

# Enhanced Fluoride over-coated Al Mirrors for FUV Astronomy

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## ABSTRACT

Technological alternatives in the Far Ultraviolet (FUV) spectral region are some of the more challenging due to the very distant and faint objects that are typically searched for in cosmic origin studies such as

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characterization of little studied rare-earth fluorides such as GdF<sub>3</sub> and LuF<sub>3</sub> that exhibit low-absorption over a wide wavelength range and could therefore be used as high refractive index alternatives for dielectric coatings at FUV wavelengths.

### Description and Objectives:

- To develop on a large scale (up to 1 meter diameter) coating of mirrors using a Al+MgF<sub>2</sub> coating process to enhance performance in the Far-Ultraviolet (FUV) spectral range
- Study other dielectric fluoride coatings and other deposition technologies such as Ion Beam Sputtering (IBS) that is known to produce the nearest to ideal morphology optical thin film coatings and thus low scatter
- Optimize deposition process of lanthanide trifluorides as high-index materials that when paired with either MgF<sub>2</sub> or LiF will enhance reflectance of Al mirrors at Lyman-alpha

### Approach for Objective 1:

Retrofit a 2 meter coating chamber with heaters/thermal shroud to perform coating iterations at a high deposition temperatures (200-300°C) to further improve performance of protected Al mirrors with either MgF<sub>2</sub> or LiF overcoats

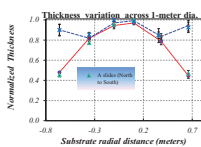
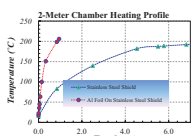
#### Tasks Description:

- Design and fabrication of internal heat shields for GSCF 2-meter Chamber.
- These wall panels were made out of stainless steel and were designed to easily interface with the existing internal configuration of the chamber.
- Optimized coating parameter for high FUV reflectance of a distribution of sides in center and out to a ~0.5 meter radius.

**Physical Vapor Deposition (PVD):**  
 Material is heated until it reaches vapor form. Material is deposited on the substrate where it condenses. Typical deposition rates are 10-100 Å/Sec.



- The images above show the fully assembled internal heat shields, power supply and quartz halogen lamps
- Heater were first tested on 08/13/13 and found maximum temperature reached was only 100 °C after 5 hours
- Doubled lamp power output from 500 W to 1000 W each (4000 W total)
- Additional testing yielded a maximum temperature of 130 °C
- Further testing done after wrapping heat shield panels with aluminum foil provide for a much quicker raise in temperature, reaching 220 °C in less than 1 hour (see graph below on the right)
- Performed a coating run with small 2x2in substrates located at various radius inside chamber (see graph below on the left)

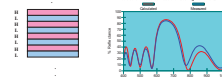


### Approach for Objective 3:

Optimize deposition process of lanthanide trifluorides as high-index materials that when paired with either MgF<sub>2</sub> or LiF will enhance reflectance of Al mirrors at Lyman-alpha

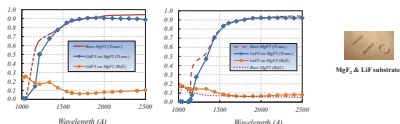
#### Tasks Description for FUV Dielectric Coatings:

- Choose a high-index (H) and low-index (L) pair combination
- Form a pair of (H,L) layers with thicknesses equal to a Quarter-Wave Optical thickness at the design wavelength.
- Repeat the stack above until desired reflectance is achieved.

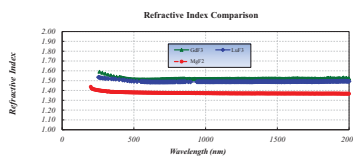


Options for FUV coating candidates: Low Index: MgF<sub>2</sub> (n=1.38)  
 High Index: LuF<sub>3</sub>, GdF<sub>3</sub>

The two graphs below show transmission and reflectance of GdF<sub>3</sub> (right) and LuF<sub>3</sub> (left) films grown on MgF<sub>2</sub> substrates. These data are used to extract refractive index for both of these materials.



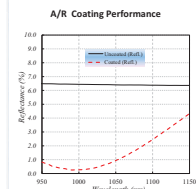
The data on the graphs shown above were used to obtain refractive index (n and k) for MgF<sub>2</sub>, GdF<sub>3</sub>, and LuF<sub>3</sub> films. The results are shown in graph



### Example of A/R coating design and fabrication:

A/R to suppress FS reflection losses near 1000 nm  
 Design includes 2 layer pairs of GdF<sub>3</sub>(H)/MgF<sub>2</sub>(L) (181 and 200 nm respectively) on both sides

Performance is 0.25% near 1000 nm

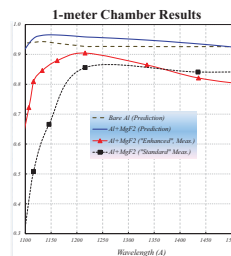


### Al+MgF<sub>2</sub> Coating Performance:

#### 3-step coating process:

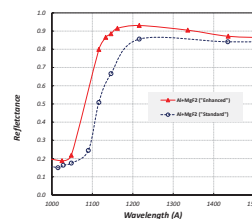
- Al is coated on the substrate at room temperature to the planned layer thickness
- As soon as possible after the Al deposition, overcoat the Al layer and substrate at room temperature with a thin 4-5 nm layer of MgF<sub>2</sub> in order to protect the Al from oxidation and contamination.
- Heat the substrate to 200-300 °C and finish the planned final MgF<sub>2</sub> thickness MgF<sub>2</sub>.

#### Results:



- Predicted vs. measured reflectance of bare Al and Al+MgF<sub>2</sub> reflectance (Al: 50.0 nm; MgF<sub>2</sub>: 25.0nm)
- Enhanced performance is obtained by heating (~220 °C) substrate during MgF<sub>2</sub> deposition
- Reflectance is > 80% even at 115.0 nm

#### 2-meter Chamber Results



- Recently started test run in center of 2-meter chambers to optimize the 3-step process for depositing Al+MgF<sub>2</sub> coatings
- Graph on left display reflectance data taken of test coupons done in this chamber.
- "Standard" test sample was prepared under normal conditions on 08/22/2013
- "Enhanced" was produced with a "hot" (220°C MgF<sub>2</sub> deposition done on 11/27/2103.
- These data represent an important milestone

### Micro-roughness Al+MgF<sub>2</sub> Films



AM2180C: <20 mag/μm²	PV (Å)	Sq (Å)
top left	75.38	6.146
top right	101.2	5.936
center	124	4.021
bottom left	206.11	3.027
bottom right	100	3.262
average	120.97	4.3344

AMCT15A: <20 mag/μm²	PV (Å)	Sq (Å)
top left	45.51	2.249
top right	40.19	2.311
center	59.96	1.364
bottom left	44.39	2.923
bottom right	39.83	2.654
average	46.34	2.022

The tables above show micro-roughness results on two classes of Al+MgF<sub>2</sub> coatings done with the MgF<sub>2</sub> layer deposited at ambient (left) and at elevated (right) temperatures. The table on the right shows the average roughness for the elevated MgF<sub>2</sub> depositions is 30% smaller.

### Conclusions

- Reported gains in FUV reflectivity of Al+MgF<sub>2</sub> and Al+LiF mirrors by employing a 3-step process during PVD coating deposition of these materials.
- Successfully demonstrated gains in FUV reflectance using a large 2-meter chamber that will allow coating up to 1 meter diameter optics.
- Characterization of lanthanide tri-fluoride material candidates to determine their FUV transparency for development of dielectric coatings.
- Will plan to refurbish a second 1-meter chamber to perform IBS film deposition of MgF<sub>2</sub>/LiF materials.

### Approach for Objective 2:

Upgrade existing Ion Beam Sputtering (IBS) chamber with a two-gas flow controller system. Krypton gas is used during IBS deposition. In addition, Freon (CF<sub>4</sub>) is used as reactive gas to replenish the targets (MgF<sub>2</sub>) stoichiometry. Finally, we added heaters to the chamber to improve microcrystalline film properties.

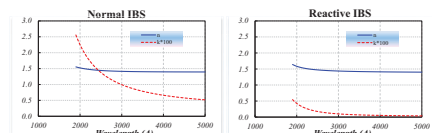
#### Ion Beam Sputtering Coating Chamber



#### Reactive Ion Beam Sputtering (RIBS)

- Non-thermal evaporation process
- Atoms from a target are ejected by momentum transfer from energetic atom-size particles
- Particles are energized by an ion gun
- Deposition rate are much lower than PVD 1-5 Å/Sec.

- Optimization and characterization of MgF<sub>2</sub> films using the IBS process did not give satisfactory results (see graphs below)
- Problem is thought to be traced at degradation of cathode filament due to reactive fluoride containing gas (Freon) in chamber



#### Perkin Elmer Lambda 950



#### PE Specifications

- Double-beam ratio recording grating monochromator
- Wavelength range: 180-3300 nm; Resolution: 0.5-5 nm
- Quartz Tungsten Halogen and D2 lamps
- Lead-sulfite and PMT detectors
- Photometric Range: Up to >8 Absorbance units (with attenuation)
- Universal Reflectance Accessory for normalized reflectance between 8° and 68° angle of incidence.

#### ACTON Research Corporation (VM-S21-SG)



#### \*ACTON Specifications

- One-meter high-vacuum monochromator (1200 grooves/mm)
- Wavelength range from 30 nm to 325 nm.
- Wavelength hydrogen-purged light source (discrete H2 emission lines between 90 nm and 160 nm and a continuum at higher Å)
- Photomultiplier Cathode Tube with light pipe equipped with a fluorescence coating (sodium salicylate) for converting FUV to visible light
- Sample compartment allows absolute transmission and reflectance measurements at varying angles of incidence (12-68°) without the need of a reference