



## Emerging Coating Technologies for Realizing High-Reflectance and Stable Mirror Coatings for Observations in the Far Ultra-violet

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➢Objectives and Goals

Plasma-based e-Beam Treatment (LAPPS at NRL)

➢ Reactive PVD/XeF₂ Fluorination (GSFC)

Broad-Band Reflectance & TRL Comparison

## ➤Conclusions



## Broad-band (90 nm – 3 $\mu$ m) High Reflectance Mirrors for Next Generation Space – Based Telescopes



### **Task Description**

Deposit high performance optical broadband (FUV -> IR) mirror coatings:

Fluorination/passivation of Al-based coatings.

Atomic Layer Deposition (ALD) layers of AlF<sub>3</sub>.

Ion assisted depositions for low-absorption metalfluoride to protect Al mirrors.

### **Driver / Need**

Broadband coatings (90-2,500 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).

The LUVOIR Final Report (https://asd.gsfc.nasa.gov/luvoir/reports/LUVOIR\_FinalReport\_2019-08-26.pdf)

#### **HWO Concept Telescope**





(HST = Hubble Space Telescope)







# Approach: Using Low T<sub>e</sub> Plasmas to Remove Oxide and Passivate Al surface with Fluorine





**Plasma Treatment** 

**Final Condition** 





# Approach: Using Ultra-Low T<sub>e</sub> Plasmas to Remove Oxide and Passivate Al surface with Fluorine



### LAPPS: Large Area Plasma Processing System

A processing system based on electron beam generated plasma







## **Basic Operation**

- High-energy (~ keV ) beam injected into background to drive plasma production
- Easily scaled to large area processing
- Advantageous plasma properties
  - Easily controlled species production
  - Low electron temperature (T<sub>e</sub>) plasma (<1 eV)  $\rightarrow$  Low ion energy (<5 eV)  $\rightarrow$  Low/No Damage

Radical Source: Inductively Coupled Plasma (ICP)



R.A. Meger et al., US patent no. 5,874,807 (Feb. 1999)

S.G. Walton et al., ECS Journal of Solid State Science and Technology, 4(6) N5033-N5040 (2015)

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## Low ion energy shows ability to grow high quality fluoride films



- Low ion energy processing capability
  → excellent reflectivity
- Demonstrates ability to grow fluoride coating without surface roughening or affecting underlying Al layer.





## Uniformity Tests: ICP vs. Non-ICP (FUV)





- The FUV reflectance displays a peak value close to 90% at Lyman-Alpha
- Excellent uniformity (standard deviation among 5 samples is < 0.20 %) over 115-180 nm
- 5 eV case had best reflectivity characteristics.
- Surface roughness does not seem to be significantly affected by ion energy.



## **Research Coating Chamber Capabilities**





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



XeF<sub>2</sub> Gas feed components capable of continuous flow or pulsed flow.



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

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

Chamber is in operation and experimentations on producing various schemes of fluorination are ongoing



## Reactive Physical Vapor Deposition (rPVD)





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## Reflectance Result rPVD: Al+LiF

Highest R at H Lyman-alpha ever reported 🙂







• R data of mirrors with and without Ti seed layer meeting HabEx and LUVOIR R requirements



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# Broadband Reflectance







# Comparison Various Coating Technologies

	Coating Properties						
Coating Technology	λ Value @ R>60%	TRL	Largest Optics Coated	Elevated Substrate Temperatures Required?	Max. Relative Humidity for Coating Stability	Dielectric Layer Deposition Process	µ-roughness
Bare Al	>150 nm	6	>1 meter	No	~70-100%	-	~0.78 nm
Al+MgF <sub>2</sub>	>111 nm	6	> 1 meter	No	~70%	PVD	~1.84 nm
Al+LiF	>101 nm	6	~0.5 meter	No	< 30%	PVD	Fresh 1.5-2.5 nm Aged >3 nm
Al+eLiF+MgF <sub>2</sub>	>102 nm	~5-6	~ 0.3 meter	Yes	~60 %	eLiF (PVD) MgF <sub>2</sub> (ALD)	1.5-2.5 nm
Al+AlF <sub>3</sub> (e-beam)	>105 nm	~4	5x5 cm <sup>2</sup>	No	~60%	E-beam Plasma	~0.81 nm
Al+XeLiF	>103 nm	~3	5x5 cm <sup>2</sup>	No	~60%	Reactive PVD	~1-1.5 nm





# Conclusions



- The Large Area Plasma Processing System at NRL has demonstrated oxide removal and fluorine passivation of Al mirrors **over 6**" **diameter area** 
  - 15 nm 25 nm thick AlF<sub>3</sub> optical coatings
  - Reflectivity approaching specs for next generation space telescope mirrors
    - o <R>(100-200nm) = 81%
    - <R>(120 3000nm) = 93%
  - Demonstrated control of FUV reflectance properties of Al with a metal fluoride overcoat (MgF<sub>2</sub>, AlF<sub>3</sub>) by varying ion energy, radical density, and plasma exposure time.
  - Verification of reflectance uniformity, environmental stability, microroughness and polarization characterization has been demonstrated to be at TRL 4.
- A second coating technology (XeLiF), which involves passivation with a fluorine containing precursor (XeF<sub>2</sub>) gas is showing promising reflectance in the FUV.
- The surface XeF<sub>2</sub> passivated Al does not show (via AFM) significant changes in low to moderate humidity even after months in nominal lab conditions.



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# Backup Slides





FUV Reflectance for Ebeam + Radical Source Treatments with varying treatment time and varying ion energy

- Correlating XPS results with process conditions seems hint at two possible trends
- First increased ion energy led to slightly higher oxygen content
- Second longer exposure time led to slightly higher oxygen content
- Overall higher oxygen content was correlated with decreased FUV reflectivity

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## Uniformity Performance Large Area Growth in 6" LAPPS Reactor



Uniformity across 6" substrate holder:

- Target thickness 25 nm
- 5 samples treated simultaneously
- Real time monitoring of center sample .
- Post treatment reflectivity analysis of all samples ٠
- Post treatment ellipsometry mapping of fluoride • thickness for all samples.

Sample	Chamber location	Average thickness
AI8A	top	24.6 ± 1.37 nm
AI5	middle	25.5 ± 1.74 nm
Al6	right	23.9 ± 2.54 nm
AI7	left	22.1 ± 0.59 nm
ECA1	bottom	24.9 ± 1.37 nm





Top: Sample configuration for the uniformity test. Bottom: Mean reflectance (red) and R non-uniformity (blue) from 6" sample array.



# UV/Vis/NIR Polarized Reflectance

2.0%



UV/Vis/NIR Average R (AOI =12°)



#### Diattenuation

••••• AOI = 20°

700

Wavelength (nm)

1200

AOI = 12°

• AOI = 8°

1700

2200