# Improving LUVOIR FUV Instrument Capabilities through Enhanced Coatings


By

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# Outline

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

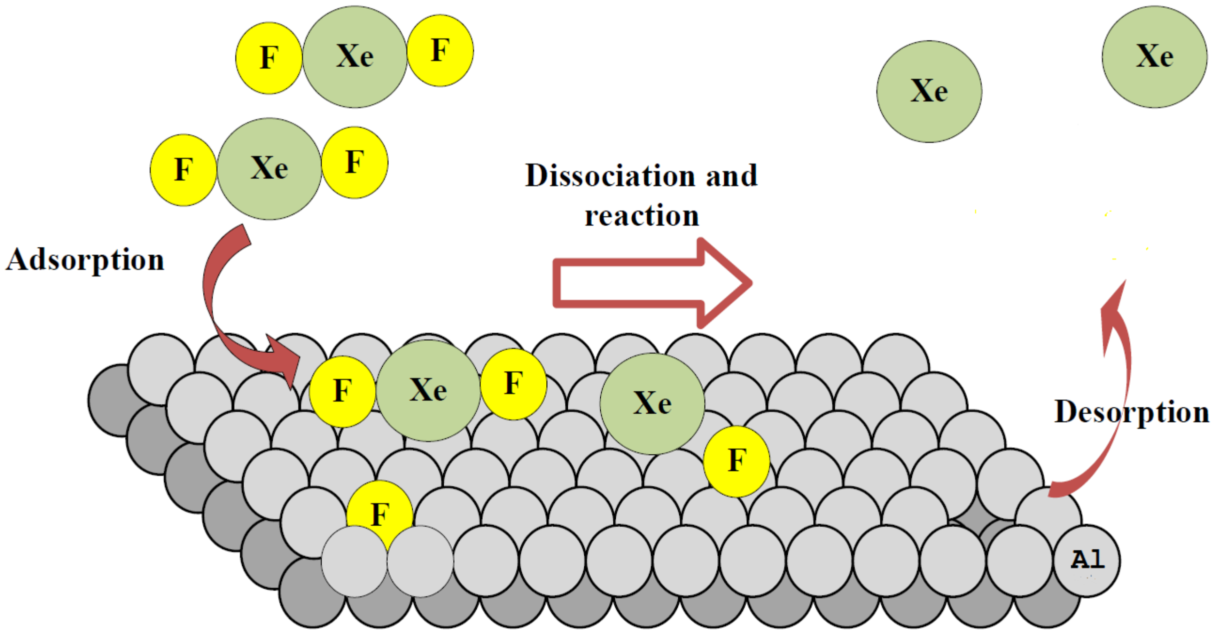
# Overview and Objectives

- **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.

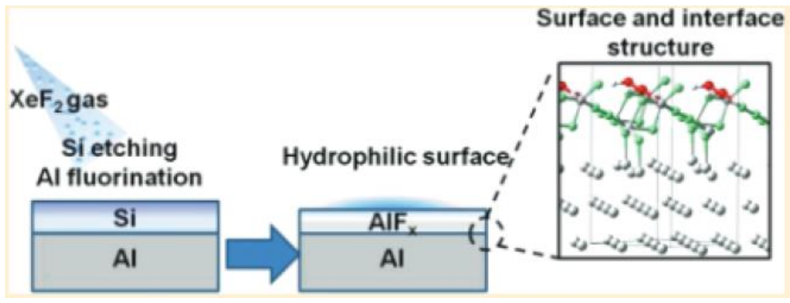
# Hybrid PVD Passivation/Fluorination Chamber




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

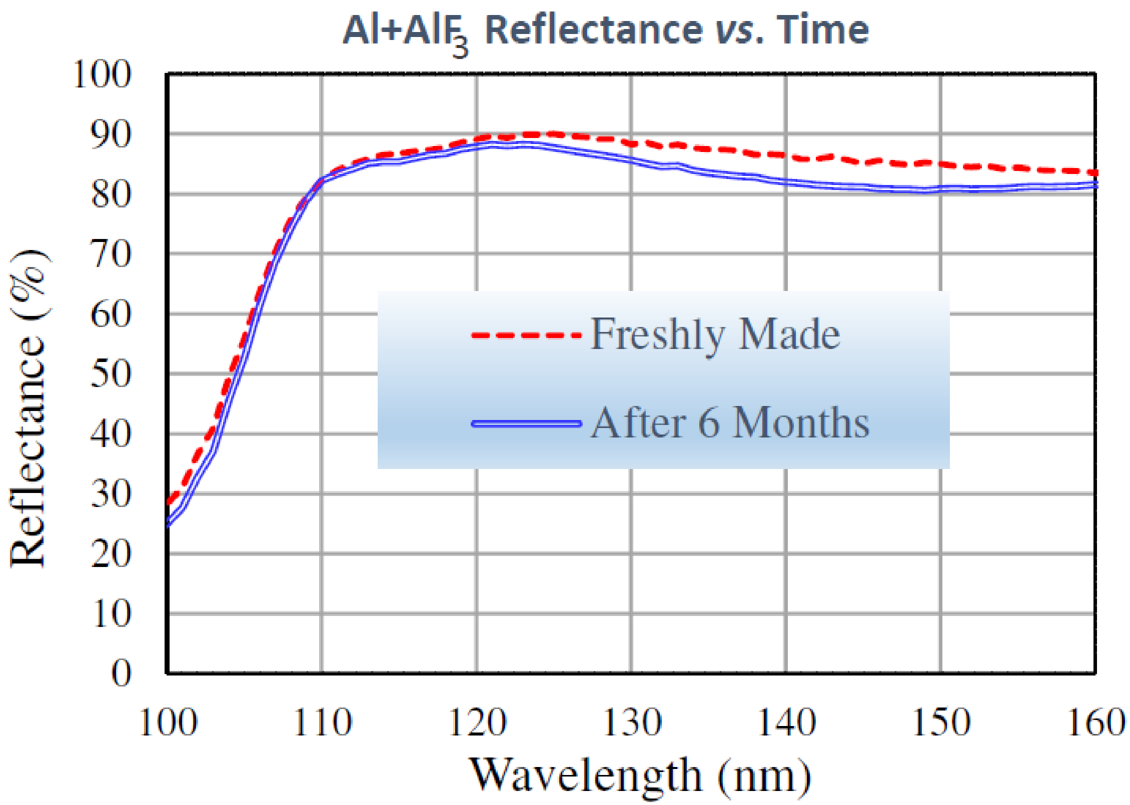
Heating of the $XeF_2$ may also be used if compound is not sufficiently reactive for increased selectivity.

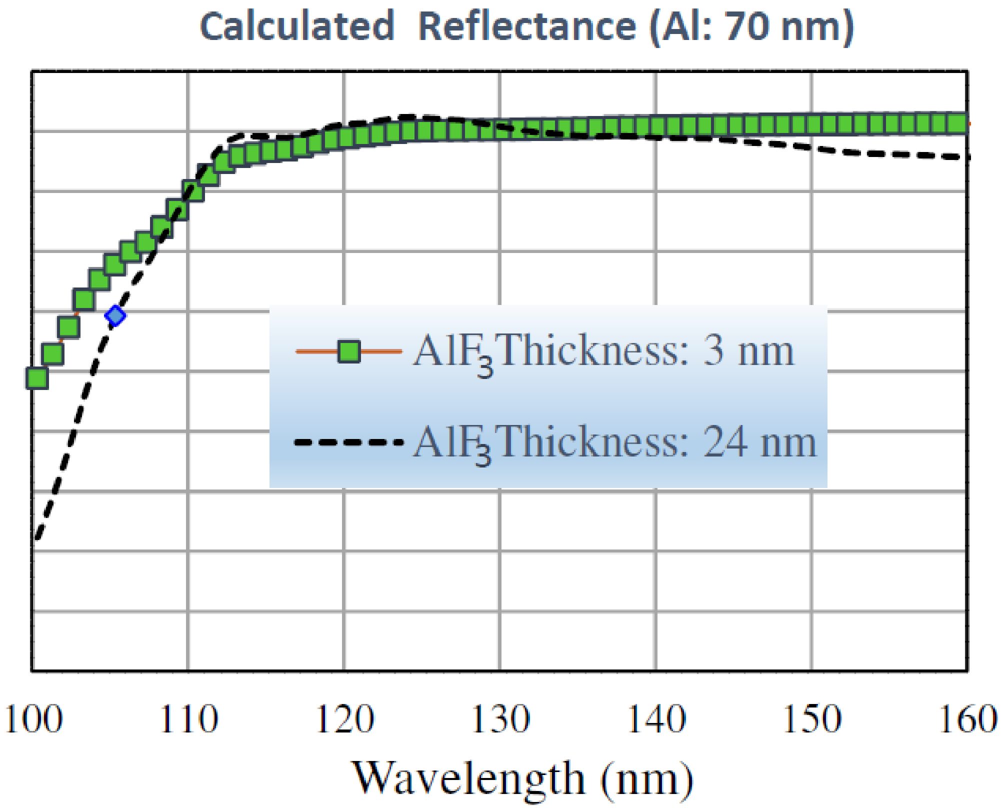
$XeF_2$ is a dry-vacuum based method of reaction and requires no plasma or other activation minimizing damage to substrate.




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

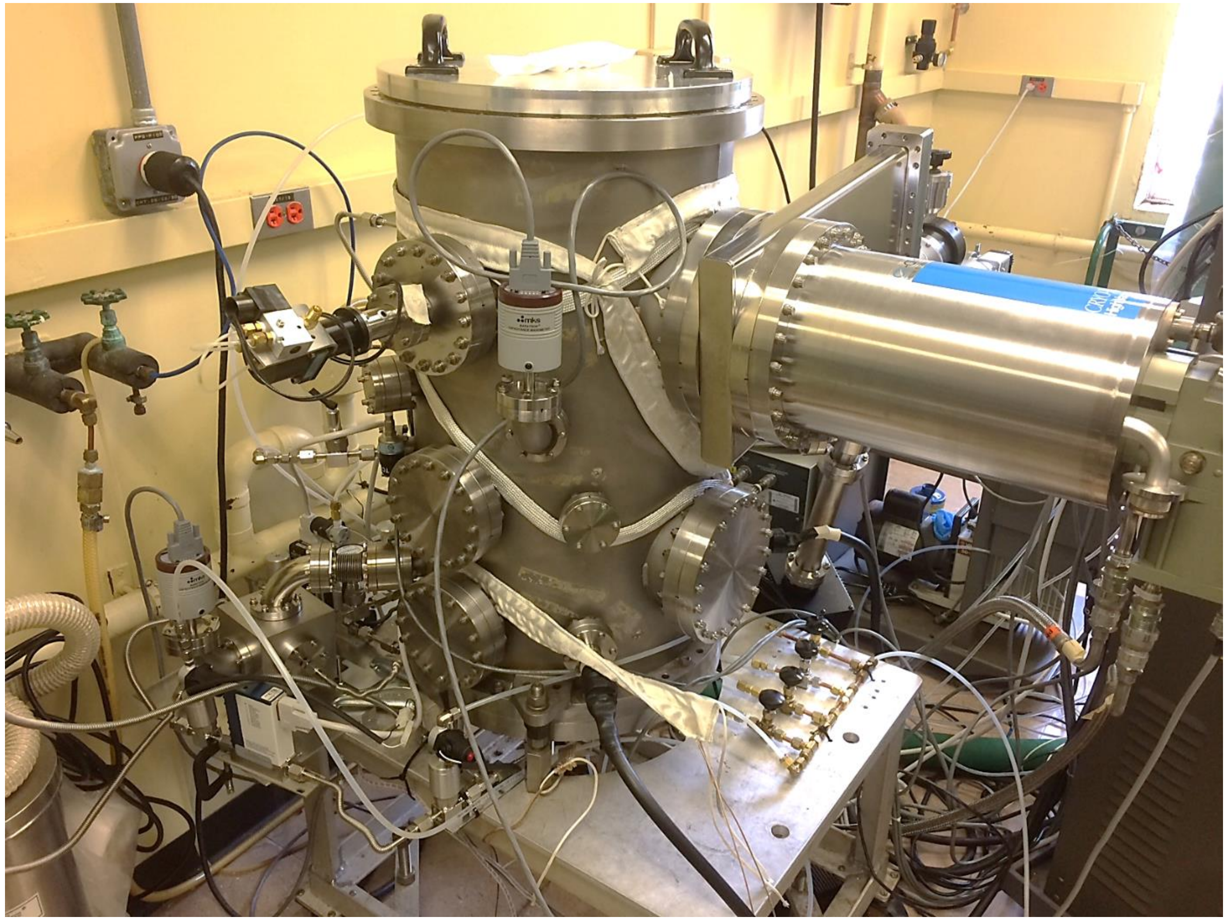
# AlF$_3$ as Aluminum Mirror Overcoat



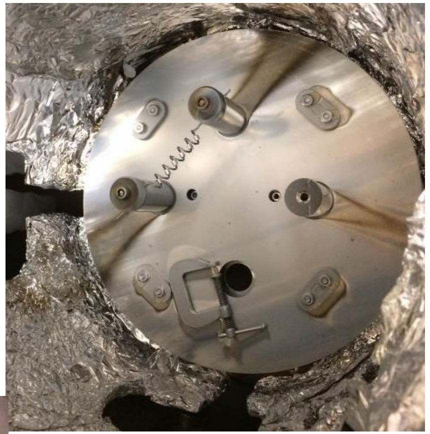

- A+AlF$_3$ PVD **3-step process**: Al (70nm) AlF$_3$ (24nm)
- Minimal changes in reflectance (after 6 months) with sample kept in ambient lab conditions (50% RH)
- Calculated data agree well with measured results
- Predicted performance shows a 50-60% reflectance at 100nm

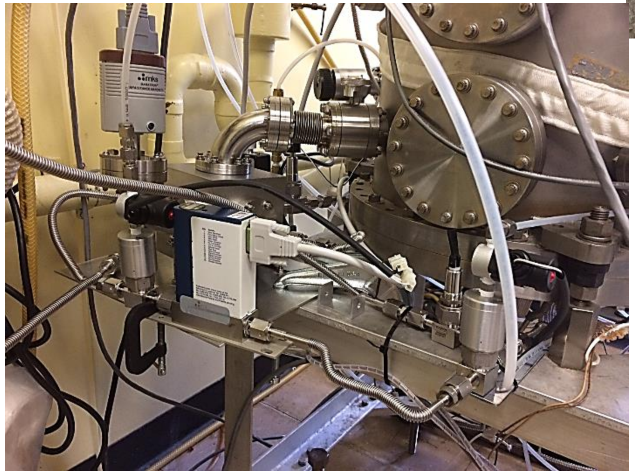
# $XeF_2$ Fluorination of Fresh Al Task



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

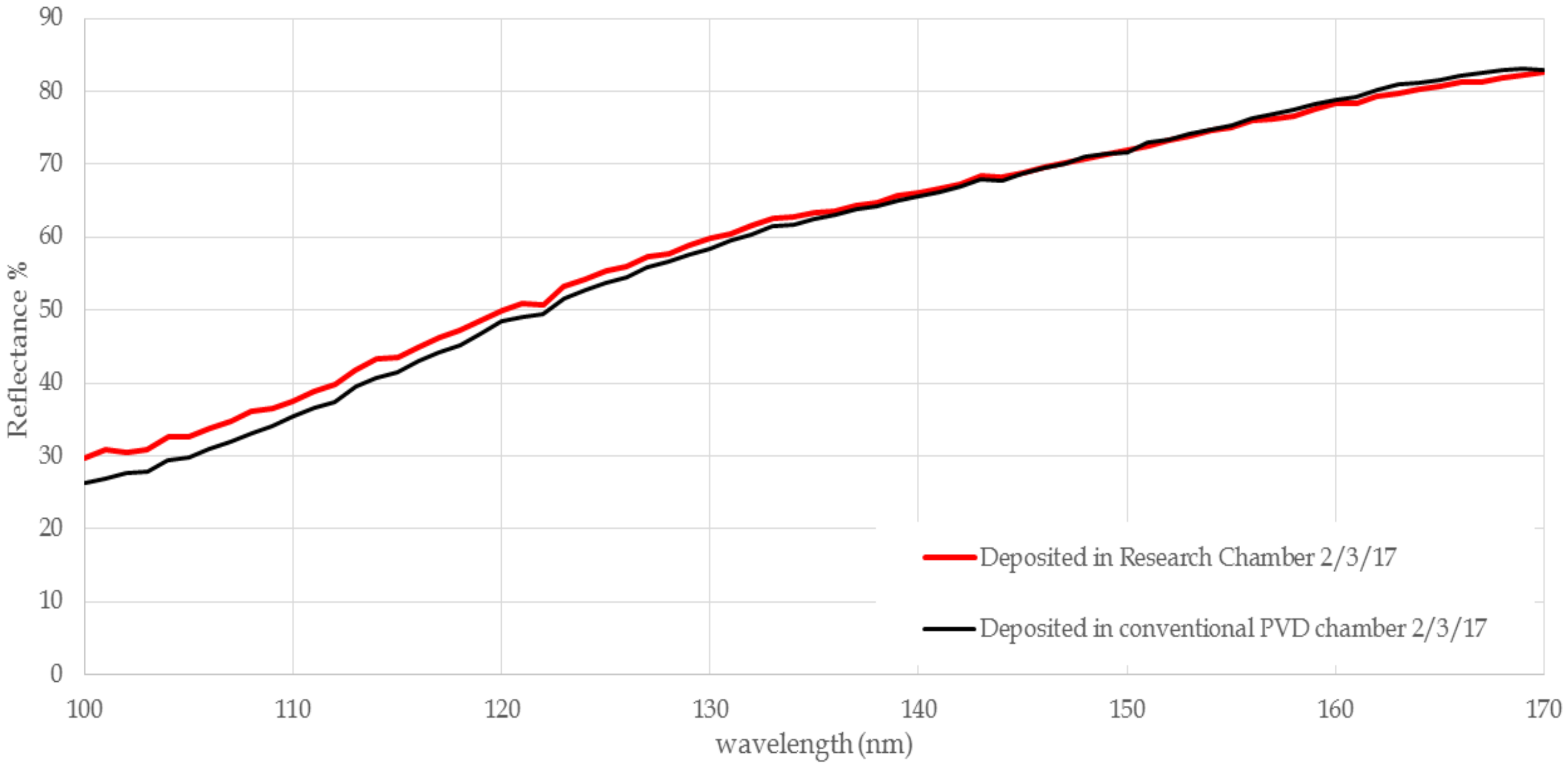
Inside of chamber PVD components.





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

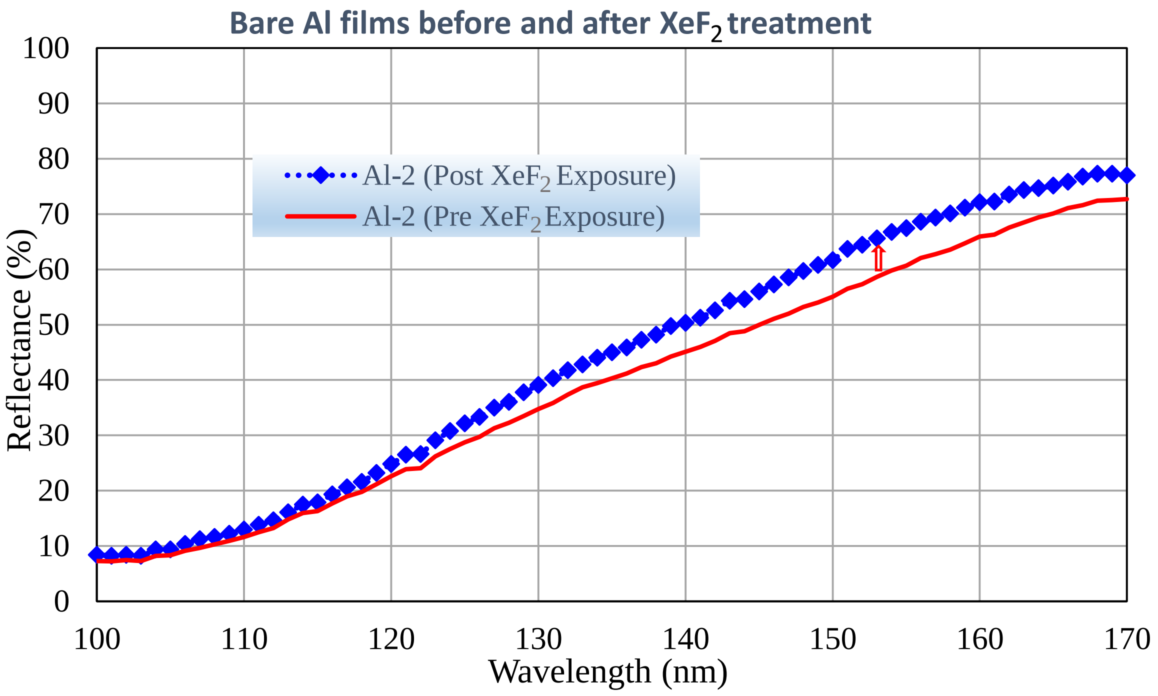
# Flash Coating of Aluminum Films



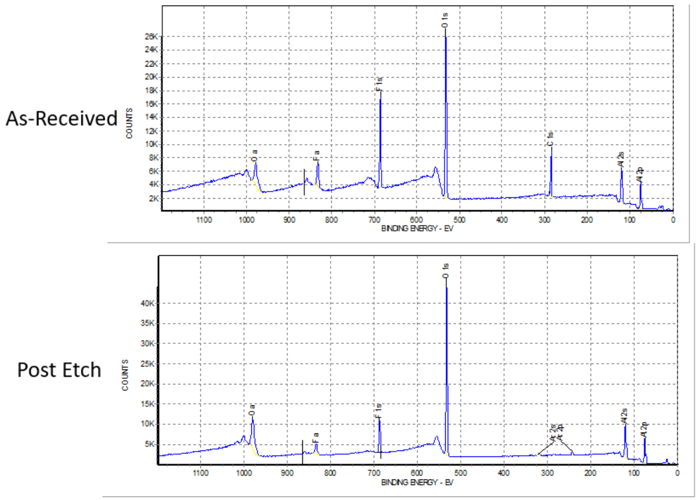

Aluminum coating thickness: 50-70nm @ 130-160 A/Sec

# Al Before & After $XeF_2$ Treatment

- 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.



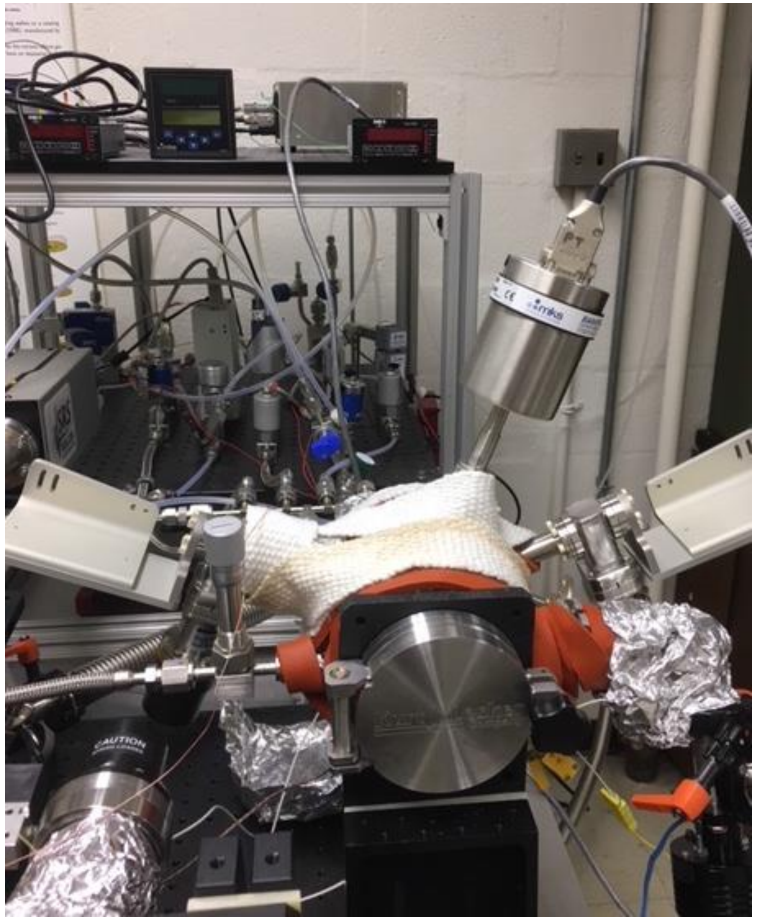

## XPS Results: 7.9% F-Al bonds after $XeF_2$ treatment




| XPS Line | As-received | Post Etch |
|---|---|---|
| Al 2s | 29.0 | 42.9 |
| O 1s | 35.5 | 49.2 |
| C 1s | 20.9 | ~ |
| F 1s | 14.5 | 7.9 |

- 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

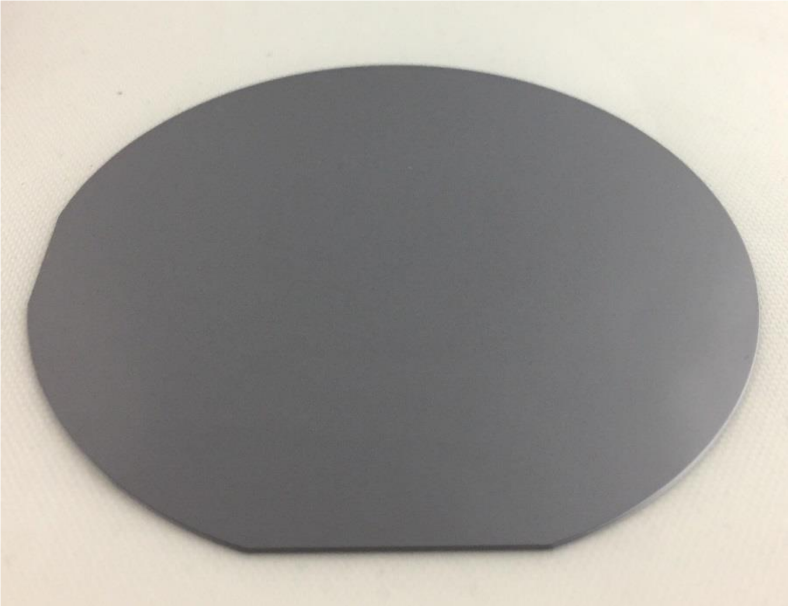
# Atomic Layer Deposition Reactor Systems

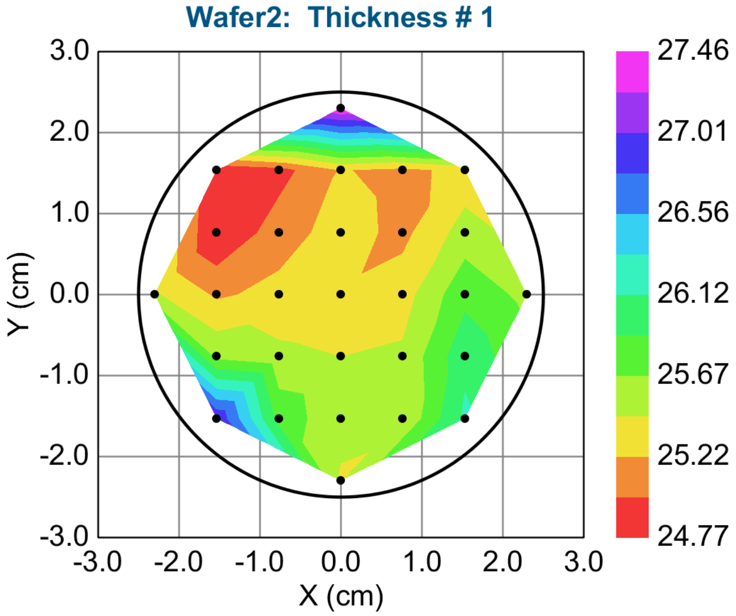


**General-purpose ALD reactor at UMD features:**

- 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

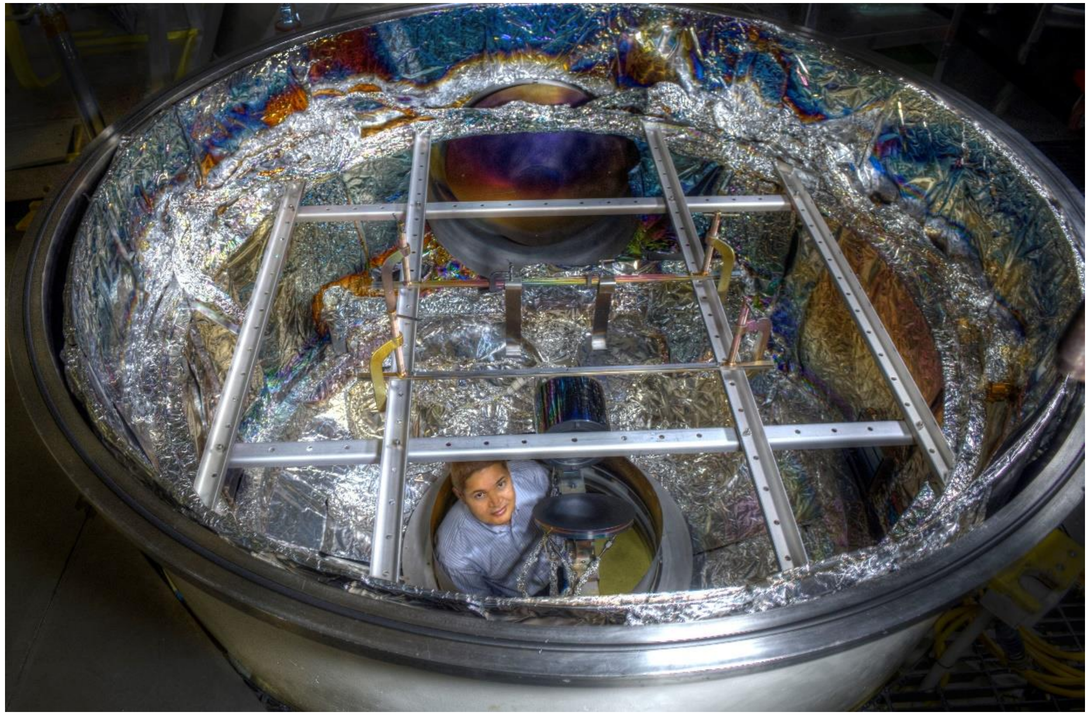





- Precursors exposure: 0.1s
- Post-exposure residence: 1s
- Purge: 20s TMA / 25s Water
- Precursors manifold T: $110^{oc}$
- Cycles: 200

gpc ~ 1.3 A/cycle (ideal is 1.1/cycle)

# Physical Vapor Deposition Ion-Assisted Process

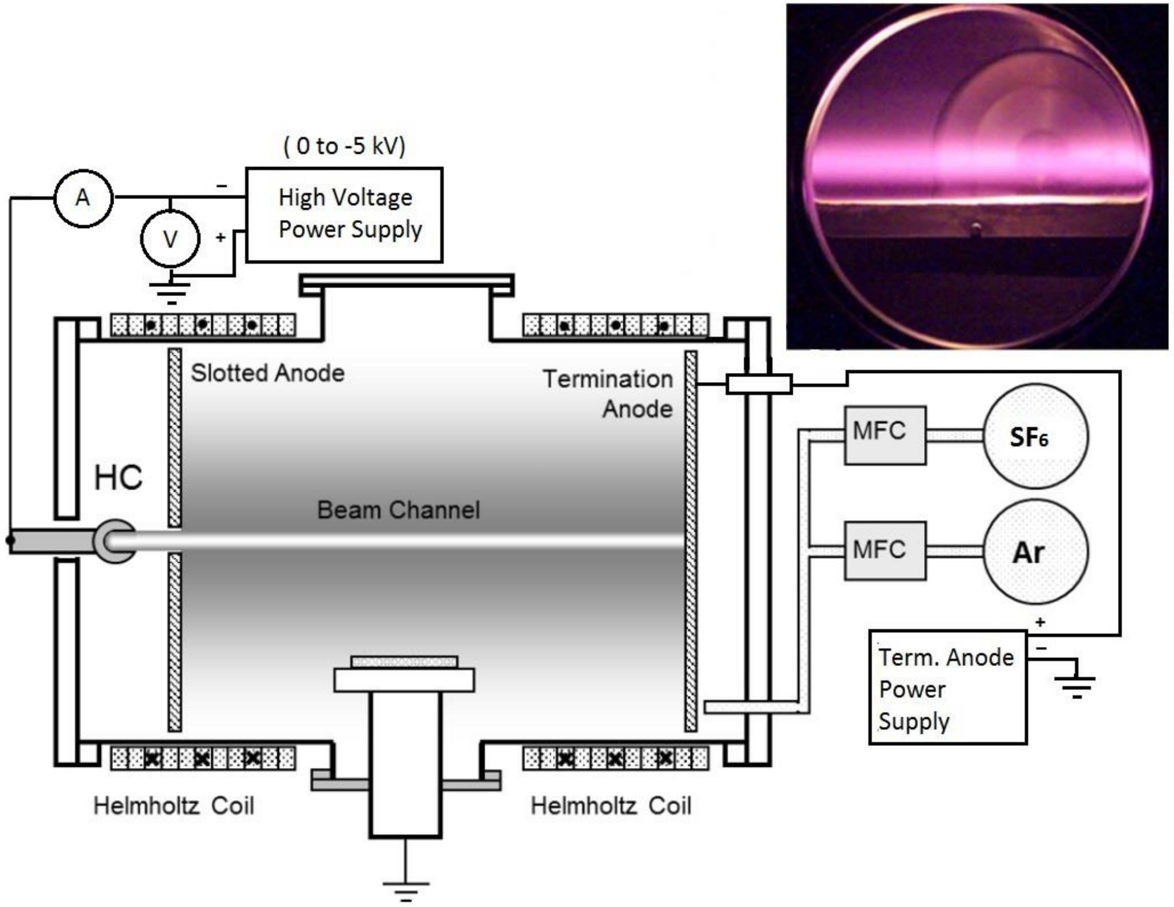


PVD/ IBS UHV coating chamber
(2-meter diameter )

- Procurement & installation of an in-situ optical monitor ($\lambda$ = 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.

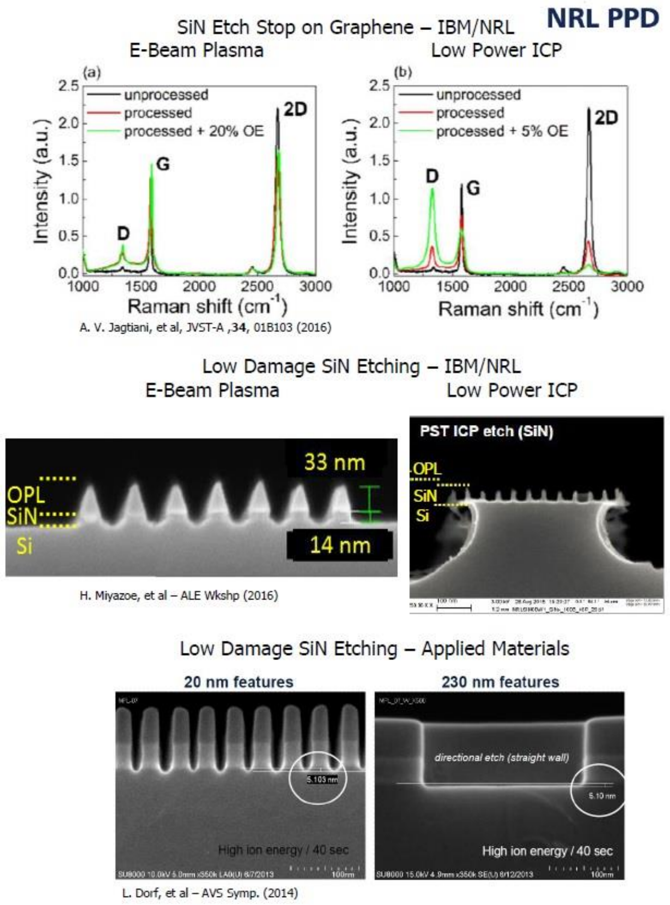
# LAPPS Reactor at NRL

- 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 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.



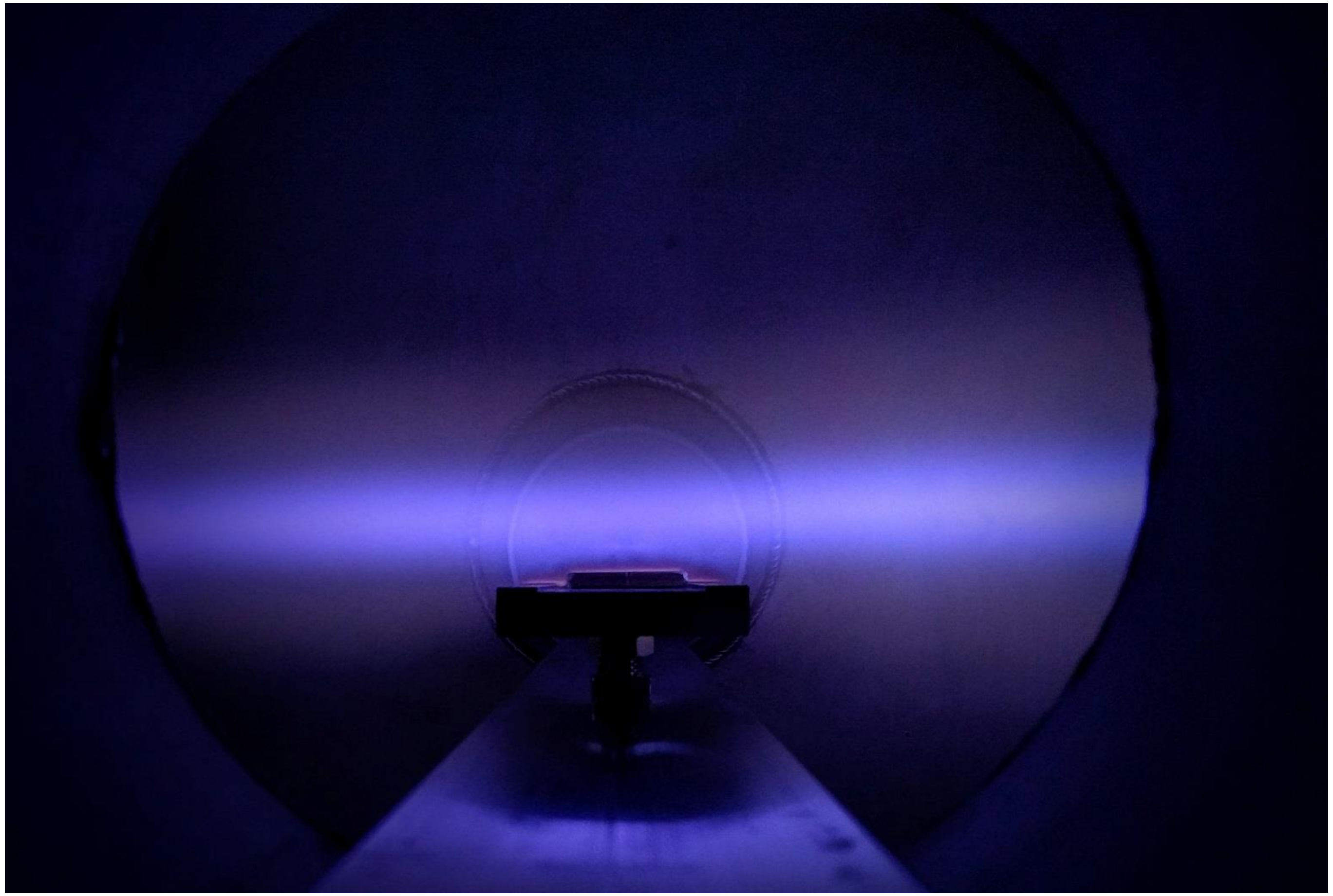
# Motivation for e-Beam Etching

- 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.



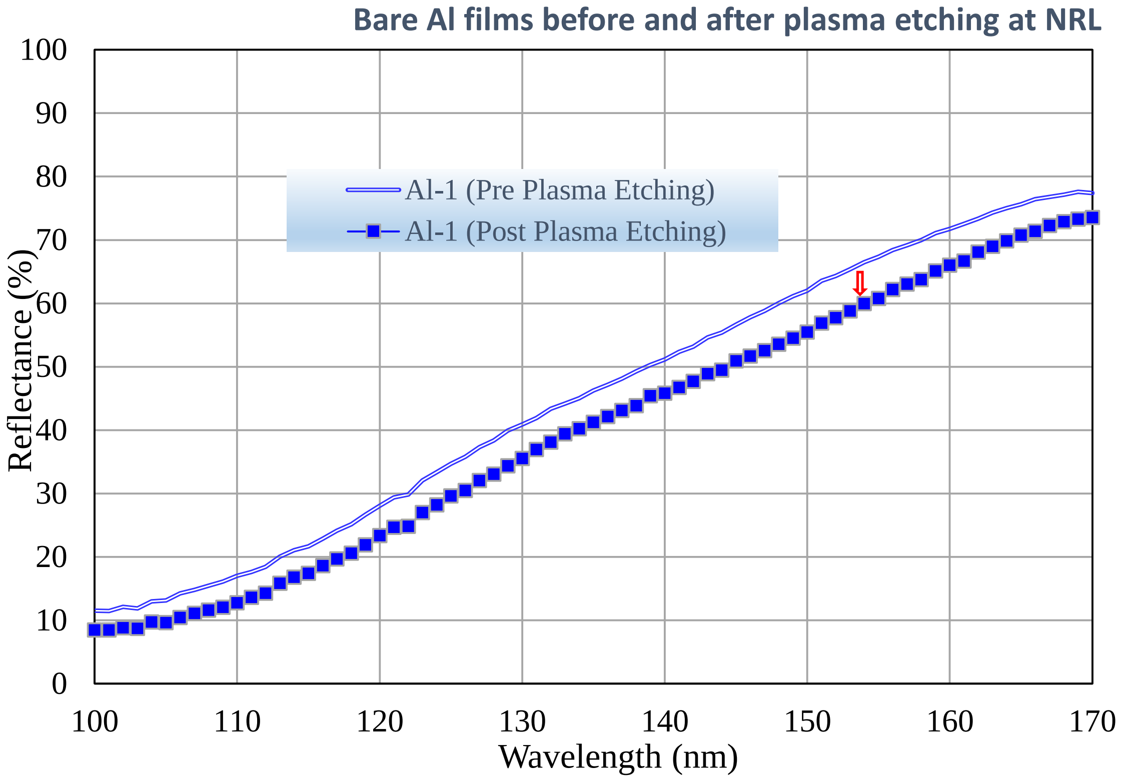
# How are e-beam generated?

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

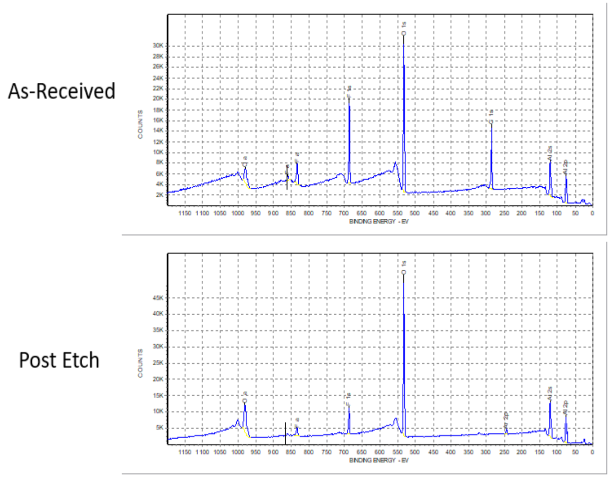


# Bare Aluminum e-Beam Results: Trial 1

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




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

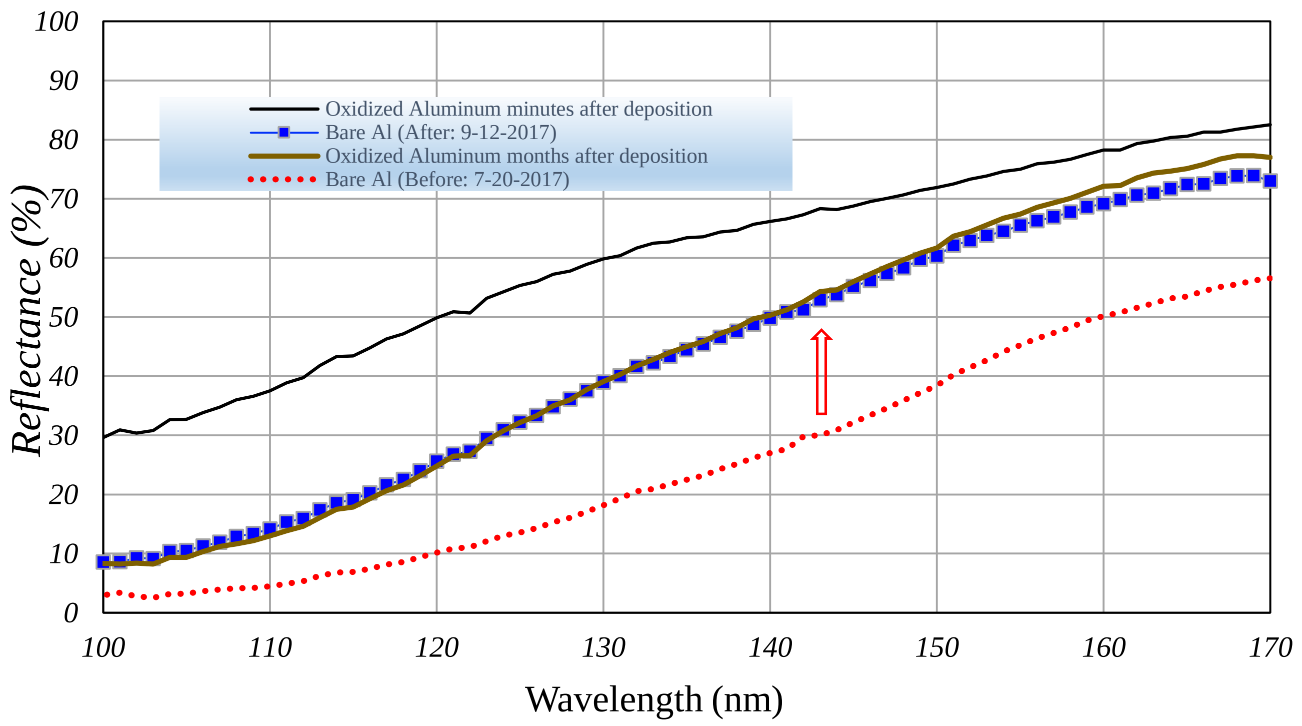



| XPS Line | As-received | Post Etch |
|---|---|---|
| Al 2s | 29.3 | 48.0 |
| O 1s | 31.3 | 45.4 |
| C 1s | 27.5 | ~ |
| F 1s | 11.9 | 6.6 |

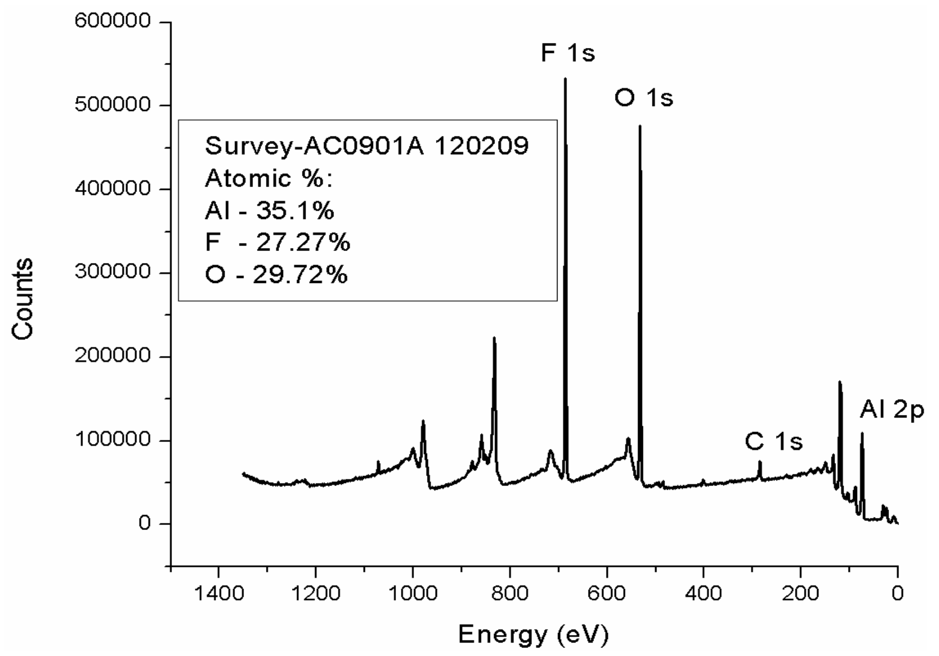
# Bare Aluminum e-Beam Results: Trial 2

- A bare Al coating made in 2009 was treated at the NRL LAPPS 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 coatings before and after plasma etch/passivation @ NRL**

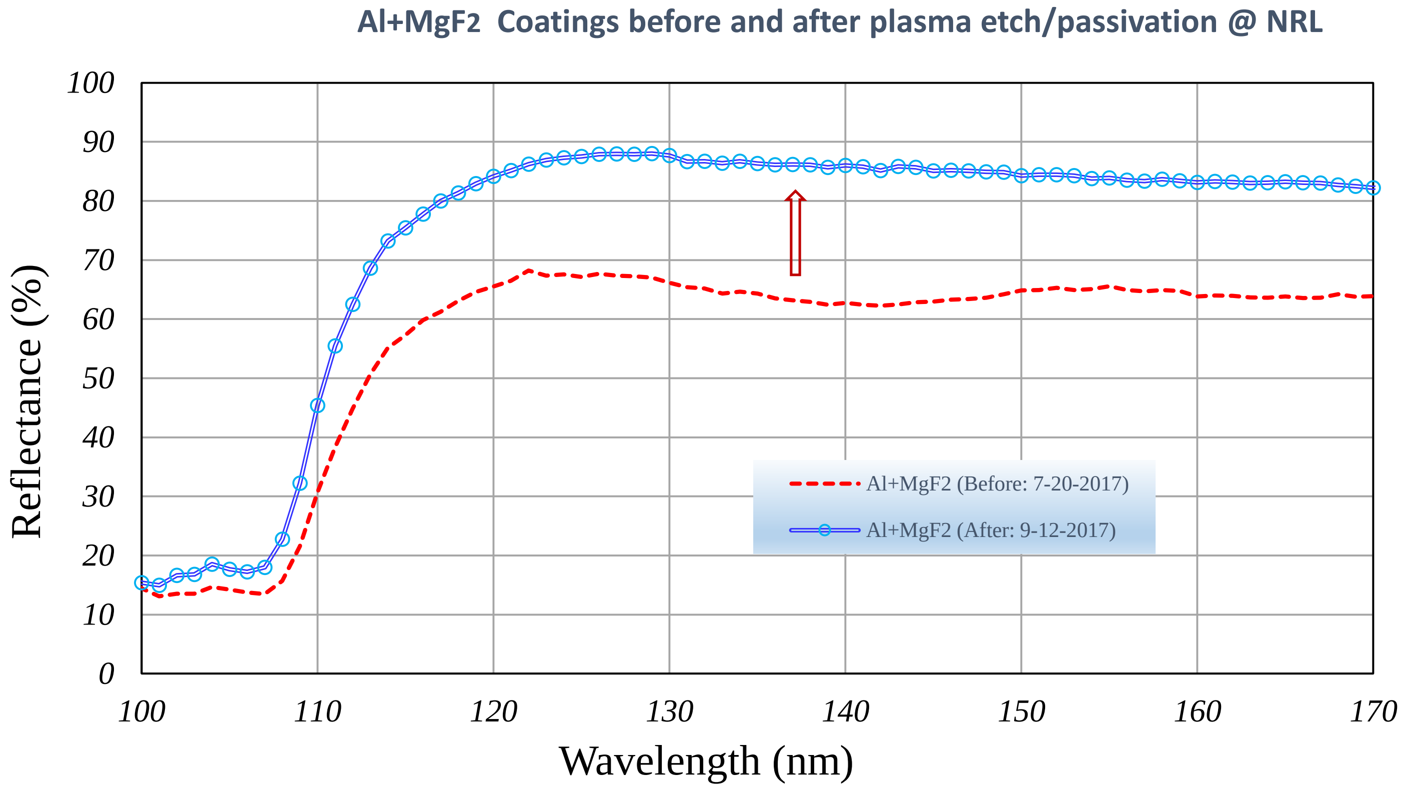



XPS Results: **27.3%** of F-Al bonds after e-beam treatment



# 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).



# 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 LAPPS 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 LAPPS (NRL) and $XeF_2$ reactor (GSFC).
- More studies with NRL e-beam reactor are planned in the future.

# Acknowledgments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