

GR-LVT - LMIS1

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CHARACTERIZATION AND OPTIMISATION  
OF SILICON NITRIDE THIN FILMS

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*Due date:*

08 January 2016



## Acknowledgments

This semester project has been conducted within GR-LVT group, part of LMIS1 laboratory at EPFL, Switzerland. This was a rich experiment for me to work in the cleanroom and to have a first approach with the equipment. I would like to thank Prof. Guillermo Villanueva and Tom Larsen for their supervision, Philippe Langlet for his helpful advices and Pierre Mettraux from MHMC laboratory for the stoichiometry analyzes.

Lausanne, January 2016

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## Abstract

Ten runs of silicon nitride LPCVD (Low Pressure Chemical Vapor Deposition) depositions were performed with a different recipe for each run. The goal of this work was to examine properties of silicon nitride and their variation with regards to the deposition parameters, which are : the ratio between dichlorosilane or DCS ( $\text{SiH}_2\text{Cl}_2$ ) and ammonia ( $\text{NH}_3$ ), the total gas flow, the pressure and the temperature. The ratio and the temperature are the two most considered parameters. The temperature was varied between  $776^\circ\text{C}$ ,  $800^\circ\text{C}$  or  $838^\circ\text{C}$ , pressure is 135 or 200 mTorr, the ratio of DCS: $\text{NH}_3$  is between 1:6 and 6:1 and total gas flow is 132 or 200 sccm (standard cubic centimeters per minute).

The affected properties we look at are: the stoichiometry of the silicon nitride layer, the internal stress, the roughness, the uniformity in thickness, the refractive index and the deposition rate.

By varying the four deposition parameters, we could obtain different set of properties. The obtained ranges are shortly exposed : Si/N ratio varied from 0.84 to 1.04, stress from 168 to 1127 MPa, roughness from 0.18 to 7.55 nm, refractive index from 1.99 to 2.14, uniformity in thickness varies from 0.39 to 1.81 % and deposition rate from 2.4 to 6.6 nm/min.

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# 1 Introduction

Silicon nitride is used for many applications, due to its remarkable optical and mechanical properties. It can for example be used as a final passivation and mechanical protective layer for ICs, as a mask layer for selective oxidation of silicon or as one of the dielectric materials in a stacked oxide-nitride-oxide layer in DRAM capacitors [5]. It is also commonly used for MEMS surface micro-machined components, such as nanopore arrays [7].

The key qualities of silicon nitride are [6] :

- Light weight.
- High strength and toughness (for a ceramic material).
- High chemical resistance to acids, bases, salts, and molten metals.
- Good resistance to oxidation up to 1500°C.
- High electrical resistivity.

Silicon nitride may be deposited by atmospheric-pressure chemical-vapor deposition (APCVD), low-pressure chemical vapor deposition (LPCVD) or plasma-enhanced chemical vapor deposition (PECVD). Dichlorosilane ( $\text{SiH}_2\text{Cl}_2$ , DCS) or silane ( $\text{SiH}_4$ ) are typically used as a source of silicon while ammonia ( $\text{NH}_3$ ) is typically utilized as the nitrogen source [5].

LPCVD is the technique used in this project to deposit thin films. The two gases used during the deposition are dichlorosilane and ammonia. The ratio between the two gases can be changed, which will affect the properties of the resulting layer. The deposition happens in a furnace at high temperature (around 800°C) which can result in a stress in the thin film. The total gas flow injected and the pressure in the furnace are also controlled.

The stress in the silicon nitride thin film is a very important parameter, as it can improve quality factors of nano-strings [9], [11], [12]. The long-term goal of this project is to get the right properties of high stress nitride to achieve high quality factor in NEMS resonators. It has indeed been seen that quality-factor is enhanced by increasing the stress in silicon nitride thin films [9]. Silicon nitride resonant micro-strings can for example be used for real-time particle mass spectrometry [8].

We are trying to understand the variation of silicon nitride's material properties depending on the deposition conditions. The different parameters that were manipulated during the deposition of silicon nitride are:

- Temperature
- Pressure
- Gas ratio  $\text{SiH}_2\text{Cl}_2/\text{NH}_3$
- Total gas flow



By varying one parameter at a time, we can determine its influence on several material properties of the nitride film of interest :

- Stoichiometry
- Stress
- Roughness
- Real part of refractive index and its uniformity
- Uniformity of the deposited layer
- Deposition rate

The Young modulus is also something we would like to measure, as the resonance frequency of a nano-beam is strongly dependent of it, but it has not been done so far.

After presenting the state of the art, it will be described how the measurements were performed. The results will then be discussed and some conclusions will be presented.

The next step of this project is to push the limits of the gas ratios, pressure and total gas flow to try to get highest and lowest stress, keeping a lower temperature to minimize the roughness.

## 2 State of the art

Many researches on the influence of deposition conditions on the material properties of silicon nitride thin films have been done in the past. For example, a study examined the stoichiometry, deposition rate, refractive index, etch rate and intrinsic strain [4]. However, as this study measured the strain and not to the stress, it was of interest to conduct this project for understanding the change in stress due to deposition parameters.

Some interesting statements can be found in an other study that showed that increasing gas flow too much may deteriorate deposition rate and thickness uniformity and that stress is inversely proportional to the refractive index [5].

It also showed there is a strong link between the silicon content and refractive index and proposed to use refractive index as a measure of the silicon content in silicon nitride thin films. It explained an interesting point about pressure, that increasing the partial pressure of reactants containing silicon reduces the stress. As the depositions parameters of this project are new, we want to make the same kind of properties study, but with new parameters.

This present work is based on what was done in another study [10]. The author has performed twelve different runs, using different gas ratios, pressure, temperatures and gas flows. Conclusions are given in this study about the dependence of different properties based on various parameters, which are shown in Table 1. The sign  $\nearrow/\searrow$  describes how the value evolves if the parameter in the first column (gas ratio, temperature, pressure, total gas flow) is increased.

	Stress	n	Deposition rate	Thickness variation	Roughness
T°C	$\searrow$	( $\nearrow$ )	$\nearrow$	$\nearrow$	$\nearrow$
Pressure	$\nearrow$	.	( $\nearrow$ )	$\nearrow$	/
Gas ratio	$\searrow$	$\nearrow$	$\nearrow/\searrow$		/
Total flow	( $\searrow$ )	.	$\nearrow$	/	

Table 1: Influence of the deposition parameters on the silicon nitride layer, according to [10]

With :

$\nearrow$  : Increases

$\searrow$  : Decreases

$\nearrow/\searrow$  : Changes in both direction

( ) : Slightly

/ : No influence

. : No conclusion

A blank : Not tested

The study of Tönnerberg [10] also gave a recipe to deposit silicon nitride at low stress and low roughness (T = 770°C, p = 150 mTorr, DCS:NH<sub>3</sub> ratio = 3:1, total flow = 200 sccm). The recipe has though not been tested by the author.

## 3 Experiment

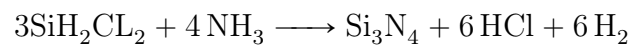
### 3.1 Deposition

The depositions were done at CMi (Centre de MicoNanotechnologie) at EPFL in a LPCVD furnace, shown in Figure 1.



Figure 1: LPCVD CMi furnace [1]

**Reaction** The reaction occurs as presented below [6]. It involves ammonia  $\text{NH}_3$ , which gives the  $N$  atom of silicon nitride and dichlorosilane  $\text{H}_2\text{SiCl}_2$ , which gives the  $Si$  atom.



The wafers used for these for deposition are single-side polished  $\langle 100 \rangle$  test wafers. Before the deposition, an RCA cleaning is done to remove oxides and other contaminants at the surface of the wafer. The thickness of the deposited layer was aimed at 200 nm to allow the laser of the stress measurement tool to work correctly.

### 3.2 Experimental Matrix

The ten runs that were performed during this study are shown in Table 2. The starting point are the two CMi recipes : Low Stress (LS) and High Stress (HS). Then the eight new recipes are labeled from Hmu1 to Hmu8.

Recipe	T [°C]	p [mTorr]	DCS:NH <sub>3</sub> ratio	DCS [sccm]	NH <sub>3</sub> [sccm]	Total gas flow [sccm]
LS	838	135	4:1	105	27	132
HS	776	200	1:6	30	180	210
Hmu1	838	200	1:6	30	180	210
Hmu2	776	135	4:1	105	27	132
Hmu3	776	200	1:1	105	105	210
Hmu4	838	135	1:1	66	66	132
Hmu5	776	200	4:1	168	42	210
Hmu6	776	200	5:1	175	35	210
Hmu7	776	200	6:1	180	30	210
Hmu8	800	135	4:1	105	27	132

Table 2: The experimental matrix

The low stress recipe (LS) deposition happens at high temperature, low pressure, high gas ratio and low total gas flow. For the high pressure recipe, it is the opposite. For the new recipes, we first want to see the effect of the gas ratio SiH<sub>2</sub>CL<sub>2</sub>/NH<sub>3</sub> which is varied between 1:6 and 6:1. To this end, the values of standard recipes for the pressure, temperature and total gas flow will be used. Hmu8 is done at 800°C to see the effect of the temperature.

As the total gas flow and the pressure are always changed together, their effects can not be independently analyzed. Furthermore, total gas flow and pressure are often varied together with temperature. Thus, it is difficult to see the effect of pressure and total gas flow. Still, as the change between Hmu2 and Hmu5 is only in pressure and total flow, we can try to see the effect of these two parameters on the properties of the material.

The partial gas flow limits were given by Philippe Langlet, responsible for LPCVD deposition at CMi. The ranges for the gas concentrations are the following : NH<sub>3</sub> gas flow between 27 and 1000 sccm and DCS gas flow between 30 and 200 sccm.

## 3.3 Equipment

### 3.3.1 Spectroscopic ellipsometer

Ellipsometry is a technique that uses the interaction of polarized light with surfaces to establish the refractive index (real and imaginary part) and the thickness of transparent thin films. Its thickness resolution is a few Å. The ellipsometry was conducted on the *Sopra* ellipsometer in CMi, shown in Figure 2.

The ellipsometer was firstly used to measure the thickness of the deposited layer which allows us to deduce the deposition rate. It is also essential to know the thickness of the layer to be able to measure the stress afterwards.

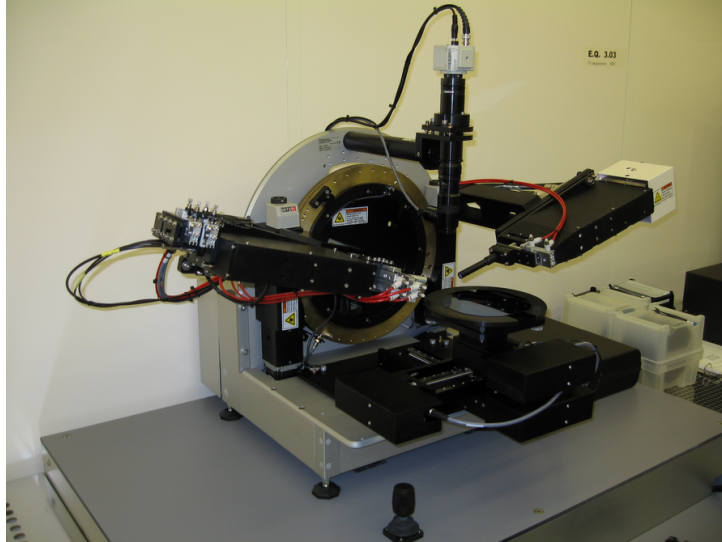


Figure 2: Sopra GES 5E, spectroscopic ellipsometer [1]

### 3.3.2 Thin film stress measurement tool

The stress in a thin film can result from a difference in thermal expansion, called thermal stress, or from the microstructure of the film, called intrinsic stress.

The stress can be either compressive or tensile as shown in Figure 3. Compressive stress is generally to avoid, because it may lead to buckling.

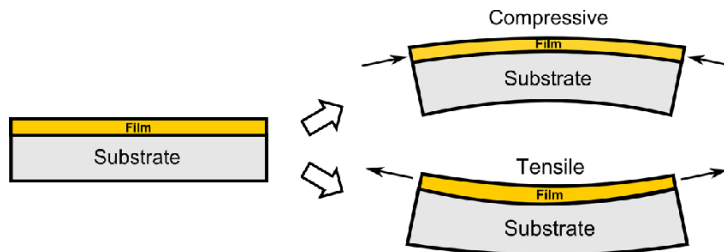


Figure 3: Compressive and tensile stress [2]

In our case, as the deposition occurs between  $776^{\circ}\text{C}$  and  $838^{\circ}\text{C}$  and the thermal dilatation coefficients of silicon and silicon nitride are not equal, we have a thermally induced stress. Nevertheless, this thermal component of the stress is a small, compressive stress, while the intrinsic component has a more significant impact on stress [5].

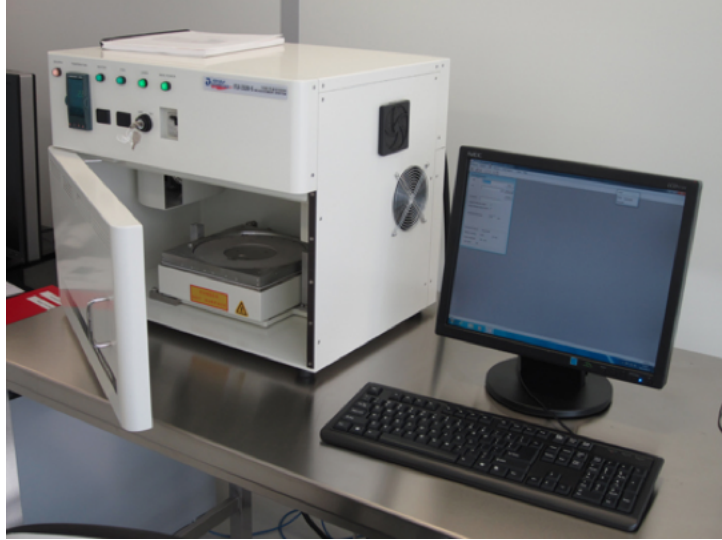


Figure 4: Toho Technology FLX 2320-S, thin film stress measurement tool [1]

To measure the stress, the following procedure was used:

1. Measure initial curvature of the wafer after silicon nitride deposition with equipment of Figure 4.
2. Etch backside of the wafer to remove silicon nitride, since it is deposited on both sides of the wafers.
3. Measure final curvature of the wafer and deduce the internal residual stress in the thin film with equipment of Figure 4.

### 3.3.3 Dielectric dry etcher

The dry etcher is used to remove the backside silicon nitride from the wafer. A  $\text{He}/\text{C}_4\text{F}_8$  gas combination is used with an etch rate varying between 57 and 158 nm/min depending on the deposited layer properties. A picture of the dry etcher, can be seen in Figure 5.



Figure 5: SPTS APS dielectric etcher [1]

## 4 Results and analysis

### 4.1 Measurements

The Table 3 presents the main measurements for the ten recipes exposed in Table 2. The details of all measurements can be found in Annexe A.

Recipe	Thick. unif. [%]	Si + N [%]	Ratio Si/N	RMS rough. [nm]	$\sigma$ [MPa]	n (780 nm)	n unif. [%]	dep. rate [nm/min.]
LS	0.42	99.1	0.92	7.55	168.8	2.11	0.14	4.6
HS	0.64	98.9	0.84	0.18	1127.4	1.99	0.23	2.7
Hmu1	0.39	97.4	0.84	0.18	979.4	1.99	0.32	6.6
Hmu2	1.77	98.5	0.93	0.27	460.3	2.10	0.06	2.4
Hmu3	0.97	98.3	0.98	0.19	946.4	2.02	0.48	4.4
Hmu4	1.81	98.0	0.86	0.18	767.0	2.04	0.29	6.5
Hmu5	1.25	99.30	0.97	0.29	580.3	2.09	0.1	3.5
Hmu6	1.31	99.22	1.00	0.27	472.6	2.12	0.06	3.3
Hmu7	1.33	98.84	1.04	0.34	397.7	2.14	0.11	3.1
Hmu8	1.49	99.56	0.94	0.64	390.7	2.11	0.28	3.1

Table 3: Measurements for the ten recipes

For the uniformity of the thickness and the refractive index, five points were measured: one in the center, and one at each extremity of the wafer, 10 mm from the edge. The thickness and the refractive index are the average of these five points, while their

uniformity is calculated on the basis of these five values. The refractive index is measured for  $\lambda = 780 \text{ nm}$ .

## 4.2 Stoichiometry

Stoichiometry is an important parameter to determine as we want to control the exact composition of the thin film. The theoretical ratio of  $\text{Si}_3\text{N}_4$  is of 0.75, but this ratio is often not obtained and therefore we want to know the specific ratio  $\text{Si}_x\text{N}_y$  for every recipe.

The concentrations of the different chemical compounds could be measured through an X-Ray photoelectron spectrometry (XPS) analysis in MHMC laboratory at EPFL .

Concerning the measurements, one wafer of each recipe was probed. For LS and Hmu2, the whole silicon nitride layer was analyzed, and the concentration of the different atoms was measured every 5 nm. For these two recipes, the average concentration Si+N [%] in Table 3 was calculated on the basis of all measurements done before reaching the oxide layer, which will be discussed in section 4.2.2. For the other recipes only the 30 first nm were analyzed, also by steps of 5 nm, which was enough to evaluate the average concentration of silicon and nitrogen.

### 4.2.1 Si/N ratio as a function of DCS/ $\text{NH}_3$ ratio

The Si/N ratio as a function of DCS/ $\text{NH}_3$  ratio is shown in Figure 6.

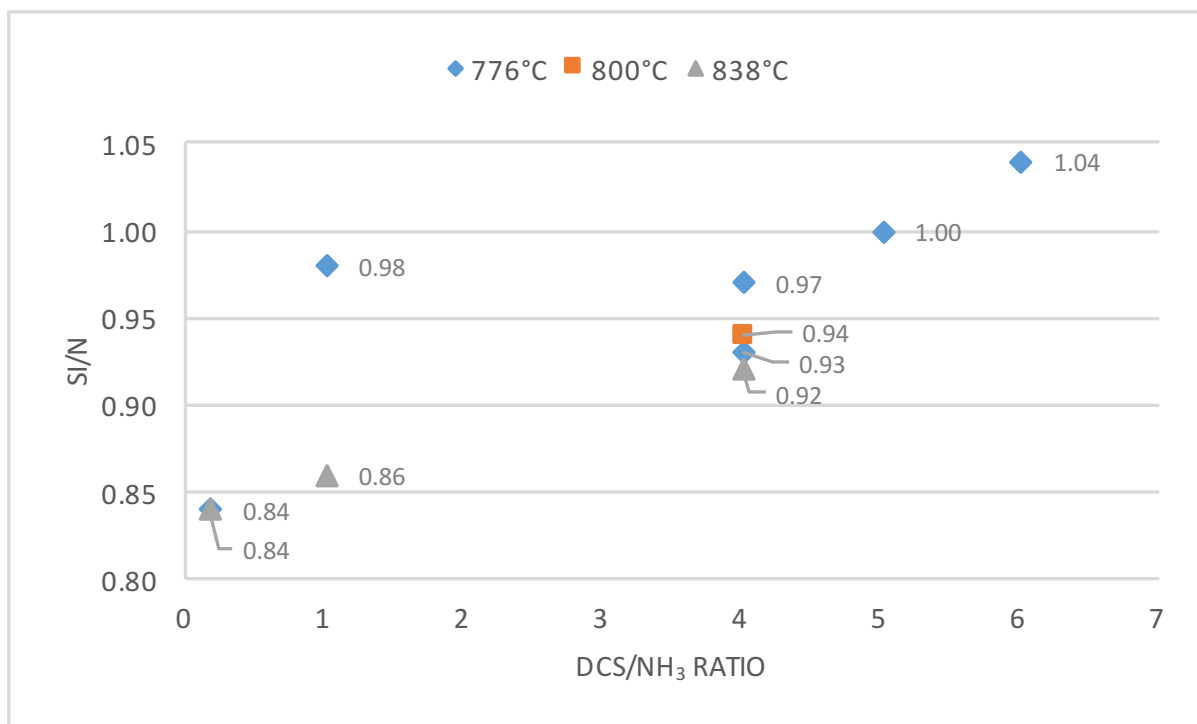


Figure 6: Si/N ratio as a function of DCS/ $\text{NH}_3$  ratio



The relationship looks quite linear, except for one point at 0.98. No explanation was found for this isolated deviation. The HS recipe is at 0.84 and the LS at 0.92, showing that none of the standard recipes respects the 0.75 theoretical ratio of silicon nitride  $\text{Si}_3\text{N}_4$ .

Keeping the depositions at  $776^\circ\text{C}$ , 200 mTorr, 210 sccm and excluding the point at 0.98, an interpolation is traced in Figure 7.

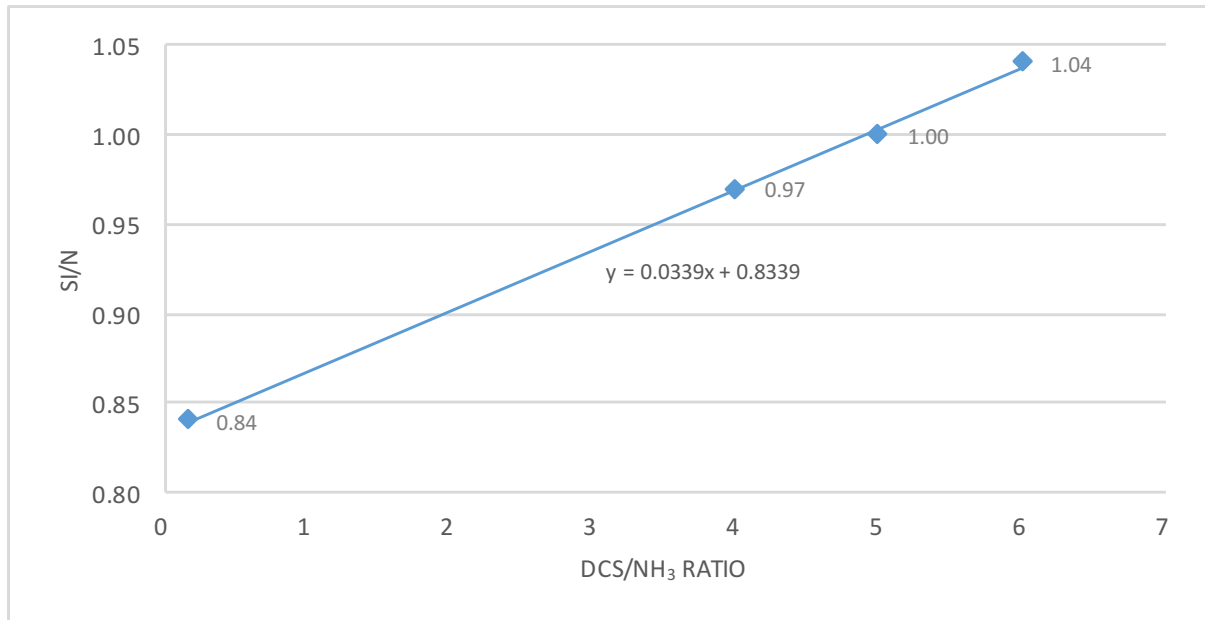


Figure 7: Interpolation for data at  $776^\circ\text{C}$ , 200 mTorr, 210 sccm

The calculated relative error for this interpolation is below 0.1%, which indicates that the ratio of Si/N is directly related to the DCS/NH<sub>3</sub> ratio, when all other parameters are kept constant. This is model however not very robust as no explanation was found for the point at 0.98 in Figure 6 and some new runs should be done to verify its reliability.

#### 4.2.2 In-depth analysis

An in-depth analysis of two wafers was performed: one of the low stress recipe of CMI and one of Hmu2. The results for these analysis are presented in Figure 8 and 9.

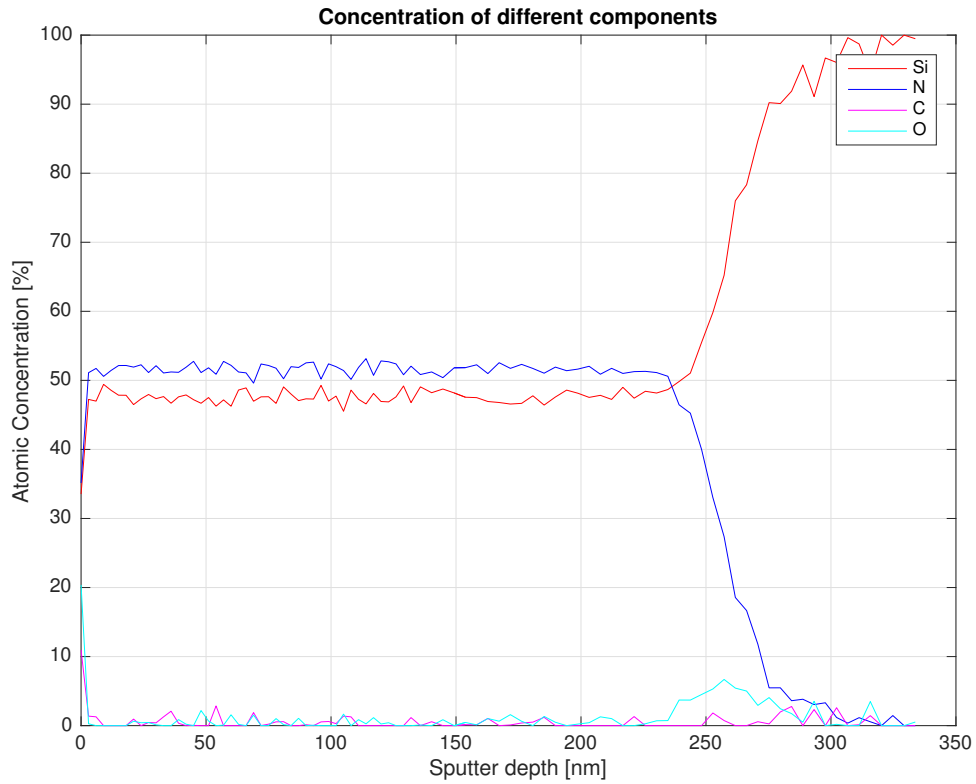


Figure 8: Concentration of the different chemical components for LS recipe



Figure 9: Concentration of the different chemical components for Hmu2 recipe

Fluorine was also detected as a contaminant during the analysis, but only as a surface contaminant on the first 20 nm with a concentration below 1%.

The concentration of contaminants (C, O) remains on the whole layer at a quite low level, but there is a concentration of oxygen close to 10 % at the interface between silicon substrate and the silicon nitride layer. A possible explanation is that the silicon nitride gets re-oxidized between RCA cleaning and LPCVD deposition. The width of these oxidized silicon layers are of 62 nm for the low stress recipe and of 35 nm for the Hmu2 recipe.

The XPS analysis gives us a view of the binding energy diagrams of the different atoms. We present here the result for Hmu2 binding diagram for silicon atom, shown in Figure 10.

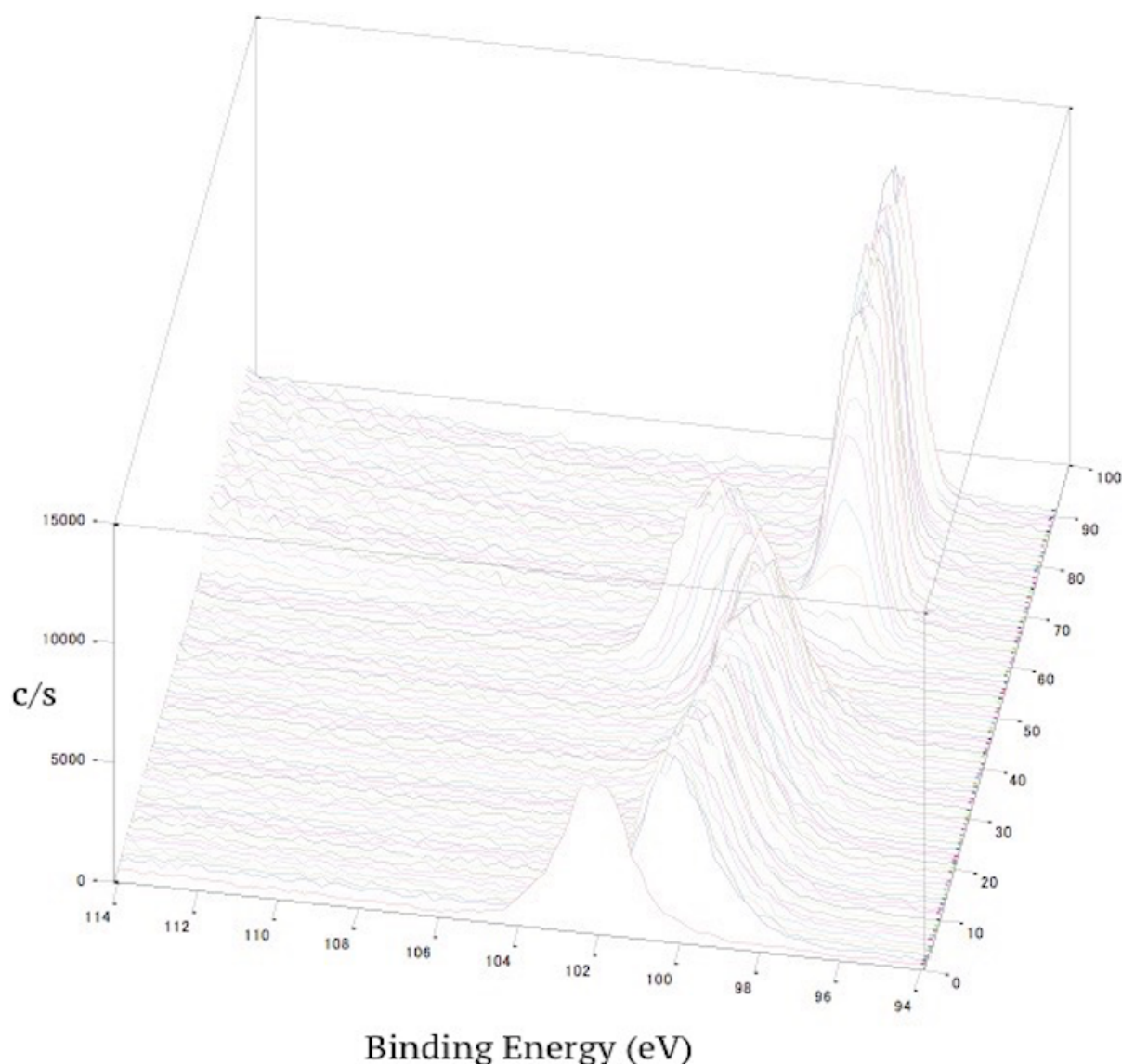


Figure 10: Binding energy diagram silicon atom, Hmu2 recipe

We clearly identify the first layer, corresponding to the native oxide of silicon, which

is around 2 nm. The curves remain then at an energy corresponding to silicon, but are then slowly shifted to the left, corresponding to oxidized silicon when getting closer to the interface with the substrate as discussed before.

### 4.3 Stress

For the stress measurements, two wafers were measured for each recipe and each wafer was measured at two angles. The stress corresponding to a recipe, as presented in Table 3, is then the average of these four values, except for the Hmu2 recipe for which only one wafer was measured and therefore only averaged two values.

The stress is plotted for different temperatures as a function of DCS/NH<sub>3</sub> ratio in Figure 11.

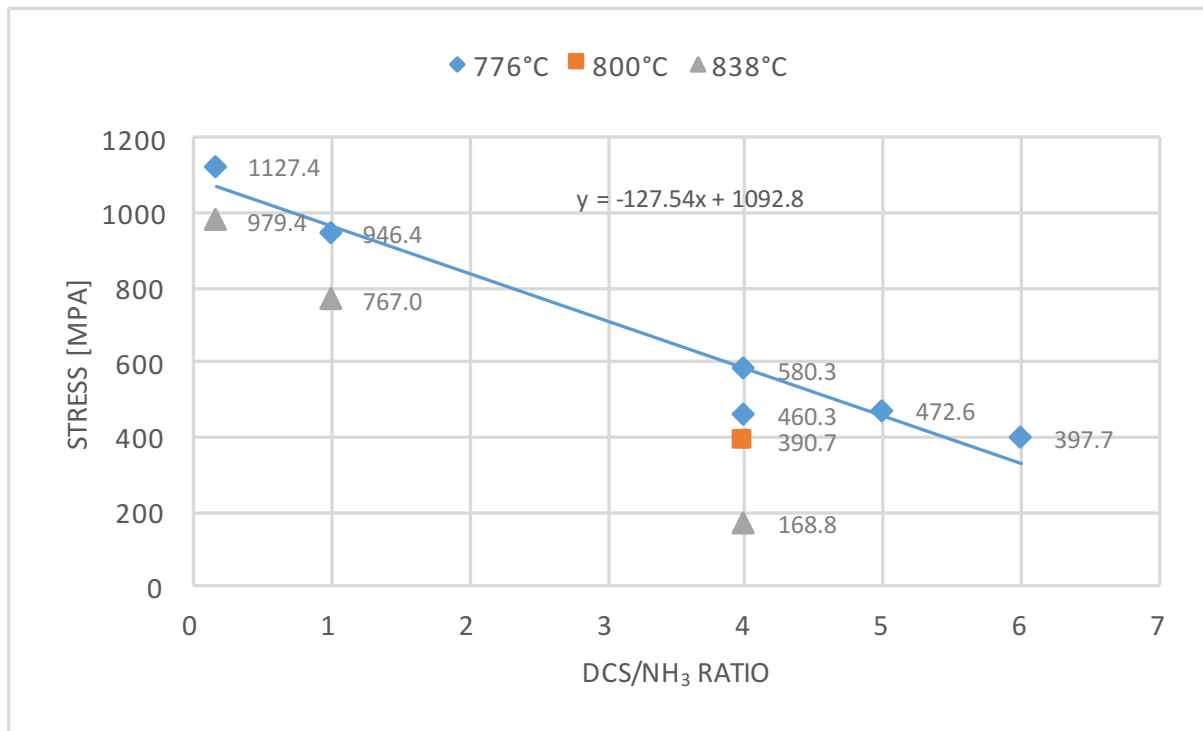


Figure 11: Stress as a function of DCS/NH<sub>3</sub> ratio for different temperatures and linear interpolation for data at 776°C

Firstly, as seen in section 2, the stress decreases if the film is richer in silicon or if the temperature is increased. The gas ratio has a greater impact on the stress as changing it from 6:1 to 1:6 changes the stress of 730 MPa, while keeping ratio constant and varying the temperature of 62°C changes the stress of only 411 MPa.

Secondly, we see that stress is increased from 460.3 MPa (Hmu2, 135 mTorr, 132 sccm) to 580.3 MPa (Hmu5, 200 mTorr, 210 sccm) when increasing pressure and total flow. We know from [10] that the stress increases with pressure, while it increases slightly with decreasing total flow. Thus, the increase in stress from Hmu2 to Hmu5 is mostly due to the increase in pressure.

A linear interpolation is traced in Figure 11 for data at 776°C. The quality of the fit is evaluated in Table 4.

Recipe	Stress [MPa]	Interpolation [MPa]	Absolute error [MPa]	Relative error [%]
HS	1127.4	1071.5	55.9	5
Hmu2	460.3	582.6	-122.4	27
Hmu3	946.4	965.3	-18.9	2
Hmu5	580.3	582.6	-2.3	0
Hmu6	472.6	455.1	17.5	4
Hmu7	397.7	327.6	70.1	18

Table 4: Quality of the fit for data at 776°C of Figure 11

The pressure and total flow change for Hmu2, which explains why it does not fit this model. The relative error increases when the stress is lowered, which indicates the model is not adapted for low stress, but it gives a first estimation of the stress. For example, for a 3:1 ratio, a tensile stress around 710 MPa can be expected. To get a more precise model, the Hmu2 point can be removed and an interpolation of degree 2 can be applied. Result is shown in Figure 12 and quality of the fit is calculated in Table 5.

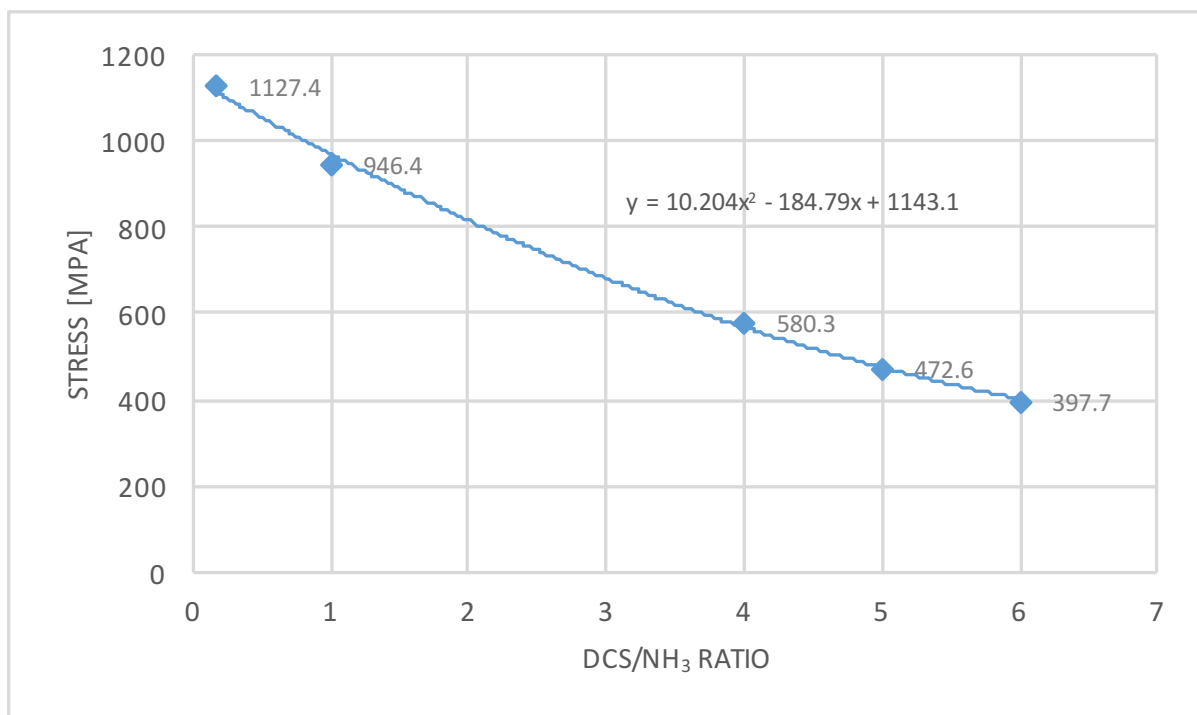


Figure 12: Stress as a function of DCS/NH<sub>3</sub> ratio for different temperatures and interpolation of second order for recipes at 776°C, 200 mTorr and 210 sccm

Recipe	Stress [MPa]	Interpolation [MPa]	Absolute error [MPa]	Relative error [%]
HS	1127.4	1112.6	14.8	1
Hmu3	946.4	968.5	-22.1	2
Hmu5	580.3	567.2	13.1	2
Hmu6	472.6	474.3	-1.6	0
Hmu7	397.7	401.7	-4.1	1

Table 5: Quality of the fit of Figure 12

We see the relative error is now kept under 2%, and a stress of round 680 MPa can be expected for a 3:1 ratio with this more accurate fit.

#### 4.4 Roughness

For the roughness, one wafer per recipe was tested with an AFM (Atomic Force Microscope) tool over a surface of  $100 \mu m^2$  was probed and the root-mean-squared value was deduced. For most applications, a low roughness is required [3].

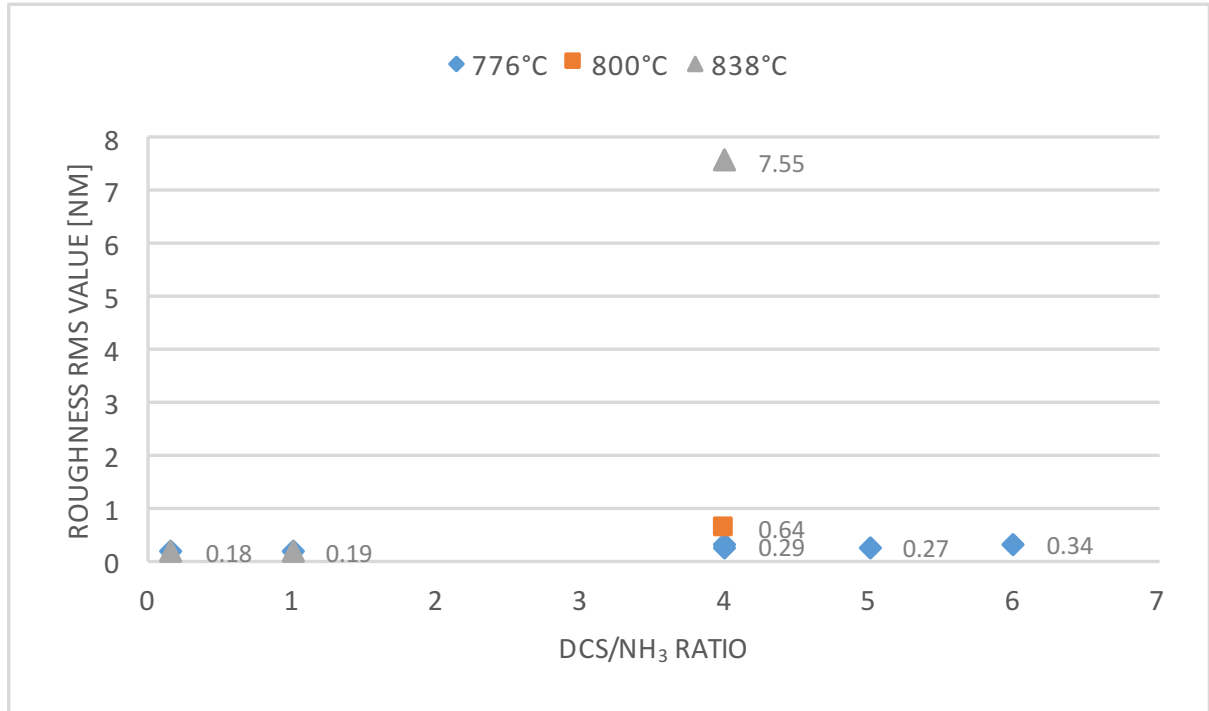


Figure 13: Roughness as a function of DCS/NH<sub>3</sub> ratio for different temperatures

In Figure 13, the standard low stress at CMi is at 7.55 nm is eleven times higher than the point at 0.64 nm for 800°C. Since a lower temperature gives a lower roughness, an idea to achieve a low stress recipe with lower stress could be to work at low temperature to minimize the roughness, and to increase the gas ratio in aim to keep a low stress.

## 4.5 Refractive index

The refractive index is an important parameter for optical applications [13]. We want to keep  $n_{Si_xN_y}$  around the value of  $n_{Si_3N_4} = 2.01$ . The value of the refractive index for  $\lambda = 780 \text{ nm}$  is plotted in Figure 14 for the different recipes. Between two wafers of the same recipe, the measure is very stable : only the second digit changes sometimes, see Annexe A.

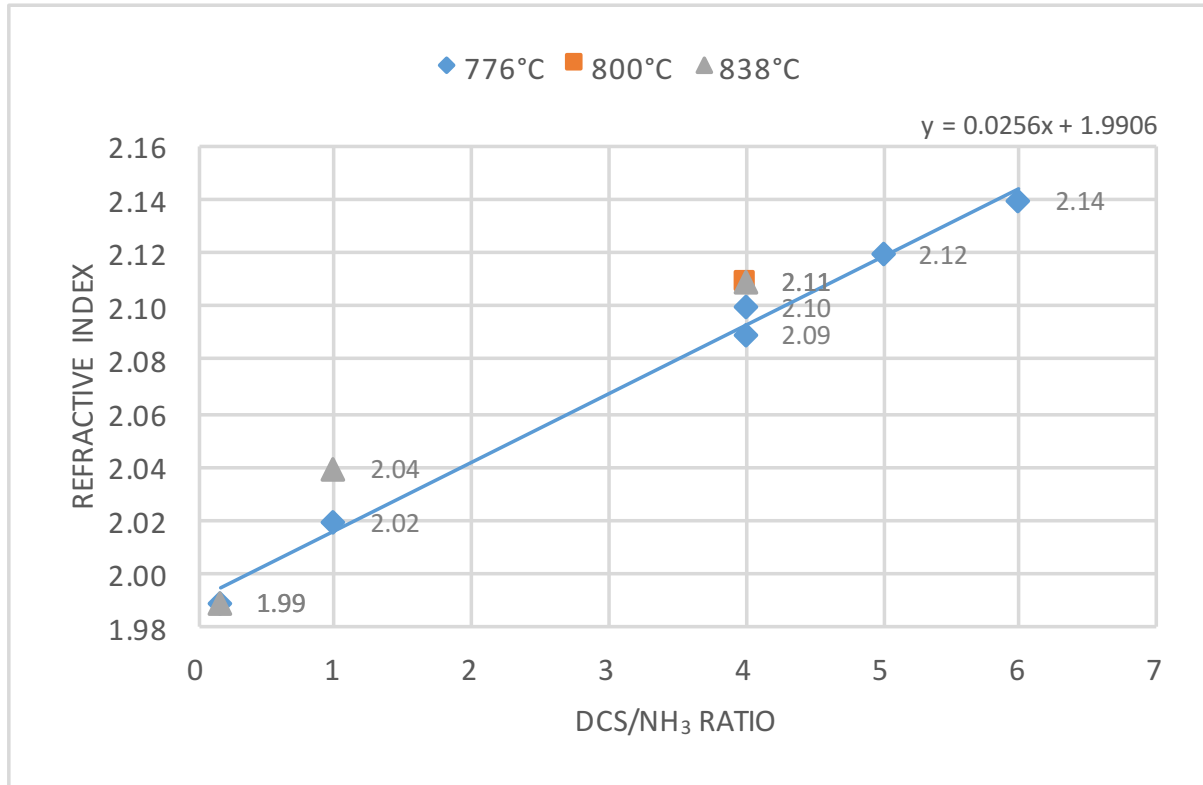


Figure 14: Refractive index at 780 nm as a function of DCS/NH<sub>3</sub> ratio for different temperatures

A linear interpolation is traced in blue for data at 776 °C and the relative error is kept under 3 % for obtained data. The refractive index tends to get closer to  $n_{Si} = 3.44$  when increasing silicon concentration, as described in [4]. It can be explained by the fact that excess silicon atoms are present in the lattice at substitutional nitrogen sites [5]. It seems that increasing temperature leads to a slightly higher refractive index. A change in pressure or total flow does not seem to have an important influence on it as we can see looking at points at 2.10 (Hmu2, 135 mTorr, 132 sccm) and 2.9 (Hmu5, 200 mTorr, 210 sccm), where temperature and gas ratio are kept constant.

The uniformity of the refractive index was determined from five points. It remains below 0.5 % as visible in Annexe A.

## 4.6 Thickness uniformity

The layer should be as uniform as possible, so that structure patterns made by micro-fabrication are stable over the layer. The variation in thickness is very well controlled, as shown in Figure 15. It remains below 2% for the ten recipes. Despite this good quality of deposition, it is hard to find any clear dependence on any parameter.

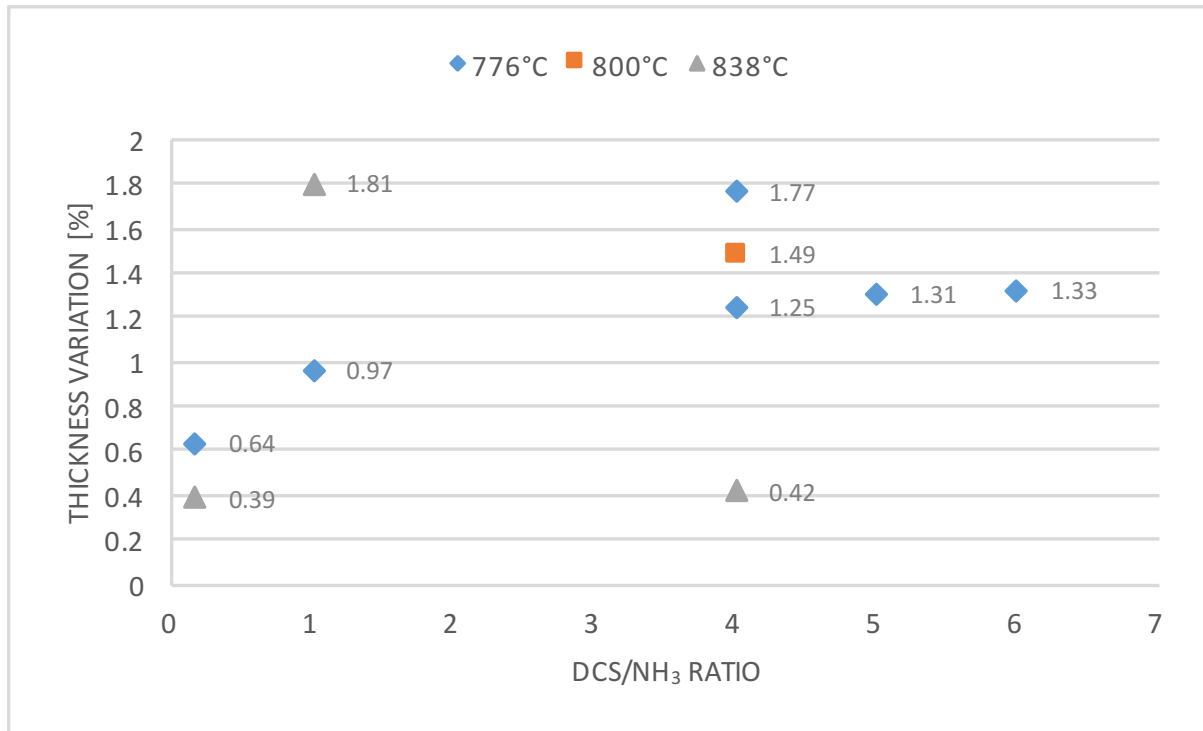


Figure 15: Variation in thickness as a function of DCS/NH<sub>3</sub> ratio for different temperatures

## 4.7 Deposition rate

The deposition rate should be as high as possible, so that deposition is shorter and cheaper. The deposition rate as a function of DCS/NH<sub>3</sub> ratio is plotted in Figure 16.



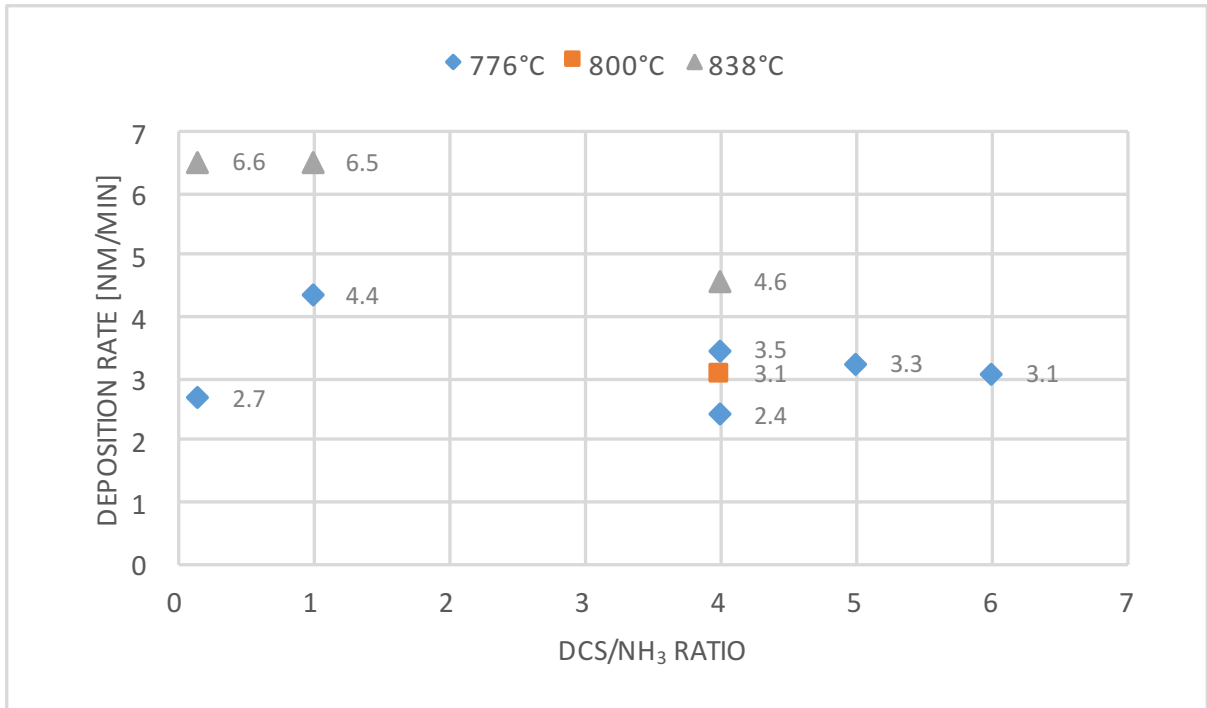


Figure 16: Deposition rate as a function of DCS/NH<sub>3</sub> ratio

We see that deposition rate is higher at higher temperature. Both pressure and total flow are changed between the points at 2.4 (Hmu2, 135 mTorr, 132 sccm) and 3.5 (Hmu5, 200 mTorr, 210 sccm). Based on this result and on [10], we can deduce that a higher flow rate and a higher pressure gives a higher deposition rate.

## 5 Discussion

### 5.1 Stress as a function of the refractive index

The relationship between refractive index and stress is plotted in Figure 17 and quality of the fit exposed in Table 6.

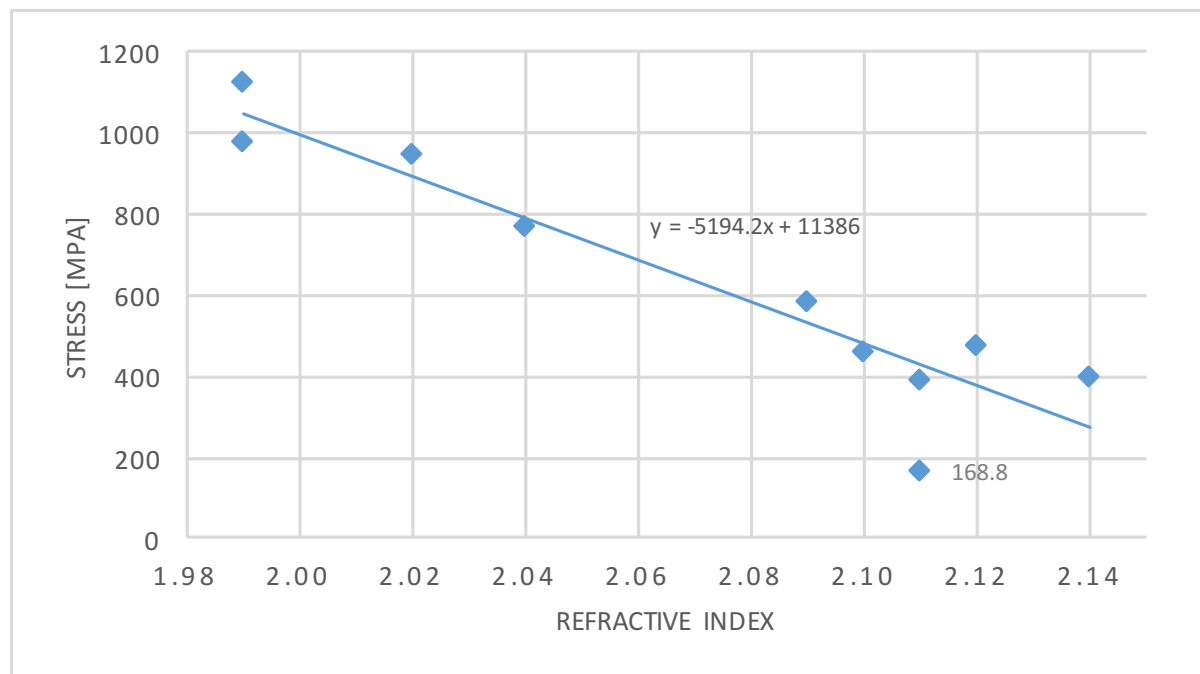


Figure 17: Stress as a function of the refractive index

Recipe	Stress [MPa]	Interpolation	Absolute error [MPa]	Relative error [%]
LS	168.8	426.2	-257.4	153
HS	1127.4	1049.5	77.9	7
Hmu1	979.4	1049.5	-70.1	7
Hmu2	460.3	478.2	-17.9	4
Hmu3	946.4	893.7	52.7	6
Hmu4	767.0	789.8	-22.9	3
Hmu5	580.3	530.1	50.2	9
Hmu6	472.6	374.3	98.3	21
Hmu7	397.7	270.4	127.2	32
Hmu8	390.7	426.2	-35.6	9

Table 6: Quality of the fit of Figure 17

The stress is inversely proportional to the refractive index. However a linear interpolation does not allow us to establish a reliable model, as we see the relative error is quite high.

From Figure 17, we keep points at  $T = 776$  °C, 200 mTorr, 210 sccm. The result is presented in Figure 18 and the quality of the fit in Table 7.

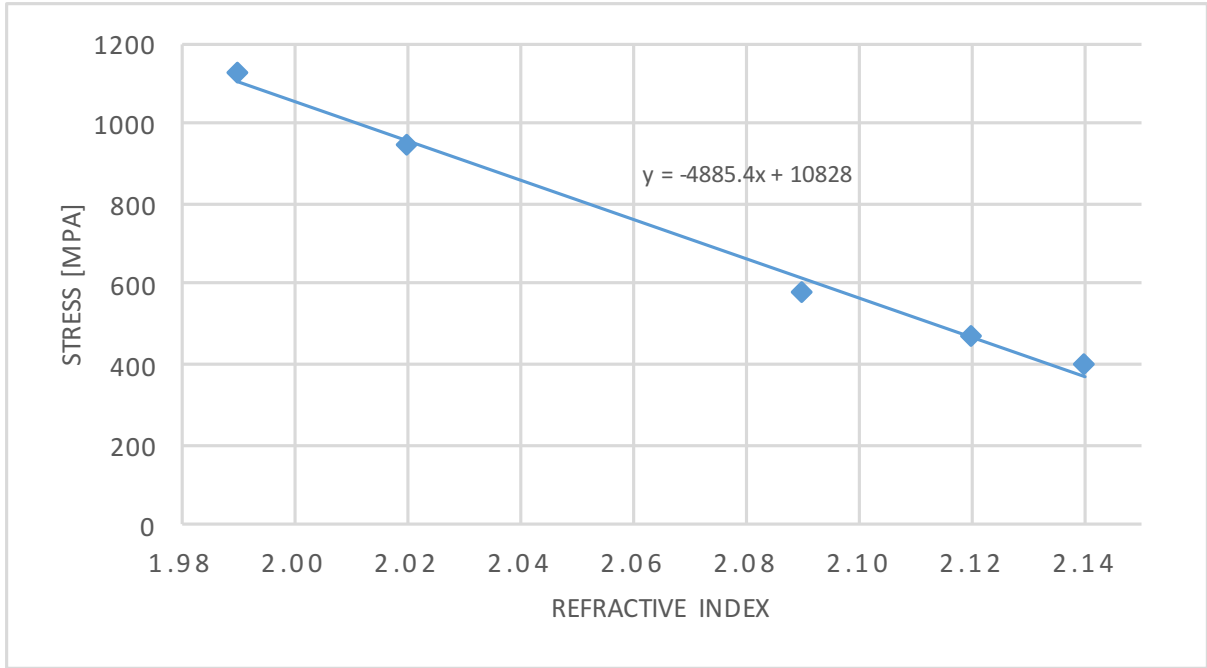


Figure 18: Stress as a function of the refractive index for data at  $T = 776\text{ }^{\circ}\text{C}$ , 200 mTorr, 210 sccm

Recipe	Stress [MPa]	Interpolation [MPa]	Absolute error [MPa]	Relative error [%]
HS	1127.4	1100.1	27.3	2
Hmu3	946.4	949.2	-2.8	0
Hmu5	580.3	597.0	-16.7	3
Hmu6	472.6	446.1	26.5	6
Hmu7	397.7	345.5	52.1	13

Table 7: Quality of the fit based on interpolation of data at  $776^{\circ}\text{C}$

We see the fit is improved if one parameter is varied while keeping the three other parameters constant. Here the relative error is kept lower, although the model is not reliable at lower stress.

## 5.2 Further depositions

The next step of this project is to use the knowledge of previous work and to test new depositions, in order to get the highest and lowest possible stress. We show in Table 8 the recipes that are planned for future depositions.

Recipe	T [°C]	p [mTorr]	DCS:NH <sub>3</sub> ratio	DCS [sccm]	NH <sub>3</sub> [sccm]	Total gas flow [sccm]
Hmu9	776	189	5:1	135	27	162
Hmu10	776	150	6:1	162	27	189
Hmu11	776	439	1:6	30	180	210
Hmu12	776	488	1:6	35	210	245
Hmu13	776	542	1:6	40	240	280

Table 8: Proposition of further depositions

The goal of Hmu9 and Hmu10 is to test low stress limit in the limit fixed by the equipment, while Hmu11, Hmu12 and Hmu13 are aimed to test high stress limit. A temperature of 776°C is chosen for the low stress recipes, although we know it might increase the stress, in order to keep a low roughness.

Some parameters of Hmu9 recipe could already be measured. A comparison with LS recipe is proposed in Table 9.

Recipe	T [°C]	p [mTorr]	DCS:NH <sub>3</sub> ratio	DCS [sccm]	NH <sub>3</sub> [sccm]	Total flow [sccm]	Stress [MPa]	n (780 nm)	Unif. of n [%]
LS	838	135	4:1	105	27	132	168.8	2.11	0.14
Hmu9	776	189	5:1	135	27	162	230.4	2.11	0.42

Table 9: Comparison between CMi low stress recipe and Hmu9 recipe

We see that Hmu9 does not allow us to reach the stress achieved by the LS recipe. In order to reach this value, we could try to increase slightly the value of the temperature or try to increase the ratio as proposed for recipe Hmu10.

## 6 Conclusion

The different depositions enabled to examine properties of silicon nitride as a function of the four deposition parameters. Linear or quadratic interpolations could be traced for most properties depending on the gas ratio. These models are reliable only if one parameter is changed at a time, showing the importance of each of the four deposition parameters.

Further depositions would allow determining the low and high stress limit achievable with CMI equipment. These recipes would hopefully lead to very high and very low stress, but the other parameters such as roughness or thickness uniformity might be deteriorated. By going at very low stress, we would also need to pay attention not to go in the compressive stress domain.

By doing more deposition, we could try to obtain a robust model where specific recipes could be obtained depending on the application: for example if a very specific refractive index is required or a very low roughness. Creating a complete model would be very long to achieve, but more specific models could be achieved, as said, by fixing three parameters and varying the last one.

An example of a particular recipe could be done in aim to obtain high quality factor resonators. Once a good recipe would be found, a study of reproducibility and between several depositions should then be made in order to have a reliable recipe.

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## Annexe A : Measurements

Recipe	Wafer ID.	Deposition rate [nm/min]	Var. in thickness [%]	Si + N [%]	Ratio Si/N	n at 780 nm	Var. of n [%]	RMS roughness [nm]	Stress [MPa] 0°	Stress [MPa] 90°	Mean Stress [MPa]
Low Stress Cmi	55569	4.6	0.33	99.13	0.92	2.11	0.09	7.55	166	165.9	168.8
	55567		0.51						167	176.3	
High Stress Cmi	55573	2.7	0.50	96.86	0.84	1.99	0.20	0.18	1125	1135	1127.4
	55571		0.79						1128.8	1120.7	
Hmu1	53979	6.6	0.31	97.42	0.84	1.99	0.29	0.18	972.6	974.6	979.4
	53978		0.48						984.7	985.7	
Hmu2	53973	2.4	1.77	98.46	0.93	2.10	0.079	0.27	452.4	468.1	460.3
	53971		1.78								
Hmu3	54046	4.4	0.89	98.3	0.98	2.02	0.48		944.3	945	946.4
	54019		1.05						947.4	948.9	
Hmu4	54015	6.5	1.61	97.97		2.04	0.35	0.18	769.3	769.6	767.0
	54013		2.01						767	761.9	
Hmu5	53983	3.5	1.33	99.3	0.97	2.09	5.06E-02	0.29	582.3	582.8	580.3
	53984		1.17						580.6	575.5	
Hmu6	55360	3.3	1.21	99.22	1.00	2.12	8.80E-02	0.27	445.4	490.5	472.6
	55362		1.41						474.3	480.2	
Hmu7	55356	3.1	1.29	98.84	1.04	2.14	0.11	0.34	393.3	395.1	397.7
	55357		1.38						398.8	403.4	
Hmu8	55351	3.1	1.47	99.56	0.94	2.11	0.45	0.64	386.8	388.8	390.7
	55352		1.51						371.2	415.8	