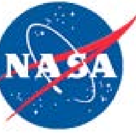


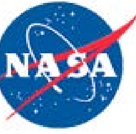
Improved Lyman Ultraviolet Astronomy Capabilities through Enhanced Coatings

By

Manuel A. Quijada

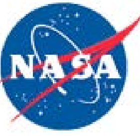
Collaborating Institutions:
- Goddard Space Flight Center (GSFC)
- University of Maryland (UMD)
- Naval Research Laboratory (NRL)

Outline



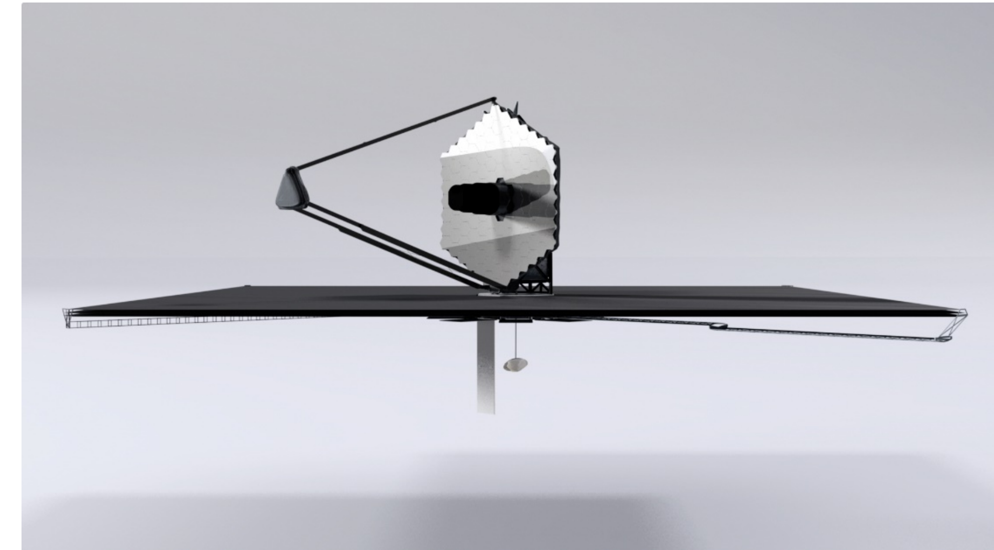
- Overview & Objectives
- Program Element Updates
  - ✓ Research Chamber fluorination updates (GSFC)
  - ✓ ALD growth of AlF_3 films (UMD)
  - ✓ 2-meter chamber Ion-Assisted deposition upgrades (GSFC)
  - ✓ E-beam plasma processing (NRL)
- Path Forward
- Conclusions
- Acknowledgments

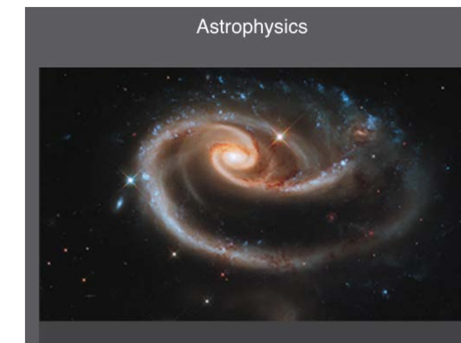
Overview and Objectives

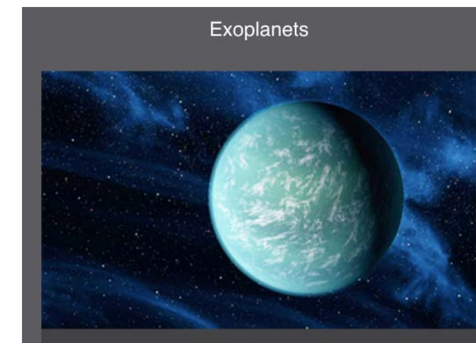


- **Task Description**
  - ✓ Deposit high performance optical broadband (FUV -> IR) mirror coatings:
    - ✓ Fluorination/passivation of Al-based coatings.
    - ✓ Atomic Layer Deposition (ALD) layers of AlF_3.
    - ✓ Ion assisted depositions for low-absorption metal-fluoride to protect Al mirrors.
- **Driver / Need**
  - ✓ Broadband coatings (90-10,000 nm) have been identified as an "Essential Goal" in the technology needs for a future Large-Aperture Ultraviolet-Optical-Infrared Space Telescope (LUVOIR and HabEx).
- **Benefits**
  - ✓ High throughput & high signal-to-noise ratio (SNR) over a broad spectral range.
  - ✓ Enabling technology for astrophysics and optical exoplanet sciences (in shared platform).

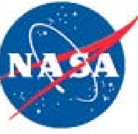
LUVOIR Concept Telescope

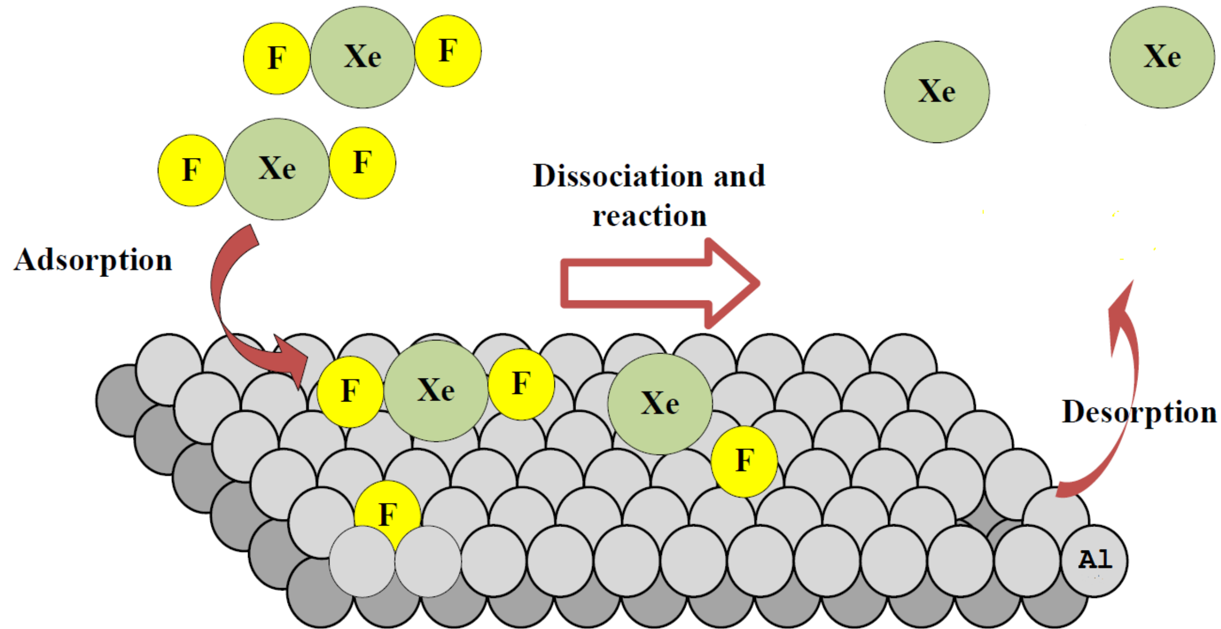






Hybrid PVD Passivation/Fluorination Chamber

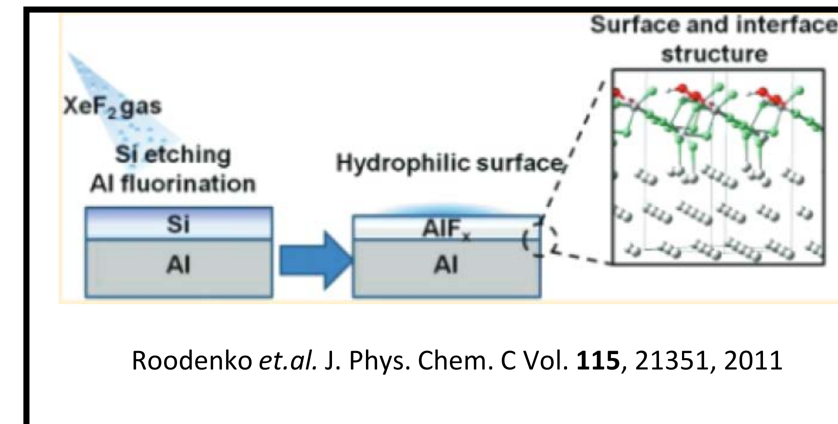




Reactive fluorine compound with low bond energy used (e.g. XeF_2 with 133.9 kJ/Mole).

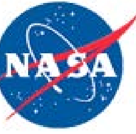
Heating of the XeF_2 may also be used if compound is not sufficiently reactive for increased selectivity.

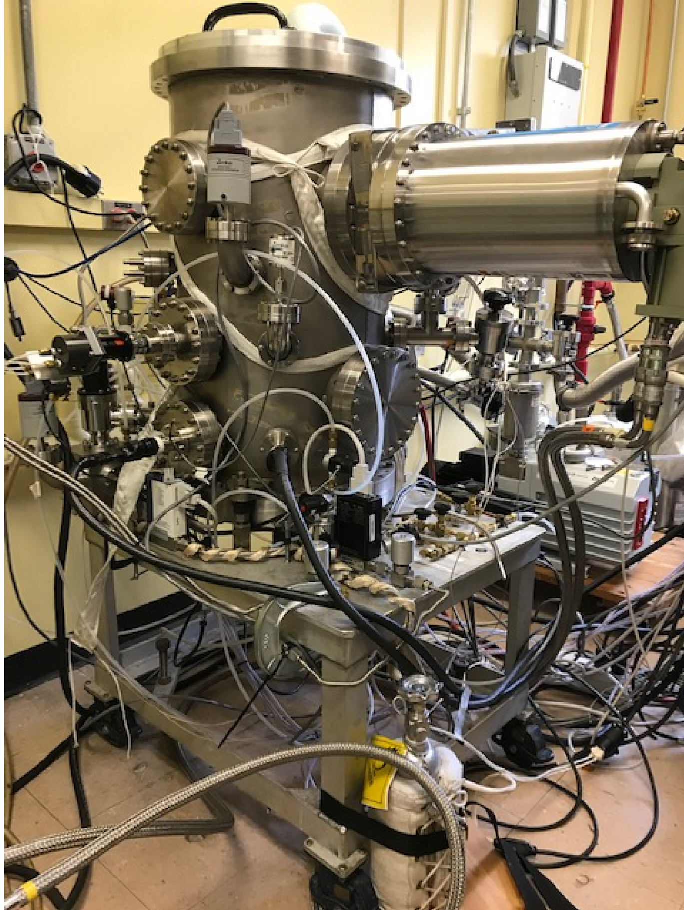
XeF_2 is a dry-vacuum based method of reaction and requires no plasma or other activation minimizing damage to substrate.



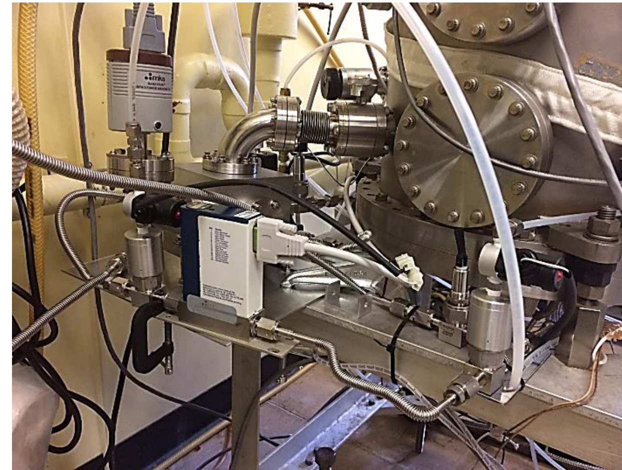
Roodenko *et.al.* J. Phys. Chem. C Vol. **115**, 21351, 2011

Research Coating Chamber Upgrades

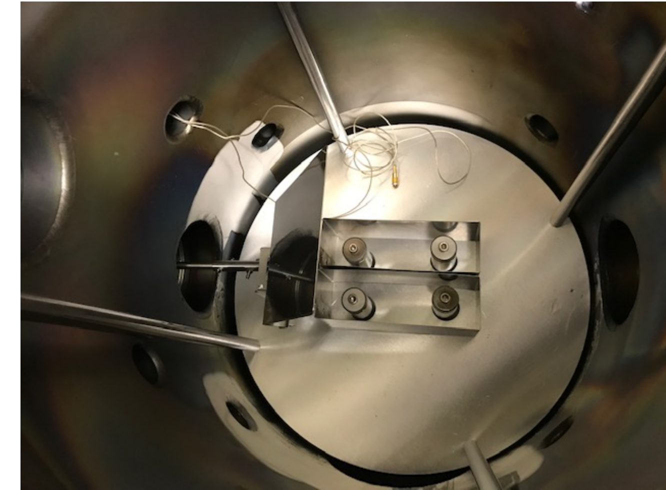




UHV Research Chamber capable of thin film physical vapor deposition (PVD) and passivation.



XeF_2 Gas feed components capable of continuous flow or pulsed flow.

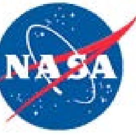


Inside view of RC with 2-materia PVD deposition system.

R&D for combined PVD & fluorination of Al-based high performance FUV coatings.

Chamber is ready to be used and experimentation will start in 1-2 weeks (when FIREBALL primary mirror re-coating is done).

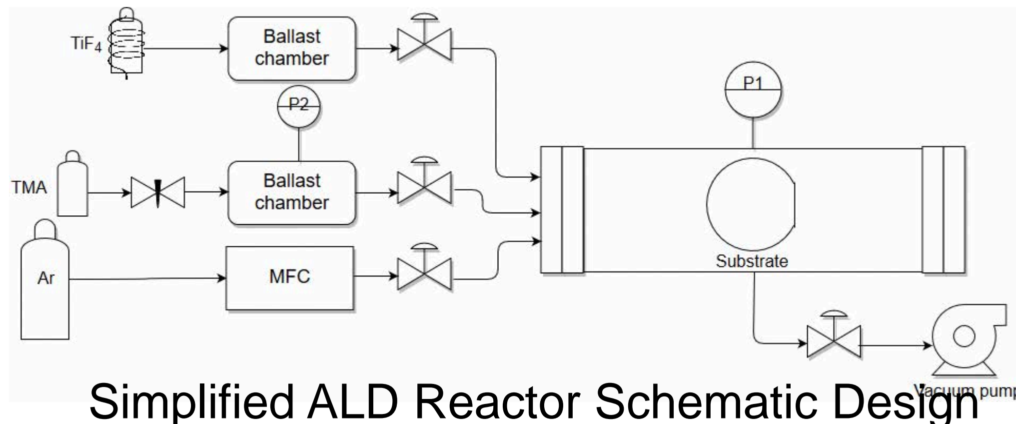
ALD Precursors For AlF_3 Depositions



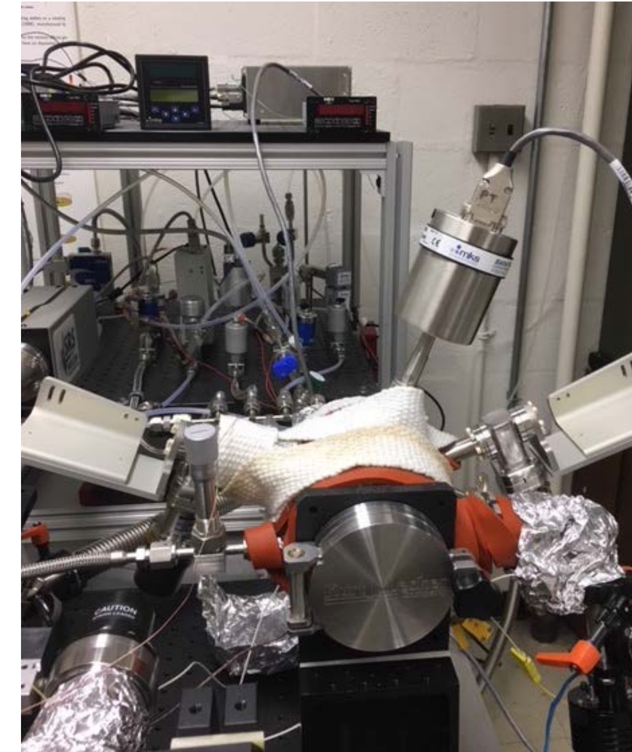
Investigation of TMA and TiF_4 precursor systems:

Trimethyl aluminum (TMA) - Aluminum source
- Has been studied extensively for ALD use.
- Good vapor pressure.

Titanium tetrafluoride (TiF4) - Fluoride source
- Has several cases of use in ALD; not as well-established.
- Requires heating to generate vapor pressure.
- Moderately acidic fluoride (as opposed to HF).
  Does not require additional activation (e-gun/plasma).



Simplified ALD Reactor Schematic Design

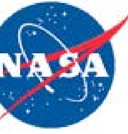


ALD Reactor at UMD

General-purpose ALD reactor features:

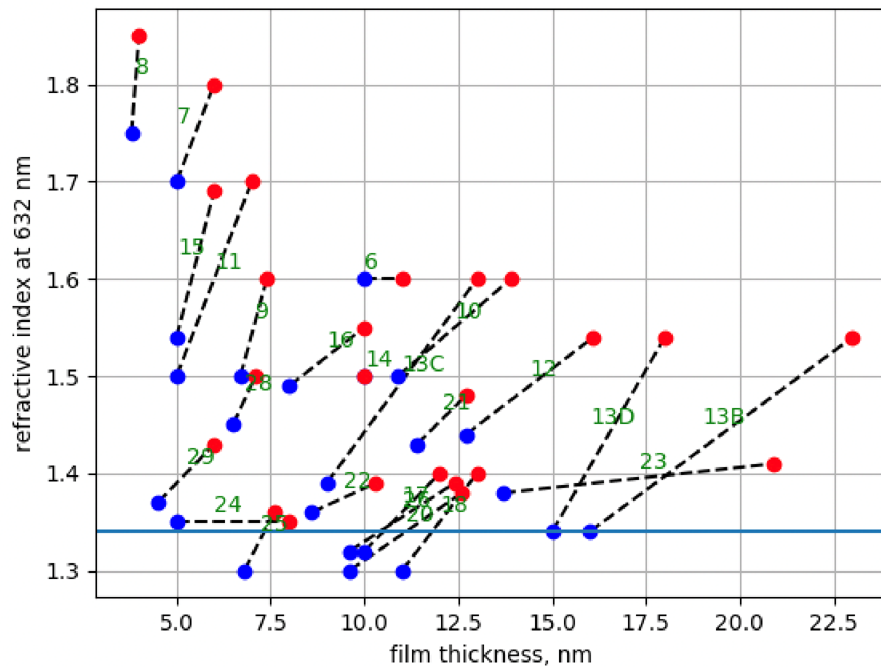
- The ALD process utilizes solid state halide precursors (TiF_4 and Trimethyl Aluminum (TMA) for the deposition of AlF_3 films.
- Solid state precursors precursor manifold plumbed for Ar, TMA, water, DEZn' room for 3 additional precursors.
- Optical access ports for real-time ellipsometry.
- Exhaust gate valve for "exposure" –mode operation.
- Accepts up to 2 inch substrates.
- Residual Gas Analyzer.

AlF_3 ALD Growth

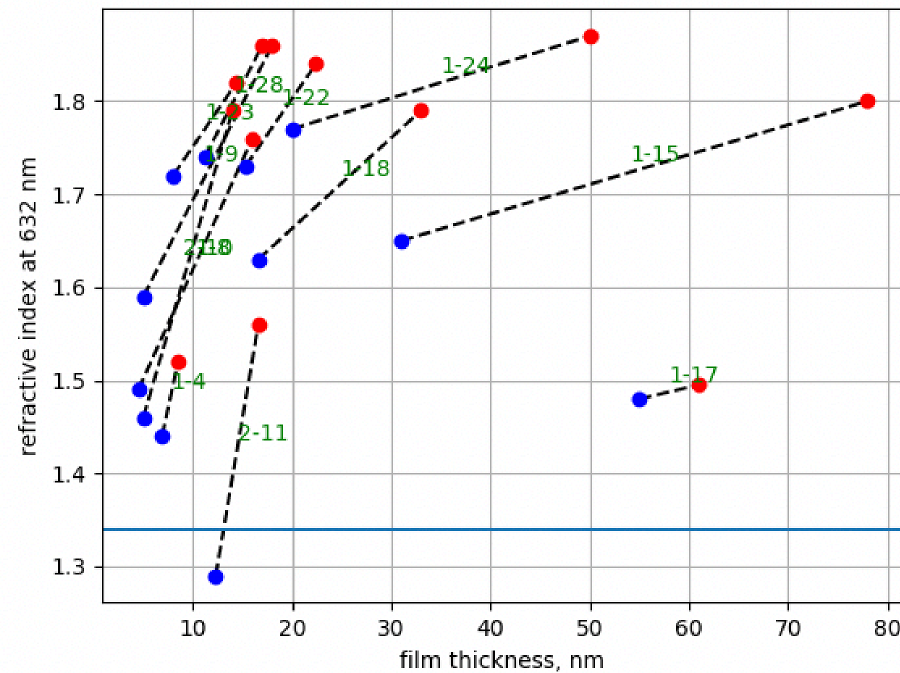


UMD graduate student (Alan Uy) who is conducting ALD growth of AlF3 as part of Ph.D. thesis.

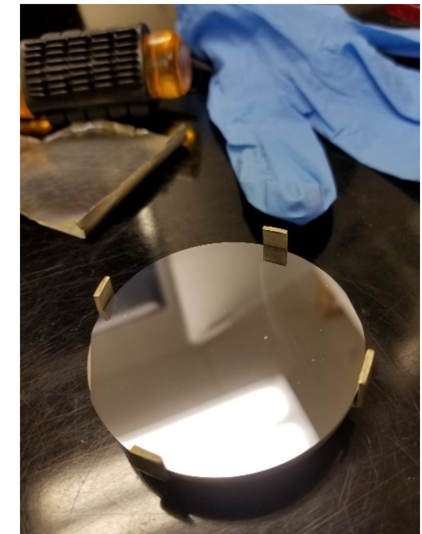
- Extensive metrology to determine film composition, thickness & refractive index.
- Reduction of particle growth and "streaks by heating the Ar gas during purge.
- Earlier deposition runs resulted in films that were thinner than desired; a refill of the TiF4 precursor and increased sublimate temperature resulted in substantially higher growth-per-ALD cycle.



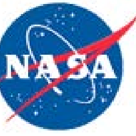
Measured refractive index vs. film thickness for initial ALD runs on Si wafer: Red/blue - Max/min value measured on wafer.



Measured refractive index vs. film thickness with significant TiF4 exposure on Si wafer: Red/blue - Max/min value measured on wafer.

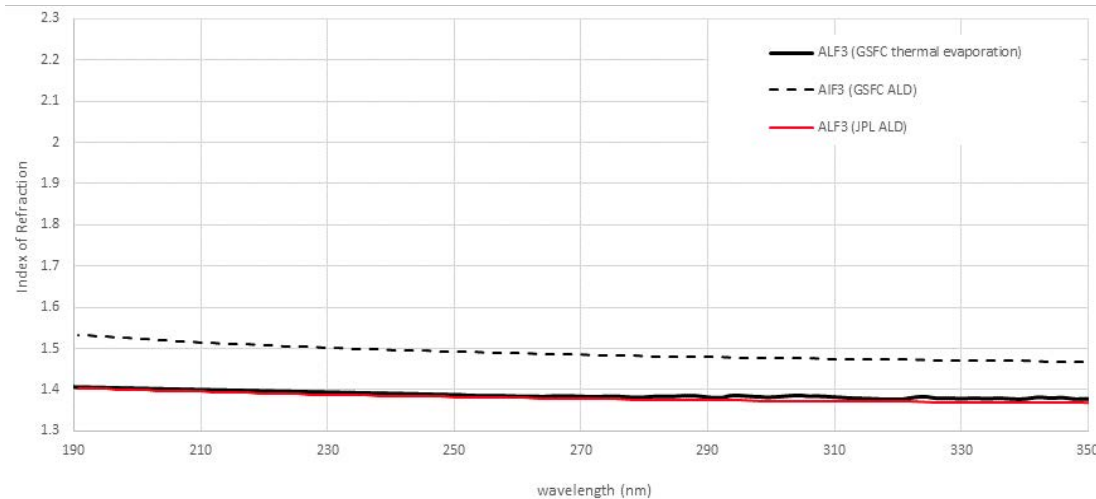


Ti and O Impurities



TiO_2 Particle generation on Film

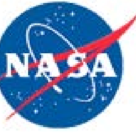
Element	At % **XPS**
Al	22
F	68
O	6
Ti	2.2 (40% as TiO_2)
C	1.5



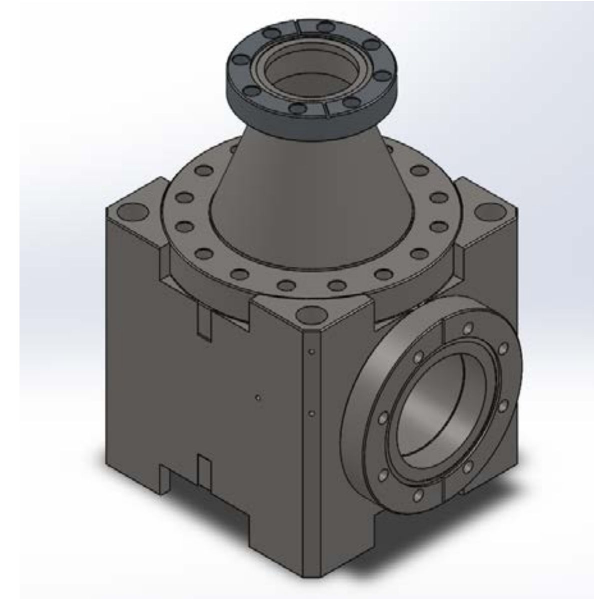
TiF_4(a) more reactive to H_2O than O_2, but only at high T
$\implies$ may occur when hot wafer is removed from reactor

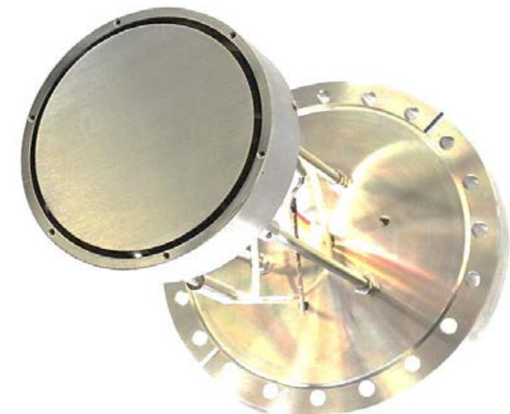
$3TiF_4(g) + 2A_2O_3 \rightarrow 4AlF_3 + 3TiO_2$
$\implies$ may be a route segment to TiO_2 contamination

ALD Reactor Re-design

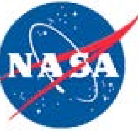


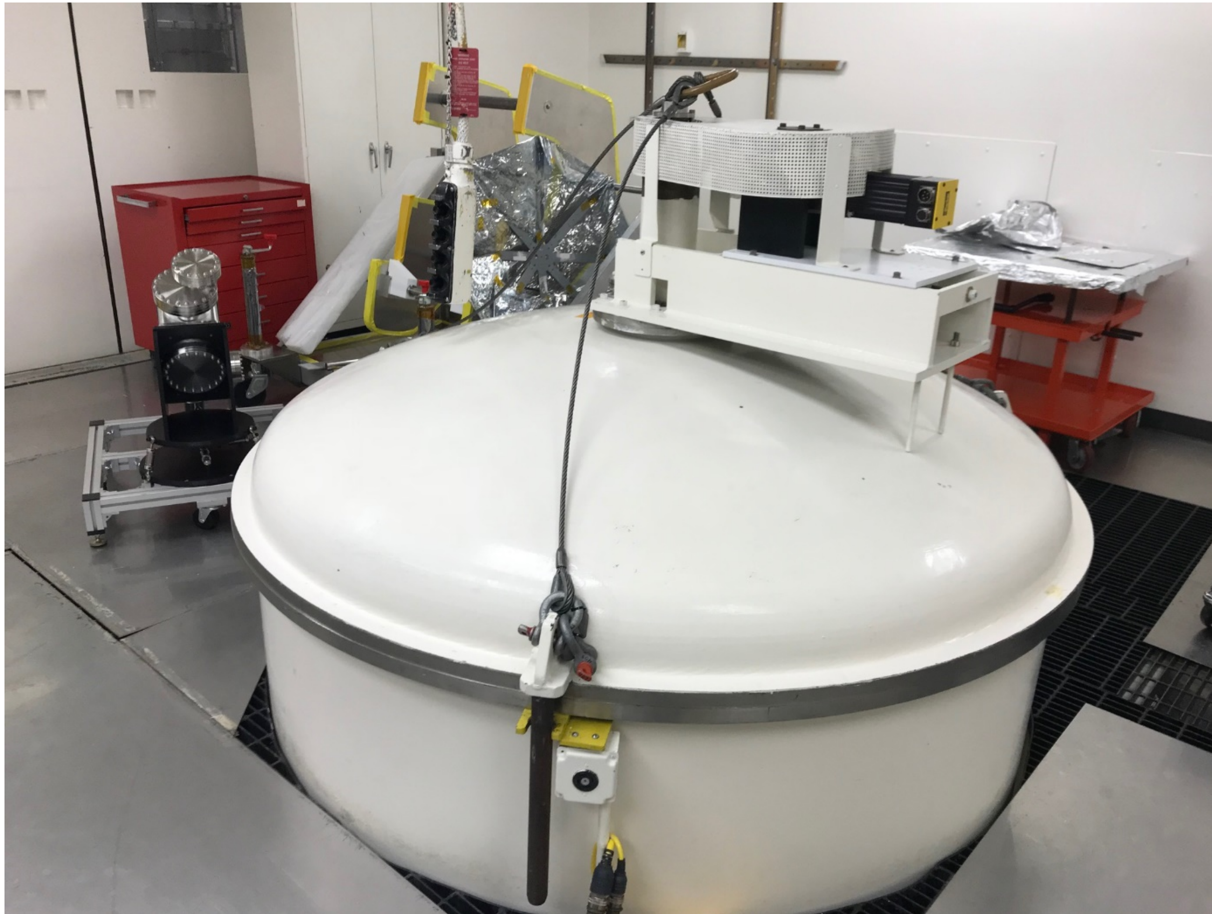
- Improve across-wafer thickness and composition uniformity by eliminating reactor flow asymmetries.
- Reduce film impurities by simplifying the reactor design to eliminate potential leaks.
- Improve reactor wall and precursor delivery system heating to assure the lack of particles and to reduce potential contamination sources.
- Consideration of a load-lock to keep as much O_2 and water out of the reactor chamber.





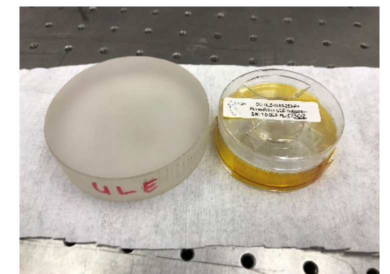
2-Meter Chamber Upgrades



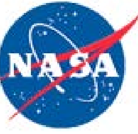


Deposition of a ion-assisted physical vapor deposition (IAPVD) of FUV-optimized Al+metal fluoride overcoats (LiF, MgF2, and Al+AlF_3) in the large 2-meter coating chamber.

- ✓ Acquisition of Ion Gun, optical monitor, deposition controller and PVD power supplies upgraded.
- ✓ Study of ZERODUR substrate to evaluate effect of heating on surface figure & wave-front error to demonstrate process on substrates traceable to a future telescope.



Ion-Assisted Coating Deposition

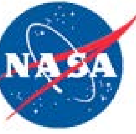


Procurement & installation of electron-gun for ion-assisted deposition to create more densely packed metal-fluoride coatings.

- Hollow Cathode (for operation without a filament).
- Deposition systems with critical dimensions greater than 1meter.
- Favorable film properties in packing density, stress, environmental stability and stoichiometry.
- Process Gases: Ar, Xe, Kr, O2, N2, Organic Precursors.
- Process: Pre-Cleaning, Surface Modification, Ion Beam Assisted Deposition, Direct Deposition.

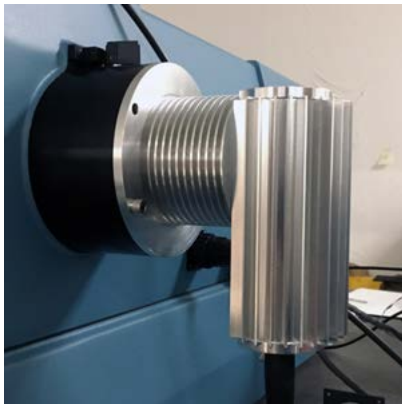
View of Ion gun in operation inside 2-meter Chamber.

Lyman-Alpha Optical Monitor



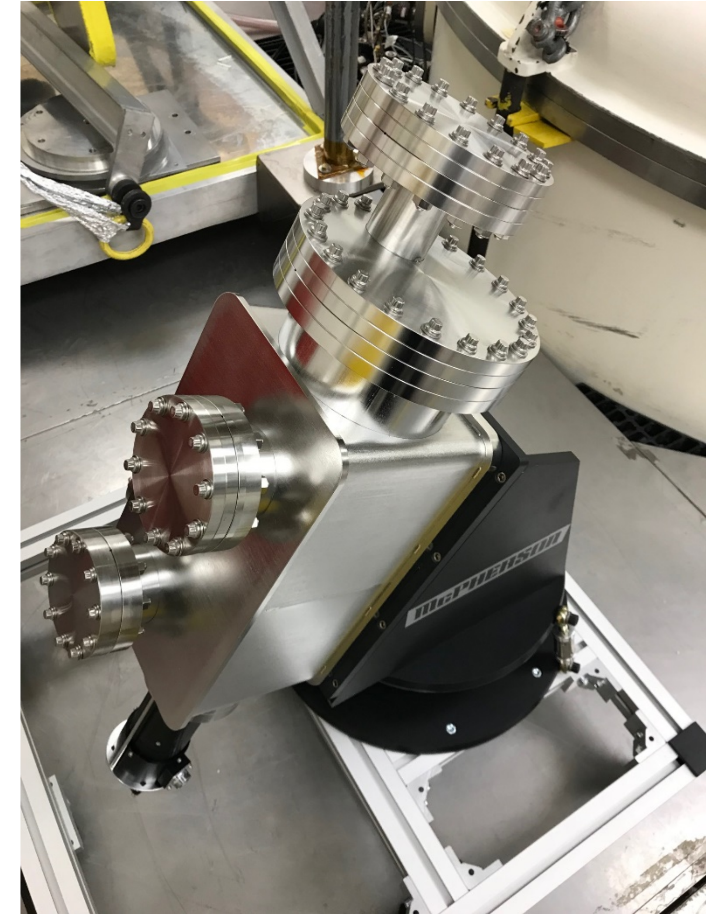
Collimator with 10 mm c.a.

- Procurement & installation of an in-situ optical monitor (λ = 121.6 nm); source, detector, port window, etc.
- System will produce a collimated beam that will manage to deliver the collected light to a 10mm spot over a meter away. It uses a ISO NW40KF flanges for mounting and beam path.



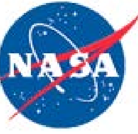
D_2 source with strong Lyman-Alpha output (MgF_2 window).

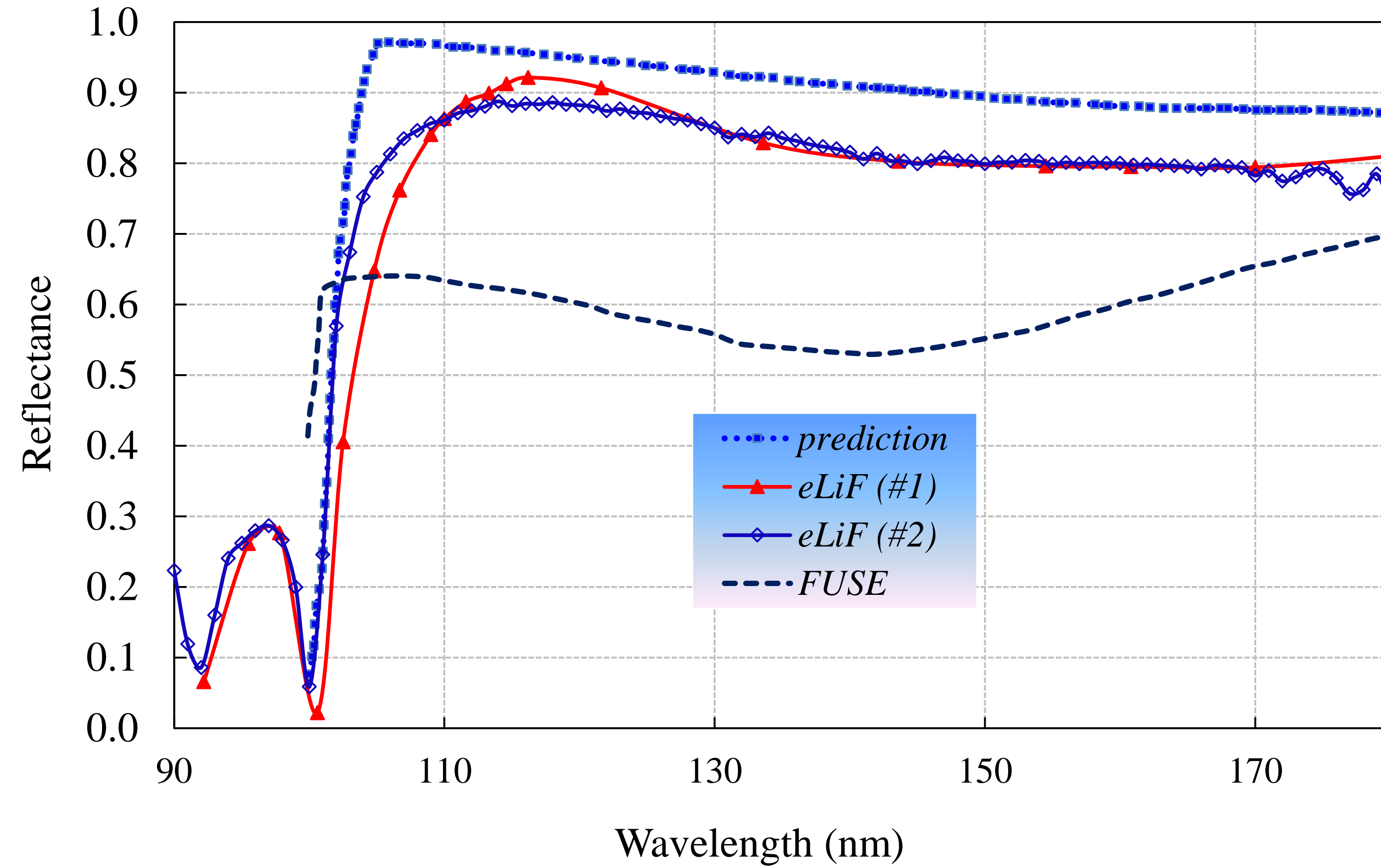
PMT Detector

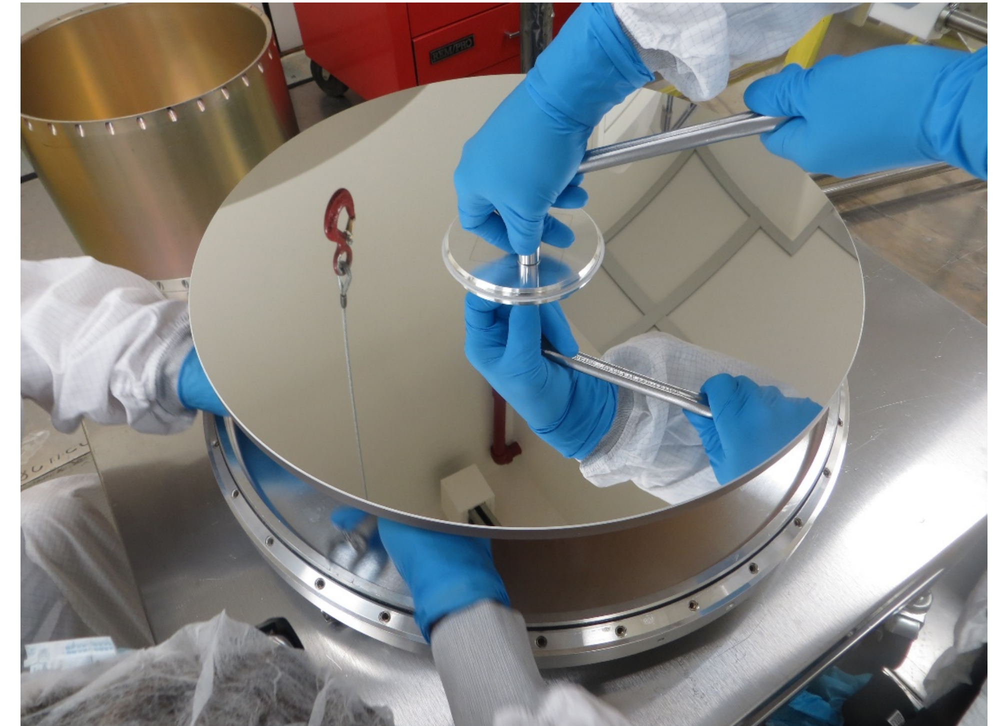


Complete Assembly as delivered

Optimization Al+LiF (eLiF) Hot Coatings

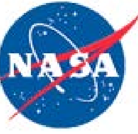


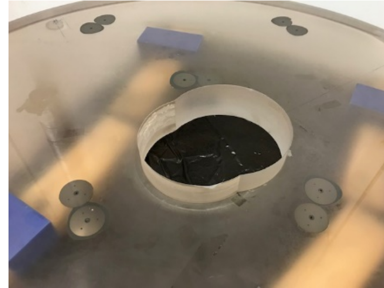


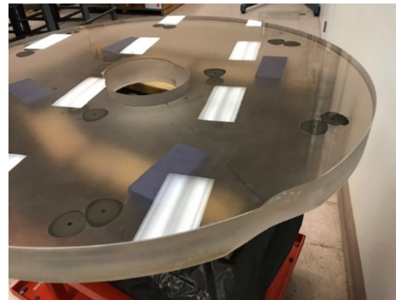


The SISTINE primary mirror after coating with Al+LiF in 2-meter chamber at GSFC.

FIREBALL-2 Primary Mirror Re-coating

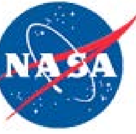


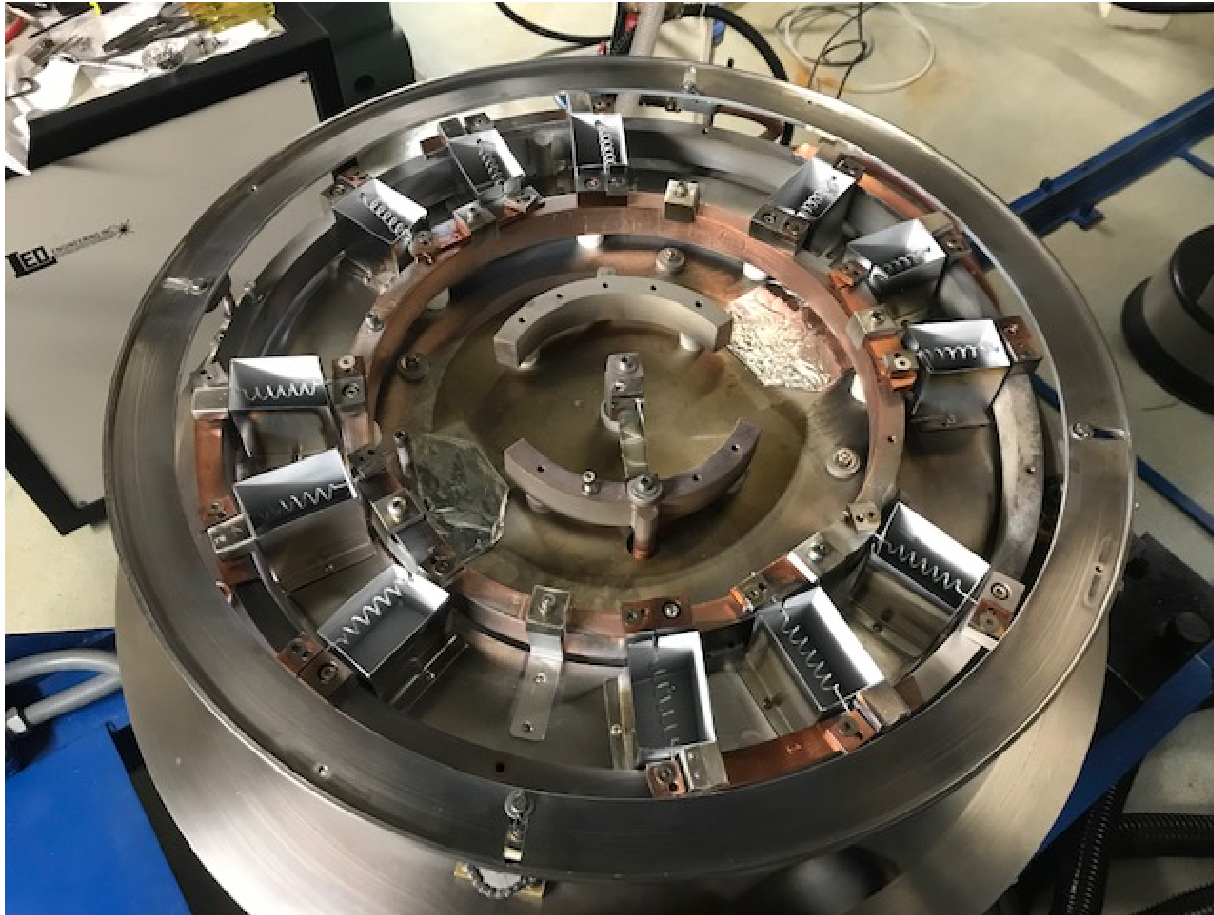




- **FIREBALL:** Faint Intergalactic Redshifted Emission Balloon
- Balloon-borne 1m telescope coupled to an ultraviolet fiber-fed spectrograph.
- Designed to study the faint and diffuse emission of the intergalactic medium.
- Primary mirror is at GSFC going through re-polishing and re-coating with Al+MgF_2 in preparation for launch in 2020.

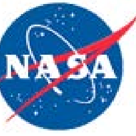
Al PVD Power Supply Reconfiguration





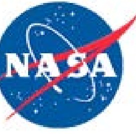
- Original power supply (5V, 3000 Amp) for Al deposition became unreliable.
- The Al deposition coils had to be reconfigured for a new power supply (30V, 500 Amp) in order to achieve the 100 A/ Sec deposition rates.
- Setup up time is much longer in this new configuration (to avoid shorting leads for each Al coil.
- Long term plan is to buy and replace original power supply (estimated cost~ $48k).

Applications of ZERODUR



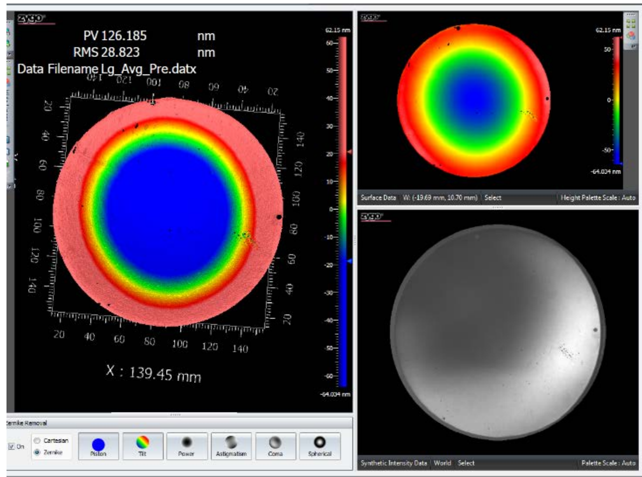
- ZERODUR® is a well-known mirror substrate material for ground and space based telescope application because of its low coefficient of thermal expansion ideal for ultra-stable optical systems.
- Coating of ZERODUR with enhance FUV coatings requires processing up to 250°C.
- Cooling ZERODUR® in the temperature range 130°C to 320°C with rates different from the typical production cooling rate of 3°C/h will change the CTE of the material with increasing temperature.[1]
- Changes in CTE can sometimes lead to changes in surface figure.[1]

[1] Ralf Jedamzik, Thomas Westerhoff, "Advices for the use of ZERODUR at higher temperatures," Proc. SPIE 10706, Advances in Optical and Mechanical Technologies for Telescopes and Instrumentation III, 1070634 (10 July 2018); doi: 10.1117/12.2311648



Process

Pre-Treat Measurement

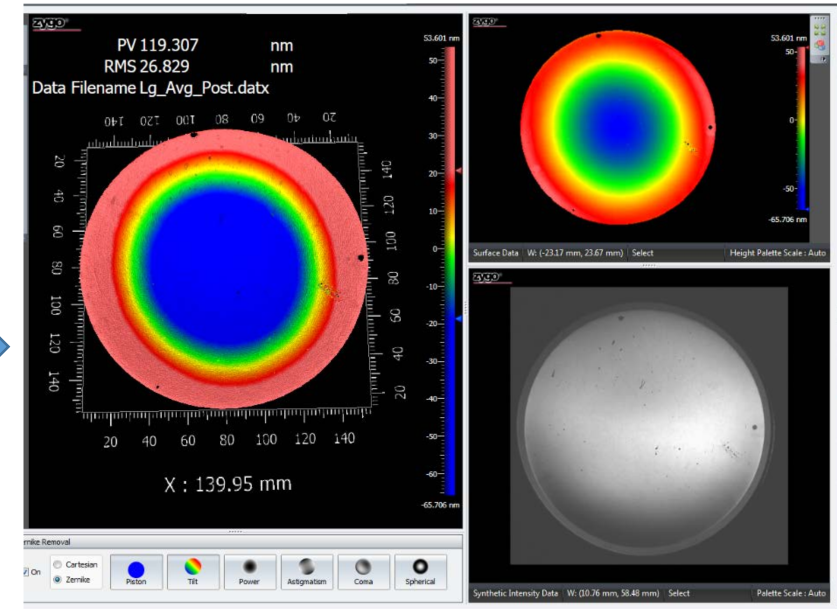


Zygo Interferometer

Heratherm Oven

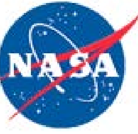
Coatings Chamber

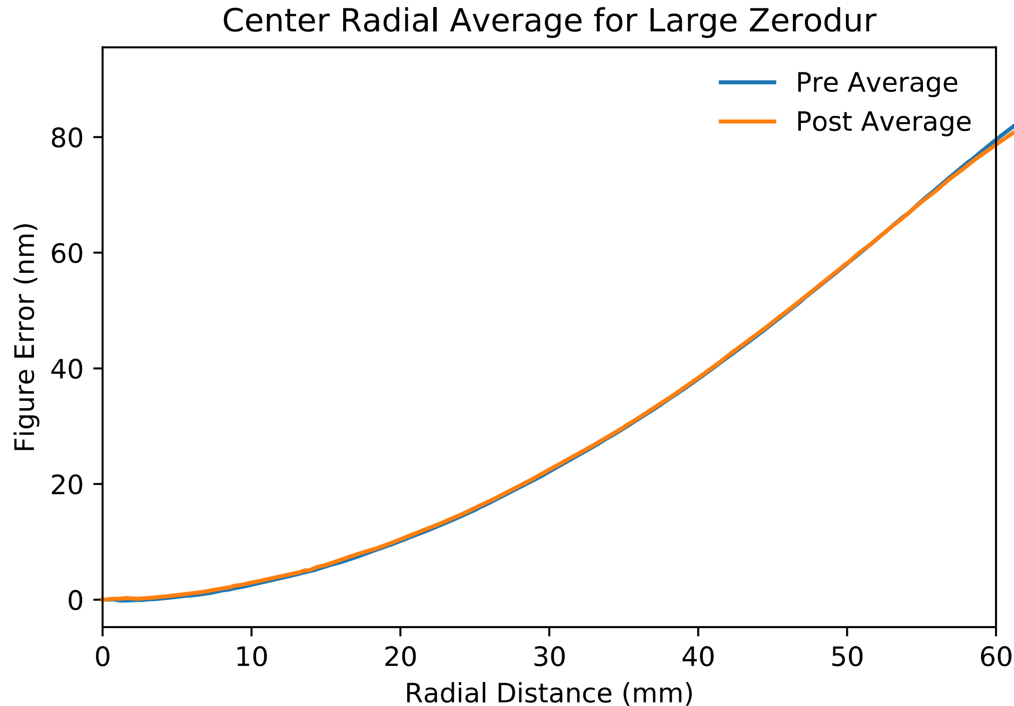
Post-Treat Measurement

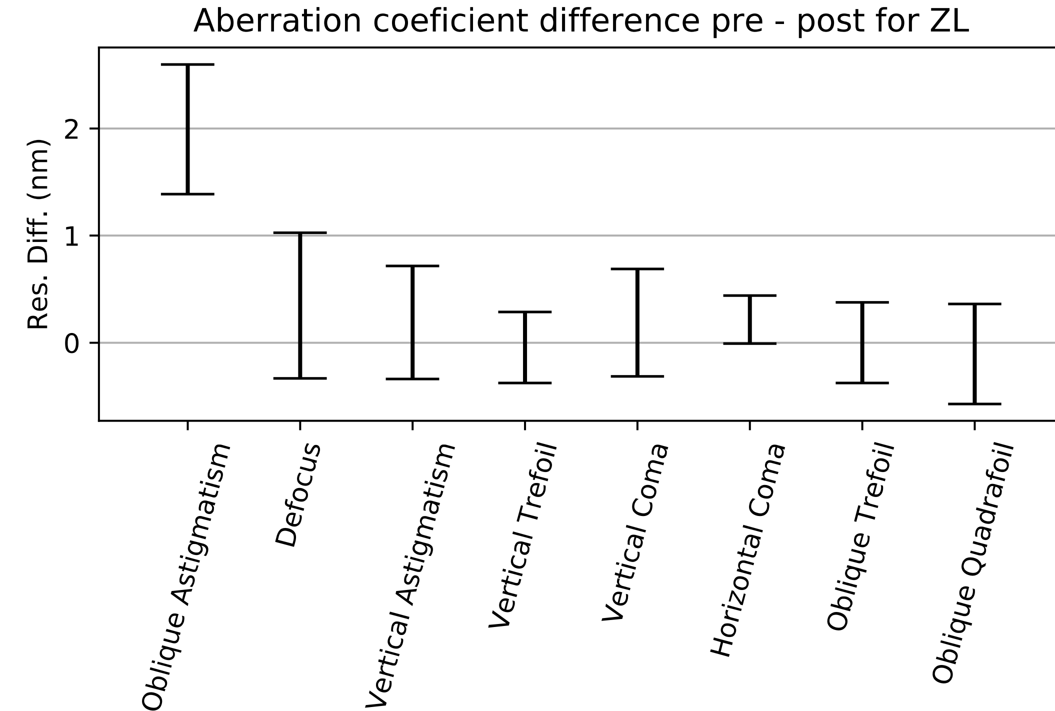


MX Software

ZERODUR Heat Cycling





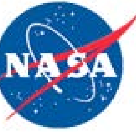


- Initial treatment is ramp down at 10 C/hr.
- Almost no difference observed in comparing CRA.
- Largest aberration change is Oblique Astigmatism (2 nm).

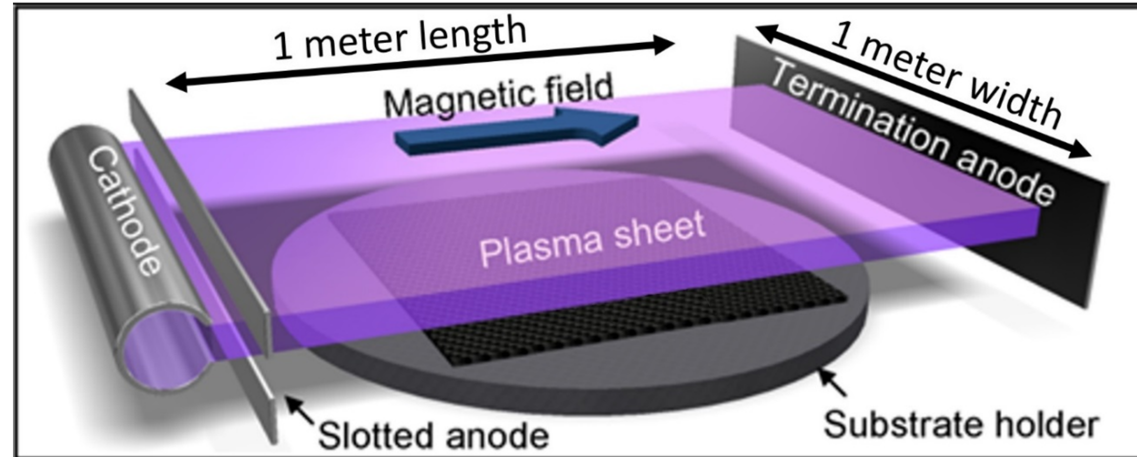
Collaborations:
Gabe Richardson (GSFC Pathways undergraduate, BYU)
Dave Sheikh (ZeCoat Corporation)

1. Manuel A. Quijada, David Sheikh, Javier del Hoyo, and J. Gabriel Richardson, "ZERODUR substrates for application of high-temperature protected aluminum far-ultraviolet coatings," Proc. SPIE 111116, Astronomical Optics: Design, Manufacture, and Test of Space and Ground Systems II, 111160T (12 September 2019); doi: 10.1117/12.2530585

LAPPS Reactor at NRL

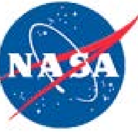


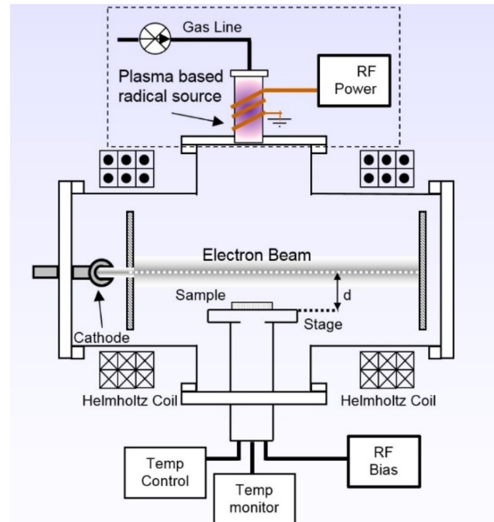
The US Naval Research Laboratory's Large Area Plasma Processing System (LAPPS), which employs an electron beam generated plasma for etching and fluorination of Al samples.

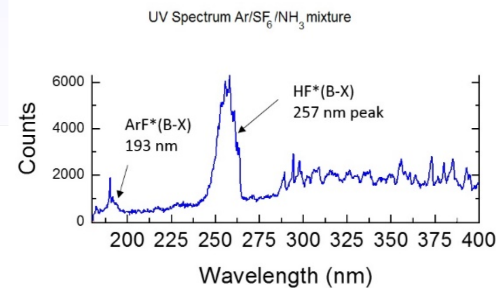


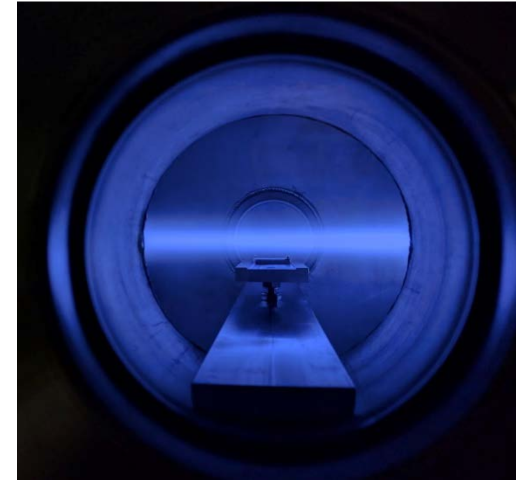
- The injection of a 2 keV beam into the background gas will directly ionize and dissociate the gas.
- Beam energy well above ionization threshold.
- Higher beam energy = more efficient ionization.

LAPPS Plasma Operation





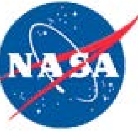


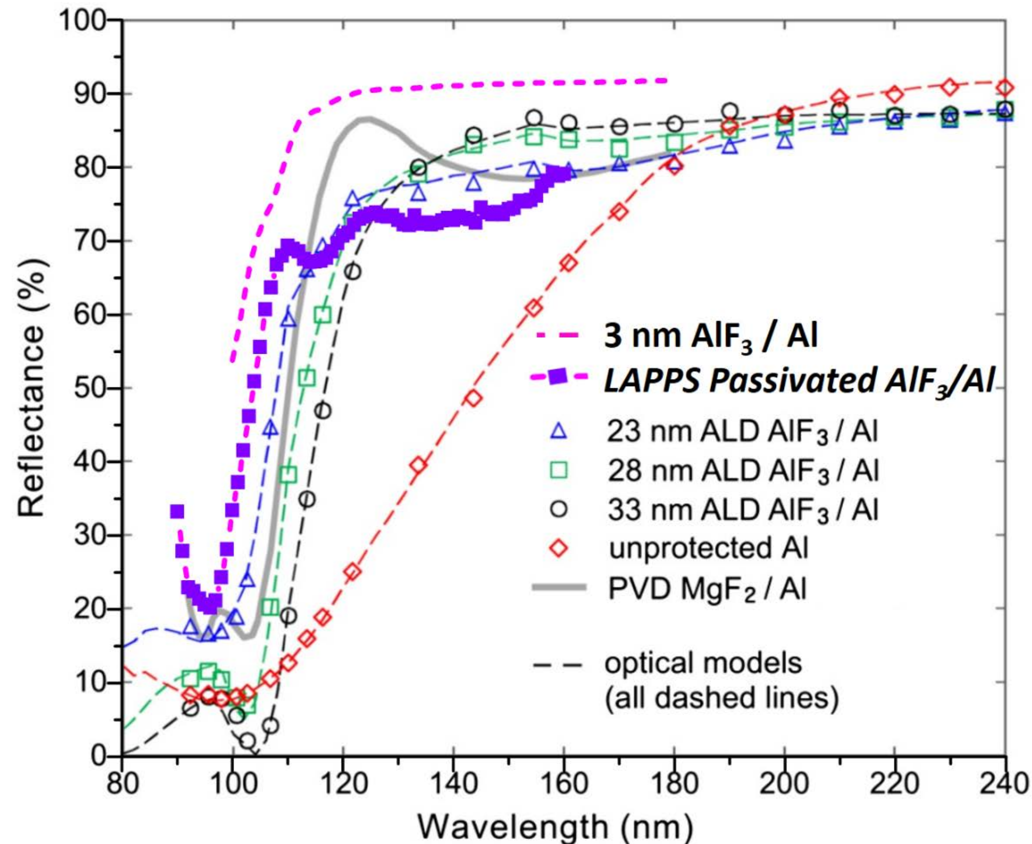


Operating Parameters

- Gas manifold: Ar, SF_6, NH_3
- Flow rates: Ar (150 sccm); Molecular gases (0.5 sccm, SF_6 and 2.5 sccm NH_3)
- Pressure: 75 mTorr
- Process Time 240 s
- Beam Energy 2.5 keV, Cathode Current 20 mA

FUV Reflectance: Latest LAPPS Results





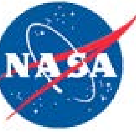
FUV Reflectance of E-beam + Radical Source Treatments of bare Al samples[1]:

- Transition the low-temperature plasma fluorination process to a new reactor at NRL to provide a much cleaner chamber .
- Reflectance for a sample treated in this reactor 70% at 110 nm (O content ~ 6.5%).
- Reflectance is compared to data in Hennessy *et.al.*[2]
- Results show promising potential for passivation of fresh Al coatings mirrors in research chamber

[1] Manuel A. Quijada, David R. Boris, Javier del Hoyo, Edward J. Wollack, Alexander C. Kozen, Scott Walton, Vivek Dwivedi, "E-beam generated plasma etching for developing high-reflectance mirrors for far-ultraviolet astronomical instrument applications," Proc. SPIE 10699, Space Telescopes and Instrumentation 2018: Ultraviolet to Gamma Ray, 106992X (6 July 2018); doi: 10.1117/12.2314391

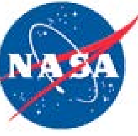
[2] J. Hennessy, K. Balasubramanian, C. S. Moore, A. D. Jewell, S. Nikzad, K. France, and M. Quijada, Journal of Astronomical Telescopes, Instruments, and Systems 2(4), 041206 (Oct–Dec 2016).

Path Forward



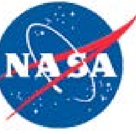
- **One-year no-cost extension in FY20:**
  - Perform XeF_2 passivation in Research Chamber.
  - Extensive re-design ALD reactor at UMD.
  - Conduct ion-assisted deposition in large (e.g. 2-meter chamber) to produce denser/lower absorption metal-fluoride films.

Conclusions



- Completed upgrades to a small (1-meter) research chamber for fluorination (XeF_2) of unoxidized Al mirrors.
- Ongoing progress in the ALD growth of AlF_3 with solid state precursors (TiF_4 and TMA).
- Complete procurement & installation of ion-gun and optical monitor for IAPVD depositions of metal-fluoride overcoats in GSFC 2-meter chamber.
- The LAPPS reactor (developed at NRL) has shown potential for oxide-removal & passivation of Al mirror.
- Initial trial runs of oxidized Al coatings with NLR LAPPS reactor showed improved FUV reflectance with treatment with the LAPPS e-beam in combination with a radical (fluorine) source.
- Chemical analysis confirmed presence of F bonds on the surfaces of Al samples (with reduced concentration of O) that correlated with improved FUV reflectance.

Acknowledgments



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