Making Aluminum Shine Again

removing polymers used to protect aluminum from oxidization

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ABSTRACT

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The LUVOIR telescope, a potential future telescope for NASA, will benefit from having a broadband mirror coated with aluminum that can allow for reflectance from the visible and infrared all the way down to the far-UV. The aluminum thin-film will need to remain unoxdized in order to reflect into the far-UV range. The application of polymers to protect the aluminum from oxidizing was investigated. We used a magnetron sputtering system and a plasma cleaner to determine whether various polymers could be removed via hydrogen etching of the thin-film. Ellipsometry was used to determine the thicknesses of the polymer thin films before and after etching occurred. The results show that controlled etching can occur with various gases. The results also show that the etching process was reasonably controlled.

Keywords: Aluminum, LUVOIR , NASA , Parylene, Polymers

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Contents

Chapter 1

Introduction

1.1 NASA And LUVOIR

NASA has always pushed the frontiers of science and space exploration through the development of new space technologies. The Hubble Space Telescope helped with the discovery of the age of the universe, learning more about dark energy, and giving us a new view of our universe by being a space based telescope (NASA Accessed July 15, 2017a). Since Hubble, NASA has made other telescopes that continue to push the boundaries of our understanding of science and our universe. NASA plans to continue to expand this knowledge and one of the possible plans is a future telescope, the LUVOIR (Large UV-Optic-IR) telescope.

The LUVOIR space based telescope or space observatory will be in NASA's decadal review where they will determine their goals for the 2020's. The LUVOIR telescope could be used to help further our understandings of the past, present, and future evolution of our universe, and could also be used to search for exoplanets. In order to be a viable candidate for NASA to choose as its next telescope to build, research needs to show that the science behind it is sound. If built, the LUVOIR will require some of the biggest mirrors ever flown in space, and they will need to reflect in the

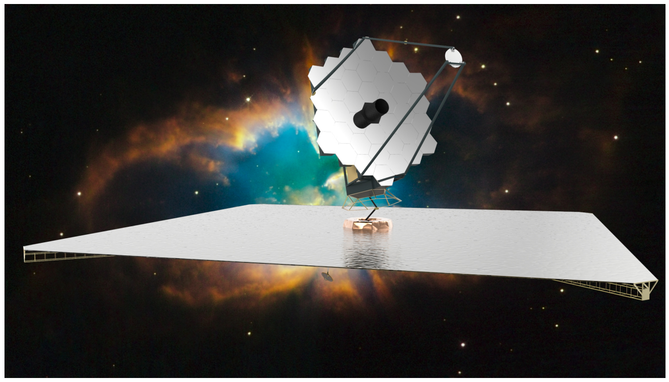

Figure 1.1 Artist rendition of the LUVOIR telescope (NASA Accessed July 15, 2017b).

UV, visible, and infrared regions of the electromagnetic spectrum.

The best candidate for a broadband mirror is an aluminum coating that is unoxidized. An unoxidized aluminum thin-film will be able to reflect wavelengths from the Infrared down to 82 nm which is into the far-UV (120-200nm) and EUV (10-120nm) range. Once the mirror is in orbit, it will be far enough away from the earth's surface that it will not oxidize (Larruquert & Keski-Kuha 2008). The challenge is how to make mirrors with an aluminum thin-film and get them to space without being oxidized. One option that has been proposed is to coat the mirror with aluminum once in space (Hass & Hunter 1967). The logistics of doing this are so great that this experiment has only been attempted once (J. A. Aznarez & Sanchez-Avedillo 1998). Opposed to this, a process called REVAP has been proposed as a means of protecting the aluminum with a volatile protective layer that can be removed once in space (Burton 1983). This experiment was only performed with zinc protective layers and it is still unclear as to whether the technique was successful for protecting the mirror's surface. This opens the door for new research on protective films. The focus of this thesis is on the investigation of using polymers as a space-removable, oxygen-barrier layer to protect the aluminum thin-film mirror.

Chapter 2

Experimental

2.1 Samples

In the pursuit of testing whether or not the aluminum coated mirrors can be protected by a polymer thin-film, we performed experiments on different samples that were obtained either by making them ourselves, or from other institutions. The samples are all silicon wafers, each having different thin-films deposited on top of the silicon.

1. Found in lab

   (a) In our research lab there are many silicon wafers that we have in stock. Most of them were obtained by buying them in bulk from other institutions. Some of these silicon wafers are secondhand, and we were able to find some wafers that had some plastic on the surface. We decided to use these wafers as a proof of concept to see what would happen when we etched the plastic thin-films and to see what parameters we could control.

2. Spin on

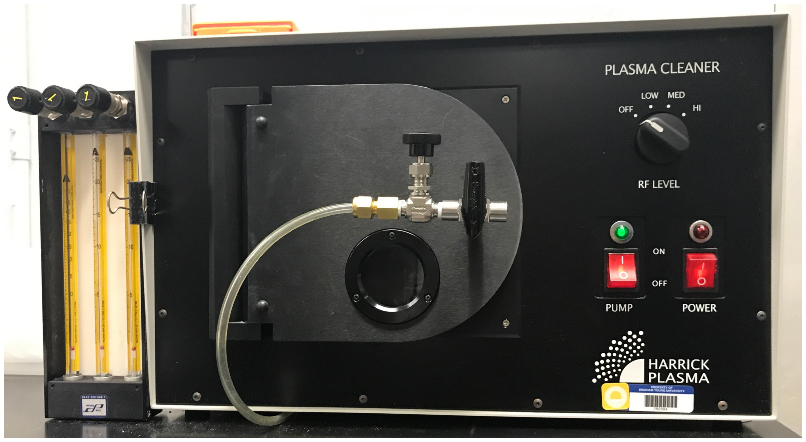

Figure 2.1 Plasma etcher used to etch silicon wafers. Harrick Basic Plasma Cleaner, Model PDC-001. Input Voltage 115 VAC @ 60 Hz, Max power 200W, Max Current 3A. A roughing pump was used to pump out the system and create a vacuum in the pyrex barrel inside.

(a) Some of our samples were prepared in our own lab. We used PMMA (polymethyl methacrylate) which is a polymer that is frequently used in microelectronics as a protective coating and as a sacrificial layer. The advantage of using this polymer is that it is well researched, inexpensive, and readily available.

i. Cleaning the surface: we cleaned the silicon wafer with a liberal amount of acetone or ethanol. We then further cleaned the sample by putting it into the Harrick Plasma Cleaner (see Fig. 2.1).

ii. Applying PMMA: after placing the silicon wafer on the center stand in the photo resist spinner, we applied 20 drops of PMMA solution in the middle of the silicon wafer. We ran the photo resist spinner (see Fig. 2.2) using program 1 from the list of preprogrammed settings.

3. Parylene From Los Alamos

(a) Another likely candidate for protecting the aluminum mirrors is the polymer parylene. Parylene is also well researched and has superior barrier properties compared to other

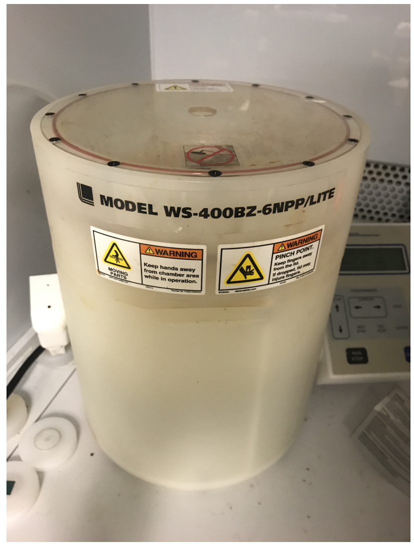

Figure 2.2 Photo resist spinner used to spin on PMMA

polymers. Parylene is also used in microelectronics as a protective layer. It is inexpensive and readily available as well. The process for making parylene requires a pyrolyzer, and other machines which were not available to us. Instead of coating silicon wafers with parylene ourselves, we obtained silicon wafers from Los Alamos that were already coated with parylene thin-films.

2.2 Etching of Samples

The following are the procedures that we followed when etching using the Harrick Plasma Cleaner and the vacuum system located in our lab.

- Procedure for using the Harrick Plasma Cleaner

  1. Turn on vacuum pump (roughing pump) and pump out the chamber.
  2. Once chamber is pumped out, allow etching gas (hydrogen) to enter through needle valve attached to the front of the plasma cleaner.

3. After turning the needle valve only a few degrees (2-6), turn on the RF power button on the right, and turn the RF power switch (top switch) to HIGH.
4. Adjust needle valve as needed after the plasma forms to keep the plasma from disappearing (if opened or closed too much, the plasma will die, so aim for 2-6 degrees and adjust accordingly).
5. Etch for desired amount of time (usually 1-2 minutes).

- Etching with vacuum system procedure

1. Place the sample to be etched on the surface of a target.
2. Pump down the system using the roughing pump, and then use the turbo pump to further lower the vacuum.
3. Connect the DC or RF sputtering machine.
4. Once the pressure is in the high vacuum range (10 mTorr to 10 nTorr), open the valve that allows the desired gas to enter and turn on DC or RF sputtering machine.
5. Observe through the window if a plasma is formed. Then etch for as long as one desires (usually 1-2 minutes).

2.3 Surface Characterization

To characterize the surface and determine the thickness of the layers on the silicon wafer we made measurements using the M-1000 Woollam variable-angle spectroscopic ellipsometer. We used a parametric model (Cauchy) to fit the data and we made the measurements at 75°.

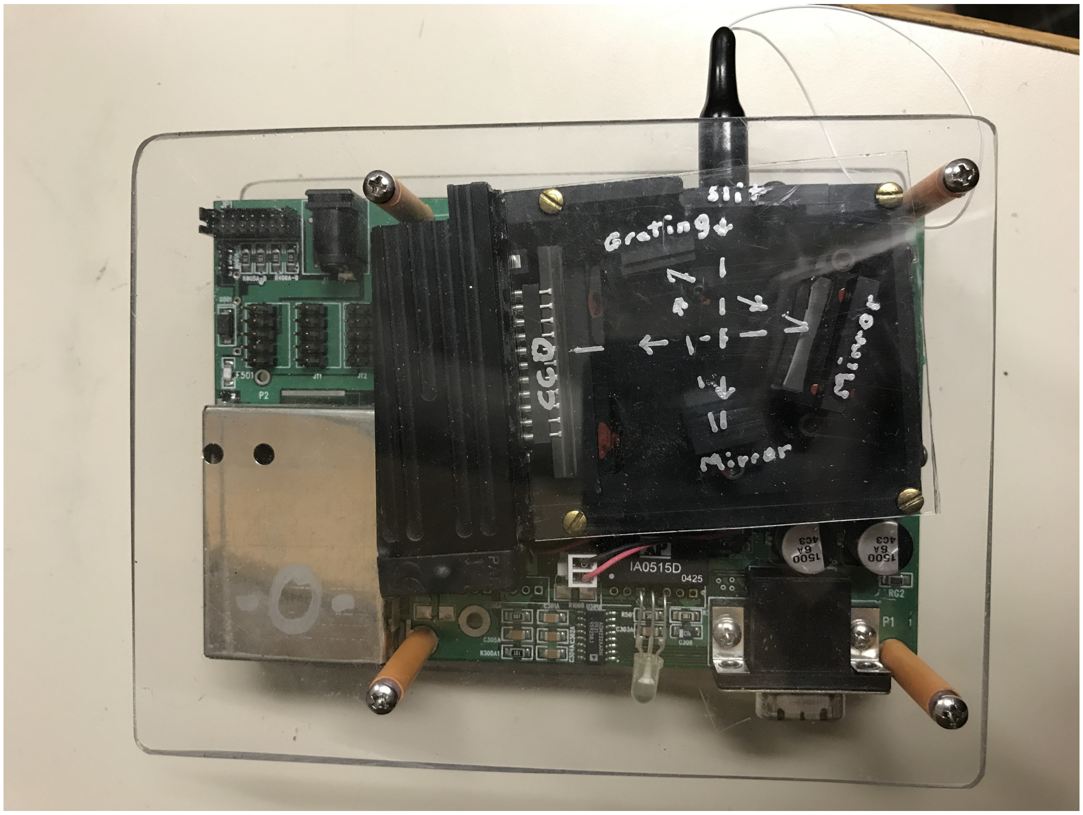

Figure 2.3 Spectrometer used to obtain the spectra which characterized the plasma's in my senior Thesis. The spectrometer grating contains 900 lines per inch.

2.4 Plasma Characterization

We used a spectrometer obtained from the BYU Physics lab stockroom. The spectrometer had a grating with 900 lines per inch. Light was coupled to the spectrometer using a glass capillary optical fiber that was placed facing the window of the vacuum system where the plasma was located. The spectrometer was calibrated with a Geissler tube containing helium.

Chapter 3

Results

3.1 Graphs and Etching Rates

We used a spectrometer (see Fig. 2.3) to perform spectroscopic analysis of the plasma's in the Harrick plasma cleaner. The measurements were performed without a sample wafer located inside. Figures 3.1 to 3.5 show the absorption spectra that were obtained. Data for the spectral lines of the elements was gathered from the NIST handbook of Basic Atomic Spectroscopic Data (NIST 2009). One problem that we faced when using the Harrick plasma cleaner was trying to keep the gas/mixture nonflammable. This is one reason why we used forming gas because it is a mixture of nitrogen and hydrogen which is less flammable.

The other figures (3.6 to 3.9) contain the results from etching with forming gas, etching in our vacuum system, and a picture of the parylene sample we etched. Each figure contains further explanation about each result.

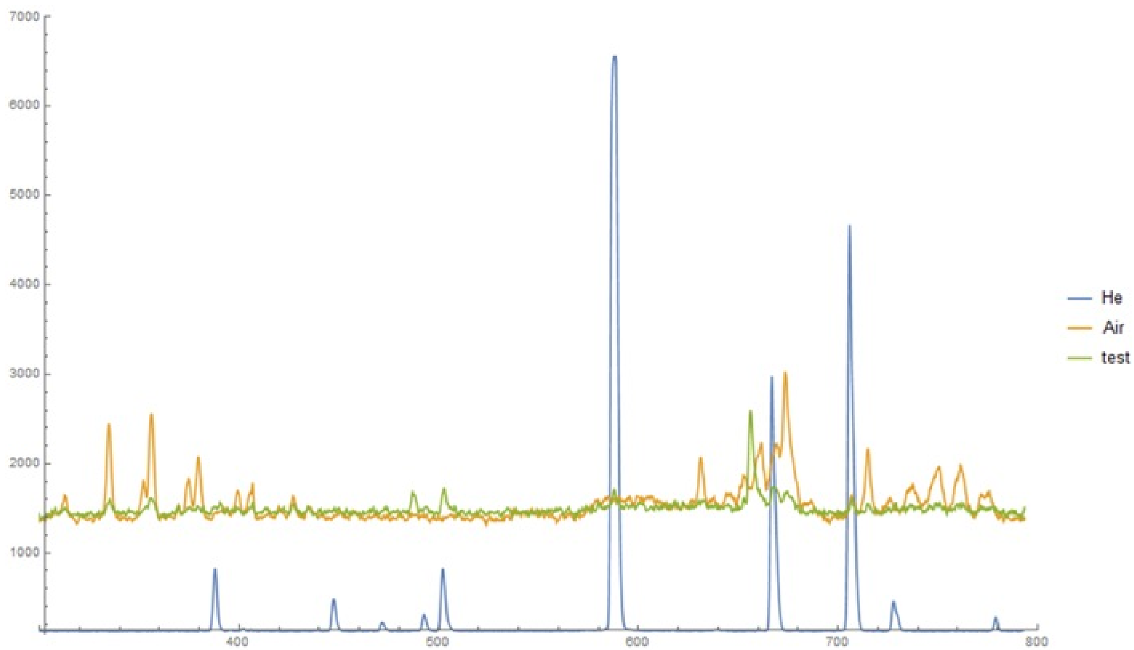

Figure 3.1 Absorption spectrum from spectroscopic scan. The range is 400 to 800 nm and data from the He calibration, air test, and forming gas test are shown.

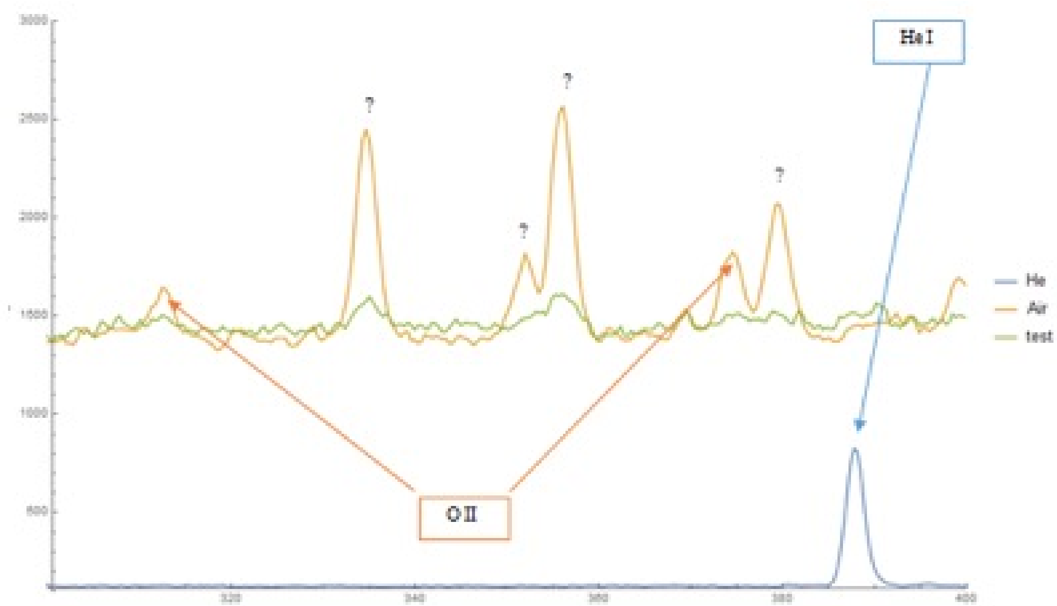

Figure 3.2 Absorption spectrum from spectroscopic scan. The range is 300 to 400 nm and data from the He calibration, air test, and forming gas test are shown. Oxygen is present and unknown peaks are present as well.

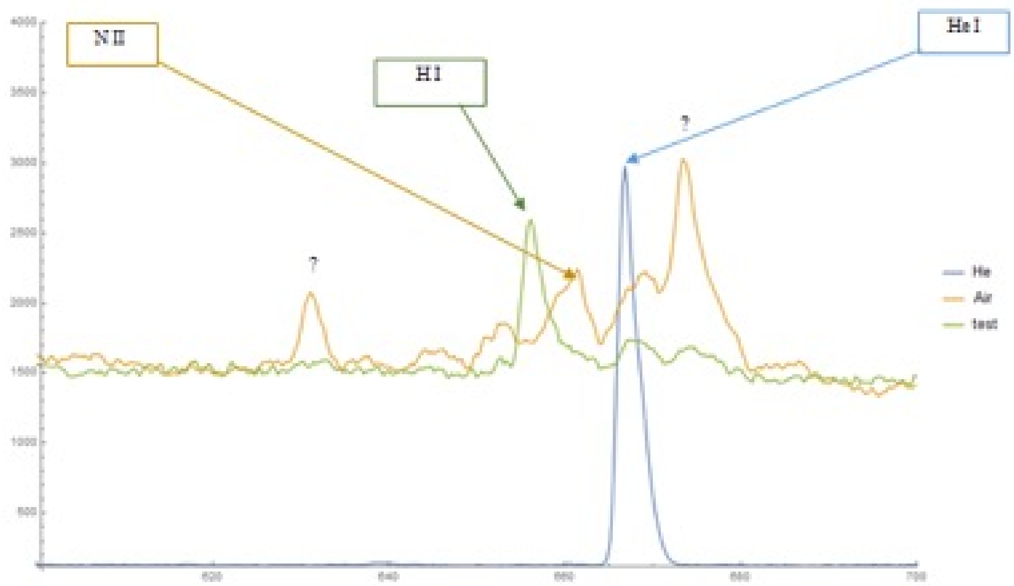

Figure 3.3 Absorption spectrum from spectroscopic scan. The range is 600 to 700 nm. Nitrogen and hydrogen are present in the test run and unknown peaks are present in the run with just air from the lab let into the system.

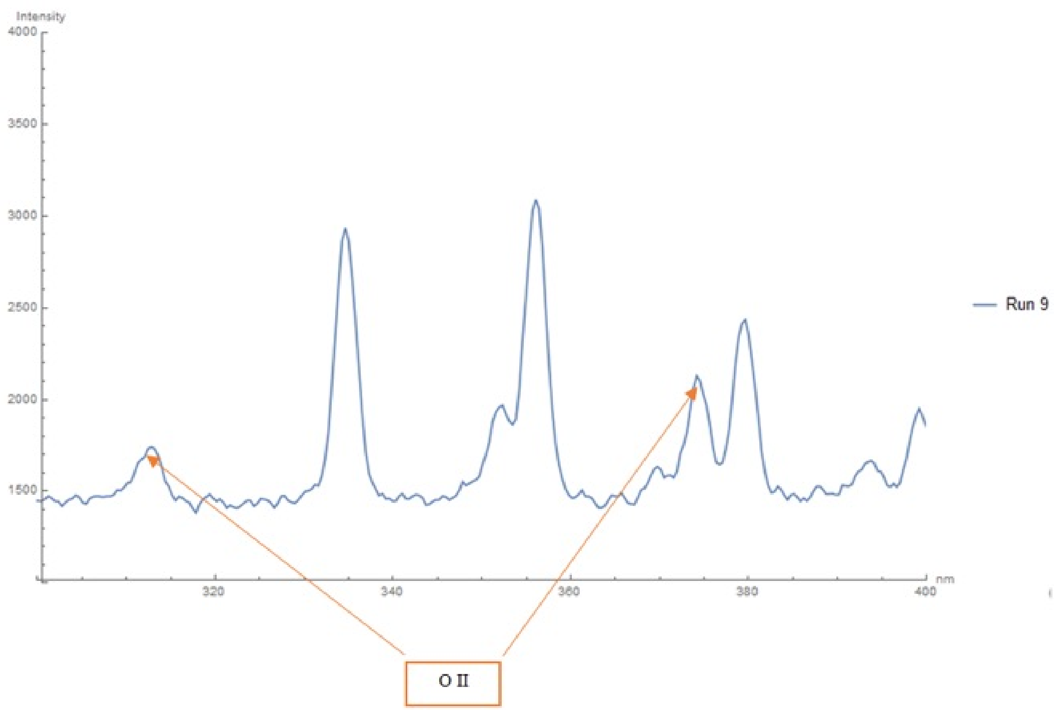

Figure 3.4 Absorption spectrum from spectroscopic scan. The range is 300 to 400 nm and it shows that oxygen was present when using forming gas in run 9.

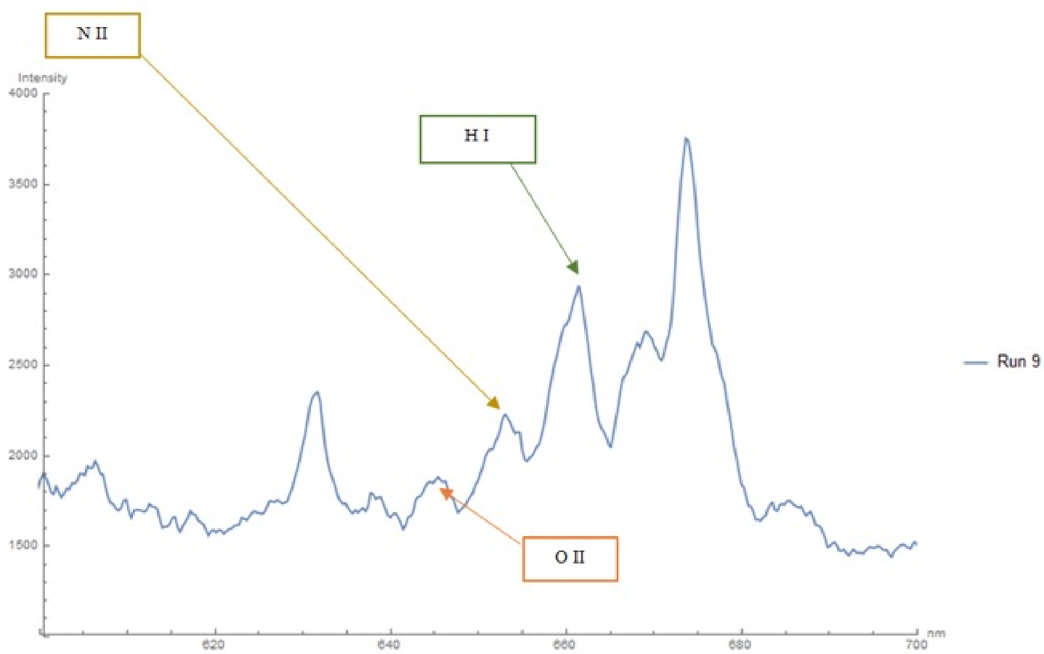

Figure 3.5 Absorption spectrum of run 9 containing forming gas. We can see the presence of nitrogen and hydrogen, but we also see some oxygen present.

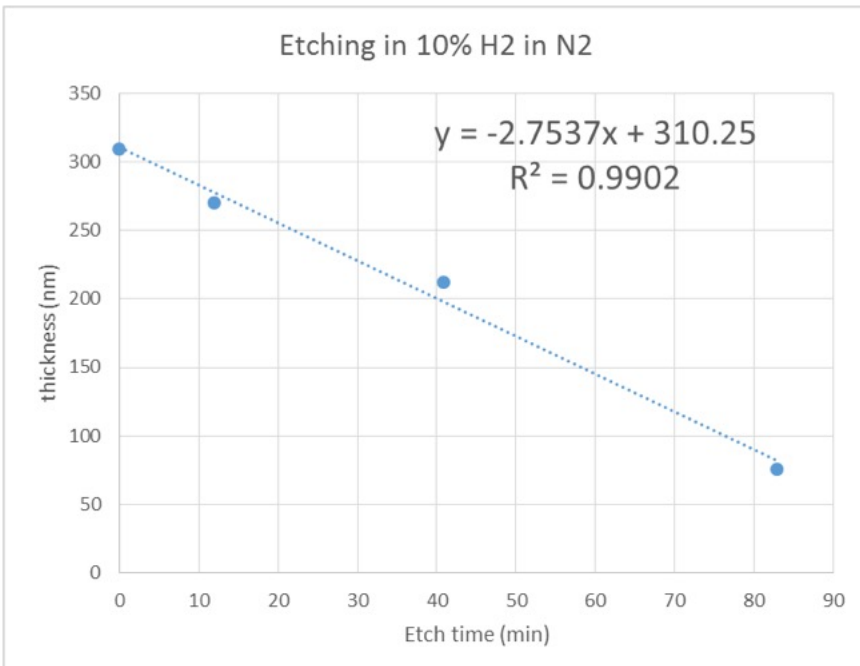

Figure 3.6 Etching rate in forming gas. (10% hydrogen in nitrogen). Shows a linear fit between the thickness in nanometers and the etch time in minutes. Shows a reasonable amount of control over the etching rate of the plastic. This was done using RF sputtering.

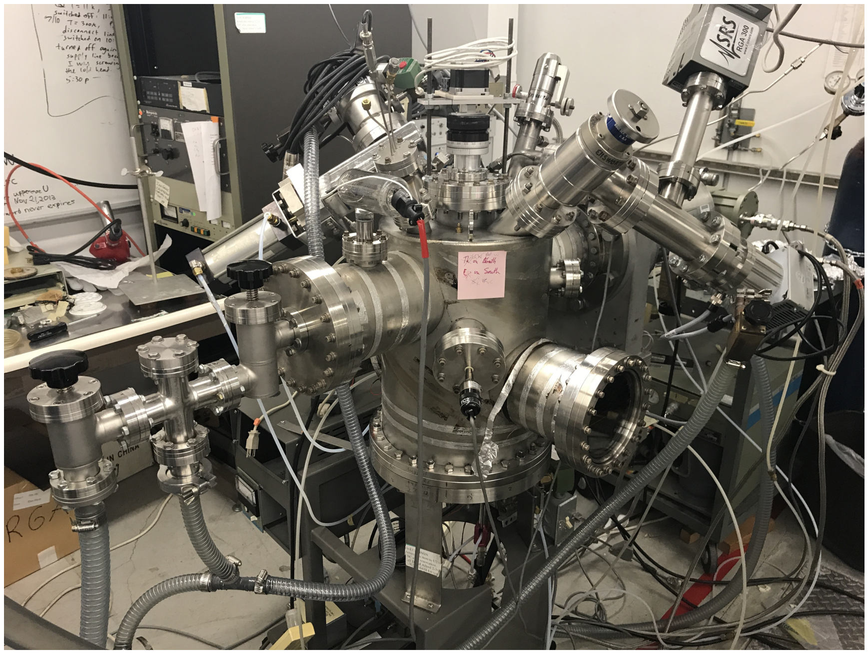

Figure 3.7 Magnetron sputtering system referred to as Joey. This vacuum system was connected to a DC or RF power supply which were used for DC or RF sputtering, respectfully.

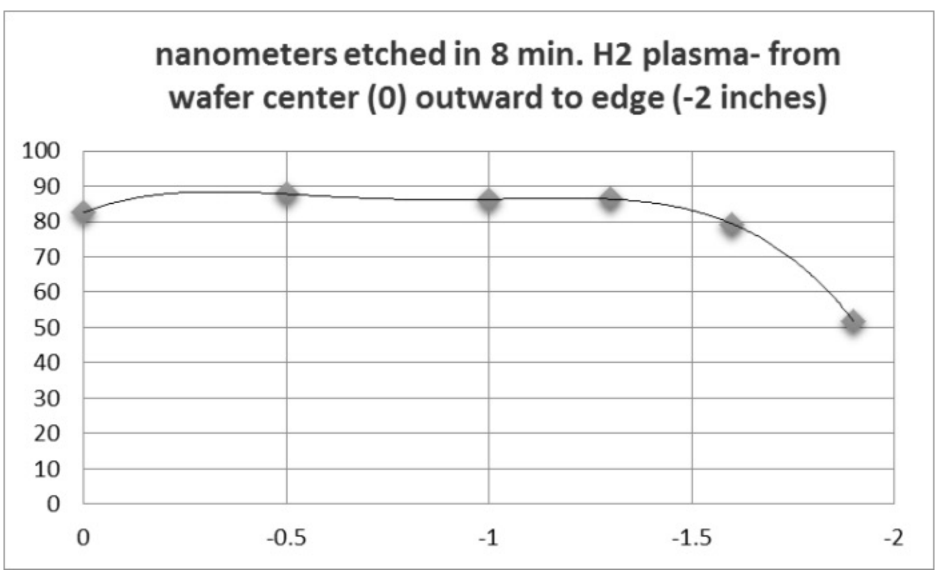

Figure 3.8 Etching thickness after placing the plastic sample in our magnetron sputtering system. The sample was placed on the south sputtering target. This etching was performed using RF sputtering.

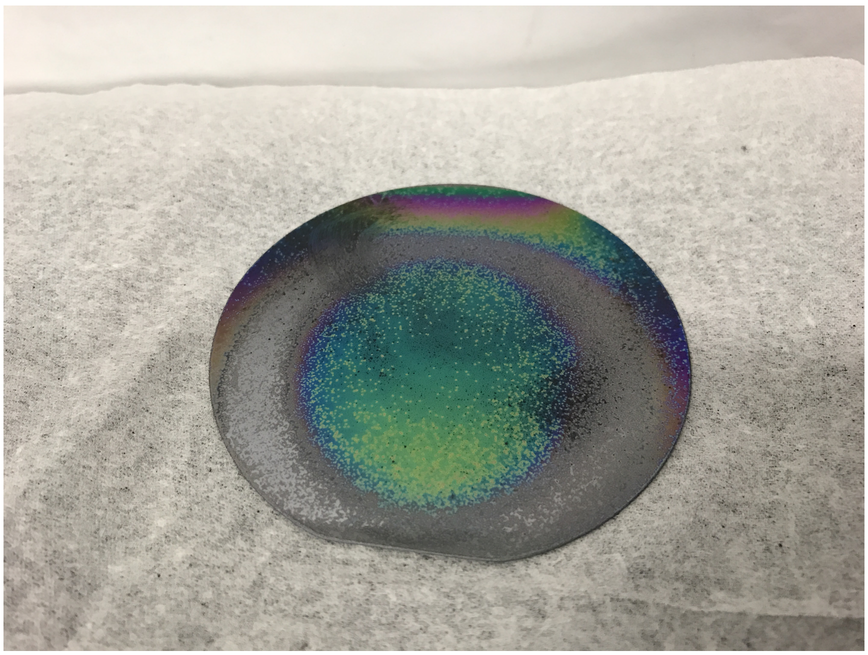

Figure 3.9 Parylene sample etched using DC sputtering where arcing occurred. The etching was nonuniform due to the arcing which occurred. The middle was left mostly untouched by the etching process while a circular ring pattern was etched. This might also be due to the shape of the target on which the sample was placed. The arcing also occurred because parylene is an insulating material. Further information on the differences between RF and DC sputtering can be found in Appendix A.

Chapter 4

Discussion

4.1 Harrick Plasma Cleaner

We can see in figure 3.6 that there is a linear relationship in the etching rate when forming gas was used. This linear relationship shows that we can have reasonable control over the etching rate of the plastic. This is good news because in order for polymer coatings to be a viable option as a removable-barrier layer it is important that there be control over the etching rate of the plastic. With this reasonable amount of control, if we know the thickness of the plastic, we could etch just that amount off and not have to continue etching after that. This would help protect the bare aluminum underneath from possibly being damaged from the etching process due to over etching of the surface.

Figures 3.1 to 3.5 show various graphs of the absorption spectra that were gathered. From these graphs we were able to determine that other gases were present in the plasma that was in the Harrick Plasma Cleaner. The reasons for this are: the needle valve on the front may have been leaking oxygen into the system, the glass walls inside may have held moisture, the seal may not have been leak tight, and the back end of the Harrick Plasma Cleaner may have been leaking as well.

The Harrick Plasma Cleaner did help because it was needed to see whether it was feasible or not to use hydrogen or other gases to etch the polymer thin-film protective barriers. From the results we can see that it did work. Later we were able to perform etching with pure hydrogen in our vacuum system under ultra-high vacuum conditions.

4.2 Magnetron Sputtering System

Basic research has been done before on sputtering aluminum, but extensive research has not been performed. Larruquert and Keska-Kuha used inert-gas ion beams to sputter silicon wafers coated with aluminum (Larruquert & Keski-Kuha 2008). They were trying to etch the oxide that naturally formed on the aluminum coating. They used argon in their ion gun to etch the surface. Their results showed that the etching process they performed did result in a roughened aluminum surface. Their research did not include attempts to etch the surface using hydrogen as the etching gas. Hydrogen has a much better probability of being used in the etching process without damaging the aluminum surface because it has a smaller mass than argon (1.0 u instead of 39.9 u). There have also been reports that hydrogen can etch the surface of semiconductors and leave the surface atomically clean (Chang et al. 1982). This is why our next step was to try etching with hydrogen in our vacuum system.

Using the magnetron sputtering system (see Fig. 3.7) we were able to obtain the etch rate of the plastic samples from the center of the wafer to the outer edge. This etching was done using pure hydrogen. The results show us that hydrogen can be used to etch the plastic. Again, hydrogen is the best candidate for etching because it is the lightest element. After having shown that hydrogen could etch polymer thin-films, we decided to see whether or not it could etch the parylene samples as well.

Arcing occurred when the parylene samples were etched in the magnetron sputtering system (This can be seen in Fig. 3.9). The arcing occurred because parylene is an insulator and we were using DC sputtering. RF sputtering is better suited for the etching of insulators. Even with the DC sputtering some etching still occurred, but it was not uniform as seen in Fig. 3.9. Further work is needed with RF sputtering to see whether or not the etching process can be reasonably controlled.

After working with RF sputtering, there still is another problem that may be present. During the etching process, the polymers that are removed from the surface may also be etching the surface as well. Further work should investigate this because if that is the case, then the carbon and oxygen present in the polymers may react with the aluminum thin-film and damage it.

Chapter 5

Conclusion

Further research is still needed to determine whether or not parylene can be used to protect the aluminum thin-film from oxidization. The next step would be to take the other parylene samples and attempt to etch them in the magnetron sputtering system using RF sputtering instead of DC sputtering. RF sputtering is better suited for sputtering insulating materials like parylene.

Once these tests are confirmed, the next step would be to perform measurements of the aluminum surface before and after the application of parylene to determine whether or not oxidization was blocked. Such testing might be performed in Dr. Turley's lab using his vacuum system. Dr. Turley's vacuum system may allow for measurements that can be done while the sample is still in the vacuum system. This would mean that the aluminum thin-film would not oxidize while under vacuum and we could see if the aluminum thin-film was damaged or not.

This research will be included in a published paper that will appear in the Proceedings of SPIE journal (International Society for Optical Engineering). It will be in volume 10398,in paper 34.

Appendix A

RF and DC Sputtering

A.1 RF and DC Sputtering

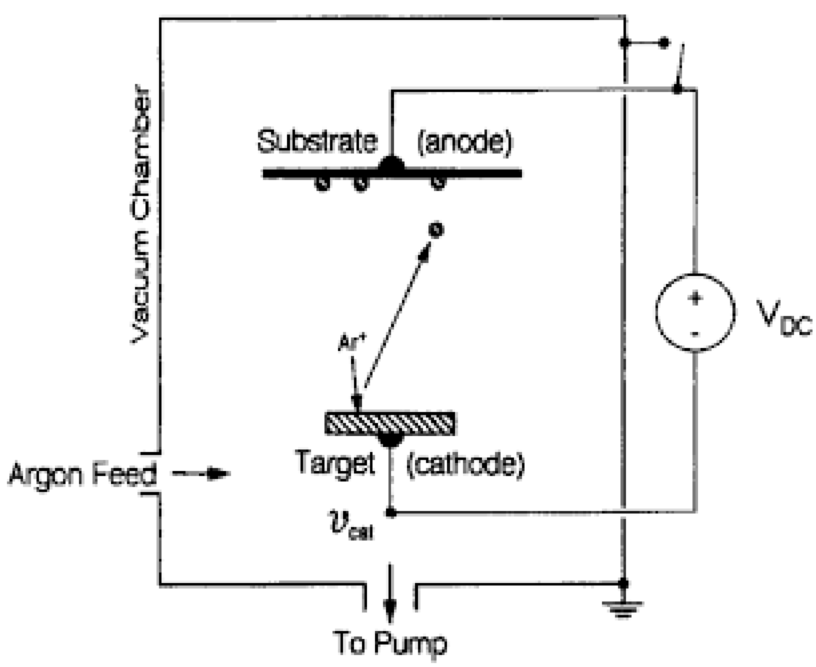

Figure A.1 DC sputtering circuit. (IFN-Trento Accessed July 17, 2017)

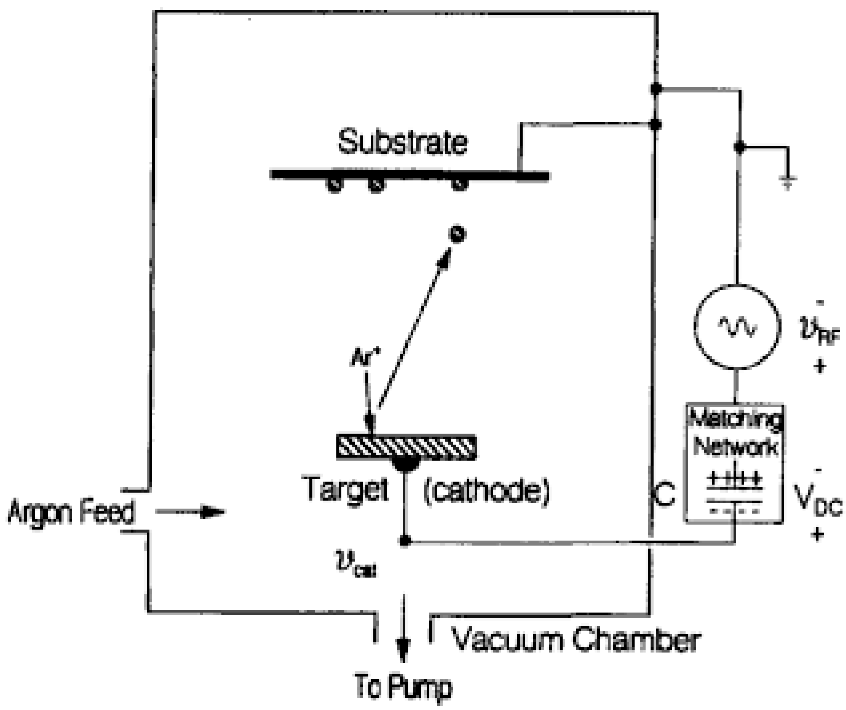

Figure A.2 RF sputtering circuit. (IFN-Trento Accessed July 17, 2017)

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