

Investigating sputter-deposition under varying conditions

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July 2018

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1 Introduction

The purpose of this project was to investigate how the different parameters of Physical Vapor Deposition affects the rate and quality of the deposition. This was investigated using series of experiments designed to create a surface response model for each of the materials tested. This project will also cover what methods are ideal to use for measuring the thickness of a deposition.

2 Theory

2.1 Lesker CMS 18 Sputter System

Sputtering is a technique used in thin-film production. Sputtering works by accelerating ionized atoms onto a target substrate. The target then ejects atoms which deposits onto a sample creating a thin film.

Magnetron sputtering is a sub-genre of sputtering. Magnetron sputtering relies on strong magnetic fields which intensifies the deposition by confining the secondary electrons near the surface of the target, creating a denser plasma with a higher current. The magnetic fields also helps direct the plasma from the target onto the sample.

In our case the Lesker has 6 magnetrons, each with their own target, which means that 6 materials are available for deposition at a given target. Each of these magnetrons has their own power supply and the magnetrons themselves and their targets are spaced around the bottom of the deposition chamber. The sample holder is placed in the middle of the chamber, surrounded by a large circular magnet. This magnet can be rotated to secure an even thickness of thin films.

During the different depositions several parameters can be changed. In our case we will focus on changing the power that the magnetron exerts on the target and the overall pressure of the chamber during the deposition. In the case of the silicon depositions we will also pump the chamber with a varying concentration of oxygen, which in combination with the silicon plasma creates silicon dioxide. For the rest of the depositions the chamber will be filled purely with argon. [2]

3 Design of Experiments

This section will concern the plans for the experiments, where we will go over why we have chosen to make the depositions we have. In the pattern column there will be a number of symbols equal to the number of variables. A '+' means that for that measurement the maximum value was used, a '-' indicates that the minimum value was used and a '0' indicates the middle value between max and min.

3.1 Gold Deposition

Gold has some very interesting and useful properties when deposited, and as such is a very important material to understand in regards to deposition. Furthermore gold is not prone to oxidizing like other materials. Because of this we have decided to make a full response surface design, and use it as a baseline for the rest of the materials. The design can be seen in table 1

Au Deposition	Pattern	Time	Pressure [mTorr]	Power [A]
Sample 1	-0	50	3	175
Sample 2	00	50	6.5	175
Sample 3	+-	100	10	50
Sample 4	--	100	3	300
Sample 5	0+	50	6.5	300
Sample 6	00	50	6.5	175
Sample 7	0-	100	6.5	50
Sample 8	--	100	3	50
Sample 9	++	50	10	300
Sample 10	+0	50	10	175

Table 1: Surface response design of the Au measurements

3.2 Chromium Deposition

When deposited onto glass, chromium is an excellent adhesive for certain metals. Therefore it is interesting to investigate chromium, as a reliable deposition technique for thin films of chromium could enhance the quality of other thin films deposited onto the same sample.

Chromium is especially interesting because if gold is deposited onto a glass and chromium wafer, it has better adhesion and a decreased amount of islands in the deposition layer. We will see that this has been a problem during the gold deposition later.

Cr Deposition	Pattern	Time	Pressure [mTorr]	Power [A]
Sample 1	--	400	3	250
Sample 2	+-	400	10	50
Sample 3	00	400	6.5	150
Sample 4	++	400	10	250
Sample 5	--	800	3	50

Table 2: Design of the Cr measurements

3.3 Aluminum Deposition

Aluminum is interesting to investigate due to its ability of passivation. When aluminum starts to oxidize the aluminum oxide layer functions like a protective barrier slowing the further oxidation of the aluminum layer, eventually stopping the oxidation completely.

Al deposition	Pattern	Time	Pressure [mTorr]	Power [A]
Sample 1	-0	100	3	175
Sample 2	00	200	6.5	175
Sample 3	+-	400	10	50
Sample 4	-+	300	3	300
Sample 5	0+	200	6.5	300
Sample 6	++	300	10	300
Sample 7	+0	500	10	175
Sample 8	0-	1200	6.5	50
Sample 9	00	1000	6.5	175
Sample 2, 1	-0	1000	3	175
Sample 2, 2	00	1000	6.5	175
Sample 2, 3	+-	1000	10	50

Table 3: Design of the Al measurements

Since it later became clear that the first three depositions had deposited in the range of 0-7nm, we decided to do them again with longer time. This was done because the low thicknesses made it difficult to measure the depositions precisely.

3.4 Ti Deposition

As a stronger and lighter version of steel, titanium is a versatile metal. Also being very resistant to oxidization, it is a metal that is interesting to investigate as a thin film.

Ti Deposition	Pattern	Time	Pressure [mTorr]	Power [A]
Sample 1	-+	400	1	220
Sample 2	+-	800	5	100
Sample 3	00	400	3	160
Sample 4	++	600	5	220
Sample 5	--	800	1	100

Table 4: Design of the Ti measurements

3.5 SiO₂ Deposition

Silicon dioxide is a widely used material in wafer production and is therefore interesting to investigate. In our case we use a target of silicon and pump the chamber with a mixture of argon and oxygen gas. The plasma from the silicon target then mixes with the oxygen in the chamber depositing silicon dioxide onto the wafer. In contrast to the more conventional deposition technique of using a furnace, the Lesker doesn't require high temperatures. This means that heat sensitive samples can have a layer of silicon dioxide applied onto them, without risk of heat damage. The Lesker does however not anneal the deposited silicon dioxide which may result in the layer of silicon dioxide having a higher roughness.

SiO2 Deposition	Pattern	Time	Pressure [mTorr]	Power [A]	Oxygen percentage
Sample 1	+ 0 0	1000	10	85	27.5
Sample 2	+ - -	1500	10	50	5
Sample 3	0 0 0	1000	6.5	85	27.5
Sample 4	0 + 0	1000	6.5	120	27.5
Sample 5	- - -	1500	3	50	50
Sample 6	+ - -	1500	10	50	50
Sample 7	+ + +	1500	10	120	50
Sample 8	0 + 0	1500	6.5	50	27.5
Sample 9	+ + -	1500	10	120	5
Sample 10	- - -	1800	3	50	5
Sample 11	0 0 +	1200	6.5	85	50
Sample 12	0 0 0	1200	6.5	85	27.5
Sample 13	0 0 -	1200	6.5	85	5
Sample 14	- + -	1000	3	120	5
Sample 15	- 0 0	1500	3	85	27.5
Sample 16	- + 0	1000	3	120	50

4 Thickness Measurements

During this project we used a number of methods to attempt to determine the thickness of the deposited material, this section will go over the theory of those methods as well as how we used them.

4.1 Weight

One way to measure how much material is deposited onto the wafer, is by measuring the weight of the wafer before deposition and after deposition. The difference in weight is then the weight of the deposited material, and knowing the density of the material and the area of the wafer the thickness can be calculated.

The equation for determining the wafer thickness from this can be written as follows:

$$t = \frac{w_{after} - w_{before}}{A \cdot f \cdot \rho} \quad (1)$$

Where A is the area of a whole wafer, f is the fraction of a whole wafer calculated as

$$f = \frac{w_{before}}{w_{wholewafer}} \quad (2)$$

and ρ is the density of the material. In order to obtain the rate, we simply divide thickness with the deposition time.

4.2 Ellipsometer, VASE

Another one of our most used methods is ellipsometer measurements, in which light with wavelengths ranging from 210-1690 nm is sent at the surface of the material at different angles, and the reflected

light is measured. The reflected light is then compared to models of materials, which can be used to estimate the thickness of the deposited film. In order to obtain the rate, we simply divide thickness with the deposition time.

The Ellipsometer used is a Variable Angle Spectroscopic Ellipsometer (VASE). [6]

4.3 Bulk Resistance

Another way we have measured the thickness of the layers, is by using a four-point probe to measure the resistivity of the sample.

A four-point probe has four probes in a line. When the probe is pressed onto the sample, the outer two probes induce an electrical current in the material, and then the inner two act as sense-probes measuring the resistivity.

For calculating the thickness, we use the equation:

$$t = \frac{\rho}{R_s} \quad (3)$$

Where $\rho = 2\pi s \frac{V}{I}$. As is known electrical resistance $R = \frac{V}{I}$, and as such can be looked up. Again in order to obtain the rate, we can divide thickness with the deposition time.

4.4 Dektak

Additionally one can attempt to use a profilometer to measure the thickness of a material. In this project the danchip Dektak was used. Since it is an instrument that tells the features of the surface of a sample, we needed a feature with the depth of the deposited layer on the wafer. We could make one such feature by either scratching the wafer with something that would only affect the deposited layer and not the material we deposited on. What we did however, was utilize the fact that the sample was held by little clamps during the deposition. Where these clamps touched the wafer no material was deposited.

The result of this was sub-par, as it was difficult to determine the height of the deposited material, because of large variances in the height of the deposited material around the hole. As a result of this, we have chosen to omit the Dektak measurements from the report and rely on the other methods. [7]

4.5 X-ray Reflectometry

X-ray Reflectometry (XRR) is a technique that can measure a single or multi-layered thin film surface of a sample. X-rays are directed towards the sample at low angles. The angle of incidence is then increased until the critical angle of incidence is found, where all the rays are absorbed into the material. This angle is different from material to material, from which the XRR technique can categorize which thin films have been deposited onto the surface. As the critical angle is passed the rays start reflecting away from the sample creating interference. From this interference the thickness of the film can be measured. XRR can also measure density and roughness of thin films using the intensity of the x-ray measurements. [3]

The machine used for XRR is the Rigaku XRD SmartLab.

4.6 X-ray Photoelectron Spectroscopy

X-ray Photoelectron Spectroscopy (XPS) is a spectroscopy technique that uses the photoelectric effect to identify the properties of a sample. The XPS sends X-ray photons into a sample which releases electrons from the sample. These free electrons have varying kinetic energy depending on the energy of the X-rays and their own binding energy. The electron spectrometer inside the XPS then analyses the electrons based on this energy. As each element has very specific binding energies for each of their electronic states, the electron spectrometer can detect which elements inhabit the sample. For each element detected a more specific survey can be done for each individual element. [4]

4.7 Transmission Kikuchi diffraction

Transmission Kikuchi Diffraction (TKD) is a microscopy technique that has many similarities to Electron Backscatter Diffraction (EBSD). EBSD is typically used in different microscopy techniques such as Scanning Electron Microscopy (SEM) to identify the texture and structure of materials on micro and nano-meter scale. The downside to EBSD is that the spacial resolution is limited to between 20-50 nm depending on the density of the material. Where TKD improves itself from EBSD is with the handling of the material sample. The sample is thin enough to be electron transparent, which is also why TKD sometimes is called transmission-EBSD (t-EBSD) as it adopt some principles from Transmission Electron Microscopy (TEM). The sample is also mounted horizontally or backtilted away from the detector, making it detectable from both sides. The ability to detect from both the bottom and top of the sample improves the spatial resolution as you can limit the diffraction source volume. [5]

5 Analysis

In the following section we will analyze the data of each material as well as comment on how well the different measuring methods are and what limitations they have. For the DOEs we have used the Ellipsometer measurements, as they seem to be most the accurate and have also been used on all wafers.

5.1 Gold sample

5.1.1 DOE

In table 5 we have calculated the rates according to the four methods: Ellipsometer, Weight, Sheet resistance and XRR. It can be seen that the measurements for Ellipsometer and weight are somewhat consistent. We are missing a lot of measurements of the sheet resistance due to errors, that may have been caused by the gold not being a continuous film, but rather small islands. We will expand on this later. The XRR measurements took a long time to make, so we lacked the time to do them for all the samples.

From the collected data we can produce a model of how the parameters of power and pressure influence the deposition rate. The parameters can be seen in figure 1.

Au Results	Pattern	Rate (Ellipsometer) [nm/s]	Rate (Weight) [nm/s]	Rate (4PP) [nm/s]	Rate (XRR) [nm/s]
Sample 1	-0	0.4384	0.4254	E30	N/A
Sample 2	00	0.4322	0.3689	-	N/A
Sample 3	+-	0.0929	0.0983	0.122	0.1218
Sample 4	-+	1.0419	0.6473	-	N/A
Sample 5	0+	0.6968	0.6467	-	N/A
Sample 6	00	0.4234	0.3300	-	N/A
Sample 7	0-	0.117	0.1133	0.211	N/A
Sample 8	- -	0.1398	0.0913	0.286	N/A
Sample 9	++	0.7034	0.6529	-	0.68083
Sample 10	+0	0.4016	0.3424	0.666	N/A

Table 5: The values of power ranges from 50 (-) to 300 (+) and the pressure from 3 (-) to 10 (+).

Parameter Estimates				
Term	Estimate	Std Error	t Ratio	Prob > t
Intercept	0,4005429	0,046025	8,70	0,0010*
Power(50,300)	0,3487333	0,031441	11,09	0,0004*
Pressure(3,10)	-0,070367	0,031441	-2,24	0,0888
Power*Pressure	-0,0729	0,038507	-1,89	0,1313
Power*Power	0,0336143	0,050418	0,67	0,5414
Pressure*Pressure	0,0467143	0,050418	0,93	0,4066

Figure 1: The parameters of the model fit for the gold experiments.

From the obtained parameters we can see from the Prob > |t|, that the power is very significant and as such must be concluded to influence the rate as described, which is positively with a factor of ~ 0.35 . What is far more surprising is that from the data we have collected the pressure is not significant enough to conclude it has an impact. In order to verify this, we could collect more data, but as it is, it can not be concluded whether the pressure has any impact on deposition rate.

None of the other combinations of the variables we have investigated were significant enough to conclude they had any impact.

In figure 2 a graph of the parameters show how they would affect the rate, as well as how desirable the different values of the parameters are in order to maximize the deposition rate. Naturally they only show how each variable changes the outcome for one value of the other parameter, but they do not change much and are a pretty good indicator of the tendency at all values. This shows us that even though other (non-significant by t-value) factors are clearly at play in the model, the significant linear dependence on power is the largest determining factor of the rate, in the variable ranges we are working with.

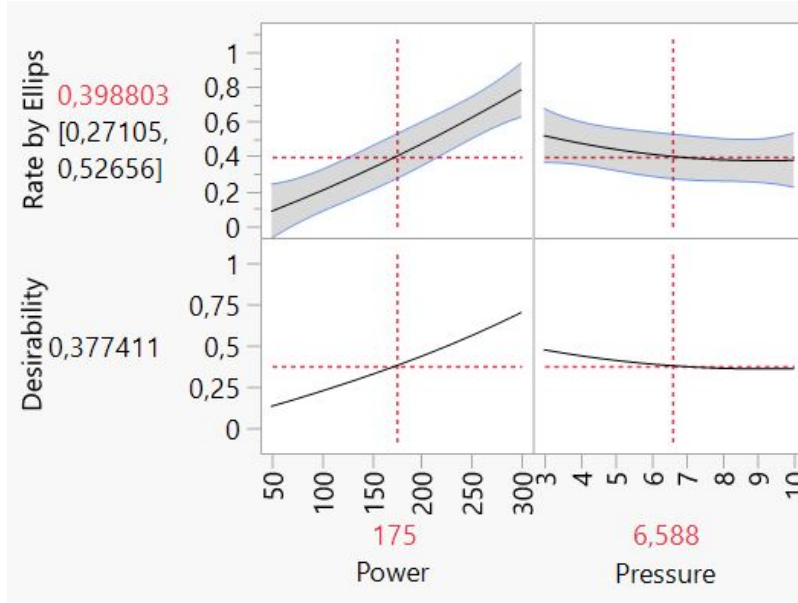


Figure 2: The top graphs show how the rate is influenced by the power and pressure, and the bottom graphs show what our ideal values should be if we want to maximize the deposition rate.

Another thing we can use to test the validity of the model is how well the model describes the data. As can be seen in figure 3 most of the data points lie within the uncertainty of the model, as such it describes the data relatively well, which is to be expected since this is what we modelled for.

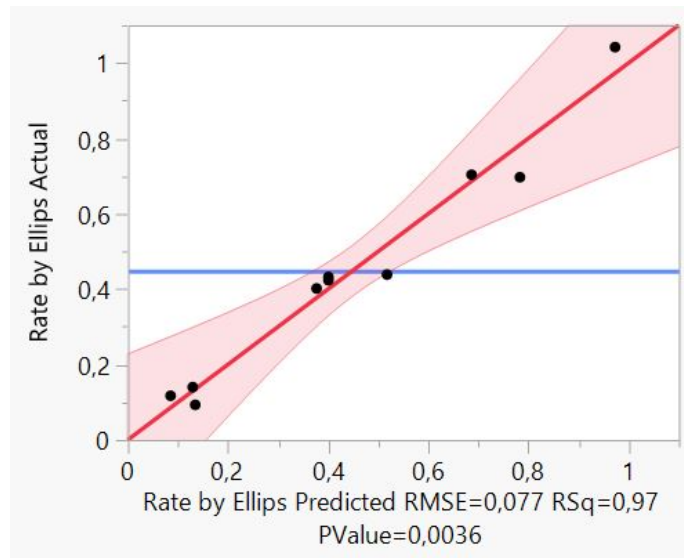


Figure 3: The predicted values of the model compared to the measured values.

5.1.2 XPS of gold

As we can see from figure 4 the gold is behaving as expected. On the left image we can see two peaks, indicating that gold is present on the surface and after each etch. On the right image we can see that there is no peaks, which means there is no oxygen in the gold. This is expected as gold does not bind with oxygen.

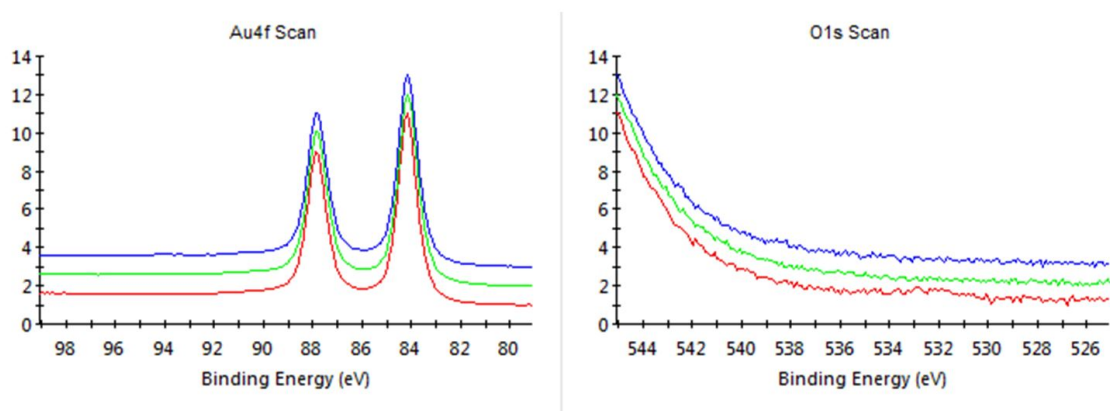


Figure 4: The XPS measurement made on the Au4 sample. The red line is the surface measurement and the green is after etching.

5.1.3 Transmission Kikuchi Diffraction

Lastly we performed a TKD on a TEM grid that was inserted into the machine alongside sample Au9. The results can be seen in figure 5. As can be seen there are large areas where we have 111 crystal orientation (blue) and those are considered the grains. Based on other TKD measurements we have been shown, it is usual for gold to deposit so the large grains have 111 crystal orientation. It can also be seen that there are elongated areas in which no data can be measured. The sample was determined to have a thickness of 34.04nm according to XRR and 35.17nm according to ellipsometer and since the film is so thin and since gold is generally unwilling to stick to glass surfaces, what we are seeing could very well be islands of gold, with nothing deposited in-between. This could explain why the sheet resistance is so inconsistent, since there is not any continuous layer to conduct electricity through.

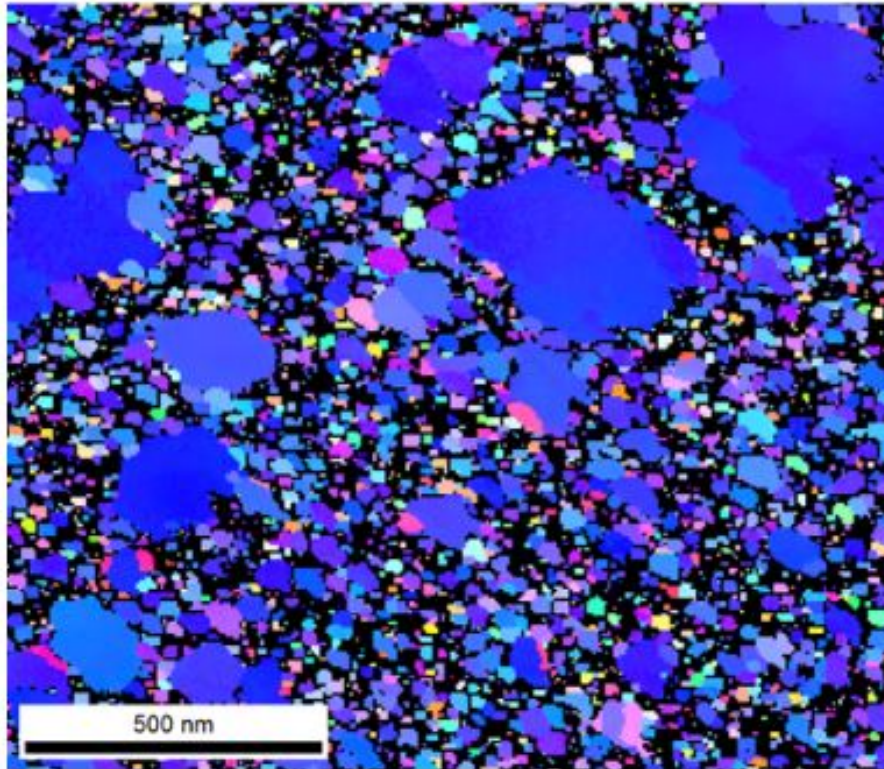


Figure 5: The results of the TKD measurements on sample Au9, which was deposited using 10 mTorr and a power of 300A for 50 seconds. Blue areas are 111 surfaces and black are areas where no direction could be fitted, other colors are other crystal orientations.

What this tells us about the depositions, is that in order to get a smoother film, that does not feature 'islands' of gold with valleys inbetween, a thicker layer than 34nm would need to be deposited. Alternatively the gold could be deposited onto something other than silicon dioxide in the hopes of increasing adhesion and lowering the number of islands.

5.2 Aluminum Sample

For aluminum it was discovered that some values could not sustain plasma, and as such we were left with fewer data points than we had hoped. This may make the response surface design a bit more unreliable. The calculated data can be seen in table 6. Again we use this data to produce a model of how the parameters of power and pressure influence the deposition rate. The parameters can be seen in figure 6.

Al Results	Pattern	Rate (Ellipsometer) [nm/s]	Rate (Weight) [nm/s]	Rate (4PP) [nm/s]	Rate (XRR) [nm/s]
Sample 1	-0	0.02491	0.043473	0.03799	N/A
Sample 2	00	0.02239	0.004032	0.02159	N/A
Sample 3	+-	0.00295	-0.1140	-	N/A
Sample 4	--	-	-	-	N/A
Sample 5	0+	0.0551	0.03977	0.08750	N/A
Sample 6	00	0.04697	0.08115	0.07120	N/A
Sample 7	0-	0.02338	0.02829	0.03172	0.0212206
Sample 8	--	0.007358	0.01690	0.004675	N/A
Sample 9	++	0.0365	0.02623	0.005265	N/A

Table 6: During the deposition on Sample 4 the parameters could not sustain plasma, and as such the data has been excluded. The values of power ranges from 50 (-) to 300 (+) and the pressure from 3 (-) to 10 (+).

Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	0,030449	0,004133	7,37	0,0052*
Power(50,300)	0,0238708	0,004772	5,00	0,0154*
Pressure(3,10)	0,000638	0,003957	0,16	0,8821
Power*Pressure	-0,001863	0,006749	-0,28	0,8005
Power*Power	-0,000224	0,004772	-0,05	0,9656
Pressure*Pressure	-0,004901	0,0052	-0,94	0,4155

Figure 6: The parameters of the model fit for the aluminum experiments.

From the obtained parameters we can see from the Prob > |t|, that the power is significant, which indicates that it has influence on the rate, scaling linearly with a factor of ~ 0.023 . The reason this number is so much lower than it was for gold, is due to the fact that Al deposition in general has much lower rates. As we also saw for gold, the data we have collected shows that the pressure is not significant enough that we can conclude it has as impact. Given the uncertainty of the data, it cannot be ruled out that the influence is simply too small to be detected.

None of the other combinations of the variables we have investigated were significant enough to conclude they had any impact. The most promising of these features seem to be pressure squared, affecting the rate negatively, since that has higher significance than pressure scaling linearly with rate.

In figure 7 a graph of the parameters show how they would affect the rate. This time we see that there is a larger uncertainty. This is likely caused by the fact that due to aluminum having very slow deposition rates, the samples may have had too thin films to get accurate readings of the thickness. Again the graphs only show how each variable changes the outcome for one value of the other parameter (the middle parameter), but varying that value does not change the tendency much.

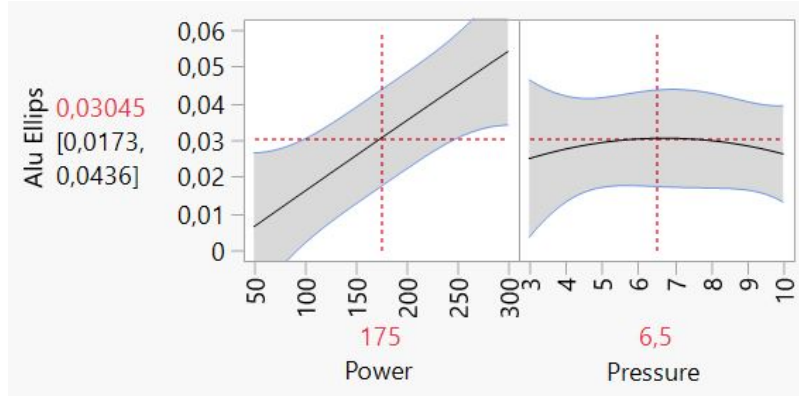


Figure 7: The top graphs show how the rate is influenced by the power and pressure.

To test the validity of the model we can plot the predicted values versus the measured values. This can be seen in figure 8. Most of the data points lie within the uncertainty of the model, as such it describes the data relatively well. If we wanted to really test if this was reproducible we could make a second set of measurements and see if this model described those data, even though it was fitted the the old measurements.

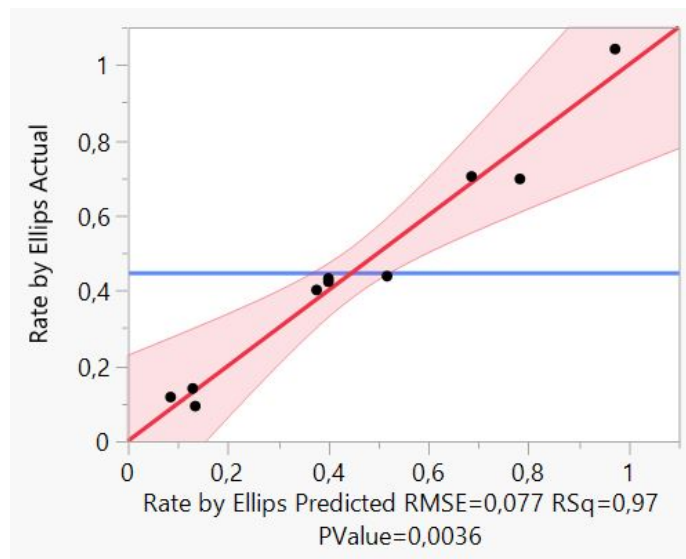


Figure 8: The predicted values of the model compared to the measured values.

5.3 XPS of aluminum

From figure 9 we see that aluminum has oxidized as expected. Once again we see the presence of oxygen both before and after the etch. However, as we only did 2 measurements we can't see the effect of passivation in the aluminum.

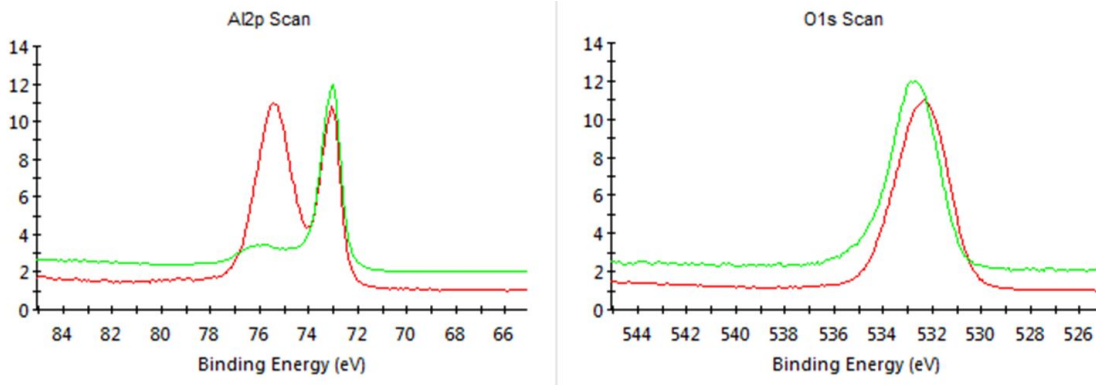


Figure 9: The XPS measurement made on the Al9 sample. The red line is the surface measurement and the green is after etching.

5.4 Chromium Sample

For Chromium we did not make samples in order to do a proper response surface, but simply made the corner cases to see what range we are dealing with. The results can be seen in figure 7.

Cr Results	Pattern	Rate (Ellipsometer) [nm/s]	Rate (Weight) [nm/s]	Rate (4PP) [nm/s]	Rate (XRR) [nm/s]
Sample 1	00	0.22335	0.131263	0.06646	0.198025
Sample 2	-+	0.011775	-0.003674	-	0.0214775
Sample 3	+-	0.055875	0.039806	0.0068720	0.09505
Sample 4	--	0.0455	0.074106	0.00049325	N/A
Sample 5	++	0.02145	0.021701	0.00006198	N/A

Table 7: The values of power ranges from 50 (-) to 250 (+) and the pressure from 3 (-) to 10 (+).

This data shows that based on all methods that sample 1 has the largest deposition rate by a lot. Which is interesting since this sample was deposited using middle values for both power and pressure. The previous two models agreed that the power increased the rate, and that if the pressure had any effect, it was to slightly increase the deposition rate at low pressure. It could be interesting to investigate whether or not this is a fluke, since the second highest rate seems to be Sample 3 according to ellipsometry, where the power is maxed and pressure is lowest.

We also see that the lowest deposition rate is sample 2 where the power is lowest and the pressure is highest. This also plays into the idea that the power and pressure have the same effect on all materials, but this cannot be concluded from the data. Nonetheless this gives an idea of the magnitude of the rate of chromium depositions.

5.4.1 XPS of chromium

From figure 10 we see that the deposited chromium layer has been oxidized by air. This is to be expected as chromium wants to bind with oxygen and does so quite quickly. On the right image we see that oxygen clearly inhabits the chromium layer, both on the surface and after the etching. On the left image we see that both chromium and chromium oxide inhabits the deposition.

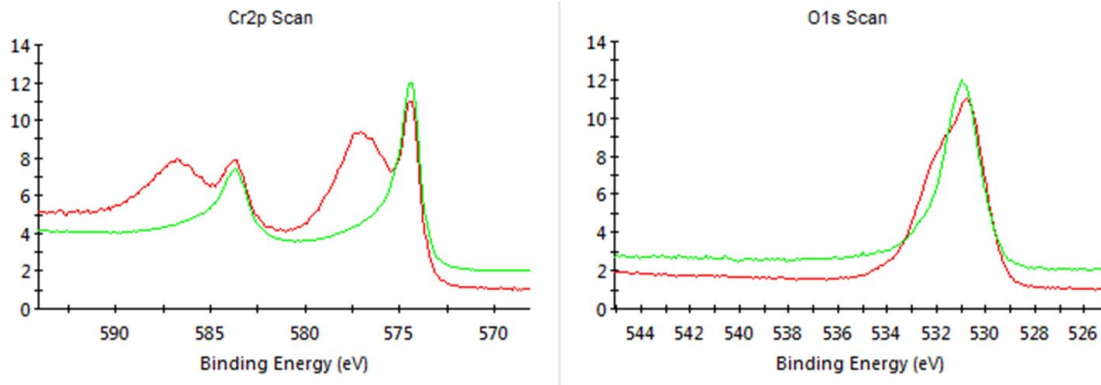


Figure 10: The XPS measurement made on the Cr1 sample. The red line is the surface measurement and the green is after etching.

5.5 Titanium Sample

Regarding titanium we also did not make a full response surface due to time constraints, but simply made the corner cases to see what range we are dealing with. The results can be seen in figure 8.

Ti Results	Pattern	Rate (Ellipsometer) [nm/s]	Rate (Weight) [nm/s]
Sample 1	00	0.0685	0.05345
Sample 2	-+	0.04339	0.02475
Sample 3	+ -	0.1028	0.06052
Sample 4	-	0.03613	0.03627
Sample 5	++	0.04698	-0.05961*

Table 8: The star indicates that a speck of the wafer broke off during the dismounting of the wafer, which may explain the lower value. The values of power ranges from 100 (-) to 220 (+) and the pressure from 1 (-) to 5 (+).

From this data, again going by the ellipsometer measurements, it can also be seen that the highest rate is found at max power and lowest pressure supporting the idea that the models for gold and aluminum has the same tendencies as other metals, with the middle measurement 00 in the middle and the only far outlier from this idea is ++ being so small. Again these measurements are not sufficient to test the tendencies as much as to give the idea of the magnitudes of the rates and how much they changed with variables. We can see that changing the values can increase the rate by a factor of three from the highest to lowest measurement.

5.6 Silicon Dioxide

During the first deposition the plasma was flickering and both the power and the voltage of the Lesker jumped in value. It was found that this problem was due to the high pressure of the deposition recipe. This resulted in us omitting the recipes in the DOE that required a pressure of 10 mTorr. From the 10th to the 14th deposition, the Lesker had increasing problems with the power output. Deposition 10 could only get half of the indicated power output. Deposition 12 had great variations

in the power output. Deposition 13 and 14 could not go beyond 18W in the power output. These irregularities in the depositions resulted in the omission of deposition 15 and 16.

We weren't sure what the increasing problems with the depositions arose from. We theorized that the Si target could have gotten oxidized from the oxygen that inhabited the chamber during the depositions. As our time was too limited to recreate the samples after an investigation of the target and the Lesker itself, we instead chose to omit the Silicon DOE in favor of some titanium samples.

We have however performed a number of XPS measurements to determine if the oxygen level had any influence on the deposited silicon dioxide in terms of what compounds were deposited.

From figure 11 we can see that the oxygen level doesn't have an influence when it comes to the deposited silicon dioxide. The XPS measurements has almost the exact same peak on all the etches and the oxygen measurements has a lot of noise. If we compare it to figure 12 we see that furnace sample is much better than the samples made in the Lesker. The reason for this could be that the furnace sample is annealed, which results in a much smoother surface, where the Lesker samples are rougher due to not being annealed.

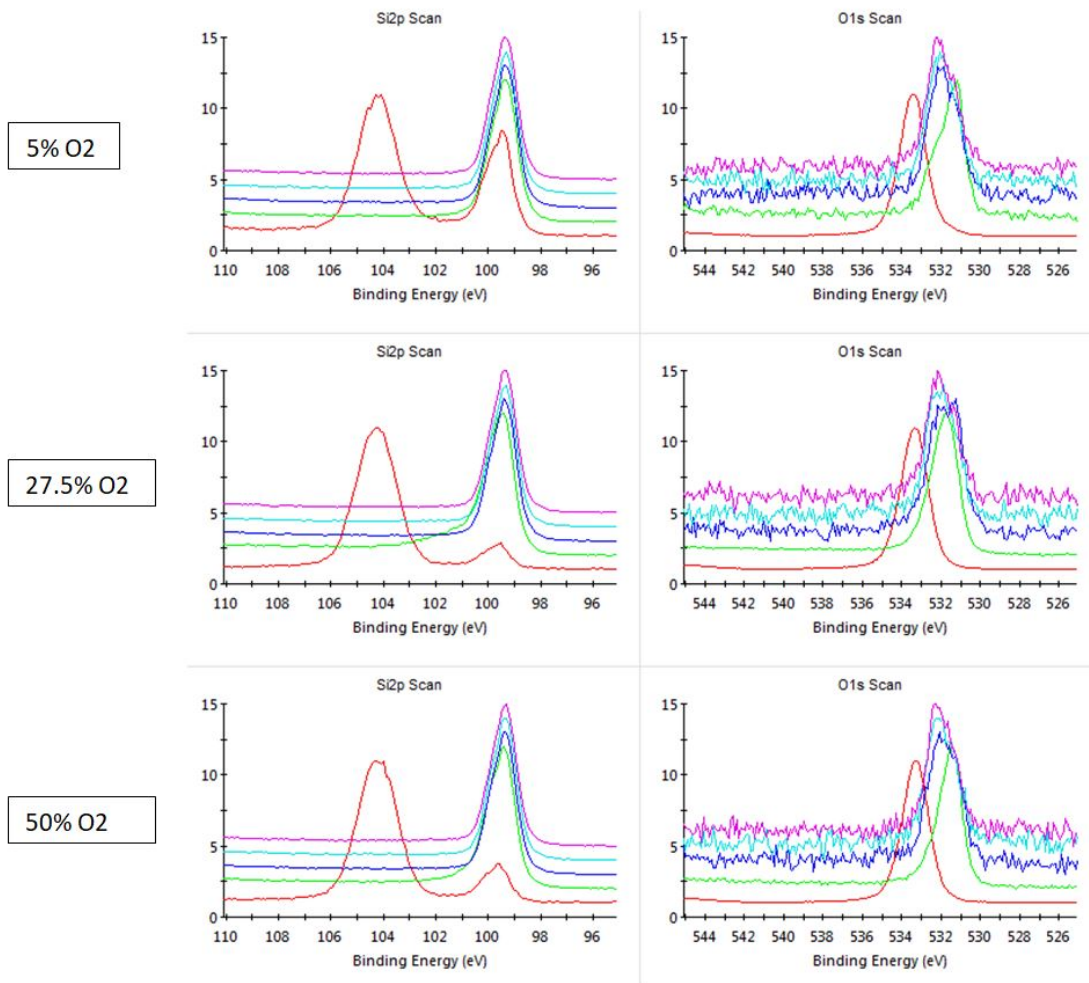


Figure 11: XPS data from the Lesker deposited Silicon dioxide samples. The samples investigated with XPS were samples S2, S9 and S4. S2 and S9 both had a power of 85W and a pressure of 6.5mTorr , while S4 had a power of 50W and 3mTorr. Ideally we would have only varied one parameter, namely the oxygen level, but due to the problems described that was not possible.

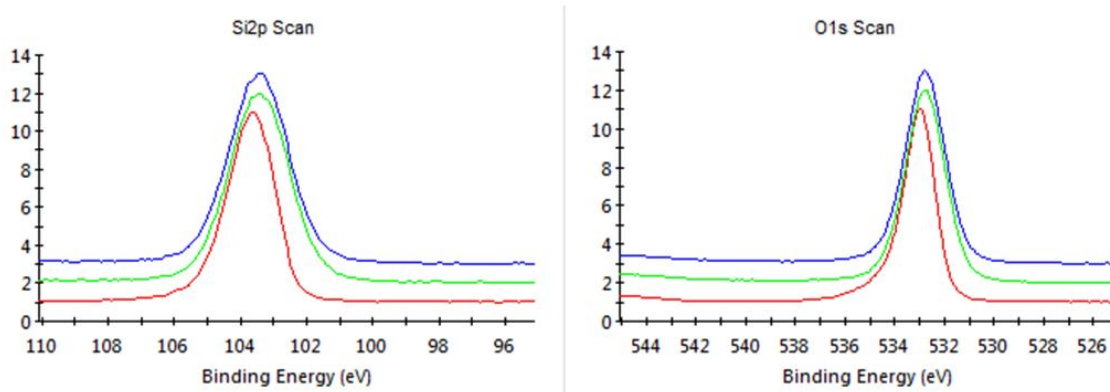


Figure 12: XPS data from Silicon dioxide grown in a furnace.

5.7 Measurement Techniques

5.7.1 Weight

As the simplest of the thickness measurement techniques, determining the thickness by weight has its pros and cons. The pros of the technique is that the measurement only takes a few seconds and the math is pretty simple. The problem with the technique is that if the density is low or the layer too thin, the requirements for the equipment increases. With materials of lower density there is also a larger chance of human error, as a small scratch of the wafer can remove more weight than the deposition added. We saw some wafers that were lighter after the deposition before, as can be seen from the number of negative rates.

Having this in mind the technique can be a good measurement option if the deposition of material is sufficiently large. The quality of weight measurements could also increase if the wafers were whole, since the edges were fragile and sometimes specks broke off, which can make big differences when we are comparing the small changes of the deposited materials.

5.7.2 Ellipsometer

This technique was the most stable of the ones used for all samples. Depending on the measurement angles and measurement points the ellipsometer can give a great deal of information regarding not only the thickness, but also how much the thickness varies should the deposition be uneven. The relative measurement time depends on how much of the sample you want to study, but a one point measurement only takes a few minutes. The only negative to this technique is that it uses optical measurement, which can set some requirements to the sample. In our case we had to deposit silicon dioxide on the wafers used for metal deposition to make the measurements more stable.

Overall this measurement technique gave really good results and only had problems with a few samples that were below 10 nm thick.

5.7.3 Bulk Resistance, 4-point probe

These kinds of measurements lets you measure the sheet resistance of a deposited material, from which you can calculate the thickness. It is a simple and quick method, but it does have some

downsides. If the material is soft it can easily be scratched. In our case this happened to some of our gold samples. As we saw with our TKD measurements, the gold samples deposited showed signs of having islands with gold. This means that if one of the probes lands on an island with deposited material, the resistance is not correctly measured.

In our case this measurement type didn't give us much information. The reason to this could be uneven depositions for all the materials, but as we only had time to do TKD on the gold samples, we cannot be sure that this is the reason.

5.7.4 X-ray Reflectometry

X-ray Reflectometry was the most time consuming measurement technique, but also by far the best one. For all the samples tested it gave quite precise measurements. It also gave some insight into the roughness and density, which suggested that the depositions were not evenly deposited. The software also gives a lot of options for refining the data.

This technique can give some amazing measurement, if enough time is spent on it. A great measurement can take many hours and the following analysis just as long. Therefore this technique is optimal if the user has unlimited time, but if you're in a rush, other methods may prove more useful.

6 Going Forward

During this project, there was a lot of things we wanted to investigate and little time do to it. As such we are left with some loose ends that we could have pursued if we had had more time. In this section we will briefly go over those.

- The Silicon dioxide depositions would definitely be interesting to spend some more time on, but to point at something concrete that we could have done, we could have attempted to anneal the samples before putting them in the XPS. This might have made the quality better than we currently see. Annealing the Silicon dioxide would also make the data from the Lesker data more comparable to the furnace SiO₂, where annealing is an automatic part of the process.
- Ideally we would also have wanted to spend some more time on the TKD, and characterize the grain size and quality of the deposited materials under the different conditions.
- Another thing that we would have done, is to make full response surface designs for every material, but this is a very time-consuming process so we had to carefully select which measurements we wanted.

7 Conclusion

7.1 Materials

The gold data was better than the aluminum data for the purpose of minimizing the uncertainties of the response surfaces. From these surface response models we could see how the variables influenced the rate.

As we can see from the analysis, the power seems to be most important feature in regards to determining the rate of deposition. The power scales linearly with different factors depending on the material.

Curiously there was not enough significant evidence to conclude that the pressure had an effect at all, but all the models had the rate go up slightly at low pressure.

No combinations of the two variables had high enough significance to suggest they had any effect.

7.2 Methods

Using the weight of the samples to calculate the thickness was a quick and easy method, but susceptible to human errors. It had large margin of errors with materials of low density. This method is optimally used for samples with large depositions at high density, where the samples are also taken well care off.

The XPS is a great tool for identifying the quality of a deposition. Although acquiring elaborate data sets is quite time consuming, the etching process gives insight into each layer of a sample, how well the compounds have mixed and how much oxidation or other types of corrosion has impacted the sample.

The ellipsometer measurements were fast and easy, although it had some problems with layers being to thin or thick. It is the one we have used to make the DOEs because the other methods were less precise or we did not have data for all samples. This is the method we would recommend unless there is a large surplus of time, which would allow for the use of XRR as well.

The XRD provided quite stable measurements, and from what we can gather it is the most precise method. However it is time-consuming and if there is a large number of samples a faster method could be applied.

Sheet resistance measurements by 4-point probe proved to be quite unreliable, as a lot of the samples would cause errors on the machine. This may have been due to the deposited layers not being continuous films. Nonetheless even when it did turn up values, the values were highly inconsistent with the other methods.

TKD gives information in an area where the other measurement techniques cannot. The TKD looks into the structure of the deposited layer on a nanoscopic level. In our case it gave insight into why the sheet resistance measurements did not work for the gold samples, as the gold had not deposited evenly across the surface of the wafer, creating islands in the process.

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