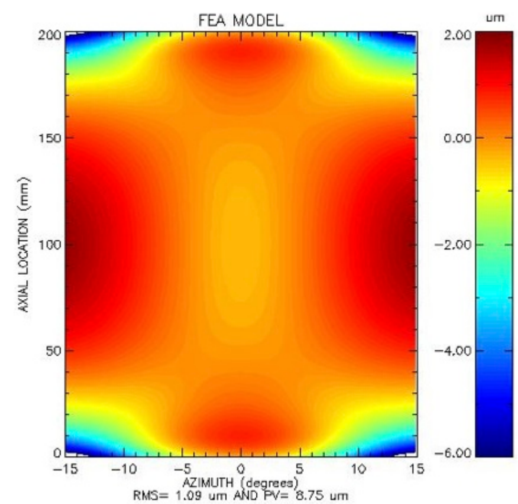
◆ The distortion can be reproduced with a film of 4-5 GPa stress with measured thickness





Measured





Modeled

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

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 σ ~ 4 GPa for ~ 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

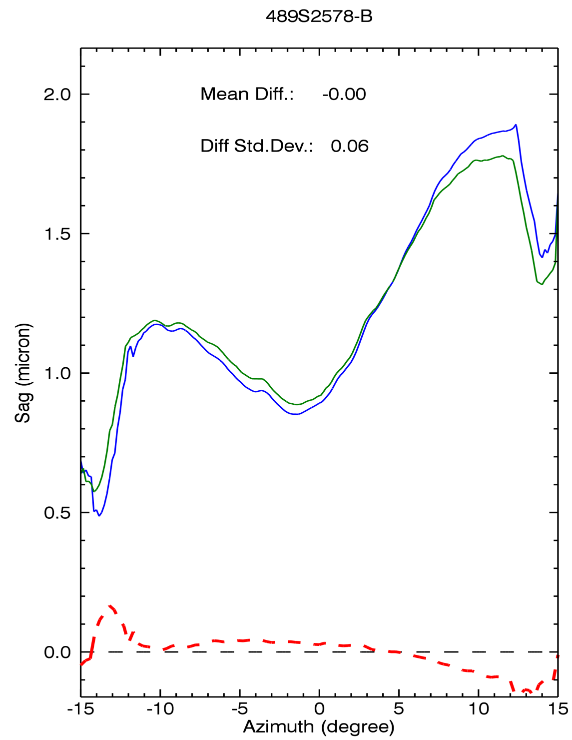
(1) Annealing

- 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

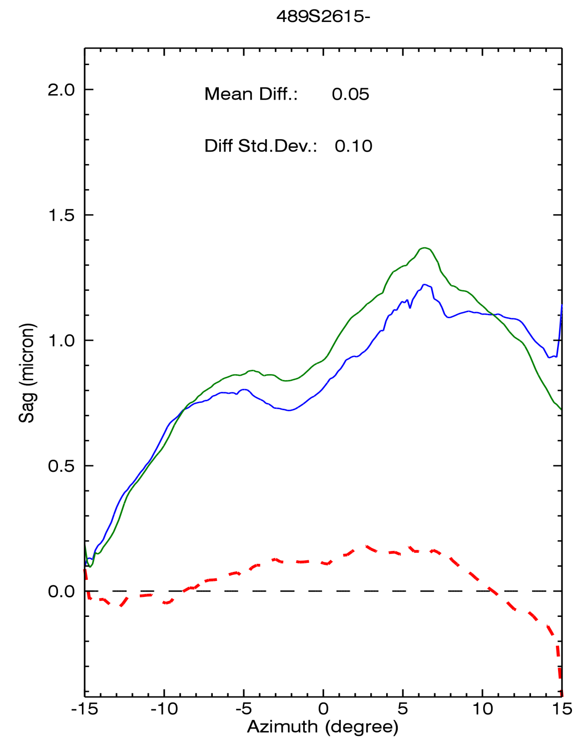
Annealing does not change substrates

Bare glass substrates heated up to 410°C

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



350 C



410 C

Substrates are not affected after heating

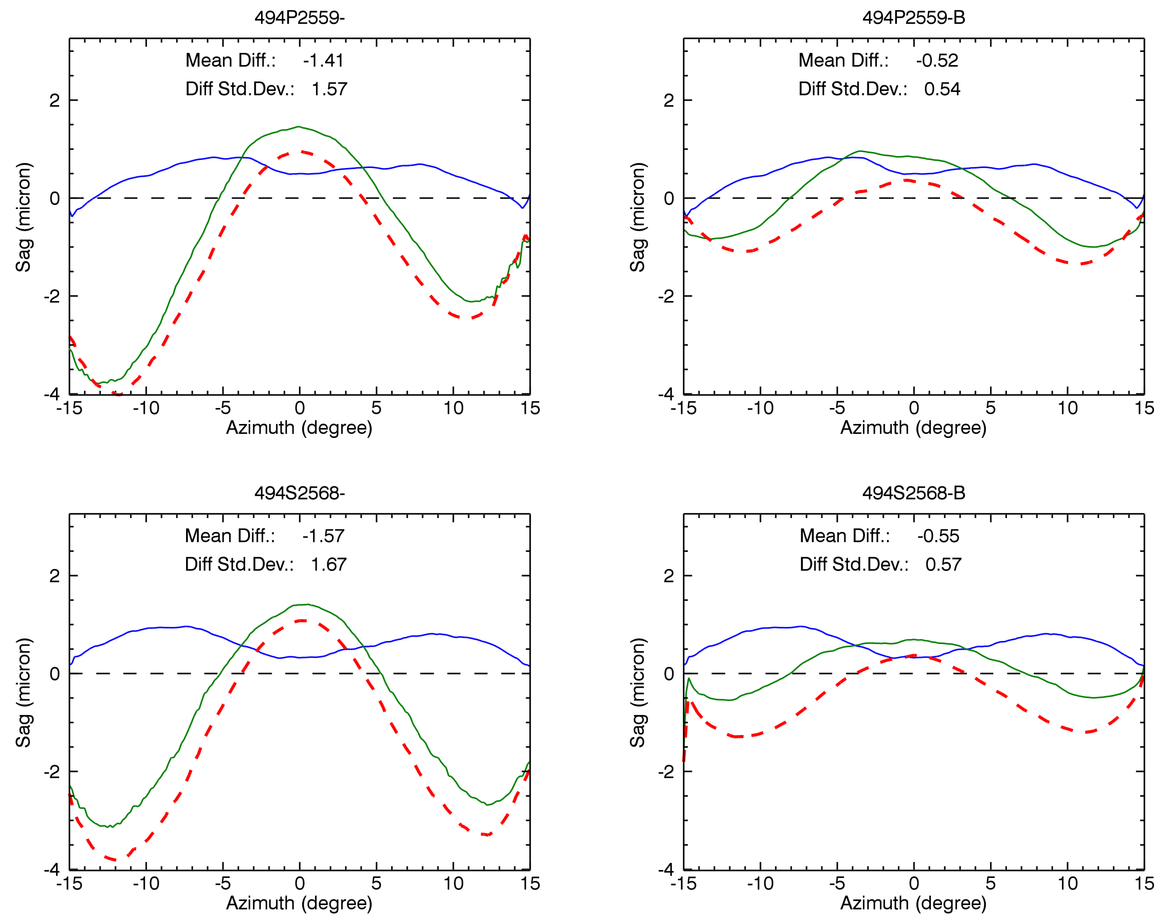
Coating and Annealing

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

Example shown: 20 nm ➔

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

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

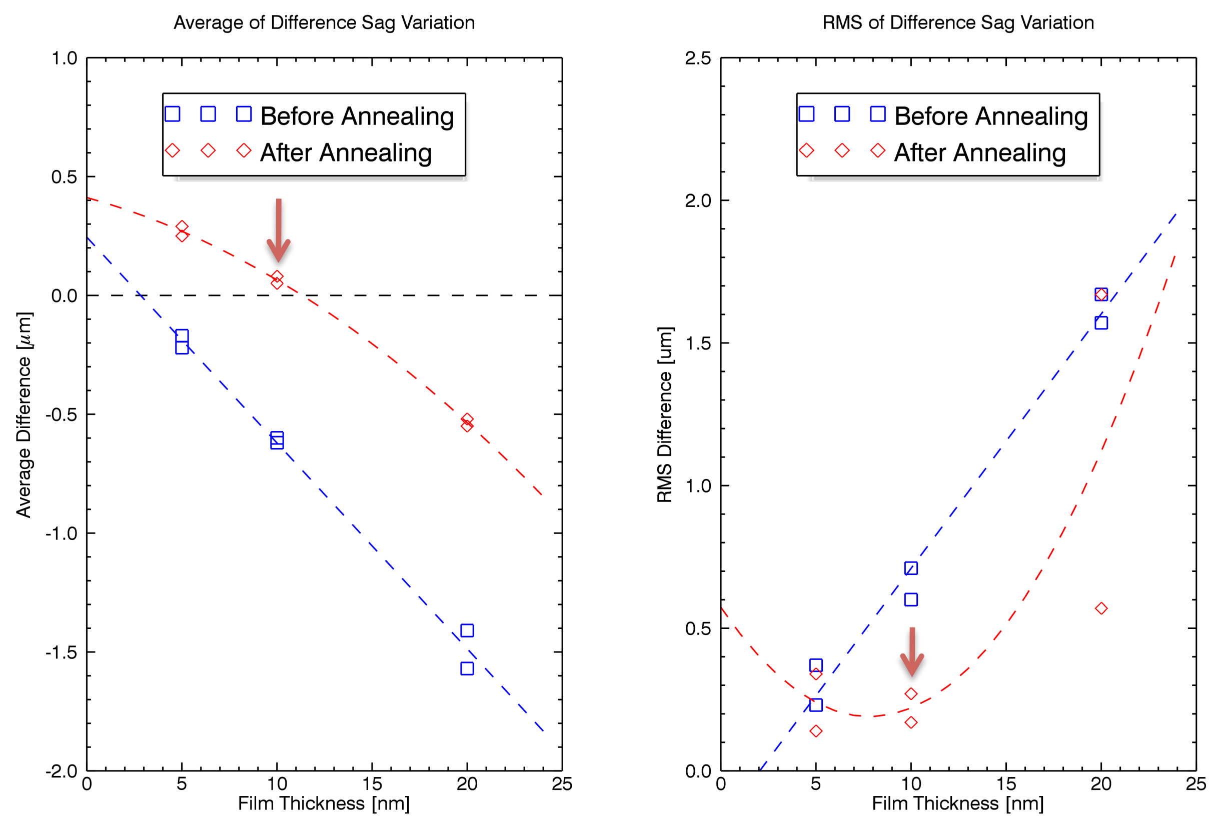


Before Annealing   After Annealing

Thickness Dependence

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 of Film Annealing on Thickness: 01/10/2013

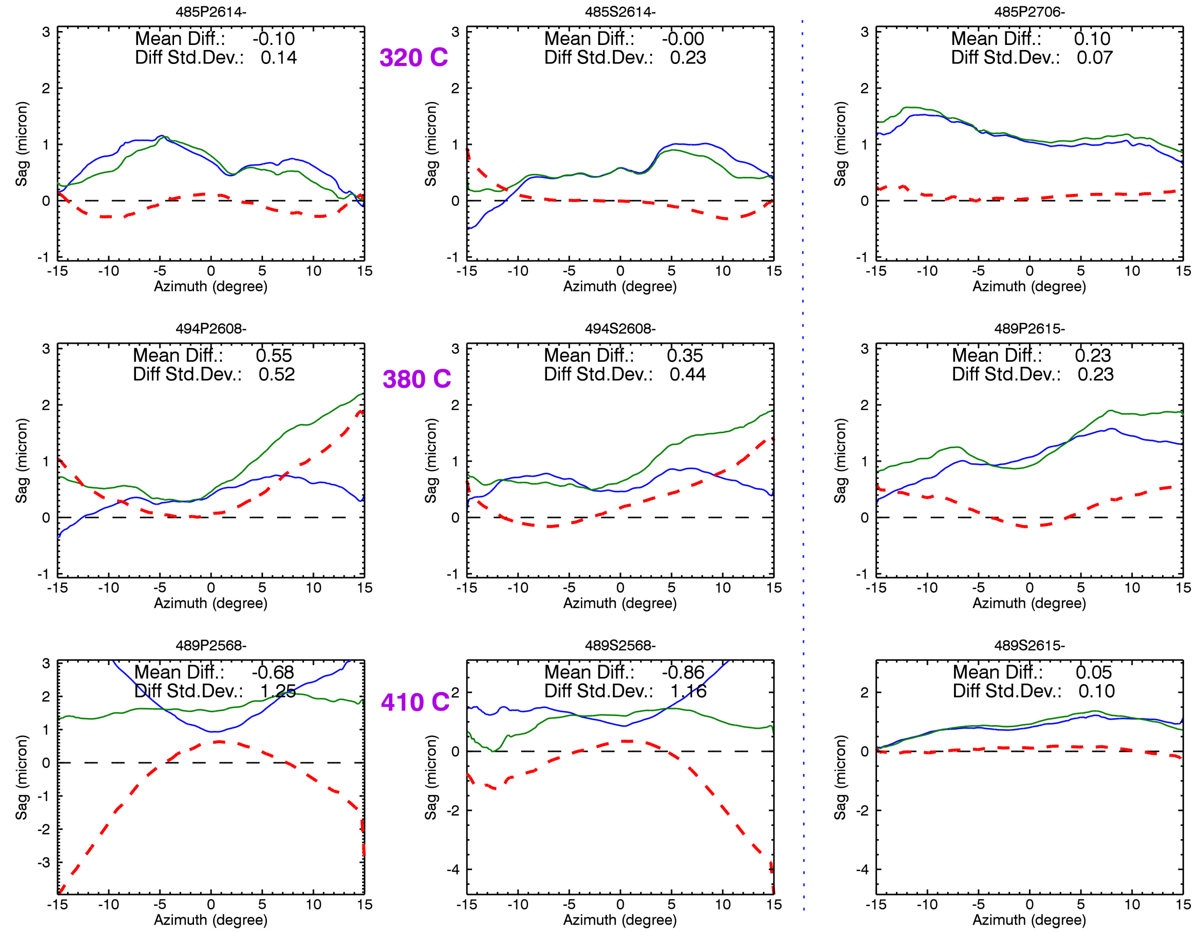
Minimal distortion at *d* ~ 10 nm

Dependence on Annealing Temperature

320°C ➔

380°C ➔

410°C ➔



2 mirrors for consistency

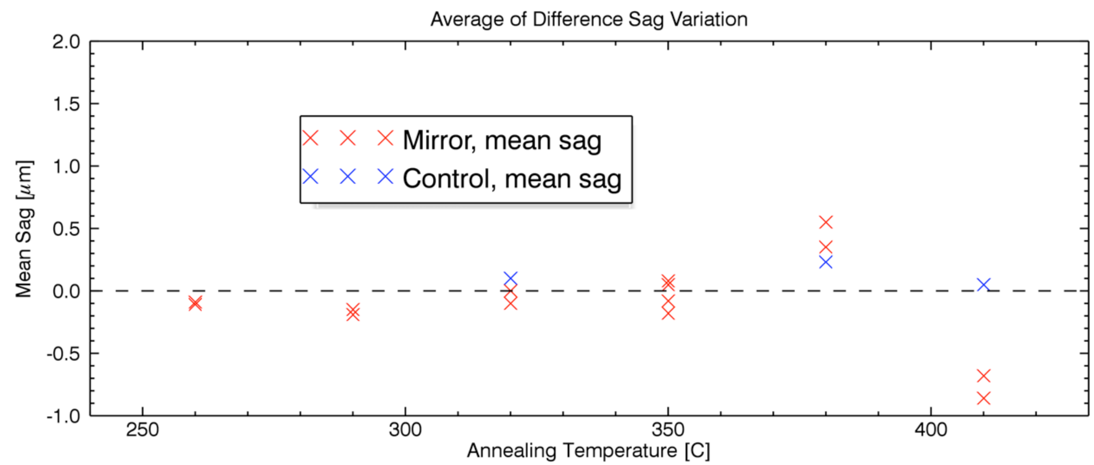
Substrate as control

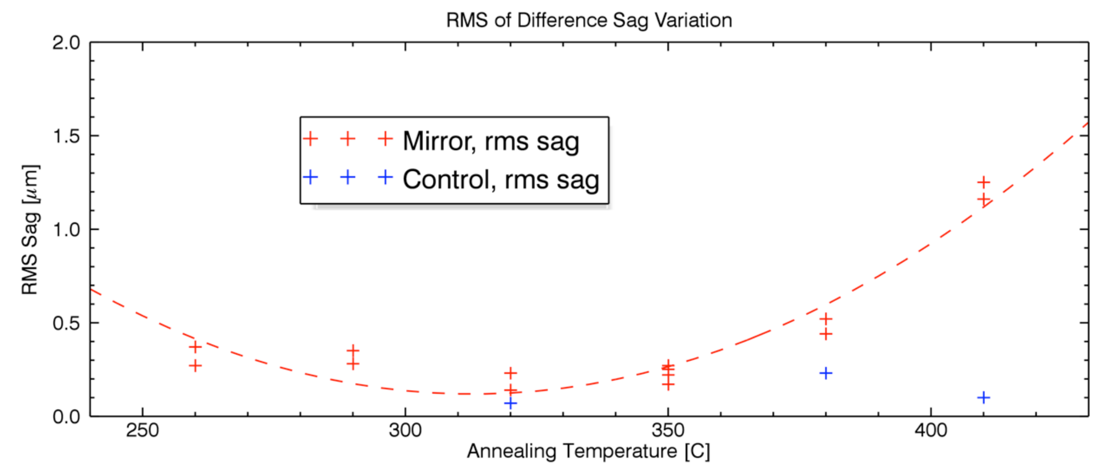
Temperature Dependence

Non-monotonic trend

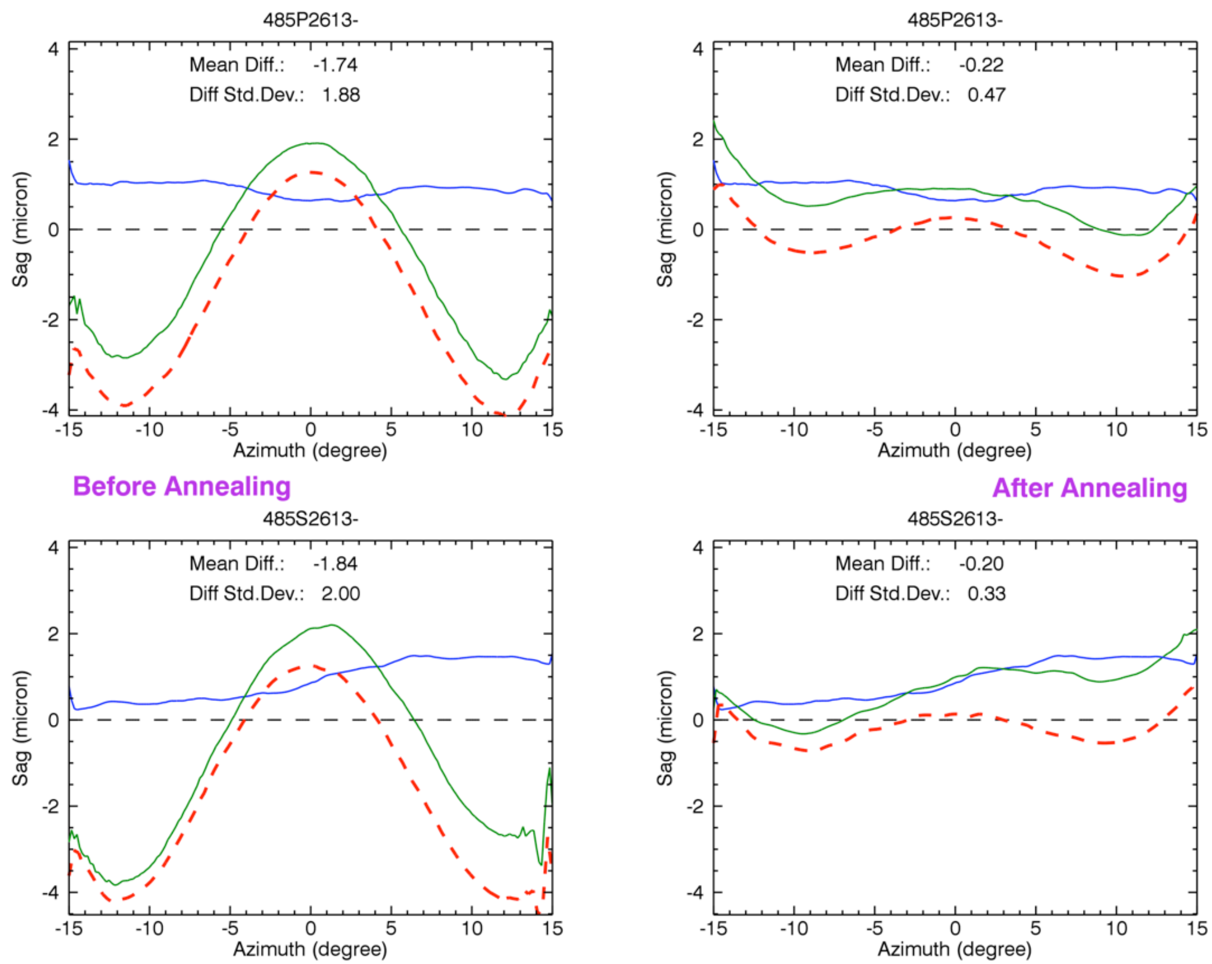
– Larger distortion may be induced by CTE-mismatch at higher temperature

Optimal Temperature at ~ 310°C





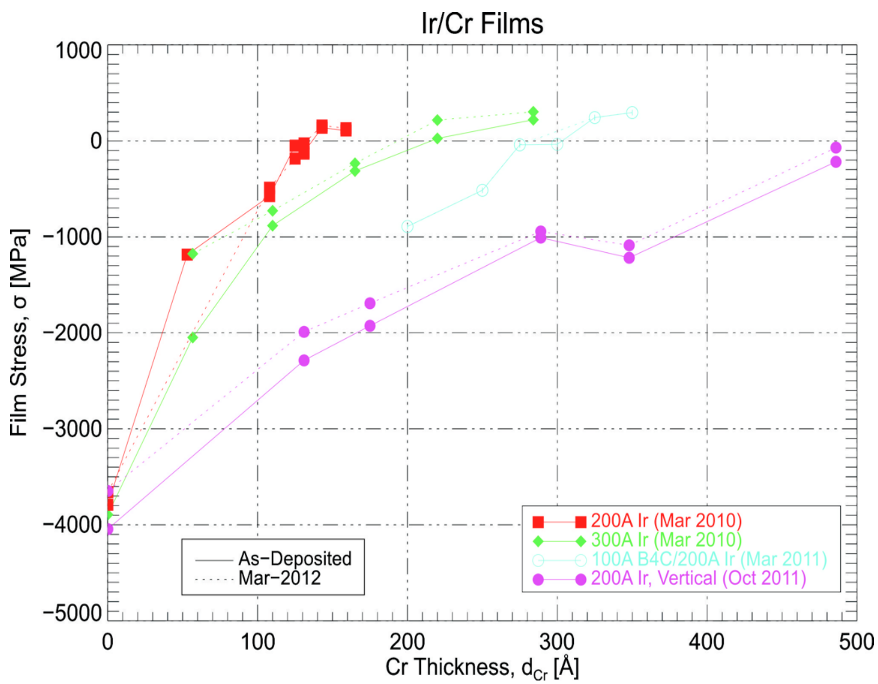
Annealing



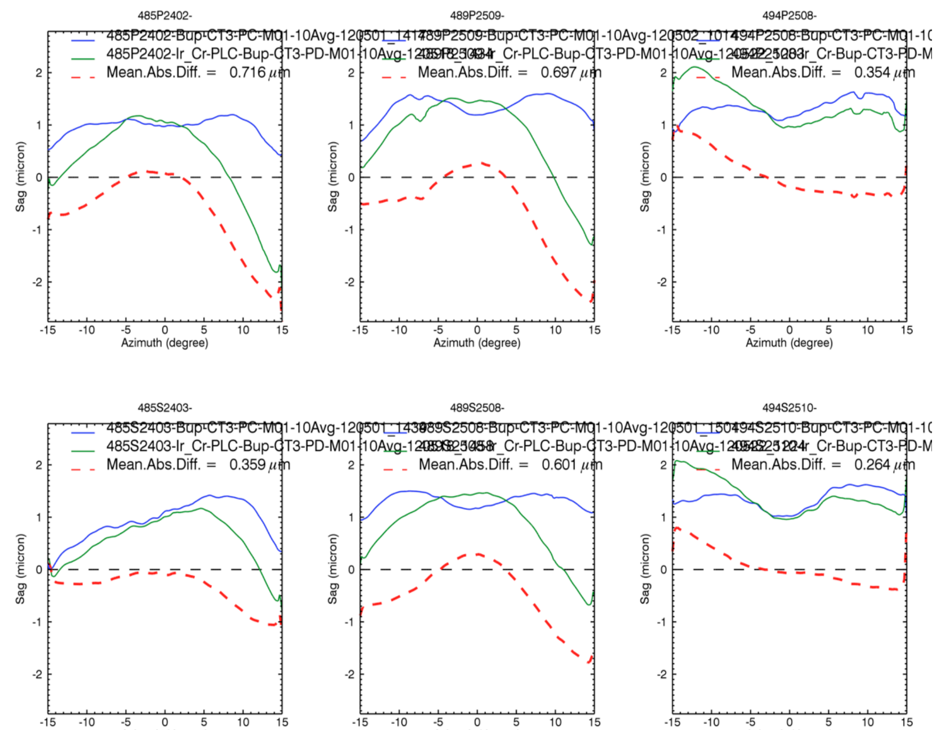
485P2613-
Mean Diff.: -1.74
Diff Std.Dev.: 1.88
Sag (micron)
Azimuth (degree)
485P2613-
Mean Diff.: -0.22
Diff Std.Dev.: 0.47
Sag (micron)
Azimuth (degree)
Before Annealing
After Annealing
485S2613-
Mean Diff.: -1.84
Diff Std.Dev.: 2.00
Sag (micron)
Azimuth (degree)
485S2613-
Mean Diff.: -0.20
Diff Std.Dev.: 0.33
Sag (micron)
Azimuth (degree)

(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)

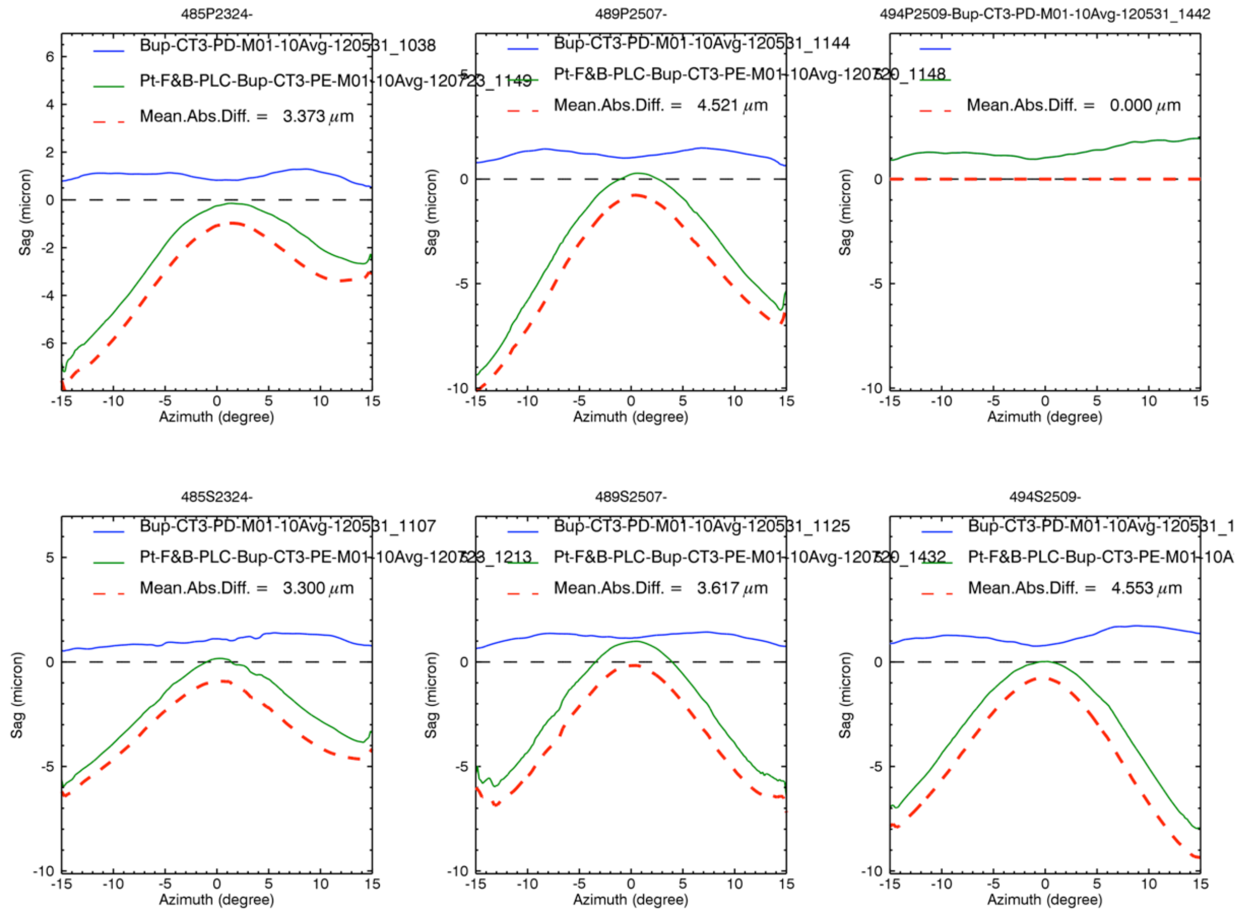


+221 MPa | -426 MPa | -887 MPa

(3) Atomic Layer Deposition

Pt coating

Did not balance mirror distortion

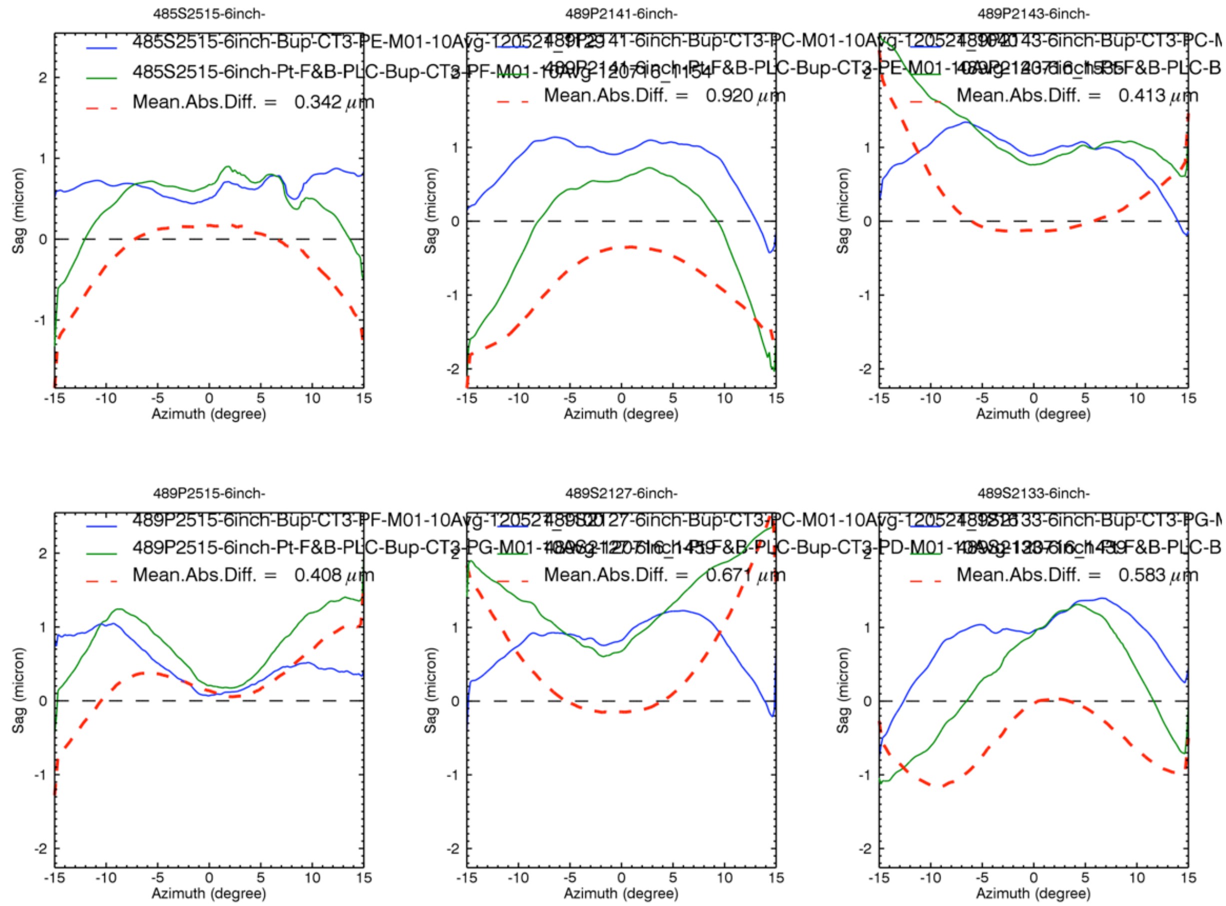


* Coating done at Cambridge NanoTech, Cambridge, MA

Atomic Layer Deposition

Pt coating

Did not achieve the desired effect of balancing mirror distortion

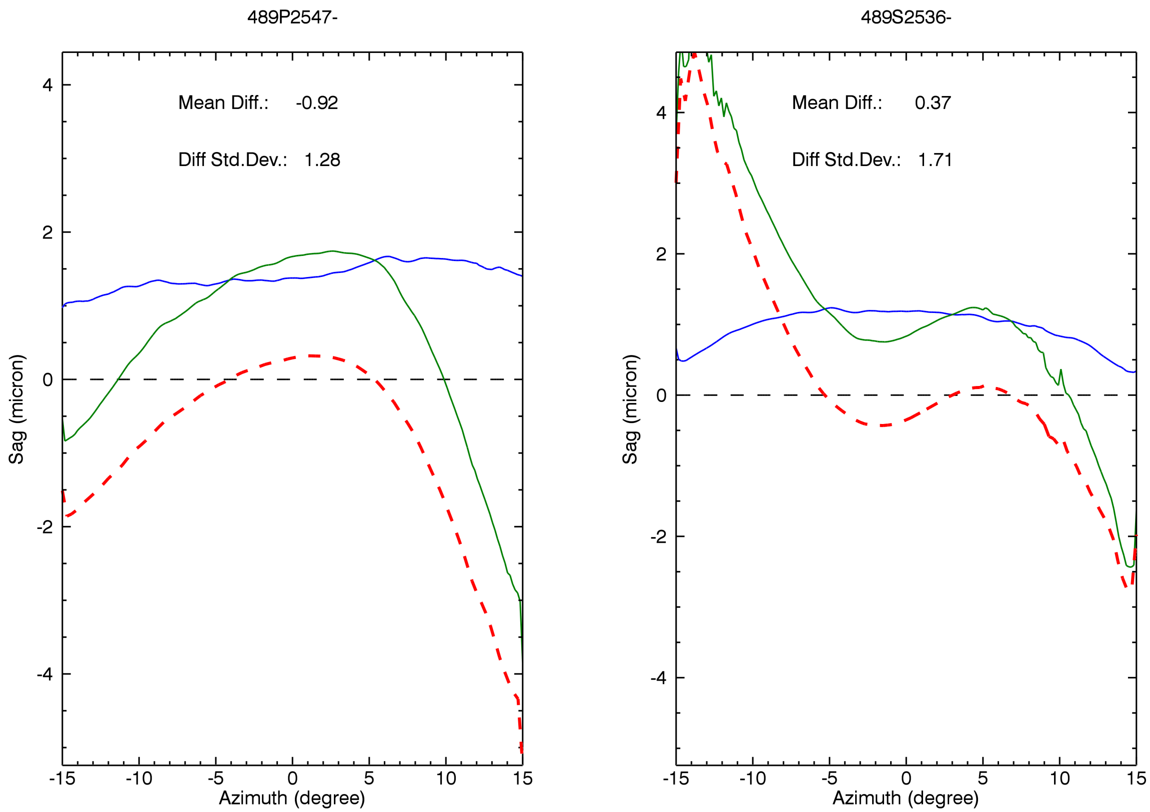


* Coating done at Arradiance, Sudbury, MA

Atomic Layer Deposition

Ir coating

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

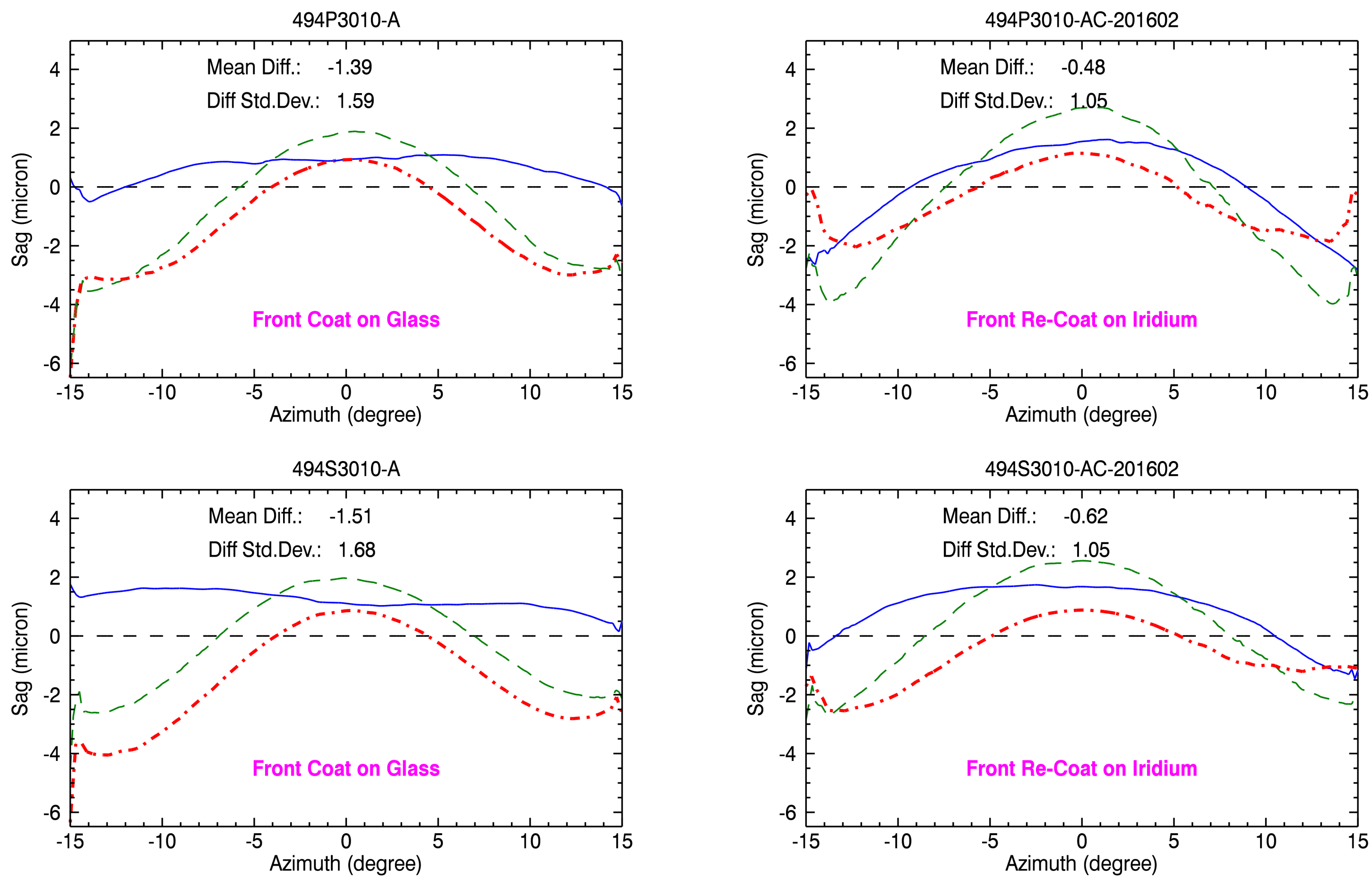


* ALD coating of Iridium done at Beneq Oy, Finland

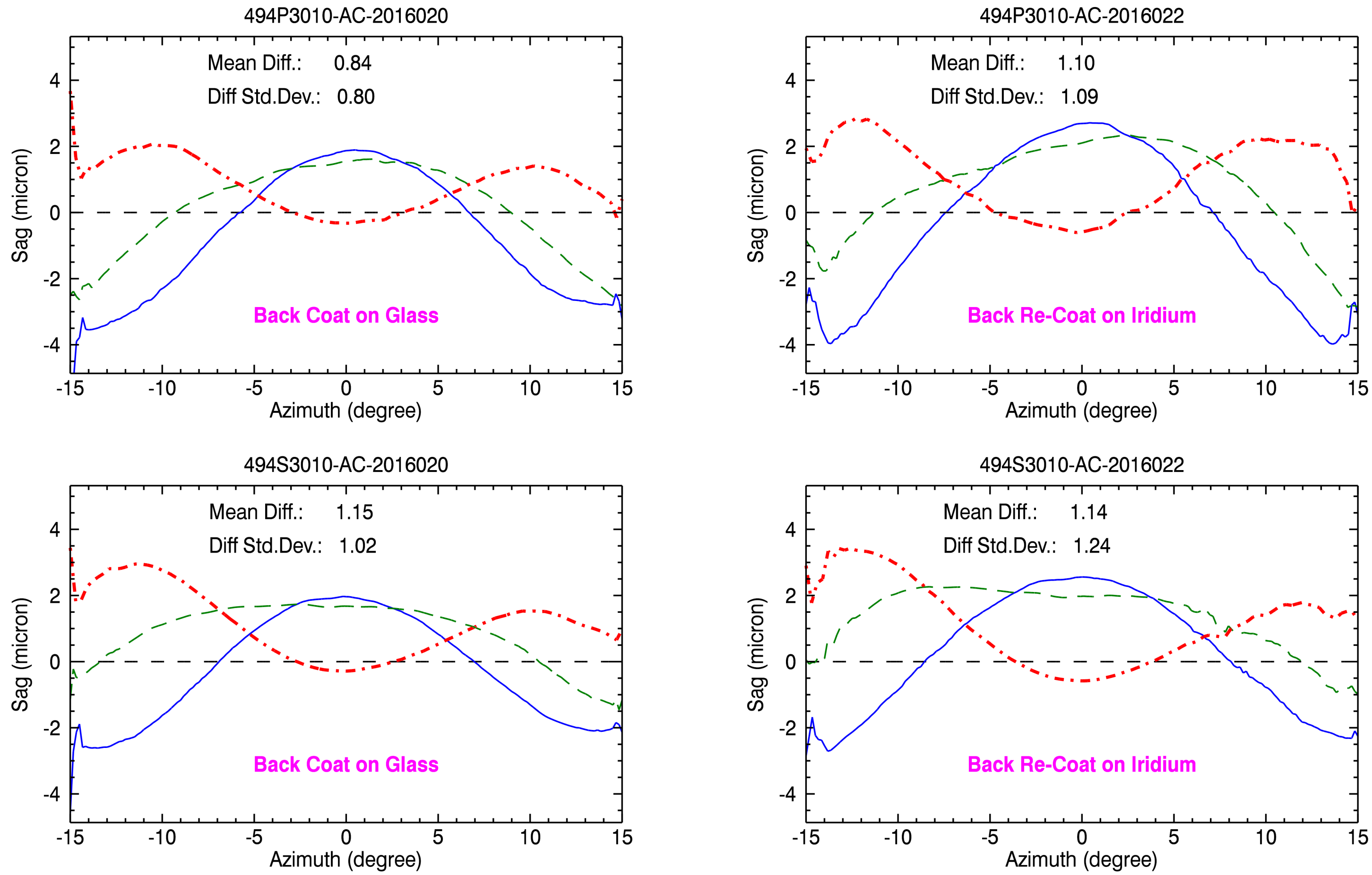
(4) Front + Back Deposition: sputtering

- 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

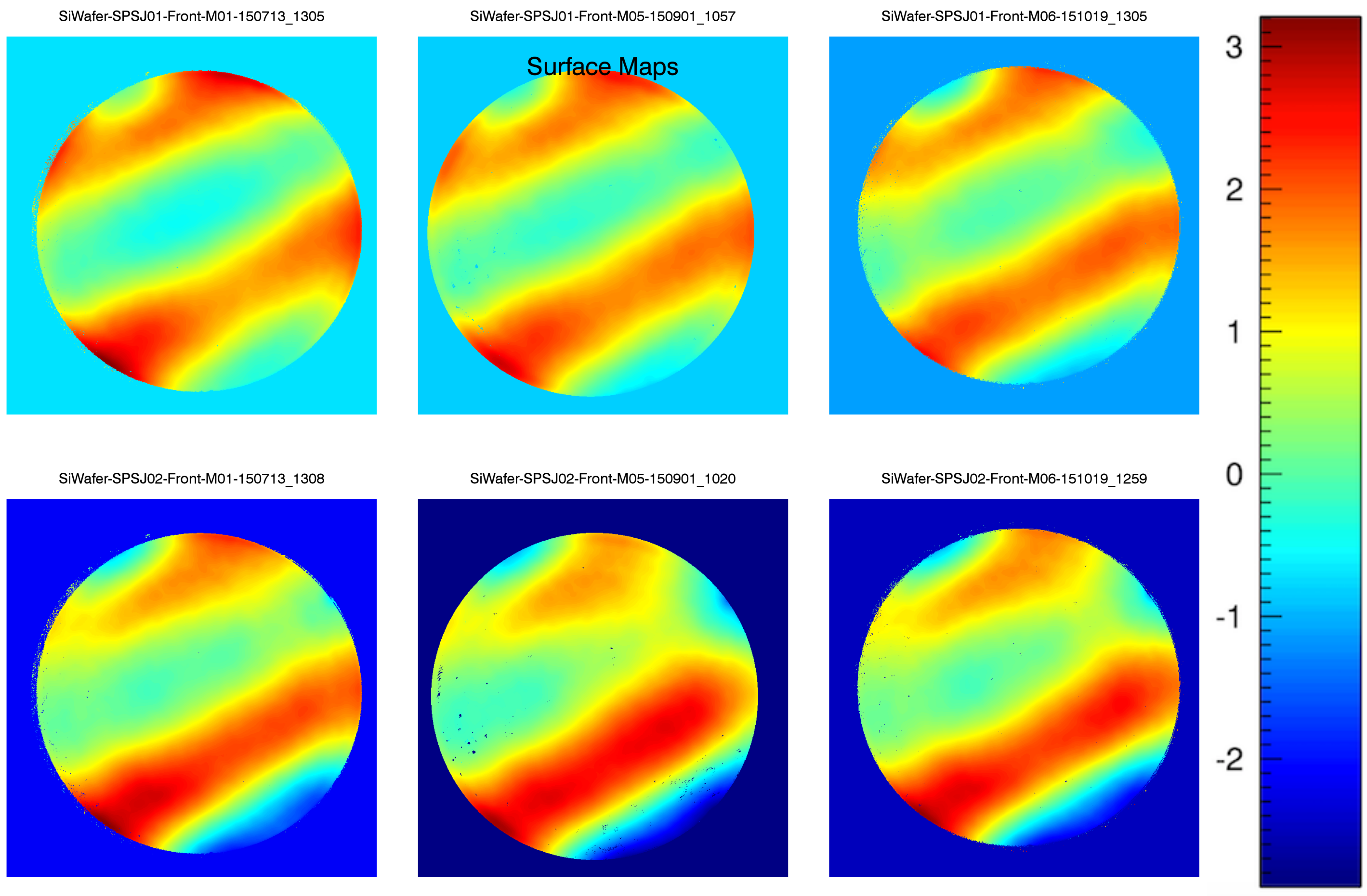


Comparing coating on bare glass (back side) vs. on Iridium-coated surface using front-and-back magnetrons



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.