PEALD deposition of AlN using TMA and NH_3

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Journal for Mikro-3W: Experimental Semiconductor Technology (33470) Technical University of Denmark June 22, 2017

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1 Introduction

Aluminum nitride (AlN) is a piezoelectric material, with various applications in optoelectronics and as a semiconductor. Specifically, AlN has a large and direct band gap (6.2 eV), high dielectric constant (9.14), large piezoelectric coefficient (5.1 pm V⁻¹), high electrical resistivity (1013 Ω cm), high thermal conductivity, and doesn't expand much at higher temperatures. An advantage of AlN, when compared to other piezoelectric materials, is that atomic layer deposition (ALD) allows it to be deposited in a CMOS–compatible process, as it can be deposited at temperatures below 400 °C.[1, 2]

The purpose of this project is to establish an optimized recipe for depositing aluminum nitride (AlN) on silicon (Si), using trimethylaluminum (TMA) and ammonia (NH₃) plasma as precursors in a Plasma Enhanced Atomic Layer Deposition (PEALD) process. The goal is to produce a highly uniform AlN layer, with low roughness, low amount of surface particles and impurities (specifically oxygen and carbon contaminants), at a decent deposition rate.

With this goal in mind, several parameters of the PEALD process will be varied. In order to limit the amount of required experiments, we will be utilizing the design of experiments (DOE) software JMP to facilitate our recipe optimization.

To investigate the PEALD-processed wafers, we will be using ellipsometry, scanning electron microscopy (SEM), atomic force microscopy (AFM), X-ray photoelectron spectroscopy (XPS) and a particle scanner, to investigate the thickness, structure, roughness and composition, and surface particles on top, of the deposited layers.

1.1 ALD & PEALD

Chemical Vapor Deposition (CVD) is a chemical process used for deposition of materials onto a substrate. During the process the substrate is exposed to a different set of volatile precursors. These combine in their gaseous form, and deposit onto the surface of the sample, to either create or decompose materials on the surface, to create the desired deposition, while by-products are removed by inert gas flows in the chamber. CVD can be used to deposit materials of various forms, such as amorphous or other crystalline states.

Atomic Layer Deposition (ALD) is a subclass of CVD, which is especially good at making depositions of epitaxial film. Epitaxial growth means that the deposited layer has the same crystalline structure, as that of the substrate it is getting deposited onto. Additionally, ALD allows the homogenous coating of structures with high aspect ratios.

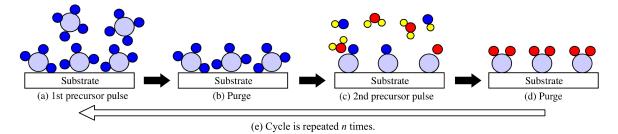


Figure 1: The ALD cycle.

ALD works by having the precursors react to the surface one at a time, in a sequential and self-limiting way. As most ALD is done with two precursors, this results in a process flow where the substrate is first exposed to precursor A, followed by a chamber purge. Then exposure to precursor B, again followed by a chamber purge. All this is repeated for n cycles, as shown in Figure 1. By repeating the cycles in this way, the ALD layers are slowly deposited, monolayer by monolayer, onto the surface of the sample. However, for this to work, precursor A must be able react with the surface of the sample, for the deposition to be initiated. It is also important that all of the remaining precursor, not attached to the surface, is purged away, before the next precursor is introduced. This ensures that the reaction only takes place on the surface, and not in the gas phase (like CVD).

Plasma Enhanced Atomic Layer Deposition (PEALD) is a different technique to thermal ALD. Here, one of the precursors uses plasma as an energy source. This allows the reaction to obtain the necessary amount of activation energy, without the need for high temperatures. The use of plasma can thus allow the deposition of materials not previously possible with thermal ALD. One downside to PEALD is that it can only deposit on one sample at a time and very slowly, unlike thermal ALD.

In this study we use TMA and NH_3 as precursors to deposit AlN: $C_3H_9Al + NH_3 \rightarrow AlN + by$ products, where NH_3 is the plasma enhanced precursor and TMA is the gas precursor.

2 Experimental Setup

The Danchip clean room PEALD is the Picosun R200 PEALD, with a Picosun Handyman load lock:

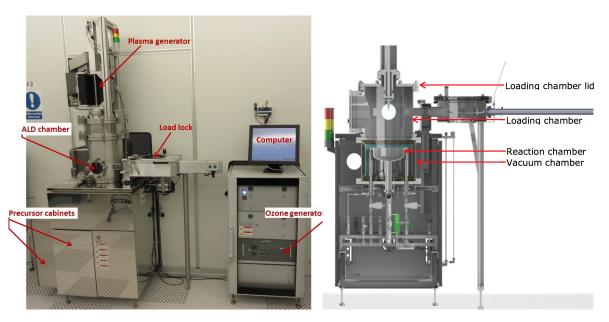


Figure 2: The Picosun R200 PEALD with plasma Figure 3: Load lock and PEALD chamber generator installed.[3] schematic. The plasma cone is not shown.[4]

The PEALD reaction chamber has a dual chamber structure, where the inner chamber is the reactor chamber, while the outer chamber is an isolating chamber. The inner chamber has no heating elements, but instead rely on heating elements placed between the two chambers. The advantage of using this setup to control the temperature flow, is that the temperature in the inner chamber is more stable and uniform, even if the temperature fluctuates slightly in the outer chamber. However, the inner chamber is 18% lower in temperature than the displayed heating temperature, since the system expects a thermal lid to be installed, whereas the plasma cone is not heated. Additionally, this setup requires some stabilization time for the temperature. Both chambers are under vacuum to further reduce the influence of the outside air, and they are also constantly purged with nitrogen.

Before a deposition process can be started, a wafer must be loaded into the chamber. This is done by placing a wafer of either 4-inch (100 mm), 6-inch (150 mm) or 8-inch (200 mm) sizes into the load lock. The lid is then closed, and from here on, the rest of the work is done through the accompanying software (with the exception of insertion and extraction of the load lock arm with the wafer wafer between the load lock and the PEALD chamber, which is done manually using buttons on the load lock).

First, the pressure of the load lock is reduced to match the PEALD chamber (typically 10 hPa). During this process, the load lock is flushed with nitrogen through a series of pump-purge cycles, which is done manually using the software. This reduces the amount of oxygen in the load lock, which could contaminate the ALD process.

After the load lock has been purged, it is time to insert the wafer into the PEALD chamber. The arm of the load lock is extended into the PEALD chamber and the sample holder picks up the wafer. After the wafer has been inserted, the arm is retracted, and the gate valve between the PEALD and the load lock is closed. The sample is then lowered into the chamber, as the lid of the PEALD moves down onto the chamber. All there is left then, is to select the desired recipe, and start the deposition.

When it comes to producing a recipe, there are several aspects of the process to consider:

- The temperature of the reaction chamber (incl. stabilization time)
- The TMA precursor pulse parameters (pulse time, purge time)
- The NH₃-plasma precursor pulse parameters (flow rate, plasma power, pulse time, purge time)
- The chamber purging parameters (flow stabilization time, purge gas flow rate)

In regards to the parameters of the plasma, we have to consider the amount of energy we want to use, to create the plasma, the gas flow used and the pulse time and purge time of the plasma.

Even before we start to consider the other parameters, we have already amassed quite a few of them, and it is quite obvious, that we will not be able to consider them all, in our experiments. Therefore we create a plan, a so called Design of Experiments (DOE), where we only choose the most important parameters to vary, outlined below.

2.1 Design of Experiments

Using the DOE software JMP, we setup a definitive screening design, which allows us to vary multiple factors at the same time, with the goal of determining which of and how the factors have an effect on the quality of the deposition.

In this experiment we vary four of the ALD-deposition parameters:

- The reaction chamber temperature, from 200 °C to 450 °C (corresponding to an 18% lower actual "in-reaction-chamber" temperature of 164 °C to 369 °C).
- The NH₃-plasma pulse time, from 1 s to 4 s.
- The NH_3 -plasma flow rate, from $50 \, sccm$ to $200 \, sccm$.
- The NH_3 -plasma purge time, from 8 s to 20 s.

The rest of the parameters are kept constant:

- We deposit using 350 cycles on 6-inch $(150\,\mathrm{mm})$ wafers.
- With TMA pulse time at $0.1 \, \text{s}$, TMA purge time (with N_2) at $4 \, \text{s}$.
- With plasma power at 3000 W.
- With all the purge and carrier gas flows held constant at standard values.

We then look at the following qualities of the produced wafers:

- Particle count on the wafer surface (measured with a TENCOR SurfScan particle scanner)
- Minimum, average & maximum thickness (measured with a VASE M2000 ellipsometer), giving:
 - The non-uniformity percentage, calculated as: $\left(\frac{\text{Max. thickness-Min. thickness}}{2 \cdot \text{Avg. thickness}}\right) \cdot 100.$
 - The growth rate per cycle (GR/C), calculated as: $\left(\frac{\text{Avg. thickness}}{\text{Number of cycles}}\right)$.
- Roughness (measured with a Bruker Dimension Icon-PT AFM).
- Composition of deposited layers (measured with Thermo Scientific K-Alpha XPS)

The higher the temperature in the reaction chamber, the more energy the precursors have for bonding. But if the temperature exceeds 370 °C the TMA precursor will begin to decompose, and if the temperature is too low the precursors will not react. Previous studies have found that the growth temperature has a "window", where the growth rate of the AlN almost is independent of the temperature.[5]

The pulse time of the NH_3 -plasma needs to be long enough to allow the bonding of the nitrogen with the aluminium on the substrate. Increasing the pulse time beyond the necessary amount is excessive, since it will not enhance the PEALD process. Same with the NH_3 -plasma flow rate: we need enough

to allow a complete monolayer to be deposited, but an excessive flow rate will not enhance the process. Finally, after one NH_3 -plasma pulse, the reaction chamber needs to be purged with N_2 and the plasma cone with Ar for long enough, such that no reactive species and byproducts from the NH_3 -plasma are left behind in the reaction chamber before the TMA pulse begins.

3 Experimental Results

3.1 PEALD Runs

In the tables below, samples that have been analysed with XPS or SEM are marked with **XPS** or **SEM**, respectively. Samples that have been photographed are marked with **IMG**. Recipes that have been changed, compared to the parameters outlined in Section 2.1, have corresponding footnotes in the tables outlining the changed parameters. Note again that the actual temperature in the reaction chamber is 18% lower than the temperature parameter.

For the experimental ALD depositions, we performed a series of "Pre-DOE" runs to analyze and thus get a feeling for what settings we want to keep constant. These are shown in Table 1.

Run	Temp.		NH ₃ -plasma	a	Avg.T.	GR/C	Particles	Roughness	Nonunf.
Tun	$[^{\circ}C]$	flow [sccm]	$pulse\ time[s]$	purge time [s]	[nm]	[nm]	#	[nm]	%
$170606C_{se}^{1}$	м 350	100	2	12	58.27	0.083	774	n/a	4%
$170607A^{2}$	350	100	2	15	30.18	0.086	409	0.860	9%
$170607B_{xr}^{3}$	s 350	100	2	15	37.72	0.094	484	1.020	6%
$170607C^4$	200	125	4	20	32.38	0.032	2347	0.457	20%

¹ With 2000 W plasma power & 700 cycles. ² With 2000 W plasma power. ³ With 400 cycles. ⁴ With 1000 cycles.

Table 1: The 4 runs before the Definitive Screening DOE.

After the main settings were selected, we created a Definitive Screening DOE in JMP, using the min. and max. values mentioned in Section 2.1, which gave us 17 runs which can be seen in Table 2.

Run	lemp.		$\mathrm{NH_{3} ext{-}plasma}$		Avg.T.	GR/C	Particles	Roughness	Nonunf.
	$[^{\circ}C]$	flow [sccm]	pulse time [s] p	ourge time [s]	[nm]	[nm]	#	[nm]	%
$170608A_{XPS}$	200	50	4	20	21.69	0.062	1029	0.443	1%
170608B	450	125	1	20	50.80	0.145	543	0.987	6%
170608C	200	200	1	20	5.03	0.014	1268	0.808	20%
$170609A_{img}$	200	200	1	8	6.09	0.017	1317	0.255	25%
170609B	325	200	4	20	37.32	0.107	394	1.190	4%
$170612A_{img}$	450	200	2.5	20	57.22	0.163	772	1.070	5%
$170613A_{\text{IMG}}$	200	50	1	20	6.57	0.019	1463	0.253	10%
170613B	200	125	4	8	23.43	0.067	1602	0.576	4%
170613C	450	200	1	8	57.40	0.164	454	1.120	8%
$170614A_{XPS}$	450	50	4	8	49.36	0.141	725	0.917	4%
170614B	325	50	1	8	13.94	0.040	620	0.515	13%
170614C	325	125	2.5	14	31.82	0.091	701	0.896	6%
$170615A_{\text{IMG}}$	450	200	4	8	60.20	0.172	374	1.08	5%
170615B	200	200	4	14	24.95	0.071	2070	0.608	5%
170615C	450	50	1	14	48.03	0.137	1288	0.857	7%
170616A	200	50	2.5	8	14.53	0.042	1506	0.373	3%
170619A	450	50	4	20	47.32	0.135	566	0.813	4%

Table 2: The 17 Definitive Screening DOE runs.

Finally, after the 17 runs were performed and analyzed, we chose the 170607B recipe as the most suitable for performing a PEALD deposition of AlN on a series of silicon wafers with, $400 \,\mathrm{nm}$ wide and $4.5 \,\mathrm{\mu m}$ deep, pitch trenches to show the unique ability of PEALD to deposit homogeneously on high aspect ratio structures. The deposition on trenches was performed two times, with the latter using increased purge times after both the TMA and NH₃-plasma precursor pulses.

Run Temp.		$\mathrm{NH_{3} ext{-}plasma}$			Avg.T.	GR/C	Particles	Roughness	Nonunf.
Tun	$[^{\circ}C]$	flow [sccm]	pulse time [s]	purge time [s]	[nm]	[nm]	#	[nm]	%
$170616B_{se}^{1}$	з50	100	2	15	61.00	0.087	n/a	n/a	n/a
$170619B_{SE}^{2}$	_{ем} 350	100	2	30	63.29	0.090	n/a	n/a	n/a

 $^{^{1}}$ With 700 cycles. 2 With 700 cycles and TMA purge time at 8 s.

Table 3: The 2 ALD depositions on silicon wafers with trenches.

It should be noted, as mentioned in Section 2.1, that the there is a typical ALD temperature window. For PEALD deposition of AlN, this is around the 300 °C-350 °C temperature range.[5] The 200 °C and 450 °C parameter choices (corresponding to an actual temperature of 164 °C and 369 °C respectively) are both outside the expected limits of this ALD window, and will thus likely result in either a too low growth rate per cycle (GR/C), for an effective monolayer growth per cycle (as is expected of ALD), or one so high that we are are actually dealing with a CVD process. The temperature 450 °C is chosen specifically so the actual temperature in the PEALD chamber is 369 °C. This is because TMA starts to decompose at 370 °C. However we have no concrete proof that the temperature in the PEALD chamber is precisely 18% lower than the input temperature. Generally, for an PEALD deposition of AlN we would expect the GR/C to be around 0.08 –0.1 nm per cycle[1]. Growth rates near this value have been made bold in the tables above, which indicate runs that likely were near or within the ALD window.

3.2 General Analysis

3.2.1 Non-uniformity

An ellipsometer is used for the uniformity measurements. It is an optical technique that measures the shift in polarization as light reflects or transmits. This polarization shift depends on the samples properties, and can thereby determine thickness. Figure 4 is an example of such a measurement.

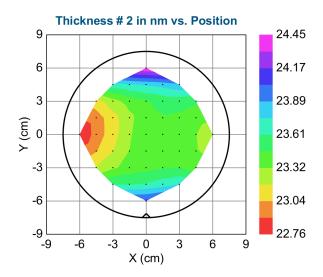


Figure 4: An ellipsometer measurement showing the distribution of the thickness of the AlN layer on the wafer. In this case it is the 20170613B wafer.

- The two major factors in the non-uniformity of the deposition, are the temperature and the plasma pulse time. From Figure 5 we clearly see that when the temperature or plasma pulse time increases, the non-uniformity decreases.
- Both the increase of temperature and plasma time seems to decrease the deviation, of the non-uniformity, although increasing the plasma time seems to do this slightly more than increasing the temperature.
- Increasing the purge time seems to have no effect on the non-uniformity of the deposition.
- It is uncertain if an increase in plasma flow increases the non-uniformity. It might make a small increase in the non-uniformity, but for the given case of the two points above 20 percent, it is probably a combined effect of low temperature and low plasma time, that makes the biggest difference.

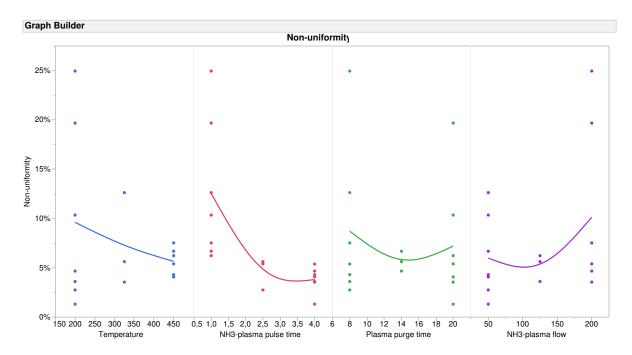


Figure 5: Non-uniformity as a function of temperature, plasma pulse time, plasma purge time and plasma flow

3.2.2 Growth Rate

- We see on Figure 6, that the temperature plays a heavy role, when it comes to the growth rate. There is also some indication that the deviation, of the growth rate, is reduced at higher temperatures, although it is hard to be certain of this, as the deviation at 325 degrees seems higher than the deviation at 200 degrees.
- An increase in plasma time slightly increases the growth rate, but it could also be that it reduces the margin of error.
- For the plasma purge time there seems to be no correlation with the growth rate.
- For the plasma flow there is a slight indication, that it affects the growth rate. However, the large deviation of points makes it difficult to be certain.

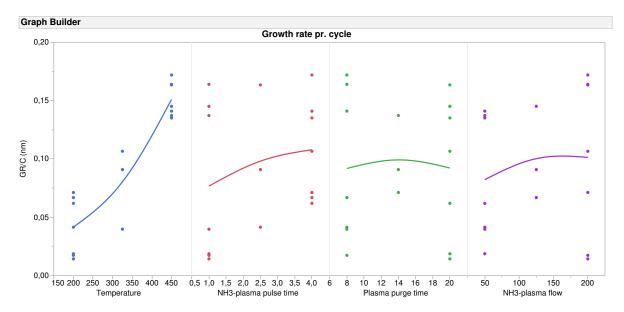


Figure 6: Growth rate as a function of temperature, plasma pulse time, plasma purge time and plasma flow

3.2.3 Particles

To measure the particles on the samples, we used the TENCOR SurfScan particle scanner. The SurfScan scans the surface of the sample for particles in a given range of size. The SurfScan then returns with a list of size intervals and the amount of particles that fits the different intervals. The size ranges are user defined, which means that you can always search for smaller and smaller particles on a sample.

- From Figure 7 it seems that the temperature has an effect on the amount of particles. The optimal temperature is around 360 °C, and either going lower or higher than this temperature increases the amount of particles.
- For the plasma time, there seems to be no correlation with the amount of particles.
- For the purge time, we see indications of an unoptimal value at 14 s. But there is no clear correlation
- The plasma flow, shows to have no influence on the amount of particles.

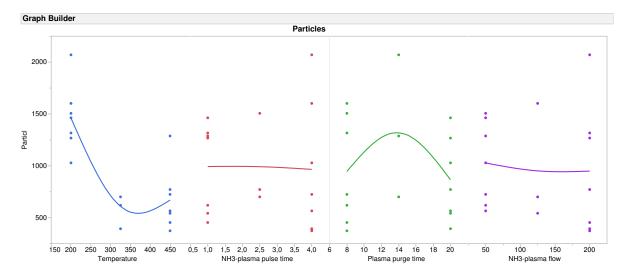


Figure 7: Particles as a function of temperature, plasma pulse time, plasma purge time and plasma flow

3.2.4 Roughness

Although an ellipsometer measurement is great for getting an idea of the overall thickness of the deposition, we need something else if we really want to know what the surface looks like. For this we use an AFM. The AFM lets us see variations on the surface, that can be as small as a few atoms. As the AFM makes such precise measurements, it is also really slow. A scan of a 500 nm times 500 nm area takes 17 minutes. Therefore we limited our search to one of these scans, for each wafer. An example of one of these measurements can be seen below:

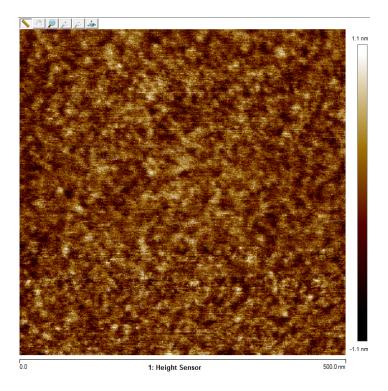


Figure 8: An AFM measurement showing the distribution of AlN on a, 500 nm times 500 nm, area of the wafer. This is sample 170609A.

- When it comes to the roughness of the sample, we can quite clearly see on Figure 9 that the temperature plays a role. An increase in temperature also increases the roughness of the sample. It is also quite interesting to note, that the increase of temperature, while increasing the roughness, also decreases the deviation of the roughness.
- Both the plasma time and plasma purge on the other hand, show almost identical tendencies. However both of these seem to have almost no effect on the roughness of the sample.
- An increase in plasma flow seems to increase the roughness. However, as the deviation of points is quite large, it is hard to say, if this is the case.

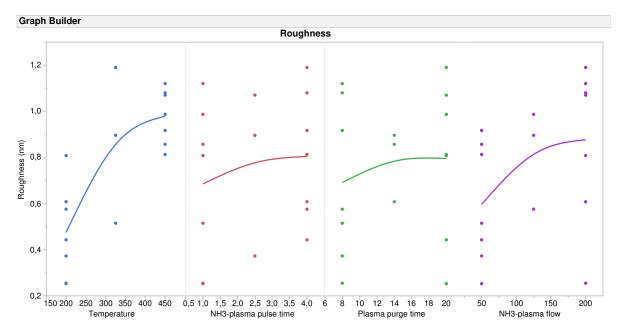


Figure 9: Roughness as a function of temperature, plasma pulse time, plasma purge time and plasma flow

3.2.5 JMP DOE optimization

From the data we have collected using the parameters of the DOE, we can use JMP to optimize the recipe. This recipe should hopefully give the best possible values for the non-uniformity, growth rate, amount of particles and roughness.

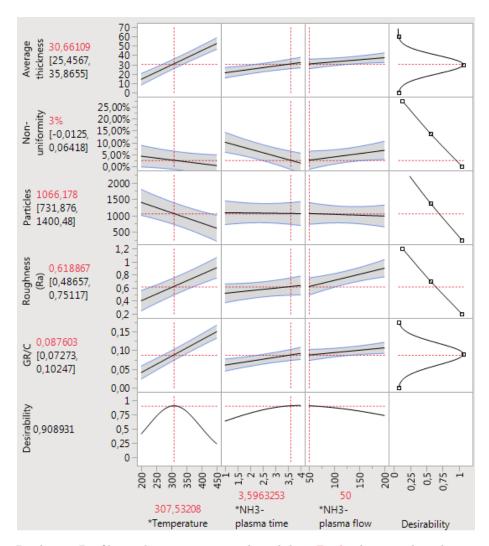


Figure 10: Prediction Profiler tool set to maximize desirability. Red values are the values at maximum desirability.

The desirabilities for our DOE were as follows:

- A deposition layer thickness of around 30 nm $\,$
- A non-uniformity as close to 0 percent as possible
- The lowest amount of particles possible
- A roughness as close to 0 nm as possible
- A growth rate of 0.08 nm pr. cycle

However, as we have seen from our collected data, these properties are all affected by temperature and some of them are also affected by the plasma pulse time, plasma purge time and the plasma flow, but not necessarily in the same way. This is also quite clear in the profiler data, as some of the qualities have suffered a bit, in exchange for better values in some of the others.

The thickness and growth rate is pretty much as we wanted it. The non-uniformity of 3% is also really good. However the amount of particles are a bit on the higher end and the roughness could perhaps be lower, but it is to be expected, since all the qualities are largely affected by the temperature.

3.3 XPS Analysis

The following four graphs are the XPS data for sample 170607B, 170608A, 170608B and 170614A.

We expect the data to show a somewhat even amount of Al and N as they react in a 1:1 ratio. When the etching exceeds the deposited AlN layers, we should see silicon (the wafer) exclusively. The oxygen and carbon are contaminations. The oxygen comes from the wafers being subjected to air. The wafers were stored in a nitrogen cabinet to prevent further contamination. First the sample is etched with a beam of argon ions for an specific amount of time, then the XPS analysis can begin. This is done stepwise through the sample. One iteration is one "step", which entails an etching and a XPS analysis. The following are sputter profiles, which means all data from each iterations is collected in one graph.

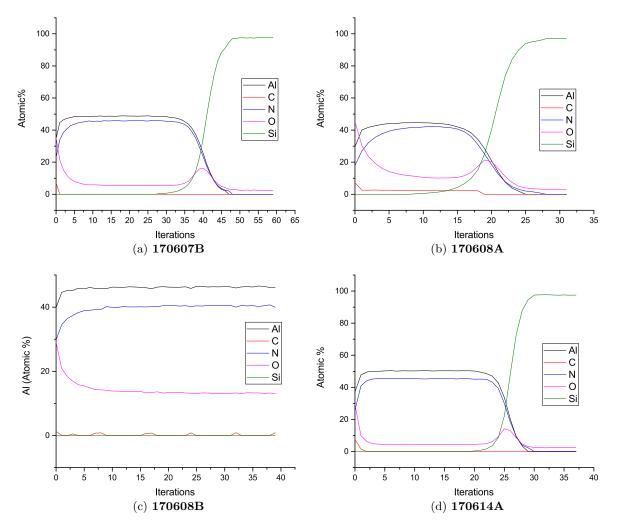


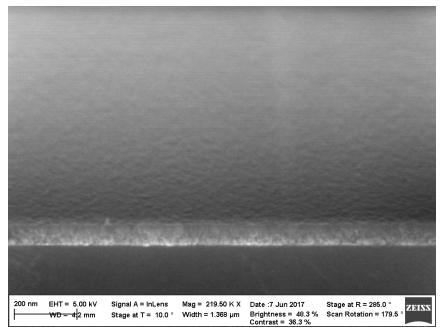
Figure 11: XPS depth profiles.

The atomic profiles of 170607B and 170614A are what we expect to see, the aluminum and nitrogen level is significantly higher than the other compounds and they are approximately the same height. The silicon wafer has a small 2-4 nm layer of native oxide, this is evident as we see a oxygen peak between the deposited AlN layers and the wafer. 170608B is noticeably different. This is because the etching argon beam did not exceed the deposited AlN layers.

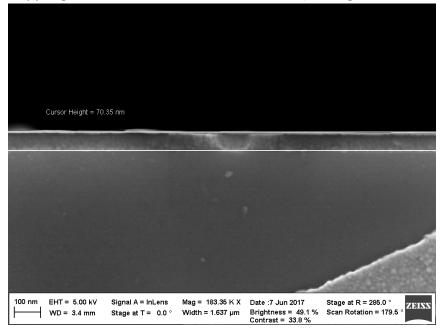
Sample 170608A was found outside the nitrogen cabinet for unknown reasons, which is likely why there is such an extensive amount of oxygen. Ellipsometer measurements showed that sample 170608A has an AlN layer of 21.69 nm. This thickness is quite thin, and can be oxidized more easily.

3.4 SEM Analysis

For some samples, we also collected images with the Zeiss Supra 40VP SEM, in order to investigate the structure and thickness of the AlN layers. Two SEM images of the cleaved 170606C sample are shown in Figure 12.



(a) Angled side view of the cleaved 170606C wafer, showing the surface.

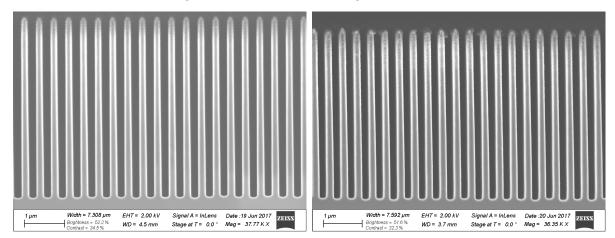


(b) Side view of the cleaved $\bf 170606C$ wafer.

Figure 12: SEM images of the cleaved 170606C wafer.

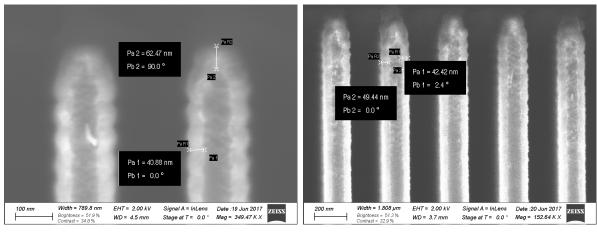
We see that the surface structure matches the AFM measurements. But, the surfaces seems to be slightly thicker when measured using the SEM (70.35 nm) vs. the ellipsometer (58.27 nm). Why this is, is not clear, but it could be due to a miscalibration of the SEM, or the ellipsometry model, or both.

The majority of the SEM analysis was focused on the two samples with 400 nm pitch trenches: 170616B and 170619B. The SEM images of these can be seen in Figure 13.

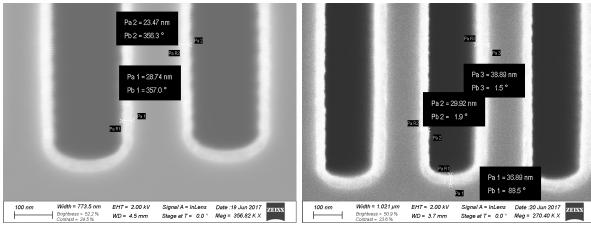


(a) Trenches in **170616B**.

(b) Trenches in 170619B.



- (c) Top of the trenches in **170616B**. Measured thickness of 62.47 nm on the top, 40.88 nm on the side.
- (d) Top of the trenches in $\bf 170619B$. Measured thickness of $42.42\,\mathrm{nm}$ on the side.



- (e) Bottom of the trenches in 170616B. Measured thickness of $28.74\,\mathrm{nm}$ on the side.
- (f) Bottom of the trenches in **170619B**. Measured thickness of 29.92 nm on the side.

Figure 13: SEM images of 170616B and 170619B, respectively

From Figure 13a & 13b we see that the AlN has generally been deposited homogeneously. But, when measuring closely, and comparing the thickness on the tops (Figure 13c & 13d) with the thickness in the bottoms (Figure 13e & 13f) there is still a difference in deposition thickness of around 30 nm. This could indicate that the TMA and/or NH₃-plasma hasn't been able to fully diffuse into the trenches, and thus that their pulse times (and/or purge time) should be increased when depositing onto high aspect ratio structures. Additionally, in Figure 13f a difference can even be seen when comparing the left and right side of the trench walls, though this could be due to a slight tilt of the sample holder in the SEM.

Accurately measuring the thickness of the tops using the SEM proved difficult, the measurements seemed to match the thicknesses measured with ellipsometry. But we also noted that using the SEM, the pitch of the trenches was measured to be around 10% lower than the expected value of $400\,\mathrm{nm}$, which seems to indicate further inaccuracies with these measurements beyond the visual difficulty of the determining where the top deposition starts and stops.

3.5 Wafer Images

During the production of the PEALD wafers, we noted that different recipes produced different colors on the wafers. It seems to mainly be due to differences in the thickness of the deposited AlN layers.

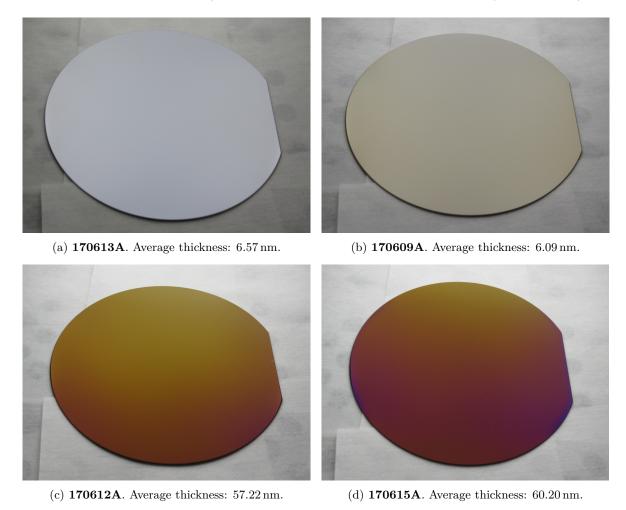


Figure 14: Pictures of a selection of produced wafers.

4 Conclusion

The temperature and the NH₃ plasma time have a significant effect on the uniformity of the deposited AlN layer. Increasing the temperature and the plasma time decreases the margin of error of the non-uniformity.

The temperature has an enormous influence on the growth rate and the margin of error is reduced at higher temperatures. The plasma time also seems to have an effect on the growth rate. The higher the plasma time the higher the growth rate.

The temperature has an effect on the amount of particles on the wafer after the deposition. This study shows the optimal temperature was $350\,^{\circ}$ C. The N₂ purge time after the NH₃ plasma flow had an effect on the number of particles, but the correlation is inconclusive. Out of the three plasma purge times applied in this study, $14\,\mathrm{s}$ had the worst results.

The temperature has an effect on the roughness of the samples. The higher the temperature the higher the roughness. A higher temperature also decreases the deviations of the roughness. An increase of the NH_3 plasma flow also seems to result in an increase in roughness. The deviation however is quite large.

This study is inconclusive due to the limited data set. The measurements with 200 °C and 450 °C are not within the previous mentioned ALD window, and thus these measurements are likely to be misleading when trying to optimize the PEALD recipe for deposition of AlN.

5 References

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