FFF042 Project
Top-Down Fabrication and Characterization of InGaAs Nanowires

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1 Introduction

Top-down processes for fabrication of FinFET transistors are commonplace in the semiconductor fabrication industry. In this project, InGaAs nanowires will be manufactured by etching of an InGaAs layer grown on an InP substrate. Subsequently, metal contact leads will be deposited over the wires and its resistance will be measured. We will also measure the leakage currents of the substrate. Due to its high electron mobility, InGaAs is a promising material for use in modern transistors.

2 Fabrication Process

2.1 Initial Sample

The samples with which the processing in this project started off, consist of a InP substrate on top of which an InGaAs layer of 16 nm thickness has been epitaxially grown. For the amount of different nanowires, fabricated in the following, a small platelet of this substrate (approx 1×1 cm) was sufficient. The reason an InP substrate is used with a layer of InGaAs, is that they have similar lattice constants, so no defects appear. InP also has a higher bandgap, which will reduce the leakage current in the sample.

2.2 Spin-coating With Resist

In this first stage of the fabrication process, we coated the surface of the sample with e-beam resist. The technique used within this project is called "spin-coating", which uses centrifugal forces to uniformly dispense the previously deposited resist. For this, we mounted the sample in a spin-coater, which is rotating the sample around its surface's normal-axis resulting in the intended, uniform covering.

However, before any resist can be applied to the platelet it is necessary to remove all impurities from its surface, since they can cause flaws in the final resist coating. We therefore placed the sample in a bath of Aceton for 5 min followed by 2 min bath in isopropanol[1] (IPA). After the substrate has been dried with a nitrogen gun, we placed it on hotplate for 5 min at 200 °C in order to evaporate any remaining drops of liquid from the surface. Otherwise these would be incorporated in and under the e-beam resist film and would thereby reduce the accuracy of the resulting masking.

Following, we mounted the sample in the spin coater, where it is held in place by a vacuum pump at the platelets bottom side. At 3000 rpm for 60 seconds the e-beam resist, which we sprinkled on the substrate's surface with a pipette before, is uniformly distributed and forms a film with the aimed thickness of ~ 100 nm. Its compounds are MIBK[2] and HSQ[3] (3 : 1), which forms SiO_2 after the curing with heat. This circumstance makes it easier to perform the selective etching later on (see section 2.5).

2.3 Lithography of Nanowires

With the sample, being coated with e-beam resist, we used Electron Beam Lithography (EBL) to define the nanowire structures. During the EBL a beam of electrons is accelerated towards the sample's surface, where it interacts with the resist layer creating cross-links. Areas that have been exposed by the electron beam

[1] Isopropanyl alcohol
[2] Methyl Isobutyl Ketone
[3] Hydrogen Silsesquioxane

(a) Horizontal wires

(b) Vertical wires

Figure 1

therefore become less soluble when being placed in a solution of resist developer, hence the classification as "negative resist". We operated the EBL machine in "low current mode", which corresponds to a range of comparable low electron dose in the beam. Before starting the measurements, the EBL needs to be calibrated regarding the astigmatism (focus orthogonal to the beam), as well as the position calibration (accuracy of beam deflection relative to position the aimed spot on the surface). We exposed 5×10 wire arrays, where each array is named by a letter (A to E) for the vertical coordinate, and a number (1 to 10) for the horizontal coordinate. Each array contains 50 nanowires with dosage varying from 2.0 to 3.8, and size varying from 5 nm to 100 nm, see Figure 1a and 1b. Each even number has vertical wires, each odd number horizontal wires.

2.4 Development of Resist

After the lithography of the nanowire structres, we started developing the resist. The part which has been exposed in the EBL will stay on the sample (since it's hardened), the rest will be dissolved in the liquid. In the procedure, we started by putting the sample in TMAH[4] for 90 seconds, then 10 seconds in H_2O followed by 60 seconds in H_2O. Lastly the sample was placed in IPA for 10 seconds, followed by 30 seconds in a different beaker with IPA. We then took out the sample and blew-dry the sample with a nitrogen gun. Using IPA after H_2O has the advantage that it is easier to rid of than H_2O. We would have to blow harder with H_2O, but that would create a risk of moving the HSQ resist.

2.4.1 First SEM Analysis

When the development was completed, we took a first look at our sample with a scanning electron microscope (SEM). We started by looking at array E2 which has vertical aligned wires. The first thing we noticed is that all the 5 nm wires are gone. Also the 10 nm wires with dosage 3.0 and below have disappeared. The cause for this could be that a 5 nm wire with a 100 nm resist-layer would need a very careful treatment, since it is fifty times as high as width. Other causes could be that the dosage wasn't enough to really hit the resist till the bottom.

At array E3 with horizontal wires, the aimed size in the EBL is more in accordance with the actual size. Also here the 5 nm wires are all gone, but the 10 nm wires have survived more. Apparently there is something that causes a smearing/broadening distortion in the SEM images.

2.5 Etching

The etching of the InGaAs layer was done by a solution with the following ratio: $H_2O_2 : H_3PO_4 : H_2O = 1 : 1 : 25$. The etching rate of this solution is $\sim 1\ nm\ s^{-1}$ in the z-direction, and we etched for 16 seconds. This way the InGaAs not covered by the resist is etched away completely. We chose the etchant so that the InP substrate remained unharmed. Afterwards we put the sample in a bath of H_2O to stop the etching process, followed by a bath of IPA.

[4]Tetramethylammonium hydroxide

2.5.1 Second SEM Analysis

We took a look at our sample again after the etching process. We noticed that we could get a much higher resolution and no horizontal smear, vertical structures have expected size. This would confirm the hypothesis of the horizontal smear-out in the first analysis.

2.6 Removal of Resist

We then removed the resist from the InGaAs wires, so that we ended up with InGaAs wires on a InP substrate. The sample was put in a beaker with HF:H_2O = 1:10 for 90 seconds, where-after we rinsed it with water and dried it with the nitrogen gun to provide physical removal of resist and HF / H_2O. HF is used since it will etch the oxide SiO_2 that is on top of the wires. (HF is commonly used to etch oxides.)

2.7 Lithography of Contacts

To be able to perform measurements on the nanowires, contacts to them need to be made. Before we started with designing the contacts, we spin-coated the substrate with a PMMA[5] positive resist on 4000 rpm for 45 seconds and we baked it for 2 minutes on 180°C. In the EBL, we started designing the contacts to the sample. The distance between the contacts placed onto the wire is held constant at approximately 1 μm.

Table 1: Placed contacts per wire array. d = dosage, t = thickness, h/v = horizontal/vertical.

	1	**2**	**3**	**4**
E	d = 3.8, t = 100 nm, h	d = 3.8, t = 20 nm, v	d = 3.8, t = 100 nm, h	d = 3.8, t = 100 nm, v
D	d = 3.8, t = 100 nm, h	d = 3.8, t = 20 nm, v	d = 3.8, t = 100 nm, h	d = 3.8, t = 100 nm, v
C	d = 3.0, t = 100 nm, h	d = 2.0, t = 50 nm, v	d = 3.0, t = 100 nm, h	d = 3.0, t = 100 nm, v
B	d = 3.0, t = 100 nm, h	d = 2.0, t = 50 nm, v	d = 3.0, t = 100 nm, h	d = 3.0, t = 100 nm, v
A	d = 2.0, t = 100 nm, h	d = 3.8, t = 50 nm, v	d = 2.0, t = 100 nm, h	d = 2.0, t = 100 nm, v

2.8 Development and Plasma Ashing

We again developed the resist as before (now instead we are using a positive resist, which gets rid of the exposed parts) and this time we used a plasma asher afterwards. This machine uses a plasma to get rid of the risidual resist which settles as an impurity layer on the sample surface. Otherwise, the following metallisation would incorporate these remains. During the ashing, the molecules of the resist are reacting with ionized oxygen atoms in the plasma and turn into ash, which is then sucked away by the vacuum.

2.9 Metallisation

The next part is to actually deposit metal onto the sample. This is done by an evaporator, which evaporates metal by letting a very high current flow through it. We started with 100 Å Ti to get a good interface with the substrate, followed by 100 Å Pd to prevent the Au from diffusing into the substrate and then 800 Å Au to make a good contact, which gives in total a thickness of about 100 nm.

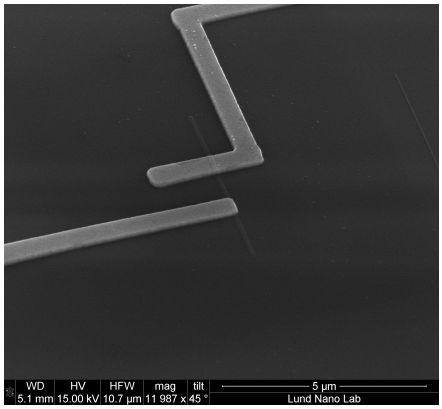
2.10 Lift-off

After the metallisation the sample was placed in a bath of Acetone to get the metal deposited on the resist off. It was placed in there for a full night. After we got rid of all the unwanted metal, we placed the sample under a microscope to check if all the connections were intact.

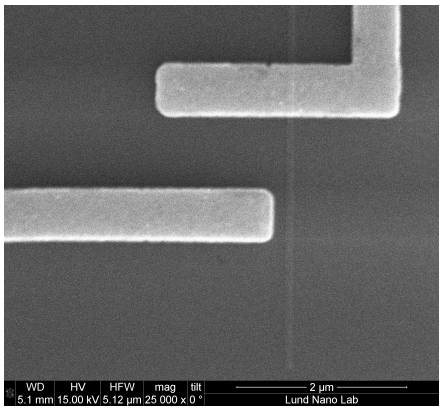
2.10.1 Third SEM Analysis

In the last SEM analysis we measured the widths of the connections and the thickness of the wires, to be used later in the data analysis. Since the metallisation gives a much higher contrast between substrate and metallised areas, it is much easier to detect the applied structures and focus the SEM. One of the structures is shown in Figure 2a, where one can see how much thinner the nanowires are compared to the metallic contacts. During the first EBL of the wires and the second one for the contacts, a set of geometric marks is generated on the samples surface as well. These so-called "alignment marks" act as reference points to

[5]Polymethyl methacrylate



(a) Vertical nanowire with contacts



(b) Missing contact due to miss-alignment

Figure 2: A vertical nanowire (slim feature centre) with metal leads (bright gray L-shapes) connected to it in (a). The image depicts a slightly tilted (45°) and rotated perspective of the structure. The nano wire causes the deposited metal to bulk above it, which generates a slim dent on the contacts. In (b), one can see a miss-matched contact with no connection between metal and wire.

achieve a well aligned match between wires and metal leads. Because this process is rather sensitive, thus difficult, it is possible to achieve a slight miss-alignment, which ends up with a disjunction (see Figure 2b).

2.11 Measuring

The completed sample was being measured for the resistances using a probe station. In the measurement we found out that there was a huge leakage current, of the same order as the nanowire current, see Table 3, Figure 3.

2.12 HCl etch

A solution to get rid of the large leakage current was to etch away the top-most layer of InP, that was suspected to be unintentionally doped. We used a $1:3$ HCl[6]:H_{20} solution to etch for 3 seconds, since it etches InP well, but not InGaAs. The dilution and short etch time was chosen due to the risk of etching away the InP underneath the wires, possibly breaking them in the process. Afterwards the leakage current was drastically decreased, as seen in the results of section 3.2.

3 Measurements

3.1 Nanowire Width

After the etching of the InGaAs layer, the width of the fabricated wires (excluding the ones that completely disappeared) was measured, see Table 2. For all wires, a higher EBL dose resulted in a higher ratio of measured width to design width. We can also see that the vertical wires retained more of the design width, meaning the etching was faster vertically than horizontally. On the horizontal wires, roughly 50-60 nm (approx. 2 nm/s) was etched away which completely removed the wires smaller than 50 nm. This etch rate is faster than the etch rate down into the sample (approx. 1 nm/s).

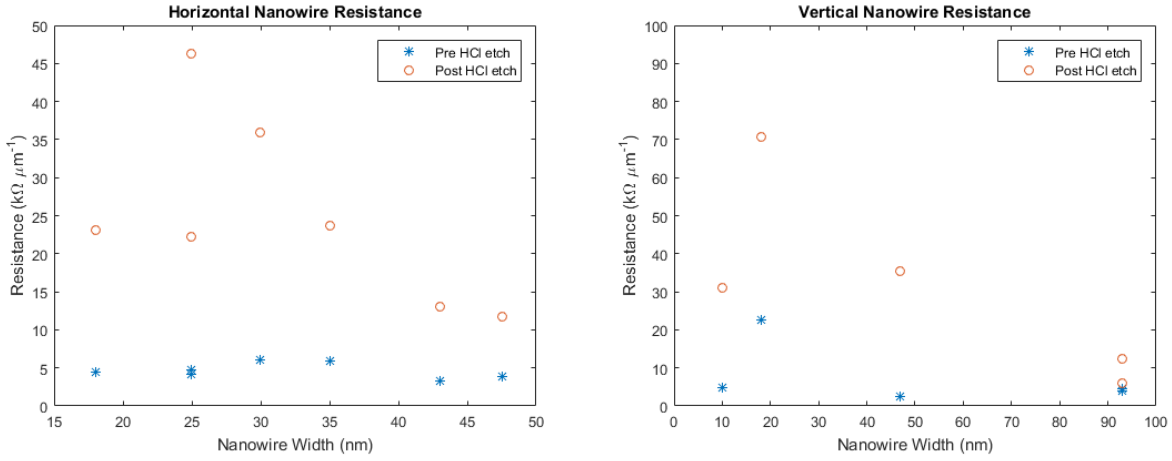
Table 2: Measured Nanowire Width as Ratio of Design Width

EBL Dose	Wire Orientation	Average Width Ratio
2.0	Vertical	0.47
3.8	Vertical	0.74
3.0	Horizontal	0.23
3.8	Horizontal	0.39

[6]Hydrogen Chloride

3.2 Nanowire Resistance

The resistance of the nanowires was measured before and after the etching with HCl, see Figure 3. For all wires, the resistance increased after the etching. One would expect the resistance to increase with reduced nanowire width, but this was not observed before etching. After etching the resistance seems width-dependant, but there are also big variations in resistance for nanowires of the same width.



(a) (b)

Figure 3: Measured resistance of the horizontal (a) and vertical (b) InGaAs nanowires as function of nanowire width, before and after HCl etching.

The leakage current between contact pads in all corners of the chip was measured, and the resistances between them calculated, see Table 3. For all measured locations, the leakage current was reduced by more than a factor 10 after HCl etching.

Table 3: Calculated Resistance Between Contact Pads

Location	Pre HCl etch (kΩ)	Post HCl etch (kΩ)
Bottom Left	16.7	182
Bottom Right	13.0	330
Upper Left	12.5	164
Upper Right	10.7	320

4 Conclusions

InGaAs nanowires were successfully fabricated using the process in Section 2. However, we encountered some difficulties regarding the wire width and leakage currents.

4.1 Nanowire Width and Etching

As seen in Table 2, the measured widths of the wires differed substantially from the design widths due to complications with lateral etching. The next step would be to change the crystal orientation of the InGaAs layer to reduce the lateral etching. By doing this it might be possible to improve the width ratios and also allow for more narrow wires.

4.2 Leakage Currents

Before the HCl etch (see Table 3), the leakage current was of the same order of magnitude as the current through the wire (see Figure 3), which is bad since the measurements are then just as much a characterisation of the substrate as the wires we are interested in. The leakage current was vastly reduced by etching the top layer with HCl. The EBL patterns used for the contact layer were not symmetric, they had varying amounts of overlap from 0.945 µm to 1.879 µm, resulting in different amounts of leakage for different wires. For characterisation of the wire properties, this is also something that should be improved.