Optimization of thin film deposition using in-situ ellipsometry

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Indhold

1 Motivation

The purpose of this project and following journal is to characterise the material thickness produced using parameters such as pressure, power and ratio of source materials used in magnetron sputtering of silicon wafers on the sputter system in DTU Danschips cleanroom. We want to investigate and optimize the sputter deposition process and map how the parameters affects the thickness of different types of metals, dielectrics or semiconductors on silicon wafers. The thickness will be measured in-situ by an ellipsometer layer by layer by fitting different types of material models to the data, which in turn predict the growth rate for each layer. An ex-situ measurement of the total multilayer thickness will also be carried out as a comparison to the model fit.

2 Setup

The general experimental set-up consists of the Sputtering machine Lesker which has an ellipsometer attached to its side.

The sputter is divided into three main parts. The Load Lock chamber in which the sample silicon wafer is mounted and from there brought into the main chamber. The main pressure chamber where the sputtering is done, which is equipped with various pumps (to keep the low pressure), six sources or 'guns' loaded with target material used for sputtering, and the ellipsometer which shines light at an angle onto the sample and collects the reflected light in a waveguide. Lastly is the rack with the computer system controlling the machine including power-supplies for the guns.

2.1 Sputtering

Sputtering is a method used for thin-film fabrication. It creates a thin layer of a chosen material, which is called the target, on a surface.

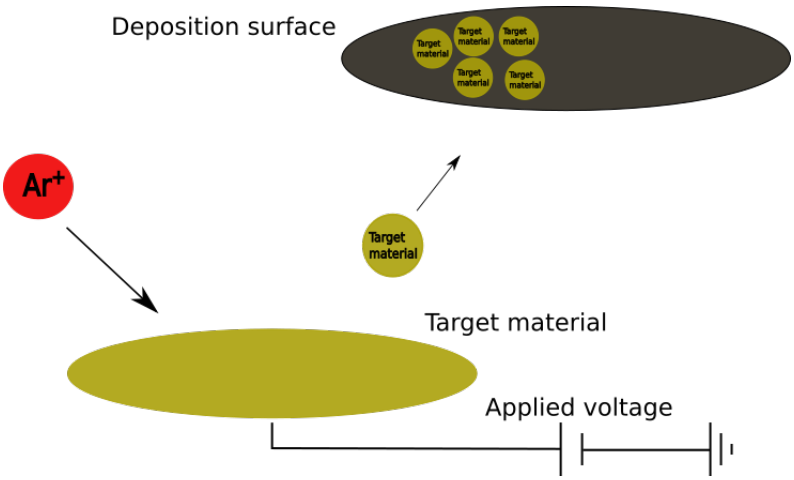
An incident ion is propelled towards the target material and at impact it dislodges some atoms from the it. Theses atoms then move through the air until they hit a surface which they bind to. In this way a layer of a material can be applied to a surface, atom by atom, and thereby very thin layers can be produced. This is illustrated on fig. 1. [1]. The process also works with molecules.

The ion is typically argon that has been ionised by turning it into a plasma. Argon is used because it is inert since it's a noble gas. It has been ionised so it will be possible to apply a negative charge to the target which will propel the positively charged argon-ion towards it. This entire process is done in a high vacuum chamber to avoid contamination from other materials [1].

We made use of 3 different sputtering techniques: DC sputtering, reactive DC sputtering and RF sputtering. DC sputtering is when a DC-voltage is applied to the source material. Reactive DC-sputtering is the same as DC sputtering except that a reactant has been added to the argon gas. In our case the reactant is oxygen [1].

RF (radio frequency) sputtering is when an AC-voltage is applied to the target material [1].

The whole process of sputtering on a silicon wafer and measuring the change in polarisation is as followed: First the systems are made ready for use by logging on the computers controlling the Lesker sputtering machine and the ellipsometer. It is typically a good idea to calibrate the ellipsometer before use. For this a wafer with a silicon oxide layer with a known thickness is used in the sputtering chamber, where the waveguide optical fibre which receives the light reflected from the wafer is aligned for maximum signal by adjusting the angle of the receiver, and afterwards the ellipsiometer program performs a system check.

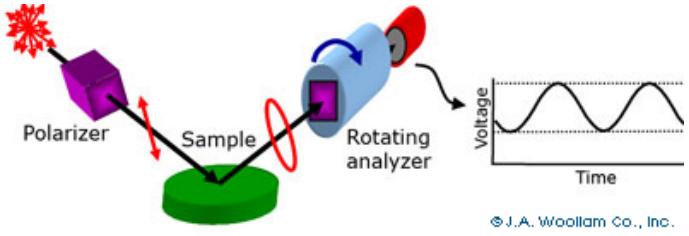


Figur 1: Illustration of the sputtering process. Ar hits the target material and dislodges atoms that bind to the depositions surface.

Then a wafer is prepared by securing on it on a holder, placing it in the load lock, pumping the load lock chamber and finally loading the sample into the main pressure chamber. The desired recipes can then be run. We had all our recipes run from a master recipe 'DC recipe stack'.

2.2 Ellipsometry

In ellipsometry a light source sends unpolarised light through a polariser, given it a specific known polarisation (by allowing only a certain polarisation of light to pass through). This light reflects off the surface of the sample wafer and depending on the deposited material and its thickness the light changes polarisation which is detected in a rotating analyser. The difference in polarisation is measured as an amplitude ratio Ψ, and a phase difference, Δ, which is recorded in the logging program controlling the ellipsometer, CompleteEASE. From these data the ellipsometer fitting program can determine the thickness of thin films and their optical constants, n and k, the refractive index and absorption loss coefficient.



Figur 2: Illustration of the ellipsometer polarisers. [3]

When depositing multiple layers it is possible to measure the change in Ψ and Δ for each layer in-situ and predict the thickness (calculated from the growth rate) of each layer. The process is done by a model constructed and fitted from the measured data. There are several ways to make models for analysing each layer. One method is stacking 'blocks' in the software, each representing a material layer chosen from a database with the bottommost layer being the silicon wafer. If done correctly this model can fit a whole continues dataset of different materials and exclude the parts with noise from switching materials when shutters in the chamber opens and closes and blocks the light.

The 'Growth Rate and Optical Constants (GROC)' model is another possible model which utilises a virtual interface for all the underlying layers other than the current layer that is being

fitted. It creates a pseudo-substrate with properties matching the combined previous layers to simplify the process. For this model you choose a set range of the dataset and fit that section to a material. We haven't looked at all the models and various ways of adjusting them available in the program. We just manually fitted each segment of the data to its corresponding material using the GROC model.

To measure the total height/thickness of the deposited layers we used a Dektak stylus height profiler, which moves a stylus with a set contact force over the surface of the wafer and picks up small vibrations and displacement. If we measure a step height difference between the top layer and the bottom silicon wafer layer we get the total height. This is done so we can compare the calculated fitting thickness and the actual thickness of the film. We use a strip of kapton heat resistant tape to cover a small section of the wafer from deposition. When the tape is peeled off afterwards it gives a nice edge for the Dektak to measure over.

3 Log

8. of June. Starting Monday we were introduced to the cleanroom and the Sputter-System Lesker. Two silicon wafers were deposited with Au and Ti respectively, for testing the deposit rates from previous log recipes. Afterwards they were measured in a Dektak profiler. The thickness for Au was three times higher than expected.

9. A wafer was prepared with three layers, Ti-Si-Ti, with no rotation of the wafer during sputtering, and we measured the polarisation with in-situ ellipsometry after initial alignment of optical fibre. The fitted growth rates and thicknesses matched profiled thicknesses. The last layer, Ti, failed due to too high pressure in chamber.

10. Loading new Si wafer sample. Now with Au and Si alternating for 6 layers total, still no rotation. Afterwards the same configuration was run with rotation. Thickness for both wafers was measured in the Dektak, as with all future wafers. This is Au-Si wafer no. 1.

11. We calibrate the ellipsometer with a Si oxide 25 nm wafer (measured as 28.1 nm). A new Si wafer sample is loaded in the sputter-system and a new different configuration of Au-Si power and pressure parameters was used based on the DOE (Design Of Experiments) method, with rotation for a total 6 layers. Au-Si wafer no. 2.

12. We started by calibrating the ellipsometer with a Si 25 nm wafer again (this is now done every time we log on to the Sputter-System before sputtering). A new sample of Au-Si sample with rotation is run, 6 layers total again. Au-Si no. 3.

15. We now try reactive sputtering by mixing in O_2 with the Ar gas and the Si target to create SiO_2. We make a single layer wafer with a mix of 10 % O_2. This is measured in the ex-situ ellipsometer which can vary the angle of incidence. Next the first wafer with Cu-SiO_2 is run in-situ with varying O_2 percentage. These are layers 1-4. A total of 16 layers, 8 for Cu and SiO_2 are planned in the JMP statistic program with DOE.

16. We continue the series of Cu-SiO_2 wafers no. 2 with layers 5-8 and no. 3 with layers 9-12.

17. Last Cu-SiO_2 wafer is made, no. 4, with layers 13-16. The last two layers of Cu and SiO_2 failed to be properly measured by ellipsometer and gave growth rate results with a very large error. This is probably due to the second last SiO_2 or last Cu layer layer being too thin. This could result in wrong readings as the ellipsometer shines through and measures the wrong layers.

18. We now try RF-sputtering with 0 and 2 % O_2 mixed in when sputtering ITO (Indium-Tin-Oxide). A Au-ITO 4 layer wafer was made. The third layer was aborted because of too high pressure in chamber, but was continued after restart. This resulted in the substrate heater turning on, heating the sample and chamber to 250°C from the normal 20°C. The data from last layer was lost.

19. Another Au-ITO wafer, no. 2, was sputtered with a total 6 layers. Limitations in the Ellipsometer software CompleteEase stopped the data recording prematurely after 2.5 hours but retained most of the data.

22. We continue the Au-ITO wafers based on the planned DOE parameters. Au-ITO no. 3, with 4 layers. The recipe we used had an infinite pressure check loop which we corrected, hereafter the power setpoint didn't set as expected and ramped down when supposed to ramp up. After the sample was done the substrate heater turned on for unknown reasons, heating the sample to 100°C. In the end the Ellipsometer software CompleteEase failed to save the data and froze. Not even leaving it overnight helped and the data was lost.

23. A last Au-ITO no. 4 (now number 3) wafer was made with 8 layers. This includes the failed 4 layers from the 22. and a single failed Cu layer from the 17. substituting an Au layer.

4 Results and discussion

For this experiment we varied the parameters power and pressure used during the sputtering process. For the SiO_2 oxide which was deposited using a Si target and a mix of Ar and O_2 gas, we varied the O_2 in Ar/O_2 ratio from 10 to 50 %. The power in watt was in the range from 50 to 180 watt and the pressure from 3 to 9 mTorr. The ranges were taken from previous runs in the log files. The lower and upper constraint on the pressure was to ensure that the Ar plasma wouldn't extinguish and because the system could not handle to high pressure. The power range was also a result of the limitations of the system. The depositing ITO, Indium Tin Oxide, we chose a lower power because the Sn, Tin, in ITO wouldn't be able to deposit at higher than 75 Watt. Here we chose a Ar/O_2 ratio of either 0 or 2 % according to older recipes.

Except from some of the first layers of Au, the planned parameters used in the experiment was taken from the JMP statistical program using Design of Experiments (DOE). The method gave the most appropriate values for testing and mapping based on the factors and their constraints. The resulting data was modelled with a linear least squares fit in JMP with the factors Power and Pressure and the response Rate. The models or their desirability could then be maximised, given the optimal parameters for the best deposition rate. See Appendix.

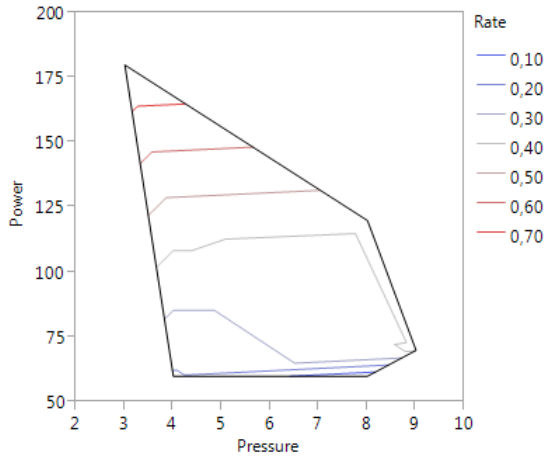
We made 3 different wafers with alternating 3 layers each of Au and Si, and 4 wafers with 8 alternating layers of Cu and SiO_2. Lastly we made 3 wafers with alternating layers of Au-ITO (gold and Indium Tin Oxide). Au layers were deposited for 480 sec., Si for 600 sec., Cu for 600 sec., SiO_2 for 1200 sec. and ITO for 1800 sec. because of earlier logged deposition times.

The model for Au shows optimal power around 180 Watt and pressure at 3 mTorr for a deposit rate of 3.81 Å/s, see fig. 10(a) in Appendix, but the model used to get these values does not fit the data very well. It has R^2-value of 0.71. It is likely that Au is not well described by a linear model, but that is difficult to conclude because we don't have a lot of confidence in our data for Au.

This is because when fitting the data from the ellipsometer we noticed that Au was very sensitive to time range chosen for the fit and therefore it is hard to figure out which calculated rate is the correct one. When fitting you want the MSE (Mean Square Error) to be as low as possible but at the same time have enough data points for a meaningful fit.

The data also shows some strange behaviour. For instance the data shows that the rate is greatest around 120 W and 6 mTorr and 180 W and 3 mTorr. In between these two points it drops to a minimum as seen on figure 7. We can't come up with any explanation for why this is the case other than that our data are incorrect. For these reasons we don't have much confidence in our data for Au.

The optimal power and pressure for Si according to the model is 180 Watt and 3 mTorr, which gives a predicted rate of 0.77 Å/s (see 10(b) in Appendix). The model has a R^2-value of 0.92. It can also be seen from fig. 3 that the max rate is found at 180 W and 3 mTorr though we are



Figur 3: Contour plot for Si

missing some points around 180 W and 9 mTorr that could perhaps be interesting to measure if the experiment is repeated at some other time to see whether the maximum is only at 3 mTorr or also at 9 mTorr.

Tabel 1: Data from Au-Si and Au-ITO wafers and the parameters used, power in Watt, pressure in mTorr, Ar to O_2 ratio and the response growth rates calculated from the model fit is in ångström per second.

Power	Pressure	Rate
110	7	2.74
150	5	1.82
120	4	3.74
80	6	1.97
70	5	2.03
140	9	3.48
180	3	3.98
50	8	1.23
80	4	2.07
50	9	1.25
180	9	1.79
115	3	2.64
180	6	4.42
180	3	3.75
50	9	1.14
50	3	1.31
50	3	1.25

Tabel 2: Results for Au

Pressure	Power	Rate
9	70	0.41
4	120	0.4
8	60	0.04
8	120	0.43
4	60	0.19
5	90	0.32
3	180	0.79
4	120	0.5
7	100	0.31

Tabel 3: Results for Si

Pressure	Power	Ar/O2	Rate
9	25	0	0.03
6	50	0	0.15
9	50	2	0.1
6	75	2	0.25
9	75	0	0.23
6	25	2	0.05
3	75	0	0.31
3	25	0	0.1
3	50	2	0.22

Tabel 4: Results for ITO

The data for SiO_2 shows a linear behaviour for the 3 Ar/O_2 ratios and it is clear that for every one of them 180 W and 3 mTorr are the points with maximum rates and 10% oxygen is the one with the greatest rate. This can be seen on fig. 6. This is also confirmed by the linear model (see fig. 11(b) in Appendix) . It shows that the maximum rate of 1.50Å/s is at 3 mTorr, 180 Watt and 10 % Ar/O_2 ratio. With an R^2-value of 0.85 it is not best of linear fits but the trend from the contour plots also shows that lower pressure and higher power gives a higher rate so we are

Tabel 5: Data from Cu-SiO_2 wafers and the parameters used, power in Watt, pressure in mTorr, Ar to O_2 ratio and the response growth rates calculated from the model fit is in ångström per second.

Pressure	Power	Rate
6	115	1.05
6	115	1.47
3	180	1.52
9	115	1.41
9	50	0.42
6	180	0.22
9	50	0.67
9	50	0.85
3	50	0.6
3	115	1.52
3	115	1.45
6	50	0.72
3	180	1.91
9	180	1.94
3	50	0.69

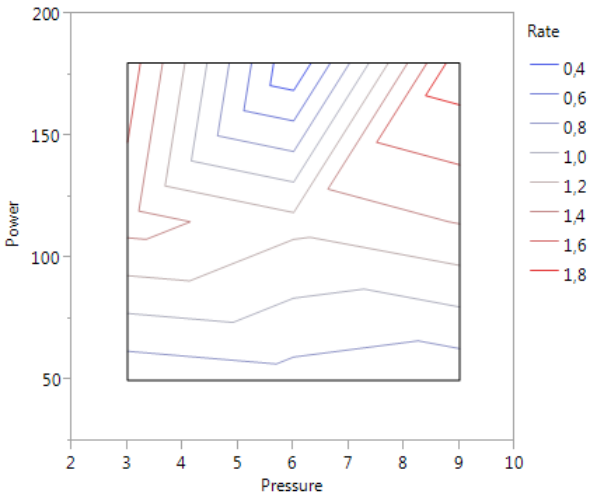
Tabel 6: Results for Cu

Pressure	Power	Ar/O2 ratio	Rate
3	50	10	0.3
6	115	30	0.53
9	50	10	0.54
9	115	30	0.48
9	50	50	0.4
6	115	10	0.79
6	115	30	0.58
6	50	30	0.37
9	180	10	1.08
3	180	10	1.45
3	115	30	0.74
6	115	50	0.21
9	180	50	0.59
3	50	50	0.3
3	180	50	1.11

Tabel 7: Results for SiO_2

confident about that the highest rate is found at 3 mTorr and 180 W.

Cu, like Au, has data that looks random as seen on fig. 4. It has 2 maxima at 180 W and 3 mTorr and 9 mTorr respectively. In between at 180 W and 6 mTorr it has a minimum. Like with Au, we can't explain this behaviour other than stating that there is some error in the data. The linear model only has a R^2-value of 0.63. It shows that the maximum is 1.66 Å/s at 3 mTorr and 140 Watt (see fig. 11(a) in Appendix) but we don't believe it because of the aforementioned reasons.



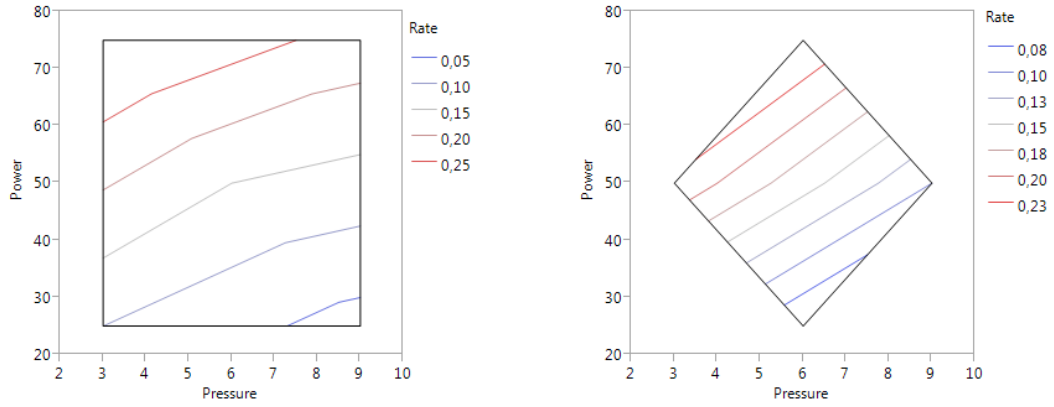
Figur 4: Contour plot for Cu

ITO as seen on fig. 5 has a linear behaviour. The R^2-value of the model is in fact 1. This is probably due to the small number of points and that ITO has a small rate and so is easy to measure. Both plots (fig. 5) show that the maximum rate is found at 180 W and 3 mTorr and

Wafer	Thickness measured	Thickness calculated
Au-Si 6 layers no. 1	379 nm	415.44 nm
Au-Si 6 layers no. 2	456 nm	449.4 nm
Au-Si 6 layers no. 3	372 nm	445.44 nm
Cu-SiO_2 8 layers no. 1	487 nm	549 nm
Cu-SiO_2 8 layers no. 2	320 nm	386.4 nm
Cu-SiO_2 8 layers no. 3	605 nm	675 nm
Cu-SiO_2 8 layers no. 4	623 nm	705+ nm
Au-ITO 4 layers no. 1	430 nm	217.9+ nm
Au-ITO 6 layers no. 2	645 nm	623.28 nm
Au-ITO 8 layers no. 3	326 nm	341.4 nm

Tabel 8: Thickness measured Dektak and calculated by model for each wafer and its materials. The plus signifies that we lost data for some of the final layers so we know that the calculated layer thickness is at least greater than the specified thickness.

the model confirms this. In fact it shows that the highest is 0.32Å/s at 180 W, 3 mTorr and 2% oxygen.



(a) Contour plot for ITO at 0% oxygen

(b) Contour plot for ITO at 2% oxygen

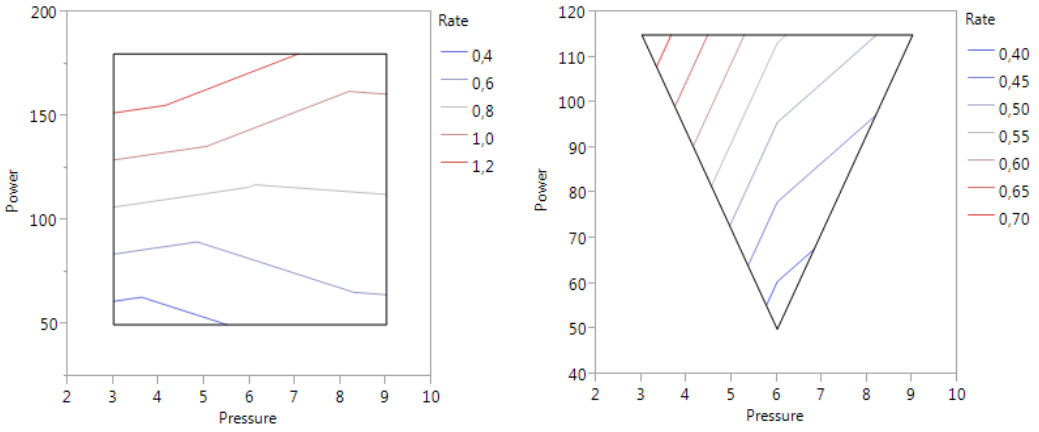
Figur 5: Contour plots for ITO with the 2 different Ar/O_2 ratios, 0% and 2%

Keep in mind that these rates are predicted from data which was fitted segmentally in the ellipsometer program and therefore accuracies may vary. In fig. 10(a) for gold the error range of the rate is quite high (around 1.03 Å/s for the upper and lower bound) while the R^2 number compared to the other materials is low.

To conclude we found that Au had its max rate of 3.81 Å/s at 180 W and 3 mTorr. Cu is at 3 mTorr and 140 W with a value of 1.65 Å/s. Si has a max rate at 3 mTorr and 180 W with a value of 0.77 Å/s. SiO_2 has a max rate at 180 W, 3 mTorr and 10% oxygen in Ar with a value of 1.50 Å/s. ITO has a max rate at 3 mTorr, 75 W and 2% oxygen in Ar. We don't have much confidence in the values for Au and Cu.

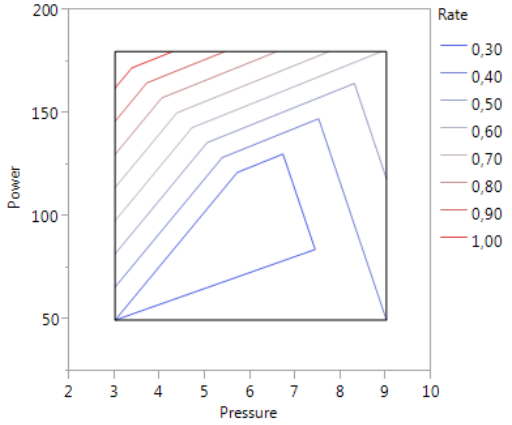
5 Errors and suggestions for future experiments

When we measured the total thicknesses of our wafers they were almost always greater than the calculated ones. An explanation for this could be that we measured the thickness with the



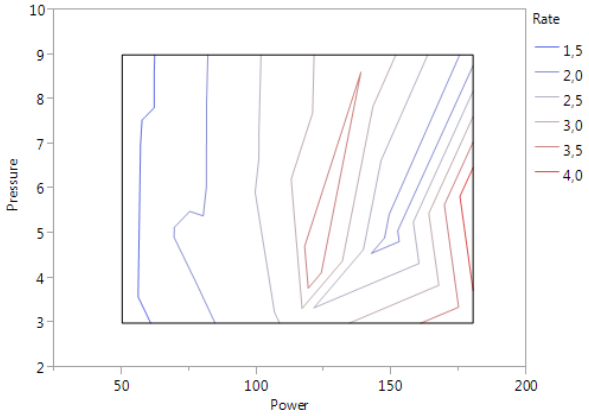
(a) SiO2 with Ar/O_2 ratio of 10 %

(b) SiO2 with Ar/O_2 ratio of 30 %



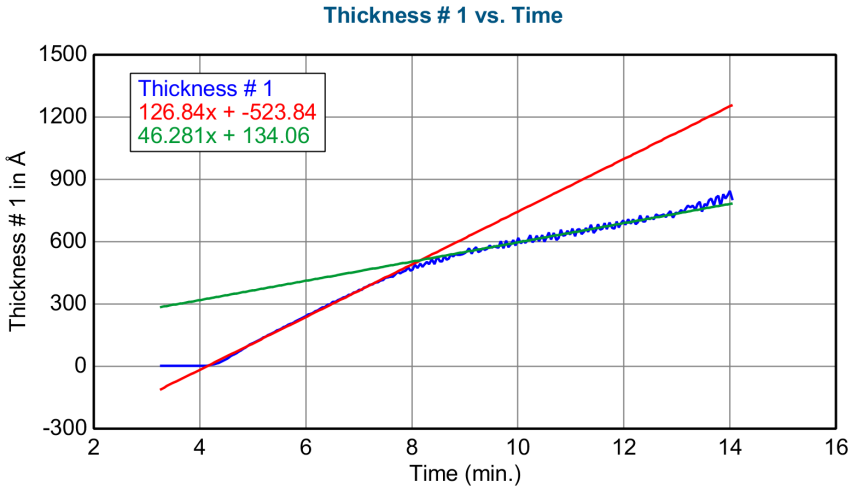
(c) SiO2 with Ar/O_2 ratio of 50 %

Figur 6: Contour rate plots of the different Ar/O_2 ratios of SiO_2



Figur 7: Contour plot of Au deposit rate.

Dektak at the edge of the wafer while the ellipsometer measured at the center of the wafer and the deposited material is not evenly distributed across the wafer. Instead more material was deposited in center of wafer than the edges. We deposited 6 or 8 layers on every wafer so the error will compound and that might explain why the difference is as high as 50-80 nm. A map



Figur 8: Figure depicting the growth rate of the first Cu layer of wafer Cu-SiO_2 no. 4. The red and green line show the two different growth rates and the change approximately at the 8th min. mark.

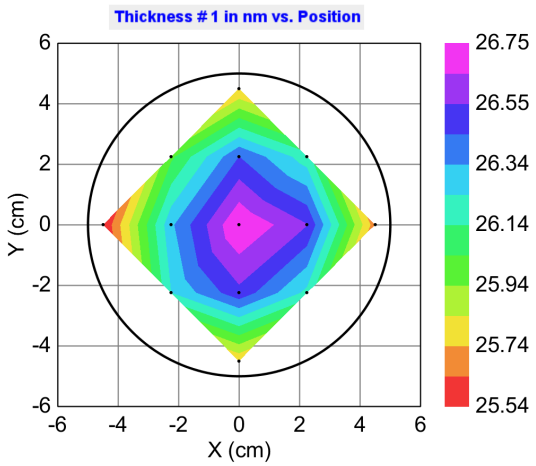
scan of a wafer with a single layer of SiO_2 can be seen down on fig. 9. Here a small difference in thickness across the wafer can be seen.

Another thing is that when fitting the model to the data, it is hard to determine where to start the fit and in some samples the start of the fit has a significant effect on the rate. If the fit starts too early the nucleations phase is included. The nucleation phase is the phase where a layer of the deposited material has not been formed yet. If the fit starts too late data is lost which might result in a different rate being calculated.

Another thing to note is that the GROC-model assumes the growth rate to be constant and that might not always be true. When we the thickness deposited material on single layer wafers as a function of time we saw that not all of them had a constant growth rate. This can be seen on fig. 8 for a Cu-layer.

Lastly we observed that when some layers became very thin or very thick, the ellipsometer had problems measuring them, this relates to the way he light has to pass though the layer and reflect at boundaries.

Suggestions for further characterisation of the sputter deposition rates could include variation of time and temperature, to see if the thickness/deposition rate could be maximised while surface roughness could be kept to a minimum. It could be interesting to examine how early a smooth layer formed for a specific material, minimum deposit time, and perhaps if a maximum recommended deposit time exists in combination with temperature, power and pressure to avoid a change in growth rate or an uneven layer. Because surface roughness adds up through the layers, the data logging with the ellipsometer would most likely give better results if fewer layers (two or even one) were stacked per wafer. We already observed problems with fitting using just 8 layers if some of the individual layers were very thin. Making fewer stacked layers of course takes more time and requires more wafers.



Figur 9: Figure depicting a map scan of a wafer wit a single layer of SiO_2. This was done on the ex-situ ellipsometer VASE, measuring in a grid on the surface.

Litteratur

[1] Andrew H. Simon, *Handbook of Thin Film Deposition - Third Edition*, Chapter 4: Sputtering Processing , 2012 Elsevier Inc.

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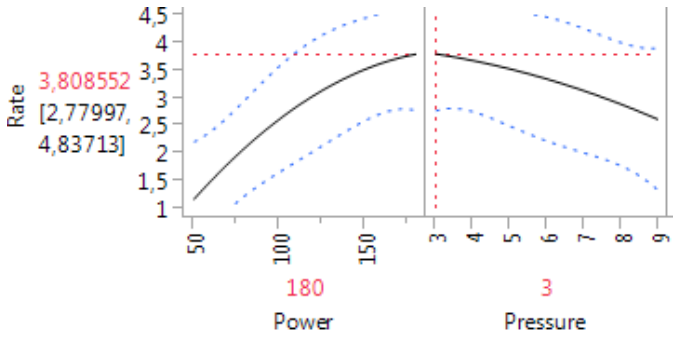
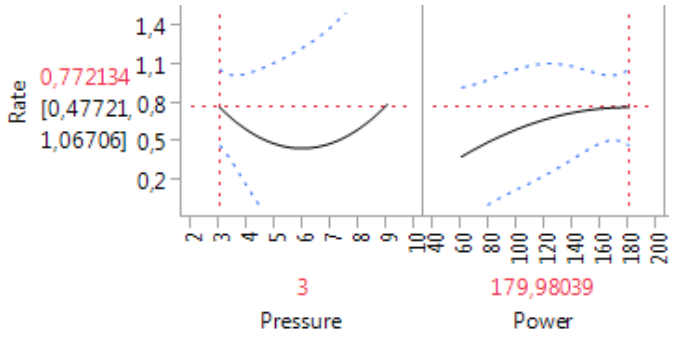
[4] J. A. Woollam Co., Inc., *CompleteEASE - Data Analysis Manual*, October 5, 2011.

A Appendix for desirability functions

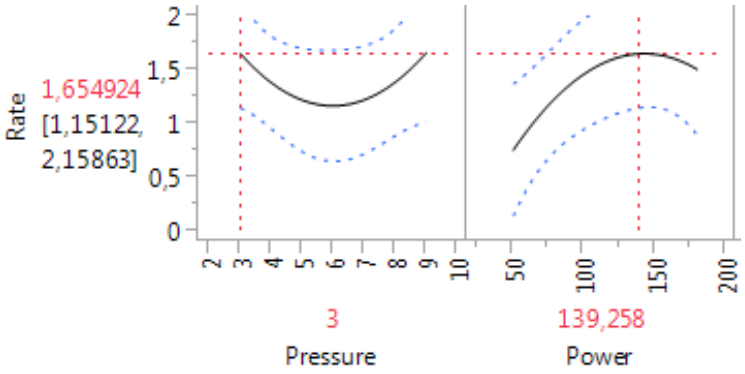


(a) Maximised Au deposit rates.

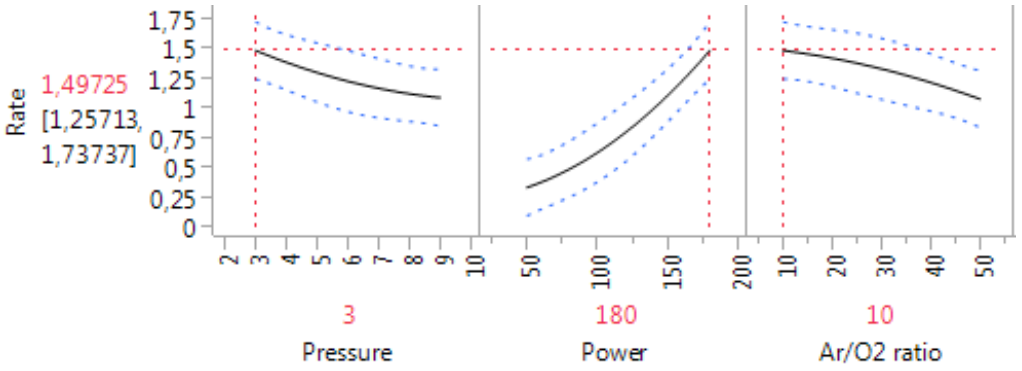


(b) Maximised Si deposit rates.

Figur 10: Maximised desirability functions for Au and Si deposit rates depending on power and pressure.

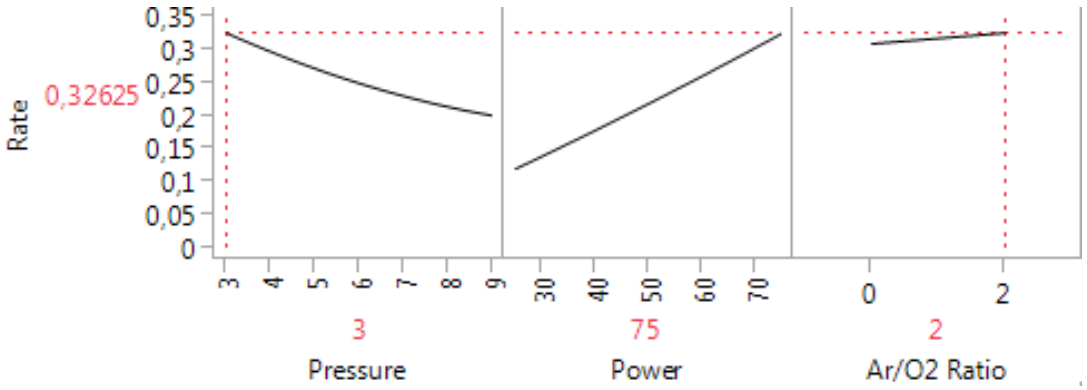


(a) Maximised Cu deposit rates.



(b) Maximised SiO_2 deposit rates.

Figur 11: Maximised desirability functions for Cu and SiO_2 deposit rates depending on power, pressure and Ar/O_2 ratio.



Figur 12: Maximised desirability functions for ITO deposit rates depending on power, pressure and Ar/O_2 ratio