At-wavelength Reflectance Measurements for Aluminum Mirrors in the Far Ultraviolet

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ABSTRACT

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Aluminum is a useful material for broadband mirrors due to its high reflectance across many wavelengths, including the far-ultraviolet. Its high reflectance in the far-ultraviolet makes it a promising candidate for use in the next generation of space telescopes. However, oxidation of aluminum poses a problem because it dramatically reduces aluminum reflectance in the far-ultraviolet. One potential solution is thin film fluoride coatings on the aluminum mirrors, though it is difficult to test these mirrors for FUV reflectance as FUV light is difficult to produce and is absorbed in the atmosphere. Here a method is developed for obtaining at-wavelength reflectance measurements using a plasma as the light source, a monochromator, and vacuum chamber. Reflectance was measured, though the values were lower than expected. The method requires further developement to acquire reliable measurements.

Keywords: FUV reflectance, aluminum mirrors, LUVOIR, thin film

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Contents

Table of Contents vii

List of Figures ix

List of Tables ix

1 Introduction 1
- 1.1 Background 1
- 1.2 Previous Work 2
- 1.3 At-wavelength Measurements 4

2 Methods 7
- 2.1 Creating Samples by Thermal Evaporation 7
- 2.2 Characterization of samples 9
- 2.3 Reflectance Measurements 9
  - 2.3.1 Monochromator Setup 10
  - 2.3.2 Taking Measurements 12
  - 2.3.3 Beam Profile 16
- 2.4 Silicon and Silicon Nitride References 18

3 Results and Conclusion 19
- 3.1 Results and Discussion 19
- 3.2 Conclusion 21
- 3.3 Future Work 21

Bibliography 23

Index 25

List of Figures

1.1 Calculated reflectance of common mirror materials 3
1.2 Aluminum and aluminum oxide reflectance 4

2.1 The evaporation chamber 8
2.2 The monochromator 10
2.3 The O-chamber 11
2.4 Octopus program 13
2.5 Sample stage alignment 14
2.6 Lithium fluoride coated aluminum reflectance 15
2.7 Beam profile without slit in chamber 16
2.8 Beam profile with slit in chamber 17

3.1 Calculated reflectance of silicon nitride wafer 20

List of Tables

3.1 Reflectance measurements for reference mirrors 20

Chapter 1

Introduction

The focus of this thesis is advancing the technology needed to provide valuable reflectance data to support ongoing research at Brigham Young University. This is done by providing a method for measuring reflectance in the far ultraviolet range. In this introduction I will discuss the motivation behind protected aluminum mirrors, the work that has been done with aluminum mirrors by other students, and how that work can be supported by at-wavelength measurements.

1.1 Background

We learn about our universe largely from what we can observe here on earth or from space-based observatories. We observe different kinds of radiation that propagate through space and bring us information about stars, planets and more. We can learn more about the universe as we increase our ability to detect this radiation and measure different kinds of radiation coming from the universe.

The ability to see more of the universe is a high priority in the future of space exploration. The National Academies for Sciences, Engineering, and Medicine published "Pathways to Discovery in Astronomy and Astrophysics," in 2020 in which they defined several goals for space exploration and plans to achieve those goals in the coming decade. One of these plans includes a need for measurements in the far ultraviolet (FUV) range [1], down to about 90nm. This is a low enough range to see the entire hydrogen Lyman series (91.2 nm – 121.6 nm).

One plan for fulfilling this need is LUVOIR, the Large Ultraviolet Optical Infrared Surveyor. As its name suggests, LUVIOR is a multi-wavelength space telescope concept being developed by NASA, and it will include the capability of getting measurements in the FUV range. This objective requires a broadband mirror coating with high-reflectivity in the far-UV [2].

The most promising material is aluminum. Aluminum is one material often used in mirrors due to its high reflectance in the visible range; however, unlike other materials commonly used in mirrors, it maintains a high reflection into the FUV range [3](Fig. 1.1) While they provide the needed reflection, aluminum mirrors are not without problems to overcome. Aluminum oxidizes quickly in earth's atmosphere, and with even a thin top layer of oxidation it rapidly loses its FUV reflectance (Fig. 1.2) [4].

1.2 Previous Work

Much research has been done in recent years to provide a solution to this oxidation problem and produce quality aluminum mirrors for use in telescopes that can obtain FUV measurements. Some research that has been done relates to possible methods of storing aluminum to slow oxidation. It has been shown that storing aluminum in certain environments, such as liquid nitrogen, hexane, and dry ice, can retard oxidation growth by as much a factor of 500 [6].

Another solution is the use of thin fluoride coatings deposited onto the aluminum to protect it from oxidation. Magnesium fluoride (MgF_2), aluminum fluoride (AlF_3), and lithium fluoride (LiF) are all potential candidates as they have low absorption in the FUV [7].

Magnesium fluoride has been used as a protective coating, but it begins to absorb at wavelengths below 115nm [8]. Work with aluminum fluoride showed that a 2.4nm coating can significantly

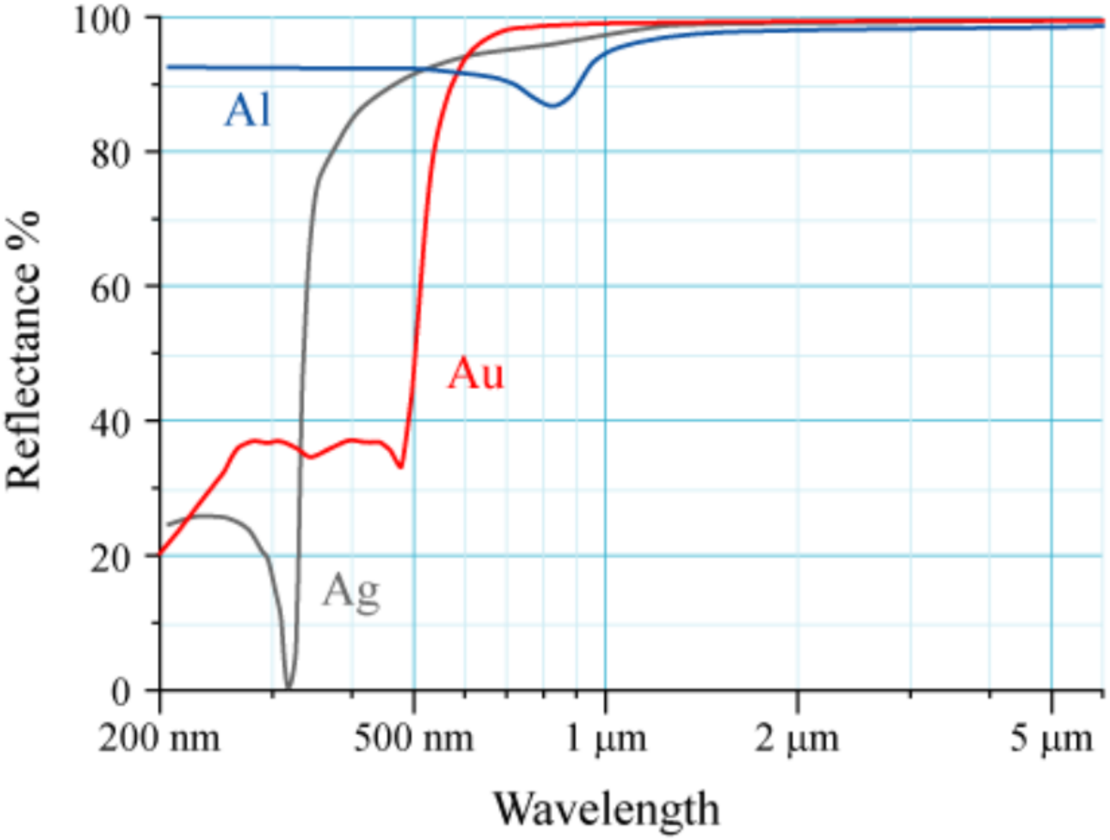


Figure 1.1 Calculated reflectance of common mirror materials. Aluminum, silver, and gold, are regularly used in mirrors due to their high reflectance in the infrared and portions of the visible range. Of those materials, only aluminum maintains a high reflectance into the FUV range (see also Figure 1.2). Figure copied from reference [5].

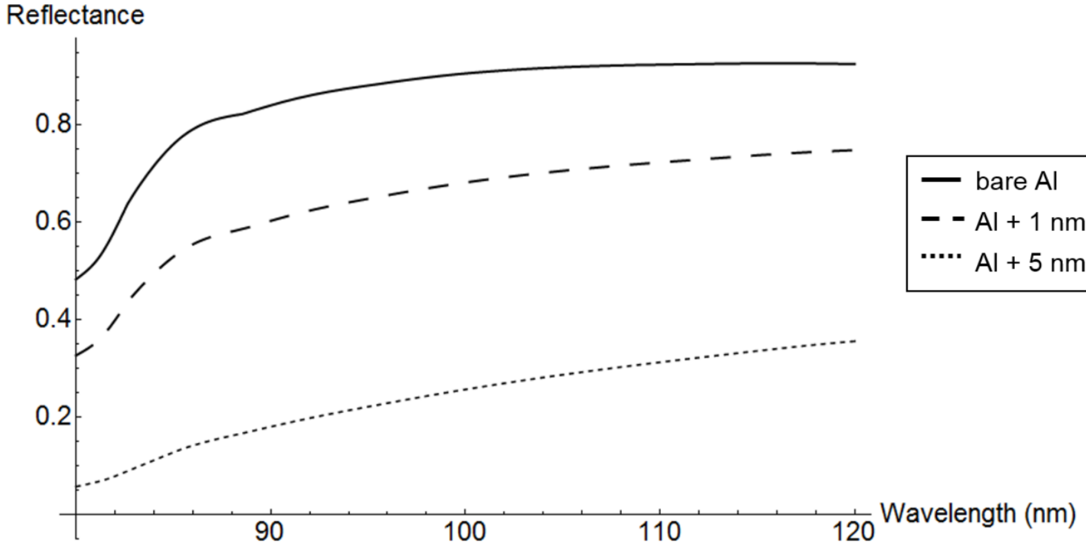


Figure 1.2 Reflectance of aluminum and aluminum oxide in the FUV range. Aluminum maintains above eighty percent reflectance down to 90nm. However, even small amounts of oxidation will significantly reduce reflectance. Figure copied from reference [4].

reduce oxidation [9], while a 9nm coating seems to stop oxidation altogether [4]. Lithium fluoride coatings have also demonstrated the necessary optical performance, but lithium fluoride is very hygroscopic, meaning it attracts water out of the air. This causes complications in humid environments. Both the degradation of the aluminum (through oxidation) [10] and the fluoride layer itself [11] have been studied.

1.3 At-wavelength Measurements

All technologies developed and used by NASA must ascend a technology readiness ladder (TRL) [12]. There are nine levels describing various competencies a new technology must achieve before it is ready to be implemented. These are crucial for NASA. As an example, the lowest TRL requires just an observation and report of basic principles. The TRL progresses as a technology concept is formed and its critical function is experimentally tested. The component must then be tested in a laboratory and then relevant environment. As it progresses even further, the whole system must be tested in a laboratory and then space environment. The highest technology readiness level is achieved when the actual system is proven through successful mission operation.

The significance of this in relation to the research done on aluminum mirrors is that showing a decrease in oxidation of aluminum is only a first step. To further develop the technology the research must be supported by at-wavelength reflectance measurements to see if it performs as expected in a relevant environment, or a laboratory environment that mimics how the mirror will actually be used. It may also bring up other questions that were not foreseen. For example, it is seen that when lithium fluoride picks up water from the air it can change the surface texture, and that can eventually lead to oxidation of the aluminum [11]. But how might the changing surface of the lithium fluoride itself affect reflectance?

My goal is to develop a method for reliably measuring the reflectance of various aluminum mirrors, including unprotected aluminum and coated aluminum, at a variety of wavelengths in the FUV. This will be an important step in continuing research on FUV mirrors.

Chapter 2

Methods

This chapter describes the methods we used for obtaining FUV reflectance measurements of aluminum mirror samples. I first describe the fabrication and characterization of samples. I then describe our setup using a vacuum monochromator to measure the reflectance of FUV light off these samples. Finally, I discuss some methods used to ensure accurate measurements.

2.1 Creating Samples by Thermal Evaporation

As our goal is to provide at-wavelength measurements for various aluminum mirrors, the first step is to create samples that we can measure. These samples should be similar to those used in previous research–these mirrors are typically a substrate with one or two thin layers deposited on top. For example, a silicon or silicon nitride on silicon substrate, a thin layer of aluminum, and then a thin layer of a metal fluoride. Because these are the same kind of samples used in previous research, the methods for creating them are already well documented. Hart [6] discusses methods for just aluminum deposition, while Lewis [11] and Davis [4] go into detail on lithium fluoride and aluminum fluoride deposition respectively.

The thin film mirrors are fabricated through thermal evaporation. In summary, a silicon or

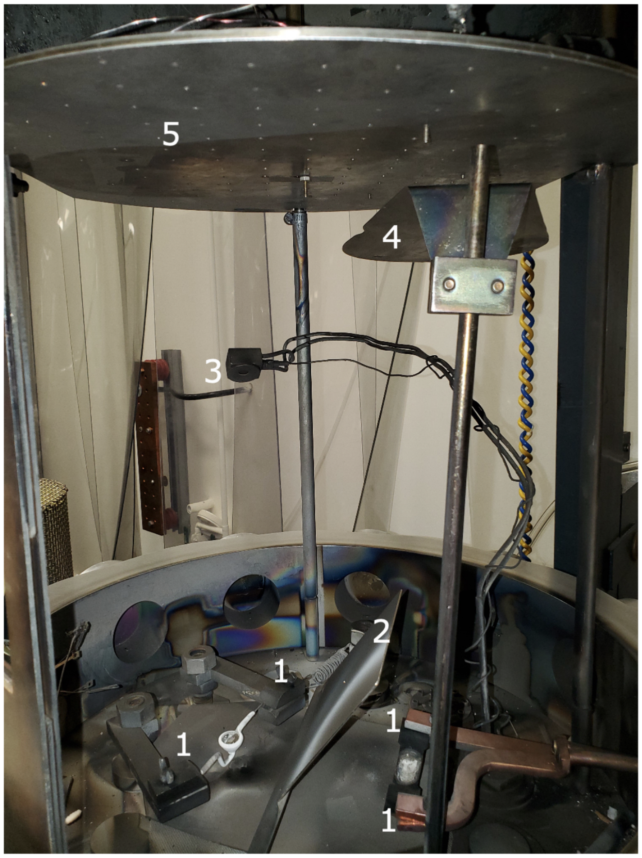


Figure 2.1 The evaporation chamber. 1) Connection points and boats where the materials to be evaporated are held. 2) Shutter used to control which material is being deposited. 3) INFICON quartz thickness monitor. 4) Shutter used to cover and expose samples. 5) Board where substrate is secured. Photo and labels from [6].

silicon nitride coated silicon wafer is held under vacuum. Aluminum is then thermally evaporated onto the substrate. When the desired amount of aluminum has been deposited, a shutter can switch to deposit another material, such as a metal fluoride if desired. Once again, after the desired amount has been deposited, the shutter can be closed to stop deposition. Once the sample is taken out of vacuum it must be quickly measured or stored, due to quick degradation of aluminum mirrors. The thermal evaporator is shown and labeled in Fig. 2.1.

2.2 Characterization of samples

Another step that must be taken before measuring the reflectance of the sample is characterization of the sample. This is an important step because the reflectance data doesn't tell us much else about the sample, and it is harder to understand if it can't be compared. For example, when we measure the reflectance of a lithium fluoride coated aluminum mirror over time, we might wonder if the changes in reflectance are caused by increasing oxidation of the aluminum, changes in the surface of the lithium fluoride, a combination, or something else.

Previous research has also documented various methods of characterization for these mirrors. These methods include the use of ellipsometry, atomic force microscopy, and scanning electron microscopy with energy dispersive X-ray spectroscopy.

Ellipsometry is useful in determining the thickness of the layers of the mirror, including the thickness of oxidation on the aluminum and the thickness of the metal fluoride coating. For more information on ellipsometric characterization, see references [4, 6, 11].

Atomic Force Microscopy (AFM) is useful in analyzing the surface texture of the mirror. This is especially relevant with LiF coated mirrors, as the surface can change as the LiF absorbs moisture from the atmosphere. For details on AFM characterization, see reference [11]

Scanning electron microscopy (SEM) with energy dispersive X-ray spectroscopy (EDX) may also be helpful. It allows us to determine the chemical content of composition of the samples and then through a program connected with the microscope, analyze that data taken. This is mostly used to confirm results from ellipsometric characterization. For more details see references [4, 11].

2.3 Reflectance Measurements

Once a sample mirror has been created and characterized, we can then measure its reflectance. Some of the methods for measuring reflectance were already established, described in the subsection

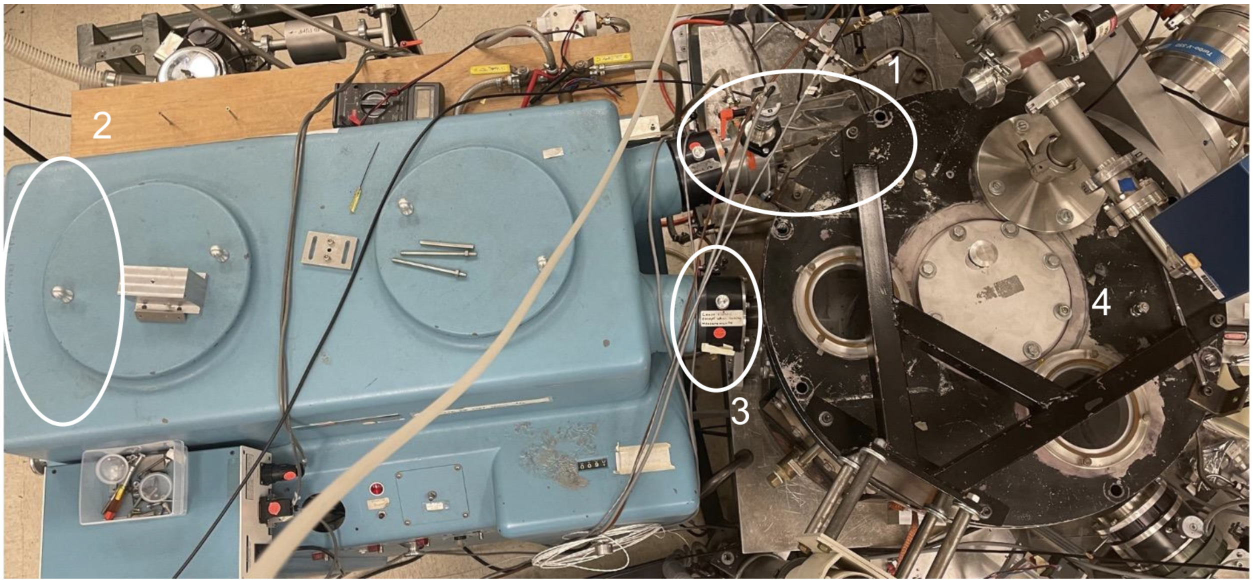


Figure 2.2 The vacuum monochromator system. 1) Gas line and high voltage for starting a plasma. 2) Light from the plasma shines into the blue chamber and reflects off a grating located here. 3) The grating is adjusted for the desired frequency to shine through this slit. This also connects the two chambers. 4) The O-chamber. Light enters here through the slit, and this is where the sample mirror is placed and reflectance is measured.

on the monochromator setup (2.3.1), while others were developed by Devin Lewis and myself, described in the subsection on taking measurements (2.3.2).

2.3.1 Monochromator Setup

The vacuum monochromator is shown in Fig. 2.2. The whole system, the monochromator and the O-chamber, must be under vacuum to get the measurements, because the air will interfere with the FUV light and produce inaccurate reflectance data. We do this with a roughing pump and turbo pump attached to both the monochromator section and the O-chamber. The two chambers are independent until the slit is opened. The system reaches a pressure on the order of 10^{-6} Torr.

Once the chamber has reached a sufficiently low pressure we introduce a gas to start a plasma at the site shown in Fig. 2.2. We use argon for its peak at 91.9 nm. The gas is let in through a metering valve to reach the desired pressure of 0.25 Torr, at which point a high voltage source supplies a

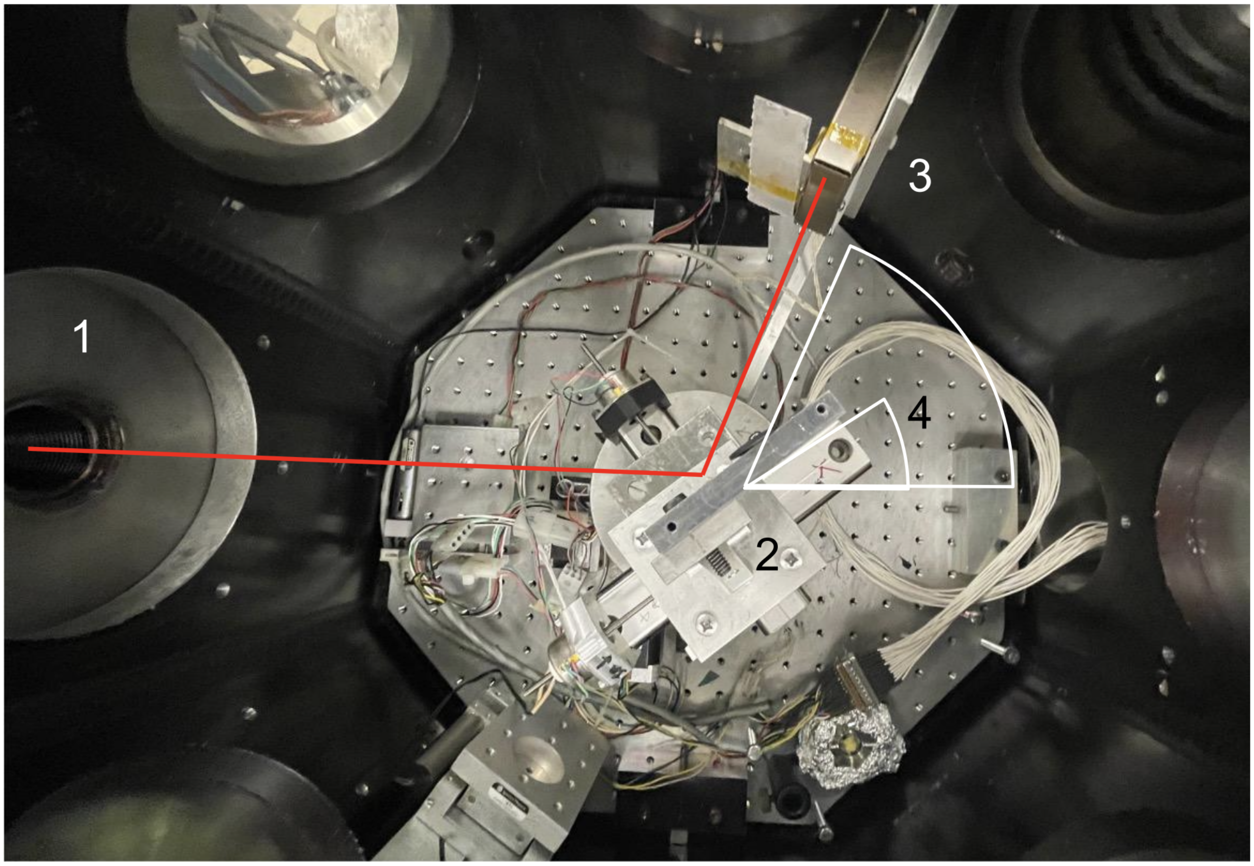


Figure 2.3 Top view of the O-chamber. 1) Light from the monochromator shines in through this slit. 2) This is the sample stage. Samples mounted here can be rotated to reflect light at various angles. 3) The detector can rotate to pick up the light reflected off the samples. 4) The angle of the sample stage and detector from 0 degrees.

sufficient voltage (up to 1000 volts) to start the plasma. We wait several minutes for the plasma to stabilize before taking measurements.

The light from this plasma enters the monochromator and reflects off a grating at the back of the chamber. The grating separates the frequencies of the light, and can be adjusted so that the desired frequency shines through a slit into the O-chamber. Any wavelength corresponding to an emission peak of some gas can be used. As mentioned we used 91.9 nm, since argon has a peak at this wavelength. The interior of the O-chamber is shown in Fig. 2.3

The sample stage is positioned in the middle of the O-chamber. This sample stage has two motors that can move it in the horizontal plane to center the sample in the beam coming from the monochromator. This also allows us to move the sample out of the way to measure the intensity of the light without reflectance. There is a third motor on the sample stage that allows us to rotated it so we can reflect light off the sample at a variety of angles. The detector also has a motor allowing it to rotate so it can be positioned to pick up the peak intensity of the light reflected off the sample.

2.3.2 Taking Measurements

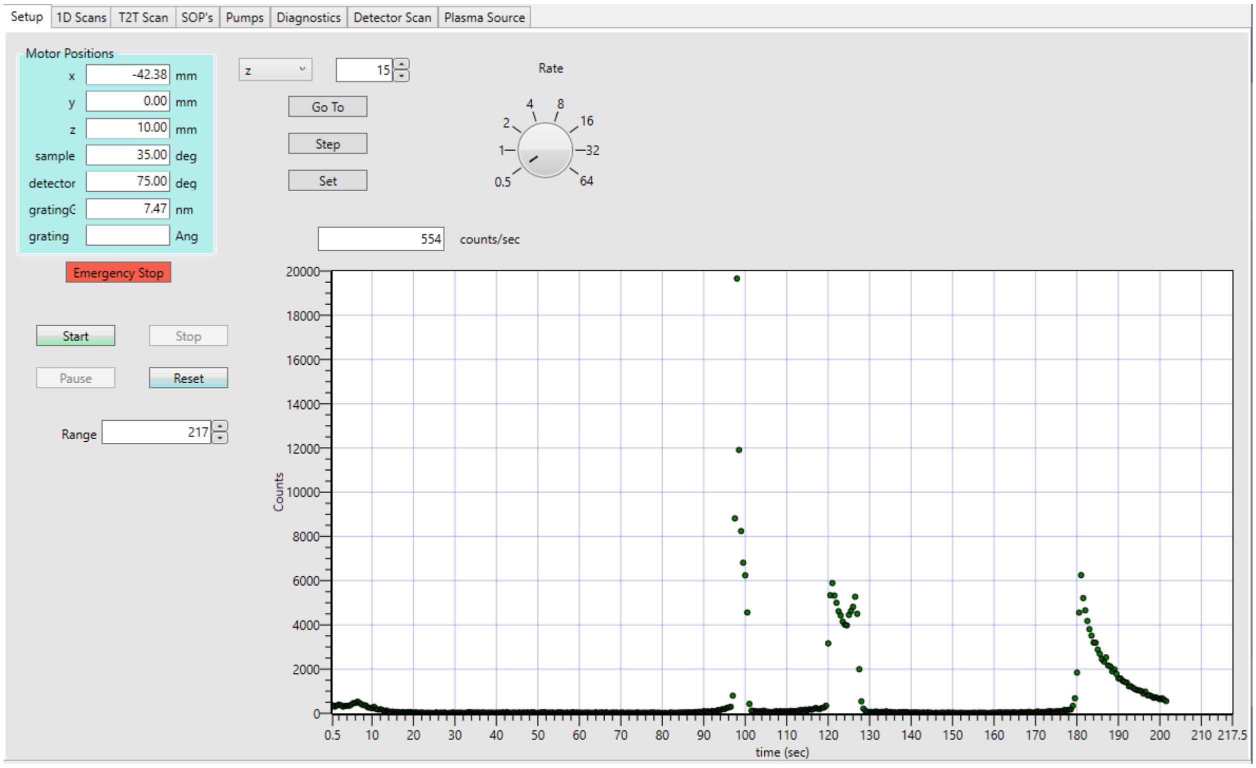
First the sample is secured to the sample stage. The system is then closed off and put under vacuum. Once the pressure is low enough, the plasma is started and left to stabilize for several minutes. After that, the slit is opened, allowing light to enter the O-chamber.

If the monochromator has been out of use for some time, it may be necessary to realign the diffraction grating. This can be done using the detector to measure the intensity of light as the diffraction grating is rotated, then matching up the measured peaks with known peaks from a database such as the National Institute of Standards and Technology's database.

A program called Octopus serves to control all the motors in the O-chamber and display the intensity of light measured by the detector. Figure 2.4 shows the Octopus display.

We first measure the maximum intensity of light, called I_0, by moving our sample stage out of the beam path and doing a sweep of about 5 degrees with the detector. This also serves to ensure the detector is centered at 0 degrees. We then slowly bring the sample stage into the path of the beam until it blocks half the light, or the intensity measured drops to half. To ensure that the sample is perfectly parallel to the beam, we rotate it by small angles. If this rotation causes an increase in measured intensity, we set this new angle to 0 and repeat the process of bringing it slowly into the path of the beam until it cuts off half the intensity (see Fig. 2.6).

Once the sample mirror has been centered in this way we can measure its reflectance. We rotate the sample stage to the desired angle, and because the light is reflecting off the samples the detector



x -42.38 mm
y 0.00 mm
z 10.00 mm
sample 35.00 deg
detector 75.00 deg
gratingG 7.47 nm
grating Ang
z 15
554 counts/sec
Range 217

Figure 2.4 The control screen of the Octopus program. Allows us to control the motors to move the sample stage and rotate the sample stage and detector. The 1D scan tab allows us to plot the intensity vs. another parameter, such as the detector angle. Other tabs are used to turn on pumps and detector.

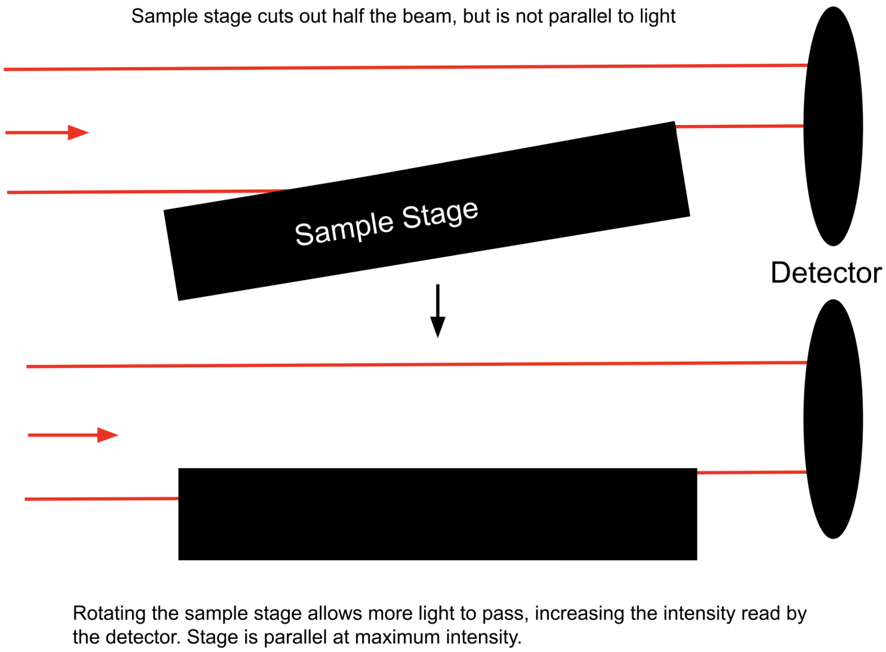


Figure 2.5 We measure the intensity as we rotate the sample stage to determine when the sample is parallel to the light. 0 degrees is set at the peak intensity.

must be moved to twice that angle. Due to imprecision in our motors, the maximum intensity of the beam is not always exactly double the sample angle, so we scan across a range of about 4 degrees with the detector near double the sample angle to include the maximum intensity.

We generally measure several reflectance angles, including one as close to normal incidence as we can. Of course, if the sample were to be measured at 90 degrees, the detector would block the incoming light. The highest angle we can measure is 80 degrees.

Once we have measure all the desired angles, a program on python, created by Devin Lewis, picks out the maximum intensity of each scan and plots them as a percentage of the I_0 measurement against the angle of reflectance, as shown in Fig. 2.6.

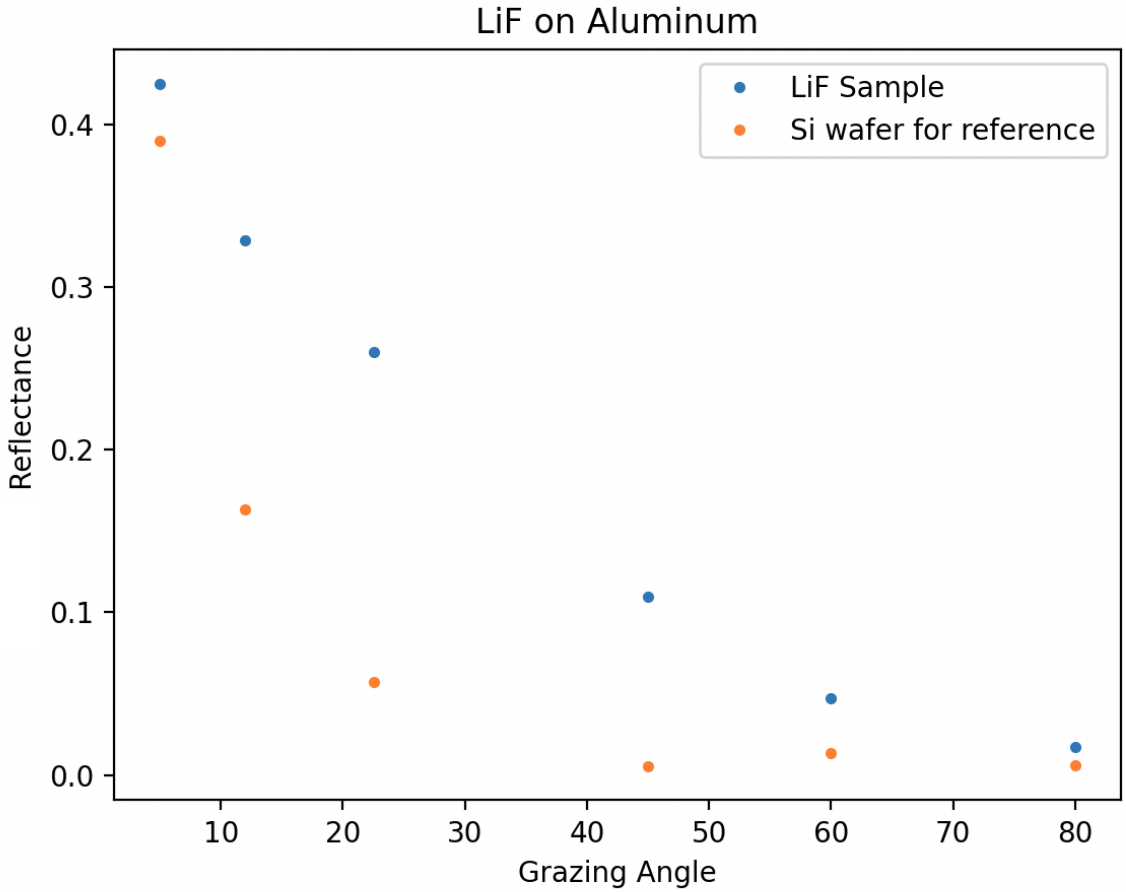


Figure 2.6 This is an example of our data from the reflectance measurements. The y-axis the measured reflectance compared to the I_0, or the intensity without any reflections. The x-axis is the angle of the sample stage, 0 degrees being parallel with the beam. The sample was lithium fluoride on aluminum, with a silicon wafer as a reference. This figure serves as an example, and as discussed later is not reliable data.

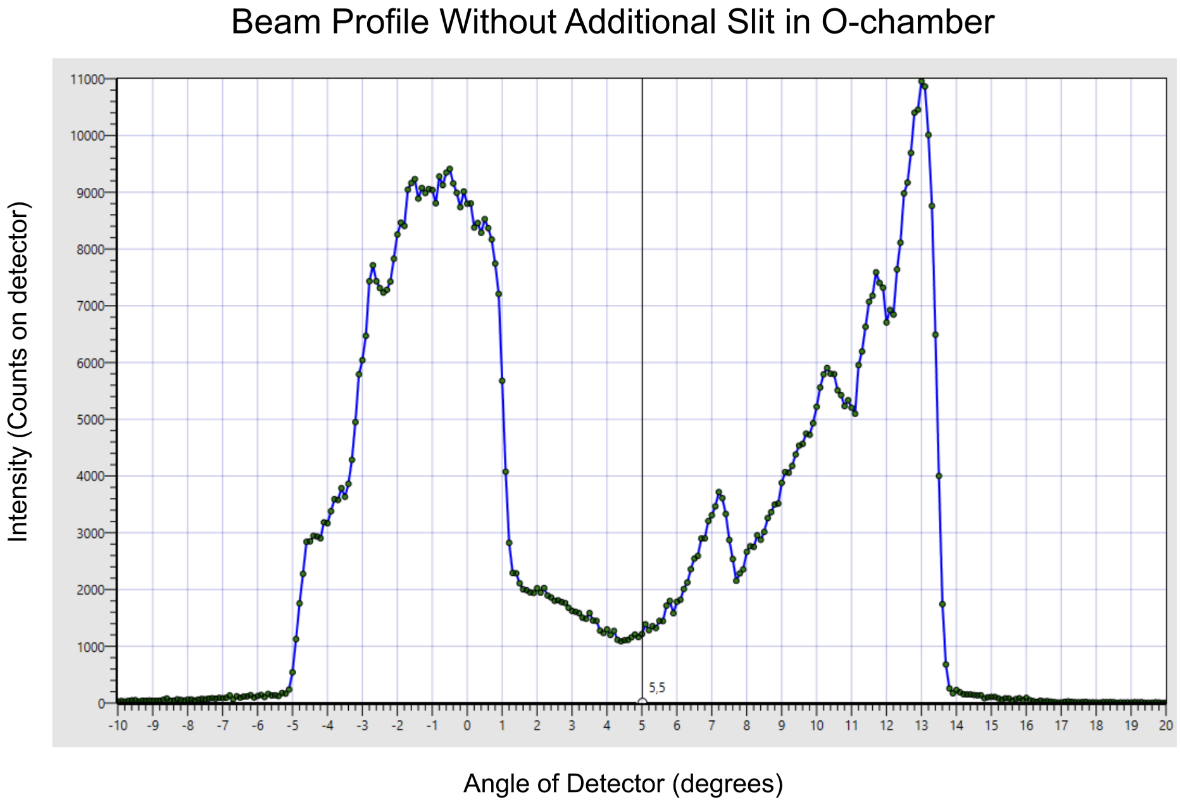


Figure 2.7 This is the light profile against the back of the chamber. The light does not enter the chamber as a clean beam, but rather separated into these two bright spots.

2.3.3 Beam Profile

The light from the monochromator does not enter the O-chamber as a clean, uniform beam. This may be caused by a misalignment of the diffraction grating and the slit. Figure 2.7 shows the beam profile as measured by the detector in the O-chamber. We could not solve the problem in the monochromator, so we placed another slit at the opening from the monochromator into the O-chamber (label 1 in Fig. 2.3 The new beam profile is shown in Fig. 2.8. This profile with just one bright spot will give us more consistent and reliable measurements.

Another thing we had to verify was that our samples were perfectly vertical so they did not reflect the light out of the horizontal plane. This would cause the light, or some portion of the light,

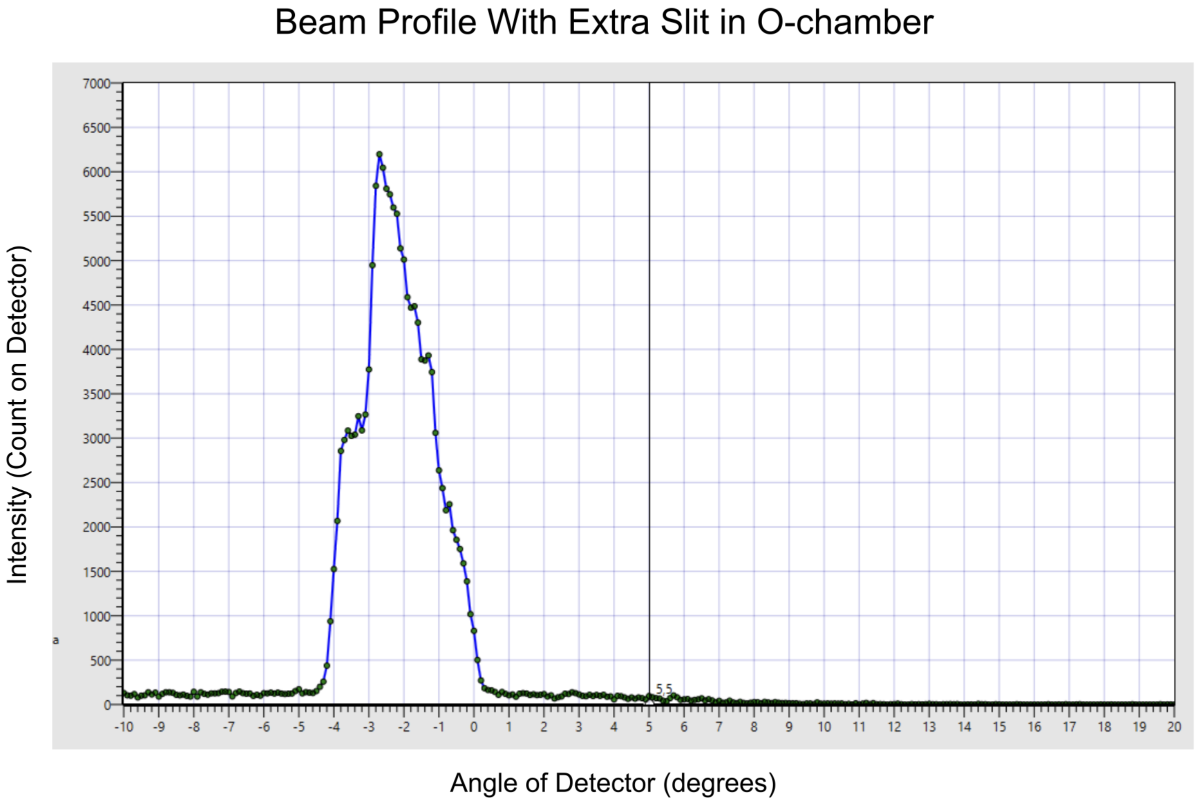


Figure 2.8 After adding a thin slit in the O-chamber, we were able to get one bright spot.

to miss the detector and give us a bad measurement. To verify this we set up a HeNe laser shining through a window in the O-chamber opposite the opening from the monochromator. The laser was aligned to be perfectly horizontal and passing through the center of the chamber, like a mirror image of our plasma light. The laser was reflected off the samples at 0, 20, 45, and 80 degrees onto the detector to see if the beam shifted up or down on the face of the detector. It did not, and we determined that our samples were vertical and not reflecting the light out of the horizontal plane.

2.4 Silicon and Silicon Nitride References

We set up the system the best we could for getting reflectance measurements, but before measuring samples, we need to verify that we are getting correct measurements. We can do this by measuring the reflectance of a simple sample with a known reflectance.

We used samples of our silicon and silicon nitride coated substrates. We used a program to calculate the expected reflectance on each substrate with our sample stage at 70 degrees. We could then measure the reflectance of each sample at the same angle and compare to the calculated values.

Chapter 3

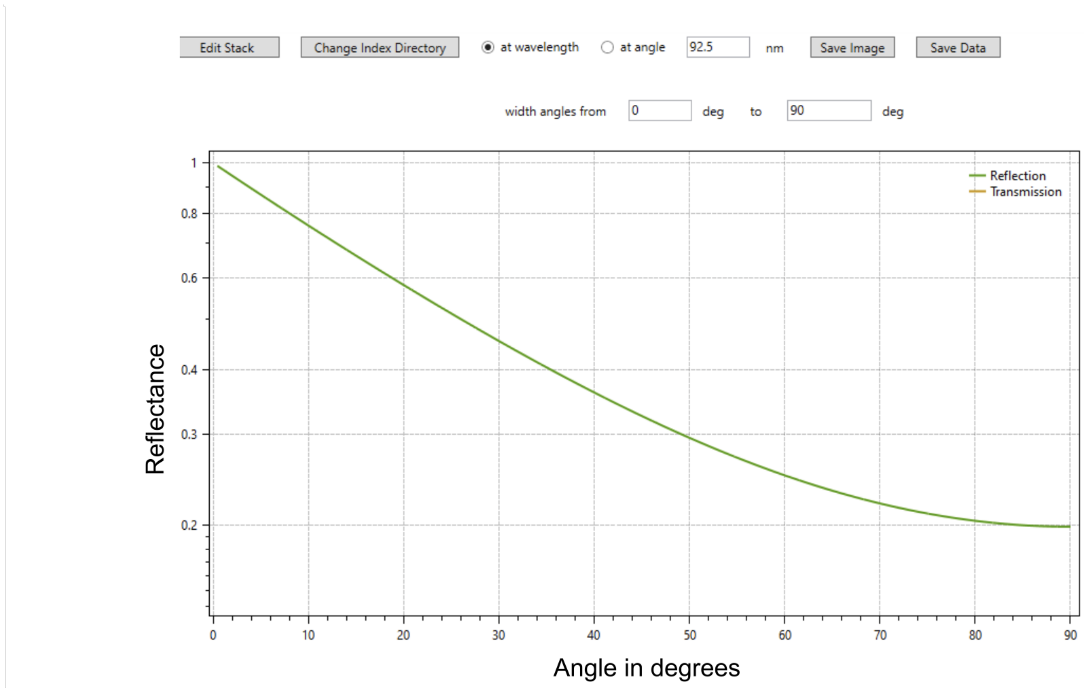
Results and Conclusion

3.1 Results and Discussion

Our initial measurements of the lithium fluoride coated aluminum mirrors were lower than we expected. Figure 2.6 shows a near normal incidence reflectance of less than 5% for a sample of lithium fluoride on aluminum. Even if there were significant oxidation the reflectance should be higher than that. Measurements of aluminum fluoride and magnesium fluoride coated aluminum mirrors showed similar results, with less than 10% reflectance in each case. To ensure that this was not a problem caused by our samples we resorted to the method of using a simple reference mentioned above.

The calculated reflectance of our silicon wafer is shown in Fig 3.1. At 92 nm and an angle of 70 degrees, we should observe 20% reflectance. Our silicon nitride coated wafer on the other hand should have a reflectance of 22% at the same angle and wavelength. When measured with the monochromator, however, our reflectance was much lower than expected. Table 3.1 shows these results.

The silicon substrate measured only about one fourth the expected reflectance, while the silicon



● at wavelength ○ at angle 92.5 nm

width angles from 0 deg to 90 deg

Figure 3.1 This program calculates the expected reflectance of our silicon nitride wafer. The calculated reflectance shown is for 92.5nm light, and the x-axis shows the angle between the light and the sample. Reflectance at 70 degrees is 22%. The same program calculated 20% reflectance for our silicon wafer at 70 degrees.

Table 3.1 Reflectance calculations and measurements for reference mirrors at 70 degrees with 92 nm light

Reference Mirror	Calculated Reflectance	Measured Reflectance
Silicon Nitride	22%	8.2%
Silicon	20%	4.8%

nitride wafer was a little closer at one third the expected reflectance. Both these measurements were taken in the same run, meaning both wafers were on the sample stage, and after measuring the reflectance of one, the stage was shifted so the other was in the path of the light, and that wafer was then measured. The plasma was not restarted and the system remained under vacuum.

3.2 Conclusion

From the measurements of our silicon references, we can conclude that our monochromator is not producing accurate reflectance measurements. We are getting some reflectance, but not nearly as much as we would reasonably expect. Whether this is caused by some misalignment in the system, a fault in our detector, or something else is not immediately clear. While we have developed useful methods and made progress in gathering reflectance data, further work will be needed to get reliable reflectance measurements.

The goal was to develop a reliable method for measuring FUV reflectance, and while further work is needed to make the measurements more reliable, we were able to develop a method for getting reflection measurements.

3.3 Future Work

Additional research will be needed to provide reliable FUV reflectance measurements for aluminum mirrors.

One difficulty is in characterizing the profile of the light coming into the O-chamber. Because UV light is invisible to our eyes, we can't see the light profile as we might with a laser to easily detect if some light is missing the detector. Additionally, our detector has limitations. Because it only moves in the horizontal plane by rotating around the sample stage, it is hard to get a full picture of how the light is behaving. Providing a way to actually see the light would greatly help us determine what might be causing our low reflectance measurements. This might be done with the use of a substance such as sodium salicylate, which fluoresces when exposed to UV light.

These reflectance measurements will also be useful on future work done with First Contact. First Contact is a polymer designed to clean optical mirrors. It is applied to the mirror, dries, and then peeled off, removing any particles on the surface in the process. Research is being conducted to better understand how First Contact affects the surface of the mirror and how well it cleans. It would be useful to see how this polymer affects the reflectance of certain mirrors after its use to see if it leaves behind any residue that impedes reflectance. In addition, it may help in slowing oxidation of aluminum mirrors.

Bibliography

[1] N. A. of Sciences Engineering and Medicine, *Pathways to Discovery in Astronomy and Astrophysics for the 2020s* (National Academies Press, 2021).

[2] NASA, “LUVOIR Final Report,” https://asd.gsfc.nasa.gov/luvoir/reports/ (Accessed March 4, 2023).

[3] J. Larruquert, J. Mendex, and J. Aznarez, “Nonoxidized Al-overcoated Ir bilayers with high reflectance in the extreme ultraviolet above 50 nm,” Opt. Eng. **41,** 1418–1424 (2002).

[4] A. A. Davis, “Oxidation of Aluminum Under Various Thicknesses of Aluminum Fluoride,” Senior Thesis (Brigham Young University, Provo, UT, 2018).

[5] M. Bass, *Handbook of Optics* (McGraw-Hill, 1994), Vol. 2.

[6] M. Hart, “Retarding the Growth of Oxide on Aluminum Thin-Films,” Senior Thesis (Brigham Young University, Provo, UT, 2022).

[7] *Handbook of Optical Constants of Solids*, E. Palik, ed., (Academic Press, 1998).

[8] “Experimental determination of optical constants of MgF2 and AlF3 thin films in the vacuum ultra-violet wavelength region (60–124nm), and its application to optical designs,” Optics Communications **283,** 1351–1358 (2010).

[9] M. Miles, “Evaporated Aluminum Fluoride as a Barrier Layer to Retard Oxidation of Aluminum Mirrors,” Master’s thesis of the Department of Physics and Astronomy (Brigham Young University, Provo, UT, 2017).

[10] M. A. Quijada, J. D. Hoyo, and S. Rice, SPIE (2014).

[11] D. M. Lewis, C. M. Plewe, A. Stapley, J. Vawdery, R. Turley, and D. Allred, “Illuminating the degradation of lithium fluoride mirror coatings in humid environments,” In *Advances in Optical and Mechanical Technologies for Telescopes and Instrumentation IV*, R. Navarro and R. Geyl, eds., 11451 pp. 746–753 (SPIE, 2020).

[12] I. Tzinis, “Technology Readiness Level,” https://www.nasa.gov/directorates/heo/scan/engineering/technology/technology_readiness_level (Accessed March 4, 2023).

Index

aluminum, 2
aluminum oxidation, 2
at-wavelength measurements, 5
atomic force microscopy, 9

ellipsometry, 9

First Contact, 22
fluoride coatings, 2
    fabricating, 7

hydrogen Lyman series, 2

LUVOIR, 2

monochromator, 10

O-chamber, 11

plasma, 10

thermal evaporation, 7