Determining the Cause of Metal Oxide Film Growth Due to Exposure to Vacuum Ultraviolet Light

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ABSTRACT

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Brigham Young University's Thin Films Research Group has been studying the exposure of reactively sputtered yttrium (Y_2O_3) and scandium (Sc_2O_3) oxide films to a 7.2 eV vacuum ultraviolet (VUV) excimer lamp. Our results show that a dramatic increase occurs to the film when it is subjected to the UV light. In some cases the film swells to nearly seven times the original thickness, which is an unexpected result. The light source used in our research is similar to those used for cleaning in the semiconductor industry. Therefore, it is important to determine the cause of growth. It has been determined that reactively sputtered samples can exhibit film growth, whereas oxide films from air oxidation of metal films do not. We have experimentally determined that the film growth is not connected to new deposition or oxidation of the silicon substrate. The growth can then be attributed to change rendered to the sample itself. Pressure from water vapor within voids does not appear to be the cause—neither does surface interaction with gas species (ozone and atomic oxygen) by themselves. Evidence suggests that atomic oxygen and/or ozone created by the lamp with VUV photons present at the same time are altering the structure of our samples and are driving the film growth.

Keywords: yttrium, scandium, film growth, vacuum ultraviolet lamp, reactively sputtered

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A special thanks to my parents and sister for their constant love and support through this process. I could not have done it without you.

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Chapter 1

Introduction

1.1 Report Overview

The purpose of this report is to identify the factors contributing to the growth of metal oxide films (Y_2O_3 and Sc_2O_3) when exposed to 7.2 eV vacuum ultraviolet (VUV) light. The following section explains the relevance of this research. Chapter 2 is the bulk of the thesis, covering the experiments and interpretations. Sections 2.1-2.2 detail our deposition system and process and the discovery of film growth due to lamp exposure. A history of initial theories tested on Y_2O_3 are introduced and discussed in section 2.3. Current theories using Sc_2O_3 are covered in section 2.4 and 2.5. Chapter 3 details our plans on future research. The conclusions that we have drawn from our data is contained in Chapter 4. Appendix A contains supplementary information on our data collection process, ellipsometry.

1.2 Application of Our Findings to Semiconductor Research

The vacuum ultraviolet lamp used in our research is specifically designed to clean hydrocarbons off of sample surfaces. Our ultraviolet (UV) light used was generated by a 7.2 eV excimer lamp manufactured by Resonance LTD. in Barrie, Ontario, Canada. It is a demo of the REX-4 model. This type of lamp is used in the semiconductor industry for the purpose of cleaning the surfaces of materials deposited onto silicon substrates. The expansion of any metal oxide during cleaning would be unacceptable. This could alter the properties of the individual components, causing the device to fail. Yttria (Y_2O_3) has recently been considered as a possible replacement for the primary gate dielectric material in metal oxide semiconductor devices [1]. Thus it is important to understand how and why yttrium, scandium, and possibly other metals react to the cleaning process.

Chapter 2

Experimental Methods

2.1 Deposition and Film Measurement

The metal-oxide film samples used as part of our research were prepared by depositing metal films onto silicon (Si) wafers using a reactive sputtering process employing a high-vacuum sputtering system. In this procedure, blank silicon wafer substrates were placed into a high-vacuum chamber. The chamber is then pumped down to pressures on the order of 10^{-6} Torr. These low pressures allow greater control of the atmosphere and ensure the chamber is free of unwanted molecules, which increases the purity of the deposited layer. It also allows molecules to move more freely within the system [2].

A low chamber pressure also creates a longer mean free path for the metal oxides during deposition. Mean free path is the average distance a molecule travels before experiencing a collision with another molecule or atom [3]. This means that the longer the mean free path, the more likely a molecule is to make it from the target onto the surface of the sample Si wafer during a run. Figure 2.1 shows the deposition system used by our group. New additions made to the chamber in the fall of 2010 now allow use to use up to three sputtering guns at a time. This means alternate layers can

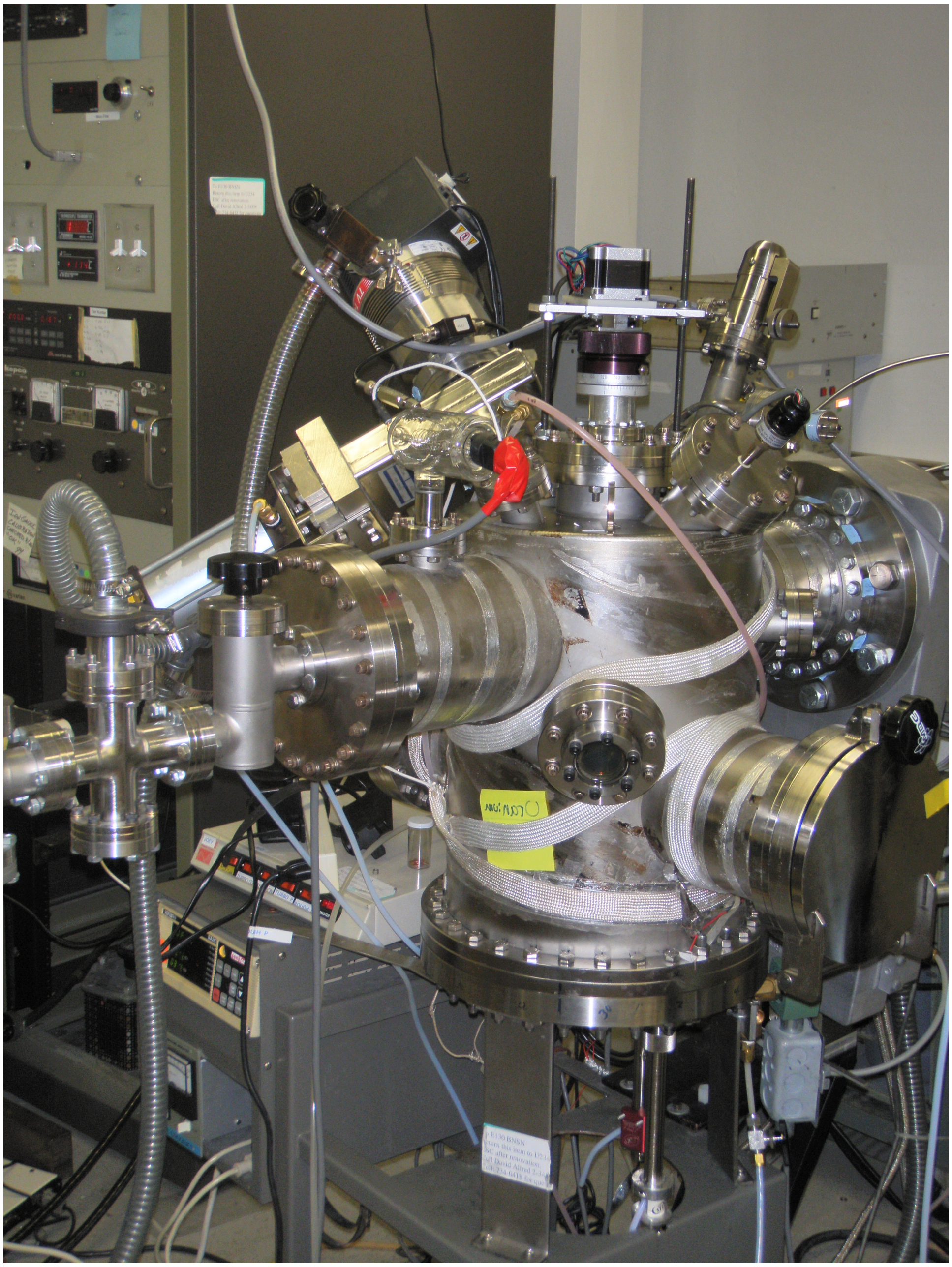


Figure 2.1 The vacuum system used to sputter metal oxide samples.

be deposited without breaking the vacuum.

The sputtering process utilizes the formation of a plasma. Our plasma is created by introducing argon gas into the chamber in the presence of a large negative potential near the surface of a sputter target. Ions are accelerated by the potential so they bombard the surface of the metal target material, yttrium or scandium, with high energy. These high energy collisions eject particles from the target material, which eventually settle in the silicon substrate [4]. Electrons sustain the plasma. Strong rare earth magnets are used to produce a hoping motion of electrons near the target surface to increase their path length so the plasma can be sustained at low pressures.

In order to produce an oxide during the process, a small amount of oxygen flows into the system. We usually run with about 6-14% oxygen (0.16-0.37 mT) and balance argon. Total system pressure is 2.5 to 2.8 mT. Reactive sputtering refers specifically to creating a metal oxide film from a metal target. The oxygen gas interacts with the yttrium or scandium to form Y_2O_3 and Sc_2O_3 [2].

The resulting samples were measured for resulting thickness and optical constants using ellipsometry. For more on ellipsometry see Appendix A. Ellipsometric data was measured and analyzed using WVASE32^{TM} software. Multiple models were created to fit the spectroscopic data [5].

2.2 History of Observed Film Growth

In previous surface studies by the thin films optics group, cleaning techniques included exposing Si wafer surfaces to a vacuum ultraviolet (VUV) lamp that generates 7.2 eV light [6]. The lamp is used to clean hydrocarbons off sample surfaces before measuring thicknesses.

The VUV lamp generates very short wavelength (high energy) photons. These high energy photons can only propagate a short distance, $\sim$1 cm in air, before significant absorption occurs. The surrounding oxygen in the air is atomized, requiring experiments to be conducted in a fume hood to extract the toxic ozone byproduct. The oxygen atoms and ozone molecules strike the surface of

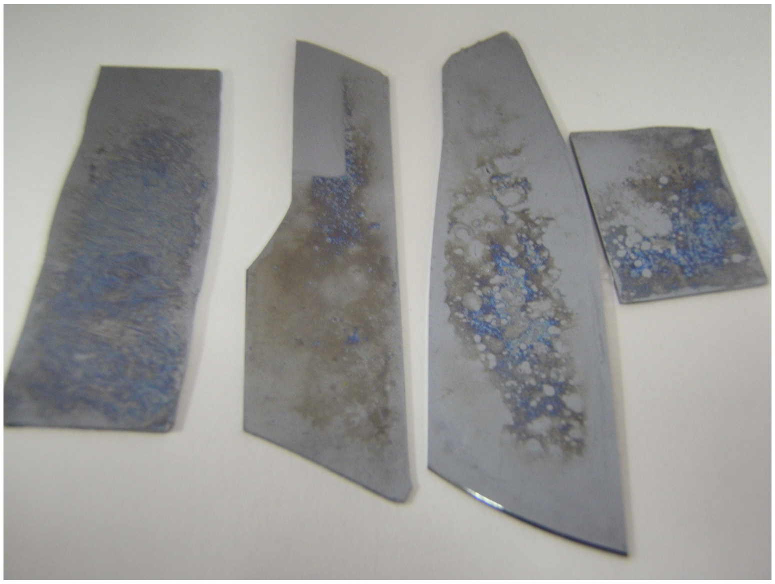


Figure 2.2 Yttrium samples after being exposed to the VUV lamp.

the sample, either breaking up the hydrocarbons or oxidizing them completely into CO_2 and H_2O. These smaller molecules can be removed easily because of their higher vapor pressure [7].

We first noted that something was different in cleaning a Y_2O_3 film. After a five-minute exposure under the lamp, the sample surface had noticeably changed in its appearance. For unknown reasons, a Y_2O_3 film acquired a dark hue, black mottled coating, and a brownish hue as can be seen in Figure 2.2. Ellipsometry revealed that the sample had become notably thicker. Before exposure, the film was measured to be 24.7±0.1 nm. After the five minute VUV photon treatment it was measured to be 31.1±0.1 nm thick. This amounted to a 25.6% increase in thickness. Since the removal of hydrocarbons would produce a thinner sample, further testing was initiated to find what caused the film to grow.

2.3 History of Previously Tested Theories on Yttrium

In the following sections I will summarize the work conducted by my colleagues Devon Mortenson [2] and Kristal Chamberlain [5].

2.3.1 Theory 1: Film Oxidation

One theory proposed that the yttrium sample was not fully oxidized, but was becoming so under the UV light. As previously explained, such particular samples were prepared through a reactive sputtering process. This means we used a yttrium target, rather than a yttrium oxide target, and allowed oxygen to flow into the system during deposition. The VUV lamp atomizes in the air, meaning there is an abundance surrounding the sample during exposure. If the yttrium film was not already fully oxidized, it could react with this extra oxygen though diffusion of oxygen into film might limit growth.

This theory was proven to be inadequate by calculating the total thickness from a completely unoxidized yttrium sample to a completely oxidized sample, while expanding only upwards. Knowing the thickness and the density of yttrium, it was calculated that the upper bound of expected film growth could be 3.3 nm. The actual growth (6.3 nm) exceeded, by a factor of two, the maximum value of the theoretical [2]. This theory was further expelled when subsequent testing showed that the sample thickness continued to increase upon even longer exposure to the UV light—a total of 82.5±0.34 nm after 35 minutes under the lamp, nearly eighteen times the upper theoretical limit [2]. While it could account for a small percentage of growth, the theory does not explain enough of the change to be acceptable.

2.3.2 Theory 2: New Deposition

The second theory put forward was that the VUV lamp somehow was depositing new material onto the sample. It could not be yttrium, since it is an element not found in the atmosphere, but we supposed organic vapors were present and polymerized rather than etched. As previously stated, the high energy photons from the lamp can only propagate a short distance before they are absorbed. To increase etch rate, we placed the sample as close as possible to the lamp by creating a supporting base of glass microscope slides. On average the stack is composed of 15 to 18 slides. We wondered whether the slides could be within range of the lamp and could they be contaminated so that the VUV photons were knocking molecules off them and depositing the new material onto the sample film. Alternatively, could the atmosphere in the hood have some contaminate that lead to deposition?

Testing this theory was straightforward. Since this theory ascribes growth to the lamp and glass slides or hood atmosphere, growth should occur regardless of the sample being used. A blank silicon substrate was subjected to the same lamp treatment and its ellipsometric thickness measured. A slight decrease in thickness was recorded [2]. This type of behavior would be expected if the VUV lamp was only cleaning the hydrocarbons off the surface as it was designed to do. The VUV lamp was therefore not depositing material onto samples from the surrounding environment.

2.3.3 Theory 3: Oxidation of Substrate

Since it appears that nothing was being added to the sample, the remaining possibility required that what is already in the film was somehow being altered. One suggestion was that the Y_2O_3 was catalyzing the oxidation of the silicon wafer underneath the film.

Silicon readily oxidizes given a kinetic pathway, since SiO_2 is thermodynamically more stable. It has been found that SiO_2 can be grown by exposing Si to ozone at low temperatures [8].

This hypothesis was ruled out in the following way. The sample was placed into the furnace. If the new growth was the very stable configuration of silicon oxide , the furnace would not cause the silicon to revert back to an unoxidized state and the thickness, therefore, would remain the same.

A sample that had been previously exposed to the VUV light with a thickness peaking at 98.3±0.2 nm was subjected to the furnace for two hours at 500^{o}C. Upon removal it was immediately measured again. The thickness was found to have decreased 83% and now measured 16.9±0.02 nm [2]. This was a significant change in thickness upon heating. The silicon oxidation could not give proper explanation to the behavior of our sample.

2.3.4 Theory 4: The Popcorn Effect

We have reasoned that the deposition process does not produce fully dense film. Instead, some void formation occurs. Within these voids water could possibly settle. The flux of VUV photons might be sufficient intensity to boil the water if absorbed within the film. This could create an effect similar to popping popcorn, where internal pressure from the water pressure causes an outward expansion. This expansion could cause the surface to grow in thickness.

We placed a sample into a Lindbergh furnace for one hour at 500^{o}C, then immediately placed the dry film in a nitrogen- rich atmosphere glove box and subjected it to a ten-minute treatment of UV light . If water was the cause of expansion, then baking out the water should hinder the growth compared to previous tests. However, measurements showed the sample increased in thickness from 21.10±1.0 nm to 93.35±3.0 nm [5]. This film growth is large enough to cast doubt on the theory that water expansion caused the increase.

2.4 Early Theories Involving Scandium

At this point in the research we were desirous to know if VUV film growth is a feature of reactively sputtered Y_2O_3 only, or if other metal oxides might experience growth when exposed to UV light.

It was determined that it would be beneficial to test scandium oxide. Scandium lies just above yttrium on the periodic table of elements, and therefore shares some characteristics. Having a target at our disposal, reactively sputtered Sc_2O_3 samples were produced for testing.

2.4.1 Furnace Oxidized Scandium

In our previous tests, reactively sputtered samples were used to study the film growth. As stated earlier, this is accomplished using a metal target while oxygen flows into the vacuum chamber creating an oxide. We decided to first test a sample of Sc_2O_3 that was produced by furnace oxidization of Sc, rather than produced by reactively sputtering. This means the sample was created by sputtering a film of scandium onto a silicon substrate, then baking it in a furnace for seventeen hours at 550^oC. This insured that the sample would be fully oxidized.

It was found that exposing the furnace oxidized scandium to the VUV light surprisingly did not cause the film to swell. Our data seems to suggest that in order for the sample to grow it must be made using the specific method outlined in section 2.1.

This sample was revisited one year after its original testing was conducted to see if any changes would occur now that the sample had aged. During this time the sample could have oxidized more, absorbed water, or formed a layer of hydrocarbons on the surface. It was found that the apparent thickness had decreased after the Sc_2O_3 was exposed to the VUV lamp. It was found that the thickness had decreased. This would be expected for hydrocarbons being removed. We concluded that reactively sputtered samples can exhibit film growth, whereas air oxidized scandium does not.

When we tried a reactively sputtered sample, we found results similar to the Y_2O_3. See below. We therefore used reactively sputtered samples for the remainder of testing.

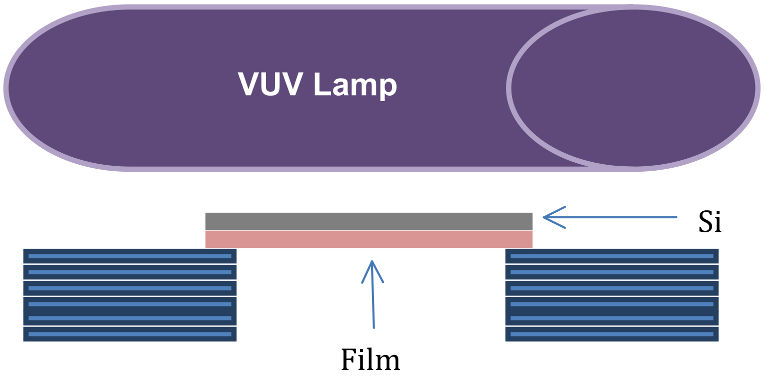


Figure 2.3 The structure set-up to test the effects of ozone to the film surface.

2.4.2 Necessity of Gas Contact Scandium Oxide

A further experiment was completed during which a sample was placed under the VUV lamp in such a way that the VUV photons could not fall directly on the sample surface, but the sample surface was exposed to the atmosphere. This was done by creating two structure towers from glass microscope slides, 15 slides high, and suspending the sample upside down over the gap like a bridge. This bridge structure would allow ozone, formed by the lamp used in air as outlined in section 2.3.1, and atomic oxygen to have contact with the scandium film. The photons , however, are unable to strike the surface and are therefore unable to effect the film. Figure 2.3 illustrates the experiment set-up.

The sample was initially measured to be 12.5±0.01 nm thick and was then subjected to UV light for twenty minutes. Remeasurement showed the sample to be 12.75±0.01 nm thick. This result seems to suggest that reactive specks in the gas alone are not sufficient, but photons are needed to interact with the surface to achieve film growth.

To confirm these initial results, the sample was exposed to VUV light using the same shielding technique for six additional time increments of ten-minutes. The Sc_2O_3 was exposed the UV light for ten minute increments and then measured. Figure 2.4 shows the results from the experiment.

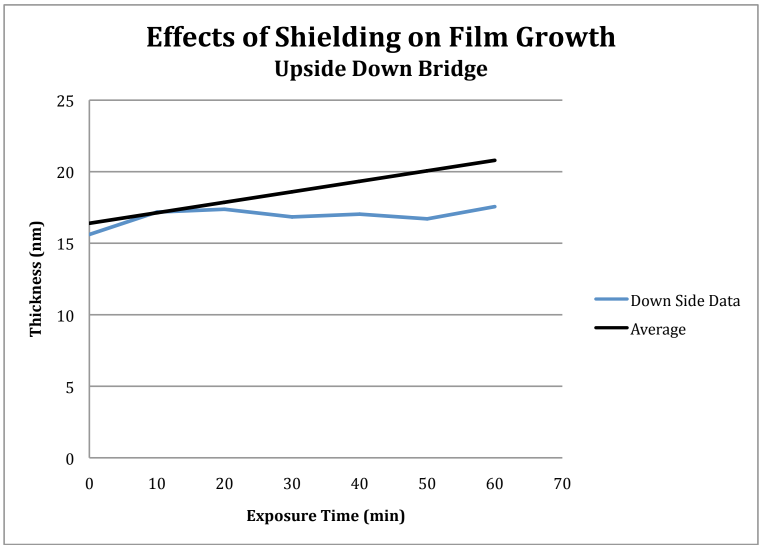


Figure 2.4 Thickness of the film vs. the exposure time when subjected to gas species. The average growth was calculated to be 0.73 nm per 10 minutes under the lamp. This theoretical data is graphed along side the data collected.

From the graph it is clear that ozone and atomic oxygen alone is not causing growth in the samples.

Notice that at some exposure times the thickness changed slightly. This data could suggest that the film grew; however, since these increases were very slight and measurements at later exposure times show decrease in thickness, it is more likely that the supposed increase can be attributed to non-uniformity of the surface or inadequacy of the ellipsometric measurements and fittings.

2.5 Current Theories

2.5.1 The Photon Effect

We next conducted another shielding experiment that would only allow the photons to interact with the sample surface. The set-up for this experiment is shown in Figure 2.5. A fuzed quartz window that was transparent to UV light was placed directly on top of the scandium sample. The quartz

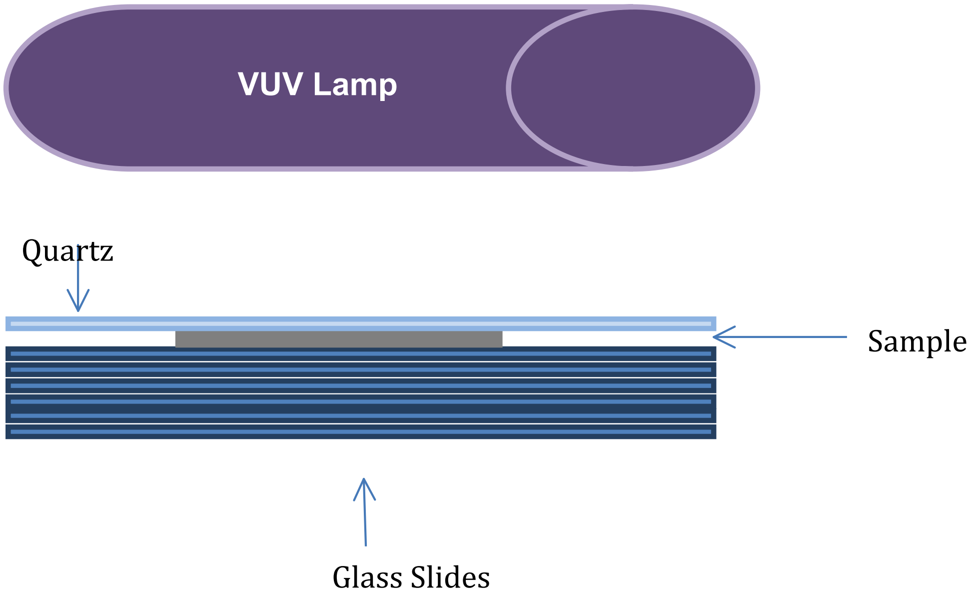


Figure 2.5 The structure set-up to test the effects of high energy photons striking the film surface.

window was 25×25×1 mm thick and manufactured by G. Finkenbeiner Inc. (1-800-552-6182). Using our ellipsometer in transmission mode, the quartz was seen to be >60% transparent from 1.24 to 6.5 eV. By placing it in direct contact with the film, we prevented the gas species from interacting with the surface.

The sample was exposed to UV light for intervals of ten minutes. After the first ten minutes the film thickness had decreased by ∼0.3 nm. Results from the entire experiment can be seen in Figure 2.6. There is no film growth. It would appear that having just photons interacting with the surface produces the results expected with hydrocarbons being cleaned from the film.

Our data suggests that the structures of the reactively sputtered oxides are being altered from the combined presence of high energy photons and the reactive gas species generated by the lamp. We realize, though, that while the quartz prevents ozone from reaching the film, it may not entirely eliminate its presence. Thus we cannot conclusively state that film growth is not due to photons alone until more strenuous testing is completed.

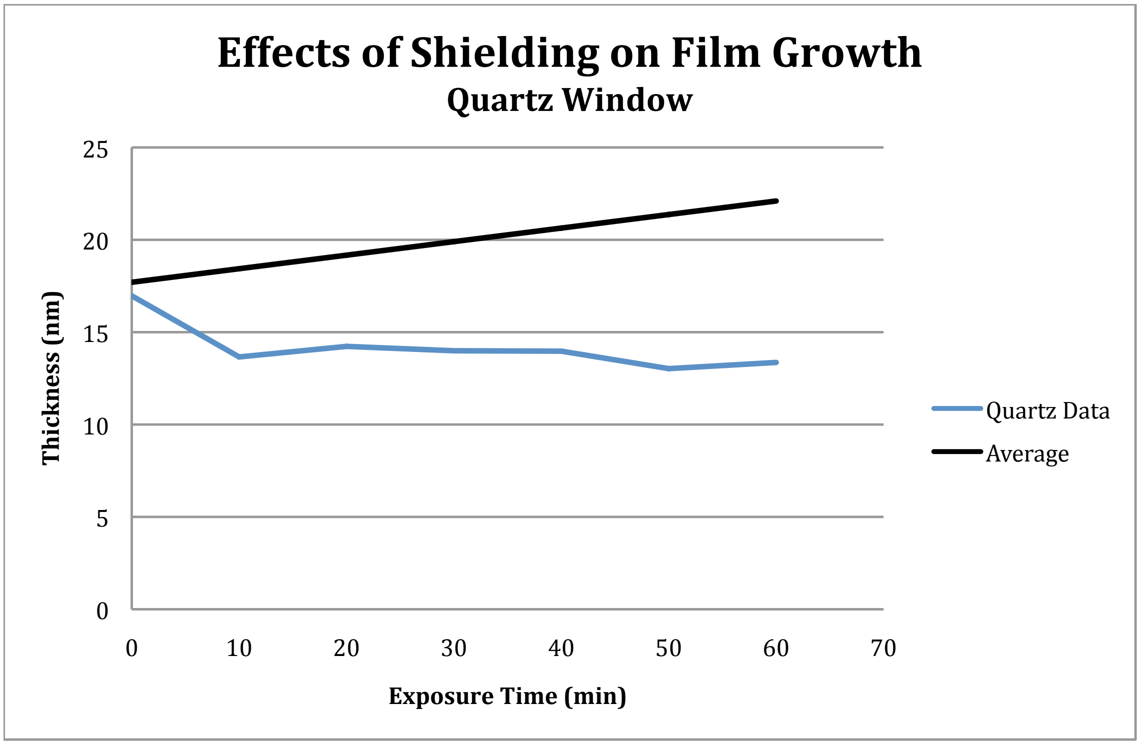
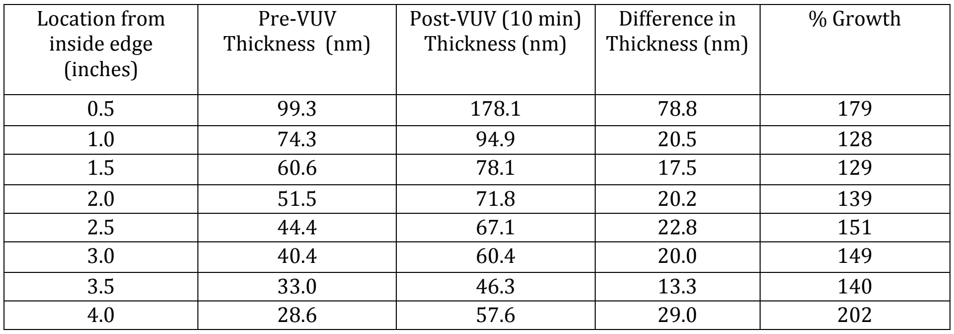


Figure 2.6 Thickness of the film vs. the exposure time when subjected to photons. Average growth observed in the absence of the quartz (the heavy black line) is graphed along side the data collected.

Chapter 3

Results and Discussion

My research puts considerable constraints on possible explanations. In fact, we have no theory on the mechanisms driving yttrium and scandium oxide film growth under VUV lamp exposure. The results have been duplicated in additional reactively sputtered samples involving both metals tested. Therefore, we know that growth is not due to some irreproducibility in the original film. The growth has continued to occur with samples sputtered in our chamber after some extensive altercations were made as mentioned in section 2.1. The growth of a scandium film sputtered in the new vacuum chamber set-up is summarized in Figure 3.1.



Location from inside edge (inches)	Pre-VUV Thickness (nm)	Post-VUV (10 min) Thickness (nm)	Difference in Thickness (nm)	% Growth
0.5	99.3	178.1	78.8	179
1.0	74.3	94.9	20.5	128
1.5	60.6	78.1	17.5	129
2.0	51.5	71.8	20.2	139
2.5	44.4	67.1	22.8	151
3.0	40.4	60.4	20.0	149
3.5	33.0	46.3	13.3	140
4.0	28.6	57.6	29.0	202

Figure 3.1 A summary of growth on a scandium sample sputtered in the altered vacuum chamber. Samples now offer a gradient of thicknesses. Sample was exposed to lamp for 10 minutes.

3.1 Future Research

3.1.1 Taking Measures to Prevent Growth

While research continues to find the cause of growth, it is also of interest to see if steps can be taken to prevent the films from growing while under the VUV lamp. Our new vacuum chamber allows us to sputter from up to four different targets. This means that layers from different materials can be put on a substrate without breaking the vacuum. We plan on taking advantage of the system by depositing a capping layer on top of future samples. It is our expectation that this top layer will block the access of gaseous specious and may further eliminate the causes of film growth when exposed to UV light .

3.1.2 Expand the Set of Metal Oxides Tested

Another approach is to branch out and test other metal oxides to see if they experience film growth. We tested scandium after our Y_2O_3 sample grew, but as stated in section 2.4, this was done because of their similarities. Yttrium and scandium are both group 3 elements. The next metal that we would like to try to duplicate our results with is nickel. It could be that a reactively sputtered nickel oxide sample would be affected by the same mechanisms driving yttrium and scandium film growth. This could possibly tell us more about what types of sample will grow and allow us to narrow the field more.

3.1.3 Characterization using TEM Imaging

We would also like to utilize transmission electron microscope (TEM) imaging. The use of TEM could finally characterize the new product being formed during the VUV exposure. This will reveal more about the structure of our sample and how, or if, it is changing. Since the TEM also performs electron diffraction, we would be able to observe any lattice structure change as a corresponding change in the diffraction pattern. This would help unravel what is happening to the sample itself.

Preparing a sustainable sample for TEM is the first step. On way might be to put the target material deposited onto coated copper TEM grids. The sputtering and oxidation process during VUV treatment destroys the carbon coating on the grids. New research conducted by Dr. Robert Davis of BYU and his research group has recently manufactured TEM grids composed of carbon nanotubes that could possibly survive the sputtering process. Electrons can penetrate only a few tens of nanometers. If this new method is satisfactory, TEM experiments will be run.

Chapter 4

Conclusions

The mechanism driving the growth of the films Y_2O_3 and Sc_2O_3 due to VUV light exposure appears to be an internal, rather than an external, change. Experimental tests have ruled out new depositions and oxidation of the silicon substrate as viable explanations for film growth. Water expansion inside the voids of the structure has also fallen short of explaining the thicker samples. The ozone and atomic oxygen by themselves have been ruled out as the cause of film increase. Instead, our evidence suggests that the structure of our metal oxide samples is being altered by the combined presence of UV photons and the surrounding ozone created by lamp use in air and atomic oxygen. In order to better understand the nature of this change, we propose that samples are studied using high resolution microscopy and electron diffraction to understand the structure and what is changing in the film. In the meantime, we wish to find ways to prevent film growth by adding silicon caps to the samples during sputtering. It is our belief that this could stop the film from swelling while being exposed to the VUV lamp. More work could be done on film expansion in environments where oxygen is scrupulously excluded.

Appendix A

Ellipsometry

A.1 Spectroscopic Ellipsometry

Measuring the polarization of light reflected from a surface can determine the thickness and optical constants (i.e. η and κ) of a thin film. This is done by measuring the incident beam's change in polarization after reflection from the film surface. Polarizers can be made to work over a wide range of wavelengths. Data taken over an additional range of incident angles can better characterize the thickness and constants. This particular process of measurement is referred to as spectroscopic ellipsometry .

Commercial ellipsometers typically employ two polarizers, one before and one after the sample in the analyzer. The first polarizer simply creates a beam with both s and p-polarizer components polarized at some angle α, thus linearly polarized light arrives at the test surface.

The effects of polarization and reflection on the light beam can be expressed using Jones vector formalism:

$$E_D\text{=[Analyzer Matrix][Sample Matrix][Incident Light Vector]}$$

where E_D is the electric field of the light entering the detector [9]. The Jones vector for the light arriving at the detector is then

$$\begin{pmatrix} \cos^2\theta & \sin\theta\cos\theta \\ \sin\theta\cos\theta & \sin^2\theta \end{pmatrix} \begin{pmatrix} -r_p & 0 \\ 0 & r_s \end{pmatrix} \begin{pmatrix} \cos\alpha \\ \sin\alpha \end{pmatrix}$$

$$= \begin{pmatrix} -r_p\cos\alpha\cos^2\theta + r_s\sin\alpha\sin\theta\cos\theta \\ -r_p\cos\alpha\sin\theta\cos\theta + r_s\sin\alpha\sin^2\theta \end{pmatrix}$$

where θ is the orientation of the analyzing polarizer and r$_p$ and r$_s$ are the complex Fresnel coefficients [10].

The detector, however, does not measure the electric field, but rather the intensity of the arriving light. Intensity is proportional to the square of the electric field. Therefore, the detector measures:

$$I_D \propto |r_p|^2\cos^2\alpha\cos^2\theta + |r_s|^2\sin^2\alpha\sin^2\theta - \frac{r_p r_s^* + r_s r_p^*}{4}\sin 2\alpha\sin 2\theta.$$

For ellipometry measurement, it is the convention to solve for the ratio of the Fresnel coefficients

$$\rho = \frac{r_p}{r_s} = \tan\psi\exp^{i\Delta}$$

where ψ contains the amplitude information and the Δ contains the phase information. They offer greater reproducibility by applying only to relative amplitudes and phases [9].

A.2 Mean Squared Error

Ellipsometric data was measured on a J.A. Woodlam M2000 and analyzed using WVASE32^{TM} software. This program uses the ψ and the Δ data collected at a number of angles (usually 1-7) and wavelengths ($\sim$1000) to fit a model for the thickness and optical constants of a film. Figure A.1 is

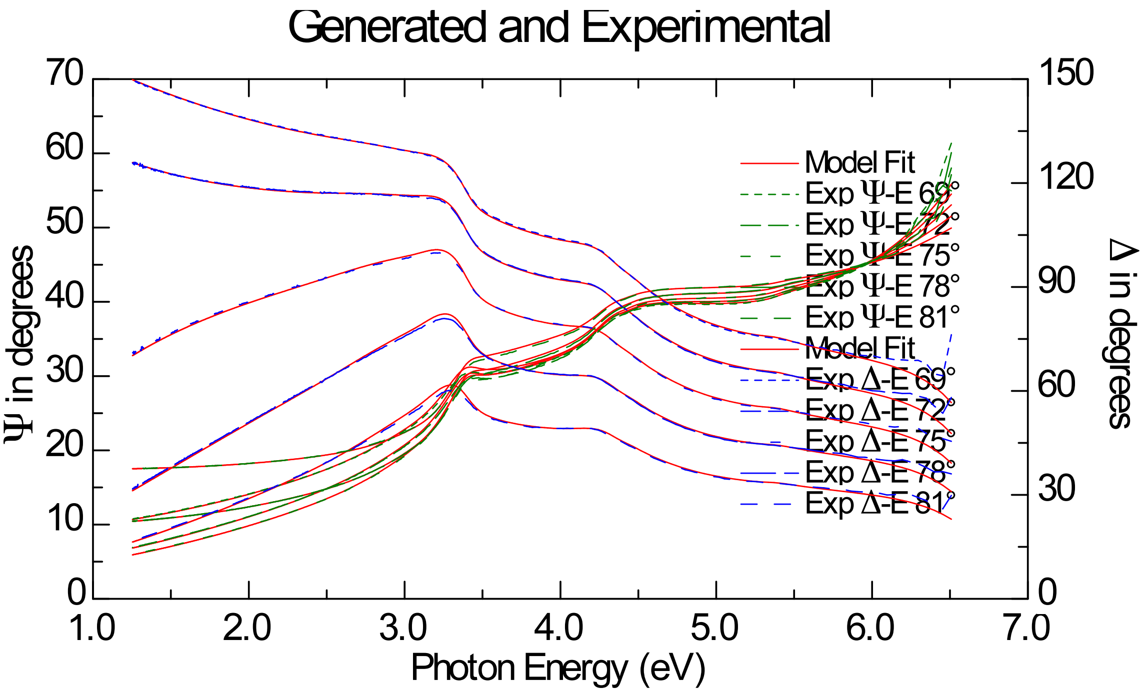


Figure A.1 Data from a Sc_2O_3 film fitted to ψ and Δ.

an example of fitted data for a scandium run. The mean squared error (MSE) expresses a measure of how closely the model fits the experimental data [11].

The MSE must approach unity for a perfect fit of the data; however, this is highly improbable when tens of thousands of data values are collected and there are only a handful of adjustable parameters in the model. A MSE of less than ten is typically considered to represent a reliable fit when many angles are used, although some fits with higher MSE values can still produce an accurate measurement of thickness. It is at the operator's discretion to adjust the model for a better MSE value.

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