Multi-Angle Reflectance Measurements in the EUV

by

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March 2001

Submitted to Brigham Young University in partial fulfillment of graduation requirements for University Honors

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Abstract

Optical constants for most materials in the extreme ultraviolet (EUV) are not well known. There are many parameters that matter when trying to determine optical constants from reflectivity including film thickness, roughness, angle, and wavelength. To know and vary the wavelength of the light does not provide enough data to determine these optical constants. Ellipsometry and X-ray diffraction can often be used to determine the thickness of the films, but not always. I have built a multi-angle reflectance measuring device for use in the extreme ultraviolet (EUV) region of the electromagnetic spectrum. This reflectometer allows its user to know and vary the angle at which the light hits the multilayer as well as know and vary the wavelength of the light. This produces many more data points for finding optical constants through fitting.
Acknowledgements

Many thanks to Matthew Squires, R. Steven Turley, and David Allred for the help, encouragement, and insight they have given throughout the time of this research. Others who deserve thanks are Wes Liffereth and Matthew Squires for helping design and machining parts; Spencer Olson and Cort Johnson for help with LabVIEW; Michael Hales, John Ellsworth, and Jason Flint for help with wiring; David Baylogh, Jennifer Vancesdal, Doug Markos, and Matthew Squires for making samples; and Matthew Squires for help measuring samples. Most especially I want to thank my husband, Hyrum, my daughter, Adele, and my parents for all the sacrifices they made for me to finish this.
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Chapter 1

Introduction

Demand is mounting for devices that can manipulate extreme ultraviolet light (EUV). Examples include an EUV microscope, which could image live cells, and mirrors and masks which would enable using the shorter wavelength EUV light in photo lithography and allow production of smaller microelectronic circuits. Countless space applications exist for these same mirrors. The index of refraction is not well known for most materials in this region. This project is a beginning step to filling this information void. One of the obstacles to research in the EUV is that light is readily absorbed by everything, including air, so all measurements must be made in vacuum, and the signals are generally weak.

Most bulk materials reflect less than 5 percent of the incident EUV light. Because their $n$ values are close to 1, there is not much contrast between the material and the vacuum that surrounds it. Their $\beta$ values are also large meaning that almost all of the light will be absorbed. Making good mirrors for this region requires careful planning using the optical constants for the wavelength you want to reflect to design a multilayer of complimentary materials with appropriate thicknesses to have the reflections of that wavelength constructively interfere. In times past this region has been mostly overlooked, so these needed optical constants are not well known.

My research group had a contract to make space flight mirrors for the IMAGE satellite that was launched in March of 2000. These mirrors needed: (1) to reflect well at 304 Å (40 eV), a line from singly ionized helium in the magnetosphere, and (2) reflect poorly at 584 Å (20 eV), a brighter line from neutral helium in the ionosphere. Most materials reflect more at 584 Å than at 304 Å making this a difficult task. Both of these lines are in the EUV.

Shannon Lunt wrote a genetic algorithm to find out what multilayer would work best using published constants[1]. These calculations suggested the best material would be an aperiodic multilayer of yttrium oxide ($Y_2O_3$) and aluminum (Al). This multilayer was not selected for use because of the difficulty of sputtering $Y_2O_3$. Instead, a much easier to make multilayer composed of sputtered uranium (U) and silicon (Si) was produced, for which the calculation showed acceptable optical results.

A U and Al multilayer was also tried, but U and especially Al were found to oxide badly. Adam Fenimore studied the manufacture and oxidation of these samples[2].

The chamber my research group first made for testing their mirrors consisted of two tunnels, which intersected at a 29° angle. The detector could be attached to the chamber either at the back to measure the direct intensity of the source when there was no mirror or on the side to catch the
BEAM REFLECTED FROM THE SAMPLE. THEY ALSO MADE ANOTHER CHAMBER WHICH COULD HOLD THREE SAMPLES AND ROTATE BETWEEN THEM. ONE OF THE SAMPLES WOULD BE MEASURED IN THE FIRST CHAMBER AND THEN INSERTED IN THE SECOND CHAMBER WITH TWO OTHER SAMPLES. DATA FROM THE TWO NEW SAMPLES WAS FOUND BY COMPARING THEIR DATA TO THAT OF THE FIRST SAMPLE.


SPUTTERING, THE PROCESS USED TO MAKE THESE MIRRORS, IS AN EFFICIENT AND NATURAL CHOICE FOR MAKING MULTILAYER MIRRORS. THEREFORE, OPTICAL CONSTANTS FOR SPUTTERED MATERIALS ARE USEFUL TO ANYONE MAKING MULTILAYERS TO STUDY THE EUV.

MATTHEW SQUIRES WORKED TO DETERMINE WHAT THE OPTICAL CONSTANTS ACTUALLY ARE, BUT HE COULD ONLY OBTAIN ONE DATA POINT PER WAVELENGTH USING THE EQUIPMENT AVAILABLE TO HIM, AND THERE ARE MANY PARAMETERS TO FIT, SO IT WAS HARD TO PIN DOWN[3].

FOR MY THESIS, I HAVE DESIGNED A REFLECTOMETER CHAMBER FOR MEASURING HOW THE ABSOLUTE REFLECTIVITY OF A MULTILAYER STACK CHANGES WITH THE ANGLE OF INCIDENCE WHICH WILL BE DESCRIBED IN SECTION 3.3. THIS WILL ALLOW FOR MANY DATA POINTS AT DIFFERENT ANGLES, YIELDING A LARGE SET OF DATA POINTS FOR FITTING. THIS REFLECTOMETER IS A SOURCE OF DATA IN ADDITION TO X-RAY DIFFRACTION AND ELLIPSOmetry, YIELDING MORE INFORMATION TO PIN DOWN PARAMETERS.
Chapter 2

Theory

2.1 Optical Constants

A simple electromagnetic wave in a medium can be represented by the oscillation

\[ E = E_0 e^{i(k_0 r - 

\frac{\gamma}{\hbar} + \phi)} \],

(2.1)

where \( k_0 = \frac{2\pi}{\lambda_0} \) so \( k = \frac{2\pi}{\lambda} \) with \( \lambda = \frac{\lambda_0}{\tilde{n}} \) and \( \tilde{n} = n + i\beta \). Because \( \tilde{n} \) is complex, \( k \) is also complex in the EUV. \( n \) and \( \beta \) depend on the material the wave is traveling through and are called the optical constants. When you plug these equations back into the original equation and expand it, you obtain

\[ E = E_0 e^{i(k_0 r - \frac{\gamma}{\hbar})} e^{-\frac{3\beta}{\hbar} z} \].

(2.2)

The first exponential in equation 2.2 still oscillates and shows that a change in \( n \) effectively modifies \( \lambda \). The second exponential is a decaying exponential which shows that the amplitude decreases as it travels in the z direction and at a given \( z \) decreases as \( \beta \) increases. This decrease in amplitude represents the energy from the wave being absorbed by the medium.

Vacuum has a real optical constant of 1 with no imaginary part by definition. This means that vacuum does not absorb light. Other optical constants are compared to vacuum. The indices of refraction of typical materials at wavelengths in the EUV are complex. The real part is very close to 1, and the imaginary part is large. This leads to two problems, (1) the contrast in the real part is small between vacuum or other materials so there is little reflection and (2) the light is completely absorbed before traveling very far in the material.

2.1.1 Electron Gas Model

The Electron Gas Model is the simplest model of what happens when a wave of light reaches a medium from a vacuum. This section is based on the work of Eberhard Spiller[4]. The scattered field emitted by a single electron when the light hits it is

\[ E_s = -\frac{E_0 e^2 \cos(\theta)}{mc^2r} \].

(2.3)
Even an electron that is bound in an atom can be thought of as free if its binding energy is small when compared with the energy of the incident light. At a point behind a film of thickness \( d \) the amplitude due to a small area \( ds \) is

\[
dE_s = -E_0 r_0 N_{at} f d \cos \theta \frac{e^{ikr}}{r} ds,
\]

where \( f \) is the number of free electrons per atom and \( N_{at} \) is the atomic density of the film. By integrating over an infinite sheet, we find the scattered field emitted by the collision of the light with a film

\[
E_s = -iE_0 r_0 \lambda N_{at} f ds e^{ikz}.
\]

The total transmitted amplitude is the sum of the incident amplitude and the scattered amplitude

\[
E = E_0 e^{ikz} (1 - i\sigma_0 \lambda N_{at} f d).
\]

In optics, the light’s phase shift due to traveling through the film is the difference of the film’s refractive index from 1 times the distance traveled. Neglecting absorption and reflection and taking \( d \) to be very thin, the amplitude after traveling through the film can be written

\[
E = E_0 e^{ikz} (1 - i k (1 - n))d.
\]

Comparison of equations 2.6 and 2.7 gives for \( n \):

\[
n = 1 - \frac{\sigma_0 \lambda^2 N_{at} f}{2\pi}.
\]

This shows that more free electrons in the film yield an index of refraction more different from 1, which means that film should have a greater reflectance. This implies that \( \lambda \) may reflect more than most substances in this region because of its high atomic number.

### 2.2 Fresnel Equations

As shown in Figure 2.1, there are two polarizations of light called \( s \) and \( p \).

The \( s \) polarization is perpendicular to the plane of incidence, and the \( p \) polarization is parallel to the plane of incidence. When light reaches a boundary between two media, some of it reflects and some of it refracts. The amount depends on the index of refraction and the incident angle. Augustin Fresnel derived equations that give the fraction of reflected light; the equations are different for each polarization. Using Snell’s law, \( n_1 \sin(\theta_i) = n_2 \sin(\theta_f) \), to combine variables, the equation for \( s \) polarized light is

\[
r_s = \frac{\cos \theta - \sqrt{n^2 - \sin^2 \theta}}{\cos \theta + \sqrt{n^2 - \sin^2 \theta}}
\]

and for \( p \) polarized light is

\[
r_p = \frac{-n^2 \cos \theta + \sqrt{n^2 - \sin^2 \theta}}{n^2 \cos \theta + \sqrt{n^2 - \sin^2 \theta}}
\]

Where \( \theta \) is the incident angle and \( n = \frac{n_2}{n_1} \). The fraction of reflected light of each polarization is

\[
R_s = |r_s|^2
\]

\[
R_p = |r_p|^2.
\]
The combined reflectance is
\[ R = \frac{R_s + R_p}{2} \] (2.13)

For materials in the EUV n is complex making \( \theta \) complex too. Obviously, this \( \theta \) is no longer the physical angle of the refracted beam.

While the Fresnel Equations still hold with a complex index of refraction, it can be helpful to put them in a new form. \( r_s \) and \( r_p \) can be expressed in terms of \( k \). Snell’s Law implies that \( k_z \) is constant from layer to layer, independent of \( n \).

\[ k_z = \frac{2\pi}{\lambda_0} \sin \theta \] (2.14)

But \( k_z \) changes
\[ k_z = \sqrt{\left(\frac{2\pi n}{\lambda}\right)^2 - k_x^2} \] (2.15)

here \( n \) is the index of refraction for a given layer.

\[ r_{s21}^s = \frac{k_{\perp 2} - k_{\perp 1}}{k_{\perp 1} + k_{\perp 2}} \] (2.16)

\[ r_{p21}^p = \frac{n_1^2 k_{\perp 2} - n_2^2 k_{\perp 1}}{n_2^2 k_{\perp 1} + n_1^2 k_{\perp 2}} \] (2.17)

### 2.3 Multilayers

Multilayers use alternating layers of different materials on a substrate to create multiple boundaries. By selecting materials with appropriately contrasting optical constants and adjusting
the thicknesses of the layers, a multilayer can be designed to reflect or anti-reflect a given wavelength of light. Figure 2.2 shows how the light reflects in a multilayer.

![Multilayer Diagram]

Figure 2.2: The light scattering through a short multilayer.

The reflectivity of a multilayer can be calculated with Parratt’s recursion formula.\[5\] This calculates the reflectance at the midpoint of a layer using the Fresnel coefficients at the boundary and the reflectance at the midpoint of the previous layer. The change in phase when the light travels a distance of $\frac{d}{2} \text{ is } \exp i \frac{k_L d}{2}$ so his formula is:

$$R'_2 = \frac{C_2(r_{21} + R'_1)}{1 + r_{21}R'_1},$$  \hspace{1cm} (2.18)$$

where $R_1$ is the reflection amplitude of the previous layer. At the substrate $R=0$, because the light is all transmitted. Starting from the substrate, recursively applying this formula will yield the reflection amplitude of the whole multilayer. $R$ in general is complex, so the actual reflectivity can be obtained by taking the magnitude squared. $R = |R|^2$
Chapter 3

Equipment

In this chapter I explain the equipment used to obtain my data. Figure 3.1 shows an overview of the entire system. The light source, monochromator, and detector have previously been used by my research group to produce, select, and detect wavelengths of extreme ultraviolet light (EUV). The vacuum chamber itself was pulled from a shelf. The base plate was Wes Liffereth's suggestion. I searched for several months to find rotation and translation stages that had the freedom of motion we wanted without higher than needed resolution and that fit our size and cost constraints with no success. As a result, I helped design the rotation and translation stages. I vacuum cleaned and installed the stages including both motor mounts made for the rotation stages. I did all the

Figure 3.1: The Measurement System
wiring from the motors to the drivers to the computer. I also wrote the computer program to control them.

3.1 Light Source

I used a McPherson Model 629 Vacuum UV Hollow Cathode Light Source as my source of EUV. It is described in a paper by Paresce, Kumar, and Bowyer[6]. As shown in Figure 3.2, pipes for cooling water wind through the system. The source must always be cooled when running.

![Figure 3.2: This is a drawing of the Vacuum UV Hollow Cathode Light Source.](image)

The cathode of this plasma lamp can be filled with any of a number of different gases to obtain varied emission spectra. I experimented with filling the cathode to pressures between 150 and 700 \( \mu \text{Hg} \). Plasma formation seemed to occur most easily at pressures of about 500 \( \mu \text{Hg} \). The cathode is actually open at both ends, so a constant flow of gas is required to maintain a certain pressure. A high voltage of about 700 Volts is applied to the gas. If the gas does not immediately ionize, shown by a current registering on an ammeter, the connection between the light source and the monochromator is closed to allow the pressure of the gas in the cavity to build up. Usually a current starts before the pressure reaches atmospheric pressure. If the current does not start, the valve between the light source and the monochromator is reopened to allow the pump on the monochromator to lower the pressure in the cathode and then closed to try again. The current is usually about 850 mA. Once the plasma forms, the valve is reopened, and the pressure gradually returns to about 500 \( \mu \text{Hg} \). This ionized gas or plasma radiates light at characteristic wavelengths of the gas from which it is produced. Many gases can be used in this light source to study many wavelengths.

To choose a gas, find one that radiates the desired wavelength bright enough to be easily detected and far enough away from other bright lines to enable unambiguous selection of the desired wavelength. The wavelengths I used were 1216 Å, Lyman\( \alpha \), the hydrogen (H) transition 2p to 1s, and 584 Å, a dominant helium I (He) line in the extreme ultraviolet (EUV), the 1s 2p to 1s\(^2\) transition. Because of my choice of wavelengths, I used H and He in the light source. Helium also radiates at 1216 Å, but H is 20 times brighter than He there, which makes He a less desirable choice for studying this wavelength. If I had wanted to look at 461 Å, I probably would have chosen neon to fill the cathode.

The largest problem with this light source is its instability. The intensity of the light can change drastically in seconds, but it usually doesn't. Normally the intensity just fluctuates up and
down within a small range. Or the intensity may change almost linearly, giving a hundred thousand count difference between the beginning and the end of a run. The light source is also non-uniform. The differences in intensity from point to point make it important to capture the same part of the beam throughout each run.

This light then enters the monochromator through the entrance slit which is typically 400 μ wide.

3.2 Monochromator

I used a McPherson 225 one meter vacuum ultraviolet scanning monochromator, pictured in Figure 3.3, to select a single line from the plasma emission spectrum. Its near normal incidence reflection grating has 1200 grooves/mm. This concave grating has a radius of 995.4 mm, a ruled width of 50 mm, a 30 mm groove length, and a blaze angle of 1° 33′. The angle of the grating can be adjusted. The incident light from the entrance slit hits the grating at an angle of 7.5° from normal. One wavelength of that light, determined by the grating’s angle, reflects at an angle of 7.5° from normal and travels out the exit slit. This monochromator has a resolution of about 0.1 nm.

Near grazing incident angles reflect the most EUV light and typically the amount reflected drops off as the light approaches normal incidence. Because this monochromator is designed for near normal incident light, only a small fraction of the EUV light that reaches the grating is reflected. My research group does have plans to buy a grazing incidence monochromator in the future.

The monochromator was pumped down to 10⁻⁵ Torr using a Varian Turbo V 550 vacuum pump. Most of the time this pump was left on at its highest speed of 54 kilorevolutions per minute (krpm).

Spencer Olson wrote a LabVIEW program called ‘channeltron’ that we use to move the grating

Figure 3.3: These are the entrance right and exit left slits of the monochromator.
to select a particular wavelength. The same program reads in the counts sent to the computer from the detector. For more about this program see Appendix A.

3.2.1 Detector

I used an Amptektron MD-501 as the detector for the system. It has a 0.1cm² aperture and can handle 4 × 10⁶ counts per second and resolve pulses 250 ns apart with its channel electron multiplier (CEM). The CEM absorbs the incoming EUV photons and sends a 220 ns, 5 V output pulse to the computer for each photon detected. The detector needs 10-15 volts direct current and typically draws 30mA. It also requires an operating pressure of 1 × 10⁻⁵ Torr to protect it from overload. To make sure the detector is not exposed to higher pressures, we have an interlock that will not let the detector turn on if the chamber is not down to pressure. We use the Varian vacuum gauge on top of the chamber to verify the pressure.

Figure 3.4 shows that the detector is particularly efficient at 584 Å and fairly efficient at 1216 Å[8].

![Figure 3.4: Detector Efficiency Curve](image)

3.3 Reflectometer

What follows is a description of the pieces of equipment I developed for this multi-angle reflectometer for this thesis. The ability to measure the reflectance of our samples at many angles is a great addition to our resources because of the extra data it will provide. Also, many applications for multilayers have a particular angle at which the mirror needs to function. We will be able to test those mirrors at whatever angle is required to assure performance without building a new chamber each time.

3.3.1 Vacuum Chamber

The aluminum chamber is 35 cm deep and 50 cm wide with an octagonal base. Each of its eight sides has a hole for a flange. Most of these holes are covered with plexiglass portals. I made a
flange to cover a hole, because its flange was missing. One hole connects the chamber to the monochromator through a short bellows which allows enough movement for fine tuning the alignment after the chamber is connected. Another flange holds an electrical vacuum feedthrough for the wires to the detector and the motors. A gate valve is attached to another side of the chamber with a Varian Turbo-300HT MacroTorr pump on the other side of the gate. The turbo pump and a Varian 300DS oil free scroll pump are used to pump the chamber down to $10^{-6}$ Torr as measured by the Varian model 800 cold cathode vacuum gauge attached to the top of the chamber. An aluminum plate (base plate) with a one inch bolt hole grid in it is on the bottom of the chamber.

The lid of the chamber is bolted to an electric hoist that aids the opening and closing of the chamber. The lid also has a vent valve in it.

3.3.2 Rotation Stages

To the center of the base plate is bolted a post that supports two rotation stages that turn on bushings. One stage is for the sample, to adjust the incident angle at which the light hits, and the other is for the detector. The post is wider at the bottom than at the top to help decouple the stages. The detector is mounted to an arm that turns around the bottom of the post on bushings. The top of the post supports a table top, also on bushings, to which the translation stages are mounted. The table top stage is used to change the incident angle, in half degree steps, of the light coming from the monochromator. The detector arm moves independently to pick up the direct beam or the reflected beam.

The rotation stages needed to have a common center, but turn independently and not be taller than four inches in combined height. No commercial rotation stages could be found to meet this criteria, so special ones had to be designed. These stages are turned by stepper motor driven worm gears that are mounted separately on the side. The motor mount was designed by Matthew Squires and Wes Lifferth. I helped design these stages and Wes built them especially for this project.

Originally, all the stages were run by 1-inch motors from Haydon Switch and Instrument, model HSI z26343-05, but these motors didn’t have enough torque to rotate the rotation stages, so they burnt out. We then measured the torque required by our stages and found that although normally 6 oz-in was all that was needed, there were spikes of up to 35 oz-in. The worst of these spikes were due to the translation stages’ motor wires catching on the pin hole in the chamber. We attached these wires to a post to hold them out of the way with enough slack not to compromise motion.

When the motors died, Matthew Squires made a new flange with mechanical feedthroughs to turn the two rotation stages. Two complete revolutions of the handle on each feedthrough turned the stage 4.5 degrees. For reliability, data measured with these mechanical feedthroughs was taken in 4.5 degree increments.

Now the rotation stages are turned with motors from Eastern Air Devices Inc. model ZB17GBK-10. They are rated for 13.9 oz-in. The new motors are larger, so Matthew and Wes redesigned the motor mount. The old mount put one motor above the other spaced so that the worm gear turned by the motor shaft lined up with the gear for the stage. The new motor mount puts the motors on different sides to make room for the larger motors, so the shafts cross at a right angle and the worm gears connect to the gears at different point of the circle.
3.3.3 Translation Stages

Figure 3.5 shows the translation stages that are used to scan the sample’s surface in millimeter steps and to move the sample out of the way for a direct source intensity reading. Each translation stage consists of 16 cm of track, a 7 cm long carriage that glides on ball bearings, an adapter plate which attaches the motor shaft to the carriage, and a mounting bracket that clamps to the track to hold the stepper motor. As mentioned before HSI z26343-05 motors move the carriages. The sample is attached with metal clips to the plate on the vertical, top stage. Each piece of equipment had to be cleaned before we put it in the chamber. Figure 3.6 shows me cleaning the translation stages.

I found a translation stage set up similar to the one we have now in Dr. Peatross’ lab. I ordered 400 mm of track from THK Linear Motion Systems and had Wes saw it in thirds. Dr. Peatross donated the vertical adapter for the z stage from his set up since he was taking his apparatus apart to use for something else. We adapted the motor mount brackets from the design used on his to fit our motors and track.

3.3.4 Motor Wiring

The four wires from each stepper motor are soldered and clamped to connector pins. The joining connector pins are attached to the wires in two multiconductors that are soldered to the vacuum side of the electrical feedthrough. We decided to use an old electric feedthrough that Scott Daniel had to save a hundred dollars. When we first placed it in the wall of the chamber, the chamber pumped down well. After I had soldered the wires to the feedthrough, however, we found a leak in the feedthrough. Jason Flint removed the wires, extended the pins, filled in between the pins with vacuum putty to fix it, and re-attached the wires.

Outside the chamber these wires connect to drivers made by HSI. We chose these drivers because they cost half of what most stepper motor drivers cost. Each driver is in a shell that is
mounted to a metal box. I found the box and drilled holes in it to mount the drivers for organization and protection. The drivers are powered by a 12 Volt power supply. This power supply came from Scott Daniel. Michael Hales and I put it in a metal box and soldered wires to pins we put on the outside. The direction input pin for every driver is wired to D1/O0 on the data acquisition (DAQ) board. Each driver is wired to its own pin for a step pulse: x is D1/O1, y D1/O2, z D1/O3, θ D1/O4, and detector D1/O5. Each step pulse wire is twisted with a wire connecting ground on the DAQ board to the driver the step pulse goes to. These wires allow the stages to be computer controlled as described in 3.3.5.

### 3.3.5 Computer Control

The stepper motors are computer controlled via a National Instruments PCI-MIO-16E-4 data acquisition (DAQ) board by a LabVIEW program called ‘stage control’, which Cort Johnson and I wrote to signal the motor drivers. Its code is in Appendix B. This program has three inputs. The first input is a ring control, to select the motor of the stage you want to move. The second input is a numeric control, for entering the distance you want that stage to move in millimeters or degrees, depending on whether you selected a translation or a rotation stage. The third input is a boolean switch for the direction you want the stage to move.

The ring control assigns a number to each entry. When a stage is selected, it sends the number to a case statement. The case statement holds two important values, (1) the empirical number to multiply the entered distance by to find the needed number of steps the motor needs to take (step factor), and (2) the channel name for the pin on the DAQ board the clock pulse will be sent from.

The distance entered in the numeric control is multiplied by the step factor and the result become the number of times a for loop is executed. This for loop uses sequence frames to generate pulses by alternately sending high and low signals to the pin on the DAQ board specified by the
channel name 1 millisecond apart. Originally, the wait time was longer at the beginning, decremented until at full speed, and incremented near the end of the motion to control acceleration, but I found that the longer wait times weren’t needed, so this complicated ramping was taken out.

The direction boolean is sent out digital line D1/O0. False is clockwise for the rotation stages and out of the beam for the x stage. This boolean is also the selector for a case statement that hold all the above code. If false is selected, the stage is actually sent 20 extra steps, and at the end of its motion it is sent back the other way 20 steps to avoid repeatability problems due to slop in the gears.

I started writing an automation program with Matthew Squires that would use ‘stagecontrol’ and ‘count’(a sub vi from ‘channeltron’) to do a whole data run at specified angles and check the source between specified angles without stopping and write the data to a file. I didn’t finish it, but Matt recently did.
Chapter 4

Procedures

4.1 Alignment

We set the body of the chamber on a metal table with adjustable height legs and adjusted the legs so that the level read level in either direction. We similarly leveled the base plate inside the chamber with screws threaded through the plate to reach the chamber floor as shown in Figure 4.1.

![Image of leveling screws](image)

Figure 4.1: Here the base plate is being leveled by adjusting the screws it rests on.

We attached the chamber to a monochromator as straight as we could by eye with a bellows nipple, as shown in Figure 4.2. Then we aligned it to within 1 mm by shining a laser through the entrance slit of the monochromator with the grating turned to zero to let all the light go straight
out the exit slit and nudging the chamber over so that the laser shone through crosshairs scratched into the center of the plexiglass plate opposite the nipple. We centered the base plate by attaching the center post of the rotation stages to the center of the plate, hanging a plumb bob through the laser's path halfway across the chamber, and moving the plate using the mechanism in Figure 4.3 to center the post under the plumb bob.

Figure 4.3: The screw threaded through this block pushes on the chamber to adjust the plate.
4.2 Venting the chamber

The following procedure is used to vent the chamber:

1. Close the flap valve between the monochromator and the chamber.
2. Close the gate valve, but do not lock it into place.
3. Turn off the detector power.
4. Turn off the Cold Cathode vacuum gauge for the chamber.
5. Turn off the Turbo pump to the chamber.
6. Wait five minutes for the turbo pump to spin down.
7. Turn on the bottle of compressed nitrogen behind the monochromator.
9. Place the nitrogen hose near the top of the vent valve in the top of the chamber lid, and open the valve.
10. It will take 3-5 minutes for the chamber to be vented.
11. Test the venting state by putting your finger over the valve. If it is sucked down, the chamber is not fully vented.
12. When venting is complete, turn off the nitrogen gas, but leave the valve open on the top of the chamber. This is a reminder the chamber is vented, and it also ensures the chamber is completely vented.
13. Turn on the hoist and make sure no part of the hoist will bump the chamber.

4.3 Sample Insertion

The following steps are used to insert a sample:

1. Vent the chamber using steps in Section 4.2.
2. Put on gloves.
3. Remove the previous sample if there was one. Always remember to use tweezers or hold sample by the edge.
4. Place the new sample under the spring clips on the vertical stage.
5. Make sure the back of the sample is fully touching the sample mount.
4.4 Pumping Down the chamber

The following steps are used to pump the chamber down to vacuum:

1. Close lid on the chamber.
2. Close the vent valve.
3. Open the gate valve.
4. Turn on the Turbo fan pump.
5. Wait until the turbo fan controller display says “Normal Operation”.
6. Turn on the Cold Cathode vacuum gauge.
7. Open the flap valve between the monochromator and the chamber.
8. Monitor the pressure until it is below $10^{-5}$ Torr.

4.5 Measurement Process

The following steps are used to measure a sample without an automated program:

1. Insert a sample using the steps in Section 4.3.
2. Pump down the chamber using the steps in Section 4.4.
3. Record the base pressure of the monochromator and chamber.
4. Once the pressure is below $10^{-5}$ Torr, turn on the detector.
5. Turn on the driver’s power supply.
6. Open the stage control program on the desktop.
7. Turn on the laser.
8. Align the sample surface with the laser by moving the x stage until the laser grazes the surface.
9. Turn on the plasma by opening two valves on the tank of helium and adjusting the needle valve so that the pressure gauge reads about 500 microns of mercury, turning on the high voltage, and then closing the valve between the hollow cathode lamp and the monochromator until a current is obtained.
10. When a current is obtained, reopen the valve between the hollow cathode lamp and the monochromator.
11. Record the voltage, current, and operating pressure of the gas, monochromator and chamber.
12. Move the sample back 15 mm, unless this has already been done.
13. Open the channeltron program on the desktop.

14. Enter the current monochromator wavelength and hit OK.

15. Adjust the monochromator grating to the wavelength you want by entering the new wavelength in the box on the lower right and clicking "GoTo".

16. When the program is done, turn the top knob to continuous and click "Take Data".

17. The counts from the detector will appear in the graph and the indicator above it.

18. Collect intensity readings from the detector while slowly moving the detector through the angles across the beam to find the peak intensity.

19. Set the angle at which the peak intensity is detected as the zero angle and record the maximum direct intensity for the beginning of the run.

20. Move the sample up 15 mm.

21. Move the mirror through the desired angle, move the detector through twice that angle scanning slowly to find the peak for that angle, and record the intensity.

22. Repeat the previous step for all desired angles.

23. Move the sample to the zero angle and back 15 mm.

24. Move the detector back to the zero angle and record the direct intensity for the end of the run.

25. Turn off the detector and the driver’s power supply.

26. Shut off the light source by turning off the high voltage and the gas.
Chapter 5

Data

To demonstrate the proper operation of the reflectometer described in this thesis and its usefulness, I will present three different sets of data. First, I will present some crystalline silicon reflectance data obtained using this reflectometer and compare it to expected reflectance based on the known optical constants. Because these optical constants are well established, this is an effective test of the reflectometer. Second, I will present reflectance data from a uranium-silicon (U/Si) multilayer, show how it disagrees with the current model in a circumstance where x-ray diffraction measurements reveal nothing about the sample. This shows how my chamber can be a source of new and useful data. Third, I will present reflectance data obtained from a Al/MgF2/TiO2 mirror being designed by my research group for a Mars Mission.

5.1 Silicon Data

The following crystalline silicon data was taken by Matthew Squires using the automated program. Data is sent from the detector continuously at evenly spaced intervals. The numbers in the time column are actually the number of readings taken so far. They can be helpful in normalizing the data. While older data showed reasonable agreement, this data shows how nice the agreement is with extra light source information since the variation in light source intensity can be a major source of error. Data from measurements of crystalline silicon at 2.5 degree angular increments from zero to 82.5 degrees is reported in Table 5.1. The counts at zero are actually straight through the chamber with the mirror holder moved out of the beam.
<table>
<thead>
<tr>
<th>Angle (degrees)</th>
<th>Counts (number)</th>
<th>Time (seconds?)</th>
</tr>
</thead>
<tbody>
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<td>95817</td>
<td>52266</td>
</tr>
<tr>
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</table>

Table 5.1: This is raw reflectance data taken at 1216 Å of a crystalline silicon substrate that oxidized, so it has 10 to 20 Å of SiO₂ on top.
5.2 Uranium-Silicon Data

This mirror was designed to be about 50 Å of U on a Si substrate capped with about 125 Å of
sputtered Si. The Si on the top undoubtedly oxidized. This oxide could be thicker than on the
crystalline silicon sample because of the amorphous structure of the sputtered silicon. Exact
thicknesses are not known because sputtering rates are not exact and the x-ray diffraction
measurement we thought would help determine the thicknesses revealed no structure in the bilayer.

Data from measurements I took of this sputtered uranium-silicon (U/Si) multilayer at 4.5
degree angular increments from zero to 76.5 degrees is reported in Table 5.2. The counts at small
angles are artificially low because the sample was small. With the sample at an angle of 5 degrees,
a 2 mm beam hits a surface about 23 mm wide. This sample was about 16 mm wide and the
alignment at the time this data was taken was not perfect, so I lost a huge fraction of the beam at
near grazing angles.

<table>
<thead>
<tr>
<th>Angle (degrees)</th>
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</thead>
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<td>2.7</td>
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</tbody>
</table>

Table 5.2: This is raw reflectance data taken at 584 Å of a uranium-silicon bilayer on a silicon
substrate.
5.3 Aluminum Capped Magnesium Fluoride-Titanium Dioxide Data

The European space Agency is sending equipment to Mars to study the possibilities of life there. One satellite is going to study the interaction of Mars’ atmosphere with the solar wind. We were asked to develop a mirror for this satellite. It needs to reflect neutral particles, but anti-reflect Lyman-α, 1216 Å which is considered noise for this project. Data from measurements of a Al capped MgF₂/TiO₂ mirror being developed for a Mars mission are reported in Table 5.3.

<table>
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<th>Counts (thousands)</th>
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</thead>
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<td>36.7</td>
</tr>
<tr>
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<td>99</td>
</tr>
</tbody>
</table>

Table 5.3: This is reflectance data from an Al/MgF₂/TiO₂ mirror at 1216 Å.
Chapter 6

Analysis

The normalized reflectance is obtained by dividing the data by the average of the two direct intensity readings. This reflectance is then compared to a model in IMD. IMD is a computer program written by David Windt to model the optical properties of multilayer films[9]. It uses a database of optical constants, including user defined optical constants, for each layer and the Fresnel equations discussed in 2.2 to find the reflectance. It can also calculate the transmittance, absorbance, phase shifts, and/or electric field intensities as a function of any parameter such as incident angle, layer thickness, or wavelength.

6.1 Crystalline Silicon

The optical properties of crystalline silicon have been studied extensively, and EUV reflectance values for crystalline silicon are well known.[7] Some of the Si on the top of our sample oxidized to SiO2 because it was exposed to air. The group’s previous experience with measuring Si samples leads us to assume an oxide thickness of about 15 Å. No measurement of this thickness was tried because both x-ray diffraction and ellipsometry have proven to unreliable in determining the thickness in this case. Comparison of the data Matthew Squires obtained with our apparatus with the known reflectance values shows good agreement as shown in figure 6.1. This confirms that the apparatus operates properly to obtain useful information.

6.2 Uranium-Silicon

Figure 6.2 shows that there is a significant difference in the reflectance of U from the model. Actually determining the optical constants of U is beyond the scope of this thesis. I leave to others the opportunity to definitively determine U optical constants in this region. My U/Si data has enough features in it that should make it possible to eventually figure out what is there. The fact that x-ray diffraction (XRD) is non-descript means that the reflectometry is especially critical. The closest optical constants to 584 Å for U are about 200 Å away. This explains why the IMD values for U/Si are significantly different from the values obtained in the reflectometer.
Figure 6.1: This is a plot of the Si data taken at 1216 Å and a model of the silicon including a 15 Å layer of SiO$_2$ on top because the Si has been exposed to air.

### 6.3 Aluminum Capped Magnesium Fluoride-Titanium Dioxide

The model that led us to make this mirror and the measured reflectance are shown in figure 6.3. This much higher reflectance shows importance and ability to produce new and useful normalized reflected data. The fact that the measurements disagree with the IMD models is good news rather than bad. It shows how critical these kinds of measurements are and why we need angular measurements to really tell what is doing on.
Figure 6.2: Here reflectance models of several reasonable thicknesses of SiO₂, Si, and U are plotted with our data at 584 Å. The data shows significant differences that cannot be explained by reasonable changes in the thicknesses of the layers in the model, although those changes do effect the reflectance.

Figure 6.3: This is a reflectance plot of the Al/MF₂/TiO–2 with the model that inspired its making at 1216 Å. The reflectance is much higher than predicted which made it an unusable choice to anti-reflect Lyman-α.
Chapter 7

Future Work

This project leaves a huge amount of work to be done. The first thing I would recommend doing is completing a computer automation program. This would make collecting data much easier and faster and avoid human mistakes like forgetting to record a data point before moving on to the next angle. The next thing would be to continue studying uranium to actually determine its optical constants in this region.

Of course there are many other materials that could be beneficially studied using this equipment. There are immediate plans to study scandium, ruthinium, carbon (diamond and graphite), and aluminum in this reflectometer.
Appendix A
Appendix B
Bibliography


