FUV Reflectance of recently prepared Al protected with AlF_3

COR Program Technology Development

By

Manuel Quijada/GSFC Code 551

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Mirror Tech/SBIR/STTR Workshop 2016

Outline

- Motivation
- Project Objectives
- Leveraging Success:
  - Acquisition of new FUV McPherson spectrometer
  - Transfer of vacuum coated to our branch
- Recent Technical achievements:
  - Al/MgF_2/LiF/AlF_3 Coating processes
  - AFM results
- Conclusions
- Acknowledgements

Enhanced UV Coating Applications

- Distant and faint objects are typically searched for in cosmic origin studies:
  - Origin of large scale structure
  - The formation, evolution, and age of galaxies
  - The origin of stellar and planetary systems
- Astronomical observations in the Far Ultraviolet (FUV) spectral region are some of the more challenging
- Very limited option of reflecting coatings to use at FUV wavelengths:
  - Modest reflectivity offered by those coatings
  - Al+MgF_2 [typically 82% at Lyman-alpha, 1216 Å) that are used on reflecting surfaces of FUV instrumentation
- Improved reflective coatings for optics at FUV could yield dramatically more sensitive instruments .
- Permit more instrument design freedom

Large UV/Optical/Infrared (LUVOIR) Decadal Mission Concept Study

LUVOIR Science Technology Definition Team (STDT) met at GSFC on 8/18-19/2016

Proposed instruments suite:

1. Optical / NIR Coronagraph:
    a) Capable of imaging and detecting bio-signatures on Earth-like planets around sun-like stars.
    b) Requires as broad a bandpass as possible (notionally 400 nm - 1.8 um, to be revised)
    c) Spectral resolving power of R~200
    d) Possibly to include polarimetry
2. UV Imager & Spectrograph:
    a) High-resolution point-spectroscopy (R~1,000 - 200,000)
    b) Multi-object spectroscopy capability (possibly including an IFS/IFU)
    c) UV Imaging with a minimum 1 arcmin x 1 arcmin FOV
3. Wide-field Optical / NIR Imager:
    a) 4-6 arcmin x 4-6 armin FOV
    b) Individual filter bands TBD, possibly to include a grism
    c) Possibly to include sub-microarcsecond astrometry capability
4. Multi-resolution Optical / NIR Spectrograph:
    a) Point-detector spectrometer, possibly to include an IFU/IFS
    b) R~100,000 for template matching & solar system science
    c) R~2,000 for transit spectroscopy
    d) Possibly to include high-precision 1cm/s radial velocity measurement capability
    e) Broad-wavelength coverage in a single shot, as red as possible (at least 3.5 um for transit spectroscopy)

Investment in developing high throughput FUV coatings is an **enabling** technology for #2

Project Objectives

- Use improved deposition processes to develop high performance mirrors in the Ultraviolet spectral range
- Specific tasks:
  - Research low-absorption materials to design and produced dielectric coatings in the FUV
  - Improve FUV mirror reflectance of aluminum mirrors over-coated with MgF_2, LiF, and AlF_3 in a large 2-meter UHV chamber
  - Coat variety of substrates using different materials covering the 90-170 nm spectral range.
- Key challenges:
  - Achieving high reflectivity (> 90-95%) in the 90 to 250 nm range requires absorption-free optical coatings
  - Low stress strong adhesion in coatings
  - Environmentally stable coatings
  - Precise & accurate techniques
  - Scaling coatings to large diameter (1+ meter class) mirror substrates

Leveraging Resources for Success

- Resource acquisition for coating testing & development
  - Acquisition of new FUV McPherson spectrophotometer for measuring transmittance/reflectance in the 90-170 nm ($225k).
  - Acquired from another branch a “turn key” Physical Vapor Deposition Kurt J. Lesker (Model # PVD 75).
  - Ongoing coating development activities of Al+AlF3 protected coatings (GSFC FY17 IRAD (PI: Manuel QUijada)

Acquisition of New FUV spectrophotometer

Completed purchase & installation of new McPherson 225 spectrometer on May, 2016

General Specifications:

- Model 225 1m focal length, f/10
- Spectral range: 90-200 nm
- Automated T/R capability permits accurate and fast data acquisition
- Windowless H_2 purged source
- PMT tube detector with vacuum tight scintillator coated window for response from 30 nm to 200 nm.

Kurt Lesker Thin Film Deposition System

- ➢ Took possession of a “turn-key” thin-film deposition system from another branch.
- ➢ Capability to perform test coatings (up to a few inches in diameter) for R & D purposes.
- ➢ Coating process includes Physical Vapor Deposition and Electron beam evaporation.

PVD 75™

PRO Line
Thin Film Deposition System

Kurt J. Lesker Company
PROCESS EQUIPMENT™ DIVISION

Applications

- Designed for university, industrial, and government lab R&D thin film deposition
- OLED/PLED and organic electronics applications
- Photovoltaics and semiconductor devices
- Optics and decorative coatings
- Small batch production

Features

- Fully enclosed "zero" clean room footprint or optional open frame design
- Box 304 stainless steel chamber with aluminum door and large viewport
- Manual touch-screen or recipe-controlled, PC based process automation
- Turbomolecular or optional cryogenic high vacuum pumping

Process Modules

- Magnetron sputtering: RF, DC, Pulsed DC
- Electron beam evaporation
- Thermal evaporation
- Organic materials evaporation
- Ion source substrate cleaning or assisted deposition

Options

- Substrate heating, cooling, or biasing
- Planetary substrate fixturing
- Upstream or downstream pressure control
- Film thickness control
- Substrate load lock
- On-site installation and training

www.lesker.com

Inside PVD 75 Coating Chamber

Substrate holder

Thickness monitor

E-Beam Source

PVD

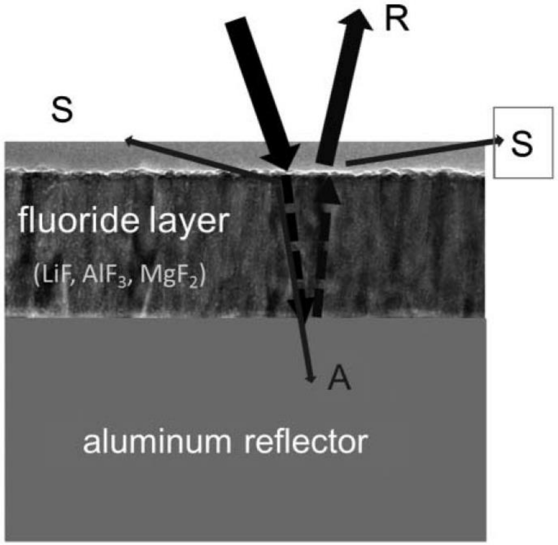
PVD 75 will be used primarily as thin-film R & D resource

Fluorides as Protection Layers

Using fluorides as protectors for Al:

(Wilbrandt *et.al. Vol. 53 No. 4 App. Opt. 2014)*:

Absorption edges: 116 nm (MgF_2), 110 nm (AlF_3), and 104 nm (LiF)



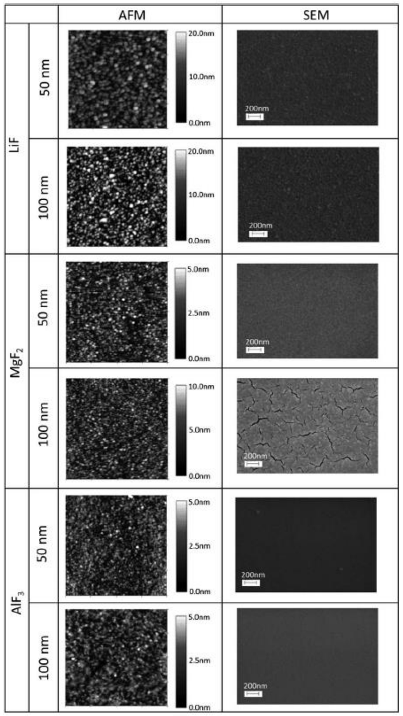
Optical Constants

$$R = 1 - A - S$$

Roughness

Overview of Roughness Values for Protected Aluminum Mirrors

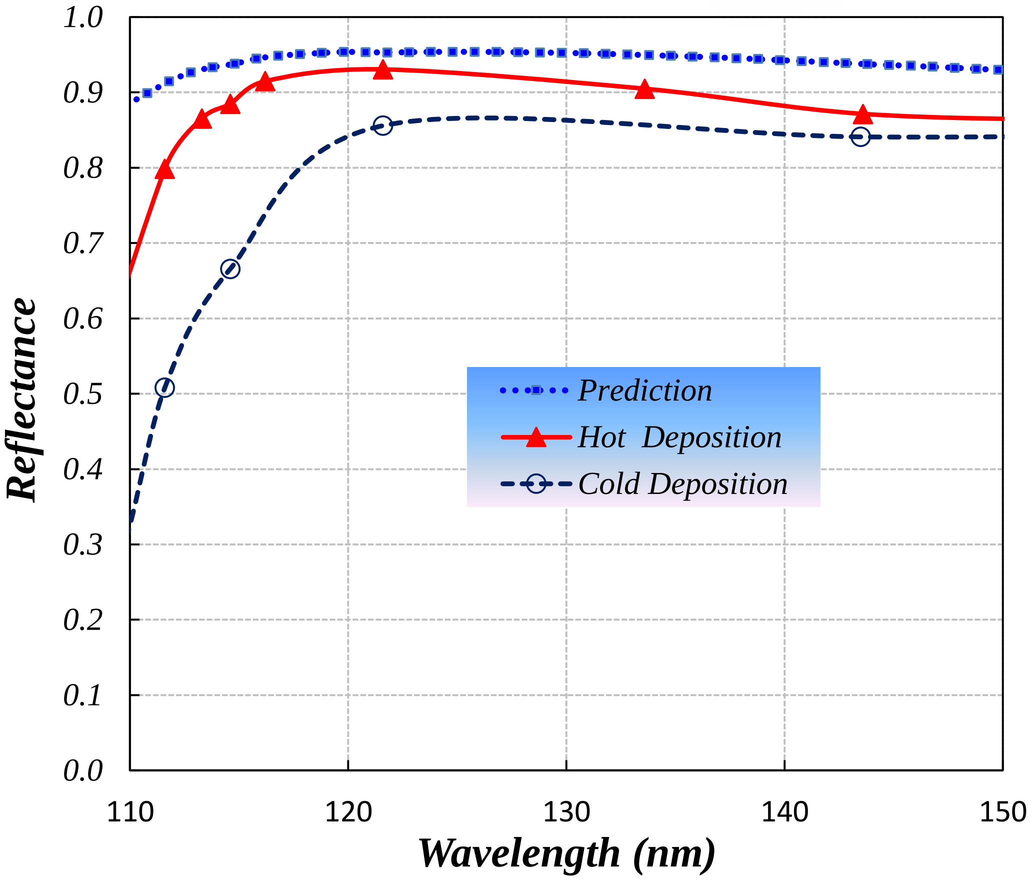
Type	Rate in nm/s	σ in Nanometer	
		1 µm	10 µm
Al+AlF_3	0.2	1.34	1.38
	2.0	1.23	1.31
Al+LiF	0.2	5.81	4.69
	2.0	5.20	4.16
Al+MgF_2	0.2	1.39	1.36
	2.0	1.70	1.51



- AlF_3 & MgF_2 exhibit the lowest (comparable) roughness.
- LiF films have significantly higher roughness.
- Surface roughness increases with layer thickness.
- Surface roughness decreases with increased deposition rate.

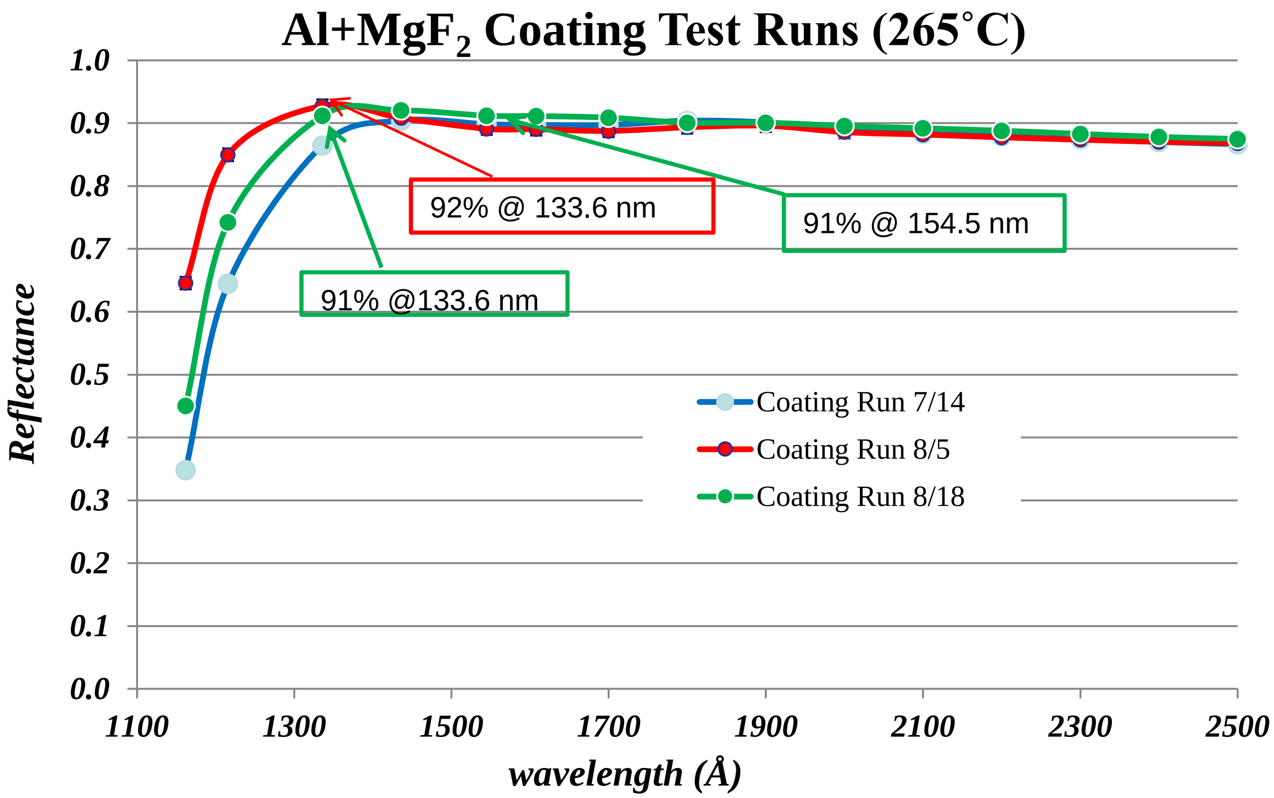
Al+MgF$_2$ Mirror FUV Performance

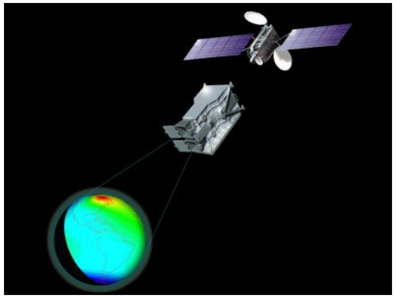
- Predicted vs. measured reflectance of bare Al and Al+MgF$_2$ reflectance (Al: 50.0 nm; MgF2: 25.0nm)
- Enhanced performance is obtained by heating (~220° C) substrate during MgF2 deposition
- Reflectance > 80% at λ > 115.0 nm (vs. 50% for "cold deposition")



ICON/GOLD Coating Tasks

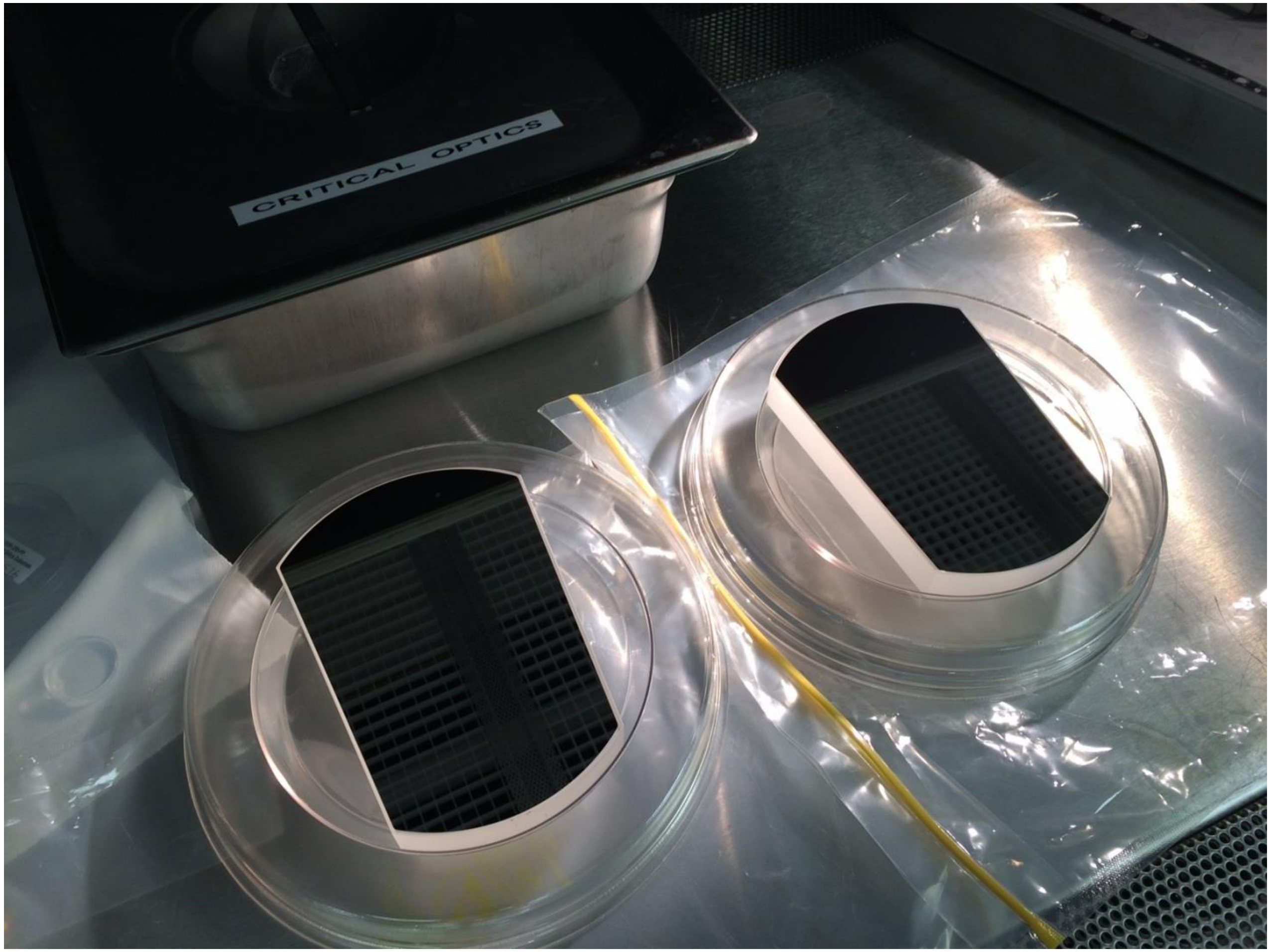
- ICON (Ionospheric Connection explorer): Study Earth’s low-orbit ionosphere sun interactions
- GOLD (Global-scale Observations of the Limb and Disk) : Imager to map Earth’s thermosphere & ionosphere



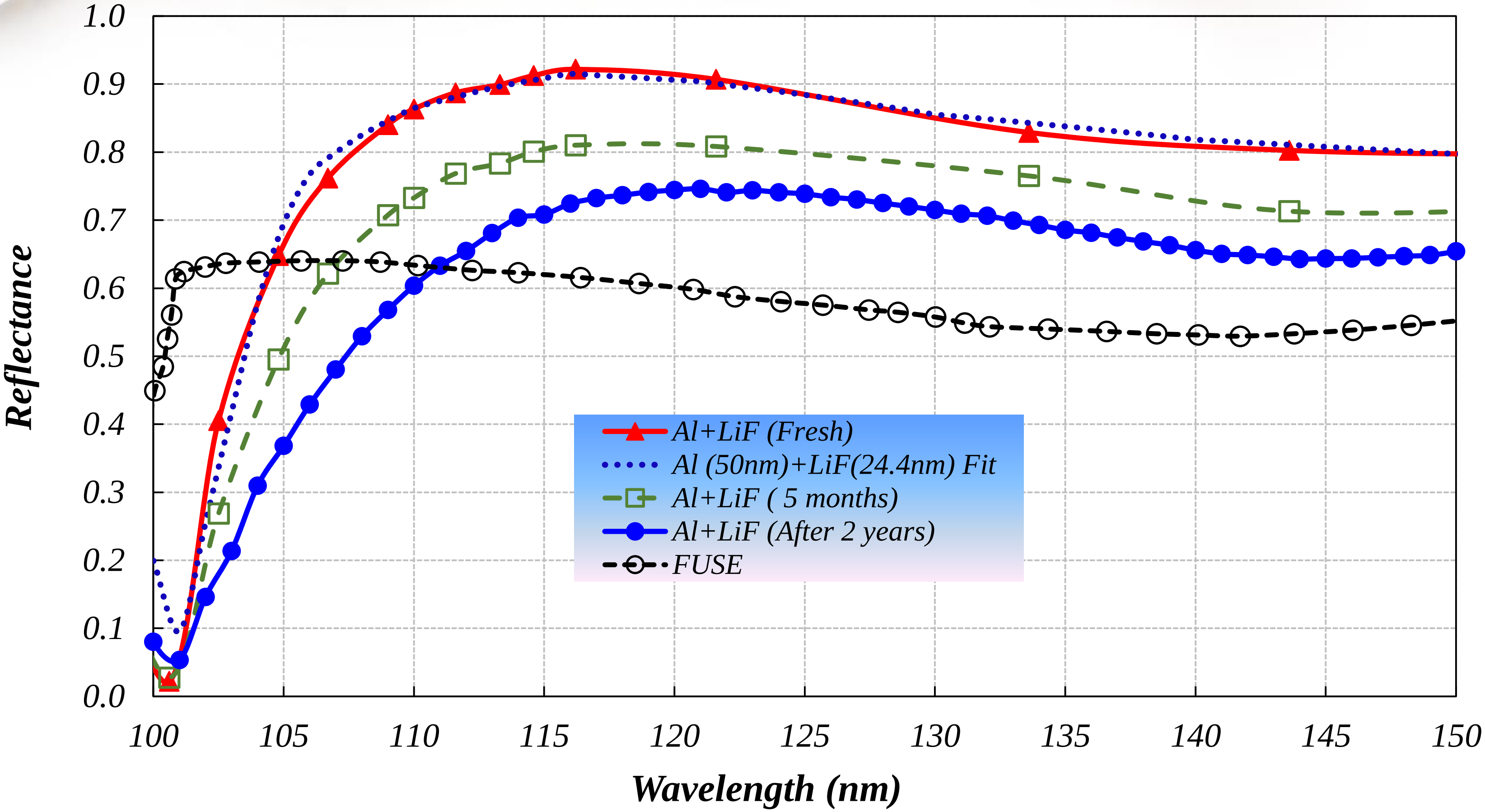


- ➢ A total of 12 optics ranging in size from 26 mm to 264 mm
- ➢ Coatings are optimized to produce reflectance over 90% in the 134-156 nm range

ICON Optics



Al+LiF Mirror FUV Performance



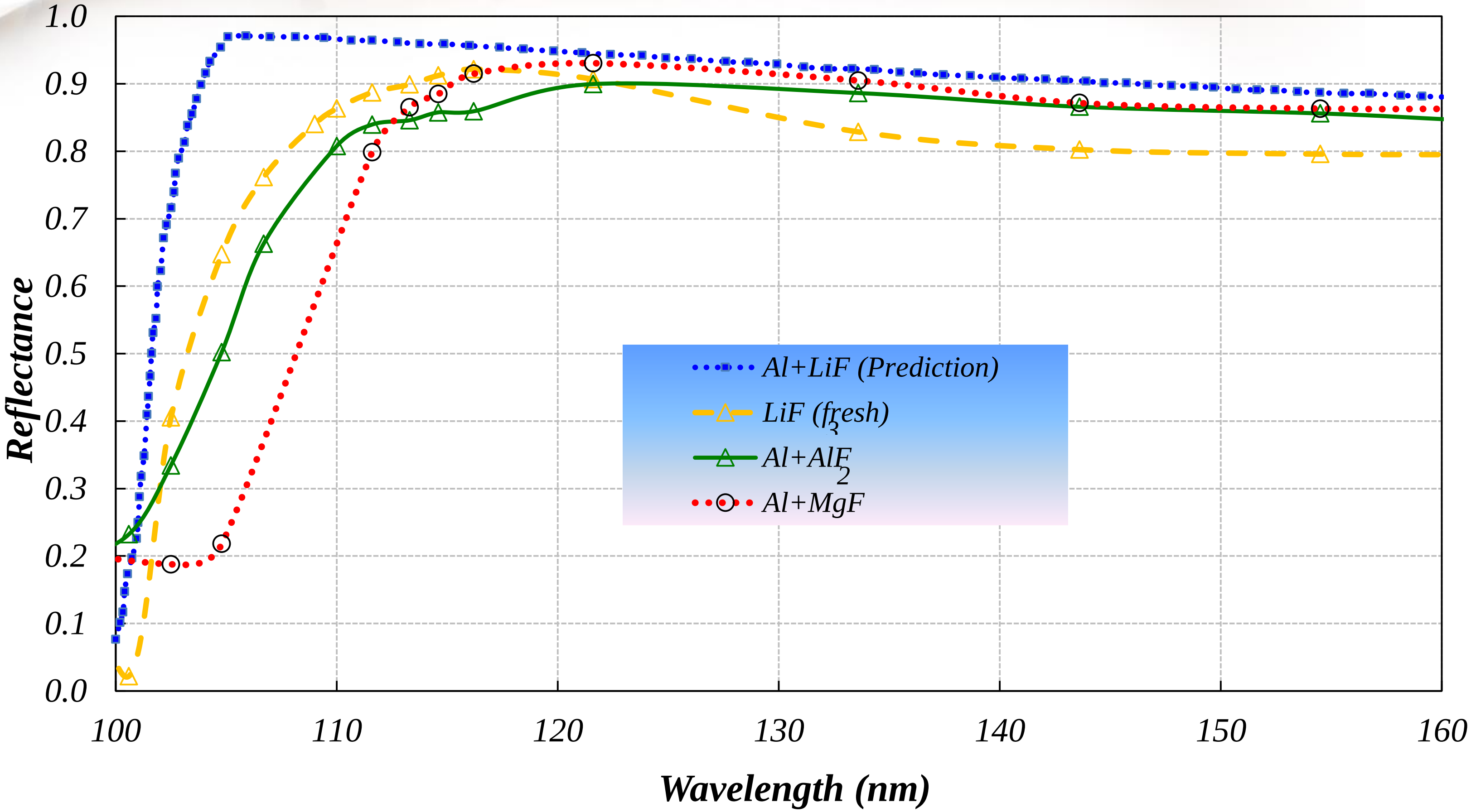
Coating recipe: Al (43nm, ambient)+LiF(8nm, ambient)+LiF(16.4nm, 250° C)
R_{ave}(100-150nm): 59% (FUSE) 75% (LiF)

LiF has to be deposited to optimize the 100-121 nm spectral range

Al+AlF_3 as FUV Mirror Potential

- AlF_3 is another low-index fluoride with good transparency in the FUV
- Absorption edge is between MgF_2 and LiF (110 nm)
- Hydrophilic material
  - ✓ The affinity to bond, at a molecular level, with water.
  - ✓ Note: This is not the same as hygroscopic, which is the ability of a substance to actively attract and absorb water vapor in the air without bonding
- Started to produce Al+AlF_3 test witness samples (to leverage success we have had with both MgF_2 and LiF
- Goal is to produce test samples:
  - ✓ To enhance FUV reflectance
  - ✓ Environmentally stable

Al+LiF Mirror FUV Performance



- AlF_3 thickness: 195 A (Al: 600 A)
- Reflectance @ 102 nm is 23% (AlF_3), 19% (MgF_2), and 2% (LiF)
- AlF_3 could be viewed as a viable alternative for FUV reflectors

Atomic Force Microscope Study

Al+MgF_2

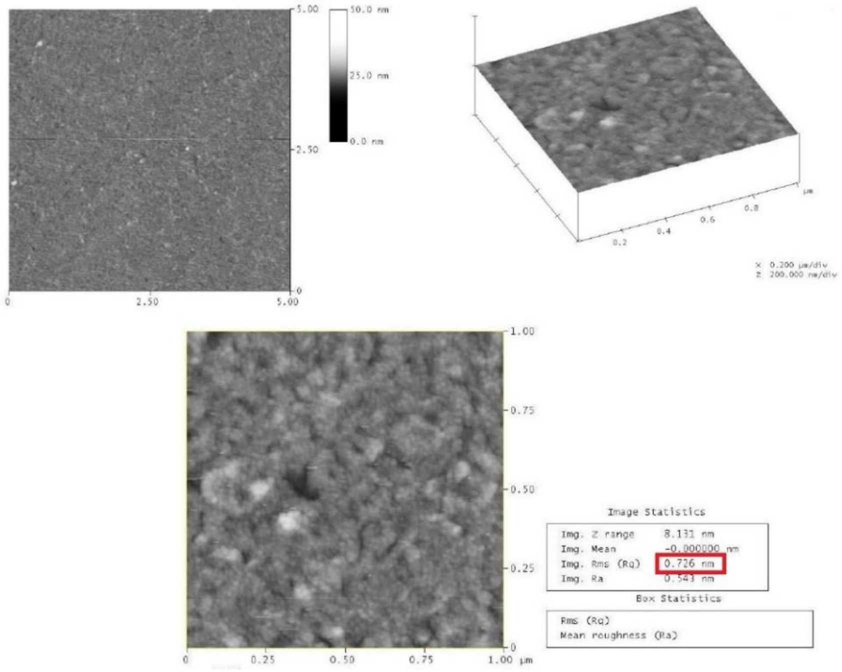


Image Statistics

Img. Z range	8.131 nm
Img. Mean	-0.000000 nm
Img. Rms (Rq)	0.726 nm
Img. Ra	0.543 nm

Box Statistics

Rms (Rq)
Mean roughness (Ra)

Al+AlF_3

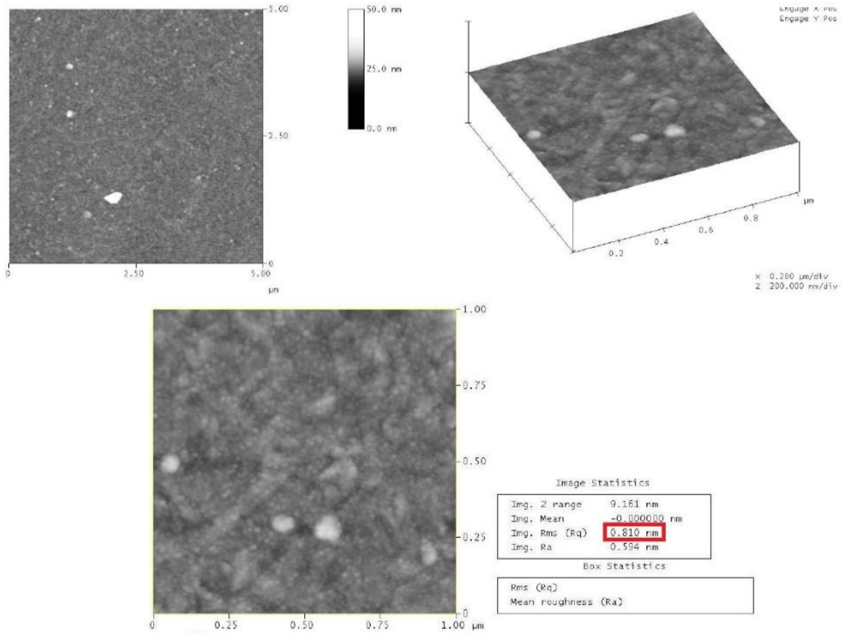


Image Statistics

Img. Z range	9.161 nm
Img. Mean	-0.000000 nm
Img. Rms (Rq)	0.810 nm
Img. Ra	0.594 nm

Box Statistics

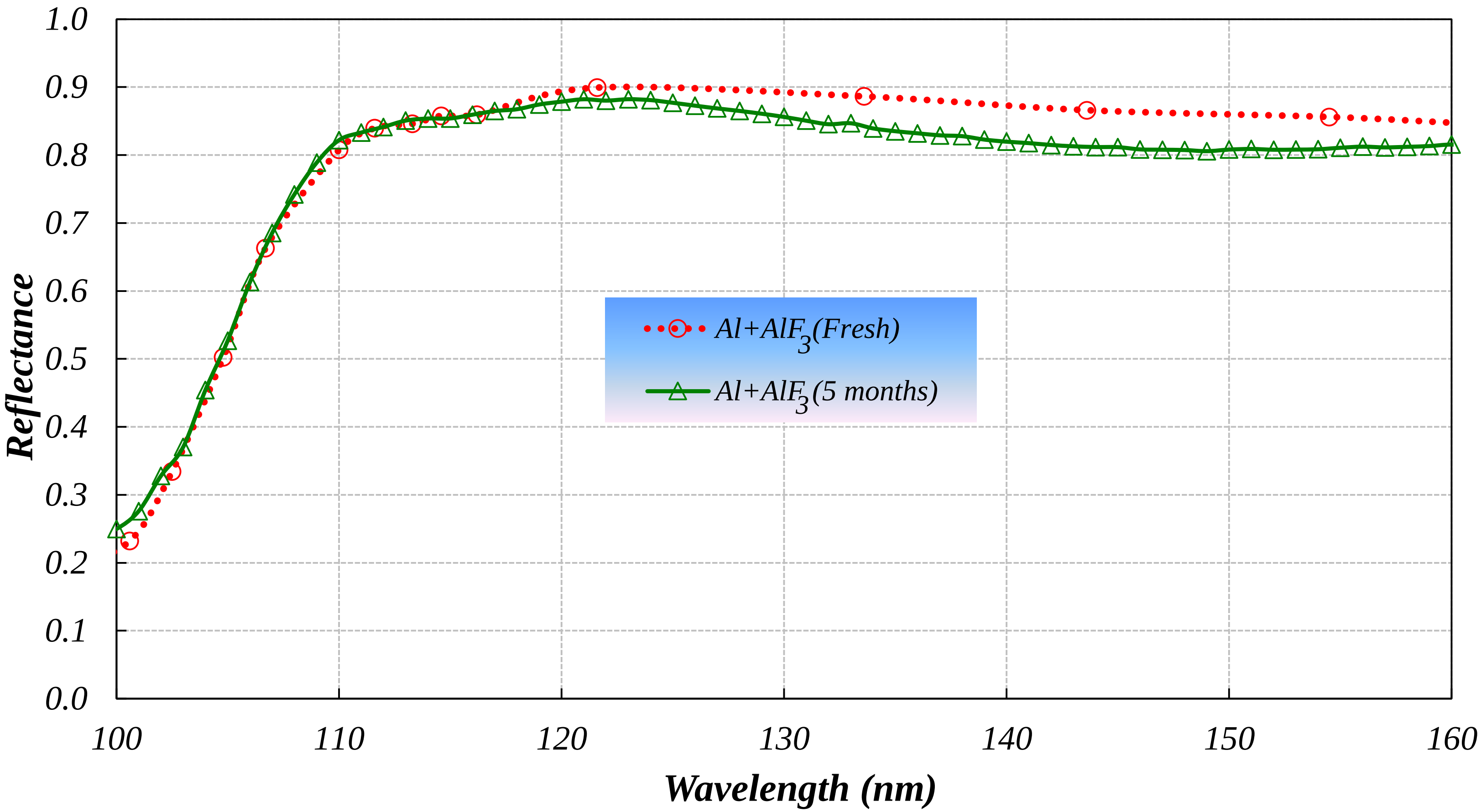
Rms (Rq)
Mean roughness (Ra)

Al+MgF_2 ⟹ Rq (RMS) = 0.726 nm

Al+AlF_3 ⟹ Rq= 0.910 nm

Results are 35 %(AlF_3) and 65% (MgF_2) lower than Wilbrandt *et.al.*

Al+AlF$_3$ FUV Reflectance Durability



Witness sample appears stable when stored in ambient lab conditions

Conclusions

- Reported gains in FUV reflectivity of $Al+MgF_2$ and Al+LiF mirrors by employing a 3-step process during PVD coating deposition of these materials.
- Applied enhanced FUV coating on ICON and GOLD projects.
- Recent acquisition of a new McPherson FUV spectrometer and transfer of a Kurt Lesker vacuum coater
- Successful demonstration of enhancement in FUV reflectance of $Al+AlF_3$ samples.

Acknowledgement

Collaborators: Javier del Hoyo/551,
Felix Threat/551,
Ed Wollack/665,
Vivek Dwivedi/545
Brian Fleming (U of CO)
Kevin Frances (U of CO)

GSFC Employment Opportunity: Optics Branch

Job posting on USAJobs:

https://www.usajobs.gov/
Vacancy Announcement Number: **GS17C0026**
Pay Plan, Series, and Grade: GS-0855-13
Code: 551
Duty Station: Greenbelt, MD
Closing Date: 11/10/2016

Enhanced MgF_2 and LiF Over-coated Al Mirrors for FUV Space Astronomy

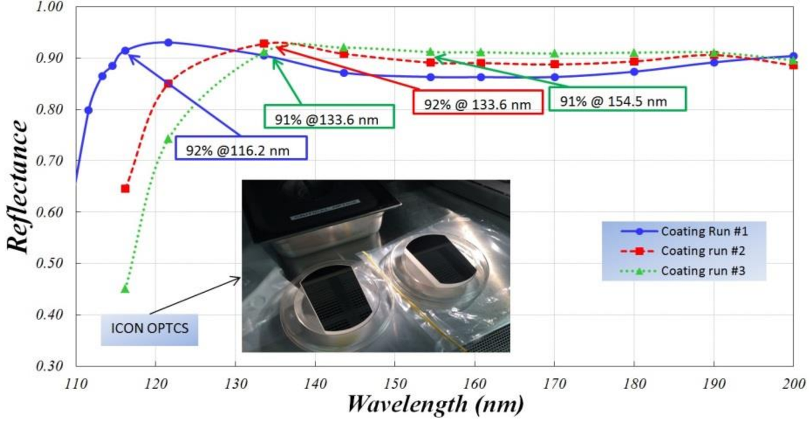
PI: Manuel A. Quijada/Code 551

Description and Objectives:

- Development of high reflectivity coatings to increase system throughput, particularly in the far-UV (FUV) spectral range
- Study other dielectric fluoride coatings and other deposition technologies such as Ion Beam Sputtering (IBS) that is expected to produce the nearest to ideal morphology optical thin film coatings and thus low scatter.

Key challenge/Innovation:

- Achieving high reflectivity (> 90-95%) in the 90 to 250 nm range
- Scaling up coatings to large diameter (1+meter) mirror substrates



Various test runs to produce coatings with over 90% reflectance for ICON optics in FUV

Approach:

- Retrofit a 2 meter coating chamber with heaters/thermal shroud to perform Physical Vapor Depositions at high temperatures (200-300 C) to further improve performance of Al mirrors protected with either MgF_2 or LiF overcoats.
- Optimize deposition process of lanthanide trifluorides as high-index materials that when paired with either MgF2 or LiF will enhance reflectance of Al mirrors at Lyman-alpha.
- Establish the IBS coating process to optimize deposition of MgF2 and LiF with extremely low absorptions at FUV wavelengths.

Collaborators:

- Javier del Hoyo, Steve Rice and Felix Threat (551)
- Jeff Kruk and Charles Bowers (665)

Development Period:

Oct. 1, 2011 – Sept. 30th, 2014

Accomplishments:

- Performed end-to-end testing of the 3-step Physical Vapor Deposition (PVD) coating process in 2 meter chamber to enable 1+meter class mirrors with either Al+MgF_2 or Al+LiF coatings for FUV applications
- Completed characterization of lanthanide trifluorides (GdF_3 and LuF_3) to pair them with low-index MgF_2 layers to produce narrow-bands dielectric reflectors at FUV wavelengths
- Production of mirrors with reflectance over 90% in FUV for ICON and GOLD projects.

Application:

- Application of these enhanced mirror coating technology will enable FUV missions to investigate the formation and history of planets, stars, galaxies and cosmic structure, and how the elements of life in the Universe arose.

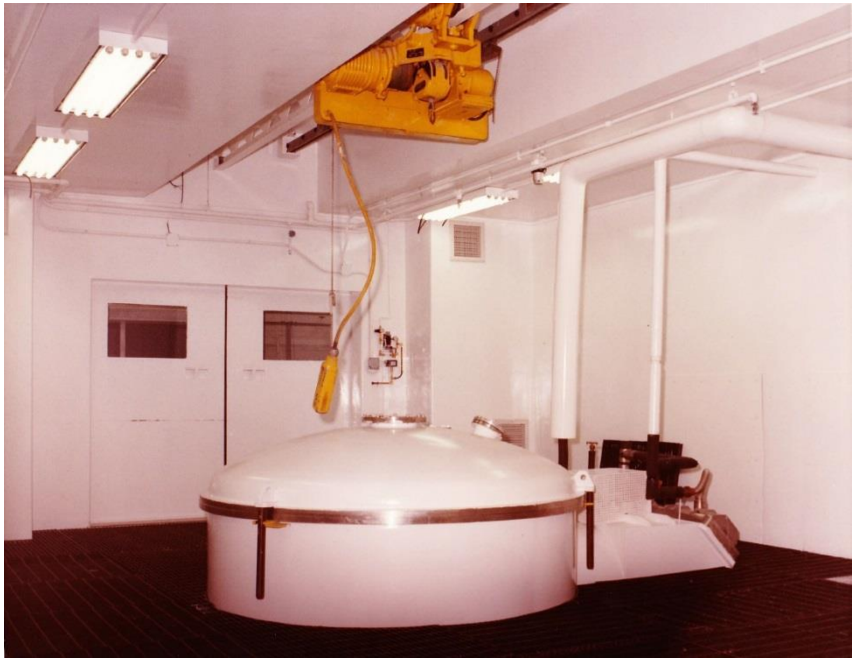
TRLin = 3 ***TRLcurrent = 4*** ***TRLtarget = 4***

Backup slides

GSFC Coating Facilities

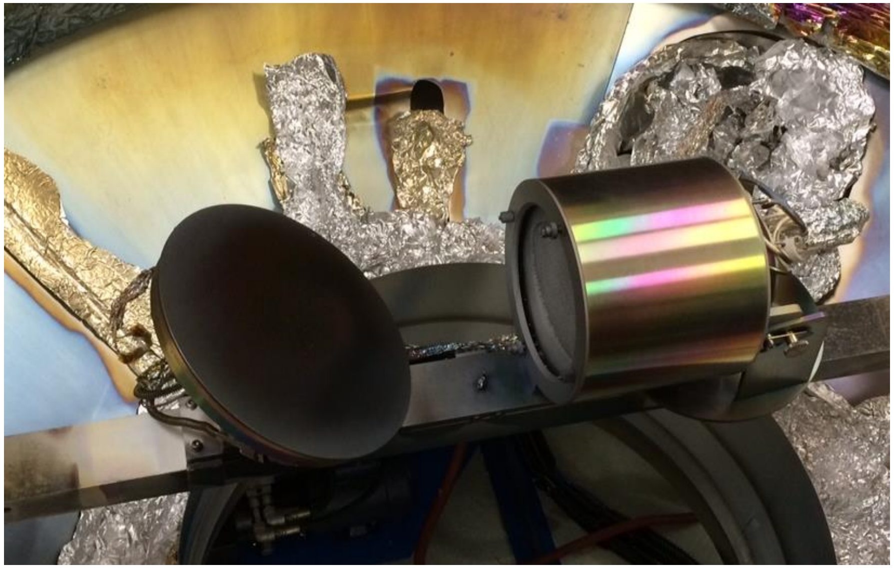
- Deposition methods: PVD, IBS, and RF Magnetron Sputtering
- Coatings produced: Al, MgF2,, LiF, SiO_x , GdF_3, LuF_3, Al_2O_3, Ag, Cr, Y_2O_3, SiC, B_4C.

PVD UHV coating chamber
(1-meter diameter)

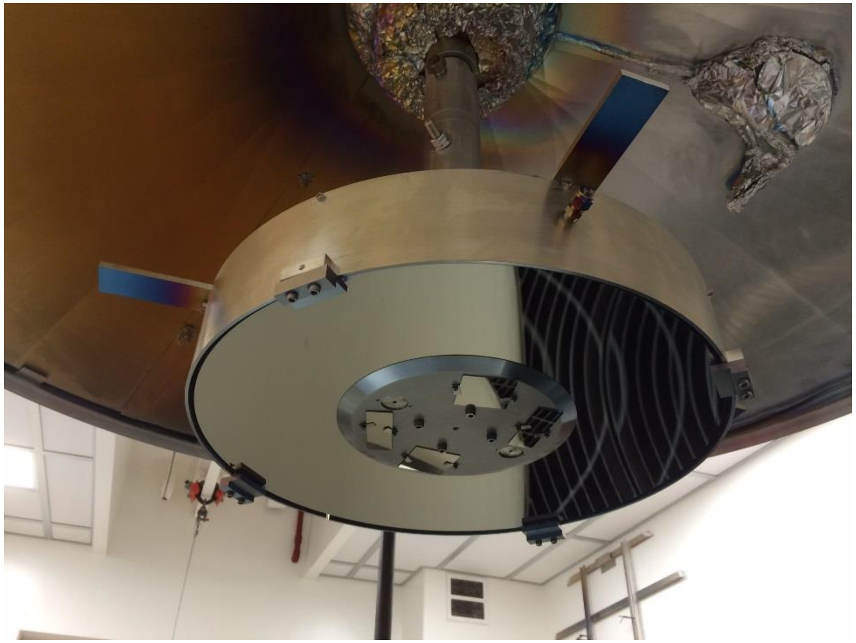


PVD/ IBS UHV coating chamber
(2-meter diameter)

2-Meter Diameter UHV Chamber



Ion Beam Sputtering Setup



Chamber top Cover with recently coated mirror substrate.

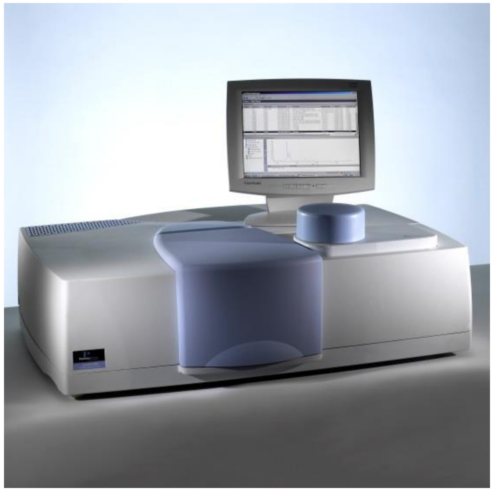
Missions supported: ICON, Astronomical Observatory (OAO) & Ultraviolet Explorer (IUE)
FUSE, HST (COSTAR, GHRS & COS), and a number of sounder rocket missions

Optical Characterization: T(λ), R(λ)

ACTON VUV Spectrometer

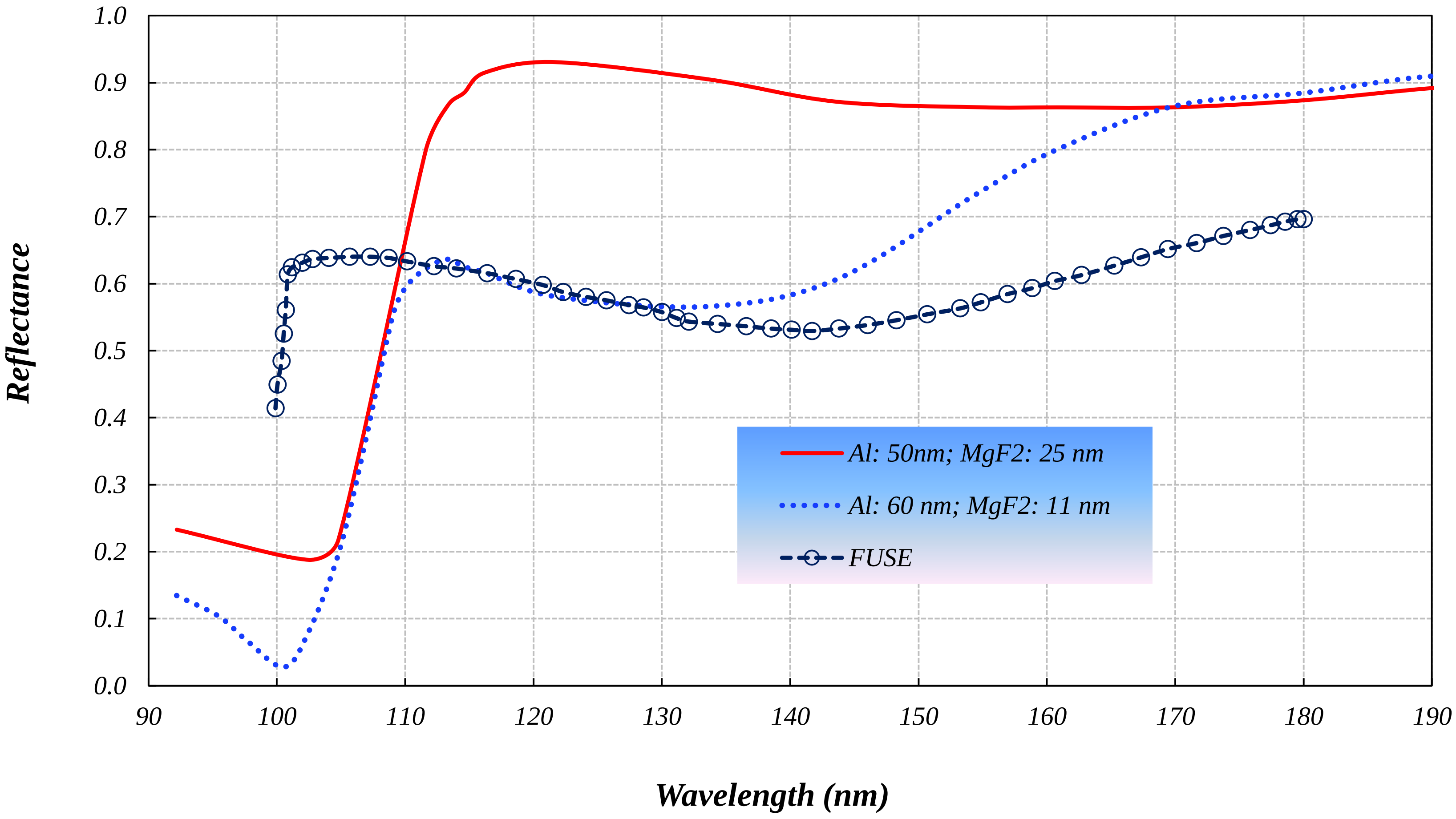
- Spectral range: 30-300 nm
- Source: Windowless H2-purged source (H2 emission lines between 90 nm and 160 nm and a continuum at higher
- Detector: PMT with fluorescence coating

Perkin Elmer Lambda 950

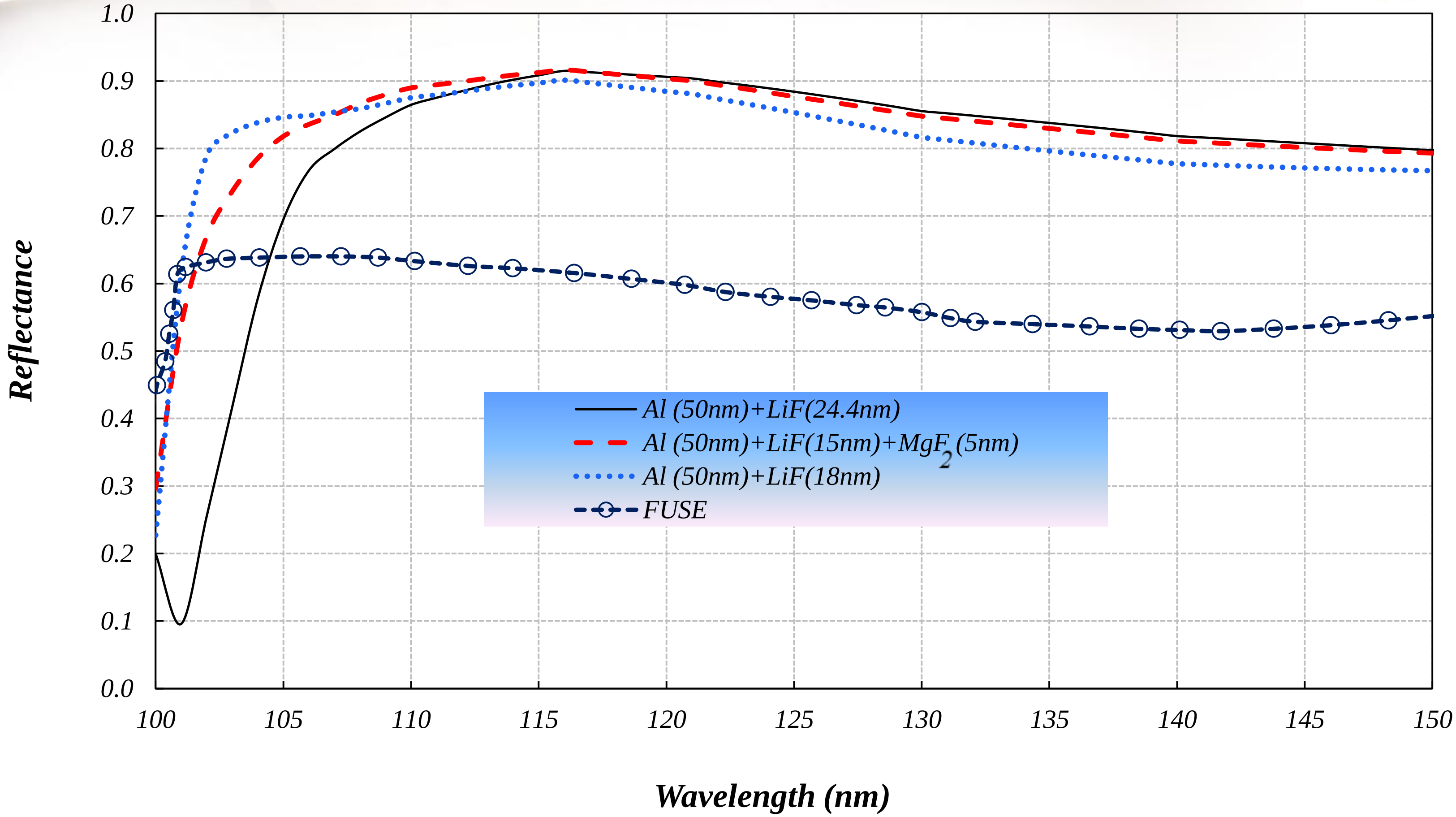


Spectral range: 190-2500 nm
Universal Reflectance Accessory

Thin MgF_2 Overcoat

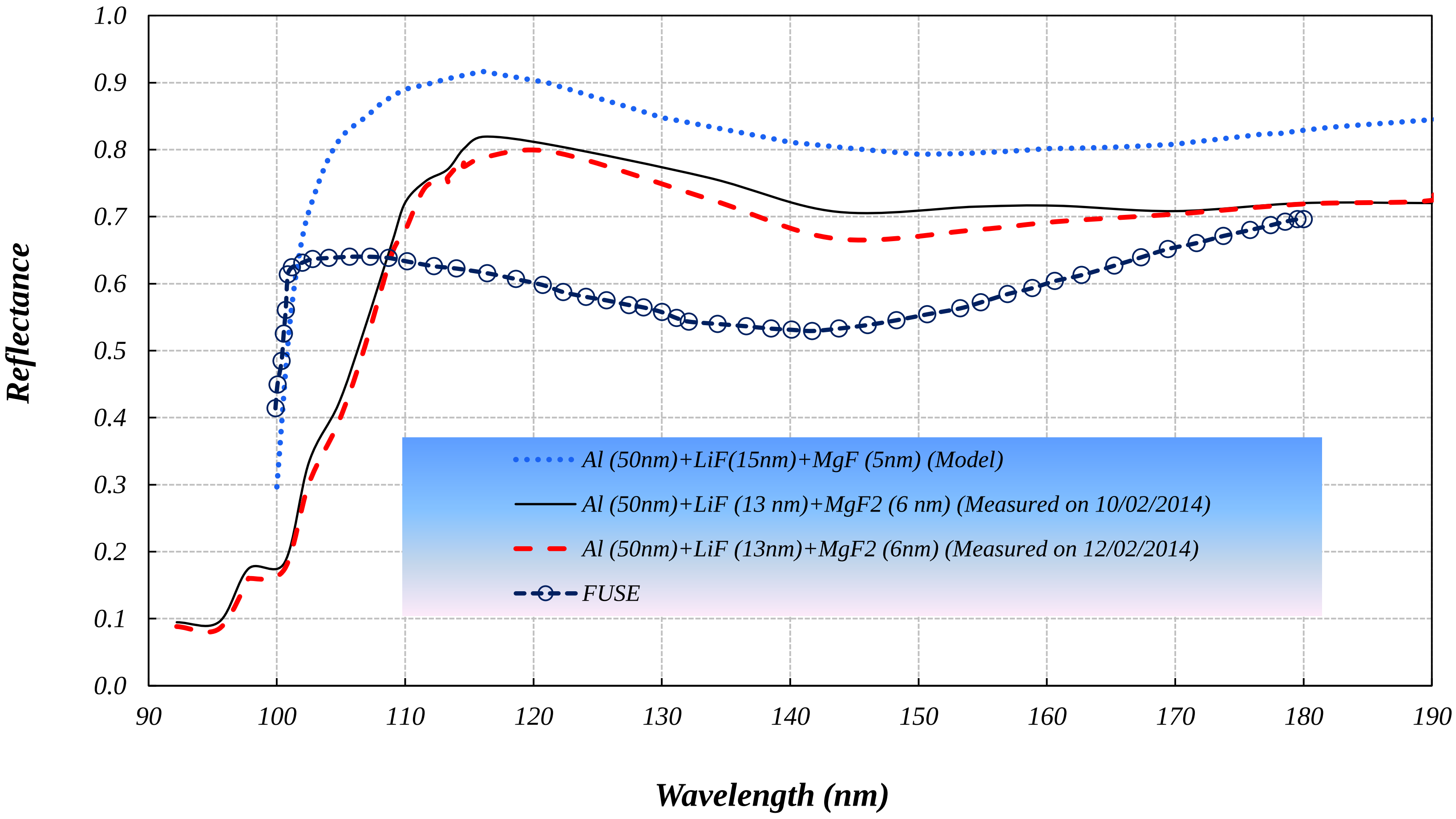


Al+LiF Mirror FUV Predicted Performance



Dual bowl fixture

Al/LiF/MgF$_2$ Test Run



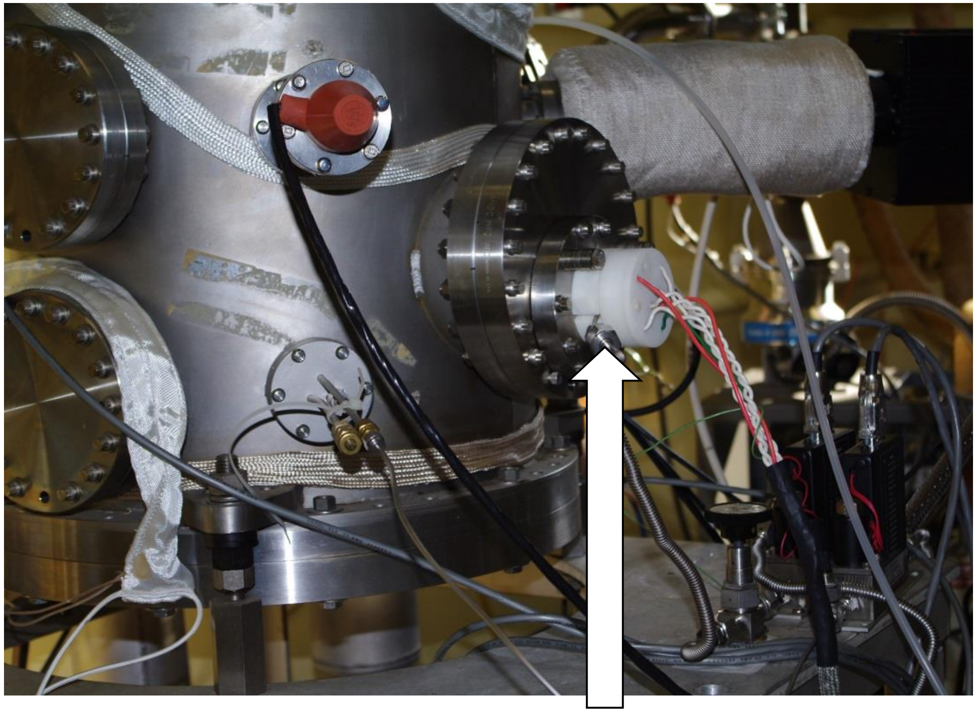
Al/LiF/MgF$_2$ Coating has been stable over a 2 month period!

Future Proposed Development

- Develop the capability to use IAPVD (Ion Assited Physical Vapor Deposition) to further enhance UV performance.
- Scale the process for larger optics (i.e. improved uniformity)
- Further develop methods to protect hygroscopic materials.

Ion Beam Sputtering Coating Chamber

- Upgrade chamber with a two-gas flow controller system.
- Krypton gas to be used in the ion-beam sputtering depositions.
- Freon (CF4) used as reactive gas to replenish the targets (MgF2) stoichiometry.
- Added heaters to the chamber: To improve microcrystalline film properties.



Reactive gas intake

Next Generation UV Spectroscopy

- Suborbital Imaging Spectrograph for Transition region Irradiance from Nearby Exoplanet host stars (SISTINE)
  - High-throughput using enhanced LiF GSFC coatings
  - 1st flown sub-arcsecond, medium spectral resolution (R=10,000) spectrograph covering 100-160 nm bandpass.