Introduction to Ellipsometry
Laboratory 1

Objectives: During this laboratory, you will become familiar with the ellipsometer as well as with the ellipsometry software. You will also learn how to calibrate the angle of reflection and make measurements on a model Si/SiO_2 system. Using this system, you will investigate how refractive index varies with wavelength.

Experiments: In this lab, you will begin to make measurements and calculations using the J. A. Woollam M-44 ellipsometer. The software uses the Fresnel equations along with the theory of multiple reflections to calculate parameters such as refractive index, thickness, and absorption coefficient. You can also use the ellipsometry software to calculate delta (Δ) and psi (Ψ) from a given model. The calculation of these quantities has been covered to a large extent in the short course on ellipsometry.

Here is how to get started.

1. Power up. The computer and ellipsometer electronics should be off during this procedure. Lighting the Xenon lamp requires a lot of power and can damage sensitive equipment. Turn on the power supply to the lamp and then ignite the lamp. Once the lamp is on, turn on the ellipsometer electronics and the computer.

Note that the ellipsometer works as described in the short course. Light from the Xenon lamp is collimated with the optical fiber and then reflected from the sample at a fixed angle. The detector contains a diffraction grating and a diode-array detector so that 44 wavelengths of light can be analyzed simultaneously. The ellipsometer also contains a rotating polarizer. The intensity of the light is recorded as the polarizer rotates and then the mathematics described in the short course are used to extract Δ and Ψ.

2. Initialization and Calibration. Carefully place the silicon wafer standard on the sample stage and start the WVASE program. Go to the hardware pull down menu and select *initialize*. During initialization, software looks for the instrument configuration, the rotating analyzer is brought up to speed and data acquisition is synchronized with the rotating analyzer. Next, in the hardware menu, select *align*. The ellipsometer contains a 4-quadrant detector. To insure that the sample is flat, the signal to each of the quadrants should be identical. Adjust the tilt of the sample stage so that the red cross is in the center of the cross-hairs as viewed through the eyepiece. This alignment step can be completed only under the condition that the sample is flat. Now go to the hardware menu and select *calibrate*. This calibration is necessary to determine the exact positions of the input and analyzer polarizers and the relative attenuation of the ac signal relative to the DC signal. (See the short course on ellipsometry for details and an explanation of the specific parameters.)

3. Calibration of the incident angle. Although the ellipsometer is built to precise standards and the angle of incidence should be 75.0°, to know this angle precisely requires calibration. We do this with the standard silicon wafer included with the instrument. The wafer consists of a well defined SiO_2 layer on Si. As you might guess, this is probably the best-characterized ellipsometric system because of its relevance to electronics. First we need to set up Si/SiO_2 as our model system.

a. Go to the model window and select *adlayer*. Select the file named si-jell.mat. You just said that the bottom layer of your sample is silicon. Make sure that you are not fitting any optical parameters in this file and that the thickness is on the mm scale. We are assuming that the literature values of n and k for silicon are accurate.
b. Select *adlayer* again and select the file sio2.mat. Now you have told the software that your system consists of a layer of SiO_2 on Si. In the model window, select the SiO_2 layer and put in 250 Å for the thickness. Check the fit box by thickness. Do not fit the optical constants. This indicates that, after we make a measurement, we will fit the data to this Si/SiO_2 model using the thickness of SiO_2 as the adjustable parameter.
c. Now go to the *hardware/experimental_data/angles* and click the 'Fit' box. This says that along with thickness, you will use the angle of incidence to fit the data.
d. In the *hardware* window, go to experimental data and click "acquire single scan". This gathers your ellipsometric data.
e. In the *fit* window, select *fit/normal fit*. You should now see the thickness of the Si/SiO_2 film along with the angle of incidence. The Δ and Ψ data were fitted by varying the angle of incidence along with the film thickness. If the film thickness isn't around 250 Å, something is not right.
f. Repeat the process to see how precisely you can determine the angle of incidence. Try a few positions on the wafer.
g. Make this the angle of incidence for future experiments.

What you just did was to assume that the literature optical constants for Si and SiO_2 are correct. Then you could fit both the angle of incidence and the thickness of your sample. You can make this determination at 44 wavelengths so it should be quite precise. Look at the graph window to see how good the fit was.

Measurement of the Optical Constants of SiO_2

1. Now that you know a little bit about the ellipsometer, you can start fitting optical constants. Go ahead and make some measurements of the thickness and optical constants of SiO_2. Print out the optical constants and return them with your lab report. You do this by going to the model and deciding to fit **n** for the SiO_2. Just fit refractive index, n, and thickness. The extinction coefficient, k, should be zero for this transparent film. Make sure that you are not fitting the angle.

2. Refresh the optical constants of SiO_2 by reloading the layer. Go to the model window and delete the SiO_2 layer. Then add it again. Use the thickness that you previously measured, but don't fit it again. This time, fit the n and k values of the base Si layer. How close are the values to the literature values in the original file.

Measurements on Gold Slides

A gold film is a simpler system to work with than a monolayer-coated slide. The reason for this is that there is no surface film (at least in principle).

1. Rinse a gold slide with EtOH and dry it with N_2. Measure its optical constants. Write down the values at 633 nm. Next, clean the slide in piranha solution for 30 seconds. Rinse copiously with millipore water. Prepare 100 mL of the piranha solution by mixing H_2O_2 with H_2SO_4 in a 1:3 ratio. **Use extreme care when working with piranha solution. You must wear a lab coat or apron, gloves and a face shield.** Mount the substrate in a plastic hemostat being careful to grab only a corner. Next measure the optical constants. Write down the values at 633 nm. Also note the Δ and Ψ values.

SAVE THESE SUBSTRATES IN SCINTILLATION VIALS FOR LABORATORY 2

In your report, describe the reasons for the differences between the sample before and after piranha cleaning.