Electron-Beam-Generated Plasma to Enhance Performance of Protected Aluminum Mirrors for Large Space Telescope Astronomy

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Summary

This three-year Strategic Astrophysics Technology (SAT) program started in October 2019. The project addresses the need to develop and advance state-of-the-art optical-coating technology that will be key to providing improvements needed to realize the large mirrors planned for advanced NASA mission concepts such as the Large UV/Optical/IR (LUVOIR) Surveyor observatory, the Habitable Exoplanet Observatory (HabEx), and the Cosmic Evolution Through UV Spectroscopy (CETUS) [1, 2, 3]. LUVOIR and HabEx architectures will have a spectral coverage from the far-ultraviolet (FUV) to the near-infrared (NIR) spectral ranges (90-2500 nm), and CETUS will be an all-UV Probe. All three mission concepts have as goals to unveil fundamental information related to primary NASA interests, such as the history of galaxies, both the Milky Way and its neighbors; the origin of stars and planets; the demographics of planetary systems; and the search for life.

Although aluminum-based reflectors have been identified as a baseline for LUVOIR, HabEx ,and CETUS, the performance of this material is still deficient in the FUV [4, 5]. Accordingly, improving optical coating technology was identified as an "Essential Goal" in the technology needs in two separate Science and Technology Definition Team (STDT) studies commissioned by NASA to study the feasibility of a LUVOIR or HabEx missions. These studies, which were delivered as part of the National Academies of Sciences, Engineering, and Medicine, “Decadal Survey on Astronomy and Astrophysics 2020” report (released November 2021, have deemed that obtaining broadband coatings with a reflectance of at least 50–70% below 120 nm is an immediate, high-priority technology investment area, fundamental to mission feasibility. This SAT program aims to develop Al-based broadband optical coatings through a joint effort between NASA’s Goddard Space Flight Center (GSFC) and the US Naval Research Laboratory (NRL). We plan to leverage improvements in FUV reflectivity that have been developed at GSFC of producing Al coupons protected with one or more LiF/MgF_2/AlF_3 dielectric overcoats [6-8] and to develop a path for coating optics as large as 1+ m diameter, using the Large Area Plasma Processing System (LAPPS) reactor at NRL and their patented e-beam plasma system [9]. One of our primary goals is to remove oxide from bare Al surfaces and passivate the surface with an AlF_3 layer.

Preliminary studies on passivating thin Al films have already demonstrated the feasibility of a plasma process based on benign precursors for effectively fluorinating the surface of small Al mirror samples without highly corrosive hydrofluoric acid (HF). Further advancement of this initial work will develop a scalable treatment of Al surfaces on a scale as large as 1+ m diameter. Successful completion of this effort would provide enhanced reflectors in the FUV (90-180 nm) that would ultimately enable new scientific imaging capabilities for LUVOIR, HabEx, and CETUS.

From the start of this SAT program, we were able to bring on an assistant scientist/postdoc (Luis Rodriguez de Marcos) who has been responsible for the production of bare and protected Al mirrors with various metal fluoride films as well as FUV reflectance and other characterizations at GSFC. This program also facilitated the engagement of two Ph.D. students in the Pathways program at GSFC (Gabe Richardson, University of Arizona and Andrew Howe, University of Central Florida). Both have performed a variety of optical measurements that have been very valuable to this project. In addition, four other scientists/postdocs supported R&D activities at NRL (Virginia Wheeler, Jeff Woodward, Alexander Kozen,

Andrew Lang, and Samantha Rosenberg). They have been responsible for X-ray photoemission spectroscopy (XPS), atomic-force microscopy, transmission electron microscopy, X-Ray Reflectance (XRR), grazing-incidence small-angle X-ray scattering (GISAXS), and grazing-incidence wide-angle X-ray scattering (GIWAXS) characterization of untreated and treated samples in the NRL LAPPS reactor.

Background

The FUV is relevant to many aspects of NASA's Cosmic Origin (COR) program. However, performing measurements in this wavelength range has historically been quite difficult for a number of technical reasons, including poor reflectance of most materials in the FUV. Consequently, the region from 90 to 115 nm has only been explored by a handful of NASA astronomy missions. The Far Ultraviolet Spectroscopic Explorer (FUSE) observing program was the most extensive by far, but it was limited by a modest effective area (20 cm^2 below 100 nm and 55 cm^2 above 102 nm) and, for some programs, modest spectral resolution (R~20,000) [8, 10]. The average coating reflectivity at launch for this mission was 60% for Al+LiF, while that of the SiC coatings was around 30%. Improved reflectivity by itself would bring enormous throughput gains, and the benefits of more capable optical designs enabled by higher reflectivity would address the shortcomings noted above, bringing further sensitivity gains.

At the moment, the FUSE instrument represents the state-of-the-art, and improved optical coatings are critical to enabling substantial improvements in either sensitivity or spectral resolution [10]. For example, limitations in sensitivity and angular resolution have confined investigations to the very brightest individual objects and our knowledge of galaxy star-formation histories. Limitations in UV sensitivity have also severely handicapped our ability to explore the cycles of gas moving into and out of galaxies. The ability to image the immediate surrounding of nearby bright stars with a large sensitive telescope equipped with a coronagraph to suppress stellar glare would allow unprecedented studies of exoplanets, and thus expand our imaging capability to include a number of potentially habitable exoplanets. This increased sensitivity will dramatically enhance detection of Earth-sized planets to statistically significant numbers and allow in-depth spectroscopic characterization of the most favorable targets. Furthermore, an order-of-magnitude increase in capabilities will pave the way for exciting new discoveries that presently cannot be imagined. We believe that developing high-efficiency coatings in the FUV will spur missions that will address the science topics mentioned above by enabling a wide range of instrument capabilities including: 1) wide-field imaging across a broad spectral range, 2) high-contrast imaging and spectroscopy of circumstellar environments, 3) single-object spectroscopy across a broad spectral and resolution range, 4) multi-object spectroscopy of thousands of objects, 5) diffraction-limited spatially resolved spectroscopy, 6) astrometry of nearby bright stars (to infer exoplanet masses) and of more numerous fainter stars [11]. Moreover, diffraction-limited imaging in the FUV provides access to finer angular scales with a much smaller telescope than in the visible or NIR, and the sky is much darker. Advancing the state-of-the-art in optical coatings is a critical component to realizing the aforementioned promise of the next generation of space telescopes. A key component of this project is the collaboration with NRL and the use of the LAPPS facility.

The LAPPS facility was developed nearly two decades ago as a means of generating large-area (>1 m^2) high-density plasmas for materials processing [12, 13] and received considerable attention recently [14] as an attractive system to meet the demands of atomic layer processing. The system uses linear hollow-cathode electron sources to generate sheet-like electron beams with typical current densities of 1-5 mA/cm^2, and beam energies between 1-5 keV. The system is relatively simple, comprising an e-beam source, entrance aperture through which the beam is injected, termination anode, and magnetic field coils. Materials are introduced on a processing stage oriented parallel to the beam propagation direction and below the beam volume. Co-axial magnetic fields of 100-300 Gauss collimate the e-beam and thus improve uniformity along its length [14]. The chosen magnetic field strength leaves the plasma ions unmagnetized while aiding in electron confinement. These parameters are sufficient to produce uniform plasma sheets compatible with typical wafer-scale systems (diameter > 300 mm). LAPPS is able to generate uniform, low-electron-temperature (T_e), plasmas with densities in the range of 10^{10}-10^{12} cm^3 [15], allowing it to access processing conditions not achievable in conventional plasma processing systems. The processing stage can either be left at ground (unbiased) resulting in substrate bombardment by very-low-energy ions (< 5 eV) or biased using either DC or RF voltage to raise ion energy. Figure 1 illustrates the basic design of an e-beam-based plasma processing system.

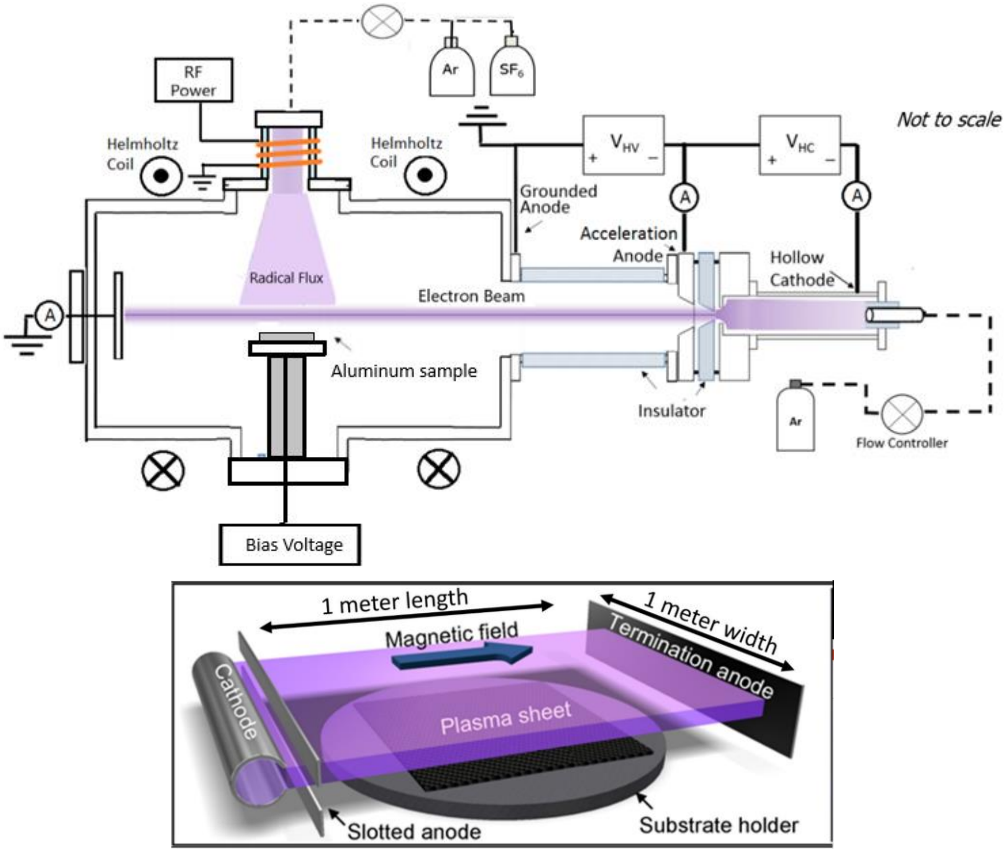


Fig. 1. Top: *Schematic of the plasma processing system used for native oxide etch and fluorine passivation of Al mirror samples. The system possesses a hollow cathode electron source allowing independent control of electron-beam energy and current over a wide range of operating pressures. The auxiliary ICP radical source also offers control over the ion/radical ratio in the processing system.* Bottom: *The LAPPS allows generation of very large area (>1 m^2), highly uniform, low-temperature plasmas. Plasma length is determined by beam energy and plasma width is determined by the length of the hollow cathode. Both length and width can be > 1 m if desired.*

Objectives and Milestones

The work plan in this project included a systematic study for determining which passivation technique performed at LAPPS has the highest potential to realize high-throughput reflectors based on Al mirrors for use in the FUV, and over a broader spectral range. Furthermore, the improvement in FUV reflectance of test samples through the successful outcome of this project provide a scalable process for enhanced FUV coatings on large optics (1-m diameter) using a facility like the GSFC 2-m vacuum chamber in combination with the LAPPS reactor.

Key milestones for Year 1:

1. Optimizing plasma parameters. In addition to upgrading the window on the LAPPS reactor, we perform a comprehensive characterization of plasma response to varying process parameters to optimize etching capabilities for Al-based coatings.
2. Oxide removal and passivation of bare Al mirrors with a thin AlF_3 layer. The main criterion for success is a process that is reproducible from one LAPPS treatment to the next in reaching a

reflectance of 60-70% at 105 nm and over 80% at wavelengths longer than 115 nm on bare Al coupons. Other metrics include a coating that (over time) is stable under ambient laboratory conditions (room temperature and 40-50% relative humidity). We also perform more controlled environmental tests to demonstrate the survival of these coatings. We believe that meeting these criteria is sufficient for advancing the TRL for this plasma process to 4.

Key milestone in Year 2:

1. Scaling up oxide removal and fluorination processes. Scaling up is demonstrated by running several small samples (25-50 mm) at the same time, spread over an area of roughly 150-mm diameter inside the LAPPS reactor. Verification of both the reflectance and stability criteria mentioned above is performed. Although scaling up to this size may seem modest, we believe that reaching this goal is sufficient to demonstrate the process is compatible for use in a 1+ m-class substrate as the only requirements will be a large enough chamber and appropriate magnet coil size.
2. Uniformity of metal-fluoride-protected Al. The target is a radial uniformity of < 5% (in a 0.5-m diameter) of the metal-fluoride layer (LiF, MgF_2, and AlF_3) in 2-m PVD chamber at GSFC.

Milestones for Year 3:

1. LAPPS treatment on protected Al coatings. Samples of Al made at GSFC with three types of metal-fluoride overcoats (LiF, MgF_2, or AlF_3) are sent to NRL for treatment in the LAPPS reactor. The criterion for success is to demonstrate controlled thinning-out of the fluoride layer to boost performance below 120 nm. Repeatability in restoring reflectance of aged samples to pristine values with all three fluoride layers is also demonstrated along with the scaling-up process. Development of this plasma-based cleaning process for removing contaminants in large mirrors advances TRL (3→ 4) and closes a technology gap relevant to the LUVOIR and HabEx concepts, as stated in the 2017 COR Program Annual Technology Report [16].
2. Detailed polarization studies [17]. Ellipsometric techniques are used to characterize the most promising coatings to assess the impact of polarization on HabEx- and LUVOIR-like missions. Results are shared with the communities studying the feasibility of these mission concepts.

Progress and Accomplishments

Year 1:

A comprehensive exploration of the parameter space was carried out. This task spanned from Year 1 to mid-Year 2, but for sake of clarity we summarize this milestone here.

The methodology for this task was as follows: First, a set of bare optical glasses were coated with Al in a single deposition run in the coating facility at GSFC. Then, the mirrors were sent to LAPPS. As mirrors were exposed to the atmosphere, the surface of bare Al films oxidized immediately after contact with air; the thin (~2 nm) but highly absorbing oxide layer strongly degrades Al reflectance below wavelengths of 200 nm. Each mirror was then independently processed at NRL using a different set of parameters, with the goal of removing the native oxide layer from the surface of the Al thin film while promoting formation of a thick and dense AlF_3 surface passivation layer. Afterwards, the processed mirrors were shipped back to GSFC where surface and optical metrology were performed.

We found that several plasma processing parameters have a significative impact on the reflectivity of Al mirrors, although for some the impact was difficult to quantify. An example of the latter is that we learned that reflectivity gain seems to improve with the number of times the chamber was run with fluorine, implying the chamber needed to be "conditioned" to remove oxygen from the walls. The process parameter optimization was pursued to minimize the O content of the protective AlF_3 layer, as FUV (100–200 nm) reflectivity is inversely proportional to the oxygen content within the AlF_3 layer. The success of this initial optimization is shown in Fig. 2, which shows steps in the reduction of oxygen content of films, and consequent enhancements of FUV reflectivity, reaching samples with no detectable O content and reflectivity ~54% at 105 nm and reflectivity over ~83% at wavelengths longer than 115 nm. In this regard, we believe we have met the main criterion for success in Year 1.

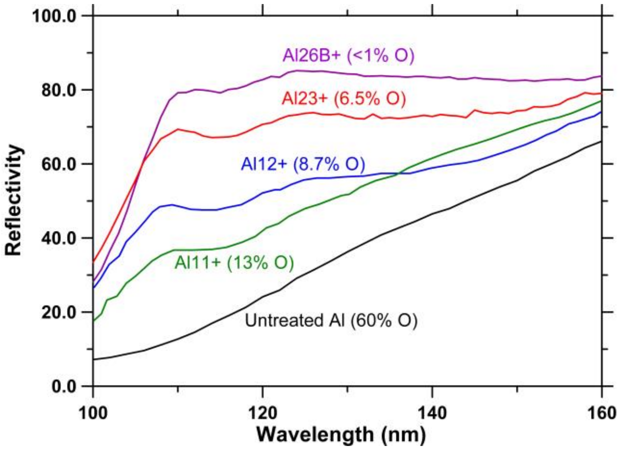


Fig. 2. *This graph illustrates the results of oxide-removal and passivation of bare Al coatings in the LAPPS facility at NRL. The latest data (purple curve) show a remarkable achievement of reaching an average FUV reflectance close to 80% and no detectable O content for a sample that was fully oxidized before treatment. The O content of samples was determined from either XPS or ellipsometry.*

Beyond the obvious improvement in coating quality evidenced in Fig. 2, we have also demonstrated the ability to control AlF_3 properties by varying process parameters. We identified three critical process parameters: process time, ion energy at the substrate, and SF_6 flow into the reactor. Control over these parameters results in the ability to control AlF_3 film thickness and surface roughness. For instance, Fig. 3 shows the effect of ion energy and SF_6 concentration on average reflectivity above 115 nm and on AlF_3 thickness. While there might still be room for further improvements, we are confident that we have identified a parameter space that will allow for optimized Al mirrors through manipulation of the aforementioned parameters.

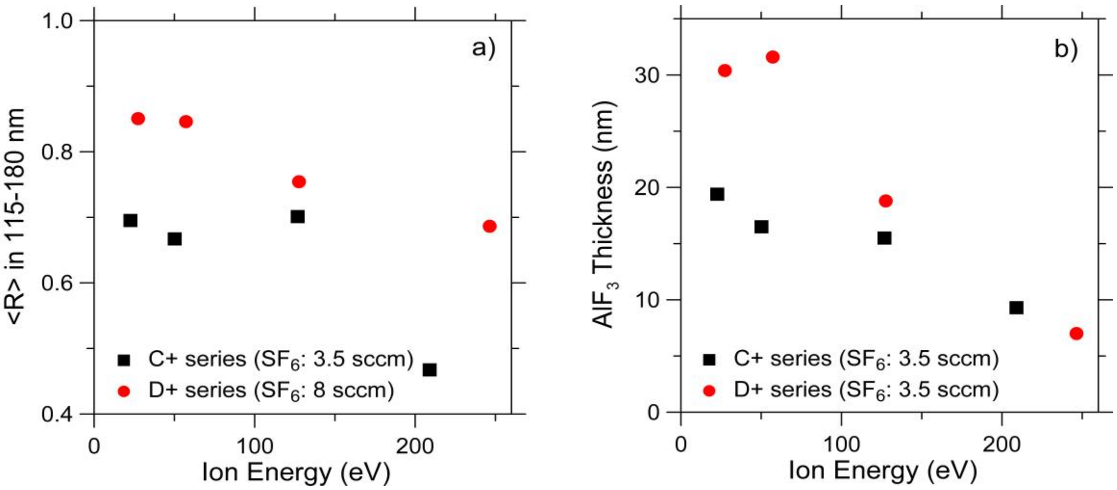


Fig. 3. Left: *Average reflectivity <R> from 115-180 nm at high (8 sccm) and low (3.5 sccm) SF_6 flows for ion energy varied between nominally 25 eV and 250 eV.* Right: *The resulting change in AlF_3 film thickness for the same set of ion energies. Note that high ion energy leads to thinner films (≈7-10nm) but degraded <R> due to increased surface roughening and oxidation. Also note that the lower SF_6 flow (corresponding to lower F atom surface flux) leads to reduced film thickness overall. (Figure reproduced from [18]).*

Presently, mirror samples treated with the LAPPS plasma consistently demonstrate R~85-90% at the H Lyman-α (121.6 nm) wavelength, an important astrophysics diagnostic often used as a reference, and R~40-55% at 105 nm. Indeed, in one sample we reached R~91% at 121.6 nm (Fig. 4, left, red curve), paired with the highest reflectivity reported to date in the literature at the same wavelength [6]. The reflectance of the latter curve was measured over the full range of interest (90-2500 nm) and is shown in Fig. 4, right (note the logarithm scale). The average full-range reflectivity for this sample is ~94%.

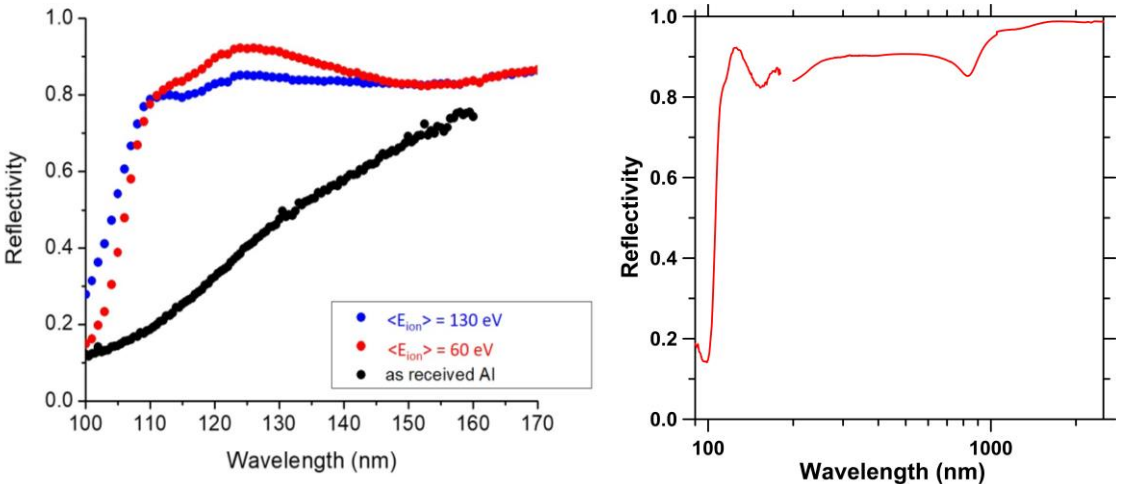


Fig. 4. Left: *Reflectivity measurements illustrating the effect of varying ion energy impacting the Al surface. Note that for the 60 eV case the H-Lyman-α reflectivity is ~91%.* Right: *Reflectivity of the 60-eV curve in the full range of interest, 90-2500 nm, in log scale.*

The main findings obtained in Year 1 were published in [18]. One aspect of this research that was put on hold in Year 1 (due to lack of the proper equipment) was the exposure of these samples under controlled environmental conditions to demonstrate the survival of these coatings under varying relative humidity. However, an environmental control system was procured and humidity-controlled tests were carried out during Year 3 as described below.

<u>Year 2:</u>

We began to explore the effects of LAPPS reactor configuration and plasma uniformity on the uniformity of the dielectric protective layer. While this work was interrupted by the COVID-19 pandemic, preliminary results indicated a 20% non-uniformity in film thickness with respect to the average thickness for a 51 mm × 51 mm Al sample, as depicted in Fig. 5. Importantly, this preliminary work demonstrated the sensitivity of film thickness to variations in plasma density. Here it was found that a factor of 10 variation in plasma density along the axis (for this sample, the beam axis goes from the upper left corner to the bottom right corner of the sample) of a cylindrically shaped electron beam resulted in a variation in AlF_3 film thickness between 40 nm and 25 nm for identical ion energy, process time, and SF_6 flow. This result illustrates that AlF_3 film thickness is highly sensitive to ion flux. As this is the first and only sample in which uniformity was analyzed, we foresee improvements by optimizing the plasma configuration within the reactor.

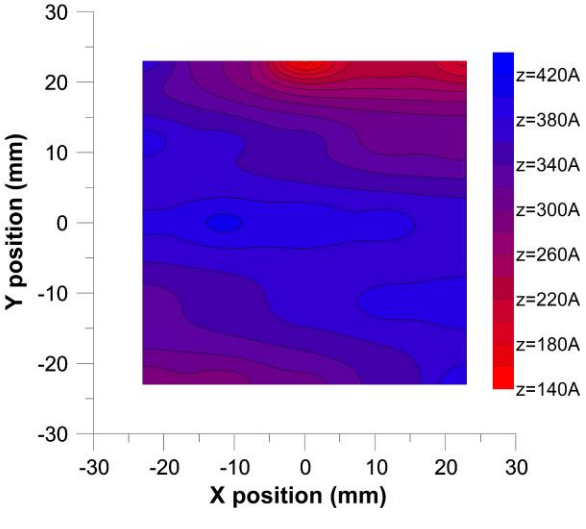


Fig. 5. *Thickness uniformity of protective AlF_3 layer over a 51×51 mm^2 Al sample determined by spectroscopic ellipsometry.*

A second accomplishment is the fact that NRL is ready to transfer the low-temperature plasma fluorination process from the old high-vacuum reactor to a new ultra-high vacuum (UHV) reactor. The goal of this transition is to provide a chamber with much lower base pressure to enhance the quality of processed Al mirrors. NRL also completed the purchase of a new in-situ spectroscopic ellipsometry system designed for real-time in-situ measurements of film thickness and refractive index of fluoride coatings on Al films during plasma processing (Fig. 6). NRL already received all the necessary parts and the ellipsometer from the vendors, so assembly will proceed in the coming months. This capital equipment was purchased using NRL funds.

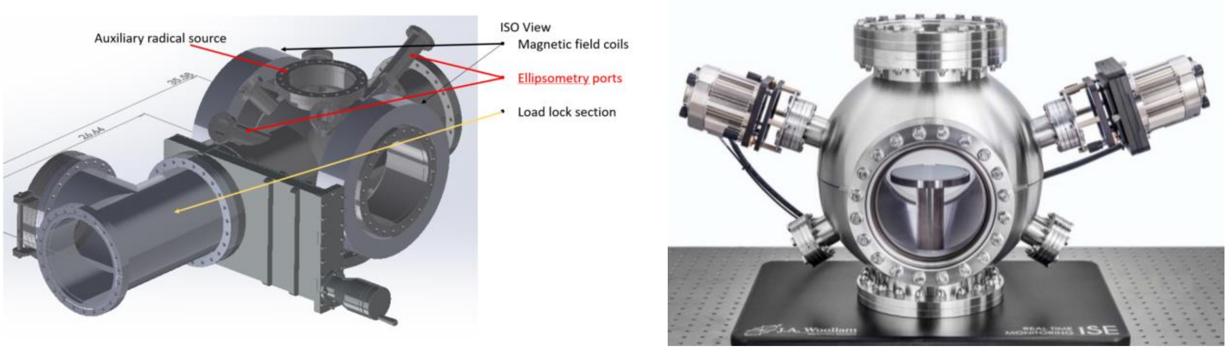


Fig. 6. Left: *Isometric view illustrating the design of the new UHV LAPPS chamber with ports for in-situ ellispometry and a load lock for maintaining chamber cleanliness.* Right: *The J.A. Woolham in-situ ellipsometry system purchased for this work.*

Year 3:

This final year of the program has seen important progress despite the difficulties associated with the COVID-19 pandemic. Recently NRL completed construction of the previously described UHV LAPPS system that includes a load lock to maintain chamber cleanliness, and an in-situ ellipsometry feature. The ellipsometer allows real-time monitoring of optical properties of a surface during plasma treatment. The completed reactor is shown in Fig. 7. To date, reactor plasma properties have been characterized and demonstrate excellent uniformity across the 150-mm sample stage. The standard deviation of the plasma density across the wafer stage was 2%.

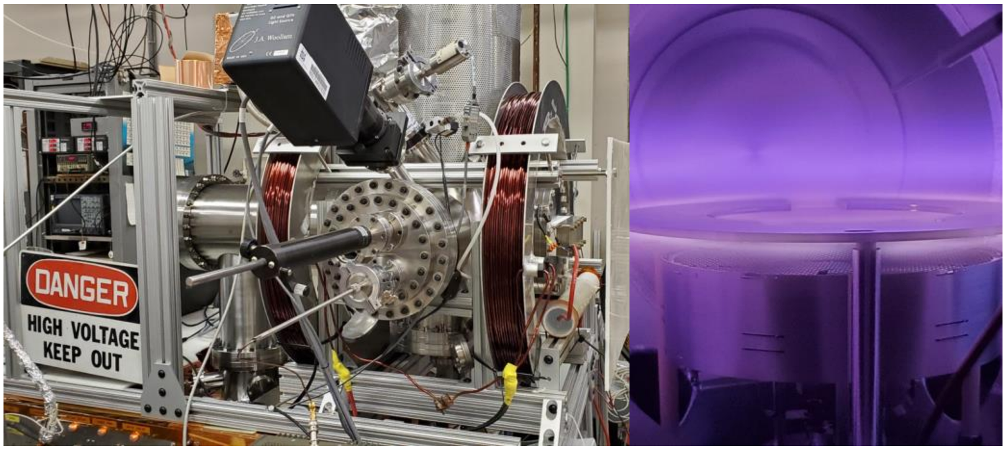


Fig. 7. Left: *Exterior view of the completed UHV LAPPS chamber with in-situ ellipometry and a load lock for maintaining chamber cleanliness.* Right: *Interior view of electron-beam generated plasma over the 150-mm diameter processing stage.*

The in-situ ellipsometry tool was also installed and tested. Testing was performed by etching a 100-mm-diameter SiO_2 wafer and monitoring the thickness change of the SiO_2 layer in real time. This system was able to resolve changes in SiO_2 thickness of < 0.5 nm indicating the ellipsometer should be a highly accurate tool for monitoring fluoride film thickness during processing.

A substantial amount of surface analysis work was also performed on the AlF_3-coated Al mirror samples during Year 3. XPS depth profiling was performed at NRL to determine the film composition as function of depth into the material. An exemplary dataset is shown below in Fig. 8. The data indicate the fluoride film is stoichiometric AlF_3 with surface carbon and oxygen contamination that originates from oxygen diffusion into the material from ambient. Oxygen remaining from the Al native oxide layer would manifest as increased O content near the AlF_3/Al interface. That is not observed, and thus indicates full removal of the native oxide layer via the plasma treatment process. This characterization effort was an important part of a recent publication on this coating method detailed in [18]

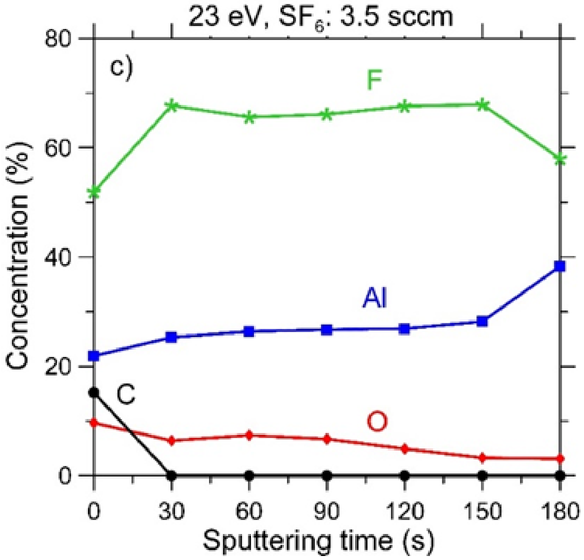


Fig. 8. *XPS depth profiling of an AlF_3/Al sample treated with 23-eV ions in LAPPS for 960 seconds. The elements present at the film surface are on the left of the plot and the elemental constituents near the AlF_3/Al interface are at the right of the plot. The profile shows the stoichiometric concentrations of Al, F, O, and C as a function of depth throughout the film. The data indicate the film is mostly stoichiometric AlF_3 with surface C and O contamination that originates from oxygen diffusion into the material from ambient.*

In addition to the XPS work, XRR measurements were performed to independently confirm the AlF_3 thickness of selected samples, where film thickness was previously found using ex-situ ellipsometry. These measurements confirmed the film thicknesses, giving us greater confidence in the ellipsometry measurements. Beyond these XRR measurements, samples were also shipped to Brookhaven National Laboratory for measurement using their synchrotron light source. This allowed remote characterization of samples during the COVID-19 pandemic, when laboratory work was not possible at either NRL or GSFC. These synchrotron measurements used GISAXS and GIWAXS to further probe the topology of the film surface. They revealed the AlF_3 coatings to be conformal to underlying structure of the Al surface on which the passivation process was performed. This important work was performed by Jeffery Woodward, a National Research Council Postdoctoral Research Associate residing at NRL.

The conformality of the AlF_3 on top of the underlying Al layer, and the crystallinity of the latter was later confirmed by transmission electron microscopy (TEM) performed by Andrew Lang at NRL. The fluoride layer thickness was confirmed to be 28 nm in thickness on top of an 85-nm-thick Al layer. This agreed very well with in-situ ellipsometry measurements taken within the LAPPS reactor, giving high confidence to the in-situ ellipsometry measurements going forward. The results of this study are shown below in Fig. 9.

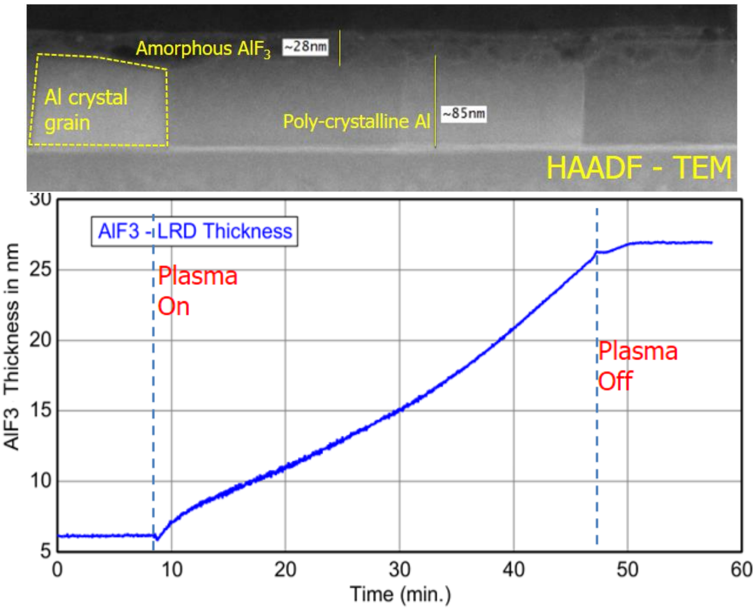
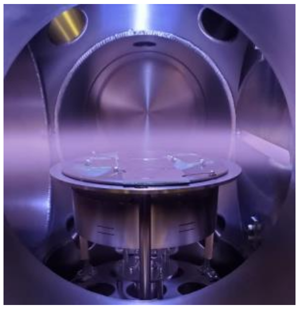


Fig. 9. Top: *TEM profiling of an AlF$_3$/Al sample showing a 28-nm-thick AlF$_3$ layer on top of 85 nm of Al. Al crystal grain size is observable and shows crystal size to be on the order of 90 nm.* Bottom: *In-situ ellipsometry for the same sample showing plasma treatment generates a 27.5 nm thick AlF$_3$ layer, in excellent agreement with the TEM results.*

As part of the final year of this effort, a uniformity study was also undertaken. This consisted of processing several 25-to-50-mm-diameter coupons on the LAPPS processing stage to simulate the treatment of a larger 150-mm-diameter mirror. These samples were treated and then examined ex-situ using ellipsometry mapping as well FUV reflectance. Figure 10 (left) shows the experimental setup, the center is a table of AlF$_3$ layer thickness at various stage locations. Figure 10 (right) shows a graph of the average FUV reflectance and reflectance non-uniformity for the samples treated. The samples were uniform to within about 15% across the 150-mm sample stage for AlF$_3$ film thickness. This translated to a reflectance uniformity of < 3% for wavelengths between 110 nm and 200 nm, and < 1% above 200 nm. This uniformity could likely be further improved with the inclusion of standard plasma processing procedures such as stage rotation, and chamber design to optimize gas flow over the substrate.



Chamber location	Average thickness
top	24.6 ± 1.37 nm
middle	25.5 ± 1.74 nm
right	23.9 ± 2.54 nm
left	22.1 ± 0.59 nm
bottom	24.9 ± 1.37 nm

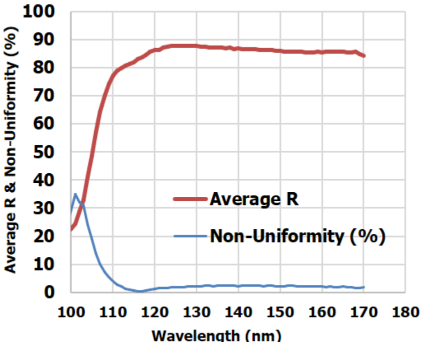
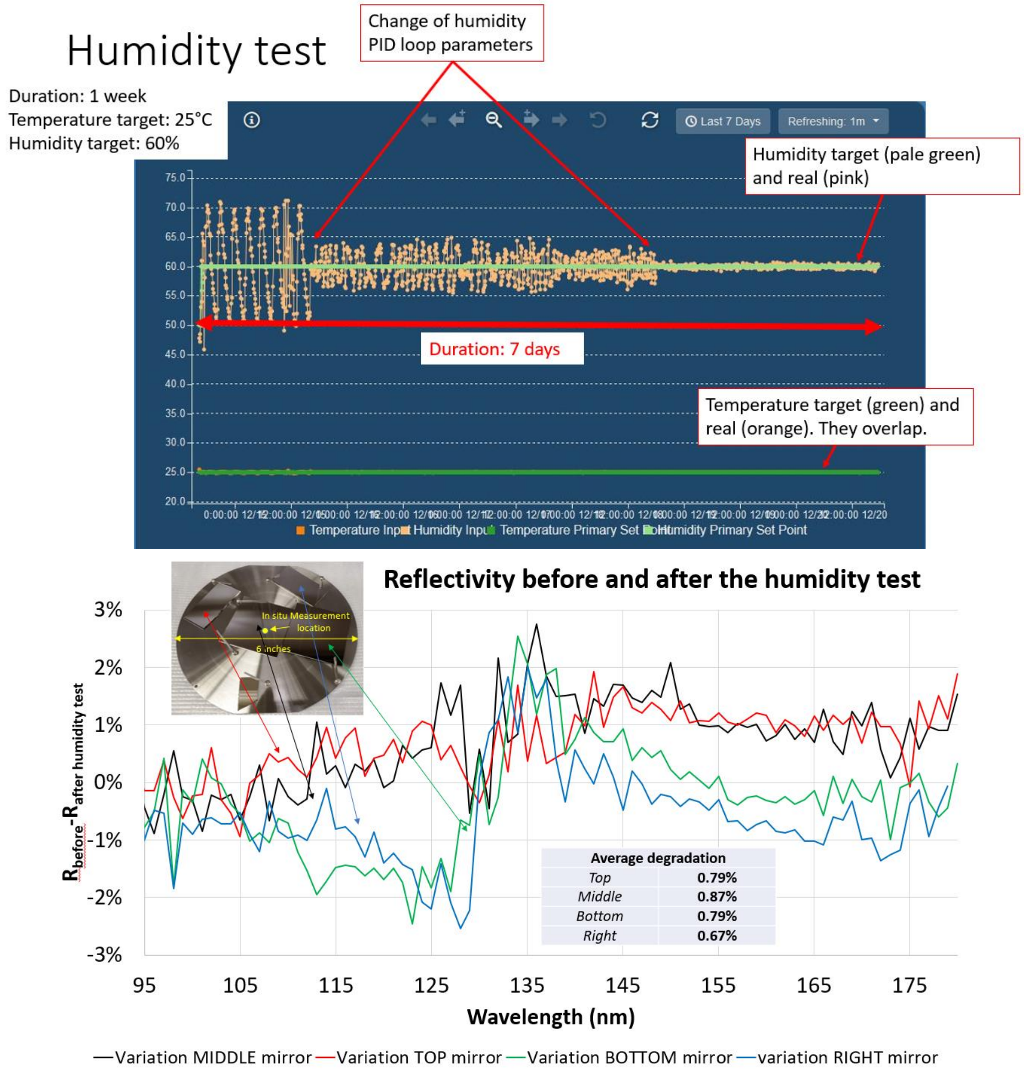


Fig. 10. Left: *Image of the processing setup for uniformity of film thickness across a 6 in. diameter sample stage.* Center: *AlF$_3$ film thickness at the chamber locations tested using ex situ elliopsometry mapping.* Right: *The average reflectivity of all the treated samples together with the reflectance non-uniformity of the samples in the FUV.*

After an environmental control system from Associated Environmental Systems was purchased, humidity-controlled tests were carried out with the samples shown in the inset of Fig. 11 (bottom). This system is equipped with a single-stage refrigerator that provides a temperature range from -20°C to 94°C, and the humidity control can be adjusted within the range from 10% to 95% RH. Figure 11 (top) shows a screenshot of the experiment in which the samples shown in Fig. 11 (bottom) were kept for a week at a constant temperature (25°C) and humidity (60%). This is a very demanding test, and sufficient to assess stability. All samples survived the humidity test, and the average FUV reflectance degradation was < 1%, which is within measurement uncertainty. With this experiment we demonstrated the environmental stability of Al mirrors protected with AlF_3 that were developed under this SAT program.



Average degradation	
Top	**0.79%**
Middle	**0.87%**
Bottom	**0.79%**
Right	**0.67%**

Fig. 11. Top: *Screenshot of the humidity test experiment for the samples shown in the inset in the bottom picture. Setpoints were 60% humidity, 25°C temperature, and one-week duration.* Bottom: *The differences in FUV reflectivity before and after the humidity tests. Differences are averaged in the 95-180 nm range and shown in the inset table.*

The etching of fluoride films (MgF_2 and AlF_3) was examined using the new UHV LAPPS reactor. When the films were exposed to the LAPPS plasma, in Ar/SF_6 mixtures, there appears to be an initial etching phase to the treatment where material is removed, particularly in the case of MgF_2 films. After about 20 seconds of plasma exposure, about 2 nm of material were removed. This resulted in increased FUV reflectivity at wavelengths <150 nm. At wavelengths <120 nm the increase was particularly pronounced, with a notable reflectivity of over 93% at 121 nm. After the initial 20 seconds etching phase, the material seemed to thicken again. This was particularly noticeable in the case of the AlF_3 films where this film growth phase nearly restored the AlF_3 layer to its original thickness. Nonetheless, the AlF_3 film showed enhanced reflectivity as well at wavelengths >125 nm and <160 nm. These preliminary results are very promising and are shown below in Fig. 12.

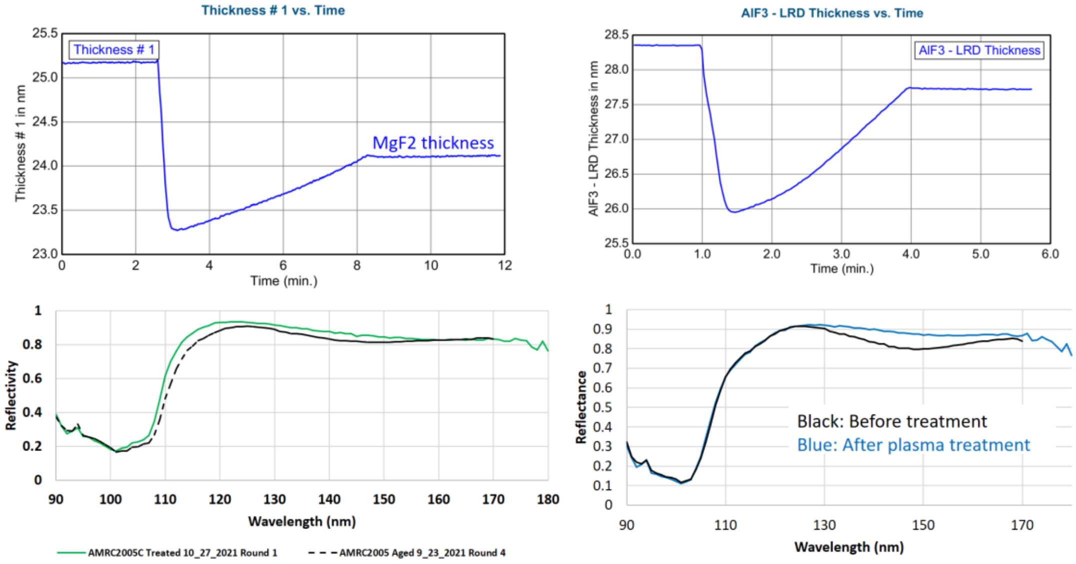


Fig. 12. Top left: *MgF_2 thickness as a function of time during plasma treatment; plasma off at 8 min.* Top right: *AlF_3 thickness as a function of time during plasma treament; plasma off at 4 min.* Bottom left: *MgF_2-coated Al reflectivity curve before (black) and after (green) plasma treatment.* Bottom right: *AlF_3-coated Al reflectivity curve before (black) and after (blue) plasma treatment.*

As a final effort, a polarization analysis was done with the samples shown in the round inset of Fig. 11. This work evaluated the impact of mirror coating on the diattenuation and retardation of polarization states in the UV/Vis/NIR. Therefore, the R_s and R_p components of coatings shown in Fig. 11 were measured in the 200-2500-nm range at a 12° angle of incidence (AOI). As reference, LUVOIR required the AOI at any given optical surface to be less than 12° to mitigate polarization aberrations. From the experimental values of R_s and R_p, the normalized diattenuation $(R_s-R_p)/(R_s+R_p)$ was obtained. Since the phase retardation could not be measured at 12˚ incidence with the available ellipsometer, high-fidelity mirror-coating models were built using optical constants derived from experimental data to predict the retardation in the spectral range of interest. We found that for the samples in Fig. 11, the average phase retardance is 0.82° and the average calculated coating-induced polarization is 0.11% at AOI=12° in the 200-3000 nm spectral range. These values are well below current requirements for coronagraphy. Figure 13 shows the calculated retardance (left) and normalized diattenuation (right) of the Al/AlF_3 mirrors developed in this research effort, along with other coating technologies (Al/LiF and Al/MgF_2) for comparison purposes. These tasks were performed by Gabriel Richardson, a GSFC Pathways intern.

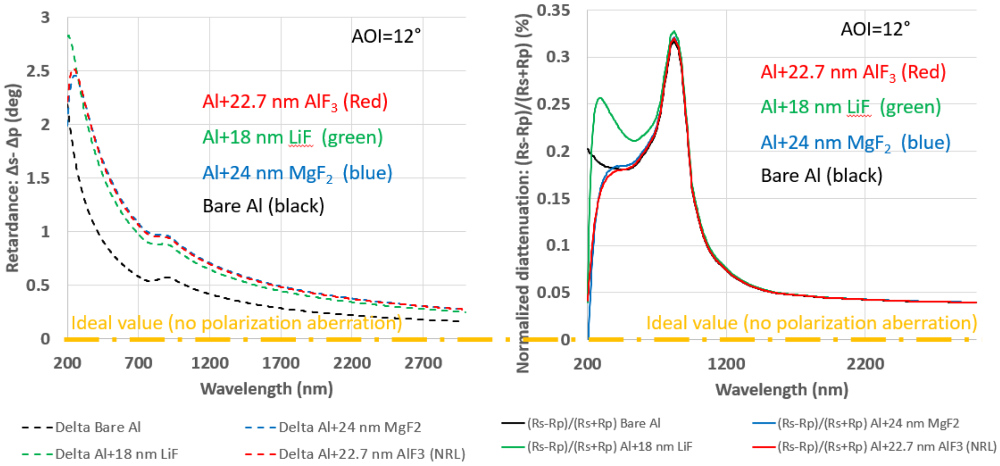


Fig. 13. *Effect of coating-induced polarization (left) and calculated phase retardation (right) for different AOI for the mirror in Fig.4 (right). The solid curves are experimental data, and the dashed lines are models.*

Finally, several efforts were made to extend the visibility of this research among the scientific community: A journal article [18], two SPIE proceedings [19, 20], and a NASA Technology Highlights entry [21] were published in which the main findings from this effort are summarized. In addition, contributed talks were presented at the SPIE Optics and Photonics [19] and Mirror Tech Days [22] conferences, and a poster was presented at the Society of Vacuum Coaters meeting and at the SPIE conference [23, 20]. A New Technology Report (NTR) was submitted and accepted and, as a result, a patent application (FAR-ULTRAVIOLET ALUMINUM MIRROR PASSIVATION PROCESS USING ELECTRON BEAM GENERATED PLASMA) was submitted. An APRA proposal with substantial follow-up content (PI: D. Boris, NRL) entitled "Plasma enhanced atomic layer deposition for precision control of thickness and stoichiometry in aluminum mirror coatings for next generation astrophysical telescopes" (FY 2021-2024) was selected for funding under the call NNH20ZDA001N-APRA 2021. Finally, JPL researchers with expertise in coronagraphy recently contacted us to inquire about the mirrors developed under this grant to reproduce some of the coatings in a model for the LUVOIR-B concept mission.

Summary

At the conclusion of this three-year effort, we successfully met nearly all of the goals laid out in this SAT research program. We have taken the concept of using large-area electron beam generated Ar/SF_6 plasmas as a means of removing the native oxide from Al surfaces and simultaneously fluorinating that surface from a proof-of-concept stage to a repeatable coating process that demonstrated high reflectivity and environmentally stable FUV mirrors at least on the scale of 150 mm substrates. With the in-situ ellipsometry techniques used in this work, combined with the surface science performed by our collaborators, we attained a robust understanding of the physical mechanisms behind this process. As such, we are confident that this process can be successfully scaled to larger areas with additional engineering of larger-scale plasma-processing equipment.

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