Improving LUVOIR FUV Instrument Capabilities through Enhanced Coatings

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Outline

- Overview & Objectives
- Methods to Enhance FUV Reflectance
- Results of E-Beam Reactor at NRL
- Conclusions
Overview and Objectives

- **Summarized Task Description**
  - Deposit high performance UV to FIR optical broadband coatings by designing/constructing hybrid thin film deposition/fluorination chamber capable of depositing aluminum under ultra-high vacuum with the capability of adding a precursor gas to fluorinate the surface and form a thin AlF$_3$ layer to protect the Al from oxidation.
  - Improved deposition processes of metal-fluoride protection coatings (MgF$_2$, AlF$_3$, LiF) on Al in order to boost reflectance performance.

- **Driver / Need**
  - High-performance broadband coatings (90-10,000 nm) have been identified as an “Essential Goal” in the technology needs for the Large UV/Optical/IR (LUVOIR) Surveyor observatory.
  - Low reflectivity and transmission of coatings in the Lyman Ultraviolet (LUV) range of 90-130 nm is one of the biggest constraints on FUV telescope and spectrograph design.

- **Benefits**
  - The development of broad-band reflectors based on Al with increased performance in the FUV spectral range will be an enabling technology for an instrumentation platform for astrophysics and optical exoplanet sciences with a shared telescope providing high throughput and signal-to-noise ratio (SNR) over a broad spectral range.
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.

AIF$_3$ as Aluminum Mirror Overcoat

- A+AlF$_3$ PVD 3-step process: Al (70nm) AlF$_3$ (24nm)
- Minimal changes in reflectance (after 6 months) with sample kept in ambient lab conditions (50% RH)
- Calculated data agree well with measured results
- Predicted performance shows a 50-60% reflectance at 100nm
XeF$_2$ Fluorination of Fresh Al Task

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 of chamber PVD components.
Flash Coating of Aluminum Films

Aluminum coating thickness: 50-70nm @ 130-160 A/Sec
A Second bare Al sample (with native oxide layer) was treated in a XeF$_2$ reactor located in the Detector Branch (Code 553) at GSFC.

- 50 cycles (10 seconds per cycle) with a XeF$_2$:Nitrogen mixture with a 1:5 ratio.
- Sample remained optically shiny with a slight improvement in FUV reflectance.

XPS Results: 7.9% F-Al bonds after XeF$_2$ treatment

- An initial as-received XPS scan was performed.
- A very etch is performed to remove light contamination and carbon: 10 sec of a 3 kV Argon Sputter raster beam
- A post etch XPS scan is performed
Atomic Layer Deposition Reactor Systems

General-purpose ALD reactor at UMD features:

- Reduced reactor volume (relative to previous reactor)
- 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 in substrates
- RGA
Alumina ALD Growth

- Precursors exposure: 0.1s
- Post-exposure residence: 1s
- Purge: 20s TMA / 25s Water
- Precursors manifold T: 110°C
- Cycles: 200

\[ \text{gpc} \approx 1.3 \text{ A/cycle (ideal is 1.1/cycle)} \]
Procurement & installation of an in-situ optical monitor (λ = 121.6 nm); source, detector, port window, etc.

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

Pumping system for this chamber is being refurbished:

- Acquisition of new cryo-pump and compressor.
- Procurement of various types of glass substrates (ULE and Zerodur) to evaluate effect of heating on figure error.
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 schematic diagram illustrates the processing reactor, whereas the image on the upper right corner is a view of the plasma through a 6-inch port.
Motivation for e-Beam Etching

- Electron beam generated plasmas have demonstrated the ability to chemically modify 2-D materials while maintaining their unique characteristics.
- Electron beam generated plasmas have shown promise as a low damage etch source. Particularly in processing devices with integrated 2-D materials.
- They have also demonstrated selective, highly directional, low damage etching in SiN without pattern dependent etch characteristics in fluorine-based chemistries.
- The e-beam provides a low-energy plasma system to etch the surface of a sample with low damage probability.
How are e-beam generated?

- 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
Bare Aluminum e-Beam Results: Trial 1

Reflectance results of bare Al sample with native oxide layer before and after treatment in the LAPPs reactor at NRL.

Bare Al films before and after plasma etching at NRL

XPS Results: 6.6% F-Al bonds after e-beam treatment

Reflectance results of bare Al sample with native oxide layer before and after treatment in the LAPPs reactor at NRL.
A bare Al coating made in 2009 was treated at the NRL LAPPSS reactor (sample was measured before and after).
Results indicate a gain in reflectivity of around 10% over most of FUV spectral range.
Reflectance performance was that of a sample with aging of just a few months (after plasma treatment at NRL).

Bare Aluminum e-Beam Results: Trial 2

XPS Results: 27.3% of F-Al bonds after e-beam treatment
Al+MgF$_2$ e-Beam Results: Trial 2

- Al+MgF$_2$ sample made in 2011 recently showed average reflectance of 60-70% in FUV.
- Sample was treated at the NRL LAPPS reactor and re-measured again.
- Results indicate a gain in FUV reflectivity of around 20% over most of the FUV spectral range.
- Samples has remained stable after a second round of measurement (after plasma treatment at NRL).

![Graph showing Al+MgF$_2$ Coatings before and after plasma etch/passivation @ NRL](chart.png)
Conclusions

- Predicted performance of an Al sample fluorinated with an AlF$_3$ overcoat would produce a sample with reflectance close to 50-60% at 100 nm and over 90% at wavelengths longer than 110 nm.

- An aluminum sample coated with an AlF$_3$ overcoat shows a stable reflectance after being kept in a normal laboratory environment (50-60% relative humidity) for a period of 6 months.

- We studied the feasibility of using the LAPPD reactor (developed at NRL) that employs a low energy- e-beam to etch away the native oxide layer from Al samples as well as thinning the AlF$_3$ and LiF layers for Al protected with these dielectrics.

- A second trial run of using a modified chemical etching at NRL provided an increase in FUV reflectance for a sample with a native oxide layer and a second Al+MgF$_2$ that had degraded after since 2011.

- Chemical analysis confirmed presence of F atoms on the surfaces of both Al samples treated at LAPPD (NRL) and XeF$_2$ reactor (GSFC).

- More studies with NRL e-beam reactor are planned in the future.
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