Lund University

Bachelor's thesis

Department of Physics
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Using gallium nitride nanowires as STM probes

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Abstract

Gallium nitride (GaN) nanowires grown using catalyst-free metal organic vapor phase epitaxy were used as scanning tunneling microscope (STM) probes. The probes were prepared by placing a GaN nanowire on a tungsten STM probe using a nanomanipulator in a scanning electron microscope (SEM) and welding them together using an electron beam induced platinum deposition. STM imaging was performed on indium arsenide (InAs) (111)B samples and atomic steps were observed. Furthermore, (I-V) scanning tunneling spectroscopy was performed which consistently showed combined band gaps of both GaN and InAs semiconductors.

Abreviations

GaN	Gallium nitride
InAs	Indium arsenide
SPM	Scanning probe microscopy
STM	Scanning tunneling microscopy
STS	Scanning tunneling spectroscopy
SNOM	Near-field scanning microwave microscopy
UV	Ultra violet light
IDOS	Integrated density of states
TIBB	Tip-induced band bending
MOVPE	Metal organic vapor phase epitaxy
NaOH	Sodium hydroxide
FIB	Focused ion beam
SEM	Scanning electron microscope

Acknowledgments

First of all, I would like to thank my family for their support and help that they provided. I would not have been able to do this without my amazing supervisors, Rainer Timm and Sofie Yngman who guided me on my work with this thesis. I would also like to thank Anders Mikkelsen for giving me advice while working in the lab. My gratitude goes to the group of my friends with whom I study and spend most of my free time. I would also like to thank Dean Bitton for asking questions which I could not answer.

Contents

1 Introduction

Scanning tunneling microscope (STM) offers great opportunities for microscopy due to a couple of reasons. Optical microscopy devices rely on light to form magnified images. Light based microscopes have a hard limiting factor defined by the wavelength that is used. This means that observing features which are smaller than the visible light wavelength is incredibly hard. STM imaging uses tunneling currents rather than light to form an image of a sample surface. This method does not have the same limitations as light based microscopes which means that a lot greater magnification can be achieved. With some fine tuning, it is even possible to observe individual atoms. These features made STM a welcome tool in surface science fields.

What this thesis seeks to achieve is to expand further upon STM technology. So far, most STMs use metallic probes due to their conductivity and a relative ease to produce. The idea in this work is to introduce semiconducting probes, which would help further characterize electrical properties of sample materials. In addition to that, some semiconducting materials such as gallium nitride (GaN) has favoring mechanical properties since they can form strong crystal structures. Similar research has already been performed using GaN nanowires as probes in atomic force microscopy [1] and using indium arsenide (InAs) nanowires as STM probes [2].

GaN nanowires were chosen for this project for a couple of simple reasons. Nanowires are high purity crystals, which can be grown in desired shapes and sizes. High mechanical strength of GaN and an ability to shape nanowires so they form sharp structures, offers GaN nanowires an opportunity to be used as STM probes. In addition to that, the 3.4 eV band gap of GaN grants it transparency to a range of wavelength of light, which allows it to be applied in near-field scanning microwave microscopy (SNOM) [3], which imply that multipurpose STM/SNOM probes could be produced using GaN.

In this thesis doped GaN nanowires were used to make STM probes, which then were used to scan on an InAs sample. For comparison, data with a conventional metallic tungsten probe was also obtained which is presented in parallel. InAs sample was chosen since it is a semiconductor with a small band gap (0.35 eV) and it forms distinct structural patterns on the surface. Small band gap is beneficial for STM since smaller bias voltages are desired. Distinct surface features of InAs makes it easy to judge the quality of the obtained scan. Research where a semiconductor STM probe is used to scan on metallic surfaces has already been done [2]. By using a semiconductor probe and a semiconductor sample this thesis wants to push the STM field ahead. What is unique about such configuration is that the presence of band gaps on both the probe and the sample allows to perform STM imaging with specific energies. This idea is further explored in section 2.4.

This thesis is structured as follows: the scientific background covers the theoretical base required to understand the obtained result. Method covers the production of GaN probes and the process of gathering the STM and scanning tunneling spectroscopy (STS) data. Results and discussion presents the obtained results which are interpreted and discussed in the same section. Outlook summarizes the obtained results and explores further possible research branches.

2 Scientific background

2.1 Semiconductors

There is no stringent definition of what a semiconductor is, however a general idea of a semiconductor is that it possesses features of both conductors and insulators. This means that to give a good understanding of semiconductors, first conductors and insulators have to be understood.

2.1.1 Band gap, Conductors and Insulators

Atoms have a number of shells of discrete energies where electrons can be present. When atoms are placed together in a bulk to form 3D solids, these discrete energy levels start stacking on top of each other since they cannot overlap duo to the Pauli exclusion principle. This causes solids to form continuous ranges of energies where electrons can be present which are known as the energy bands. The highest fully occupied energy band is called the valence band and the lowest unoccupied energy band is called the conduction band. Metallic objects have their conduction band partially filled. This means that electrons within this band can be easily exited to higher energy levels within the band, which makes metals good conductors. If, however there is an energy gap between the two bands, known as the band gap, the electrons in the valence band will need greater energies to be moved to the conduction band, which an insulating property. [4] [5]

A more stringent definition of what is a metal and what is not can be obtained with an introduction of the density of states within a solid. The density of states is approximately proportional to the square root of the energy. The highest energy value of filled states at the temperature of 0 K is known as the Fermi energy E_F. If the Fermi energy is within the conduction band, then the object is metallic and is a conductor. Alternatively, if E_F is within the band gap, then the solid is an insulator (see Figure 1). [6]

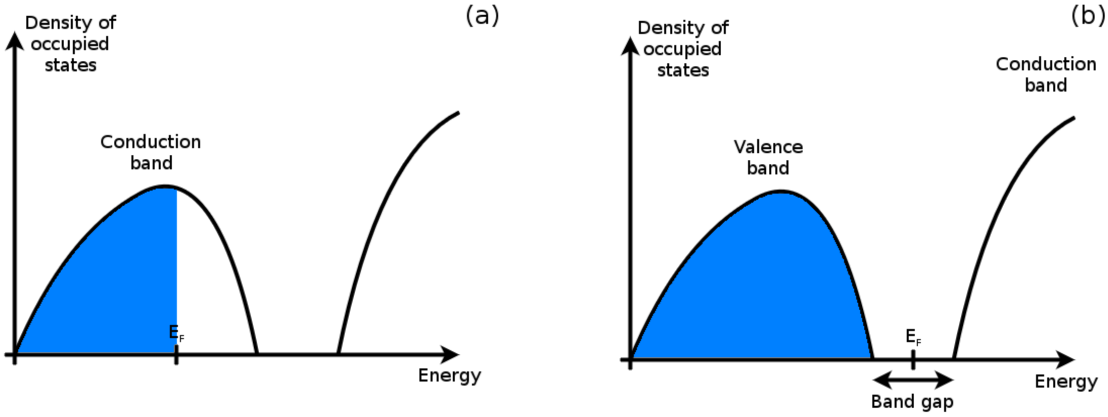

Fig. 1: (a) shows a metallic object which has partly occupied density states (blue) in a conduction band. (b) shows an insulator/semiconductor object which has fully occupied density of states in the valence band and an empty conduction band.

2.1.2 Semiconductors

Semiconductors are not directly defined by the conductivity of the material. The definition of the semiconductor comes from the band gap within a solid. If a solid has a band gap which is small enough for thermally excited electrons to move to the conduction band, then what should be an insulator becomes a conductor. As mentioned in the introduction to this section, the definition of a semiconductor is not clear, and this is the reason why. The energy of a band gap which defines a semiconductor will vary depending on the literature source, but generally it is said to be up to 3 eV. [7]

The easiest way to create semiconductors is to use elements which have four electrons in their valence shell and use covalent bonds to form 3D solids. An example of such elements would be Silicon or Germanium, which are also known as group IV semiconductors. The same effect can also be achieved by forming compounds of elements which have three and five electrons in their valence shell, or two and six electrons. Such materials are known as group III - V semiconductors and group II - VI semiconductors respectively [7]. Examples of group III - V semiconductors are InAs and GaN, which are the semiconductors used in this work. [7]

2.1.3 Doping

Although semiconductors do conduct electricity at room temperature, the conductivity is very poor. This can be worked around by introducing impurities within the semiconductor in a process called doping. The impurities, also called dopant, have one extra electron or one electron less per atom than the elements used to create the semiconductor. Such dopants are called donors (n) and acceptors (p), respectively. In the case of n doping within the semiconductor bulk, there will be one extra electron per dopant which will be bound to the impurity. The binding energy of the excess electron will be low meaning that it will be liberated by thermal excitations. This essentially causes the electron of the dopant to contribute to the conductivity of the semiconductor. [8] [9]

In the case of p doping, an electron hole is introduced. This hole can be filled by any neighboring electrons, however after it is filled in, another electron hole will open up. If this process keeps repeating, it will appear as if the hole is moving, effectively seeming as if a positive charge is propagating through the semiconductor. [9]

2.2 Gallium Nitride

GaN is a group III - V semiconductor which caught interest of a lot of scientists in recent years. GaN possesses great features regarding its mechanical [10] [11], optical [3] and electrical [12] properties.

The reason for the high mechanical strength of GaN crystals comes from their structure. Gallium bonds to three nitrogen atoms by ionic bonds which require a lot of energy to be broken. Furthermore, GaN nanostructures prefer wurtzite crystal structure which offers further mechanical strength.

GaN has a lot to offer in light related research as well. Due to its large band gap, which is 3.4 eV, GaN crystals are used to produce ultra violet (UV) light. This combined with its high luminescence intensity makes GaN crystals a popular choice in the production of white LEDs [13]. For a GaN based LED to produce white light it has to be covered in luminescent coating which is activated by UV light and emits a multitude of lower energy light waves, combination of which to human eye appears as white light. In addition to producing UV light, large band gap also makes GaN opaque to visible light. This allows for coated GaN crystals to be used in light-based SPM, such as SNOM.

The doping level in GaN can be easily varied, what gives great flexibility in electrical properties of the material. Good conduction is an essential feature for STM applications.

2.3 Quantum Tunneling

The phenomena of quantum tunneling can be described by the Schrödinger equation [14]. Consider a one-dimensional potential well, with the potential $V(x) = 0$ within the well and $V(x) = V_0$ outside of the well. The Schrödinger equation within a well will be

$$E\phi = -\frac{\hbar^2}{2m}\phi'' + V(x)\phi,$$

where E is the energy eigenvalue of the particle, ϕ is the wave function of the particle, $\hbar$ is the reduced Plancks constant, x is the spatial position and m is the mass of the particle. A solution for the Schrödinger equation is

$$\phi(x) = Ae^{ikx} + Be^{-ikx},$$

where A and B are constants and k is the wave number $k = \sqrt{2mE/\hbar^2}$. Outside of the well, the Schrödinger equation will be

$$E\phi = -\frac{\hbar^2}{2m}\phi'' + V_0\phi. \tag{1}$$

Introducing a constant $\kappa = \sqrt{2m(V_0 - E)/\hbar^2}$ in equation 1 simplifies it to $\phi'' - \kappa^2\phi = 0$. Alternatively, this equation can be expressed in an exponential form:

$$\phi(x) = Ce^{-\kappa x} + De^{\kappa x} \tag{2}$$

where C and D are constants. Taking a look at the equation 2 from a physical perspective, it can be observed that the negative exponential will contribute to decreasing the $\phi(x)$ value and the positive exponential will increase this value. It does not make sense for probability density to increase within a potential barrier, hence only the negative exponential will contribute. This means that the constant $D = 0$ giving the equation 2

$$\phi(x) = Ce^{-\kappa x}.$$

This means that instead of a particle being reflected when it encounters a barrier, the probability of finding a particle exponentially decays the further in the barrier you go as showed in the Figure 2.

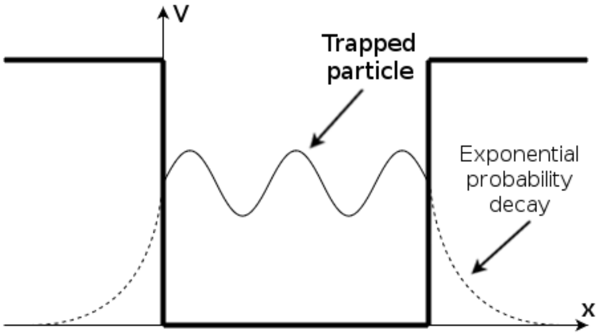

Fig. 2: A particle trapped in a potential well. The dashed lines show the exponential probably decay of finding the particle within the barrier.

If a barrier happens to end before the probability of finding a particle decays to an unmeasurable value, there will be a probability to find that particle on the other side of the barrier as displayed in the Figure 3. This results in a particle passing through what classically seemed an impassible barrier and this action is called quantum tunneling.

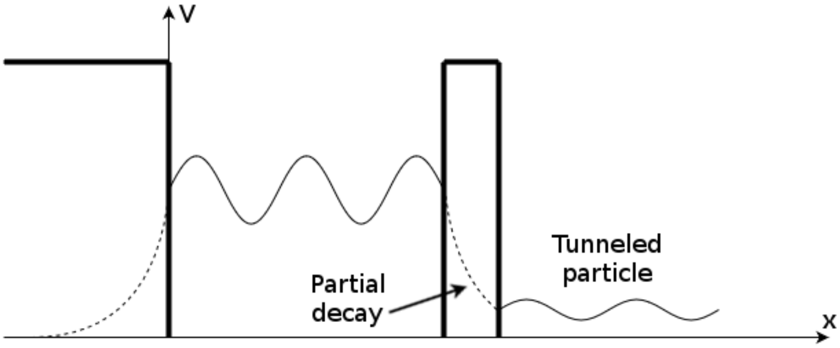

Fig. 3: A particle tunneling through a barrier.

2.4 Scanning Tunneling Microscopy

The STM is a tool which employs the phenomenon of quantum tunneling to produce an image of a sample surface [15]. The resolution of the tool is high enough to observe individual atoms, however, more importantly it gives measurements which are accurate even at sub-nanometer scale. Since its invention, STM features have attracted a lot of interest from surface scientists and a lot of effort is made towards further improvement of this method.

The key element in an STM is its scanning probe. It is made of a hard conductive material such as tungsten or platinum-iridium. The most important aspect of the probe is that the tip is etched to be as sharp as possible. The reason for this is that the part of the probe which conducts must be consistent throughout the whole scan. Usually 90% of the tunneling current goes through a single outermost atom. The fine movements of the tip during the approach to the sample and during the scanning of the sample are done using piezoelectric actuators. Piezoelectricity is a phenomenon which causes some crystals to accumulate electric charge when subject to mechanical stress. This process is reversible which means that if an STM tip holder is attached to crystals which possess piezoelectric properties, electric fields can be used to move the probe in fine increments. This method allows movements of the probe on a scale which is below 10^{-10} m.

To perform a STM, an electrical bias has to be applied either to the probe or to the sample while the other part is grounded. The applied bias is typically in the range of a few volts when semiconductors are involved in the scanning as to avoid the band gap. When scanning is performed between metals, a bias of a few mV is sufficient. Lower voltage is desirable since a higher resolution is obtained if the probe approaches closer to the sample before current threshold is reached. With the bias applied, the probe is moved towards the sample in a perpendicular plane, until the flow of the current is detected. Once the scanning of the sample is initiated, the probe moves to another position, where using a feedback loop its height is adjusted to obtain a constant current. The scan continues by performing this action at different points in a straight line until the end of the scan area is reached. From there the probe moves to a new line and the scan continues until the designated area is filled with point scans. A picture is formed by plotting the values of the probe height at which a desired current was detected and the positions where it was obtained.

The tunneling current when STM is performed on semiconductors is given by equation 3

$$I = \rho_{tip} \int_0^{eV_T} \rho_{s,loc} \left(\vec{R}, E_F + \varepsilon \right) d\varepsilon, \tag{3}$$

where I is the tunneling current, ρ_{tip} is the local density of states of the probe apex, eV_T is the sample energy, $\vec{R}$ is the position of the STM probe, $\rho_{s,loc}$ is the local density of states of the sample at $\vec{R}$, E_F is the Fermi energy and ε is the applied bias voltage.

The use of semiconductor probe and sample creates a unique interaction between their electronic densities of states. Namely, scanning can be performed at specific energies as opposed to metallic probe/sample, where scanning is performed on a range of energies. Figure 4 shows the density of states interactions between a semiconductor probe and sample. What can be observed in Figure 4 is that with no applied bias there will be no tunneling current between a probe and a sample. However, if a sufficiently high negative bias is applied to the sample, its valence band will be shifted to the same level as the conduction band of the probe where the tunneling will occur. In this case, the imaging will be performed on the filled electronic states of the sample. In the case of a positive bias applied to the sample, the imaging is performed on the empty sample states.

It is worth noting that the STM scans do not directly show the atomic structure. What exactly is shown in such images is the electronic structure of a sample surface. This can be realized by comparing images from scans performed on conductors and semiconductors. Since electrons are most locally confined within semiconductors, it is a lot easier to observe individual atoms in the scan images. In metallic samples, which have non-localized electrons, observing individual atoms is difficult.

It is advantageous to perform STM scanning in a vacuum chamber. Many samples and more exotic probes are subject to oxidation, which will not just alter the topography of the sample surface but will also act as an insulating coating which increases the risk of crashing the probe. If oxidation or other reactions which with air could alter the system

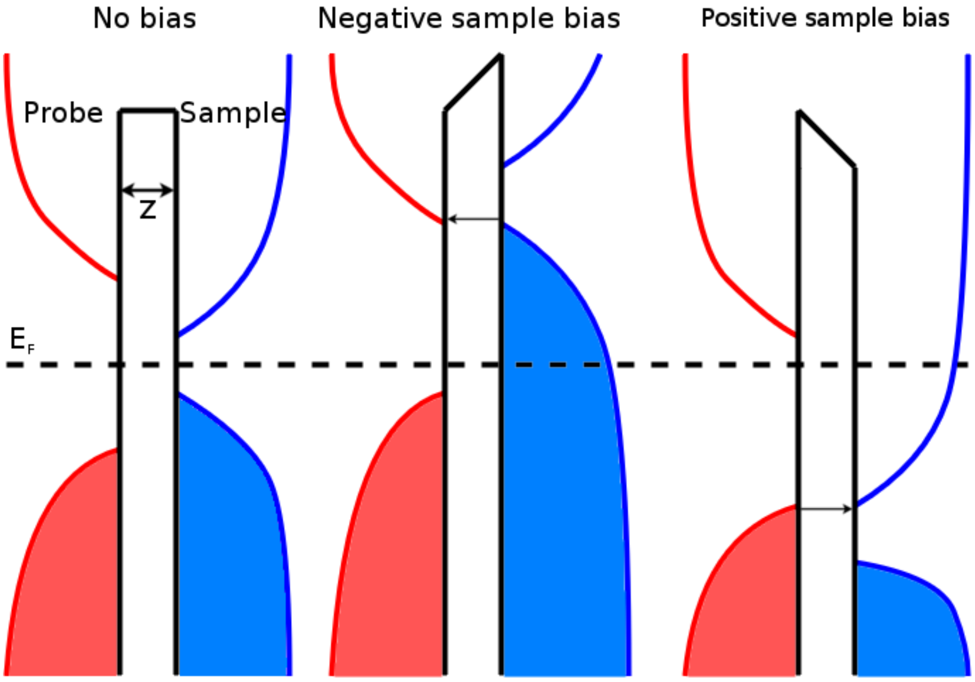

Fig. 4: Three different bias situation between a semiconductor STM probe (red) and a semiconductor sample (blue). Solid color bands indicate conduction bands and hollow bands are the valence bands. E_F is the location of the Fermi energy level and Z is the tip-sample separation. Small arrows indicate the direction of tunneling.

are unlikely to occur, STM can be performed in room conditions. Such machines often have advantage of being a lot smaller and often portable.

2.5 Scanning tunneling spectroscopy

Scanning tunneling spectroscopy is a method of SPM, which allows to inspect the density of states of a sample surface as a function of energy [16] [17]. There are multiple branches of STS, which are current $(I - V)$ spectroscopy, differential conductance $(dI/dV - V)$ spectroscopy, current imaging tunneling spectroscopy and differential conductivity imaging. Although all methods have their uses, only the $(I - V)$ spectroscopy is going to be applied here.

The $(I - V)$ is performed when the probe is at rest and the feedback loop is turned off. Then a range of voltage bias is applied between the probe tip and the sample. As the voltage is applied, reading of the tunneling current is recorded and plotted in an $(I - V)$ graph. When dealing with semiconductor probes and/or samples, their band gap must be accounted for when choosing a range of voltages so that both, the conduction band and the valence band, would appear in the scan. The obtained $(I - V)$ graph shows an integrated density of states (IDOS) over the sample at the position of the scan.

A phenomena called tip-induced band bending (TIBB) is always observed when STS is performed on a semiconductor [18]. The band bending is caused by the electric field between the tip and the sample surface and the low charge density in the semiconductor surface. This causes the surface energy bands to bend upwards for positive sample bias and downward for negative sample bias. Since the STS scans covers negative and positive voltages in a single spectrum, both downwards and upwards bent energy bands are observed. Due to this, TIBB can make the band gap appear a lot greater than it actually is.

2.6 GaN nanowires

Nanowires are nanoscale structures which have a large length to width ratio. III - V semiconductor GaN nanowires have a width which can range anywhere between 60 and 400 or more nanometers[1], depending on the growth process. For the nanowires used here, the growth is done using catalyst-free metal organic vapor phase epitaxy (MOVPE) using a two-step process similar to the one described by Z. Bi [19].

The first epitaxy step produced a nanowire core. It used sapphire wafers which were covered in undoped GaN, followed by doped GaN layers. A SiN mask was applied on top of the wafer and using electron beam lithography holes for the nanowires were etched. With a resist applied the nanowire cores were grown.

The second step applies a high quality GaN layer, which is the main conductor in the nanowire. Additionally, this layer forms a sharp tip which enables the nanowire to be used as an STM probe. Figure 5a shows a sketch of a produced nanowire structure. It is worth mentioning the nanowire cores produced for the purposes of this thesis were hexagonal shape, rather than the circular shape which is presented in the figure 5a. Figure 5b shows an SEM picture of the GaN nanowire wafer with the nanowires produced with a method described in this section.

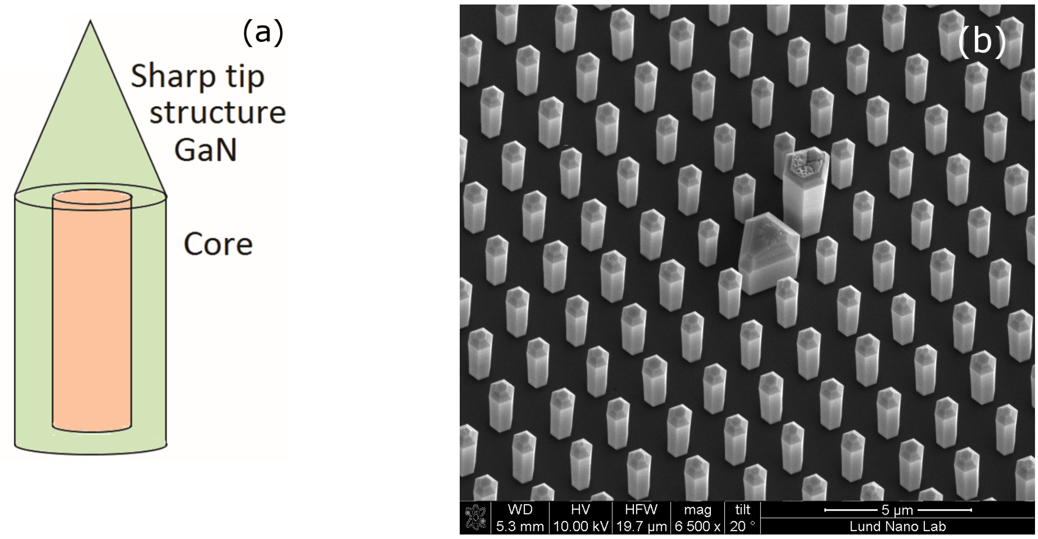

Fig. 5: (a) The design of a GaN nanowire, where the core is grown in the first epitaxial step and the outer layer is added in the second step [20]. (b) An SEM image showing a wafer of produced GaN nanowires. Observe that they are not grown to be perfectly sharp as indicated in figure (a). Although (a) shows the nanowire to be perfectly cylindrical, they are actually hexagonal in (b).

3 Method

3.1 GaN probe production

GaN nanowires have convenient properties to act as an STM tip, however their small size creates a challenge to prepare them for use in an STM tip holder. Since it is only the material at the very end of the probe that decides the properties of the whole probe, it was decided to use a prepared STM probe which is made of tungsten and attach a GaN nanowire to it so that the nanowire would perform the scanning. The advantage of using this approach is that STM probe holders which are designed for tungsten tip can be used here without any further modifications. STM probe holders are used for quick setup and exchange of probes within a vacuum chamber in an STM machine.

3.1.1 Tungsten probe production

The etching of the tungsten tip was done using the Omicron W-Tek Tip Etching Tool. Tungsten wires, which had a diameter of 0.38 mm, were used in the etching. The wires were dipped 2 mm into the sodium hydroxide (NaOH) solution, which had 0.005 mol/cm^3 concentration. Then a voltage of 10.0 V was launched through the tip the etching process was initiated.

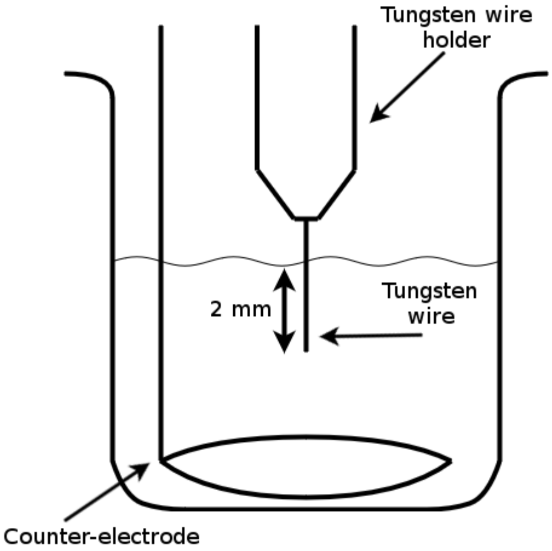

Fig. 6: A sketch of tungsten wire etching setup. A tungsten wire is set 2 mm into an NaOH solution and has a 10 V voltage launched through it. The sketch is not to scale. Note that the tungsten wire holder is not the same object as an STM tip holder.

Once the threshold current of 2.1 mA was reached the etching process was done. The wire had to be rinsed with distilled water to remove leftover NaOH. Cleaning of the wire is necessary to prevent the tip from being etched further and to maintain good vacuum condition inside an STM chamber. An optical microscope was used to inspect the etched tip for any faults, such as improper etch angle, blunt tip or an incomplete etch. Then the length of the wire was treated to fit an STM holder, in which later the wire was set and secured (Fig 7).

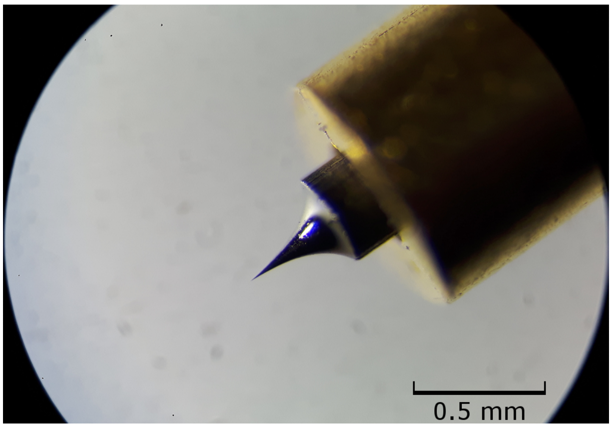

Fig. 7: Etched tungsten probe set inside of an STM tip holder.

3.1.2 GaN probe production

To make a GaN probe, a GaN nanowire had to be attatched to the tungsten probe. The probe was produced using a focused ion beam and scanning electron microscope (FIB/SEM) Nova NanoLab 600. In the SEM, etched tungsten probe was placed next to a clean wafer of GaN nanowires. The nanowires on this wafer were grown to be 3.197 µm long and 0.777 µm wide. Using a nanomanipulator, a GaN nanowire was broken off the wafer mask (see Figure 8). The adhesive forces were enough to pick up the nanowire with the probe.

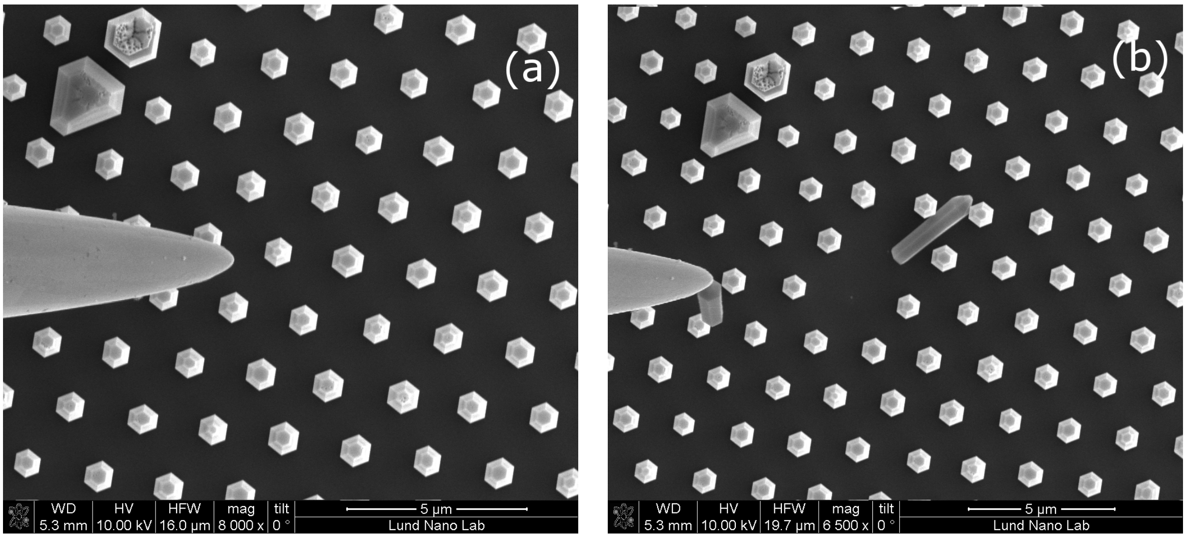

Fig. 8: SEM pictures of a top-down view of (a) a nanowire wafer with a nanomanipulator coming from the left and (b) Two nanowires broken off from the wafer where one of them sticks to the probe

The nanomanipulator with the nanowire was moved to the tungsten tip, where the nanowire was placed at the tip of the tungsten probe. The nanowire is attached to the probe due to the adhesive forces.(See Figure 9).

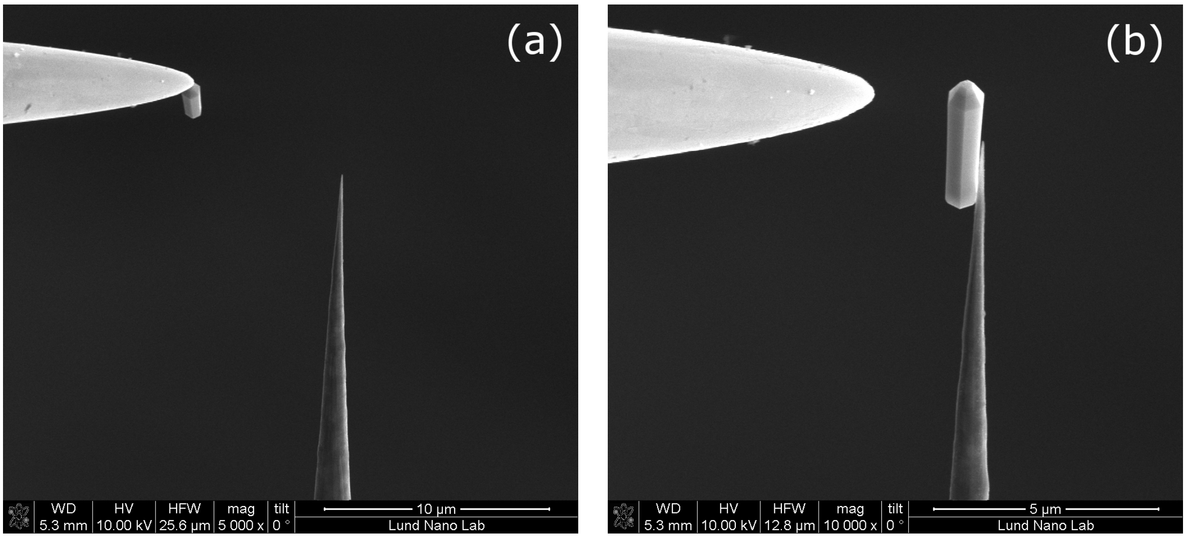

Fig. 9: (a) A probe with a nanowire attached to it (left) moved to the etched tungsten tip (bottom). (b) A nanowire is attached to the tungsten probe tip.

In order to further secure the nanowire to the tungsten tip, electron beam induced platinum deposition was used to weld them together (See Figure 10).

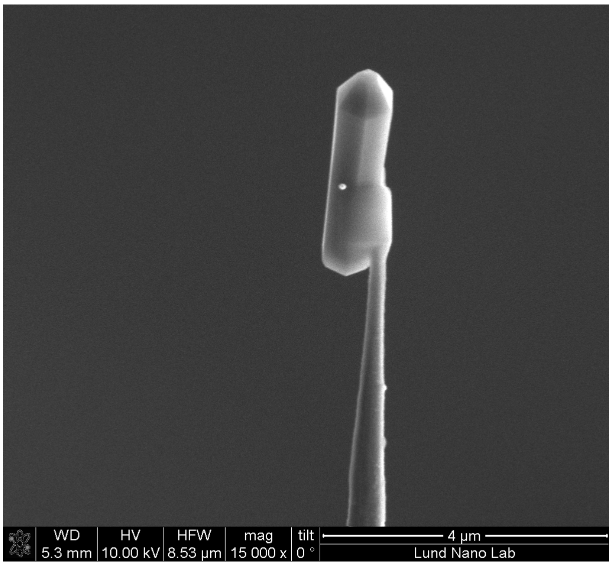

Fig. 10: A prepared STM tip with GaN nanowire. Note the area in which the nanowire comes in contact with the tungsten tip. This area was covered with Pt to secure the nanowire to the tungsten tip.

Without welding the nanowire to the tungsten probe, there is a high risk that the nanowire could be lost in the imaging process.

3.2 STM and STS

A Variable temperature Omicron XA STM machine was used which can sustain a pressure bellow 10^{-11} Pa. The scanning was performed on an InAs sample, which has a band gap of 0.35 eV. Since the band gap within GaN is 3.4 eV, a voltage of ±4.0 V was used to approach the sample. Lower voltage risked being within the band gap, which would have caused the tip to crash into the sample during an approach process.

The scanning current was set to 80 pA and the scanning process was initiated. Using a scanning speed between 20-450 nm/s, a wide variety of different sized areas at a different resolution were scanned. Together with GaN probe, a metallic tungsten probe was also used to obtain a reference STM image for comparison. During the scan process, spectroscopy measurements were performed in parallel. Various scanning voltages were used, however most of the measurements were performed using a voltage range between 5 to -4 V. Higher voltage range was chosen so that the STS scan would include entirety of the band gap, together with the conduction and the valence bands.

4 Results and Discussion

Primarily two types of data gathering were performed, STM and STS. Although both actions were performed simultaneously they are going to be treated separately in this section.

4.1 STM results using the GaN probe

GaN nanowire STM tips were used to scan on InAs (111)B surfaces. Figure 11a shows the images when scanning in a 500 nm x 500 nm area. The observed triangular patterns in Figure 11a are expected in InAs (111)B [21]. In the STM image, differences in brightness indicate a difference in the height at which an STM probe scanned. Figure 11a shows up to four consistent levels at which the scanning process was conducted. This implies that atomic steps were observed.

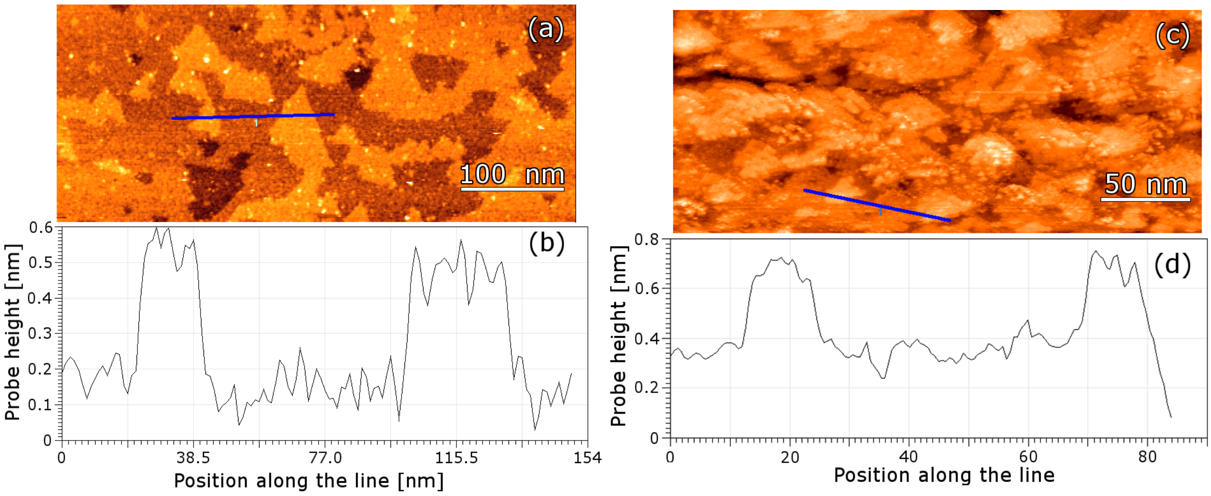

Fig. 11: (a) shows a topographical image of InAs (111)B using a GaN probe in an 500 nm x 500 nm area. Scanning was perform with an applied bias of -4.0 V and the tunneling current set to 80 pA. (b) Shows the variation in the height of the GaN probe along the blue line in figure (a). (c) shows a topographical image of GaAs (111)B using a metallic probe in 300 nm x 300 nm area. Scanning was perform with an applied bias of -2.5 V and the tunneling current set to 100 pA. (d) shows a line scan along the blue line marked in figure (c).

The line scan in Figure 11b shows the changes in the height of the probe along the blue line in Figure 11a. The changes in height are roughly 3 to 4 Å and since the distance between subsequent InAs atomic planes in the (111)B direction is 3.5 Å [21] it further suggests that the atomic steps the atomic steps of the InAs(111)B surface are visible using a GaN STM probe.

Figures 11b and 11d show the received data when scanning was performed using a tungsten probe. The width of the scan image is 300 nm and the scanning was performed on a GaAs (111)B surface. GaAs (111)B has the same crystal structure as InAs (111)B hence similar features are expected from both samples, allowing for good probe comparison. Triangular structures can be observed in Figure 11c however it is also observed that the sample was contaminated since unwanted dirt features can be observed in the figure as well.

When comparing the GaN probe image (Figure 11a) with a conventional tungsten probe image (Figure 11c), the GaN probe shows as good of a result as a metallic probe. The GaN probe image displays clear atomic steps and displays surface features in good resolution. The line scans (Figures 11b and d) look different, where the line scan of the tungsten probe is smoother than that of the GaN probe, however that is due to different feedback loop parameters used while scanning with GaN probe.

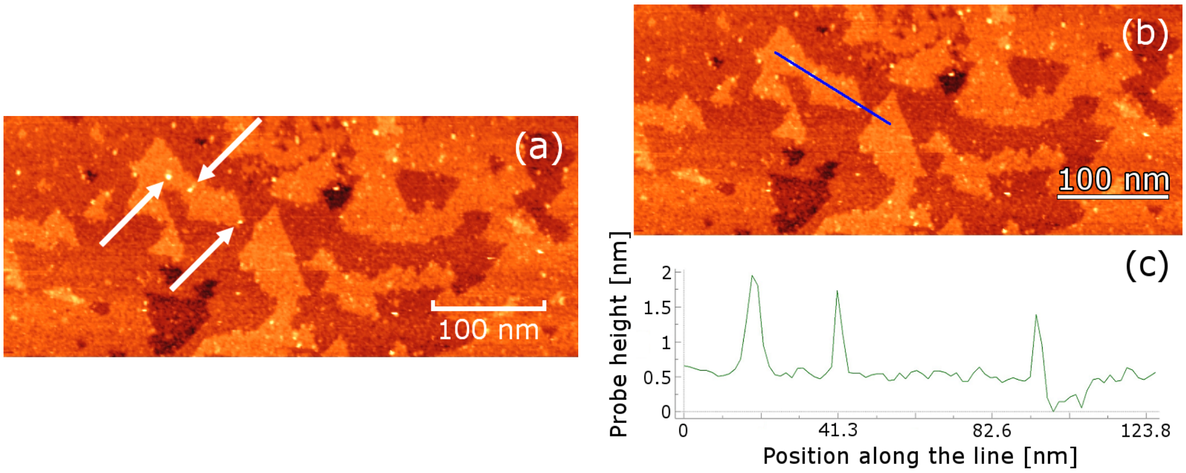

Fig. 12: (a) shows three examples of bright spots. (b) shows the scan line, (c) shows the relative hight of the scanning tip along the scan line.

Figure 12a shows a lot of small bright spots that are visible in the Figure 11a. Figure 12c shows that the elevation in these areas is about 1.0 to 1.4 nm. Since the line scan is inconsistent with the height of these spots, it is a more likely a result of an STM scan error than individual atoms on the sample surface.

Figure 13 shows scans performed in 50 nm x 50 nm area using the GaN probe. A lot of visible disturbances can be seen in the scan. Although some topographic features can be vaguely seen, no conclusions can be drawn simply due to the fact that it is too distorted. It is not completely clear whether the distortions in the figure were caused due to using a GaN tip or due to noise while scanning, however a lot of scanned images were obtained

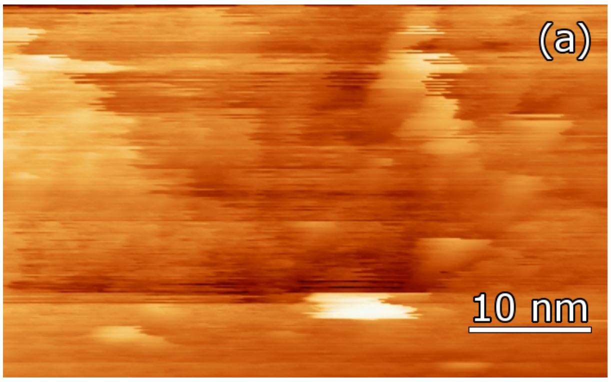

Fig. 13: A topography image obtained when scanning in a 50 nm x 50 nm area on an InAs (111)B sample. Scanning was perform with an applied bias of 4.0 V and the tunneling current set to 80 pA

which yielded similar results what hints towards the probe being at fault. Although the scan was performed in a lot smaller area than in the Figure 11a the difference between the quality is too large to be caused only by that. One likely reason for this change is the fact that in the process of obtaining the Figure 11a the tip crashed into the sample. The effects of the crash are shown in Figures 14a and b.

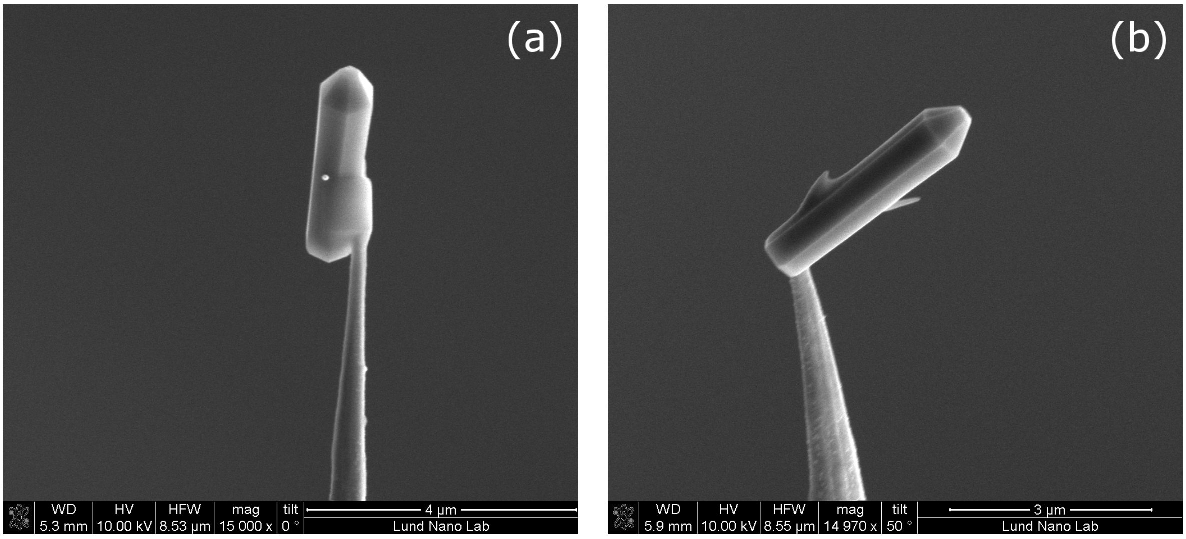

Fig. 14: SEM images in (a) shows the initial state of the STM probe and in (b) shows the probe after the crash.

In Figure 14 it can be seen that only the tungsten part of the probe was damaged during the crash and the nanowire was virtually unaffected. This shows that the GaN nanowire is much more structurally sound than a standard tungsten tip. Since the nanowire was still at the tip of the probe, it was decided to continue using it for further scanning. The fact that the probe is now bent meant that a side of the nanowire tip was conducting current between the sample and the probe. That part of the nanowire is blunter than the scan area before the crash, which is most likely reason for the distorted scan images.

Additionally, during the STM scans it was noticed that the probe alterations were happening as the scans were going on. Namely the resolution of the obtained figures differed, while keeping the same scan parameters. A likely cause for that could have been that during the scan progress the probe picked up some substance from the surface. Since the scanning is made by a single atom on the probe, picked up dirt could be the one performing the scan, or cause whats called ”double tip”. Double tip effectively scans the same sample area using a different part of the probe, giving a distorted view of the surface. Tip conditioning was used to attempt to fix this problem, which launches bursts of voltage through the STM probe which can cause desorption, ridding the probe of dirt particles.

To conclude this section, GaN nanowires can be used as STM probes and obtain detailed images, which display atomic steps, such as in the Figure 11a. The obtained image quality is comparable to the conventional tungsten probes. However, when attempting to scan in small areas, for example in a 50x50 nm such as in Figure 13, stable images which would show distinct surface features could not be obtained. It is likely that this was not caused by the nature of GaN itself, but due to the blunt side of the nanowires used to scan. This shows great opportunities for GaN in the STM field in near future.

4.2 STS results using the GaN probe

During the STM process, STS were performed as well. The bias ranges were chosen to be high enough, so that the combined band gap of InAs and GaN would be covered by the (I-V) spectra.

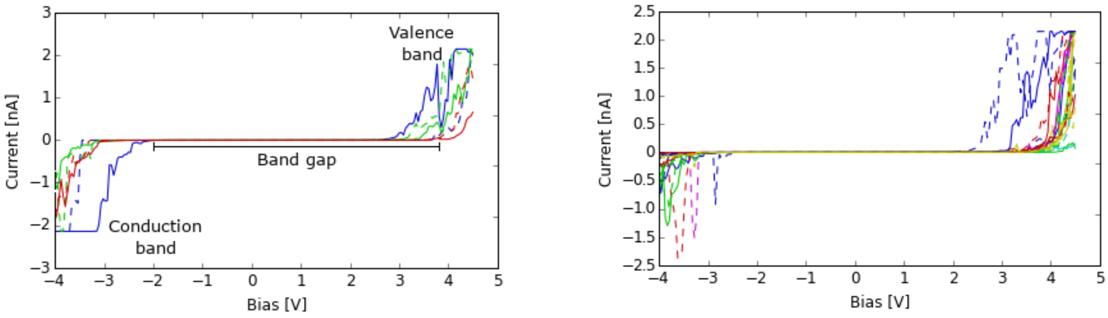

Fig. 15: Graphs showing the results of multiple (I-V) spectra represented by different colors performed on the InAs sample using a GaN probe. Both graphs show the data acquired at different positions on the InAs sample with a GaN probe. Solid lines represent (I-V) spectra scanned from negative to positive bias voltage and dashed lines represent reversed spectra scan, which goes from positive to negative bias voltage. Spectra were plotted using a Python script developed by Olivier Sholder.

Figure 15 shows the results from the performed (I-V) spectra. Figure 16 shows a representative example of an STS (I-V) spectrum from Figure 15 with line fits of the valence band and the conduction band onsets for more accurate reading of the band gap. Here the band gap is estimated by finding the difference between the line intercepts with the x-axis. The band gap in the Figure 16 is 6.6 V. The large band gap indicates that the

STS was performed using GaN nanowires, however, the combined band gap of GaN and InAs is expected to be around 4 eV. The enlarged bang gap is observed due to tip-induced band bending.

Most of the spectra in Figures 15 and 16 also had clearly visible surface states. These states possess specific energy levels, which will conduct current between the sample and the STM probe when a specific bias is applied. An example of such states can be observed in Figure 16. Surface states can be caused by the different arrangement of atoms at the surface when compared to the bulk crystal, a displaced bulk atom or atomic steps [16]. What can also be observed in Figure 16, is that the band gap is not fully symmetric around the Fermi energy at 0 V. On the positive bias, the band gap is almost 4 V, while on the negative bias the band gap is above -3 V. The band gap shift is caused by the n-doped GaN nanowire. It is worth noting that the bands presented in all of the (I-V) spectra correspond to the bands of the probe. In the sample, the valence band and the conduction band swap positions meaning that the conduction band would be within positive voltage bias and the valence band would be within negative voltage bias.

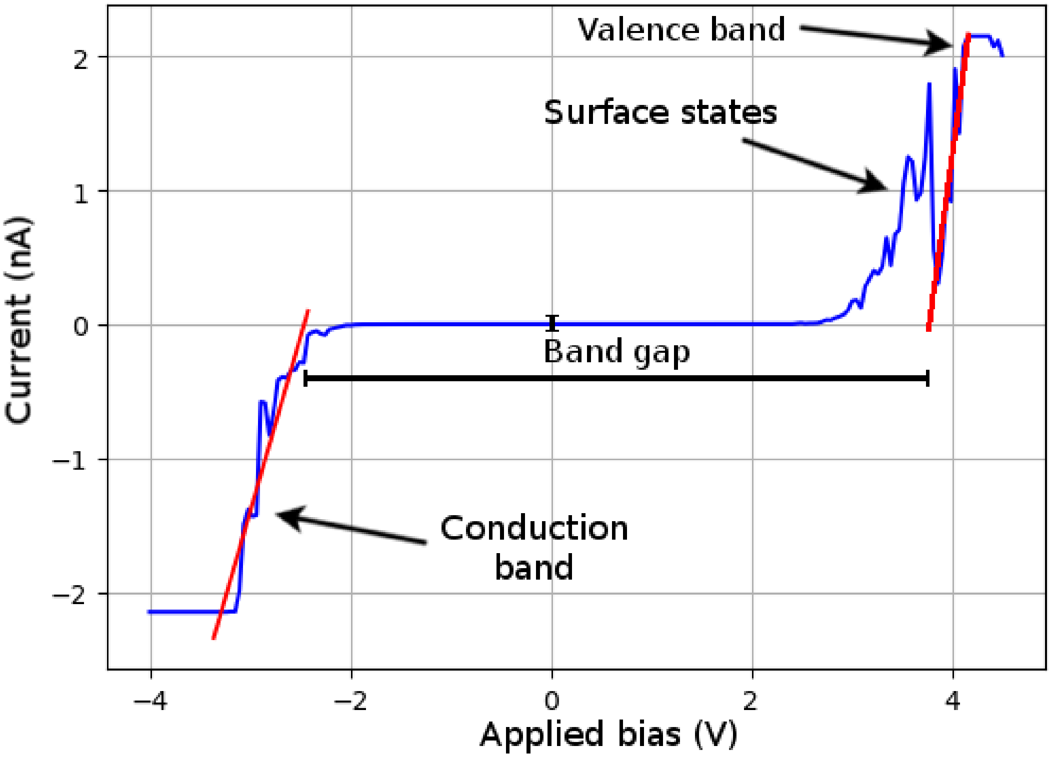

Fig. 16: An STS scan over the InAs sample in a range between -4 to 5 V. Line fits were made over the onsets of the valence band and the conduction band to estimate the band gap. The band gap was found to be 6.6 V. The data was plotted with line fits using python 3 with matplotlib and scipy packages.

To conclude this section, STS spectra show consistent results which fit the expectations of using a doped GaN nanowire as the STS probe. Performing STS in parallel with STM yields additional information about the scanned surfaces, such as the energies of local surface states or the position of the Fermi level relative to the band onsets.

5 Outlook

Overall the GaN probe showed good results in STM and STS fields. Clear atomic steps were observed in the STM images which is a first milestone in developing STM probes. It was unfortunate that stable images could not be obtained when the scanning was performed in smaller areas, however, the cause of this was most likely not the fault of the GaN nanowire, but rather by the deformed probe after it crashed into the sample.

The next step in this research would be to try and achieve atomic resolution images, where individual atoms in the sample could be observed. This should be achievable with a new GaN probe made with a same method as used in this work which would be used to scan a semiconductor sample. If the atomic resolution is achieved, the GaN STM probe would perform on par with a conventional tungsten probe which could eventually make semiconductor probes a preferable alternative in further research.

Further related research that GaN nanowires show promise is SNOM. GaN nanowires have been tested as SNOM light guides in a paper by J.C. Weber [3]. With GaN showing promising results in STM as well, multi-purpose devices which could perform both SNOM and STM operation, which could give the chance to obtain local optical information with nm or atomic scale resolution. In addition to that high mechanical strength of the GaN nanowires, which was demonstrated in Figure 14, offers a possibility to use them in AFM applications.

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