Developing an Ar milling process to improve the contact quality to InAs nanowires

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Contents

Abstract

The aim of this work was to develop a stable and reproducible argon milling process for InAs nanowires to remove the native oxide layer that increases electrical resistance. This was done by identifying a few milling parameters and studying them in relation to the milling rate of silicon dioxide (SiO_2). After further experiments with different milling parameters, a set of parameter values was found to give a milling rate of about 6-8 nm/min. The milling rate of Polymethyl methacrylate (PMMA) for the same set of parameter values was 60-80 nm/min which is 10 times more than that for SiO_2. PMMA is used during device fabrication and was exposed to the Ar^+ beam, thus it's important to compare it to the milling rate of SiO_2. Finally, argon milling was done to a set of nanowires, and the contact resistance to Ti/Al metal was calculated. Two-probe and four-probe measurements were done to the set of InAs nanowires and the data obtained was used to calculate the contact resistance. The contact resistance was found to be around 90 Ω which is relatively small compared to the internal resistance of InAs nanowires which is around 1.5-3 kΩ. For reference, the contact resistance for a set of nanowires that were not treated with argon milling was measured and was found to be around 1 MΩ. Hence argon milling is a critical process that can reduce contact resistance by a significant amount. Finally, the milling rate of InAs nanowires was calculated to be about 25 nm/min for the same set of parameters used for SiO_2 and PMMA. This is a relatively high milling rate with respect to the thickness of the oxide layer in InAs nanowires, to get a lower milling rate one would need to lower the acceleration voltage and/or reduce the amount of time of exposure of the sample to the Ar^+ beam.

Acknowledgement

Chapter 1

Introduction

In the last few decades, technological advancement took a major leap as most of the common devices and appliances got smaller, faster, and more efficient. This is a result of our increased understanding of the intrinsic properties of semiconductors at very small scales which are the building blocks of our current technology. Nanowires are semiconductors with very small diameters (nanometer, 10^{-9} m) and a length of about a micron that are grown in a special laboratory environment. Due to the small size and the properties they possess as semiconductors, nanowires have been used in scientific research in many fields including thermometry, manufacturing of transistors, nanowire lasers, and solar cells[3, 4, 6]. In chemistry and biology, protein nanowires are used as a sensor, injection devices, and model systems for toxicity studies.[1]

In spite of all the success of nanowires, they do come with challenges, especially during device fabrication. One of the challenges is getting rid of a native oxide layer on the outer surface of the nanowires that appears when in contact with air[8]. This changes the electrical properties of the nanowires significantly. The oxide layer act as a barrier between the contact metal and the nanowires, increasing the electrical resistance. Hence it is of great importance to make sure that the oxide layer is removed from the nanowires as precisely as possible. Precision is the key point here, too much and we are likely to remove parts of the nanowires, too little and we still have part of the oxide layer left. In both cases, the electrical resistance will be high, due to the decreased cross-sectional area of the nanowires in the former case, and due to the presence of the oxide layer left behind in the latter.

Different methods have been explored in hopes to treat nanowires from the readily formed oxide layers. The most common method is the use of wet chemi-

cals such as hydrochloric acid (HCl) and hydrofluoric acid (HF) which react with the oxide layer, dissolving it in the process[5]. The amount of oxide dissolved depends on the concentration and duration of exposure to the chemicals. Highly concentrated solutions will have a large effect which may increase the chances of eroding the nanowires, while low concentrations might leave behind some traces of the oxide layer on the nanowire. Thus the optimal concentration of the chemical solutions should be used in order to get the best results. This process is referred to as the wet etching process because of the use of chemicals in a liquid state to remove the native oxide.

An alternative method is the argon milling process, where argon ions (Ar^+) extracted from the plasma, are accelerated and bombarded on the nanowires, mechanically removing the oxide layer. Argon is an inert gas thus the process is purely kinetic and no chemical reactions can take place. The argon milling process is not quite a stable process in comparison to the wet etching process since it includes a number of parameters that have to be set at precise values for optimal milling rates. Parameter values such as ion energy, argon pressure, and the position where the samples are placed with respect to the Ar^+ beam have a large impact on the amount of oxide layer removed as they directly affect the milling rate. Hence it is important to understand the effects of various parameters we have at hand and how to better control them for optimal milling.

The aim of this work is to establish a stable and reproducible argon milling process by finding an optimum milling rate for the native oxide layers on InAs nanowires. This is done by first identifying the important parameters/variables that could potentially have an effect on the milling rate. The effect of each parameter was then studied independently, keeping other parameters constant/fixed, until a suitable milling rate is obtained for a given set of parameter values. To test how good the argon milling process is, electrical measurements are performed on a set of InAs nanowires before and after milling. Both two-probe and four-probe measurements are conducted and the contact resistance is calculated from the measured electric currents and voltages.

Chapter 2

Experimental details

As a way to save resources in determining the optimal milling rate for InAs nanowires, experiments were performed on a set of small rectangular silicon samples with a thin film of silicon dioxide (SiO_2). Since it is much easier to characterize film thickness than the thickness of the oxide layer on InAs nanowires. A total of five parameters were identified as potential candidates that could affect the milling rate in one form or another. These include the pressure of argon gas, the extraction voltage, the ion energy, beam exposure time, and the placement of the samples with respect to the Ar^+ beam. We generally searched for the settings that gave a milling rate of a few nm/min as the amount of oxide layer on InAs nanowires is estimated to be 3-4 nm.

The milling rate for Polymethylmethacrylate (PMMA) resist was determined using the same set of parameter values. This is important because PMMA has different chemical and physical properties to the oxide layer thus different milling rates. PMMA is used to define the areas/pathways where the oxide on nanowires should be removed and metal contacts deposited, hence it is important to determine its milling rate. Finally, contact design to InAs nanowires was done using the electron beam lithography (EBL) followed by argon milling, metal deposition, and lift-off process leaving behind nanowires with proper metallic contacts on them, ready for electrical measurements. The experimental details for each step are thoroughly explained below.

2.1   Ar milling of SiO$_2$ and PMMA

Argon milling and metal evaporation for contacts was carried out inside a tool for electron-beam assisted metal film deposition ("Temescal"), which provides good vacuum conditions for milling experiments while also having full control over the parameters that determine milling rates such as argon pressure, extraction voltage, and ion energy.

Argon ions are extracted from a plasma that is created by microwaves with a frequency of 2.45 GHz generated by a microwave magnetron. As the microwaves are absorbed by the gas, the valence electrons get ejected from the neutral Ar atom which can further be accelerated to knock off more electrons leading to a cascading event where more Ar$^+$ ions are created. The plasma is confined in a chamber by a magnetic quadrupole arranged around the discharge chamber.[9] The plasma is left to stabilize for 2-5 minutes before an extractor voltage is applied which separates positive ions from the electrons. The positive argon ions are now ready to be accelerated by applying an electric field toward the samples placed in a vacuum chamber.

Small rectangular samples of silicon dioxide (SiO$_2$) with dimensions of about 1cm by 0.5 cm were half covered before argon milling experiments, this enables proper characterization of the film thickness of SiO$_2$ before and after milling. With the samples mounted in the vacuum chamber of the Temescal, the effect of different parameters on the milling rate of SiO$_2$ was studied. The first parameter to be investigated was the ion energy (accelerating voltage) of the Ar$^+$ ions. This controls the average energy with which the Ar$^+$ ions deposit upon collision with the SiO$_2$ samples or InAs nanowires. The ion energy was set to a range of 500 V - 1250 V in increments of 250 V. For every value of the ion energy set, a new sample was mounted in the vacuum chamber.

To investigate the effect of extraction voltage on the SiO$_2$ layer, the sample was left for 10 minutes inside the vacuum chamber with the sample facing the direction of the plasma, the extraction voltage set to -300 V but with zero acceleration voltage. This way we can determine if SiO$_2$ is milled away just by having the extraction voltage. To get an even better understanding, argon milling was performed on two samples with the extraction voltage set to -180 V and -300 V while having the ion energy set to 500 V. The milling rate was then calculated and compared from this set of parameter values.

Argon pressure was another parameter that could be varied and studied in relation to the milling rate of the SiO$_2$ layer. The pressure of argon gas was set to 0.5, 1, and $2 \cdot 10^{-4}$ Torr while having the ion energy and extraction voltage set to

500 V and -200 V respectively. The milling rates obtained from these pressures prompted us to investigate the effects of pressure variations around $0.5 \cdot 10^{-4}$ Torr. The experiment was then repeated with pressure values of 0.4, 0.5, and $0.7 \cdot 10^{-4}$ Torr. It was noted that there is a very high risk of the plasma extinguishing when argon pressure was set below $0.4 \cdot 10^{-4}$ Torr.
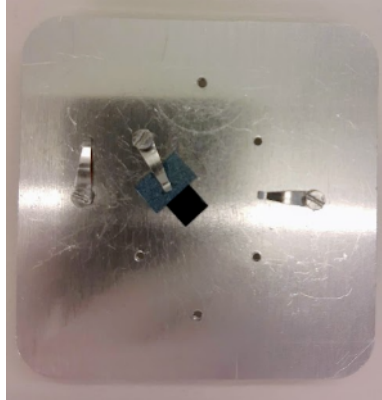

Figure 2.1: Sample on the holder ready to be mounted in the Temescal for argon milling experiment.

Samples alignment with respect to the beam is of great importance as it was noted that the beam has a varying intensity across its cross-sectional area. The Ar^+ beam is more intense at its center and decreases as one moves towards the edges. A silicon wafer of about 2 inches in diameter with a known thickness of SiO_2 was used to estimate the beam's position and cross-sectional area by exposing the wafer to the beam for 2 minutes with ion energy, extraction voltage, and argon pressure set to 500V, -200V, and $0.5 \cdot 10^{-4}$ Torr respectively. The difference in thickness of SiO_2 before and after milling was measured with the ellipsometer and the corresponding milling rates as a function of position on the wafer were calculated and plotted.

Lastly, the effect of the time of exposure of the beam to the SiO_2 samples was determined by doing several runs of the argon milling experiment on test samples, varying the time of each run while keeping other parameters fixed. Three runs of argon milling experiments were performed with times of exposure set to 1, 4, and 7 minutes. Again the difference in thickness was measured and the milling rates were calculated from which the relationship with respect to time can be determined.

Before moving on to nanowires, the milling rate of PMMA for the same set

of parameter values was determined. PMMA is a soft organic material that can be milled away with ease, it's important to understand this so as to have enough PMMA resist left for the lift-off process even after exposing it to the Ar^+ beam. A 2-inch silicon wafer was coated by the A6 PMMA resist and spun at 4000 rpm for about 1 minute to ensure an even redistribution of the resist across the diameter of the wafer. The wafer is then baked at $180°C$ for about 5 minutes so as to dry the resist making it solid enough for Ar^+ ion exposure. During argon milling, a rectangular slit was chosen to expose the wafer coated with PMMA while keeping the rest of the wafer protected from the Ar^+ ions. By rotating the rectangular slit at $45°$ angles, more test runs could be performed on the same sample while varying the time of exposure of the beam to the wafer. Using the ellipsometer, the thickness of the PMMA resist left after argon milling could be measured and thus determining the milling rate of PMMA.

Figure 2.2: Silicon wafer coated with PMMA having two rectangular stripes after argon milling, 4 minutes of milling on the horizontal stripe, and 1 min of milling on the other.

2.2   Ellipsometry

Before and after carrying out argon milling on the test samples, the thickness of the SiO_2 layer has to be determined. This can be achieved through the optical method known as ellipsometry. It is a non-destructive optical technique used to determine the thickness of thin films by measuring the optical constants of a material such

as the refractive index n, and the extinction coefficient k. Extraction of n and k optical constants rely on concepts of reflection of light and the measurement of the polarization of light. A linearly polarized beam of light is illuminated onto the surface of the material to be characterized where it will be reflected. The light which is polarized parallel to this plane of reflection is named $p-$polarized while that perpendicular is called $s-$polarized. The reflected light will undergo a phase shift and amplitude change relative to the incident beam resulting in an elliptically polarized light, thus the name ellipsometry. The changes in the wave properties correlate to the properties of the film being analyzed. Some films will give big changes in amplitude and phase shift while some will give small changes. The phase shift and the amplitude change are measured as Δ and Ψ by the ellipsometer. A wide range of wavelengths of light is being reflected off the sample at different angles giving more reliable model-fitting results.
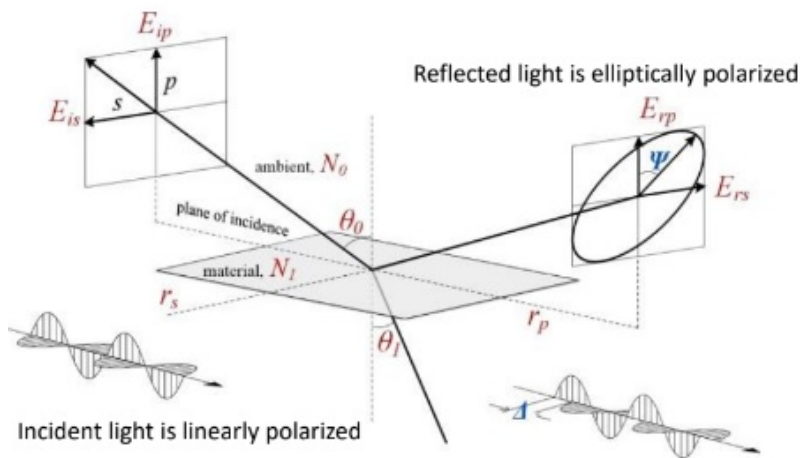

Figure 2.3: The working principle of the ellipsometer. The p and s polarized light form a resultant linearly polarized light before being reflected on the surface of a material changing the resultant wave to be elliptically polarized.[2]

The measured Ψ and Δ parameters are matched to those of the sample model representing the layers of the material. The sample model usually contains a list of optical constants n and k from which the Ψ_{model} and Δ_{model} are computed and compared/fitted to that measured by the ellipsometer. A good sample model will match to a higher degree with the data obtained experimentally. The sample model can be adjusted accordingly to fit the experimental data if there is a significant

difference.

Samples were cleaned and loaded on the Woollam RC2 ellipsometer. A model representing the layers of samples used was created together with the scanning pattern on the samples. For each data point on the sample, 3 angles of measurements were chosen from 65° to 75° in increments of 5°. This gives more data such that a better fit for the model is obtained. Finally, data were filtered out to include only the wavelength of light that is transparent to the material and eliminating data where the light is absorbed.
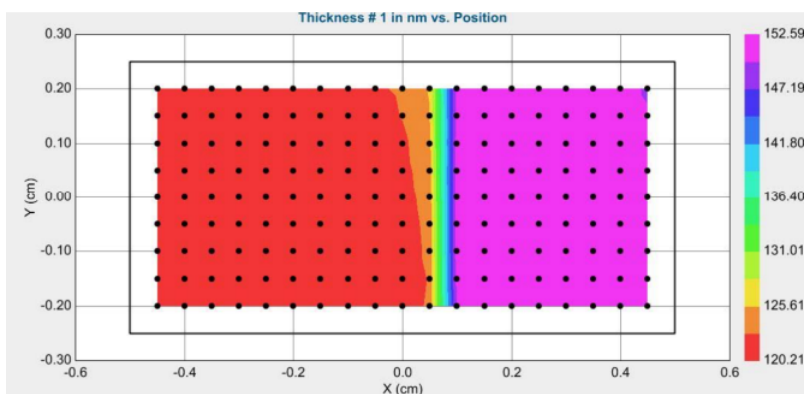

Figure 2.4: Measurement of SiO_2 thickness as a function of the position of a rectangular sample after Ar milling. The red side shows the region exposed to Ar^+ ions while the purple side was the region protected from the beam.

2.3   Contact design

Metal contacts to nanowires are fabricated on special chips with small golden metallic pads to which we can bond to the external circuitry. These chips have doped silicon which is highly conducting as the substrate and a silicon dioxide (SiO_2) layer at the top which is a poor conductor of electricity acting as an insulator. This insulation is very important as it prevents short circuits in nanowires. Nanowires are deposited onto the chips using a well-crafted indium metal tip which can scoop nanowires from where they are grown with little damage to the nanowires. Nanowires can stick very well on the indium tip and on the chip when deposited due to the strong Van der Waals forces as a result of the high surface area to volume ratio of nanowires. The growth number of the nanowires used was

3385 and the array number (dose) was 40.

The next step is to spin-coat the chip with an electron-sensitive film called polymethyl methacrylate (PMMA) or simply resist. PMMA is a synthetic polymer that changes its properties when exposed to an electron beam. The containers of resist are usually labeled with a number that represents the molecular weight of the polymer, and the percentage dilution of the resist. The dilution is expressed in the percent solids dissolved in the anisole; thus "A5" is a 5% dilution of PMMA in the Anisole[11]. The viscosity of PMMA along with spin speed determines the resist film thickness on top of the chip. Spin-coating is done on special spinners where the silicon chip with nanowires is firmly held in place. Small droplets of the resist are then applied onto the chip and the chip at spun at 4000 rpm for about a minute until an even distribution of the resit is attained. The chip is then baked on a hot plate at 180°C for several minutes, evaporating the solvent and leaving a more solid and dry film that is ready for electron beam lithography (EBL) and argon milling.

Electron beam lithography is a technique used to pattern well-defined pathways to an electron-sensitive resist using a focused beam of electrons. It's a very high-resolution method that can achieve small spot sizes of up to < 10 nm. For this, the beam must be focused to a very high degree by controlling x and y deflection, the electron dose per pixel, and the whole tool must be suspended on springs to eliminate effects from external vibrations. The electron beam changes the solubility of the resist in a developer at the desired areas. This allows selective removal of the resist. The solution used to dissolve the exposed bits of the resist is a $1 : 3$ MIBK: isopropanol solution. MIBK is responsible for dissolving the resist while isopropanol acts as a cleaning reagent helping to speed up the process. This process is known as development.

The chip is unloaded from the EBL and then dipped into a beaker containing the developing solvent for 40 seconds and then transferred to another beaker containing isopropanol for 30 seconds rinsing the sample chip. Under a microscope, the newly developed pathways can be observed. To remove any residues and clean the pathways, the chip is exposed to oxygen plasma inside a chamber for 30 seconds. Oxygen radicals are created in the chamber which quickly react with organic residues (hydrocarbons) forming carbon dioxide (CO_2) and water vapor that escape as gas.

2.4   Ar milling of InAs nanowires and Metal evaporation

The Temescal is used for both the argon milling process and in-situ metal deposition. Sample chips with deposited nanowires and well-defined passages for metal contacts to nanowires are mounted on the Type-D holder. The holder was then loaded in the vacuum chamber of the Temescal and the parameter values identified in chapter.2.1 for optimized milling rate were set, ie $0.5 \cdot 10^{-4}$ Torr argon pressure, -200V extraction voltage, 500V ion energy, and 30 seconds of beam exposure.

After argon milling, the plasma is extinguished and the sample is rotated away from the Ar^+ beam source and towards the metal evaporator. The sample is always in a good vacuum condition thus preventing the formation of the oxide layer onto the nanowires. Firstly, 5 nm of titanium (Ti) metal is deposited, followed by 95 nm of aluminium (Al). Titanium acts as an absorbent of oxides that might still be present on the nanowires while aluminium is the conducting metal preferred due to its intrinsic properties such as superconductivity when cooled down to very low temperatures.

During metal deposition, the entirety of the sample chip gets covered with the metals which have to be removed except at the contact areas to the nanowires. Thanks to the presence of the PMMA resist, the unwanted metal can be removed by dissolving the resist underneath. The process of dissolving the resist to clear out the unnecessary metal on the chip is known as the lift-off process. Lift-off is achieved by dipping the chip in acetone heated at about 60°C for 10-15 minutes. Acetone reacts with the polymer chain present in the resit which completely dissolves it, the metals on top of the resist do not have a surface to adhere to and hence are easily washed away. The chip is placed in another beaker containing acetone solution for a few minutes before rinsing it with isopropanol.

2.5   Bonding and Electrical measurements

The quality of the metal contacts to nanowires can be inferred from the measurement of the electrical resistance across the nanowires. The lower the resistance the better the contact quality, which translates to having less oxide layer in nanowires. For each nanowire, a total of four metallic contacts were made from which two-probe measurements were done on the two inner contacts and four-probe measurements using all four contacts. This way we can isolate the resistance due to metal contact with nanowires from the resistance of the nanowires themselves by calculating the differences of resistance obtained via four-probe measurement from two-probe measurement. It is important for the inner probes in four probe mea-
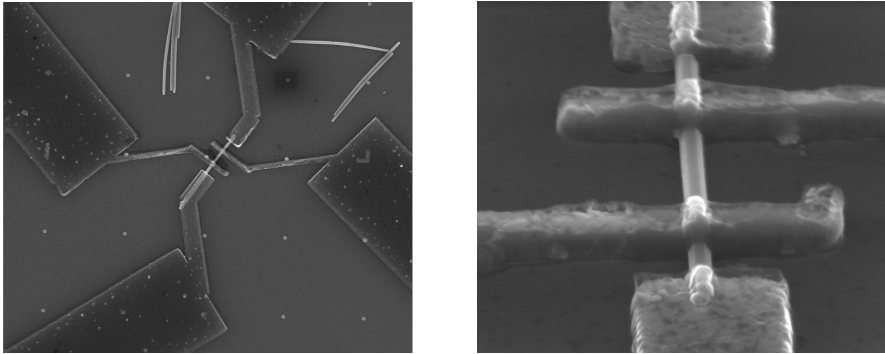
Figure 2.5: SEM images of the nanowire with Ti/Al contacts. The left image is a top-down view while the right image is tilted by 52°. The images were taken from two different devices.
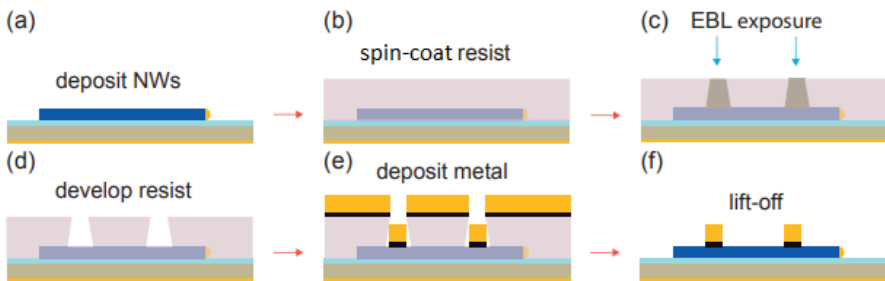

Figure 2.6: Schematic illustration of the processing steps involved when defining metal contacts on nanowires. (a) Nanowires were deposited on a measurement substrate. (b) The substrate was spin-coated with EBL-resist. (c) The contact design was projected onto the sample using electron-beam writing. (d) The exposed regions dissolved during resist development. (e) A thin layer of metal was evaporated on the substrate. Here, two metals were used, 5 nm of Ti for better adhesion, followed by 95 nm of aluminium.[5]

surements to have high impedance so as to prevent current flow through the measuring device (voltmeter). Low impedance in the inner probe will show the same result as with two probe measurements.[10]

Taking electrical measurements on the chip requires bonding to a package that has protruding pins at the edges from which we can probe the electrical charac-

teristics of the nanowires. A silver paste is used as an adhesive to stick the chip to the package. Then metallic bonding from the small golden pads on the chip to the much larger golden pads on the package is made with aluminium wire. The package is placed in a small vacuum chamber where it is connected to the voltage source, voltmeter, and ammeter through its pins. Each gap containing a nanowire is then probed using computer software that controls electrical measurement equipment where the potential drop and the current across the nanowires are measured. The electrical resistance is then calculated using Ohm's law.

Chapter 3

Results and Analysis

3.1 Optimization of Ar milling process

The effect of ion energy on the milling rate of the SiO_2 layer was examined while keeping other parameters fixed. The relationship between the milling rate and ion energy is depicted in Fig.3.1.
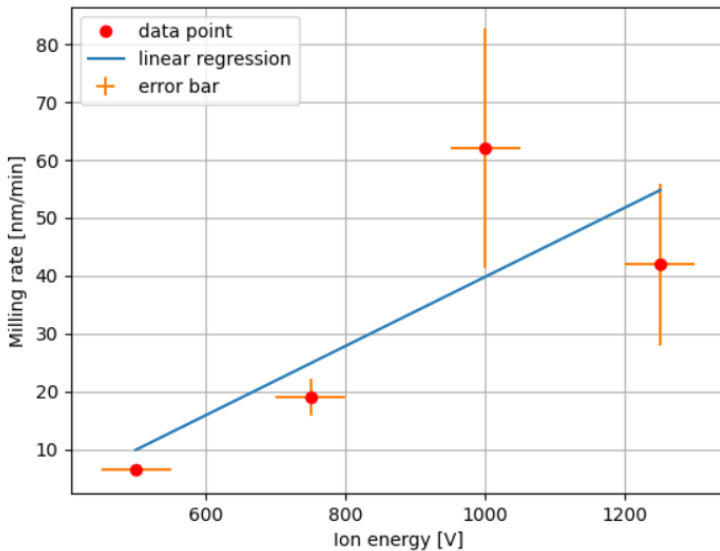

Figure 3.1: Shows the milling rate as a function of ion energy. The error bar represents a 10% deviation from the set value in the ion energy and 10s compound error in milling rate.

It can be observed in Fig.3.1 that there is a linear relationship between the milling rate of SiO_2 and the ion energy of Ar^+ ions. Having a slow and steady milling rate will remove a few nm of the oxide layer present in InAs nanowires, thus an acceleration voltage of 500 V was chosen for milling. The values of the ion energy and the corresponding milling rates used to plot Fig.3.1 are tabulated in Table.4.1 where other parameter values are recorded.

To understand how pressure variations affect the milling rate of SiO_2, an acceleration voltage of 500 V was used and the argon gas pressure was varied between $0.4 \cdot 10^{-4}$ Torr and $2 \cdot 10^{-4}$ Torr. Data obtained are plotted in Fig.3.2.
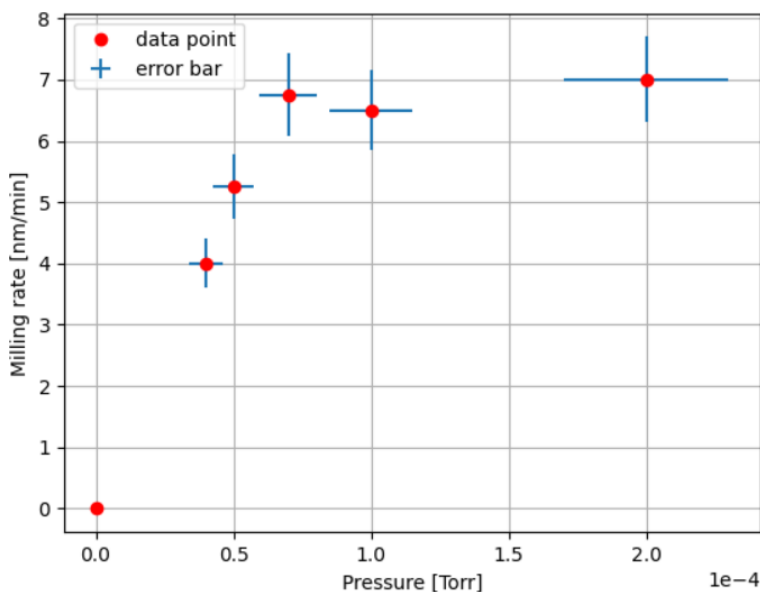

Figure 3.2: Shows the milling rate as a function of argon pressure. The error bar represents 10% deviation from the measured pressure value and 10s compound error in milling rate.

A linear relationship between the milling rates and argon pressure for pressures below $0.7 \cdot 10^{-4}$ Torr is observed while above this pressure the milling rates tend to be constant. We need a low milling rate which can be achieved by setting the argon pressure to $0.5 \cdot 10^{-4}$ Torr. There is a risk of extinguishing the plasma for pressures below this value. The milling rate tends to be almost the same at high pressures due to increased internal collision among Ar^+ ions as a result of the increased number of atoms. The mean free path of the Ar^+ ions is much shorter

as the gas pressure is increased. This results in the loss of kinetic energy of Ar^+ ions as they travel toward the sample.

The time of exposure of the sample to the Ar^+ beam was another parameter of interest and a set of experiments was done to understand its effect. The argon pressure and ion energy were set to $0.5 \cdot 10^{-4}$ Torr and 500 V respectively while varying the time of exposure as seen in Fig.3.3.
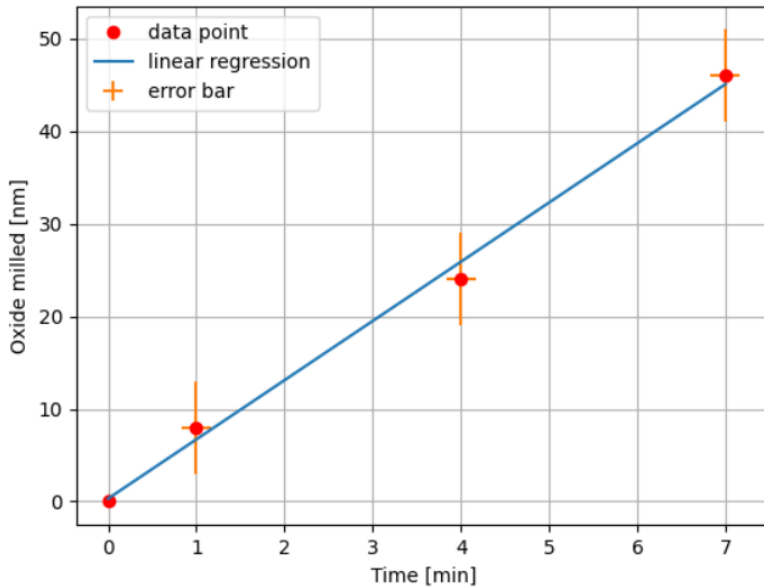

Figure 3.3: Measurement of oxide layer removed as a function of time of exposure of the sample to the Ar^+ beam. The error bars represent a 5 nm deviation in the y-axis and 10 seconds deviation in the x-axis.

Fig.3.3 shows the amount of oxide layer removed as a function of time, a linear relationship between the two quantities can be observed. The longer the exposure of the sample to the beam, the more oxide gets removed, linearly.

The extraction voltage had no direct effect on the milling rate of the SiO_2 layer as can be seen in samples 6 and 7 in Table.4.1 where the extraction voltage was set to -180 V and -300 V respectively giving the same milling rate of 5.50 nm/min. Sample 3 was left in the chamber with the plasma having been ignited for 10 minutes but without applying the ion energy. Here we could also see if there was any sort of milling happening from the UV light of the plasma and having only the extraction voltage which was set to -300 V. The thickness of the

sample measured by the ellipsometer was found to be the same before and after the experiment suggesting that the was no milling of the oxide layer. Since the extraction voltage had no direct effect on the milling, -200 V of extraction voltage was chosen to be used for InAs nanowires.

With this set of parameter values (500 V ion energy, $0.5 \cdot 10^{-4}$ Torr argon pressure, and -200 V extraction voltage) the milling rate of the SiO_2 layer was found to be 6-8 nm/min.

3.1.1   PMMA

Having chosen a set of parameter values for the milling of the oxide layer (ie 500V ion energy, -200V extraction voltage, and $0.5 \cdot 10^{-4}$ Torr of Ar pressure), it was time to understand how the same set of parameter values affects the resist by comparing the milling rate to that of the oxide layer. Data obtained from the experiment are plotted in Fig.3.4 and Fig.3.5.
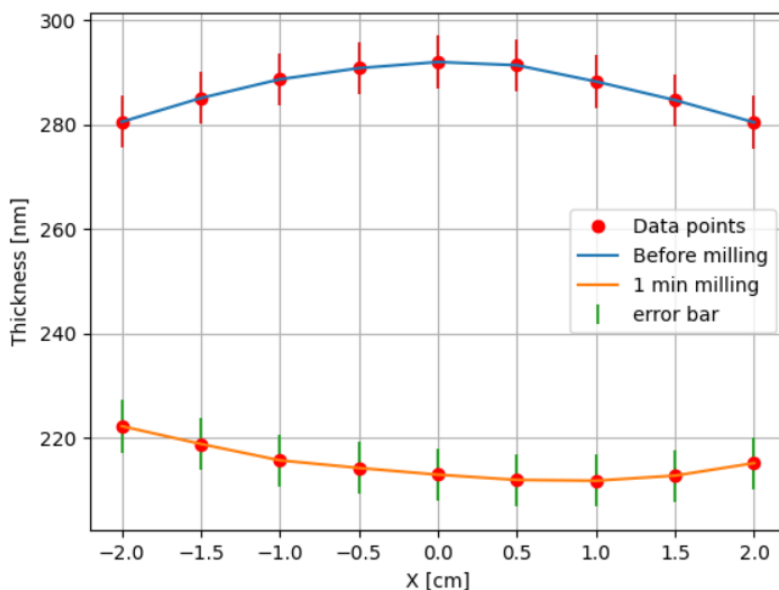

Figure 3.4: PMMA thickness as a function of position on the 2-inch wafer before and after milling. The error bar represents a 7 nm deviation from the recorded value.
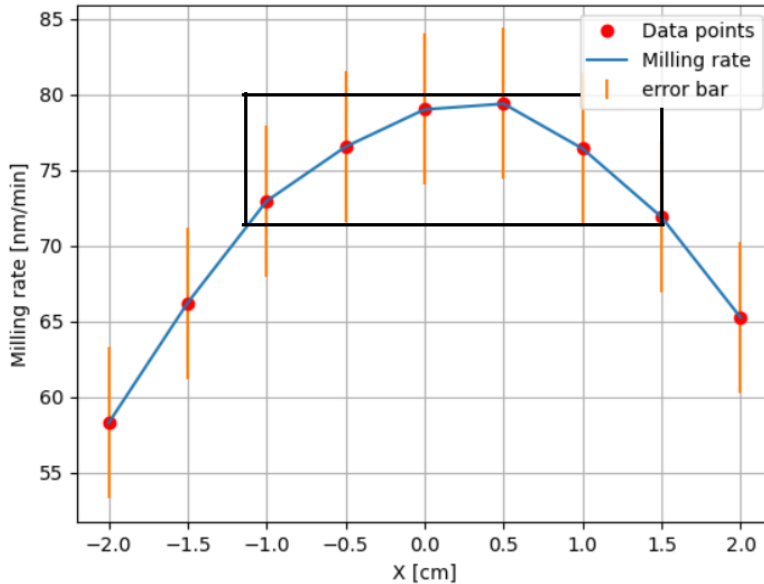
Figure 3.5: Milling rate as a function of position on the 2-inch wafer. The error bar represents a 5 nm/min deviation from the recorded value.

For the same set of parameter values, the milling rate of PMMA is 60-80 nm/min which is 10 times more compared to that of the SiO_2 layer. From Fig.3.5, it can be seen that the highest milling rate is at the center of the Ar^+ beam. To get consistent milling, samples should be placed close to the center of the beam as much as possible. Allowing for a 10% variation from the highest milling rate (80 nm/min) would translate to having a radius of about 1.5 cm from the center of the beam as shown by the black rectangle in Fig.3.5

3.2 Electrical measurements

Two probe measurement was performed on a set of InAs nanowires before and after milling. The resistance is calculated from the slope of the voltage versus current curve. Fig.3.6 shows the graphs obtained from two probe measurements for a single nanowire.

The electrical resistance without argon milling is 1000 times higher than that
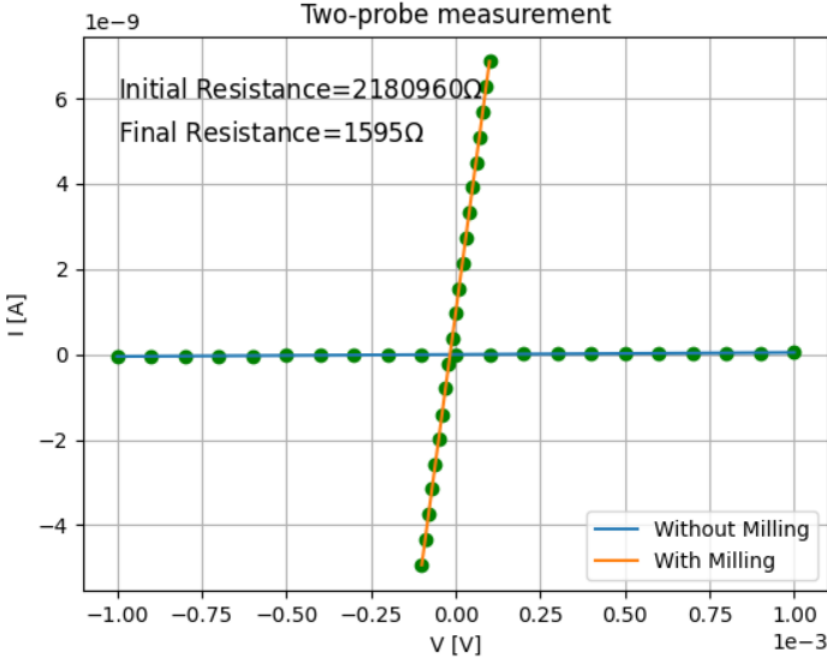
Figure 3.6: Potential drop across the nanowire as a function of current. The current recorded for the milled nanowire was scaled down by a factor of 100 when plotting.

with milling. The difference is very significant which shows how important it is to remove the oxide layer to nanowires before making contacts.

Two probe measurements include the resistance of the contacts and the nanowires themselves, hence to get quantitative data for contact resistance only, four probe measurement was performed to the same set of nanowires where the resistance of the nanowires can be inferred from the potential drop across the inner probes.

Fig.3.7 show the resistance of the InAs nanowire measured using four probes. The contact resistance with and without milling can be calculated as follows;

$$R_{ref} = \frac{R_{2p} - R_{4p}}{2} = \frac{(2.18 \cdot 10^6 - 1310)\,\Omega}{2} = 1.10\,\text{M}\Omega,$$

$$R_{milled} = \frac{R_{2p} - R_{4p}}{2} = \frac{(1594 - 1415)\,\Omega}{2} = 90\,\Omega,$$
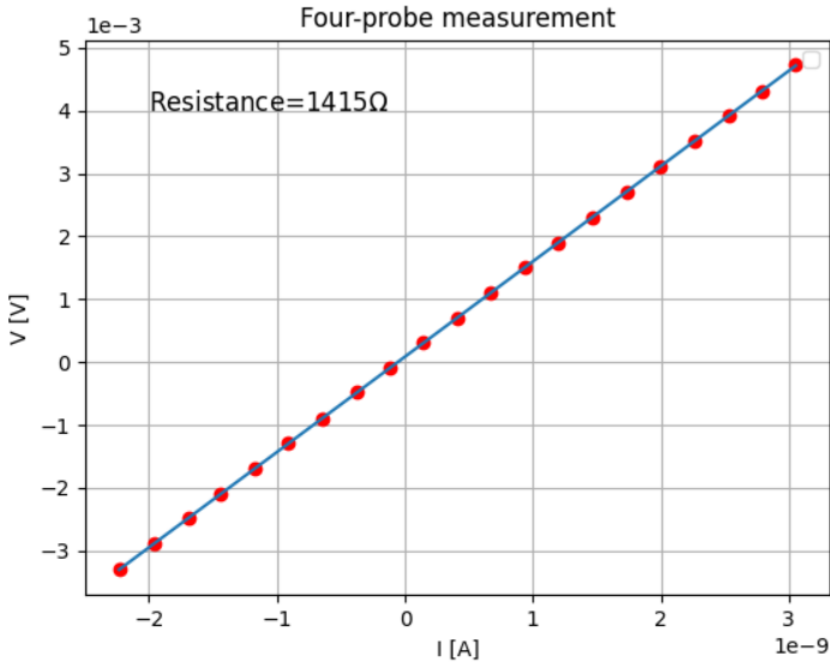
Figure 3.7: Measurement of the potential drop across the nanowire as a function of current.

and the average contact resistance over three nanowires is;

$$\overline{R_{ref}} = 0.84 \pm 0.26 \, \text{M}\Omega,$$

$$\overline{R_{milled}} = 76 \pm 10 \, \Omega,$$

where R_{ref} and R_{milled} are the contact resistance without and with the argon milling process respectively. R_{2p} is the resistance obtained through two probe measurements and R_{4p} is the resistance from four probe measurements.

The contact resistance without argon milling is very high ($\sim$ 1 MΩ) while that obtained after performing argon milling is very low ($\sim$ 90 Ω) that it can be ignored compared to the resistance of InAs nanowires themselves which is about 1-2 kΩ. Thus it is vital for the oxide layer to be removed in order to reduce contact resistance and the argon milling process provides a suitable way to achieve low contact resistance without having to damage the nanowires significantly.

3.2.1 Ar milling rate for InAs nanowires

Since the optimization of the Ar^+ milling process was done to SiO_2 samples where the milling rate of 6-8 nm/min was obtained for a set of parameter values, the milling rate of InAs nanowires for the same set of parameter values was determined. For this, argon milling was performed on a set of InAs nanowires where only a small section of the nanowires was exposed to the Ar^+ beam. This way the difference in height between the milled and the unmilled region can be measured and the milling rate can be calculated from the amount of time of exposure.
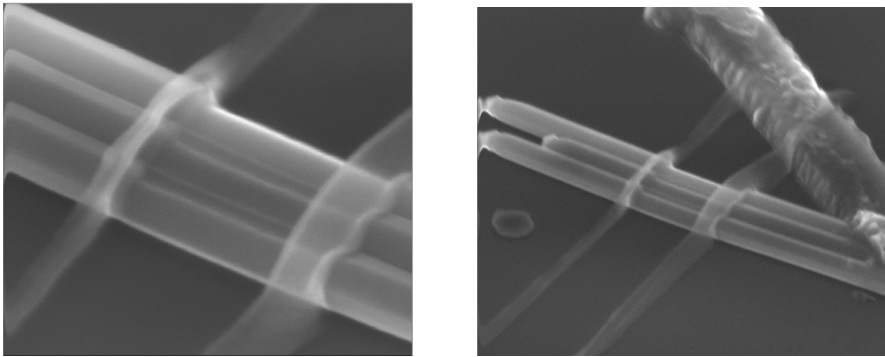

Figure 3.8: SEM images of the InAs nanowire showing a step in height after exposure to Ar^+ beam for 1 minute (view angle is $52°$).

Fig.3.9 shows the height difference after argon milling on a single InAs nanowire. The height difference is more visible when viewed at an angle rather than a top-down view. At $52°$ angle, the height difference of 20 nm is visible, to calculate the real height difference, we have to account for the angle at which the image was taken. This is done by dividing 20 nm by the $\sin(52°)$ which gives 25 nm, and since the milling was done for a minute, the milling rate for InAs nanowires with the same set of parameter values as SiO_2 is 25 nm/min.
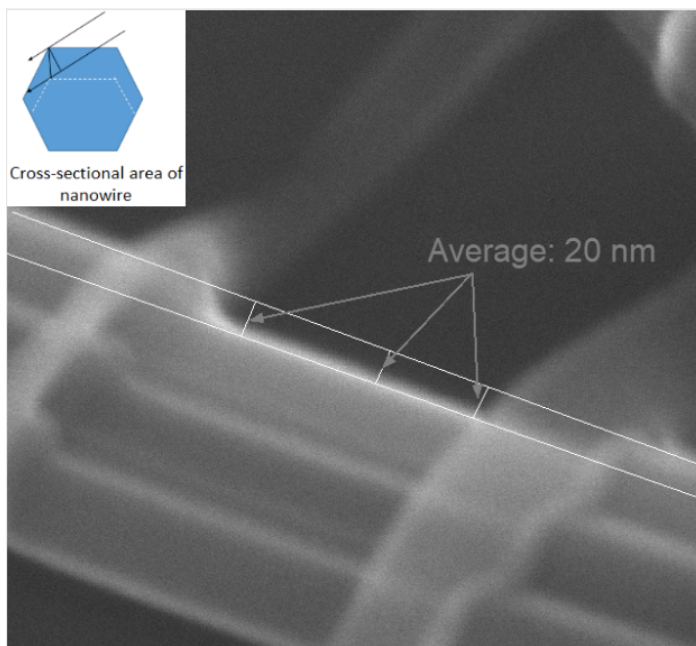
Figure 3.9: Zoomed-in SEM image (viewed at 52°) at the step height showing the 20 nm of the nanowire removed after argon milling. The inset shows a schematic diagram of the cross-sectional area of the nanowires with black lines representing the line of sight.

Chapter 4

Outlook

We have managed to develop an argon milling process by finding the milling parameter values for silicon dioxide samples (SiO_2) where a milling rate of 6-8 nm/min was recorded. The same set of parameter values was used for PMMA and for InAs nanowires where the milling rates are 60-80 nm/min and 25 nm/min respectively. Since the estimated amount of oxide layer in InAs nanowires is about 3-4 nm, 15 seconds will ideally be enough to remove about 6 nm of the oxide layer while still having most of the nanowire intact. One other option is to lower the ion energy, however, one should be aware that there could be a step function where there is no milling happening for very low energies. During the experiment we did argon milling to a set of nanowires for 30 seconds which correlates to about 12 nm of the nanowires milled, as seen in Fig.3.8.

With 30 seconds of argon milling, the contact resistance was very small compared to the resistance of the nanowires themselves that it can be ignored. The contact resistance of about 50-90 Ω was measured after milling while InAs nanowires had an internal resistance of about 1.5-3 kΩ. The contact resistance without milling is very high ($\sim$ 1-2 MΩ) meaning the argon milling process can remove the oxide layer and give low contact resistance in a substantial amount. A small set of nanowires were used during the experiments and it is always a good idea to perform the experiment with a larger set of nanowires where more statistics can be obtained. It is also worth comparing argon milling to other processes such as the wet etching process where chemical substances react with the oxide layer[7, 8]. One could also combine wet etching and argon milling processes can compare the contact resistance to those obtained by the two methods separately.

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Appendix

Sample	Milling parameters					Ellipsometry		
	Extraction energy [V]	Ion energy [V]	Time [min]	Pressure [Torr]	Initial thickness [nm]	Final thickness [nm]	Milling rate [nm/min]
1A	-300	1250	0.5	$1.0 \cdot 10^{-4}$	148	127	42.00
1B	-300	1250	0.5	$1.0 \cdot 10^{-4}$	148	145	6.00
2A	-300	1000	0.5	$1.0 \cdot 10^{-4}$	148	117	62.00
2B	-300	1000	0.5	$1.0 \cdot 10^{-4}$	148	140	16.00
3	-300	0	10	$1.0 \cdot 10^{-4}$	148	148	0
4	-300	750	1	$1.0 \cdot 10^{-4}$	148	129	19.00
5	-180	750	2	$8.4 \cdot 10^{-5}$	150	121	14.50
6	-180	500	2	$9.5 \cdot 10^{-5}$	145	134	5.50
7	-300	500	2	$9.6 \cdot 10^{-5}$	147	136	5.50
8	-200	500	4	$5.0 \cdot 10^{-5}$	151	137	3.50
9	-200	500	4	$1.0 \cdot 10^{-4}$	148	122	6.50
10	-200	500	4	$2.0 \cdot 10^{-4}$	146	118	7.00
11	-200	500	1	$5.0 \cdot 10^{-5}$	155	147	8.00
12	-200	500	4	$5.0 \cdot 10^{-5}$	149	125	6.00
13	-200	500	7	$5.0 \cdot 10^{-5}$	164	118	6.57
14	-200	500	4	$4.0 \cdot 10^{-5}$	146	130	4.00
15	-200	500	4	$5.0 \cdot 10^{-5}$	148	127	5.25
16	-200	500	4	$7.0 \cdot 10^{-5}$	154	127	6.75

Figure 4.1: Milling rates a function of different parameters used in the experiments. A and B on the sample column represent the position of the sample on the stage, A for samples placed at the center and B for samples placed slightly near the edge of the stage.