Use of NIL for the fabrication of nano wires for possible application in biotechnology

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Abstract:

Within the scope of this project, investigations were made into the possibility of replacing Electron Beam Lithography (EBL) with NanoImprint Lithography (NIL) for creating arrays of gold dots on substrates, with the aim of nanowire growth. The attempted nano wires grown were aimed to be of use in the field of biotechnology. The main focus and aim of this project has been to perfect a fabrication route for creating organized arrays of nano wires based on NIL. NIL had been considered as a replacement due to the many advantages it hold over EBL, such as high throughput to name one.

Inom ramen för detta projekt undersöktes möjligheten att ersätta Elektronstråle Litografi (EBL) med Nano Imprint Litografi (NIL) för att skapa uppställning av guld prickar på substrat, i syfte att växa nanotrådar. Avsikten med nanotrådarna var att använda dem inom bioteknik.

Tyngdpunkten och ändamålet med projektet har varit att fullända en tillverkningsprocess för att skapa organiserade grupperingar av nanotrådar baserade på NIL. NIL har ansetts som en ersättning på grund av de många fördelar den har över EBL, såsom hög produktionskapacitet för att nämna en.

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1.Introduction:

Nanotechnology is thought to be an adequate improvement for many standard devices in the future. There are many hopes that in coming years the technologies will become far more compact with an increased performance compared to what is currently available.

Research has shown that these technologies are within our grasp. However, one of the main stumbling blocks in the commercial viability of the processes is the fact that devices produced in this manner usually have quite a low throughput.

One fabricated structure with many applications in various disciplines is the nanowire. Nanowires have an intrinsic property, due to their small size, in which quantum effects are dominant, thus it is possible to observe many new properties for various materials.[1]

There are many kinds of nanowires, metallic (Ni, Pt), insulating (SiO2, TiO2), and semiconductor nanowires e.g. Si, GaN etc.[2]

To achieve nanowire growth, gold seed particles need to be deposited on the surface of the desired substrate material. These particles act as catalysts for wire growth.

In this project, GaP nanowires grown using the Metal-Organic Vapour Phase Epitaxy (MOVPE) are the semiconductor wires of interest. More information about MOVPE and wire growth can be found in section 2A.

There are various ways of depositing gold on the material of interest for wire growth. Some deposition methods, such as aerosol deposition, are fairly quick. However, this results is a random distribution of gold particles which leads to the random placement of wires. In order to obtain an arranged order of nanowires, one must create a pattern and form this pattern in the resist prior to deposition of gold. The standard method for this pattern formation at Lund Nanolab is electron beam Lithography (EBL). A short explanation of EBL can be found in appendix B. EBL is extremely sensitive and accurate, however the process is slow and expensive, and as such, not commercially viable.

The work presented herein describes a method which can be used as a viable replacement of EBL in order to obtain ordered nanowire arrays. Instead of EBL, one should have an alternative, faster method which could produce a pattern with fidelity and high throughput. An alternative method which is employed for this aim is known as nanoimprint lithography (NIL)[3]. More information regarding the NIL can be found section 2B.

The wires produced within this project were targeted for use in cellular force measurement studies. This is necessary in order to understand how cell growth is effected by its surroundings, and the stress and tension applied to it. [4][5] For this it is necessary that the wires are arranged in a hexagonal array, as in this case the nanowire distribution (which during cell force measurements would act as measurement probes) would be ordered and equidistant from one another, meaning that the cell growth would have no preferential direction to grow in . As mentioned briefly previously, the wires used in this experiment will be GaP nanowires.

If the NIL method proves successful however, the produced wires could be adapted for various applications and uses, and so would not be limited to only this application.

Production of wires using NIL has been attempted before, with successful results[6]. One of the aims of this project was to observe whether these results could be duplicated, and if so what standard parameters are required for this.

Section 2 will be focusing on the general principals used within the fabrication of this project, whilst section 3 will discuss further the optimization methods and parameters used.

2.Methods:

2A: Nanowire growth Using MOVPE,

Metal-Organic Vapour-phase Epitaxy (MOVPE), is one method for growing nanowires. Its use was first demonstrated in the early 1990's by Hiruma et al.[9]

Once gold particles are deposited onto the substrate, the sample is placed within the crystal growth chamber (as shown in figure 2.1). The temperature is increased and growth material are introduced in vapour form (TMGa as a source of Gallium and Phosphine as a source of Phosphorus). The Gold particles become supersaturated (act as collectors for material) and crystallization takes place between the metal and substrate (Figure 2.2).[10][11]



Figure 2.1- Aixtron AIX 200/4, used for MOVPE growth of nanowires at Lund Nanolab (picture courtesy of www.nano.lth.se)

The diameter of the wire is determined by the diameter of the deposited Gold particle. Zincblende nanowires grown using MOVPE have either a zincblend crystalline structure or a wurtzite crystal structure, or a combination of the two.[10][11]

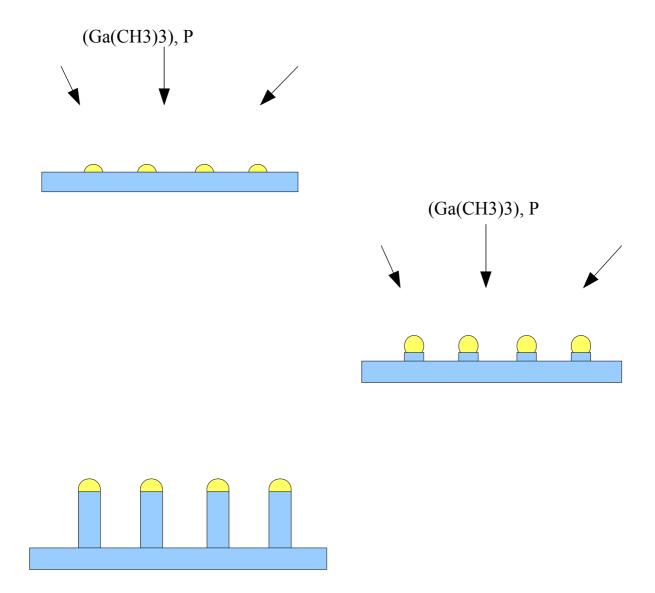


Figure 2.2- nanowire growth takes place when substrate with deposited Au is placed in a growth chamber. The introduction of the precursors (trimethylgallium along with phosphine in the case of GaP growth) and heat trigger nucleation and nanowire growth.

2B: NIL procedure,

In order to achieve nanowire growth on the substrate, Gold particles need to be deposited to act as growth catalysts. As previously mentioned in section 1, it is desirable to achieve an ordered arrangement of gold particles (which in turn would lead to an ordered array of nanowires).

The lithography method frequently used for this purpose has been EBL however, Nano imprint lithography is an alternative lithography method with higher throughput and lower running cost in comparison.

Use of NIL was first published by Stephen Y. Chou, Peter R. Krauss and Preston J. Renstorm in 1996 [3]. Briefly, NIL is simply imprinting a desired pattern into resist using a stamp, where pressure and heat may assist the process (especially in the case of thermal NIL). NIL is an ideal alternative to EBL. Its usage enables mass production of structures, currently only available for research through EBL. NIL brings nano fabrication a step closer to being commercially viable.

The NIL method used here was thermal NIL. In this method, which can be seen in step 1 of Figure 2.3, the resist is heated to a temperature above the glass transition temperature. The design is imprinted into the resist by using pressure and temperature.

In order to perform imprint, a stamp needs to be fabricated for use in NIL. This stamp has a base of Si/SiO2 and its fabrication is explained in part 2C (for reference please look at Figure 2.4).

In order to avoid damaging the stamp, or breaking the substrate during the imprint when pressure is applied, the stamp and the substrate should have a similar size and shape. This was not possible in this project due to shortage of GaP at the time of this particular experiment.

The stamp and substrate are placed on top of one another facing each other, and the temperature is raised above the glass transition temperature, followed by application of pressure to the stamp and substrate.

The resist flows and so the imprint is made, and then the system is cooled down. This is followed by the removal of any possible residues of PMMA left behind on the imprinted structure using oxygen plasma ashing.

Once the PMMA has been removed, MF319 1:1 is used to wet etch the LOR layer, and the residual LOR is removed using oxygen plasma ashing to ensure that no contamination exists between the Gold and GaP in the following step.

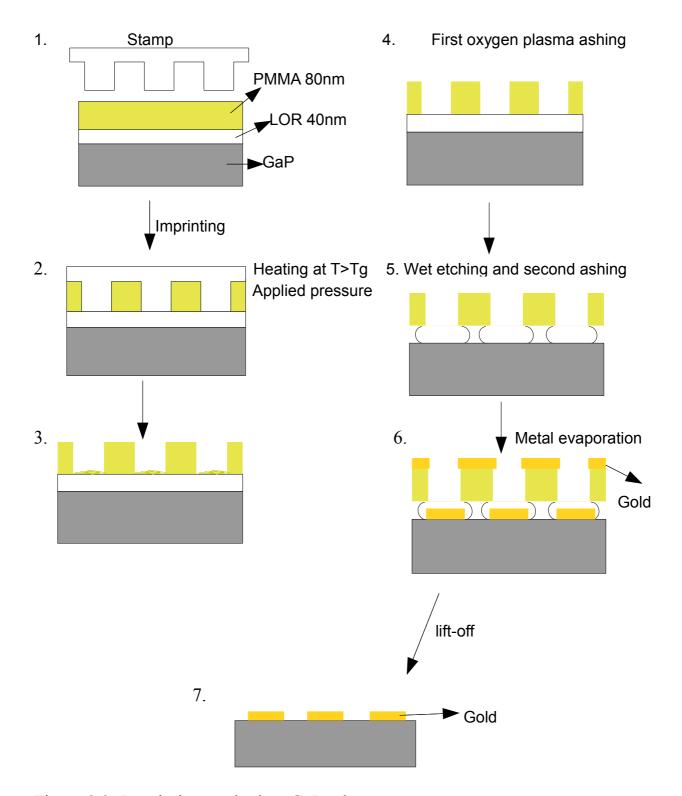


Figure 2.3- Imprinting method on GaP substrate.

2C: Stamp preparation method,

The first step in producing the desired gold pattern for NIL, is the NIL stamp fabrication. This stamp, a Si/SiO₂ substrate, will consist of a hexagonal array of dots, a pattern which will be transferred to the GaP using heat and pressure during NIL. In order to transfer such a pattern using NIL a stamp with this pattern must be fabricated first. Figure 2.4 shows how the stamp is fabricated.

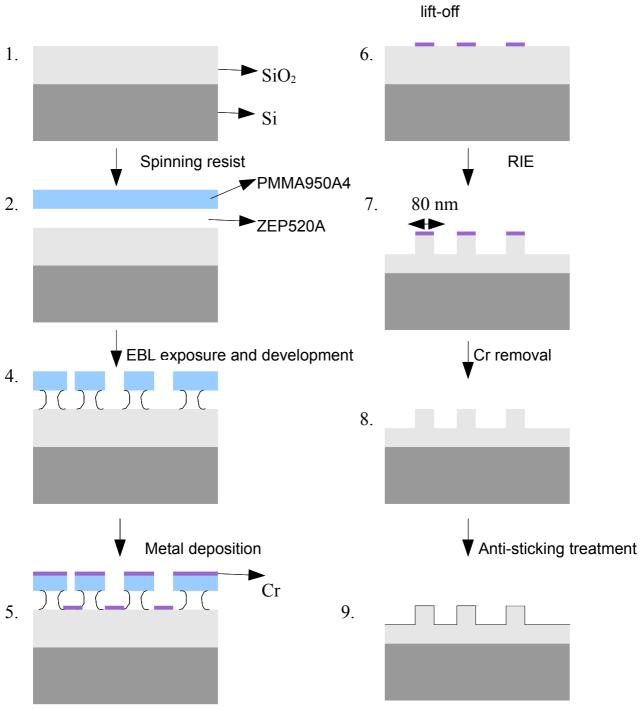


Figure 2.4: Fabrication steps of NIL stamp.

The stamp is a Si substrate, with an upper SiO₂ layer of approximately 300nm in thickness. A double layer of resist is spun on the sample, first ZEP520A and then PMMA950A4. The parameters of both are shown in section 3.

A design of hexagonally arranged dots is subsequently produced using Electron Beam Lithography (EBL) and developed in Isobutyl methyl ketone (MIBK), after which the entire sample is covered in Chromium (Cr). Lift-off is performed using warm acetone, after which only Chromium dots of approximately 80nm in diameter are remaining.

The next step is dry etching performed using the Reactive Ion etcher (RIE). Area's covered by the Chromium are protected from the etchant gases (they do not react with Chromium) while the exposed areas are etched, so by controlling the gas type, pressure, power and etching time, one may etch the sample to the desired height profile.

The gases used for this purpose were CHF_3 and O_2 . The full recipe parameters for the Trion etch can be found in section 3 and appendix A.

After the dry etching procedure, wet etching was performed in order to remove the Chromium. This is done due to the poor adhesion between Cr and F_{13} -TCS, the antisticking chemical that is applied.

The anti-adhesion chemical used for the anti-sticking procedure is known as tridecafluoro-(1,1,2,2)-tetrahydrooctyl-trichlorosilane (F_{13} -TCS). Chlorosilanes are known to react with Si/SiO₂(Figure 2.2).[8]

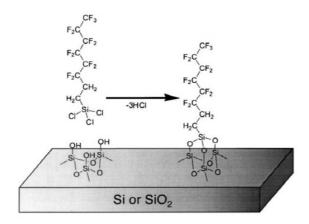


Figure 2.5-Reaction between F₁₃-TCS and Si/SiO2 surfaces. Silane connects covalently to the substrate. [8]

The procedure used is a vapour phase deposition method, since by using liquid phase deposition for nano structures, one runs the risk of insufficient wetting of the hydrophobic areas on a hydrophilic stamp (Figure 2.6). The procedure takes place in a glove box (nitrogen atmosphere, water content <1 ppm), as it is important that this procedure is performed in a water free environment.

The hot plate was heated to 250° (above boiling temperature for silane, which is 192° C). F_{13} -TCS is injected into a petri dish through a hole which must be covered with haste (amount may vary depending on the size of the petri dish). The sample is left on a hot plate for 2 hours, and so when the gaseous molecules come into contact with the stamp surface they immediately form covalent links. After the stamp is removed from the hot plate, it is washed in anhydrous hexane (solvent for silane molecules) so that the excess F_{13} -TCS can be washed away, and then blow dried with nitrogen.[8]

The anti-adhesion layer is of great importance since, without it, separating the Stamp and substrate after the NIL procedure would be tremendously difficult. It may even lead to the stamp being damaged, which would render this project pointless, as each stamp would have to be reusable a number of times for this method to be feasible.

Once the anti-sticking procedure is done, a working stamp is finished and ready to be used in the NIL procedure.



Figure 2.6- wetting defects occurring during liquid phase deposition.[8]

3. Experimental procedure:

3A: Stamp preparation,

The preparation for creating The stamp for use in NIL is as follows; Firstly a Si/SiO₂ sample (SiO₂ approximately 300nm) is used. ZEP520A and PMMA950A9 are spun on the sample at 9000 rpm and 6000 rpm respectively, and baked for 10 and 15 minutes at 160C respectively, before EBL is performed.[8]

Before Electron Beam lithography is performed on the Si/SiO₂ sample, a hexagonal array of dots is designed using the Raith 150 software, and sent to the EBL computer, after which E-beam lithography can be performed.

Originally, a dose 230 μ As/cm² was used in order to create dots of ~50 nm in diameter. However it was soon clear by trial and error that this dose was rather unstable, with no definite chance of success in the creation of the hexagonal array pattern on the stamp. Due to this the dose was increased to 560 μ As/cm², which was stable. However due to this increase in dose, the dot diameter size also increased from approximately 50 nm to around 80nm.

After lithography, the sample was developed in MIBK for one minute, rinsed in Isopropanol (IPA) for about 30 seconds, and then developed in Oxylen for 2 minutes and again rinsed in IPA for 30 seconds before it was subsequently blown dry with a nitrogen gun. Then using the Evaporator AVAC, 30 nm of Chromium (Cr) was deposited onto the sample.

After this a Lift off procedure was preformed by heating the sample to 40°C in Remover 1165, and then cleaning it in acetone and IPA, followed by blow drying the sample with a nitrogen gun.

This process left Cr behind on the exposed dots from the pattern only, as one may observe from figure 3.1. The next step was dry etching, in order to etch the surroundings of the dot structures, leaving behind pillars on the stamp. This was done using the Trion dry etcher.

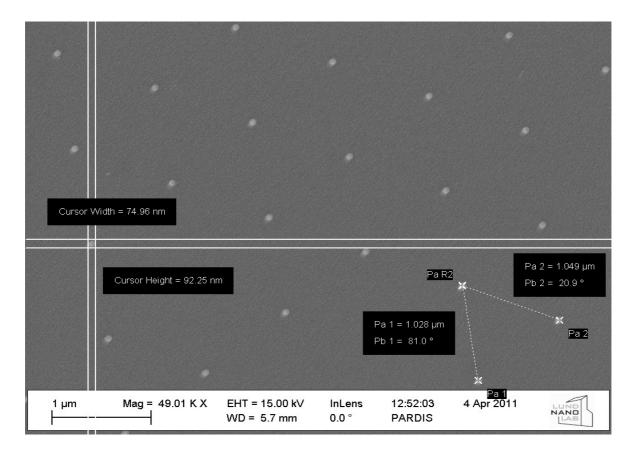


Figure 3.1: Dots after Evaporation, from stamp sample ST02.

At the beginning, going by article [6] it was decided to etch the dot to achieve a height of 180nm. This would have been achieved using gasses CHF₃ and O₂ in plasma etching with 16 and 2 SCCM amounts respectively. Table 1 below shows values used for this etching process.

CHF ₃	O_2	Pressure	RIE	Process time
16 SCCM	2 SCCM	90 mT	50 W	1500 s

Table 1- First recipe used in Trion dry etching for stamp production.

However, it was found that, as one may observe in Figure 3.2 (curtsey of Mariusz Graczyk, Lund nanolab) that there seemed to be a growth on the surface of the sample unrelated to the design. This growth was probably due to the chemical reaction of the sample and plasma over the long process time.

The exact nature of the growths is still unknown; however, what could be stated with great certainty was that these growths could cause problems in the future, as they would make it difficult to separate the stamp and the GaP sample after the NIL process and may even lead to parasitical growth of nanowires.

One way of reducing the chemical reaction leading to parasitical growth would have been to reduce the pressure at which the process took place; however, due to the structural design of the Trion this was not possible while there was gas flow.

The other option was to reduce the process time, although this would cause a reduction in the etched height. As one may observe from paper [12] the maximum height one may etch without the growth of the unwanted structures was 100nm.

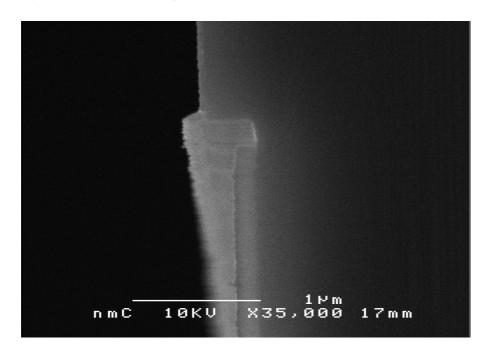


Figure 3.2- shows unintentional growths as a result of long etch times once etch depth surpasses 100nm. (Image taken by Mariusz Graczyk)

In order to use a stamp of such height there is need for certain adjustments to be made to the GaP sample during NIL. These adjustments will involve changing the resist thickness layers for the NIL substrate, and will be discussed and explained further in section 3B.

An over all summary of the final etching values can be found in table 2 below (to look at the detailed etching steps, please refer to appendix A).

CHF ₃	$O_{2.}$	Pressure	RIE	Process time
16 SCCM	2 SCCM	90 mT	50 W	420 s

Table 2- Amended Trion dry etching recipe for stamp production.

The above parameters gave the pillars a height of approximately 80 nm. Figure 3.3 shows the pillars after etch.

Once etched, the Cr should be removed from the tip of the now 80nm heigh dots. This is to ensure that the anti-sticking treatment works, as the anti-sticking chemical cannot stick to metals.

To achieve this, the stamp is placed in Cr etch for about 5 minutes, and then rinsed in running DI water. The first attempt at this did not remove the Cr fully, and despite increasing the time (all the way to 3 hours, as Cr etch does not harm the stamp itself) part of the Cr still remained persistently.

A conclusion was drawn that as a side affect of the dry etching, some fluro-carbons were left behind, covering the dots, which made it difficult for the Cr etch to reach the Cr. In order to conteract this problem, an extra step was added to the dry etch, where oxygen ashing was performed using parameters, shown in Table 3.

O_2	Pressure	RIE	Process Time
50 SCCM	250 mT	50 W	60 s

Table 3- Parameters used for Oxygen Prim in Trion.

This ensured that the fluorocarbons were removed, so that the Cr would be etched away fully. This attempt proved to be successful. As a result, the Cr etching time was reduced to 5 minutes again.

After Cr was successfully removed, the anti-sticking treatment was preformed to help ease the separation of the stamp and substrate after NIL .[7][8]

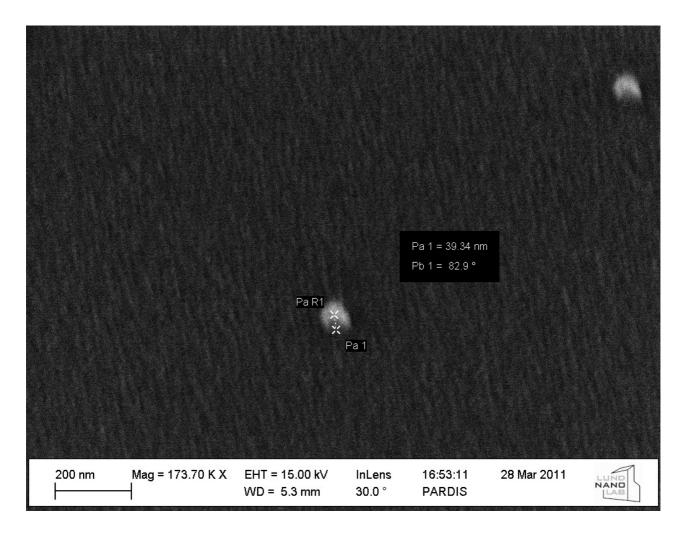


Figure 3.3: One of the dotes from Stamp sample ST14 after Dry etching. the sample is tilted at an 30° angle, height translates to approximately 80nm flat.

3B: NIL procedure,

Using the method explained previously, around 20 stamps were created, in case the stamps were broken during NIL, a possibility increased by the fact that the stamp and the GaP substrate did not have the same size (this was due to shortage of GaP at the time of the experiment). The pressure applied during NIL made it almost inevitable that the stamps or indeed the substrates themselves could break if utmost care was not taken in their placement on top of one another before NIL was performed.

Once the stamps were all prepared, the NIL procedure was performed. Initially the GaP substrate was cleaned using acetone in an ultrasonic bath. Once the substrate was sufficiently clean a double layer resist was spun on the surface.

Initially LORA0.7 3:2 was spun at 5000rpm, which gave thickness of 63nm (various speeds and dilutions were tested in order to achieve the most favourable thickness). The layer was then baked for 2 minutes on a hot plate at 180°C.

PMMA950A4 1:1 was spun on top, at 4000rpm, giving a thickness of 71nm (various speeds were tested, with this offering the closest value of ideal thickness.) This was baked for 90 seconds at 180°C on the hot plate. Step 1 in image 2.3 gives some idea as to how this step is performed.

As mentioned previously, the height of the resists should be relative to the height of the stamp. As the height was reduced to around 80nm, the PMMA layer needs to be slightly thinner. With PMMA4 1:1 in Anisol, spun with the parameters above, the height achieved was 71nm, which is ideal.

However the LOR layer must be approximately half of the height of the PMMA. To achieve this some variation of the LOR was tested and the heights were measured.

The results can be observed in Table 4. All the shown variations of LORA0.7 compositions in Cyclotene were spun at 5000rpm and baked for 2 minutes at 180°C.

LORA0.7 3:1	109nm
LORA0.7 3:2	63nm
LORA0.7 1:1	56nm

Table 4- Varies dilutions of LORA0.7 in Cyclotene, tested for height adjustment.

The best dilution to use, from observing the table above was the LORA0.7 1:1 dilution. Once the optimum parameters for the resists were determined, the NIL

procedure was preformed. The parameters for the NIL where taken from [6] and were prove to be the optimum values for imprinting dots. These values can be seen below in Table 5.

Pressure (Bar)	50
Temperature (C)	180
Imprint time (s)	180

Table 5- optimum imprint parameters for dots on PMMA, during NIL.

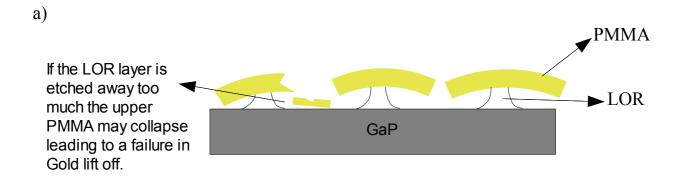
After each imprint, there was one ashing process to free the sample from the remainder of the PMMA left behind from the imprint. Then wet etching was performed in MF319 1:1 to clear LOR, followed by another attempt at ashing to insure there is no LOR left.

The ashing rates and etching rate are of great importance, and getting the timing correct is vital in order to produce nanowires.

Should the first ashing be too little, the PMMA would not be sufficiently removed, and the MF319 would not be able to penetrate to the LOR layer and etch it. However too much ashing would result in widening of the gap, which would lead to bigger nanowires than anticipated.

The MF319 etching and the second ashing are also vital, as not enough etching and ashing would lead to contamination between the Gold layer and the substrate, which may obstruct the nucleation and growth of nano wires during MOVPE.

However, too much of the etch and ash may lead to the widening of the gap resulting in an increase in diameter of the nanowires. In the worse case it may even lead to lift off failure, as too much under etching can lead to the PMMA layer collapsing. The two cases of possible failure are illustrated in figure 3.4.



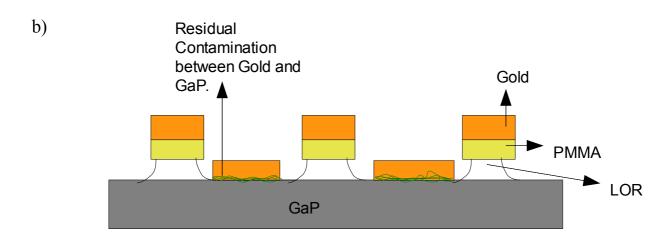


Figure 3.4:a) illustrating the possibility of the PMMA layer collapsing if the LOR layer is etched away far too much. This would result in a Gold lift off failure as double layer resist is needed for lift off. b) showing the possibility of having residual contamination from LOR layer trapped between the Gold and GaP, which may result in growth failure.

Once the sample has been ashed and etched, around 20nm of Gold is deposited onto the sample using the AVAC evaporator. Lift off is performed by placing the substrate in acetone on a hot plate at 80°C for 60 minutes.

Subsequently after lift off has been performed, a hexagonal array of Gold dots is left on the surface. The sample is now ready for nanowire growth (section 2A).

4. Results and analysis:

The list of various samples and the parameters used in ashing and etching them are shown below in table 6. The ashing refers to the oxygen plasma ashing performed, and the cage refers to the Faraday cage that could be used during this process optionally. The use of the Faraday cage reduces the thickness of resist etched away. Ashing is performed at 2 separate points, once directly after imprint to remove any PMMA resist not removed from the imprinted area, and another time after wet etching the LOR resist, in order to remove any LOR resist that may be left behind.

Sample names	WW01	WW02	IW01	IW02	NI01	NI02
First ashing time (s) (with or without Faraday cage)	45 (cage)	45(no cage)	60 (cage)	44 (cage)	45 (cage)	45(cage)
Etching time (s)	30	40	50	50	30	40
Second ashing time(s)	15 (cage)	15(no cage)	30 (cage)	30 (cage)	15 (cage)	15 (cage)
Stamp used	ST05	ST04	ST07	ST07	ST09	ST09
result	No growth	Growth	No lift-off	No lift-off	Complicated lift-off	Difficulty with lift-off

Table 6-wet Etching and oxygen plasma ashing parameters used for significant samples.

Then they were handed over to the epitaxy group and after MOVPE inspected under the optical microscope and SEM (scanning electron microscope).

The most successful sample was sample WW02, for which the hexagonal array dots grew into nanowires successfully. Images 4.1-4.4 show the nanowires grown on sample WW02.

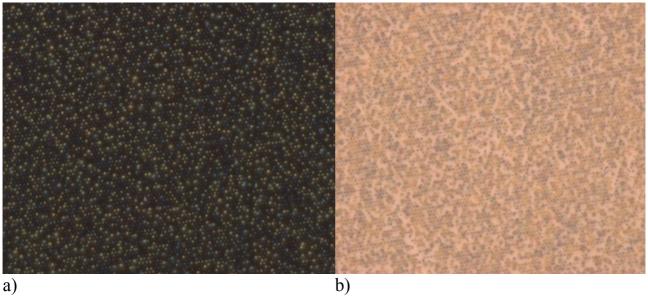


Image 4.1: Optical images from sample WW02 showing the hexagonal array of wires in a) dark field, b) light field.

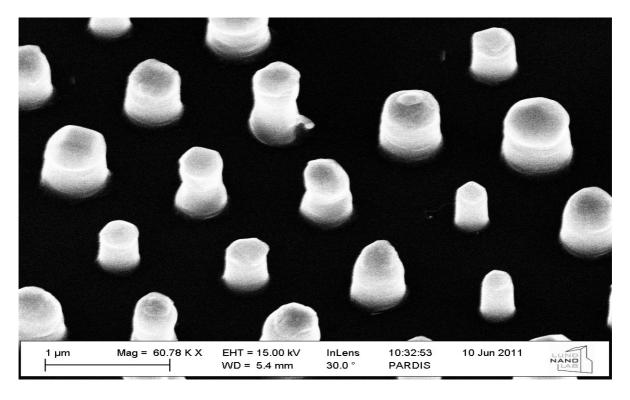


Image 4.2: hexagonal Array of nano-wires in sample WW02.

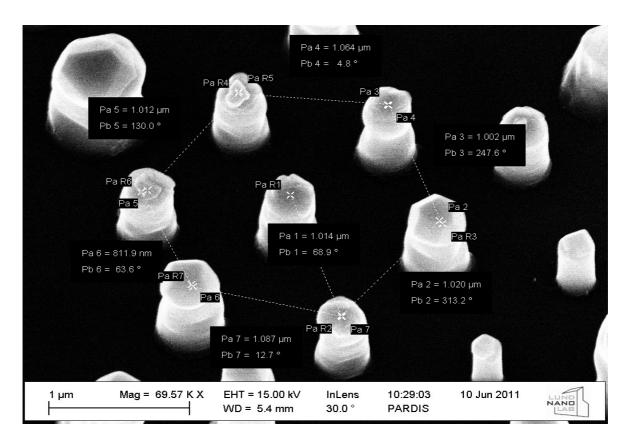


Image 4.3: sample WW02, all wires are 1µm away from their nearest neighbour.

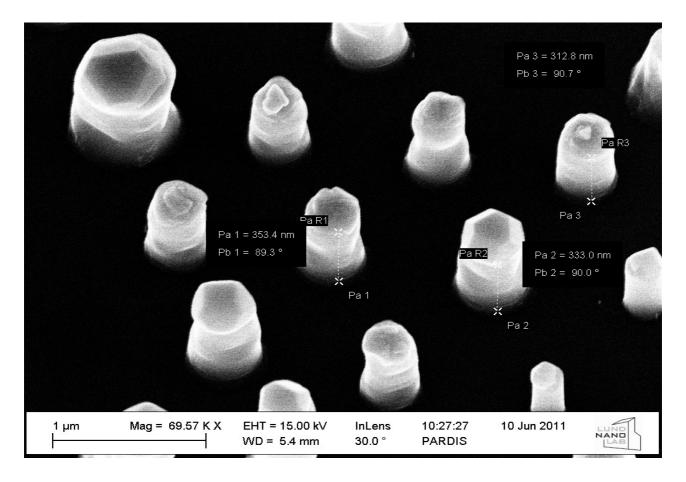


Image 4.4: sample WW02, the height of the nano-wires is measured to be ~300nm.

The wires have held their arranged structure well, and there are no bush growths (many wires sticking out from the same spot), and almost non-existent side growths (wires grown from the side of other wires). However, the diameter of the wires does not seem quite constant, and the average diameter of about 400nm, its four times larger than intended.

This is most likely due to two factors. One contributing factor for the increase in size would be gap widening due to excessive etching and ashing. The variation in size was most likely due to difficulty during gold lift off. Further investigation is needed to overcome these problems.

The experiment can be deemed as a partially successful project.

5. Conclusion and Further development:

The aim of this project was to create nanowires using NIL method in order to increase through put of samples and so, take a step closer to making nanotechnology commercially viable.

In essence, the project was successful in creating an array of Gold dots which were successfully used as catalysts during MOVPE for the growth of nanowires. However, there were some problems. The size of the grown nanowires were larger than intended. This is most likely due to gap widening after NIL, produced from excessive ashing and etching.

Also, although many wires had a standard size of approximately 400nm in diameter, variations could be observed. This was due to problems during gold lift off.

Many problems were faced during the process of this project, from shortage of vital substrates to problems with vital equipment and microscope. Some were unavoidable, however there is room for improvement, and need for someone to perform further investigation into this experiment.

Through some repetition it can be confirmed that this growth method is stable, and one may find solutions to the problems faced. However this will not be possible in this project due to lack of time. Most importantly, what has been demonstrated is that there is a clear possibility for NIL to replace EBL for lithography of arranged nanowires, but there is simply need for some more calibrations and validations.

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Appendix A,

Table A1 below shows what each step in the Trion dry etching consisted of. Steps 5 and 6 were introduced for Oxygen ashing the surface, as mentioned in section 3 of this report.

The etch rate for SiO₂ at the parameters given below for pressure, RIE and the chosen gases was found to be 12nm per minute (around 62nm for 5 minutes), please refer to the document [7], curtsey of Mariusz Graczyk.

	Pressure (mT)	RIE (W)	Time (S)	O ₂ (SCCM)	CHF ₃ (SCCM)
Step 1	20	0	30	0	0
Step 2	90	0	45	2	16
Step 3	90	50	420	2	16
Step 4	20	0	60	0	0
Step 5	250	0	45	50	0
Step 6	250	50	60	50	0
Step 7	20	0	60	0	0
Step 8	500	0	180	50	0
Step 9	20	0	120	0	0

Table A1-Procedure steps used for dry etching Si/SiO₂ Stamp in the Trion etcher.

Appendix B,

Electron Beam Lithography (EBL), is a method of nanopatterning whereby electrons emitted by an electron source are used to pattern resist on a substrate for a variety of applications.

This method is quite desirable as it can produce nano scale patterns without the use of a mask and it has great precision and depth of focus in comparison to other contending lithography methods. It is useful for research; however, low throughput and expensive running costs make it a bit of luxury that industry usually cannot afford.

A schematic of an EBL can be seen in Figure B1.

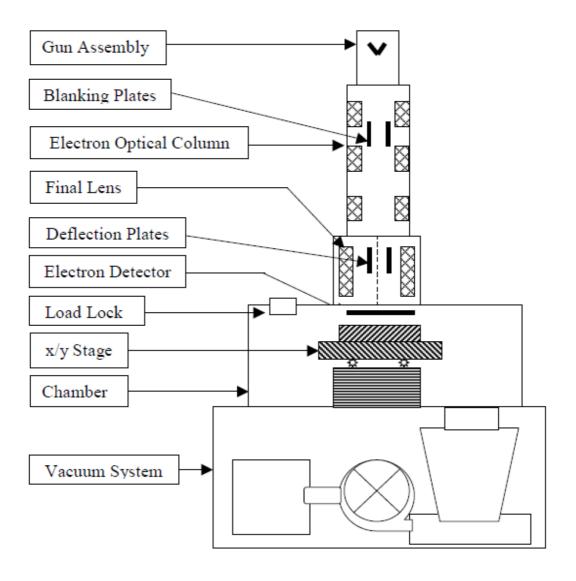


Figure B1: A basic schematic of a generic EBL machine.[13]

The condenser lens is used to focus the electron beam, and deflection coils help beam to be directed to anywhere on the surface of the wafer. The blanking plates are used to blank the beam when required, so that desired surfaces are exposed whilst the rest remains intact.

There are many electron sensitive resists, polymers such as PMMA (a positive resist), or HSQ (negative resist). Positive resists are resists where the exposed areas have the polymers break down and wash off to expose a pattern, were as negative resists are the exact opposite (exposed area cross links and remains whilst the rest of the resist is washed away). Positive resists are more often used. [8]

The beam dose (μ As/cm²) needed to expose a certain pattern on desired resist is calculated as a function of beam current I_{beam} , dwell time T_{dwell} and exposure area S², as shown below in Equation B.1,

Equation B.1;

$$Dose = \frac{(I_{beam}. T_{dwell})}{S^2}$$

The EBL used at Lund nanolab is the Raith 150, which can be seen in Figure B2.



Figure B2: EBL at the Lund nanolab, Raith 150 (picture courtesy of www.nano.lth.se)