

# Aluminum Mirror Coatings for UVOIR Telescope Optics including the Far UV

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## ABSTRACT

NASA Cosmic Origins (COR) Program identified the development of high reflectivity mirror coatings for large astronomical telescopes particularly for the far ultra violet (FUV) part of the spectrum as a key technology requiring significant materials research and process development. In this paper we describe the challenges and accomplishments in producing stable high reflectance aluminum mirror coatings with conventional evaporation and advanced Atomic Layer Deposition (ALD) techniques. We present the current status of process development with reflectance of ~ 55 to 80% in the FUV achieved with little or no degradation over a year.

**Keywords:** Large telescope optics, Aluminum mirror, far UV astrophysics, ALD, coating technology development

## 1. INTRODUCTION

It has been recognized that in the mid to far ultraviolet wavelengths ( $90 < \lambda < 300$  nm), it is possible to detect and measure important astrophysical processes, which can shed light into the physical conditions of many environments of interest in space. The astrophysics of a variety of Cosmic Origins science drivers requires access to ultraviolet emission diagnostics down to the Lyman edge at 912 Å. For example, in the local interstellar medium (LISM) all but two (Ca II H and K lines) of the key diagnostic of resonance lines are in the ultraviolet (Redfield<sup>1</sup> 2006). In addition to the fruitful science areas that ultraviolet spectroscopy has contributed since the early 1970s, France<sup>2</sup> *et al.* (2013) have emphasized the role of ultraviolet photons in the photo-dissociation and photochemistry of H<sub>2</sub>O and CO<sub>2</sub> in terrestrial planet atmospheres, which can influence their atmospheric chemistry, and subsequently the habitability of Earth-like planets. However, only limited spectroscopic data are available for exoplanets and their host stars, especially in the case of M-type stars. Similarly, new areas of scientific interest are the detection and characterization of the hot gas between galaxies and the role of the intergalactic medium (IGM) in galaxy evolution (Shull<sup>3</sup>, *et al.*, 2012). The Hubble telescope throughput cuts off around 1150 Å. The Far Ultraviolet Spectroscopic Explorer (FUSE) is “no longer operational and as such the community has lost its window on a set of critical spectral diagnostics, such as for H<sub>2</sub> and O VI, that are only available below 1150 Å.” [McCandliss<sup>4</sup>, *et al* (2010)].

The NASA Cosmic Origins Program Annual Technology Report (COR Technology Needs<sup>5</sup>, Table 7, Item 8.1.3., page 43, Oct 2011) defined the need for the “Development of UV coatings with high reflectivity (>90-95%), high uniformity (<1-0.1%), and wide bandpasses (~100 nm to 300-1000 nm)”. More recently, the Advanced Technology Large-Aperture Space Telescope (ATLAST) technology team assessed and stressed the technology development for maturing mirror coatings for the Far UV spectral range (Stahle<sup>6</sup>, *et al.*, 224th AAS Meeting, Boston, June 4, 2014 and Bolcar<sup>7</sup> *et al*, SPIE conference on UV/Optical/IR Space Telescopes and Instruments: Innovative Technologies and Concepts VII, 2015, paper 9602-8). A comprehensive summary of the Far UV science requirements (a Science Traceability Matrix (STM)) compiled by Paul Scowen<sup>8</sup>, can be found at <http://cor.gsfc.nasa.gov/RFI2012/rfi2012-responses.php>. Table 1 lists some of the important spectral lines in the FUV region for general astrophysics. Thus high reflectivity coatings covering the 100-300 nm spectral range are considered important for studying intergalactic matter (IGM). “The COPAG is considering a future large UVOIR mission for general astrophysics that would also perform exoplanet imaging and

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characterization. Some technologies may be specifically required to make these two missions compatible, for example telescope coatings” [COPAG Technology Assessment<sup>9</sup> 2011, page 5].

A successful pathway to achieve the objectives, namely to develop durable mirror coatings that will provide high reflectance over the extended spectral band in the far ultraviolet (FUV) to near infrared (NIR), requires the best choice of materials and processes. Void-free thin films of absorption-free materials are required to protect and maintain high reflectivity and durability of aluminum mirrors in laboratory and pre-launch environments. Precisely controllable and scalable deposition process is also required to produce such coatings on large telescope mirrors.

## 2. BACKGROUND

The main objectives are thus, a) to explore materials and processes to produce protective coatings for Al mirrors to perform with high reflectivity over a wide spectral range from the far UV to NIR, and b) to demonstrate fabrication of durable mirror coatings with chosen processes on distributed coupons representing a meter class mirror. To begin with, we investigated the applicability of common dielectric materials and known processes and identified promising candidates (Bridou<sup>10</sup>, *et al.*, 2010, Keski-Kuha<sup>11</sup>, *et al.*, 1999, Yang<sup>12</sup>, M., *et al.*, 2005). MgF<sub>2</sub>, LiF, AlF<sub>3</sub> stand out as primary promising materials for protective coatings while GdF<sub>3</sub>, LaF<sub>3</sub> and LuF<sub>2</sub> are other potential materials to be considered. We produced coatings of some of these materials by conventional vacuum deposition process and measured their basic properties. (Balasubramanian<sup>13</sup>, *et al.*, 2014). Over the past year, we conducted several coating experiments with conventional coating techniques as well as Atomic Layer Deposition (ALD) to produce thin MgF<sub>2</sub> and AlF<sub>3</sub> protective coatings. With the 1.2m coating chamber at Zecoat Corp., Torrance, CA, we produced a number of samples with chosen process conditions. Similarly, we produced several samples of newly developed ALD protective coatings on Al at JPL (Hennessy<sup>14,15,16</sup> *et al.*, 2015). We employed a Perkin Elmer UV-VIS spectrophotometer at JPL and an ACTON FUV spectrophotometer at GSFC to measure the reflectance properties of these samples. A spectroscopic ellipsometer was also employed at JPL to characterize the films. Theoretical model fits of measured characteristics were analyzed. Key advances have been on the ALD front with successful process development for MgF<sub>2</sub> and AlF<sub>3</sub> coatings at faster rates and lower temperatures.

We focused on the following main approaches initially:

1. Fabrication and testing of protected Al coatings (Al+MgF<sub>2</sub>, Al+AlF<sub>3</sub>, Al+LiF) by conventional coating processes
2. Producing Al mirror samples with bi-layer protective overcoats with MgF<sub>2</sub>, LiF and AlF<sub>3</sub>, the promising candidates.
3. Developing and optimizing ALD process for absorption-free thin MgF<sub>2</sub> and AlF<sub>3</sub> coatings, and fabricating Al mirrors protected with MgF<sub>2</sub> and AlF<sub>3</sub> layers.
4. Measuring time dependent changes in the reflectance characteristics of these mirror samples to establish performance stability.

### Single layer coatings of applicable transparent protective materials by conventional deposition process

Single layer coatings of MgF<sub>2</sub>, LiF, AlF<sub>3</sub>, LaF<sub>3</sub>, Na<sub>3</sub>AlF<sub>6</sub> and GdF<sub>3</sub> were produced initially with conventional coating processes in a 1.2 m size chamber fitted with resistive sources, electron gun and ion gun besides heater lamps, LN traps, cryo pumps, residual gas analyzers and computer controls, at pressures in the range of  $2 \times 10^{-7}$  to  $1 \times 10^{-6}$  Torr and temperatures in the range of 20 to 200 °C. Figure 1 shows the coating chamber employed for this purpose. A sample transport with masking mechanism was installed in the chamber. This enables multiple coatings on different samples without breaking vacuum. An FUV optical monitoring system was also installed in the system to measure reflected signal from the growing film during deposition and post deposition conditions such as total pressure, water vapor and oxygen content etc., for diagnostic purposes.

MgF<sub>2</sub>, LiF and AlF<sub>3</sub> are considered the most promising ones based on their UV transparency as evidenced by results from



Figure 1. A 1.2 meter coating chamber fitted with process controllers, thickness monitor and gas analyzer. (courtesy: Zecoat Corp)

these initial experiments. The other materials, particularly the high index fluoride materials, could be employed in other multilayer devices such as filters and beam splitters. Several coatings were prepared on fused silica and silicon substrates. Spectral performance characteristics of these coatings were measured with a state of the art UV-VIS spectrophotometer (Perkin Elmer Lambda 1050) as well as with an ACTON FUV spectrometer.

### Protected Al mirror coatings

Employing  $MgF_2$ ,  $AlF_3$  and  $LiF$  materials, single layer and bilayer protected Al mirror samples were produced earlier in 2014 with conventional deposition process in the chamber described above. Figure 2 shows the reflectance of 3 samples over the 200 nm to 1000 nm spectral range. This experimental data indicates a preference for  $AlF_3$  as protective layer. Similarly, Fig.3 shows the reflectance of a bi-layer protected Al mirror samples from initial experimental runs in the same chamber. These samples remained in the lab in a dry nitrogen flow box except during measurements involving a few days of exposure to normal lab environment with ~30 to 50% humidity. Figure 4 shows the spectral reflectance performance of a bi-layer ( $LiF+AlF_3$ ) protected Al mirror sample over the FUV to NIR spectrum measured over a period of 14 months after fabrication. Excellent stability is seen. While UV to NIR (200 to 1000nm) reflectance measurements were done at JPL, FUV (50 to 200 nm) measurements with an ACTON FUV spectrophotometer were done at Goddard Space Flight Center (GSFC). These samples were produced with conventional deposition techniques. Optimization and enhancement of reflectance in the 100 to 200 nm range is a subject of further experimental investigation of process conditions and layer structures. In this context, research done at GSFC has been reported (Quijada<sup>17,18</sup> *et al*, 2012 and 2104). ALD deposition of such coatings is now in progress at JPL.

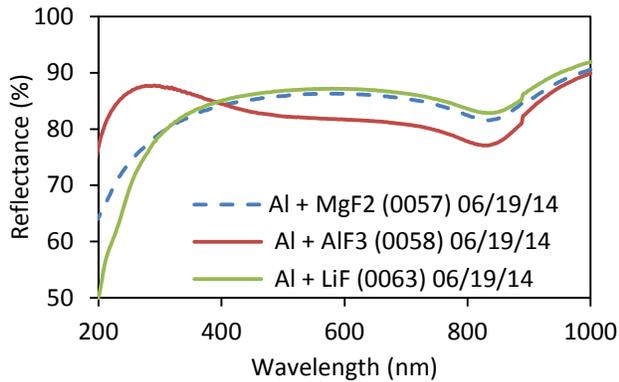


Figure 2. Reflectance of single layer protected Al mirror samples on fused silica substrates. Measurements in the 200 to 1000nm range after about 8 months from the date of fabrication.

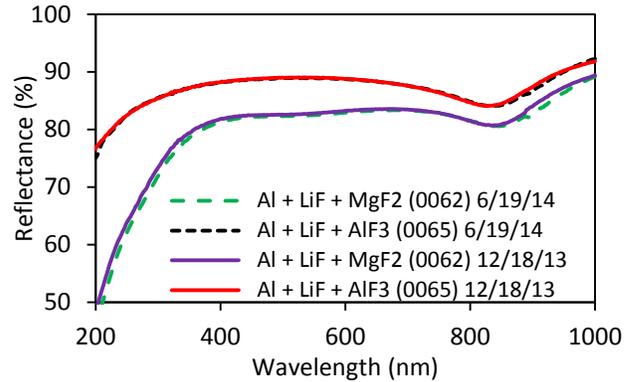


Figure 3. Reflectivity of Al+LiF mirror samples with  $MgF_2$  and  $AlF_3$  protective layers. Measured after 6 months, these samples show little degradation.

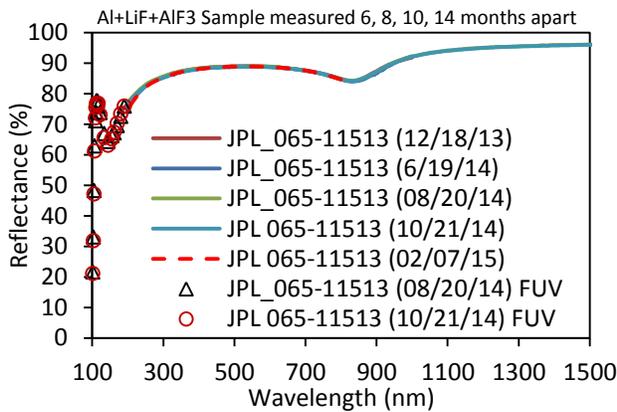


Figure 4 (A). Measured reflectance of a bi-layer protected Al mirror sample measured 6, 8, 10 and 14 months after fabrication showing excellent stability.

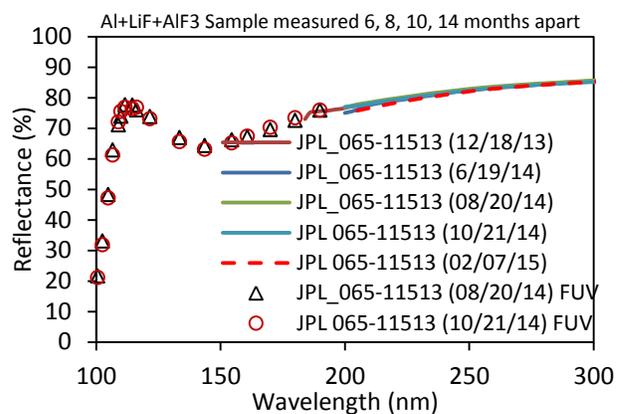


Figure 4 (B). Expanded view of the data in Fig 4(A) showing details in the FUV range.

### 3. ATOMIC LAYER DEPOSITION (ALD)

ALD process is under development at JPL to produce  $\text{MgF}_2$  and  $\text{AlF}_3$  protective coatings for high reflectivity mirrors with an Oxford OpAl showerhead-style ALD reactor shown in Fig. 5 and a Beneq ALD system shown in Fig. 6.

ALD films were deposited using bis(ethylcyclopentadienyl) magnesium ( $\text{Mg}(\text{EtCp})_2$ ) and trimethylaluminum (TMA) as the metal-containing precursors and anhydrous hydrogen fluoride (HF) as the fluorine-containing precursor in our Oxford reactor. Although metal fluorides are not common ALD materials, there have been several previous reports of their deposition often using metal fluorides such as  $\text{TaF}_5$  or  $\text{TiF}_4$  as the fluorine-containing source. [Pilvi<sup>19</sup> 2007, Mantymaki<sup>20</sup> 2013] This tends to result in residual metal contamination which degrades the absorption properties in the far UV and results in a process which can only be performed at substrate temperatures greater than 250 °C. As a result of this high deposition temperature, the fluoride films deposited with this method tend to crystallize readily resulting in significant surface morphology which is undesirable for many optical applications. In contrast, the JPL-developed ALD process using HF results in fluoride materials with lower residual contamination that can also be deposited at low temperature resulting in smoother, denser films. Further AFM studies will more precisely investigate the surface roughness of these materials as a function of process conditions.

As part of this effort,  $\text{MgF}_2$  and  $\text{AlF}_3$  were deposited at substrate temperatures ranging from 100 °C to 250 °C. Film thickness and refractive index were measured by spectroscopic ellipsometry and monitored as a function of process conditions such as process purge times and substrate temperature. Recent results on the same JPL ALD materials have been reported showing good optical performance at wavelengths down to 90 nm. [Moore<sup>21</sup> 2014, Hennessy<sup>14,15,16</sup> 2015].

Typical ALD conditions involve heating of the  $\text{Mg}(\text{EtCP})_2$  precursor which is then bubbled with Ar into the process chamber at exposure times of approximately 1 sec. TMA and HF are delivered by vapor draw at room temperature at shorter exposure times of 15-30 ms. The chamber is purged with Ar between each half-cycle exposure in the ALD process in order to ensure saturated, self-limiting deposition. We have demonstrated both  $\text{MgF}_2$  and  $\text{AlF}_3$  films with thickness uniformities better than 1% over six inches in diameter. Initial XPS measurements suggest that the films are approximately stoichiometric and further experiments will investigate how material composition changes as a function of process conditions. Deposition of LiF, a crucial material for FUV coatings, is also under development now with initial promising results.

A key goal in the development of ALD process is to optimize the process acceptably low temperature, i.e., to be below 100 °C, in order to enable large mirror coatings at high vacuum. Our recent experiments indicate that this is achievable in the near term for the relevant fluoride materials.



Figure 5. ALD coating system at JPL; gas feedthroughs and process controls enable  $\text{AlF}_3$  and  $\text{MgF}_2$  coatings development



Figure 6. Beneq ALD deposition system at JPL

### Reflectance degradation of Al with an oxide formation

To assess the nature and progression of oxide formation on fresh Al coating, we conducted a series of control experiments with Al coatings of different thicknesses deposited at different rates at a high vacuum of  $\sim 2 \times 10^{-9}$  Torr in an UHV chamber at JPL. Figure 7 shows the measured (symbols) and modelled (lines) performance of an unprotected Al mirror in the wavelength range from 190 nm to 290 nm over a period of about 1500 minutes after deposition. A nm of oxide formation is estimated to be sufficient to degrade the reflectance as shown. Figure 8 shows the measured reflectance in the far UV of an unprotected Al mirror and its model fit with an oxide formation at different thicknesses. These measurements and simulations show that an oxide layer of  $< 2$  nm thickness affects the FUV reflectance dramatically.

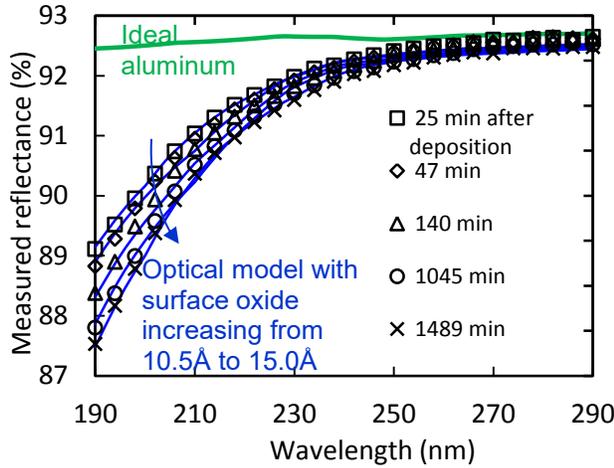


Figure 7. Oxidation induced reflectance reduction in the near UV of an Al mirror sample; model fits (lines) match (measured data symbols) a progressive increase of oxide formation.

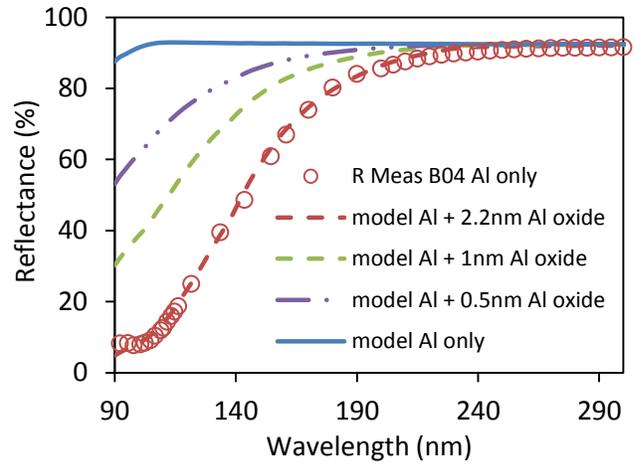


Figure 8. Unprotected Al reflectance (ideal) and modelled with a thin oxide layer matched measured characteristics in FUV spectral range

Rate of deposition of the Al layer is a critical parameter that affects the reflectance as well as its stability over time. Samples were fabricated by e-beam evaporation at different rates in high vacuum with a base pressure of  $\sim 2 \times 10^{-9}$  Torr. While further experiments are in progress, initial measurements indicate that a rate of about  $20 \text{ \AA/s}$  is favorable for obtaining better reflectance in the UV (see Fig 9) due to denser microstructure and lower oxidation in the bulk of the layer compared to lower rate samples. Unprotected and protected samples with ALD coatings were also fabricated and measured within about 20 minutes and thereafter for several days to assess the nature of degradation and the effectiveness a thin protective layer.

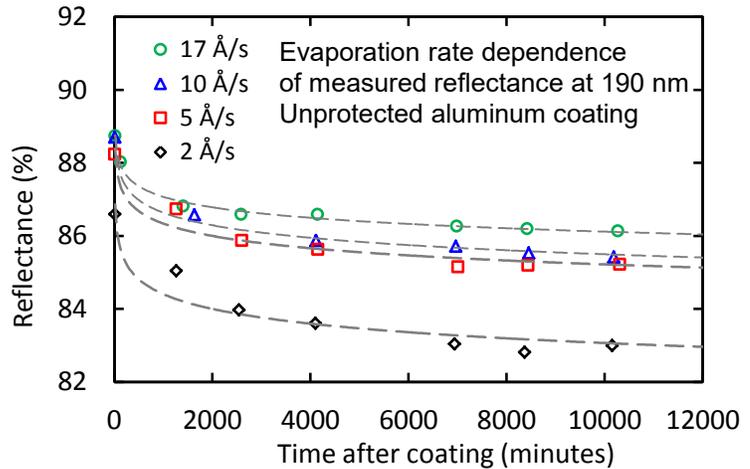


Figure 9. Measured reflectance at 190nm wavelength vs time after coating of unprotected Al samples fabricated with different evaporation rates

The drop is significant in the first 2 hours and especially within the first few minutes. Encouragingly, as Fig. 11 shows, a very thin protective layer of  $\text{AlF}_3$  preserves the reflectance adequately in laboratory conditions. This ensures a pathway to enhance and preserve the FUV reflectance of Al below 120 nm with an appropriate thin protective layer.

## Protected Al mirrors

A series of  $\text{AlF}_3$  coated Al mirror samples were prepared with different thicknesses of the protective fluoride coated with ALD process. The UV reflectance of four of these samples was measured over several days. Figure 12 shows the stability or change in reflectance from immediately after fabrication to over 6 days after fabrication. Figure 13 shows the FUV reflectance of the samples measured after several days. Model fits indicate a smaller level of oxide formation in the Al layer when thicker protective coating is applied. This is also compounded by the delay in coating the protective layer, a subject of further study.

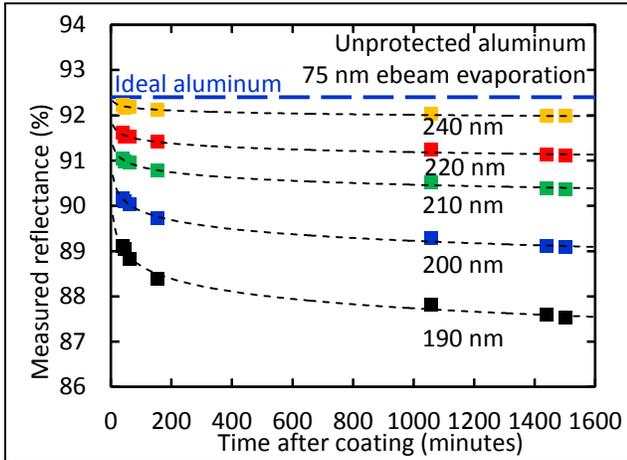


Figure 10. Unprotected aluminum degradation over time. Shorter wavelengths (below 190nm) would suffer greater degradation, not captured in the above data.

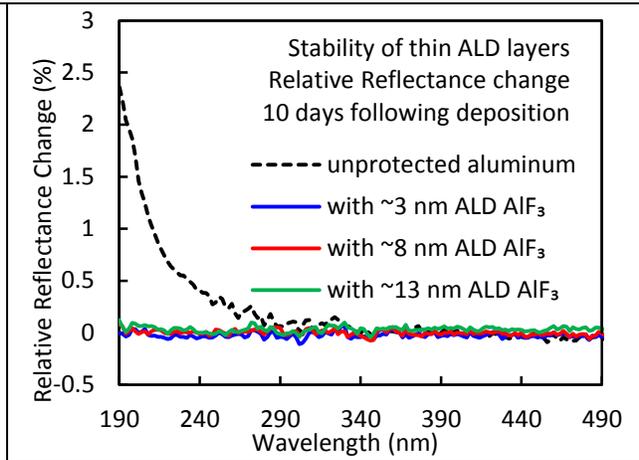


Figure 11. Stability of Al mirror (sample K series) coated with thin  $\text{AlF}_3$  layer by ALD; Even 3nm of  $\text{AlF}_3$  provides adequate protection against reflectance drop.

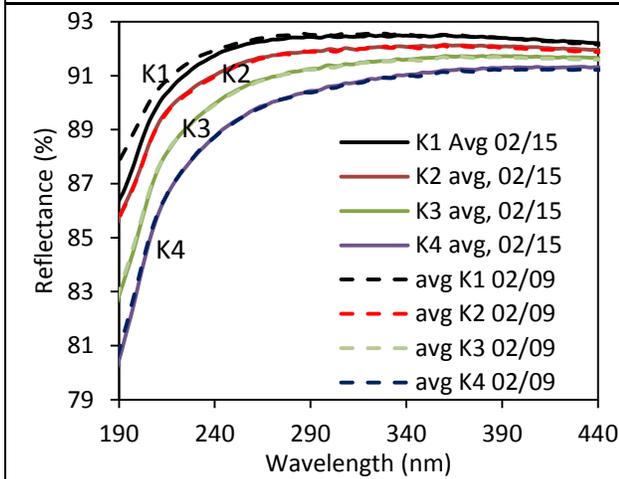


Figure 12. Aging performance of  $\text{AlF}_3$  protected Al samples compared with unprotected sample K1. K2 with  $\sim 3\text{nm}$   $\text{AlF}_3$  and K3 with  $\sim 8\text{nm}$   $\text{AlF}_3$  show no degradation over 6 days after fabrication. The initial drop from 92% to 86% at 190nm as seen above is due to the oxidation of the bulk of Al layer and a surface layer of  $\text{Al}_2\text{O}_3$  formed during and immediately after deposition.

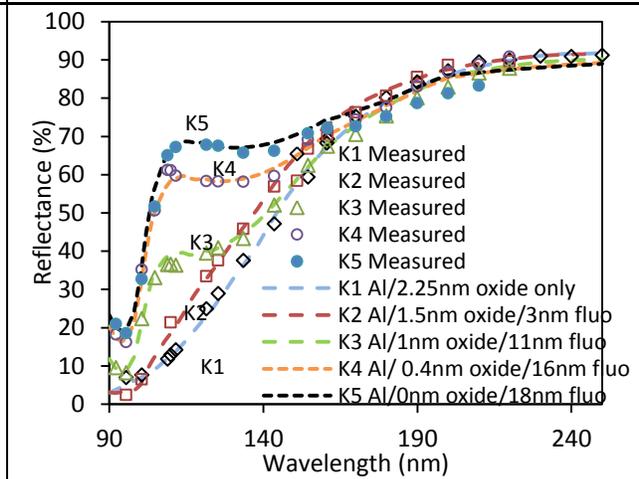


Figure 13. Model fits (dotted lines) of measured (symbols) FUV reflectance of unprotected (sample K1) and  $\text{AlF}_3$  protected samples (K2 to K5). Formation of an oxide layer before the application of a protective  $\text{AlF}_3$  layer is matched by models showing that the oxide formation is arrested by the fluoride layer, though not adequate yet. This is primarily due to the few minutes delay in transferring the sample to ALD chamber.

#### 4. RESULTS WITH CONVENTIONAL COATINGS AND ALD COATINGS

Figure 14 compares the FUV performance of three different coatings. Samples Z11 and Z15 were produced by conventional thermal evaporation techniques in one chamber. Sample K5 was produced with a combination of e-beam evaporation of Al in a UHV chamber and ALD coating of  $\text{AlF}_3$  in an Oxford ALD chamber. The sample had to be transferred from the UHV chamber to the ALD chamber during which time it was exposed to ambient conditions. Such exposure for a few minutes causes oxide formation on Al surface and reduces its reflectance inevitably. Yet the reflectance is  $> 50\%$  at 100nm and about 70% at 110nm as seen in these measurements. Further experiments and optimization are expected to yield  $>80\%$  reflectance at 100nm as predictable by models.

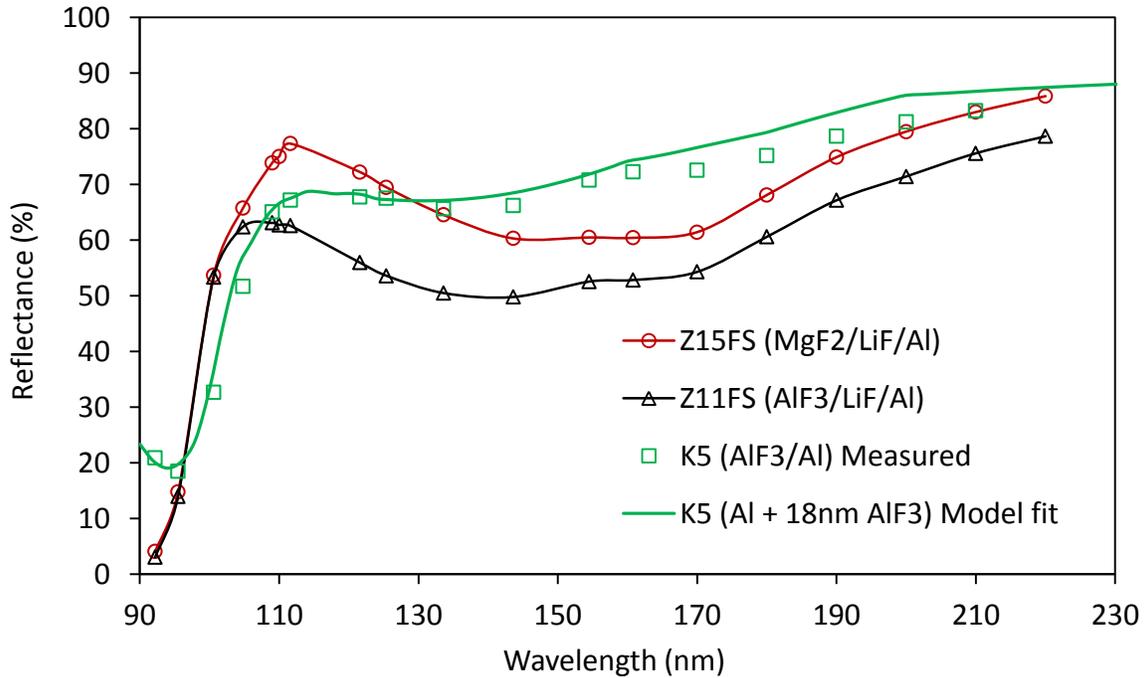


Figure 14. FUV reflectance of protected Al mirrors recently fabricated with conventional thermal evaporation (samples Z11 and Z15) in one chamber and by UHV and ALD processes in different chambers (sample K5).

#### 5. SUMMARY

Conventional vacuum deposition of Al mirrors with  $\text{AlF}_3$  and LiF as protective layers have shown stability of performance for over a year. Measured reflectance of these samples are in the range of 55 to 80% in the 95 to 120 nm FUV range while further improvements can be made with optimum layer thicknesses and process conditions. ALD coating processes have been developed at JPL for  $\text{MgF}_2$  and  $\text{AlF}_3$  coatings while LiF coatings are in development. Deposition rate, temperature and vacuum level have significant effect on reflectance as well as performance stability. More experiments are currently in progress to investigate these process parameters. In addition, we plan to coat a set of samples in the large chamber representing a meter class mirror to assess performance uniformity.

#### 6. ACKNOWLEDGEMENTS

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**Table 1:** Significant Diagnostic Spectral Lines in the UV (50-250nm)  
(Courtesy: Paul Scowen)

Wavelength (nm)	Species	Significance	Bodies of Interest
<b>68.1, 69.4</b>	Na IX	Coronal Gas ( $> 10^6$ K) Diagnostic (density, ionization state, etc.)	Intergalactic Medium, QSO sight lines
<b>77.0</b>	Ne VIII	Warm-Hot Gas ( $5 \times 10^5 - 10^6$ K) Diagnostic (density, ionization state, etc.)	Intergalactic Medium, QSO sight lines
<b>99.1, 175.0</b>	N III	Gas Temperature Diagnostic	Stellar Atmosphere Abundances
<b>102.6</b>	H, Ly- $\beta$	Lyman Series H Recombination Line	Plasma Diagnostics for Ionized Gas in Astrophysical Contexts
<b>103.2, 103.8</b>	O VI	Recombination Line Doublet	Diagnostic for Presence of Coronal Gas and the Boundaries between such gas and cooler gas envelopes or media
<b>108.5, 164.0</b>	He II	Balmer- $\gamma$ line for He	Stellar Atmosphere Diagnostic used to trace flares and CMEs
<b>117.5</b>	C III	Gas Electron Density Diagnostic	Stellar Atmospheres and Stellar Winds
<b>120.6</b>	Si III	Optically thin emission line of Silicon	Used as a diagnostic line sensitive to time variability in emitted or ionizing flux
<b>121.6</b>	H, Ly- $\alpha$	Lyman Series H Recombination Line	Plasma Diagnostics for Ionized Gas in Astrophysical Contexts – especially used for Cosmological Targets such as Reionization Era Galaxies and Stars
<b>123.8, 124.3</b>	N V	Gas Emission Diagnostic	Used to study extended stellar coronae
<b>133.5</b>	C II	Absorption Line for ionized Carbon	Used as a diagnostic and tracer for stellar chromospheres and planetary atmospheres
<b>139.4, 140.3</b>	Si IV	Emission Line of Silicon	Used to perform diagnostics of stellar coronae including density, temperature and abundance
<b>140.7</b>	O IV	Gas Density sensitive doublet	Used to study upper chromospheres in stars
<b>148.8</b>	N IV	Gas Diagnostic Line – sensitive in particular to electron collision strengths	Used to study stellar coronae and changes in their emission and bulk motion
<b>154.8, 155.1</b>	C IV	Gas density-sensitive doublet	Used to diagnose most ionized gas phases including stellar atmospheres and nebulae
<b>166.3</b>	O III	Gas temperature and density sensitive diagnostic	Used to diagnose nebula gas emission
<b>175.0</b>	N III	Gas temperature sensitive diagnostic	Used for stellar plasma observations and diagnosis
<b>189.5</b>	Si III	Gas density sensitive diagnostic	Used to study astrophysical plasmas
<b>190.9</b>	O III	Gas temperature and density sensitive diagnostic	Used to diagnose nebula gas emission
<b>232.6</b>	C II	Absorption Line for ionized Carbon	Used as a diagnostic and tracer for stellar chromospheres and planetary atmospheres
<b>233.6</b>	Si II	Gas density-sensitive diagnostic	Magnetic field diagnostic in stellar atmospheres, density diagnostic in stellar chromospheres

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