ÉCOLE POLYTECHNIQUE
FÉDÉRALE DE LAUSANNE

CMi EPFL Center of MicroNanoTechnology

Lab Report: Cleanroom practical work

with individual work on *High Resolution Photoresist* by Aladin Guillaume

Professeur : Abdeljalil Sayah

Assistant : Atakan Baris

High resolution resist processing, mask alignment exercises and aluminum wet-etching

Group 7

Ahaggach Yassine

Aladin Guillaume

Georganopoulos Alexis

Hémon Christopher

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Contents

1 Introduction

1.1 Cleanrooms

The micro-structures built for the most recent technological applications tend to become smaller and smaller. Some of them have reached the size of a micrometer. As a result, such structures contain elements which are smaller than some of the particles that can be found in the ambient atmosphere. Therefore, such component need to be built in controlled atmosphere environment such as cleanrooms.

A cleanroom is a closed place where the atmosphere and the water used are very well controlled, in order the suppress any kind of contaminant, regarding the fabrications of micro-structures. Such room are classified by a number which correspond to the maximum amount of particle under a certain diameter that can be found in a one square-foot sample of the air.

This report synthesize two sessions of practical exercises made in such environment, which complete the course *Technologie des microstructures*"[1]. During the first session, the realization of an alignment mask will be created using High-Photoresist processes. The second session present the realization of an anisotropic wet-etching of an aluminum layer. All this maneuvers are being performed on a 10'00 class cleanroom. The next chapter present the equipement that need to be worn to enter such an environment. (Note that the 1000 class cleanroom required a slight different suit which is not presented here).

1.2 Equipment

Despite of the fact that air injected into cleanrooms is filtered, a large amount of contaminant arise from humans working inside. As a consequence, every worker need the wear a specific suit. This equipment prevent particle to emanate from researcher, but also protect them from the product used during the processes.

The following steps presents the equipment required, and the critical instructions need to get dressed:

- At first, overshoes need to be placed, taking care walking on a specific sticky carpet when placing each foot back on the floor.
- A one piece over-suit is then worn, taking care not to let the sleeves touch the floor. (As they are going to get close to the maneuverer inside the cleanroom)
- A second type of overshoes is then placed over the first ones.
- A mobcap is then worn.
- The researcher then need to wear a surgical mask. (Considering that the elastic need to face the figure and the metallic part need to be bend correctly, in order to prevent steam to form on the protective glasses)
- Gloves need to be chosen between 3 sizes: S,M,L, considering that they need to be tighten, considering other plastic gloves will be placed over during the procedure of the exercises inside the cleanroom.
- The subject finally wear some protective glasses if he don't usually where eyeglasses, or if those are too small to protect the eyes.

Figure 1: Illustration of a students, wearing the CMI 10'000 class cleanroom suit[14]

A video[2] presenting the steps to put on the 1000 class cleanroom can be found in the MOOC[3] completing the course *Technologie des microstructures*[1].

2 Session 1 : High Photo-Resist Processing & Alignment exercises

2.1 Objectives

Photolithography is a process through which one can transpose a mask's geometric pattern to a wafer. It is mainly used for the micromanufacturing of integrated semi-conductor circuits. The core component behind this process is called a photoresist, it is a light-sensitive material that can either be positive or negative.
After exposition to UV-light a positive photoresist is weakened while a negative one is strengthened.Therefore, a positive photoresist after development will see it's exposed part dissolve and disappear since it has been previously weakened , a negative photoresist will do the opposite after development the exposed part remains although the rest (non-exposed part) dissolves. In order to achieve a better quality result, the photoresist used must exhibit certain characteristics : a high contrast, good sensitivity to UV-light and a high resistance to chemical etching.
The goal of the first session is to practise photolithography in cleanroom conditions (in respect to the strict apparel and cleanroom protocol), master the process flow and be able to characterize the end result (layer height, contrast and sensitivity of the photoresist). After identifying and understanding the photoresist principal characteristics, the second phase of the session is to efficiently (since the characteristics of the photoresist are known) align, expose and develop a second layer by respect to the first one as well as verifying at the end the error at alignment.
As this constitutes our first experience in a cleanroom, the importance of the protocol and apparel cannot be stressed enough. The tutor's and the professor's instructions are key to the success of the whole process.

2.2 Machines & Equipment

We were introduced to a complete set of tools for the photolitography, each has a specific role along the process flow and specific parameters that need to be set in order to achieve the desired result.

- Substrate : The wafers constitute the base of photolithography as they are the substrate upon which the resist is deposited, exposed and developed. They can either be made of pure Si or Si/SiO2, two of

the first type were used for the photoresist characteristics study while two of the latter were used for the alignment exercises. The wafers' doping, orientation and ID number can be geometrically identified. For this session, the wafers used were P type, had a diameter of 100 $\pm$ 0.5 [mm], a height of 525 $\pm$ 25 [μm].

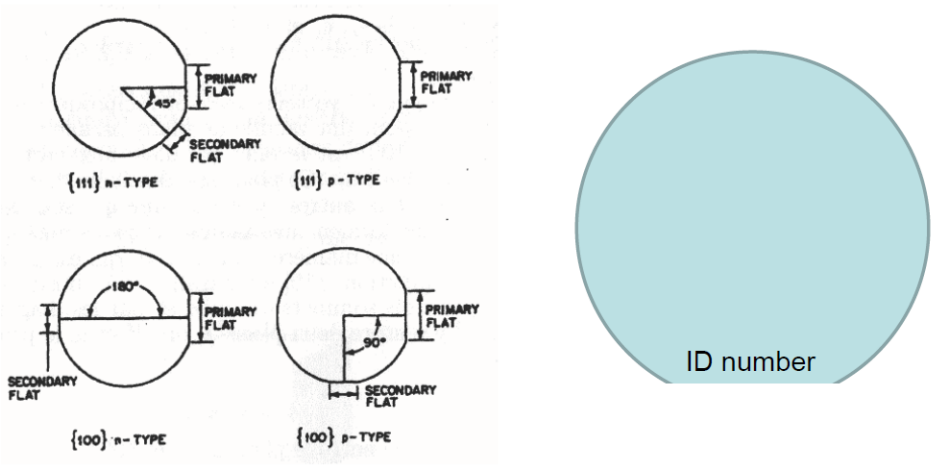


Figure 2: Wafer's doping, orientation and ID number[6]

- Photomask : In order to selectively expose our photo-resist and be able to reproduce desired patterns, photomasks were used. The photomasks are made of quartz on top of which exists a layer of metal, in our case chrome depicting the desired patterns. Through the photomask (through its patterns), UV light will shine on the photoresist resulting in the patterns being replicated on our substrate. We used a positioning photomask with different patterns for the alignment exercice and used a contrast photomask with different numbered circles in order to study our photo resist contrast (a different dose being shined through a different circle each time whilst covering all other circles).

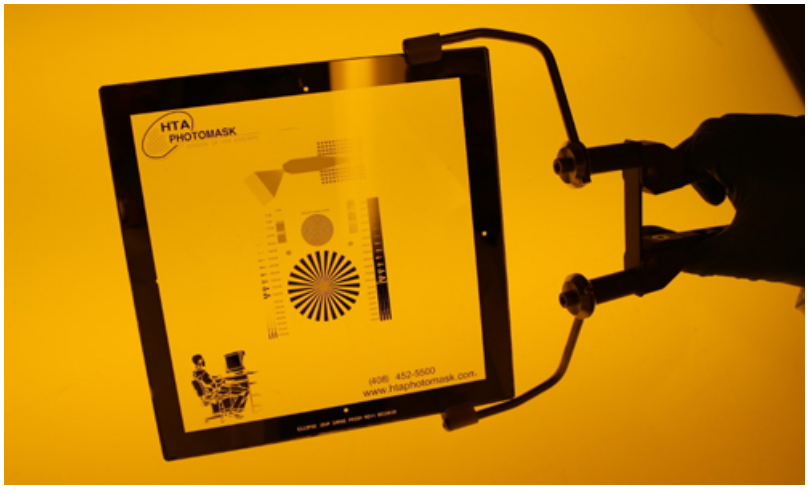


Figure 3: Photomask with different patterns[8]

- Resist : The resist used for this session is the AZ1512HS, it is a positive photosensitive resist. It also

boasts great adherence and coating capacity.The resist is made up of three key components :

- Inactive polymer : base of the novolac resist (10-40%)

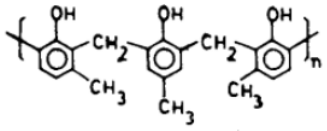


Figure 4: Novolac[6]

- Photo Active Compound (PAC) (1-5%), we used the diazonaphthoquinone (DNQ). The PAC modifies the solubility of the Novolac polymer.

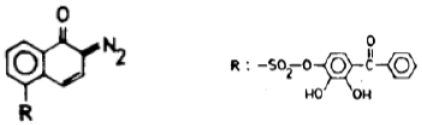


Figure 5: DNQ[6]

- Solvant (50-85%) controls the viscosity of the resist. Here, Propylene glycol methyl ether acetate is used.

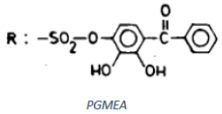


Figure 6: PGMEA[6]

- Surface treatment : In order to make the substrate surface hydrophobic so that we can easily deposit and spread resin on it we used HMDS (Hexamethyldisilazane).

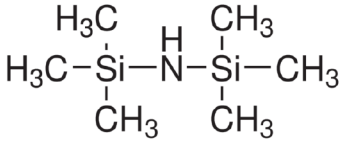


Figure 7: HMDS formula[6]

- Developer : After exposing the resist, it is developed using AZ351B. It is an alkaline aqueous solution designed to achieve the utmost contrast and wall profile.

- Solvant : We used deionized water to dilute the developer.

- Optibot VB20 : The wafer by default does not exhibit great adherence. This machine allows us to clean the surface of the substrate and coat it with HMDS making it hydrophobic and thus maximizing adherence and uniformity of the resist. It operates under vacuum and at a controlled temperature.

Figure 8: SSE VB20[4]

- Optispin SB20 : This machine allows us to spin-coat the substrate with our photo-resist. Different RPMs result in different layer thickness'.

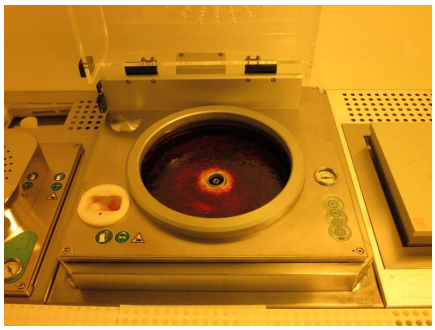


Figure 9: SSE SB20[4]

- LHG 2860 SR : We can soft-bake our wafer in order to eliminate the resist solvant and relieve the internal constraints created during the spin-coating of the resist.

- SUSS MJB4 : This machine allows us to expose our wafer after aligning it with the photo-mask.

Figure 10: SUSS MJB4[4]

- Optiphot 200 : It is an optical microscope with different lenses for different zoom magnitudes. We used it to observe the end result geometrical patterns on our wafer.

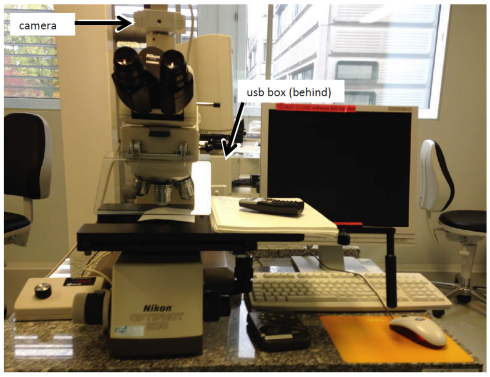


Figure 11: Nikon Optiphot 200[5]

- F20 UV : This machine has the ability to measure the thickness of thin layers using a non destructive optical method based on the reflectance of the film and the wavelength of the light emitted.

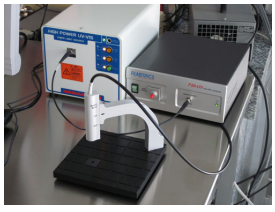


Figure 12: Filmetrics F20 UV[5]

2.3 Process flow

In this section we will discuss the whole process of photolithography step by step as well as the theory behind it. The photolithography was done in cleanroom conditions at a humidity level of 43.2% and a temperature of 21.4°C.

The steps presented here correspond to the steps of the process flow sheet received at the beginning of the session. The previous steps were prepared by the assistant.

Step 2.1:Wafer's surface cleaning and Silanization
The first step of the Photolithography process flow ensures the surface of the substrate is clean and has good adherence. By default, the native oxide of silicon makes the surface hydrophilic and absorbs thus humidity. To solve the problem we need to dehydrate the substrate and treat its surface. We use the SSE VB20 where we deposit the wafer and close the lid on it. A vacuum is created inside, the temperature is regulated at 135°C to cause dehydration and afterwards HMDS is released in the form of gas inside the chamber. What happens next is that the HMDS will be deposited the surface transforming it from hydrophilic to hydrophobic. Setting up the machine was pretty easy, as we only had to choose the HMDS standard in the menu and let it do the job.

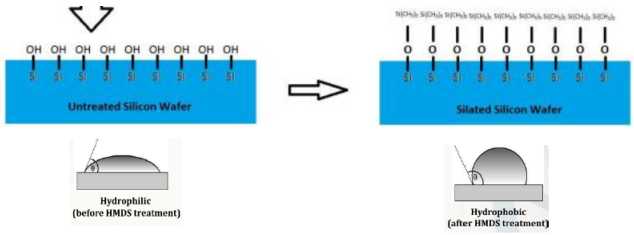


Figure 13: Wafer before and after silanization with HMDS[6]

Step 2.1 *bis* : Cooling down the wafer
This step does not officially appear on the process flow but is only common sense. After the HMDS priming the wafer is still more or less hot. If we happened to apply the resist on it directly, its solvant would start to evaporate and we would not be able to control its viscosity.

Step 2.2: Positive photoresist spin coating
The next step consists of applying the positive photoresist (AZ1512HS) uniformly and homogeneously on the wafer. The technique used is spin coating, it relies on centrifugal force to spread the resist on the surface. To do that we used the SSE SB20 and set it up using a program our assistant prepared for us. The main setting was the RPM program which can be divided in three significant steps, its first phase was a constant 500 RPM for 10 sec through a 100 RPM/s slope, the second phase was set at 3000 RPM for 45 sec through a 1000 RPM/s slope and finally the last step was set at 0 RPM for 3 sec through a -1000 RPM/s slope. All that in order to achieve the 1600 [nm] desired thickness. The machine would hold the wafer in place using vacuum suction and we would deposit the photoresist right in the middle using an eyedropper while carefully avoiding the formation of bubbles in the process. We would then close the lid and start the machine and wait for the program to finish. We did notice some small defects at the end, comets and particles mainly but nothing that significant according to our assistant.

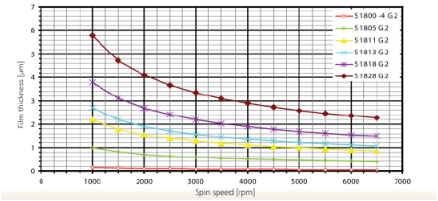


Figure 14: Film thickness in regard to spin speed[6]

Step 2.3: Softbake

After the photoresist has been spread uniformly across the wafer's surface a soft bake is needed. Mainly, the soft bake : allows us to eliminate the photo resist's solvants (they are no longer needed), helps make the adherence of the photoresist to the substrate better, relieves the internal constraints created during the centrifugation. For us to achieve the desired result, the temperature must be set carefully : we went with 100°C for 50 sec under the advice of our assistant.

Figure 15: Solvants evaporating during the softbake[6]

Step 2.4: Rehydratation (Relaxation time)

Once the softbake is done and the solvants have evaporated, we now need to let the wafer cool down in order to induce a rehydration. The cleanroom's humidity provides H2O which will diffuse in the resist. Its presence is necessary for the chemical reaction to happen during the UV exposure.Rehydration also affect the development time making it shorter. We left our wafer on top of an inactive Ceran 500 hotplate until it reached the temperature of 25° more or less the ambient temperature in the room.

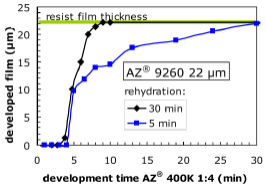


Figure 16: Influence of rehydration time over time needed in order to fully develop a resist[6]

Step 2.5: Exposure

The core step of the whole photolithography process is the exposure. When our positive photoresist is subjected to UV light through a photo mask the exposed part is weakened and when developed will disappear. We still had to figure out the adequate wavelength and dose for this process to happen in the best circumstances. As for the wavelength we used UV light at around 365[nm] which corresponds to the peak max relative intensity at the lowest wavelength for the best resolution.

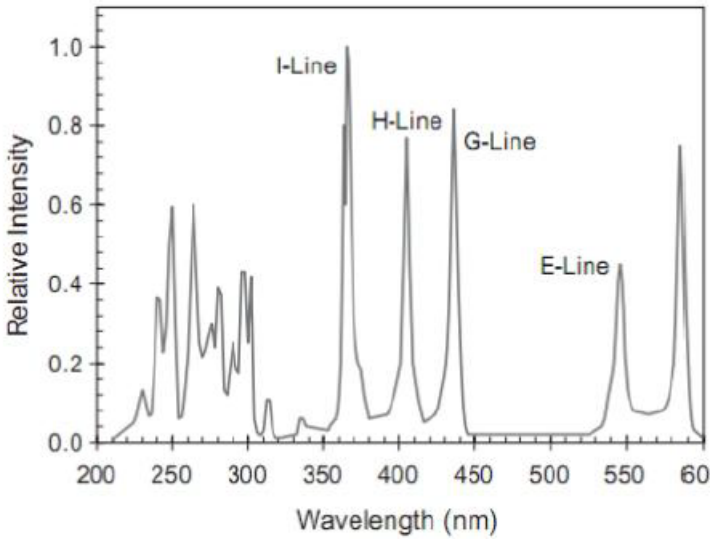


Figure 17: Relative intensity in regard to wavelength[6]

As for the dose, we know the following rule applies :

$$Dose = Intensity * Exposuretime$$

Intensity is fixed by default at 20 $[mW \cdot /cm^2]$ and thus to influence the dose the only left parameter is the exposure time.
We are using the SUSS MJB4 to expose our wafer and the two types of photomask available.We set the machine's parameters at 'Test Exposure', 'Hard Contact' and 6 , we also used the WEC setting on the machine to ensure the wafer and the mask are completely parallel to each other.For the contrast exercise, we used two different photomasks with 6 exposure cycles each ; one with pair numbered openings and another one with odd numbered openings. We took turns at exposing the wafer through each opening while making sure the others were covered. Each opening represented by a number was exposed for a different exposure time and thus at a different dose. We later studied the thickness of this wafer at each exposed area and compiled the data to figure out the the best approximation of D_{100} (the dose for which 100% of the thickness has disappeared).
For the alignment exercise, we now already know the correct dose to use in order to fully make the resist disappear after development.

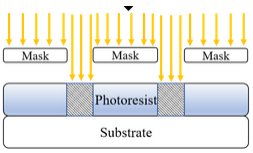


Figure 18: Photoresist exposed to light through a photomask[10]

Step 2.6-2.8: Development, cleaning and drying

Once the exposed photoresist has been weakened by the exposure to UV light, we need to proceed with development. Development consists of putting the wafer in a diluted developer solution. This solution will dissolve the weakened part of the resist and leave the rest intact.For that, we prepared a solution of 200 ml of AZ351B developer and 50 ml of water which corresponds to a ratio of 1:5. We then plunged the wafer inside the solution for approximately 90 seconds. The development time chosen was given to us by our assistant and corresponds to the ideal time for the dose used and the type of the resist. After the 90 sec have passed, we took out the wafer and rinsed it with deionized water before drying it with ~~tissues~~. We can also mention that the photoresist we are using has a higher development rate thanks to the PAC (DNQ) that becomes a catalyst to the dissolution reaction after exposure.

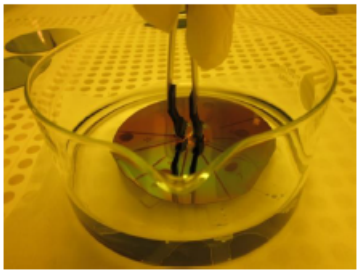


Figure 19: Wafer being developed[6]

Step 3.1: Thickness measurement

In light of the contrast exercise after exposing the wafer through different openings at different doses we took the wafer and measured the thickness of the film at each opening and compiled the data. We used the Filmetrics F20 UV. The machine measures the thickness of the resist using reflectometry. It measures the reflectance of a thin film and through the maximas and minimas it can figure out the thickness. The machine outputs the thickness and a goodness of fit percentage, the higher it is the closer the data is to the theoretical values. Through thickness measurement we were able to identify the first opening at which no thickness could be observed and knowing what dose went through which opening we figured out D_{100}.

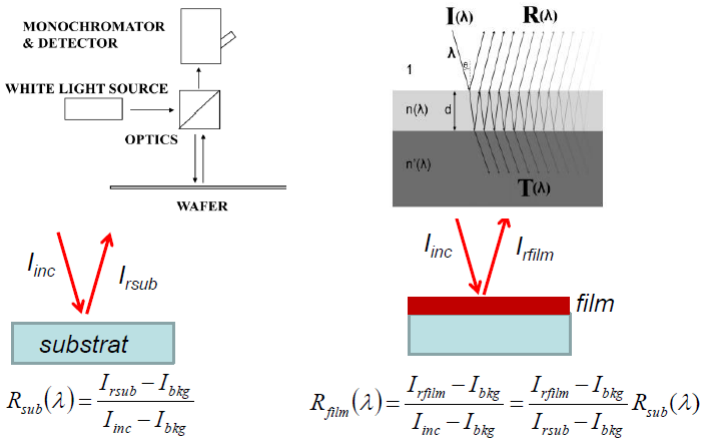


Figure 20: Reflectometry and formulas[6]

Step 4: Alignment and resolution observation

After figuring out the contrast characteristic of our photoresist, we did a second exercise where the objective was to deposit a second layer of photoresist expose it and develop it all while it is aligned with the first layer. When the whole process has been followed, it was time to check our results and see if we managed to align the second level with the first one using the Nikon Optiphot 200. It is a microscope with different zoom lenses. We took pictures of the alignment geometrical patterns as well as the resolution patterns using the 100x lens. In order to figure out the error at alignment we use the Vernier structures. They work exactly like a calliper and are made of a series of lines, the middle one being the reference point. We look right or left of the reference line looking for the first matching line between our first and second level vernier's structures. We count the number of lines n between the matching line and the reference one. The error at alignment is then found using the following formula :

$$\delta = \pm n * 200$$

The error at alignment can be defined at both axises x and y.

Figure 21: Alignment geometric patterns and vernier's structures[6]

2.4 Results

- Contrast characteristic of photoresist

Exposure time (s)	0.1	0.5	1.0	1.5	1.7	2.1	2.5	2.7	2.9	3.1	3.7	5.0
Dose ($mW \cdot s/cm^2$)	2	10	20	30	34	42	50	54	58	62	74	100
Thickness (nm)	1654	1584	1065	545.9	520	319	122	54	0	0	0	0
Goodness of fit		0.86	0.86	0.77	0.97	0.98	0.97	0.99	0.91	0.55	0.84	0.83

Figure 22: Thickness of film after different exposure times at constant intensity of $20mW/cm^2$

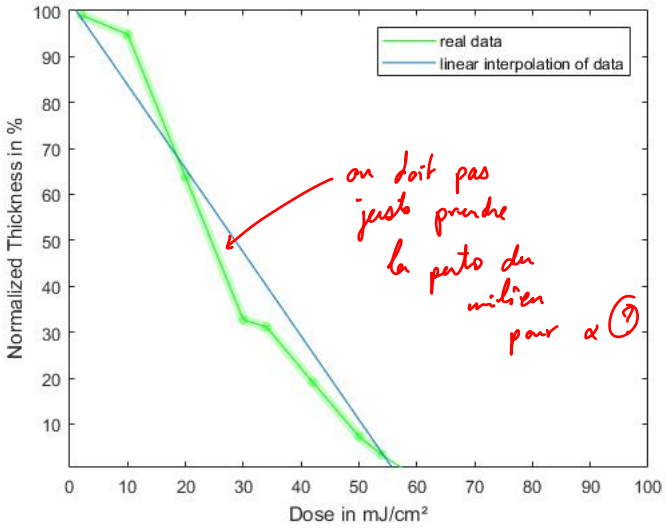


Figure 23: Graph showing Figure 22's data visually and the linear interpolation of it [12]

From the following graph we can calculate the contrast:

$$\alpha = \frac{1}{log(\frac{D_{100}}{D_0})} = 0.58; D_{100} = 56; D_0 = 1,1$$

This value for the contrast is very far from the value of 2.3 in the datasheet of the photoresist. We unfortunately have an inconsistency regarding our measured values since we do not have enough point we are trying to compute the complexity of the theoretical curve using only 12 measurement. We see our graph fend itself and break starting at the 5th measurement which makes it really hard to take the right and correct slope in order to find the contrast. Assuming this should follow a linear model, i used matlab to compute a linear interpolation of the data close to the 0% thickness but could only get 0.58 for the contrast. If we restrained ourselves to the measurement 2,3 and 4 and calculated the slope we find a contrast of 2.34 which is an exact fit to the datasheet. In a nutshell, we would need more data (12 points are a little short) and a little bit more precision in order to compute a concrete and coherent value for the contrast. If the problem really isn't from the data, we expect the small defects we saw during the spin coating to be the reason behind the machine not being able to identify the real thickness (small defects or particles could interfere with the measurement).

- Resolution In order to study the resolution, we had to study the differences between periodical patterns on our wafer and on the photomask. The periodical patterns on our wafer consist of resist lines and empty lines consecutively arranged in groups of different gaps. We had to chose the correct dose when developing so that the resist line between two empty lines in a gap would not be developed and remain identical to the photomask. The longer we expose and thus the bigger the dose, the smaller is the resolution.

Dose (mJ/cm²)	Pitch (Wafer) (µm)	Pitch (Mask) (µm)
54	4.9	5
58	3.62	5
62	2.42	5

Figure 24: Dose impact on wafer pitch size in comparison to Mask's pitch size

We can clearly see that the bigger the dose the narrower the gap between two line gets. The measurement has been done on the picture using photoshop and couting the pixels while comparing them to the scale. We can see some inconsistencies alongside the lines at some points the lines seem narrower (see following picture).

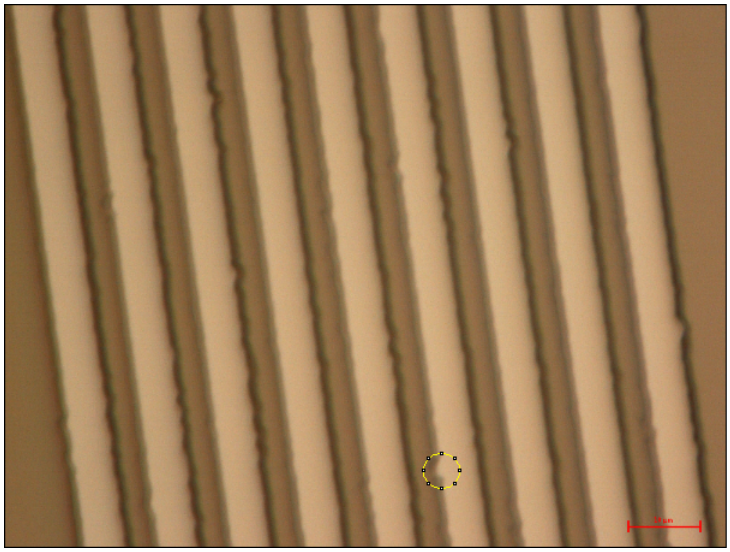


Figure 25: Pitch of the 4th gap at 100x , we can see encircled a small defect[11]

From the resolution perspective we understand the importance of the choice of the dose as too much exposure can seriously harm the resolution.

- Alignment Working on the second level of a wafer can be tricky since we have to take into account the first one. Alignment is important because the geometry of the two levels needs to be on point. Vernier's structures appear at 5 different positions in the wafer and allow us to figure out the alignment error.

X (mm)	Y (mm)	ΔX (nm)	ΔY (nm)
0	32	800	1000
0	-32	200	600
0	0	600	1000
28	0	400	800
-28	0	600	800

Figure 26: Alignment error

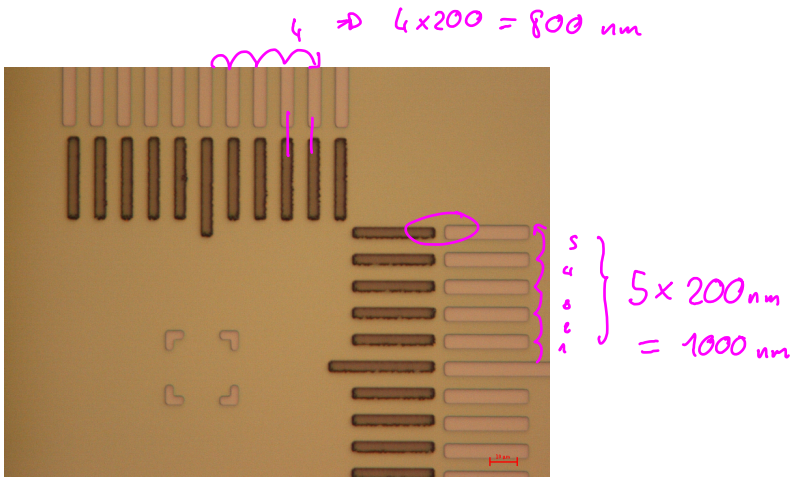


Figure 27: Alignment pattern at(X:0 Y:0)[11]

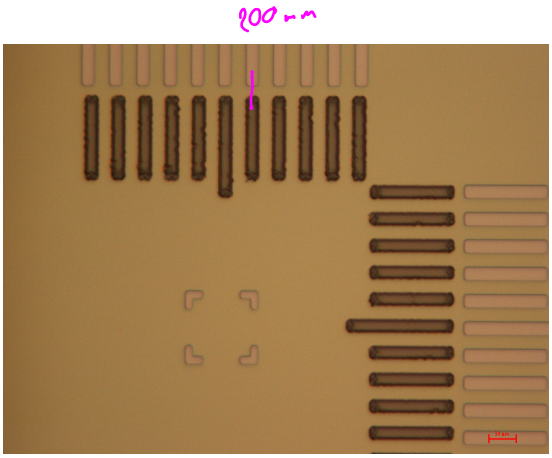


Figure 28: Alignment pattern at(X:0 Y:-32)[11]

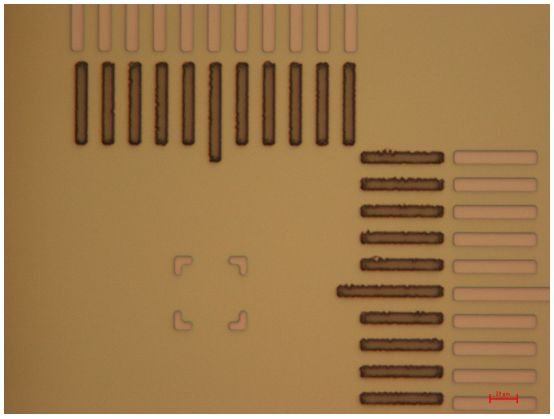


Figure 29: Alignment pattern at(X:-28 Y:0)[11]

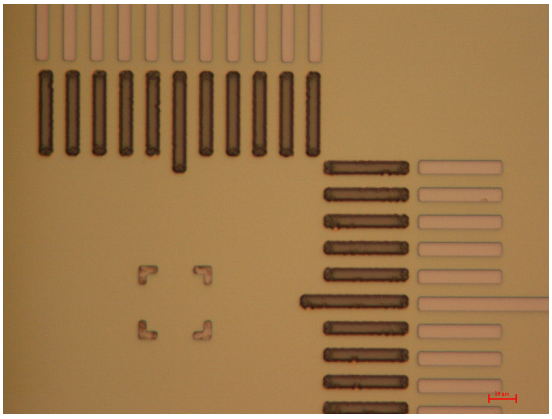


Figure 30: Alignment pattern at(X:28 Y:0)[11]

We believe our alignment has been more or less coherent and correct we don't seem to have any alignment error off the charts. The error can easily be explained by the human manipulation of the different levers. The levers are also too sensitive and we found it really hard to reach perfection.

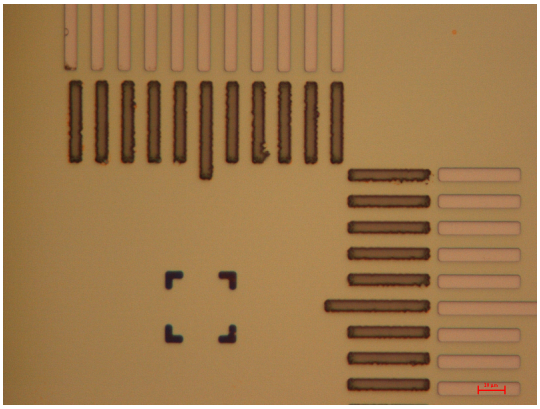


Figure 31: Alignment pattern at position (X:0 Y:32)[11]

3 Session 2: Aluminium wet-etching

3.1 Objectives

The primary goal is to create conducting routes onto a wafer of aluminium to demonstrate the fabrication of micro-electronic connectors between generic integrated circuits. This is done via an Alu-etching process, where the prepared aluminium is etched, resist-stripped, and measured.
To set measurement references, we photograph the the mask and the substrate wafer (with resin intact). The engraving procedure requires dipping the prepared aluminium wafer in ANP solution for a regulated time. This time is a critical factor for guaranteeing optimal dimensions of the conducting routes. Leaving the wafer too long or too little in the ANP will significantly vary the local structure of the routes. In extreme cases this can lead to short circuits and systemic overheating (significant geometry variation when under a voltage load, will wear out much faster).
The reaction itself is highly exothermic, extra protection is necessary.
Stripping the resist will then remove the photo-resist layer off the aluminium so that testing can begin. We then use optical and electronic measurements to test the quality each wafer over the etching time. To do this, we inject currents onto multiple control routes in the aluminium, and from this deduce the local square resistance via the van der Pauw method. Finally, we measure the nominal dimension of each route to determine the resistance (TLM method, under microscope). Using the reference, we can deduce the accuracy of the etched aluminium, and discuss the local deformities of the geometry.

3.2 Machines & Measuring tools

- Aluminium wafer: pre-prepared Al 800nm on glass wafer.
- ANP solution: composed of HNO_3, H_3PO_4, and CH_3COOH diluted in H_2O to control the etch rate. In our case, UN 3265 (AL 80) is used. This solution defines the relative concentrations of each agent.
- Resist stripping: SVC-14 was is used to strip the resist, a better alternative to acetone (dries too fast, must be rinsed with IPA before removing with deionized water).
- Rinsing: de-ionized water used after the resist striping procedure.
- Etching support: Arec. X hot plate is used to maintain the etching temperature, stir the solution, and connected with a temperature sensor. Using a timer the etching process can be monitored.

- Electrical measurement: KSM PM8 Karl Suss workstation, using 4 electrical probes placed at each motif, we use 2 to impose a current, and 2 to measure the voltage. An on-board camera and screen allows for substrate coordination.

- Optical measurement: Nikon Optiphot 200, used to photograph the micro-structures along with the corresponding magnification and scale.

3.3 Process flow

Prior to all experiments, we first took photographs of the mask and resined substrate using the Nikon Optiphot 200 under 100x magnification on the 6 and 8 μm checker squares to use as a reference.
Now we will focus on specific steps in the etching process. References will also be made to the process flow sheet of this practical, **except steps 1 though 3**, as they were completed by the assistant.

Step 4: These protocols aim to optimize the etching rate of the ANP into the aluminium substrate, in theory without causing significant under etch.

Figure 32 demonstrates what we expect to happen after immersing the wafer. Prior to immersion, safety protocol must be followed due to the reactions' highly exothermic nature. A second layer of latex gloves, a swell as a third layer of long, chemical resistant gloves are required. Them a full body protective apron, and finally a transparent plastic visor that covers the entire face. The reaction chamber (wet-etch-bench) was prepared with the equipment set up, it was our responsibility elevate the temperature to 35^oC using the Arec.X. It also contained the ANP solution in a beaker. A magnetic stirrer guaranteed solution homogeneity during the reaction.

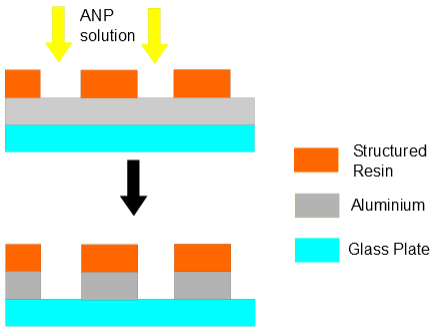


Figure 32: Theoretical consequence of wet-etch[9]

After maintaining a stable temperature, the substrate was vertically supported by a structure to prevent it from moving. Upon immersion, a timer was started, and the reaction began. Quickly we noticed changes in the colour and transparency of the substrate. The expected etch-rate is 300nm/min, however the temperature fluctuated between $[34, 37]^oC$ which implies non-optimal reaction variations. After 10 minutes, the etched aluminium was removed from the bath,washed with deionized water, and dried with a nitrogen gun. This step was repeated to removing as much of the leftover ANP as possible. At any time, if any wafer had liquid on it, we could not assume it was deionized water, and so we had to keep track of when each substrate was keeping cleaned(since the liquid could have been ANP).
The bench was cleaned with paper drenched in deionized water, as to avoid fire erupting from residual reaction of the ANP on the paper.
At this point, we expect the substrate to have the profile shown in the bottom half of figure 32: the aluminium

has been etched everywhere except where the photo-resist is directly on top of it.

Step 5: Now we seek to remove the structured photo-resist from the etched surface: this was done by applying SVC-14 planar to the substrate until it was entirely covered and left in a basin with deionized water for a few minutes. Removing it with the tweezers, it was again cleaned off with deionized water and dried with a nitrogen gun. Our substrate somehow cracked as it was dried,and broke into 2 pieces. This was not detrimental, we could still measure the routes that were not along the crack. Figure 33 demonstrates how we expect the surface to appear: only the glass plate and aluminum, upon which we previously had the structured resin.

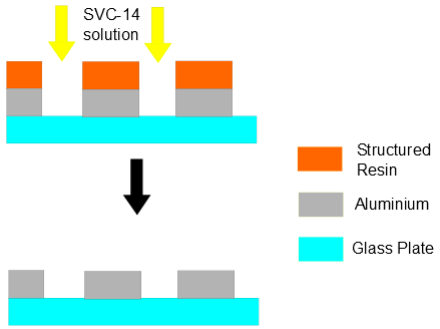


Figure 33: Resist-Striping[9]

Step 6: Finally we measure the electrical properties of the substrate using the KSM PM8 Karl Suss device to determine the route dimensions. To do this, we use the properties of square resistance and the data from the local resistance of the micro-structures. From the theory, we know that:

$$R_s = \frac{\pi}{ln2}\frac{V_{12}}{I_{12}} = W\frac{R}{L} \leftrightarrow W = R_s\frac{L}{R}$$

By measuring R_s we can plot the graph(calculate the gradient) of (R, L) values to find the true width W of the routes after etching. This was done by securing each substrate fragment under vacuum onto the stage. Arranging the 4 electrodes on the contact pads and calibrating the program SW on the computer, we looked for figure 34 under the on-board microscope. The measurements were taken on the 30μm route.

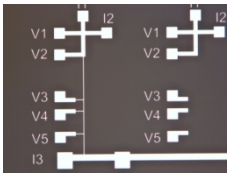


Figure 34: Electrode Contact Pads[7]

V_{12} is the measured potential from V_1 to V_2 and I_{12} is the imposed current entering I_1 and exiting I_2. Placing the 4 electrodes across these contact pads and measuring the potential defines the square resistance R_s. By moving the first two electrodes at points (V_2, I_2), we successively reposition the other two on the other points (V_3, I_3), etc. and measure the resistance at each point. We then repeat the process by moving the fixed electrodes to (V_3, I_3), and measuring from (V_4, I_4). The full set of permutations is found in our

result table(Figure 38). The length of each route is known since the wafer resin was pre-prepared in those dimensions. Furthermore the etch process would mainly affect the W dimensions since they expose the most surface across the length of the route.

3.4 Results

Using the Nikon Optiphot 200, we compiled pictures of checkered patterns left by the mask, the resin on the wafer and the aluminium left over after etching. Using the ImageJ measuring application, we can view and the width of the checkers before and after etching and view determine the corresponding under etching that has occurred after the etching. These photographs are of the 8μm pattern on the substrate. For all the photographs, along with their calculated under-etch, please refer to annexe 6.1

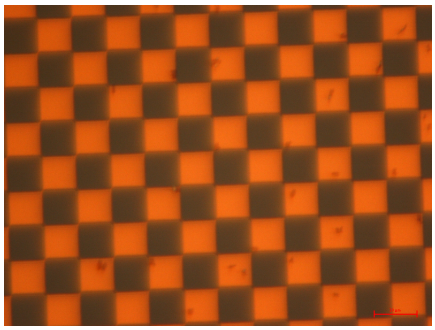
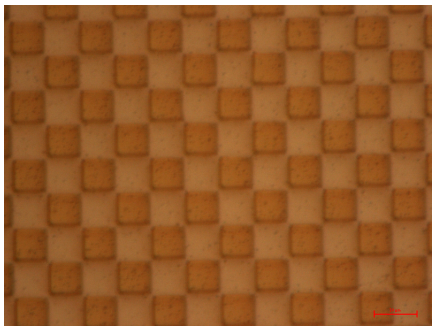


Figure 35: Checker pattern 8 as seen on the mask[11]

The checkered here seen on the mask is fabricated to be exactly 8μm in width. using ImageJ we measure them to be about 8.02 μm. This difference can be explained by human error during the measuring process, as well as image focusing issues.



ces tests c'est juste pour regarder en vitesse ce que ça donne ?

Figure 36: checkers pattern 8 in resin[11]

The resin should have the exact same pattern as the mask did after exposure. Using ImageJ, we measure 8.03 μm in width. Again, this difference can be explained by human error. But it may also be attributed to imperfections during the exposure process.

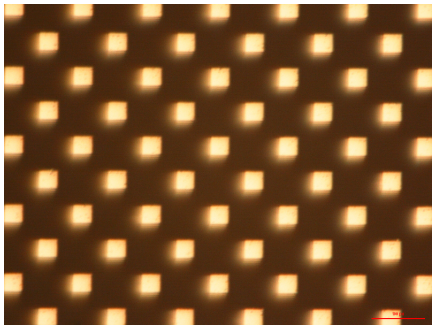


Figure 37: checker pattern 8 after wet etching[11]

This image shows us the resulting aluminium pattern after the etching. Using ImageJ, we measure a width of 3.83 μm and a gap between checkers of 12.13 μm.

Etching time (min)/ $W_{mask}(\mu m)$							
Contact pads	12	23	24	25	35	45	34
Length (μm)		400	500	700	300	200	100
Resistance ($m\Omega$)	39.4	609.4	759.1	1062.0	453.3	303.3	150.0
Local width (μm)		25.83	25.95	25.97	26.08	25.98	26.27

Figure 38: Resistance between two pads and calculated width of the 30μm route

Figure 38 describes the full set of data taken from the electrical measurements. For simplicity, we have included the square resistance R_s and the local width values just to indicate what set of widths to expect.

3.5 Analysis and conclusion

Now we will compare the under etching values from the electrical and optical testing.
In figure 39 we see the values of figure 38 plotted against each other, and interpolated in a linear regression:

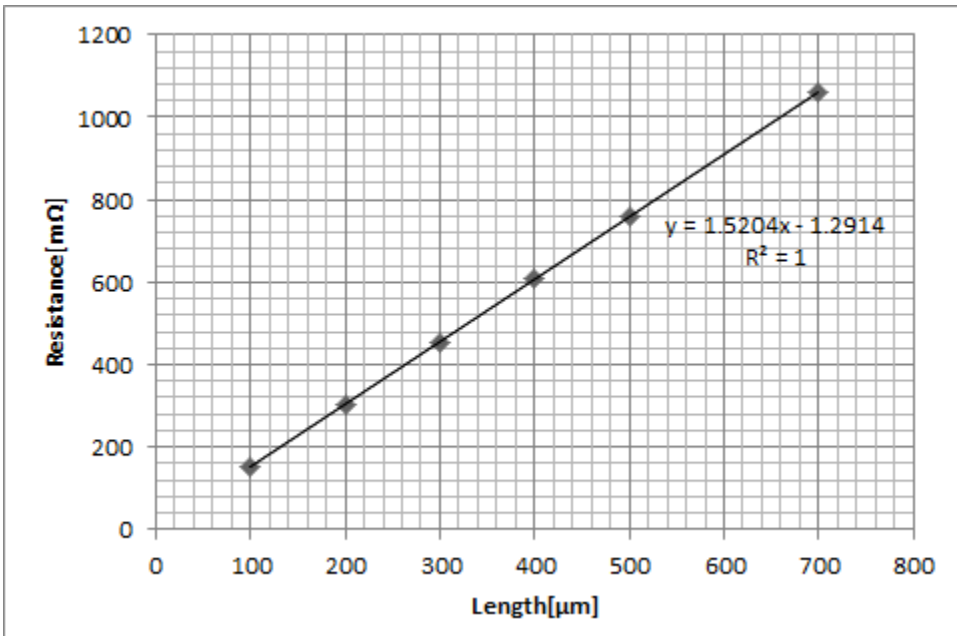


Figure 39: Electrical resistance depending on the Length[13]

The data is a perfectly linear relation, as indicated by the coefficient of regression $R^2 = 1$. This implies precise data collection and a correct model for the system. Furthermore, the gradient $\frac{Resistance}{Length}$ directly gives us the value needed to determine the true width of the route. It's worth noting that the y intercept is not 0, which is what we would normally expect. This is likely from internal resistances of the apparatus, and so our data is **precise** but not **accurate**. It's easy to compensate for this by ignoring the intercept and assuming it to be 0. So by ignoring -1.2914 and shifting the graph vertically up to the origin, it would be equivalent to "removing" the apparatus resistance connected in series to the substrate resistance. Consequently, the gradient will not change.
Finally, we calculate the final width of the 30μm route to be:

$$W_{30,exp} = 39.4\frac{1}{1.5204} = 25.91\mu m$$

Since $25.91 < 30$ we clearly have under-etching taking place, whose amount is calculated to be:
$t_{under-etch,electrical} = t_{ue,el} = \frac{30-25.91}{2} = 2.05\mu m$
Figure 40 shows the compiled data of all optical measurements in annexe 6.1 which we will now use to determine the under etch across other points on the wafer.

Etching time (10min)				
	W_{Al-opt} (μm)	W_{resine} (μm)	W_{mask} (μm)	$\Delta W_{opt} = W_{resine} - W_{Al-opt}$ (μm)
8 μm checkers	3.83	8.02	8.04	4.19
6 μm checkers	2.09	6.06	6.0	3.97

Figure 40: Width of checkered patterns on the mask and wafer

From the ΔW values we can deduce the under-etching:

$$\frac{\Delta W_8}{2} = \frac{4.19}{2} = t_{ue,opt8} = 2.09\mu m$$

$$\frac{\Delta W_6}{2} = \frac{3.97}{2} = t_{ue,opt6} = 1.99\mu m$$

Now we take the average of these values to find a generally descriptive under-etch value for the whole substrate:

$$t_{avg} = \frac{t_{ue,opt8} + t_{ue,opt6} + t_{ue,el}}{3} = \frac{2.09 + 1.99 + 2.05}{3} = 2.04\mu m$$

We can go one step further an examine the isotropic nature of the under-etching rate:

$$\frac{t_{avg}}{10minutes} = r_{ue} = 204.33nm/min$$

Despite expecting an etching rate of $300nm/min$, r_{ue} indicates a more anisotropic behavior, relatively speaking. This leads to the conclusion that the ANP behaves about $\frac{2}{3}$ as isotropic in the lateral dimensions from the vertical ones.

4 Conclusion

This workshop has been a good introduction to the world of working in a clean room. We were lucky enough to discover certain essential concepts and procedures that are used everyday in such an environment. From photo-lithography and the choice of the correct resin; wafer photography and the alignment process; and not forgetting aluminium etching and the measurement of under etching.
Overall, we can say that we learned a great deal during these two practical sessions in the clean rooms. It was an enriching experience that allowed us to better understand all the theoretical notions and elements that we have seen in class. However it is important to note that all the procedures we preformed by hand are done mechanical on an industrial scale. This insures a higher quality in the final product and eliminates the biggest element of pollution in clean rooms: humans.

5 Individual work: High Resolution Photoresist Processing

5.1 Introduction

This section synthesize a small document called *High Resolution Photoresist Processing* [15]. The different steps of such a process will be presented, emphasizing on the most important point that can deteriorate the result.

A resist is a liquid polymer, which can selectively change his state when exposed to a certain kind of light. This effect is due to the presence of photo-active molecules which enhance the polymerization of the resist when activated by light. We use resist to cover wafers, and to partially protect them, in order to selectively etch the places where resist is not present. The pattern to etch is produced in the wafer using the following process: The liquid resist is first dispensed on the wafer. The product is then backed, in order to evaporate, and to increase its viscosity. After a small rehydration step, the resist is exposed to light, in order to draw the pattern. Finally, the wafer is placed in a specific solution that will dissolve the unexposed part of the resist, conserving the exposed one.

5.2 Diversity of high-resolution resist

Resist are characterized by wavelength. They are separated in three family. The emphg-line concern resist that react with wavelength around 435 nm. *H-line* concern resist reacting around 405 nm and *i-line* resist

work around 365 nm.

There are two type of resist. The first one called *positive resist* behave as presented in the introduction [5.1], which mean that only the unexposed resist will dissolve. On the other hand, it also exist resist that dissolve only where it has been lightened.

5.3 Resolution and selectivity

The purpose of this technique is to draw a pattern on the resist, which will replicate on the layer above during the anisotropic etching step. The result of this drawing can be characterize by *resolution*. The resolution is the property of a finished product, to present a clear *contrast* between the etched and non etched area, at the lowest possible scale. The resolution and the contrast are directly linked to the *selectivity* of a process.

The resist is exposed to light, and then placed into a solution which will dissolve the unexposed part of the resist (exposed part in case of using *negative resist*) at a certain rate. This rate is called *development rate*. This dissolution is not perfect and will still degrade the exposed part at a very low rate. This rate is called *dark erosion rate*. The *selectivity* is the ratio between the *development rate* and the *dark erosion rate*. The highest is the selectivity, the better are the contrast and the resolution at the end.

The next section present the different steps, and the critical aspect to consider, regarding their impact on selectivity and final result.

5.4 Detailed analysis of the steps

5.4.1 Resist dispensing

At first, the wafer need to be coated with a thin and regular film of liquid resist. This layer is obtain by rotating the wafer while pouring resist in the center. This technique is not further described here, because it is part of another document called *Spin Coating Photoresist*[16]

5.4.2 Softbake

The resist is composed with solvent, which allow it to flow smoothly during the *spin coating step* [5.4.1]. The layer is soft-baked in order to evaporate those solvent and increase the viscosity to confer a more stable aspect to the layer during the *exposure* [5.4.4].

The time of the softbake is very important and need to be controlled precisely. If the softbake is performed too short or too cool, there will be too much solvent remaining which will induce a too high *dark erosion rate* during the *development step*[5.4.5] and lead to a bad *selectivity*. On the other hand, if the softbake is performed too long or too hot, part of the photo-active molecules will be thermally decomposed, leading to a low *development rate* inducing a bad selectivity.

5.4.3 Rehydratation

The softbake step evaporate the solvent, but also lead to almost water-free composition of the resist. The problem is that the *development step*[5.4.5] need water to attain a correct *development rate*. As a result, the wafer need to wait in specific air condition for water to resorb from the air to the resist layer. The humidity of the room must be over 40 % to allow a good and fast resorption.

5.4.4 Exposure

The exposure is the principal part of photoresist processing. Many parameters need to be taken into account to achieve a good result.

The first parameter is the wavelength. Optics laws shows that the final resolution is linked to the square-root of the wavelength. As a consequence, reducing the wavelength allow achievement of a better resolution. Unfortunately, the existing resists does only absorb light within a specific spectrum. Thus, it is useless to use a wavelength smaller than 340 nm (corresponding to *i-line resists*). Contrariwise, using a smaller wavelength will involve exposing the resist longer to obtain the same amount of excited photo-active molecules. On that account, the edges of the pattern will be exposed by reflection and refraction, and this will lead to a none-accurate shape of the pattern.

The same problem appear when using a too high *exposure dose*. Conversely, a too low *exposure does* imply a longer *development step*[5.4.5] which lead to a greater amount of dark-eroded matter.

The properties of the substrate can also influence the result. For example, a structured wafer can cause light to scatter or refract, leading to unwanted exposure. Moreover, transparent (to the considered wavelength) and glassy substrate can act as waveguide and cause underexposure leading to a bad result.

The gap between the exposure mask and the layer of resist also influences the size of the projected pattern because of diffraction.

Many other elements such as particles, bubbles, edged bed or reversed-mounted mask can involve a bad resolution.

5.4.5 Development

After exposure, the mask is placed into a diluting solution to remove unexposed resist (exposed in case of use of *negative resist*). This solution is obtain by mixing water with an active diluter component called *the developer*.

If the dilution ratio of *developer* is miscalculated, the final product will be affected by too much dark-erosion or underdeveloped part whether the concentration of diluter was too low or too high.

5.5 Conclusion

To conclude, let remember that the final result is drastically affected by *selectivity*. As a consequence, one must know the main aspects and their relation with *selectivity*, in order to obtain a clear pattern on the resist layer, inducing a good contrast in the etching step that follows.

6 Annexe

6.1 Aluminium Substrate Photographs

All photographs were analyzed used ImageJ to determine the lengths of each dimension, and so we also give the thickness of the final etched aluminium. All samples are under x100 magnification ($10\mu m$ scale)

6.1.1 6μm Pattern

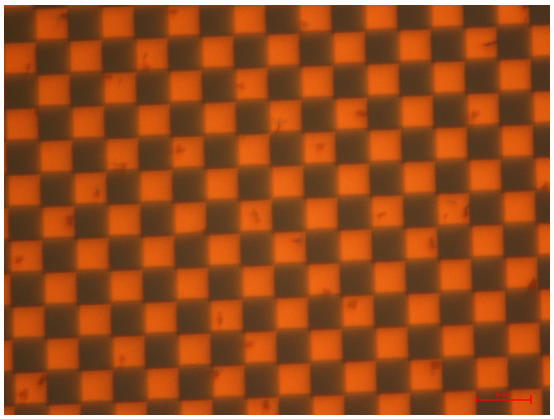


Figure 41: 6μm Pattern of the mask[11]
Measured side length of squares: 6.0 μm

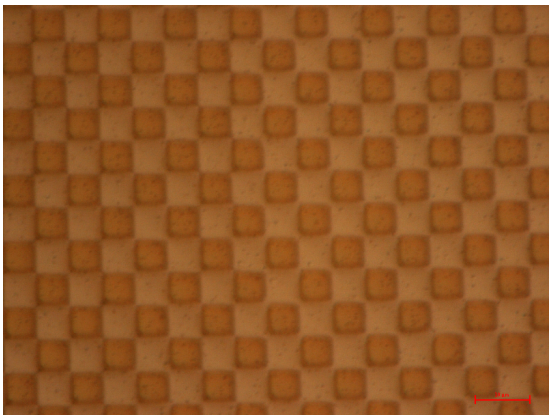


Figure 42: 6μm Pattern of the resin[11]
Measured side length of squares: 6.06 μm

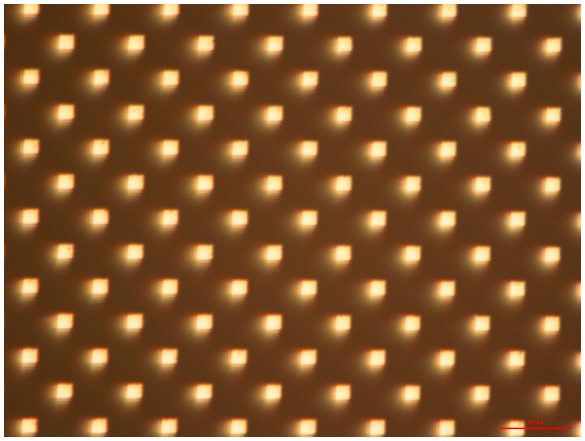


Figure 43: 6μm Pattern post-etching substrate[11]
Photograph scale indicates magnification not length
Checkers width: 2.09 μm Gap Width: 10.15 μm

6.1.2 8μm Pattern

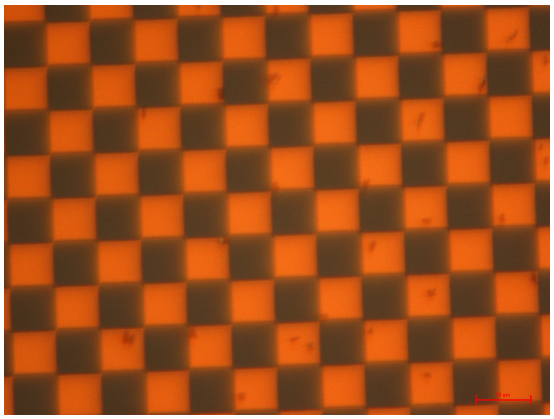


Figure 44: 8μm Pattern of the mask[11]
Measured side length of squares: 8.02 μm

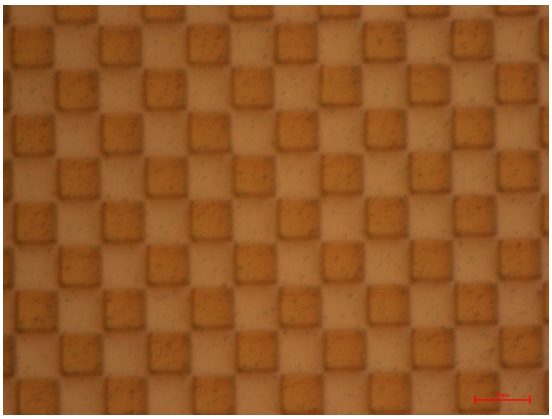


Figure 45: 8μm Pattern of the resin[11]
Measured side length of squares: 8.03 μm

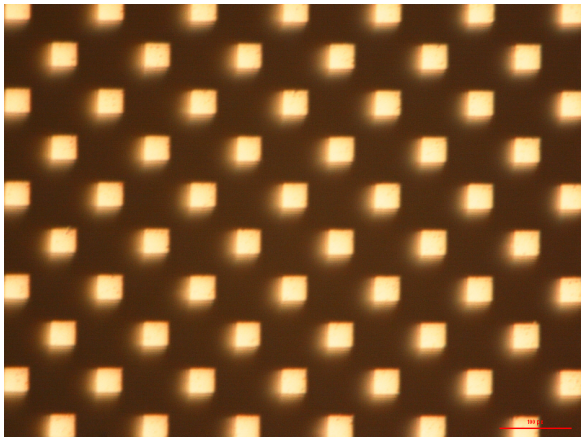


Figure 46: 8μm Pattern post-etching substrate[11]
Photograph scale indicates magnification not length
Checkers width: 3.83 μm
Gap Width: 12.13 μm

6.2 Mentionned documents

All mentionned documents were furnished during the *Travaux Pratiques en Salle Blanche*[1]course. The process flow sheet can be found after the *references* section.

References

[1] *Technologie des microstructures*, Course given by Pr. Brugger Jürgen & Pr. Gijs Martinus, completed by 2 practical work session given by Pr. Sayah Abdeljalil and Assistant Atakan Baris http://edu.epfl.ch/coursebook/fr/technologie-des-microstructures-i-MICRO-331 and https://edu.epfl.ch/coursebook/fr/tp-en-salle-blanche-automne-MICRO-332

[2] *Clean room equipment wearing steps*, This video present the steps required to put on the 1000 class cleanroom suit and has been taken from the MOOC[3] linked to the course: *Technologie des microstructures*[1] https://youtu.be/y739O8By_Hw?t=8m1s

[3] *MOOC lessons of* Technologie des microstructures[1], The MOOC which present the content of the course *Technologie des microstructures*[1] can be found at this address https://courses.edx.org/courses/course-v1:EPFLx+memsX+3T2017/course/

[4] *Photolithography Equipment*, original pictures and details about this equipment can be found on the CMI website:https://cmi.epfl.ch/photo/home_photo.php

[5] *Metrology Equipment*, original pictures and details about this equipment can be found on the CMI website:https://cmi.epfl.ch/metrology/home_metrology.php

[6] *Travaux Pratiques en Salle Blanche*, this picture was taken from the instructions document called *TP_AL.pdf* and given with the course *Travaux Pratiques en Salle Blanche*[1]

[7] *Travaux Pratiques en Salle Blanche*, this picture was taken from the instructions document called *TP_Micro_322_al.docx* and given with the course *Travaux Pratiques en Salle Blanche*[1]

[8] *Photomask with different patterns*, this illustration was taken from the following website:https://htaphotomask.com/

[9] *Self-made picture*, this picture was generated by our team using : *Inkscape*

[10] *Photoresist exposed to light through a photomask*, this illustration was taken from the following website:https://en.wikipedia.org/wiki/Photoresist

[11] *In lab pictures*, this picture was taken during the session in cleanroom using the microscope called *Nikon Optiphot 200*[5]. More details about this equipment can be found in section 2.2

[12] *Graph showing Figure 22's data visually and the linear interpolation of it* Team-generated graph from collected data made with *Matlab*

[13] *Electrical resistance depending on the Length* Team-generated graph from collected data made with *Excel*

[14] *Students wearing CMI 10'000 class cleanroom suit* This picture was taken inside a 10'000 class clearoom during a 3^{rd} year Microtechnic bachelor cleanroom practical work session in 2016.

[15] *High Resolution Photoresist Processing*, This document is a small PDF presenting the different steps of High Resolution Photoresist Processing. All steps are quickly presented, pointing out the aspect that can deteriorate the final result. The document can be found in the Annexe section [6] or on the following website https://www.microchemicals.com/technical_information/high_resolution_photoresist_processing.pdf

[16] *Spin coating Photoresist*, This document explain in detail the spin coating step and can be found on the following website :https://www.microchemicals.com/technical_information/spin_coating_photoresist.pdf

Projet : TP Micro332 - Photolithographie
Student : Guillaume Aladin **Group:** 7
Date : 20.11.17

Step	Description	Equipement	Program / Parameters	Target	Actual	Remarks
1	**WAFER PREPARATION**					
1.1	Stock out "litho" wafers					
1.2	Stock out "align" wafers					
1.3	Check	Z15/F20 Thin-Film Analyzer				First calibrate on sample Si wafer
2	**PHOTOLITHOGRAPHY: "LITHO"**					
	Check machines	Z13/SSE coater		100mm chuck in position		
		Z13/SSE hotplate		**100°C**		
		Z13/MJB4		Lamp on		
2.1	HMDS priming	Optiphot VB20 P	T° : 135°C / Others : 0	135°C	135 ± 0,5 °C	A bit oscillating around 135
2.2	AZ1512HS coating	SSE SB20/213	3000 rpm for 45s	3000 rpm for 45s		Small defects (particles)
2.3	AZ1512HS softbake	LHG-2860 SR	100°C for 50s.			
2.4	AZ1512HS relaxation time	Coran 500		Ambient T°	25,6°C	
2.5	AZ1512HS expose	Süss MJB4 (single side Mask aligner) [Cmi Zone 13]	Increase light Test exposure Create hard-contact	See table below		
2.6	AZ1512HS develop	Diluted AZ351B	Ratio 1:5	50 ml AZ351B 200 ml Water		
2.7	Water cleaning	de-ionised water				dry with nitrogene blow gun
2.8	Inspection					→
3	**MEASUREMENTS: "LITHO"**					
3.1	AZ1512HS Thickness	FilMetrics F20-UV (214)		1600	1669 nm - 1671 nm	
4	**MEASUREMENTS: RESOLUTION**					
4.1	Resolution of structures	Nikon Optiphot 200	Increase light	Correctly exposed, underexposed and overexposed regions.		50x objective can touch mask because it's thicker than a wafer.
4.2	Alignment quality	Nikon optiphot 200		Measurements on Vernier microstructures.		

One of the wafer looked very under exposed, seems like it moved between the exposure (not used for the future manipulation)

(20 mW/cm²)

	1	2	3	4	5	6	7	8	9	10	11	12
Exposure time	0,1	0,5	1,0	1,5	1,7	2,1	2,5	2,7	2,9	3,1	3,7	5,0
Dose												
Thickness (nm)	1654	1584	1065	545,9	520	319	122	54	0	0	0	0
Corelation ratio	X	0,8611	0,8592	0,77	0,97	0,98	0,97	0,99	0,91	0,55	0,84	0,83

Projet : **TP wet etch (Wet etch, Al/Float)**
Student : Guillaume Aladin **Group:** 7
Date : 24.11.17

WORK BEFORE STUDENT SESSION

Step N°	Description	Equipement	Program / Parameters	Target	Actual	Remarks
1	**WAFER PREPARATION**					
1.1	Stock out			Al 800 nm on Glass		
2	**PHOTOLITHOGRAPHY**					
2.1	Dehydration	Z1/ ACS200				Included in AZ1512 coating recipe
2.2	AZ1512 coating/bake	Z1/ ACS200	Std recipe AZ1512.2um0.Dehydr.	Coat 7 "wet etch" wafers		First dummy wafer (PR height = 2um)
2.3	AZ1512 expose	Z6/ MABA6	Alignment level 3 (**TP Micro**). Hard contact time: 10s, light intensity: **CP**	Exposure dose: 60[mJ/cm^2]		all 7 "wet etch" wafers
2.4	AZ1512 develop	Z1/ ACS200 followed by Z6/ EVG150	Std recipe **Dev.AZ1512.2um0** followed by **AZ1512_1to2um_Std_1_5**			First dummy wafer. Development incomplete after first run. Completed development on the EVG150
2.5	backside cleaning	Z1/SRD	10min program			Not done
2.6	Inspection	Z13/ µScope				
3	**DESCUM**					
3.1	O2 plasma	Z11/Tepla	Std recipe **"program 29" - 500W for 10s**			Not done

WORK @ STUDENT SESSION

Step N°	Description	Equipement	Program / Parameters	Target	Actual	Remarks
4	**ETCHING**					
4.1	Wear protection	extra glove + apron + face protection				
4.1	Prepare hot plate + sensor + magnetic stirrer to reach 35°C	Arec. X	Spinning + heating	35 °C	35 °C	to do in advance to reach temp.
4.2	Etching	Arec. X	35°C for 10 min.	35°C	~ [34;37]	etching rate: 300nm/min
5	**RESIST STRIPPING**					
5.1	Resist stripping	SVC - 14	planar to the wafer			
5.2	Rinse	de-ionized water				Wafer broke in two pieces when using the tweezer
6	**ELECTRICAL MEASUREMENT**					
6.1	switch on microscope, analyzer, screen and amplifier	KSM PM 8 Karl Suss				

- switch on microscope, analyzer, screen and amplifier
- load wafer on the stage and apply vacuum
- set 4 POINTS configuration: choose pos. IV with the knob and check 4 wires connecting with corresponding colours on the tips
- bring carefully stage UP and position 4 probes checking on the screen and in the binocular
- open SW, load TP workspace and choose 4points measurement
- export data
- $R_{\square}$ = 39,4 [mΩ] (contacts 12)

Temps de gravure (min)/ W $_{mask}$ (µm)						
Numéro des contacts	23	24	25	35	45	34
Longueur (µm)	400	500	700	300	200	100
Résistance (mΩ)	609,4	759,1	1,0620	453,3	303,3	150,0