

Charge transport measurements at GaAs-nanowires and development of a low-temperature four-tip scanning tunneling microscope

Von der Fakultät für Mathematik, Informatik und Naturwissenschaften der
RWTH Aachen University zur Erlangung des akademischen Grades eines
Doktors der Naturwissenschaften genehmigte Dissertation

vorgelegt von

Diplom-Physiker

Hubertus Hartmut Robert Junker

aus Engelskirchen

Berichter: Apl. Prof. Dr. Bert Voigtländer

Univ.-Prof Dr. Markus Morgenstern

Tag der mündlichen Prüfung: 29. November 2016

Diese Dissertation ist auf den Internetseiten der Hochschulbibliothek online
verfügbar.

Kurzfassung

Es wurden Ladungstransportmessungen an freistehenden GaAs-Nanodrähten mit einem Raumtemperatur-Mehrspitzen-Rastertunnelmikroskop durchgeführt. Vierpunktwiderstandsmessungen an einzelnen Nanodrähten wurden, mit hoher räumlicher Auflösung durchgeführt. Nacheinander wurden drei Proben untersucht, die mit unterschiedlichen Temperaturverläufen hergestellt wurden. Detaillierte Widerstandsprofile entlang mehrerer Nanodrähte von jeder Probe wurden erstellt. Drei unterschiedliche Regionen wurden dabei in den Widerstandsprofilen identifiziert: Eine Region am Sockel, die aufgrund von fehlendem Dotierstoffeinbau einen hohen Widerstand hat, eine Übergangsregion in der Mitte mit noch höherem Widerstand und eine Region mit niedrigem Widerstand an der Spitze der Nanodrähte, in der der Dotierstoff wie vorgesehen in den Draht eingebaut wurde.

Die Berechnung der Verarmungszonengröße entlang der Nanodrähte hat ergeben, dass die Drähte in den Zonen mit hohem Widerstand vollständig verarmt sind. Nur in der Region mit geringem Widerstand floss der Strom durch den Leitungskanal im Zentrum der Drähte, in den anderen Bereichen floss der Strom durch die Oberflächenzustände. In der Übergangsregion wiesen die Vierpunktmessungen eine nicht lineare IV-Kennlinie auf. Bei hohen Spannungen konnte der Strom von der Oberfläche in den besser leitenden Kern fließen, bei niedrigen Spannungen verbleibt als stromführender Kanal die Oberfläche. Während der Messung der Widerstandsprofile konnten sowohl in der Übergangsregion als auch auf dem Substrat um die Sockel der Nanodrähte elektronenstrahlinduzierte Ströme beobachtet werden.

Ein neues Tieftemperatur-Vierspitzen Rastertunnelmikroskop wurde realisiert, um damit Ladungstransportmessungen durchzuführen. Die Bewegungen der vier Spitzen werden dabei mit einem Rasterelektronenmikroskop überwacht. Bilder, die mit diesem SEM von einer Eichprobe gemacht wurden, konnten genutzt werden, um die Abschirmung gegen äußere Vibrationen zu optimieren.

Ein Nanoantrieb (KoalaDrive), wurde benutzt, um die STM Spitzen an die Proben anzunähern. Der KoalaDrive funktioniert bei tiefen Temperaturen (5 K) und im Ultrahochvakuum mit hoher Präzision funktioniert. Mit diesem STM wurden sowohl atomare Stufen auf Graphit als auch die 5×25 Rekonstruktion von Pt(100) aufgelöst. Einzelne freistehende GaAs-Nanodrähte wurden mit dem Tieftemperatur-Vierspitzen-STM mit einzelnen Spitze kontaktiert. Die elektrische Aufladung eines Spitzenhalters führte zu Verzerrungen in den SEM Aufnahmen und verhinderte so Messungen mit mehreren Spitzen.

Abstract

In order to perform charge transport measurements on freestanding as-grown GaAs nanowires, a room-temperature multi-tip scanning tunneling microscope was utilized as a nanoprobe. Four-point resistance measurements along individual nanowires were performed with high spatial resolution. Subsequently, three different samples, grown with different temperature patterns, were investigated. Detailed resistance profiles along several nanowires on each sample were recorded. Three different regions were identified in the resistance profiles: A high resistance region at the nanowire bases (resulting from a suppressed dopant incorporation), a transition region in the middle with higher resistance and a low resistance region at the nanowire tops, with a desired amount of dopant incorporation.

A calculation of the size of the depleted zone revealed that nanowires were completely depleted in the high resistance segments. Only in the low resistance region, the current could flow through the conduction channel in the nanowire core. In the high resistance region, the current was flowing through surface states. In the transition region, the measured four-point IVs showed a non-linear characteristic. At high biases, the current could transfer from the surface to the better conducting core, while for low biases, the current had to flow via the surface. During the resistance profiling, electron beam induced currents were present in the transition region as well as around the nanowire base on the substrate.

A new customized low-temperature ultra-high vacuum four-tip scanning tunneling microscope capable of charge and magneto transport measurements has been realized to carry out charge transport measurements on semiconductor nanowires at liquid helium temperature. In order to navigate the four STM tips to desired positions, an SEM was used. SEM images of a test sample were used to optimize the vibration isolation of the experiment. A new nanopositioner (KoalaDrive) was developed to approach the tips to the sample. It could be shown that the KoalaDrive is capable of performing at low temperature (5 K), in ultra-high vacuum with high precision. In images taken with this STM, atomic steps on graphite as well as the 5×25 reconstruction of the Pt(100) surface could be resolved. The low-temperature multi-tip scanning tunneling microscope was used to contact single as-grown GaAs nanowires with one tip. Charging of a part of the tip holders prevented SEM images and conduction measurements with more tips.

Abstract

Contents

Kurzfassung	III
Abstract	V
I Charge Transport Regimes and Experimental Methods	1
1 Motivation	3
2 Charge Transport	5
2.1 Diffuse Charge Transport	5
2.1.1 Drude Model	5
2.1.2 Sommerfeld Theory and Boltzmann Equation	6
2.2 Ballistic Charge Transport	10
3 Experiential Methods	11
3.1 Scanning Tunneling Microscopy (STM)	11
3.1.1 1D Tunneling	13
3.2 Scanning Electron Microscope	16
3.2.1 Beam generation	17
3.2.2 Acceleration and focusing	18
3.2.3 Contrast in an SEM	19
3.3 Sample preparation	20
3.3.1 GaAs-Nanowires	20
3.3.2 Bead crystal	21
3.4 STM-Tips	22
3.5 Four-point resistance measurements	23

II	Temperature dependence of Dopant incorporation into GaAs Nanowires measured with a Room-Temperature Multi-Tip Scanning Tunneling Microscope (RT-MT-STM)	25
4	Experimental Setup	29
4.1	RT-MT-STM	29
4.2	Scanning Electron Microscope (SEM)	30
4.2.1	Contrast in an SEM	32
4.3	Vibration isolation	33
4.4	Measurement Electronics	34
5	Resistance measurements of p-doped GaAs nanowires on n-doped GaP and GaAs	37
5.1	Analyzed samples	43
5.1.1	Growth process	44
5.1.2	First overviews (SEM)	45
5.2	Two-point resistance measurements	49
5.2.1	Constant Sample (M4586)	50
5.2.2	Step Sample (M4509)	50
5.2.3	Gradient Sample (M4580)	51
5.2.4	Comparison of the Two-Point Results	51
5.3	Three-point resistance measurements	52
5.3.1	Constant Sample (M4586)	52
5.3.2	Step Sample (M4509)	53
5.3.3	Gradient Sample (M4580)	54
5.4	Four-point measurements	55
5.4.1	Constant Sample (M4586)	58
5.4.2	Step Sample (M4509)	59
5.4.3	Gradient Sample (M4580)	62
5.5	Discussion	63
5.5.1	Temperature dependence of the resistance profiles	65
5.5.2	HR-SEM	67
5.5.3	One-dimensional transport model	68
5.5.4	Common features of all samples	74
5.5.5	Surface conductance	78
5.5.6	Offset voltage	80
5.5.7	Resistance profiles	81
5.5.8	Interpretation of the non-linear voltage current relation	83
5.5.9	Peaking Resistance	87
5.6	Electron Beam Induced Current (EBIC)	88

6	A closer look on Resistance measurements using a Multi-Tip Scanning Tunneling Microscope as Nanoprober	93
6.1	Stability Measurements	94
6.1.1	Long time run	96
6.1.2	Repetitive Measurements	97
6.2	Gate Measurement	99
7	Nano Manipulation	105
7.1	Snap to contact	105
7.2	Harvesting	105
7.3	Precise cutting	106
8	Conclusion part II	107
III	The Low-Temperature Multi-Tip Scanning Tunneling Microscope (LT-MT-STM)	109
9	Experimental Setup	113
9.1	Scanning Tunneling Microscope(s)	115
9.1.1	Magneto-Low-Temperature Multi-Tip Scanning Tunneling Microscope (magneto-LT-MT-STM)	115
9.1.2	Ultra compact Low-Temperature Multi-Tip Scanning Tunneling Microscope (LT-MT-STM)	117
9.1.3	The KoalaDrive	122
9.2	Scanning Electron Microscope	124
9.3	Cryostat	127
9.3.1	Temperature Measurement	130
9.3.2	Wiring	130
9.3.3	Thermalization	132
9.4	Measurement electronics	133
9.5	Vibration isolation	133
9.5.1	Vibration Measurements	135
9.5.2	Additional measurements and final configuration	138
10	PN Sample (M4523)	141
11	Measurements	143
11.1	Electron Beam Induced Current (EBIC)	143
11.2	Multi-Tip measurements	147
12	Conclusion and Outlook part III	151

Contents

IV	Appendix	IX
	Acknowledgments	XI
	List of Figures	XII
	List of Tables	XV
	Bibliography	XVII

Contents

Part I

Charge Transport Regimes and Experimental Methods

Chapter 1

Motivation

Electronic devices are omnipresent in today's modern world. Channels of conducting paths and active electronic structures (diodes, transistors) can be realized, by adding small amounts of dopants to an otherwise nonconducting semiconductor crystal. Electronic devices can be fabricated by lithographic processes including etching and chemical vapor deposition (CVD). In lithographic processes light or electron beams are used to write electronic structures into a photo sensitive material. After etching and rinsing, the structure can be used to deposit metals on the surface or to implant additional dopants or ions into the semiconductor. Naturally lithography is limited by the width of the used beam and, thus, by its wavelength.

A different way of creating electronic devices is currently explored by using self-assembly of nanostructures. Using the characteristic of a semiconductor surface and its growth characteristics, electronic structures can be realized during the growth of the crystal rather than afterwards. The limitations of lithographic processes do not apply for self-assembly of crystals. Smaller and new structures can be fabricated in such a process, the GaAs nanowires [subsection 3.3.1](#) used in this thesis are just one example. While doping during lithographic processes is well understood and can be done with high precision and high reproducibility, the incorporation of doping atoms into the semiconductor nanowires is not well known [\[1\]](#). Further investigations are needed to fabricate high quality self-assembled nanostructures which can be used for nanoelectronic devices.

A key factor in the self-assembly of nanostructures is the temperature during its growth. Three samples with GaAs nanowires were grown with different temperature profiles. In this study, a multi-tip scanning tunneling microscope was used in order to determine the resistance profiles along these nanowires. High resolution four-point measurements were performed to eliminate the contribution of cabling and contact resistances between tips and nanowires. From the resistance profiles the doping along the nanowire axis can be calculated.

Chapter 2

Charge Transport

Ohm's law $\vec{J} = \sigma \cdot \vec{E}$ describes the linear relation between the electric field $\vec{E}$ applied to a specimen, the conductance matrix σ of the specimen and the resulting current $\vec{J}$ flowing through it [2]. As useful as this equation is for large wires, in other vicinities Ohm's law may not hold. Since electric currents are transmitted via charge transport and electric charges are accelerated quadratically in an electric field, the fact that the resulting current is linear proportional to the applied electric field is surprising. Two different regimes of charge transport can be identified. The diffuse regime is characterized by large statistics. Charges move diffuse around in the conductor and scatter with each other and impurities. The ballistic regime occurs in clean homogeneous samples, where single (or just a couple of) charges can move freely through the sample and do not (or only very rarely) get scattered.

2.1 Diffuse Charge Transport

If a wire is connected to a charge source and sink (battery), a huge difference in charge concentration is present, before a current starts to flow. The charges move according to their temperature in random directions and scatter on their way. The random movement tends to balance the concentration difference in the wire, as described in thermodynamics. Due to the large number of charges carriers and the high probability of scattering at high currents, moderate temperatures and large sample sizes instead of the behavior of every single charge carrier only the behavior of their entity can be discussed.

2.1.1 Drude Model

The first attempt to describe the mechanism of charge transport was carried out by Paul Drude in 1900 [3]. He described a conductor as a sea of electrons. The electrons move randomly through the conductor with the velocity v_{th} according to the

temperature of the conductor. For each electron moving in $\vec{x}$ direction with velocity $\vec{v}_x$ and carrying the current $-\vec{J}_x$ an electron is moving in $-\vec{x}$ direction with velocity $-\vec{v}_x$ and carrying the current $\vec{J}_x$. In this way the contribution of all the electrons is compensated by each other and no net current is flowing. As a consequence of an outer electric field a drift velocity $\vec{v}_d$ is added to the thermal velocity $\vec{v}_{th}$. Drude started by deriving the force $\vec{F}$ acting on a charge $-e$ in an electric field $\vec{E}$ and the response of the charge carrier with effective mass m

$$\vec{F} = m \frac{d\vec{v}}{dt} = -e\vec{E} - m \frac{v_d}{\tau} \quad (2.1)$$

He introduced scattering of the electrons by $-m \frac{v_d}{\tau}$ which has the form of a friction. The time τ in this equation is characteristic for the transition from the non-equilibrium to the equilibrium state after the electric field is switched off.

In this picture, the electron ensemble - and with it the scattering rate - is accelerated continuously until an equilibrium between the energy gain from the outer electric field and the energy loss due to scattering is established. In this dynamic equilibrium equation [Equation 2.1](#) reads:

$$0 = m \frac{d\vec{v}}{dt} = -e\vec{E} - m \frac{v_d}{\tau} \quad (2.2)$$

$$\Rightarrow m \frac{v_d}{\tau} = -e\vec{E} \quad (2.3)$$

$$\Rightarrow v_d = \frac{-e\tau\vec{E}}{m} = -\mu e\vec{E} \quad (2.4)$$

$$\Rightarrow \vec{J} = ne\mu\vec{E} \quad (2.5)$$

With n the total number of electrons and μ the mobility of electrons in the sample. For the first time the linear relation between current and applied voltage could be explained by Drude. But even though a reasonable result has been derived, all electrons seem to carry the current through the sample.

2.1.2 Sommerfeld Theory and Boltzmann Equation

With the development of quantum mechanics and solid state physics at the beginning of the 20th century, new insight in the nature of charge transport could be gained. Arnold Sommerfeld described the effect of an outer electric field $\vec{E}$ on the electron distribution in k -space in a conductor [\[4\]](#). In absence of an electric field the electron distribution can be approximated by a (Fermi) sphere centered at the origin of the k -space. In [Figure 2.1](#) the equilibrium electron distribution is shown as a dashed circle. An outer electric field will induce a change in the electron distribution according to:

$$\vec{F} = \hbar \dot{\vec{k}} = -e\vec{E} \quad (2.6)$$

The force $\vec{F}$ acting on the electrons leads to a shift Δk of the sphere as visible in

Figure 2.1. This displacement is growing continuously with time τ , as described in [5].

$$\Delta k = \frac{-e\tau E}{\hbar} \quad (2.7)$$

The empty states at the left side of Figure 2.1 can be filled by scattered electrons from the occupied states on the right side. In this way the scattering probability is increasing with increasing displacement Δk . By shifting electrons from the occupied states at the right to the unoccupied states on the left, scattering tends to restore the equilibrium state. At some point a new, dynamic equilibrium is established between the continuous displacement and the scattering and the displacement of the sphere stops.

2.1.2.1 Boltzmann Equation

After Sommerfeld's qualitative description, Ludwig Boltzmann started a more analytical attempt. He described the change with time of an electron distribution $\phi(\vec{k}, \vec{r}, t)$ in the presence of an electric field $\vec{E}$ [6]. Three different parts contribute to the time evolution of the electron distribution $\frac{\partial \phi(\vec{k}, \vec{r}, t)}{\partial t}$:

1. Drift $\Rightarrow \frac{e\vec{E}}{\hbar} \cdot \vec{\nabla}_{\vec{k}} \phi(\vec{k}, \vec{r}, t)$
2. Diffusion $\Rightarrow \vec{v}(\vec{k}) \cdot \vec{\nabla} \phi(\vec{k}, \vec{r}, t)$
3. Scattering $\Rightarrow \left[\frac{\partial \phi(\vec{k}, \vec{r}, t)}{\partial t} \right]_{\text{scattering}}$.

All contributions combined result in the Boltzmann equation:

$$\vec{v}(\vec{k}) \cdot \vec{\nabla} \phi(\vec{k}, \vec{r}, t) - \frac{e\vec{E}}{\hbar} \cdot \vec{\nabla}_{\vec{k}} \phi(\vec{k}, \vec{r}, t) + \frac{\partial \phi(\vec{k}, \vec{r}, t)}{\partial t} = \left[\frac{\partial \phi(\vec{k}, \vec{r}, t)}{\partial t} \right]_{\text{scattering}} . \quad (2.8)$$

To solve this integro-differential equation several simplifications are needed. The first approach [5] is to merge all scattering processes into an "average scattering" and to analyze the behavior of the system to a sudden change of the outer electric field $\vec{E}$. If the field is switched off abruptly at time t_0 , the electron distribution $\phi(\vec{k}, \vec{r}, t)$ will change to the field-free distribution $f(\vec{k})$, which is the Fermi distribution, but does not have to be. The field-free electron distribution $f(\vec{k})$ will not be restored immediately after the sudden changes in the field $\vec{E}$, the electron distribution $\phi(\vec{k}, \vec{r}, t)$ will change slowly (compared to the change in $\vec{E}$) due to scattering which is the only available mechanism to change the distribution. The time scale of this change is τ .

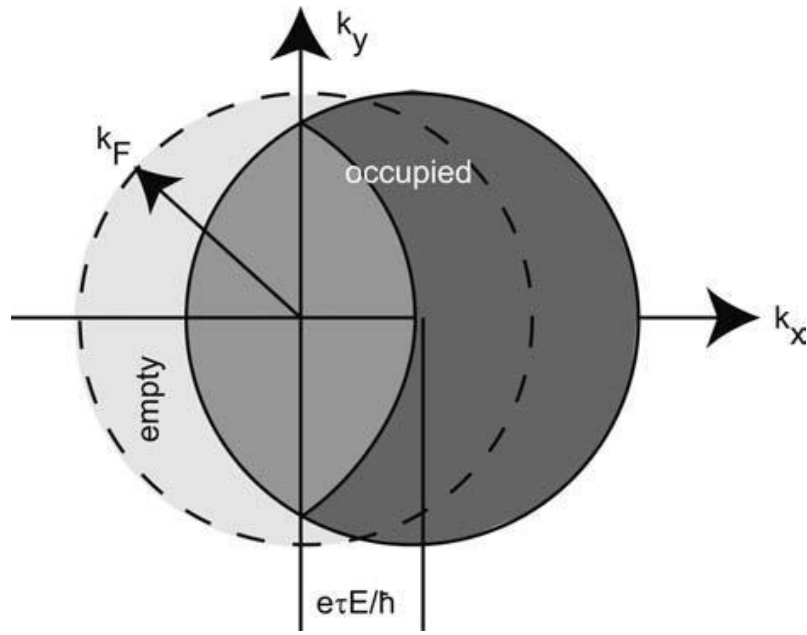


Figure 2.1: Electron distribution in k -space. In absence of an outer electric field the electrons are distributed in a sphere (dashed circle) centered at the origin of k -space. In presence of an outer electric field (dark gray) the electron distribution is shifted with respect to the equilibrium by an amount of $\frac{-e\tau E}{\hbar}$
(from [5])

For an exponential relaxation to $f(\vec{k})$ and neglecting the spacial dependence $\vec{r}$, the electron distribution $\phi(\vec{k}, t)$ can be written as a function of the electron distribution $f(\vec{k})$:

$$\phi(\vec{k}, t) = f(\vec{k}) + (\phi(\vec{k}, t_0) - f(\vec{k})) \cdot e^{-\frac{t}{\tau}}. \quad (2.9)$$

With the above simplification and $\vec{E}=0$ Equation 2.8 reads:

$$\frac{\partial \phi(\vec{k}, \vec{r}, t)}{\partial t} = \frac{\partial f(\vec{k})}{\partial t} + \frac{\partial \phi(\vec{k}, t_0) \cdot e^{-\frac{t}{\tau}}}{\partial t} - \frac{\partial f(\vec{k}) \cdot e^{-\frac{t}{\tau}}}{\partial t} \quad (2.10)$$

$$= -\frac{\phi(\vec{k}, t) - f(\vec{k})}{\tau} = \left[\frac{\partial \phi(\vec{k}, \vec{r}, t)}{\partial t} \right]_{\text{scattering}}. \quad (2.11)$$

In the homogeneous (no spacial charge differences $\vec{\nabla} \phi(\vec{k}, \vec{r}, t) = 0$) and stationary ($\frac{\partial \phi(\vec{k}, t)}{\partial t} = 0$) case and using Equation 2.11, Equation 2.8 can be simplified to:

$$\frac{e\vec{E}}{\hbar} \cdot \vec{\nabla}_{\vec{k}} \phi(\vec{k}, \vec{r}, t) = \frac{\phi(\vec{k}, t) - f(\vec{k})}{\tau}. \quad (2.12)$$

For a small electric field $\vec{E}$, $\phi(\vec{k}, t)$ can be considered as a nearly linear deviation of the Fermi function $f(\vec{k})$, so $\vec{\nabla}_{\vec{k}} \phi(\vec{k}) \approx \vec{\nabla}_{\vec{k}} f(\vec{k})$. In this case $\phi(\vec{k})$ can be written as

$$\phi(\vec{k}) = f(\vec{k}) + \vec{\nabla}_{\vec{k}} f(\vec{k}) \cdot \frac{e\tau \vec{E}}{\hbar} \quad (2.13)$$

a Taylor series in $\frac{e\tau \vec{E}}{\hbar}$. For $\frac{e\tau \vec{E}}{\hbar} \ll \vec{k}$,

$$\phi(\vec{k}) \approx f(\vec{k} + \frac{e\tau \vec{E}}{\hbar}) \quad (2.14)$$

is a good approximation. The electron distribution ϕ (including the above approximations) is identical to the Fermi function f , shifted by $\frac{e\tau \vec{E}}{\hbar}$. In Figure 2.1 f (dashed circle) as well as ϕ (dark gray) are depicted.

A dynamic equilibrium of the electric force due to $\vec{E}$ and scattering is established in the sample. Electrons from the high energy end of the electron distribution ϕ are repositioned in Figure 2.1 by scattering events. Electrons can only be (elastically) scattered as long as a adequate unoccupied state exists; As described by Sommerfeld.

A drawback of this evaluation is the treatment of scattering. Different sources of scattering and their effect cannot be distinguished. τ differs for different materials, samples, even for different enviroments (pressure, temperature etc.).

2.2 Ballistic Charge Transport

At low temperatures and very homogeneous samples (at least on the length under investigation) the probability of scattering events is considerably reduced. If in a sample the mean free path is much larger than the size of the sample, charge carriers can be thought of as propagating unchanged through the entire sample. This regime of charge transport is called ballistic. The pure ballistic case is one of the extremes of charge transport and can be achieved on small lengths. Nevertheless for sufficiently reduced temperatures and good samples, the probability of scattering can be reduced and features can be observed, which originate the ballistic regime.

Chapter 3

Experiential Methods

In the following the experimental methods used in this thesis will be briefly explained. In [chapter 4](#) and [chapter 9](#) the arrangement of the components is shown. A huge difference in size gaps from structures in every days life to the size of the atomic structures under test in present surface science. [Figure 3.1](#) shows a scale down to the atomic scale ($\text{\AA}$), starting at the typical every days size meter. Also some steps in between are presented to get a feeling for the size of an atom. A human eye can see things as small as 0.1 mm, to image smaller things, an optical microscope can be used. The resolution of an optical microscope is basically limited by the wavelength of the used light as shown in [Figure 3.2](#). Electrons can have, depending on their energy, smaller wavelengthes as light and so can resolve smaller structures down to several nanometer. Atomic resolution can be achieved with either a Transmission Electron Microscope (TEM) or a Scanning Probe Microscope (SPM). Many different types of scanning probe microscopes have been developed. In common, all SPM's have a small sensor that can be moved perpendicular to the surface of a sample. The sensor is sensitive to the physical attribute of the sample that has to be mapped. Using piezo ceramics, the sensor is moved perpendicular to the sample and the acquired data is recorded. The data are typically presented is form of a picture, where the color of a pixel corresponds to the amplitude of the measured data. Here only the scanning tunneling microscope (STM), used in the experimt is described. The other scanning probe microscopy methods are described in either [\[7\]](#), [\[3\]](#), [\[8\]](#), [\[9\]](#), [\[10\]](#), [\[11\]](#), [\[4\]](#), [\[12\]](#) or [\[13\]](#).

3.1 Scanning Tunneling Microscopy (STM)

The Scanning Tunneling Microscope (STM) was the first SPM developed. Gerd Binnig and Heinrich Rohrer invented the first STM at the IBM Zürich Research Laboratory in 1981 ([\[14\]](#)). Five years later in 1986 they were honored with the Nobel Price in Physics for this invention.

The sensor of an STM, called tip, is a "simple" conducting wire prepared to have a

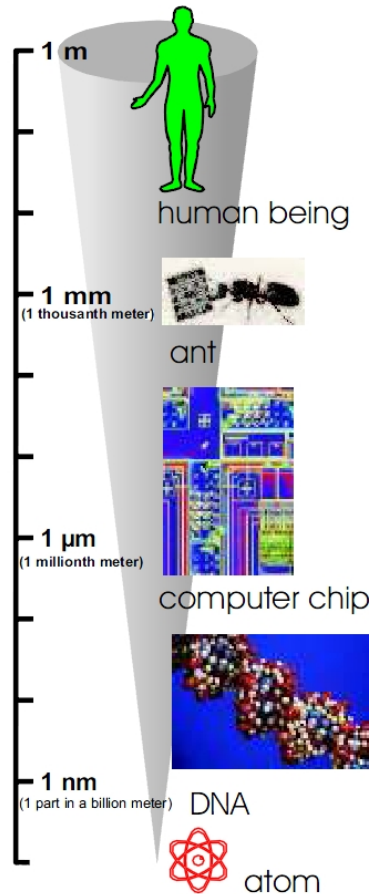


Figure 3.1: Different sizes from everyday scale meter down to the Å regime. An ant is tiny ($\sim \text{mm}$) compared to a human ($\sim \text{meter}$). The very same ant is typically one million times larger than the human DNA ($\sim \text{nm}$). The size of an atom however is in the Å regime [7].

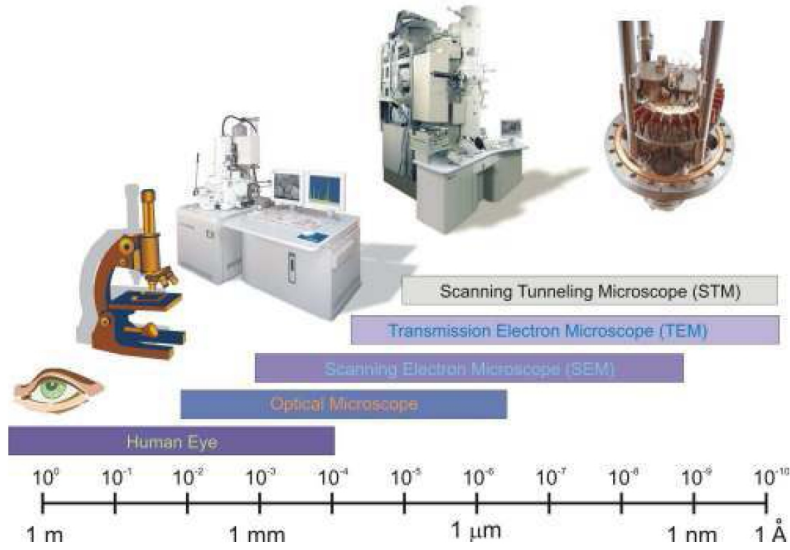


Figure 3.2: From the human eye to the STM; working areas of different types of microscopes. Different types of microscopes differ in many ways, not only in the here presented working area presented here. Also effort and time needed to generate a picture, the costs for the microscope and the working environment are important facts to find the matching microscope [7].

very sharp end (see section 3.4). Ideally the tip has only a single atom at the end. A voltage is applied from tip to sample. The STM tip is brought close to the surface of the sample, until a predefined current -typically in the nA range- is flowing. During scanning, this current is kept constant by a feedback system. To kept the current constant, the hight of the tip (z-signal) is adjusted using piezos carrying the tip. The resulting picture displays the z-signal at each point of the picture. Alternatively, the extension of the piezos can be kept constant. In this case the measured current will be displayed in the picture.

If the tip approaches to the sample in a vacuum, classically no current can flow as long as tip and sample do not touch. In a quantum mechanic treatment this situation changes. Outside of a conductor the probability of finding an electron is different from zero. To illustrate the basic principal of the STM the Schrödinger equation shall be solved for a *one-dimensional problem single electron* in the following.

By varying the applied voltage or the height of the STM tip, data additional to the topography can be gathered with an STM. Since the measured currents and the distance between sample and tip are very small, small disturbances, like vibrations or even heat can have a large influence.

3.1.1 1D Tunneling

Each particle can be treated as a wave as pointed out by quantum mechanics ([15]). In Figure 3.3(a) a one-dimensional rectangular barrier as model for tunneling is

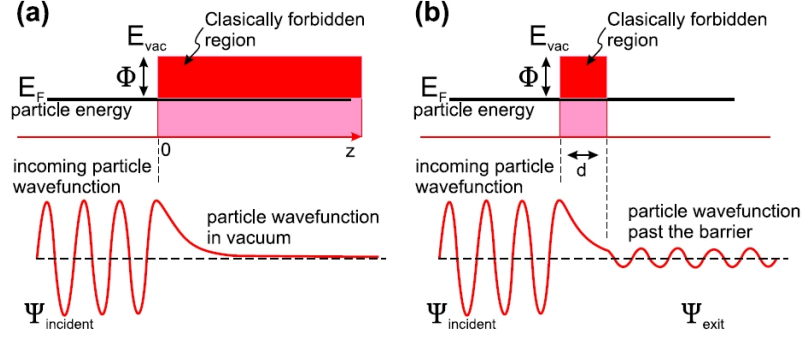


Figure 3.3: An electron with energy E_F hits a potential barrier of height $A (> E_F)$ - upper panels. In a quantum mechanical description, it can penetrate the barrier and the electron wave functions (in the lower panels) result. If the thickness of the barrier is large compared to the wave length of the electron (a), like for a transition from a conductor into the vacuum, the wave function decays. But if the distance is small (b), like in a conductor vacuum conductor configuration (STM), also an oscillating wave function can be found behind the barrier [7].

shown. An electron (or general a particle) coming from the left side in [Figure 3.3](#) with energy E_F is impinging on a rectangular barrier with height E_{vac} and thickness ∞ for (a) and d for (b). Case (a) is a model for the end of a one-dimensional wire, while case (b) represents an STM, for example.

For this "simple" problem Schrödinger's equation:

$$\frac{\hbar^2 \partial^2}{2m \cdot \partial z^2} \Psi(z) = (V(z) - E) \Psi(z) \quad (3.1)$$

with $V = E_{vac}$ inside the barrier and 0 elsewhere, can be solved. As an ansatz, the wave function Ψ can be approximated as a plane wave e^{ikz} for electrons in the conductor. After carrying out the differentiation and cancelling the wave function Ψ , [Equation 3.1](#) reads:

$$-\frac{\hbar^2}{2m} k^2 = V - E \Rightarrow k = \sqrt{\frac{2m}{\hbar^2} (E - V)}. \quad (3.2)$$

Outside of the barrier, [Equation 3.2](#) results in an oscillating wave when inserted into the ansatz. Inside the barrier, where $V > E$ the ansatz leads to a exponential decay ([16]), as can be seen in [Figure 3.3\(a\)](#). One feature of the exponential decay outside of the barrier is that the probability of finding an electron $|\Psi|^2$ drops fast, but never vanishes totally. If the potential barrier has the finite length d , as depicted in [Figure 3.3](#), the wave function goes back to being an oscillation wave behind the

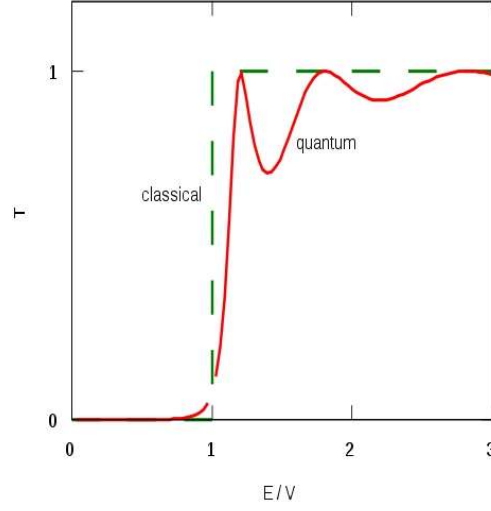


Figure 3.4: Comparison of quantum mechanical transmission probability of a finite potential barrier (Solid line) shown in Figure 3.3 and the classical result for the same situation (Dashed line). Quantum mechanics predict a non zero transmission also for $E < V$, while the classical result is a perfect step function. For $E > V$ the quantum mechanic result show oscillation which are not present in the classic case [17].

barrier. The wave function differs in the three different areas of the problem.

$$\Psi(z) = \begin{cases} A \cdot e^{\frac{ipz}{\hbar}} + B \cdot e^{-\frac{ipz}{\hbar}} & \text{for } z < 0 \\ C \cdot e^{-kz} + D \cdot e^{kz} & \text{for } 0 < z < d \\ A \cdot S(E) \cdot e^{-\frac{ip}{\hbar}(z-d)} & \text{for } d < z \end{cases} \quad (3.3)$$

$$\text{with } p = \sqrt{2mV} \text{ and } \hbar k = \sqrt{2m(V - E)}$$

The coefficients A to D and $S(E)$ have to be determined that, at the boarder of the barrier ($z=0$ and $z=d$), the wave function is continuous and continuously differentiable. One of the coefficients can be freely chosen, so $A=1$. On the left side ($z<0$), as well as in the barrier ($0<z<d$), the solution is a superposition of electrons coming from the left and electrons reflected at the two boarders of the barriers. No electrons are entering from the right side of Figure 3.3(b). $S(E)$ is the tunneling matrix and can be written as:

$$S(E) = \frac{2i\hbar k p}{2i\hbar k p \cdot \cosh(kd) + (p^2 - \hbar^2 k^2) \cdot \sinh(kd)}. \quad (3.4)$$

The probability of finding an electron is $\Psi(z)\Psi^*(z) \equiv T(E)$ and in this example proportional to the tunneling current. $T(E)$ is the so called transmission coefficient.

According to Equation 3.3 and Equation 3.4, for $T(E)$ follows:

$$T(E) = \Psi(z)\Psi^*(z) = (A \cdot S(E) \cdot e^{-\frac{ip}{\hbar}(z-d)}) (A \cdot S(E) \cdot e^{-\frac{ip}{\hbar}(z-d)})^* \quad (3.5)$$

$$= \frac{|S(E)|^2}{|A|^2} = |S(E)|^2 = \left[1 + \frac{4(\frac{E}{V}) \cdot (1 - \frac{E}{V})}{\sinh(kd)} \right]. \quad (3.6)$$

In Figure 3.4 $T(E)$ is compared to the classic transmission coefficient, which is zero for $E < V$ and one for $E > V$. $T(E)$ starts to grow at electron energies smaller than the barrier height. At higher energies $T(E)$ approaches one and starts oscillating, as shown in Figure 3.4.

For electrons with a de Broglie wavelength (Equation 3.8) much smaller than the barrier thickness, Equation 3.6 can be further simplified:

$$T(E) = 16 \frac{E}{V} \left(1 - \frac{E}{V} \right) \exp \left(-\frac{2}{\hbar} \sqrt{2m(V-E)} d \right) \quad (3.7)$$

At Equation 3.7, some of the features of STM's can be seen. Most important may be the fact, that the tunneling probability decays exponentially with growing barrier thickness d , or tip sample gap. For an STM tip with a single atom at the end, only this last atom can be considered to participate in the charge transport, because of this exponential decay.

The work function $\Phi = (V - E)$ enters as square root in an exponential function. For electrons from lower energy levels than the Fermi level E_F , the energy gap grows and the transmission coefficient decreases.

3.2 Scanning Electron Microscope

The coarse movement of the four STM tips is realized by slipstick motion, as described in section 4.1. Each tip can move freely in X-, Y- and Z-direction. The relative position of the tips is in general unknown. To contact a nanostructure with all four tips the position of each tip has to be known with high precision. Figure 3.2 shows an rough overview over the working regimes of different types of microscopes. Tip exchange and coarse approach require a field of view in the millimeter regime, while contacting requires a high resolution. In some experiments ([18] for example), optical microscopes with resolution $\gtrsim 1 \mu\text{m}$ are used. To determine the tip positions with higher precision, a scanning electron microscope (SEM) can be used, as it is done in this experiment.

In an optical microscope, for example, only details comparable in size or larger than the wavelength can be resolved. Light has a wavelength in the range of 380 nm to 780 nm. To resolve smaller details a shorter wavelength is necessary. The same is true for an SEM. The resolution of an SEM depends on the energy of the electrons

as can be seen in [Equation 3.8](#). In the used SEM (*FEI BDS 200 2LE*) electrons are accelerated by an electric field of 5 kV up to 25 kV. The electrons have a wavelength in the range of 10^{-11} m. With higher acceleration voltages, higher resolution is possible. Other effects than the wavelength of the electrons limit the resolution of an SEM to the nanometer range.

Electrons are not purely particles but waves too, as described in quantum mechanics. In many experiment (found partly in [\[19\]](#) for example) the wave nature of electrons has been proven and effects like diffraction of electron waves are widely used. Accordingly a wavelength λ (De-Broglie-Wavelength) can be assigned to a particle with mass m and energy E :

$$\lambda = \frac{h}{\sqrt{2mE}} \quad (3.8)$$

with h being the plank constant.

In the following a short overview of the basic physical principles of scanning electron microscopy shall be given.

3.2.1 Beam generation

Any light being reflected from the sample into the optics of an optical microscope is used to image the sample. A scanning electron microscope (SEM) on the other hand has to produce and accelerated an electron beam to image the sample by itself. The electrons are generated by heating up a single-crystal, zirconium oxide coated tungsten emitter with $\langle 100 \rangle$ orientation [\[20\]](#). The electrons in the emitter crystal gain energy by heating the emitter. Once an electron has enough energy it can tunnel (see [subsection 3.1.1](#)) out of the emitter into the vacuum. The barrier electrons have to overcome to leave the crystal is called work function. The hotter the emitter is the more electrons leave the emitter. Additionally a voltage is applied to the emitter.

Not only the work function is reduced by the applied voltage but the electron are emitted and accelerated in a preferred direction, too. Since the electrical field strength is highest at the tip of the emitter most electrons are emitted there. The emitted electrons are filtered before they are accelerated. So all the electrons have the same energy. The mono energetic electron beam is then accelerated and focused subsequently.

First of all free electrons have to be "produced". A voltage applied between emitter and its surrounding (called extractor) can pull electrons out of the emitter. The energy barrier an electron has to overcome to leave a metal is called workfunction. It describes the energy difference between the energy of the electron occupying the highest energy level in the crystal and an free electron. In principal the workfunction is a material property but the number of emitted electrons depend on several additional parameters. Shape and material of the emitter have a big influence, for example. It is advantageous to take a sharp metal tip as emitter. Additional manipulation of the crystal's energy bands increases the flux of electrons.

In the described experiment a zirconium-oxide covered tungsten tip is heated to

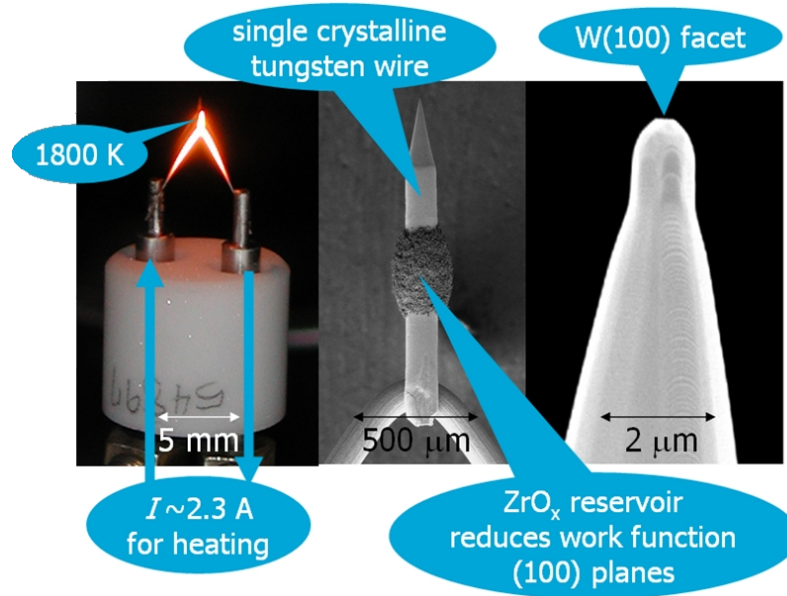


Figure 3.5: The thermal field emitter (TFE) is the origin of the electrons that are used in an SEM. By heating a small single crystal tungsten wire with a current up to 1800 K, the thermal energy of the electrons in the wire is increased. To reduce the work function, the wire is covered with an additional layer. In order to generate an electron beam in the preferred direction, the tip of the TFE is a (100) tungsten facet. [21]

$\sim 1800^\circ\text{C}$. A voltage up to 9000 V (typically 5740 V) sucks the electrons out of the emitter.

3.2.2 Acceleration and focusing

After the electron beam is produced and accelerated to the desired energy, the beam has to be focused. Electro-magnetic fields are applied to the electrons on their way through the SEM gun.

The electrons emitted by the gun have to be focused on the sample by a set of lenses. These lenses generate an electromagnetic field forcing the electrons to focus on the sample surface. Additional to the emission and the focusing of the electrons, they have to be deflected. The SEM hosts several pairs of plates for deflecting the electron beam. Two of these deflection plates create an electrical field to deflect the electron beam leaving the SEM gun. A picture is taken by sweeping the focal point across the sample surface using the deflection plates. At each point the response of the sample to the electron bombardment is different. The different response is recorded. The for now unspecified response of the sample will be discussed in the following.

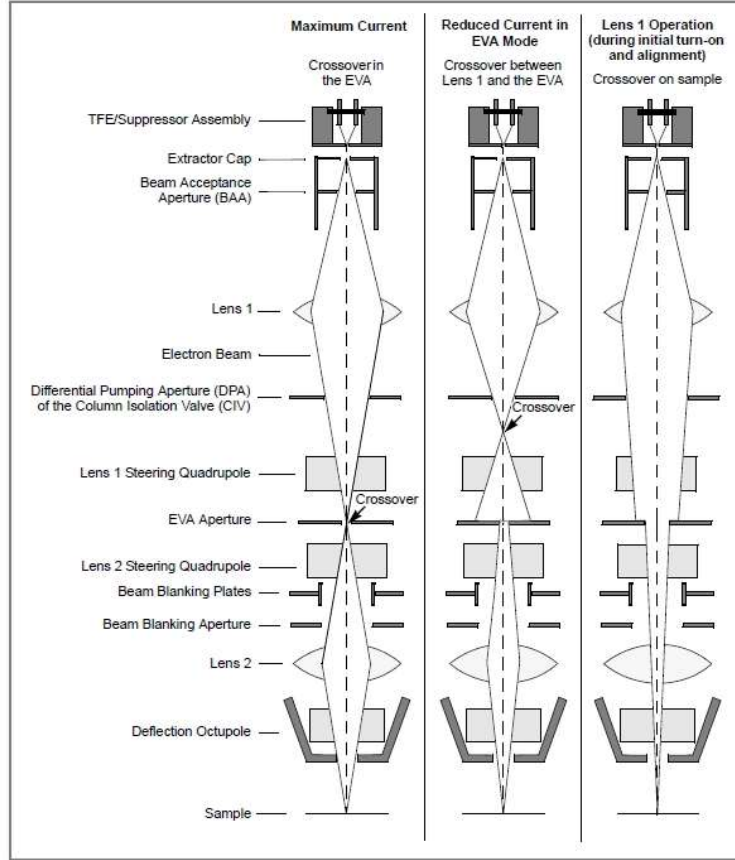


Figure 3.6: The schematic drawing the SEM column from the SEM users guide [20] is shown. The electron beam is generated in TFE on top. On the way to the sample (bottom), it is focused twice. Between the two electrostatic lenses, the beam undergoes a crossover. To scan the focal point over the surface, a deflection octupole is installed at the end of the column.

3.2.3 Contrast in an SEM

At the sample the electrons emitted by the SEM interact with surface atoms and remove electrons out of their shells. These electrons are called secondary electrons. The sample is connected to an electrical ground, so the void in the atomic shell can be refilled. The secondary electrons are collected by a detector. The detector amplifies the collected charge by a factor of roughly 10^5 (*HAMAMAZSU Electron Multiplier R5150-10*). The number of secondary electrons is displayed as contrast of the resulting image.

A second way of imaging is to measure the amount of charge deposited in/ extracted from the sample by the electron gun. The sample is connected to ground via a low noise current amplifier (Femto DLPCA-200). The amplifier converts the charge flowing from the sample to ground or vice versa into a voltage signal and amplifies it by up to $10^9 \frac{V}{A}$. The current rate is recoded as a function of the beam position.

3.3 Sample preparation

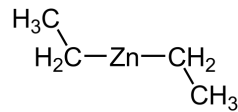
In the course of this thesis two different kinds of samples were used. In [Part II](#) a room-temperature scanning tunneling microscope is used as a nanoprobe to perform charge transport measurements on freestanding, as-grown GaAs nanowires ([subsection 3.3.1](#)). Such a nanowire sample is also used as specimen for a low-temperature multi-tip scanning tunneling microscope in [Part III](#). Additionally a Pt(100) bead crystal ([subsection 3.3.2](#)) is used for calibration measurements.

3.3.1 GaAs-Nanowires

The nanowire samples have been prepared in metal-organic vapor phase epitaxy (MOVPE)[[22](#)], at the university of Duisburg-Essen [[23](#)]. Substrates, highly p-doped ($\sim 1 \times 10^{19} \text{ cm}^{-3}$) with zinc are used. For the experiments described in this thesis, GaP (gallium-phosphide) and GaAs (gallium-arsenide) substrates in (111) orientation were used.

After the substrate is cleaned, gold particles are deposited from colloidal solution as nuclei for the growth of nanowires. Then the sample is introduced into an AIX200 RF reactor. By annealing the sample at 600°C for 5 minutes in a nitrogen atmosphere (50 mBar), the gold particles are melted and start to absorb gallium atoms from the substrate. An eutectic system from gold and gallium is created.

To enable conductivity in the intrinsically nonconducting GaAs, a dopant has to be included into the nanowire. All nanowires analyzed in [Part II](#) are p-doped with zinc, like the substrate, but with a lower charge carrier density. In [Part III](#) nanowires with a n-p junction in the middle are introduced. Nevertheless, the base of all nanowires is p-doped. Before the nanowire growth can be started, the eutectic gold-gallium droplet has to be saturated with the dopant. At 450°C reactor temperature, diethyl zinc (DEZn)



is added into the nitrogen atmosphere. Due to the high temperature in the reactor (450°C) the DEZn decomposes to free Zn and residual gases. The free zinc then can be absorbed into a gold-gallium droplet[[24](#)].

The actual nanowire growth is initiated by adding the two additional precursor gases Trimethylgallium (TMGa) and tert-Butylarsine (TBAs). Alike DEZn, TMGa and TBAs decompose due to the high temperatures. When one of the gases comes into contact with the hot surface, free gallium or arsine is created. The two components diffuse over the surface of the sample until they are absorbed into a gold droplet. In the droplet, gallium and arsine react to form GaAs. When a GaAs molecule comes

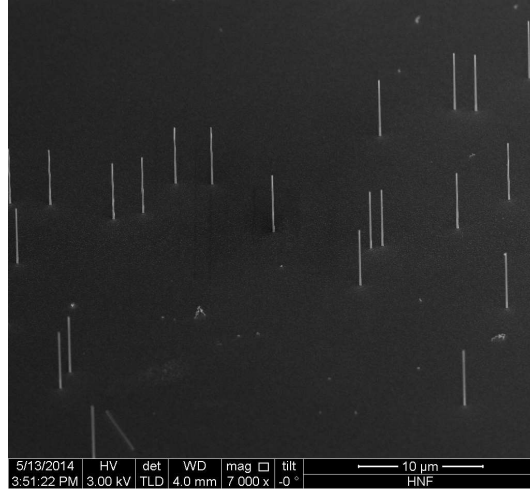


Figure 3.7: SEM image of a nanowire sample. Several upright standing nanowires are visible on the substrate.

into contact with the substrate, it is incorporated into the substrate crystal directly beneath the gold droplet. When more and more GaAs is deposited, the gold droplet is pushed upwards; the nanowire growth.

To grow straight, upright standing nanowires, a approx $1\text{ }\mu\text{m}$ high base of the nanowire is grown at 450°C . At 450°C the nanowires do not only grow vertical, but horizontally too. To reduce the unintended horizontally growth (called tapering), the temperature is reduced to 400°C for the rest of the nanowire is growth.

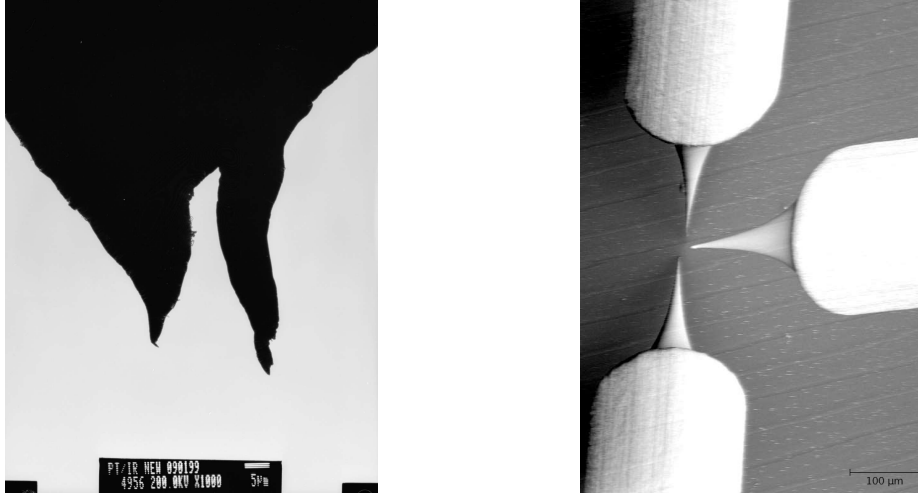
While at 400°C (nearly) the entire DEZn is decomposed ([25]), only a part of the other two precursor gases is still intact. The amount of gallium and arsine, which is available for nanowire growth cannot only be tuned by adjusting the flow of the precursor gases but via the temperature too. Only a few K difference can increase or decrease the resistance of a nanowire by an order of magnitude([26]).

An SEM image of a nanowire sample (Constant Sample) grown in such a process can be seen in Figure 3.7.

3.3.2 Bead crystal

Bead crystals can be formed from a wide choice of metals and metal alloys. A Pt(100) bead crystal was used to perform calibration measurements with the low-temperature magneto scanning tunneling microscope. It was fabricated in the Grünberg-Institut 3 of the Research Centrum Jülich according to a process described in detail in [27].

By melting a metal wire, a liquid droplet or bead of metal is created. During the careful cooling, a single crystal bead is formed.



(a) An STM tip produced by cutting [30] (b) Four-point measurement configuration

Figure 3.8: To bring the STM tips during multi-tip measurements as close as possible to each other the shape of the tip plays an important role. The tip in (a) was produced by cutting. Its irregular shape prevents the realization of a multi tip measurement configuration such as shown in (b).

3.4 STM-Tips

Each conducting material can be used to fabricate a tip for scanning tunneling microscopy ([28]). For the resolution of an STM it is crucial to form the tip as sharp as possible. Depending on the material, different techniques are known to fabricate sharp tips mostly from metal wires.

For different applications, different tip types are beneficial ([29]). At ambient conditions, for example, a slow oxidizing tip can be obtained by cutting a wire made of Platinum-Iridium alloy ([30]).

For multi-tip measurements also the shape of the STM tips is very important. Pt-Ir tips that were fabricated by cutting have a asymmetric and irregular shape. In Figure 3.8a a transmission electron microscope image of a Pt-Ir tip for an STM is shown. While high resolution STM images can be taken with such a tip, it is impossible to bring two (or even more) of such tips close together, to perform multi tip measurements.

Tungsten tips, that were fabricated by electrochemical etching show a symmetric and regular shape. In Figure 3.8b an SEM image of three tungsten tips is shown. Due to the conic shape of the tips, it is possible to bring them very close together to perform multi tip measurements on very small objects.

The STM tips used for the measurements presented in this thesis were fabricated according to a direct-current (dc) drop-off electrochemical etching procedure based on a paper by J.P. Ibe et al [31]. A tungsten wire with a diameter of $250\text{ }\mu\text{m}$ is dipped in a NaOH solution. Together with the wire, a steel contact is placed in the solution. A voltage of 8 V is applied between the wire (anode) and the steel contact

(cathode). The resulting OH^- -ions react with the tungsten of the wire to WO_4^{2-} and hydrogen. The heavier WO_4^{2-} sinks down at the wire cutting of the supply with fresh OH^- . As a consequence, the etching only takes place at the interface between solution and air, resulting in the conic shape. After about 10 minutes, the wire is etched so much at the interface, that the lower part of the nanowire falls off. At that point, the etching is finished.

During the etching process a current of approx 15 mA is flowing between the tip and the steel cathode. When the lower wire falls off, this current is suddenly reduced to about 5 mA. An electronic is installed to detect the sudden change in the flowing current and cuts off the applied voltage, to prevent the etching process from continuing.

When the etching is successfully finished, the tip is cleaned and a first inspection with an optical microscope is done.

3.5 Four-point resistance measurements

In everyday life resistances are measured using a multimeter. Two metal tips, for example, are pressed against the contacts of a resistor and the result can be read from the display of the multimeter. The multimeter applies a small voltage between both tips and measures the resulting current. The resistance is calculated from these two values (see [Figure 3.9b](#)).

If one takes a closer look, not only the designated resistance is shown, but the resistance between the two inputs of the multimeter. When the resistance of the wires and the contacts is small compared the resistance that shall be measured (as it usually is in everyday life) the result is close enough. But if this is not the case, the result is useless. In STM ([section 3.1](#)) contact resistances are in the order of up to several $\text{G}\Omega$.

To avoid measuring the sum of all the resistances, a four-point resistance measurement (see [Figure 3.9b](#)) can be performed. By adding two additional measuring points ($2 \rightarrow 4$) the current path can be separated from the voltage measurement. A current is injected into the resistor. The voltage necessary to create the current is according to the sum of the resistance of the cabling, the contacts and the resistor itself.

The voltage measurement can be performed totally current free. Its input resistance is practically infinite (up to $10 \text{ T}\Omega$). With the absence of a current flowing in or out of the voltage measurement, the resistances in this path have no influence on the result.

In this way, the voltage drop is measured only over the designated resistor, while the current flow is left unaltered. In [Figure 3.9](#) a sketch of the two measurement techniques is shown.

In both cases, the measured resistance is a fraction of the measured voltage over the injected current $\text{Resistance} = \frac{\text{Voltage}}{\text{Current}}$. While the current through the resistor is the total current in both cases (2 or 4 point measurement), the measured voltage differs. In a 4 point measurement, only the voltage drop over the designated resistor

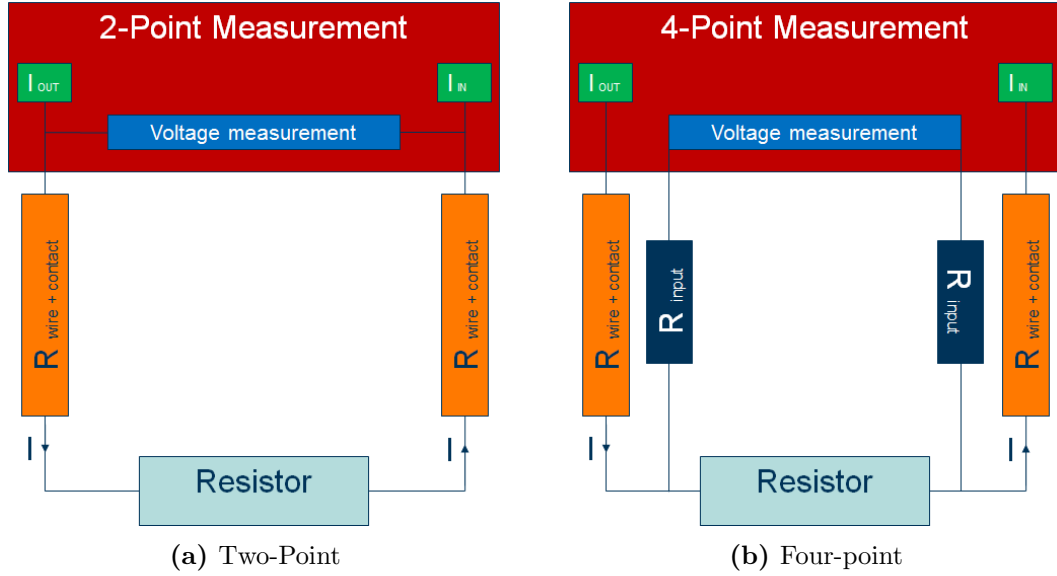


Figure 3.9: Difference between two- and four- point resistance measurements

is taken into account, while in a 2 point measurement uses the voltage drop over the entire system (resistor + cables + contacts).

The above discussed method can be applied for one-dimensional cases, like the nanowires in [Part II](#) and [Part III](#). For two or more dimensional cases, the interpretation is more complicated. In principle, also the ratio of the flowing current and the measured voltage drop is derived, but when the current can spread in a layer or volume, not all the current will pass through the region where the voltage drop is measured. First the current density has to be modeled and the fraction of the current in the voltage measurement region has to be computed before the result can be interpreted.

Part II

Temperature dependence of Dopant incorporation into GaAs Nanowires measured with a Room-Temperature Multi-Tip Scanning Tunneling Microscope (RT-MT-STM)

At the university of Duisburg-Essen GaAs-nanowires were grown for next generation solar cell applications. Before such devices can be produced, it is necessary to understand the fabrication process. For such applications, the electronic properties of the nanowires have to be adjusted carefully. During the fabrication of semiconductor devices the control of doping is essential to control the electronic properties of the device ([32]).

In this part of the thesis, the experimental findings are presented that were obtained with a room-temperature multi-tip scanning tunneling microscope (RT-MT-STM), equipped with a scanning electron microscope (SEM). With this device, four-point resistance measurements can be performed along single nanowires, without detaching the nanowires from the substrate they were grown on (“as-grown”). While the resistance measurements are carried out with the RT-MT-STM, the SEM is used to provide spatial resolution on the nanowire to determine the measurement configuration (positions of all STM tips) for each measurement.

In a preceding study, Dr. Stefan Korte investigated the influence of different substrates on the doping profile of p-GaAs nanowires with the same machine [33] [34]. The nanowire samples have been prepared by *metal-organic vapor phase epitaxy* (MOVPE) by Matthias Steidl from the University of Duisburg-Essen using an AIX200-RF reactor [23]. To obtain conductivity, the nanowires were p-doped with zinc. The process of growing nanowires used here is based on a process proposed by Joyce in paper [35].

In the course of Korte’s measurements, it turned out that the influence of the substrate on the resistance profile of a nanowire is negligible, offering a broad spectrum of possible material combinations for further studies. The features found in the resistance profile along the nanowires could be associated with the temperature step during the production process. The dopant concentration could be calculated using a charge transport model.

In the current study, the influence of the substrate temperature during the growth process on the doping concentration in the nanowire is investigated. The temperature pattern was identical to that of the samples used by Korte. In the current study, three different samples with different temperature patterns were analyzed, p-doped GaAs nanowires on n-doped GaP or GaAs substrates.

The samples will be described in more detail in [section 5.1](#). First, a short overview over the experimental setup is presented in [chapter 4](#). The scientific basics for the applied experimental method are described in [chapter 3](#), while a description of the measurement itself can be found in [chapter 5](#). The charge transport experiments are described in sections [5.2](#) to [5.4](#) for the three samples sorted by measurement complexity. The results are discussed in [section 5.5](#).

During the four-point charge-transport measurements, an influence of the position of the SEM beam on the conduction measurements was discovered. This feature is further investigated in [section 5.6](#). Emphasis is put on the influence, the measurement has on its result and the reliability of this method in [chapter 6](#). At the end of this part, some additional features and capabilities of the machinery used are presented in [chapter 7](#).

Chapter 4

Experimental Setup

The room-temperature multi-tip scanning tunneling microscope (RT-MT-STM) has been developed in the Forschungszentrum Jülich. It has been extensively used and upgraded by several PhD students. The present setup is described in detail in [33], [34] and [36]. To give the reader a basic idea of the experiment, a short overview over the device shall be given in the following section.

4.1 RT-MT-STM

The RT-MT-STM combines four individual STM units in one system. Additionally, it is equipped with an SEM. Each STM unit can move its tip freely in X and Y direction approximately 2 mm as well as 5 mm in Z direction.

The design of the RT-MT-STM is based on the beetle-type STM by Besoke [37]. Each STM tip is fixed to a ring. This ring is resting on three tube piezos. If the piezo tubes bend (slowly) in the same direction, the ring moves. Also rotations can be performed by having all piezos moving along the same circle.

Since the bending capability of a piezo tube is quite limited, only small movements ($\sim 8 \mu m$) can be performed in this way. To travel larger distances slip-stick motion has to be performed. If the piezo tubes bend fast enough, the friction between piezo and tube is not strong enough to move the ring, the ring starts to slide over the piezos. If the bending is now carried out several times back and forth, while one of the directions is fast and the other is slow, the ring will move only in one, the slow, direction. In this way the ring can be moved distances much larger than the bending range of the piezos tubes. One cycle of a slow motion in one direction and a fast motion in the opposite direction is referred to as a step. On top of each piezo a ruby half sphere is glued in order to have a low and defined friction between ring and tube. But still this friction can be different at each point of the ring. Consequently, each step has a different size and slip-stick motion is quite imprecise, but effective. In the original beetle-type design a large scale vertical motion is realized by rotating along ramps. These ramps are shaped into the lower side of each ring. Unfortunately,

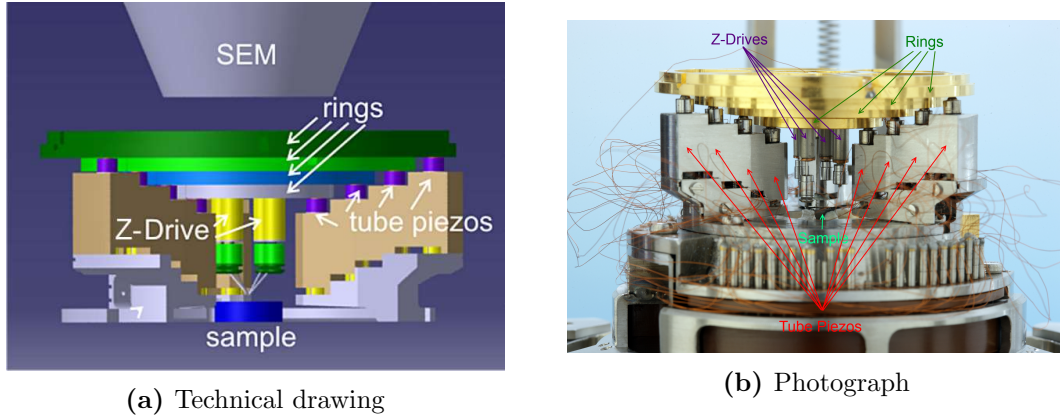


Figure 4.1: In (a) the technical drawing of the RT-MT-STM is shown. The four rings can be seen as well as the STM tips, the sample and the SEM. In (b) a photograph of the real RT-MT-STM is showing the same features except for the SEM. [36]

each vertical movement also results in a horizontal movement. For small, precise Z-motions the piezo tubes can contract or expand.

In this design, Z motion is decoupled from XY motion with a so-called Z-drive based on slip-stick motion. The STM tip holder is mounted on top of a rod. This rod is held by two springs, one at each end of a piezo tube. To move the rod, and with it the STM tip, along the piezo tube axis, the piezo extends (or contracts) slowly and contracts (or extends) very fast. During the slow movement the friction between the springs and the rod is strong enough to hold the rod in its position (relative to the spring). When the piezo tube moves fast, the frictional force is too small to hold the rod and it starts gliding. As a consequence, the position of the rod relative to the tube changes. The rod makes a step.

In Figure 4.1 the RT-MT-STM can be seen from the side. Figure 4.1a shows a technical drawing of the RT-MT-STM. The four rings (dark green, light green, light blue and silver) are resting on three tube piezos. Due to the support structure only the bottom part of the scanning tube piezos is visible (yellow) as well as their top cover (violet). In the center of the figure the Z-drives can be found. Here, only two of four are depicted due to perspective. Directly above the sample (dark blue), the four STM tips are shown. In Figure 4.1b a photograph of almost the same view is shown. Except for the SEM, all features that are shown in the technical drawing are also visible in this photo.

4.2 Scanning Electron Microscope (SEM)

For sample and tip exchange, as well as for determining the position of the four STM tips relative to each other and to the sample, high resolution sight onto STM and the sample is essential for operating an MT-STM. For sample and tip exchange the

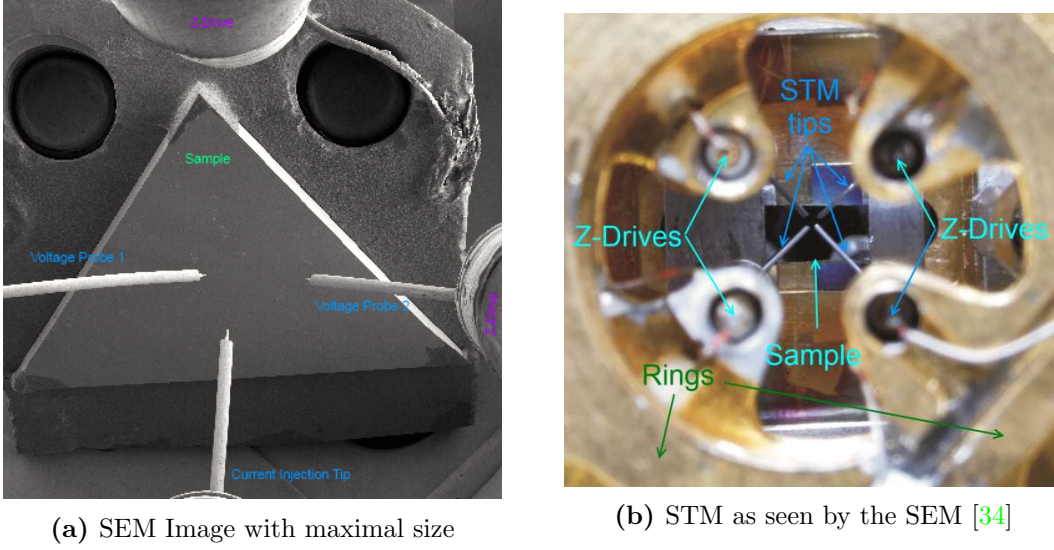
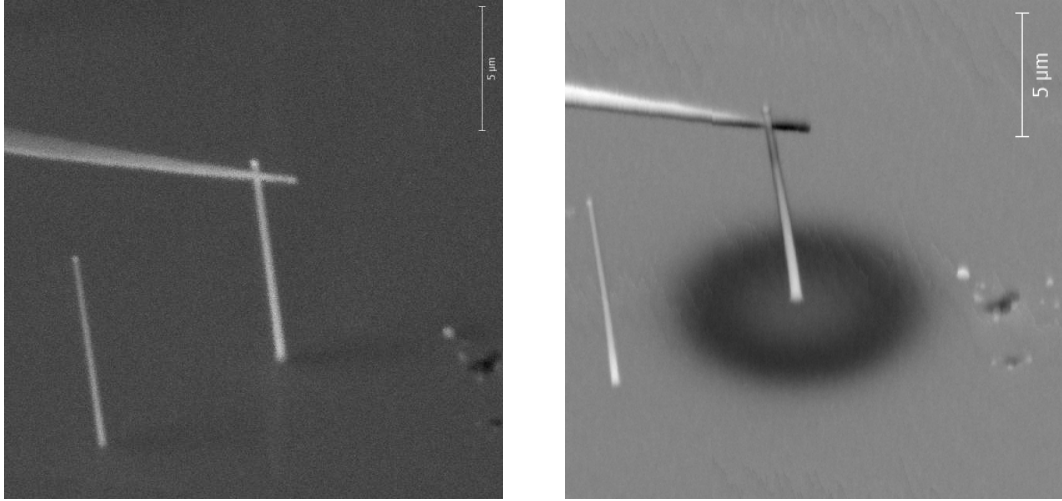


Figure 4.2: In (a) the maximum field of view of the SEM is shown. Such images are used for tip and sample exchange. A triangular sample, three STM tips and two of the Z-Drives are shown. In (b) a photograph of the view the SEM has on a (rectangular) sample is shown. Here, additionally, the inner ring and a small part of the second ring are visible.

resolution is not critical, but a relatively large area ($\gtrsim 10 \text{ cm}^2$) needs to be visible. The STM tips can be positioned with subatomic precision, but their position over the sample and relative to each other can only be determined by an additional device. In some experiments [18], optical microscopes with resolutions of $\gtrsim 1 \mu\text{m}$ are used. In order to contact structures with more than one STM tip, a view onto the sample is needed. Consequently, only structures comparable in size to the resolution of the microscope can be contacted for measurements.

For higher resolutions a scanning electron microscope (SEM) is used in the presented experiment. It is installed on top of the RT-MT-STM, looking downwards onto the sample. In section 3.2 the theoretical background of an SEM is described. The resolution of an SEM depends on the energy of the electrons as can be seen in Equation 3.8. In the SEM used here (*FEI BDS 200 2LE*) electrons are accelerated by an electric field of 4 kV up to 25 kV. With higher acceleration voltages, a higher resolution is possible.

In Figure 4.2a, the triangular sample can be seen along with three installed STM tips. On top, the fourth z-drive is present without a tip, since the fourth contact is realized by the substrate. Conducting glue is used to attach the sample to its holder, an electrically isolating wedge. The sample is tilted 45° and isolated from the STM ground. To contact the sample, a wire is glued to this wedge, close to the sample (top right in Figure 4.2a). The same conductive glue is used to attach sample and wire to the wedge and build an electric connection between the two. Figure 4.2b shows a photograph taken from the perspective of the SEM. A different sample is installed and all four tips are mounted.



(a) SEM image taken with a secondary electron detector

(b) SEM image taken using the sample current as contrast

Figure 4.3: The two images show the same nanowire contacted by the same STM tip. Also, bias and SEM focusing are kept constant. The only difference is the signal used as contrast for the SEM image. Different features are visible (nanowire shadows or dark ring) due to the different contrast mode. Also, the form of the nanowire seems different in the two pictures. Depending on the application it is advantageous to use the one or the other.

While [Figure 4.2a](#) shows an SEM image with maximal size, [Figure 4.3b](#) shows a zoom close to the maximum resolution. A GaAs nanowire is put in contacted with an STM tip. The nanowire has a length of roughly $6\text{ }\mu\text{m}$ and a smallest radius of $\lesssim 45\text{ nm}$. The size of the small particles on the right side is $\sim 30\text{ nm}$.

Thus, the SEM meets the requirements mentioned previously. It can provide large scale images for tip and sample exchange as well as high resolution images to define the tip configuration during a four-point resistance measurement on GaAs nanowires.

4.2.1 Contrast in an SEM

Electrons emitted by the SEM interact with surface atoms of the sample and remove electrons out of their shells. These electrons are called secondary electrons. The sample is connected to an electrical ground, so the void in the atomic shells can be refilled easily via the wire.

The first way of imaging is to collect the secondary electrons with a detector. The detector amplifies the collected charge by a factor of roughly 10^6 . In an SEM image, the number of secondary electrons is displayed as a function of the position of the focal point of the SEM.

A second way of imaging is to measure the amount of charge deposited in or extracted from the sample by the electron beam. The sample is connected to ground via a low noise current amplifier (Femto DLPCA-200). The amplifier converts the current

flowing from the sample to ground or vice versa into a voltage signal and amplifies it by up to $10^9 \frac{V}{A}$. The current rate is recorded as a function of the beam position. In [Figure 4.3](#) two SEM images with different contrast modes are shown. The two images were recorded shortly one after another and show the same “setup”. The two nanowires along with the STM tip and the particles on the right side are visible. In the Sample Current image an extra feature is present that cannot be found in the SED image. The dark ring around the nanowire base as well as the color shifts on the nanowire and the tip are present. This effect will be further discussed in [section 5.6](#). Also, on the SED image a feature is present that is missing on the Sample Current picture. Secondary electrons that are generated behind an obstacle in the line of sight to the detector cannot reach it directly. This creates the effect of a shadow on each nanowire.

At first glance the Current image seems to be sharper than the SED picture. Background (white) noise decreases the contrast and blurs out the edges. Sample current images, on the other hand, suffer from 50 Hz noise, omnipresent in the current to volt amplifier. Hence, the same nanowire shows different structures in the two images in [Figure 4.3](#). While the contacted nanowire for example seems to be fairly straight in the SED picture, its diameter is clearly increasing top to bottom in the sample current image.

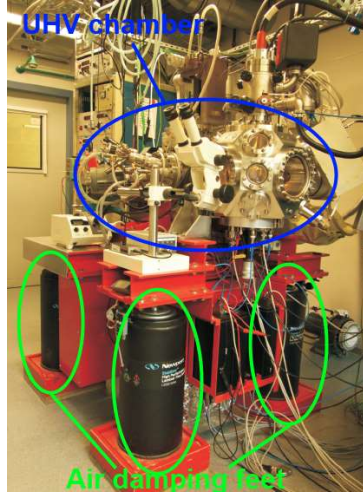
Since the structure is known to have a nearly constant radius from images taken with high resolution SEM ([subsection 5.1.2](#)), one may conclude that the SED pictures are advantageous for measurements, while the Sample Current images show clearer structures and can be used to adjust the focusing. Hence, the SED images are used for four-point resistance measurements in the next sections.

4.3 Vibration isolation

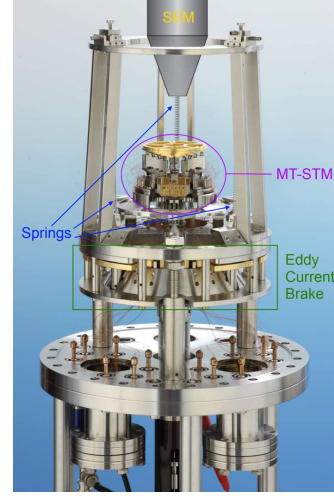
The vibration isolation of the RT-MT-STM is twofold. To isolate it from vibrations, the entire UHV chamber rests on air damping feet. Additionally the STM is damped inside the measurement chamber by a spring suspension.

People walking by, closing of doors, pumps, other electrical devices produce a lot of vibrations that can travel long distances through the ground. To decouple the experiment from these vibrations, the UHV chamber hosting the RT-MT-STM is resting on four Newport air damping feet ([Figure 4.4a](#)).

In [Figure 4.4b](#) the RT-MT-STM is shown with the second damping stage. The STM can be pressed down to be tightly fixed to the chamber. It is used for tip and sample exchange as well as for SEM imaging. Since the STM is tightly attached to the chamber, disturbances like sound can easily translate into the STM. To decouple the STM from the chamber, it can be hung from three springs. The springs act like the damping feet. To prevent oscillations of the STM eddy current damping is installed. Intrinsic oscillations induce an eddy current which damps the STM vibrations. To perform resistance measurements with more than one tip, the SEM resolution is crucial, so the standard configuration in this work is to press the STM against the chamber.



(a) The UHV chamber of the room temperature multi-tip STM



(b) The room-temperature multi-tip STM and its vibration isolation

Figure 4.4: The RT-MT-STM is isolated from vibrations in two stages. (a) The entire UHV chamber stands on air damping feet, reducing the influence of vibration running through the floor. The second stage is a combination of three springs and an eddy current damping. The STM is hanging on springs, while the eddy current damping damps the intrinsic motion of the spring.[36]

While the spring suspension is active, motion of the RT-MT-STM relative to the SEM is possible. In this configuration, the SEM resolution is reduced. For high quality SEM images, the spring suspension is disabled, while it is active during high resolution STM measurements.

4.4 Measurement Electronics

Each of the four STMs is equipped with a complete set of measuring electronics. Its tip can be moved freely and perform measurements independently of the other three STM units, as demonstrated in [36].

To perform four-point measurements (section 3.5), the STM tips have to fulfill different tasks. A current has to be injected and withdrawn from the sample, while at the same time a voltage has to be measured. To inject a current, a conducting structure has to be in contact with an STM tip (called **Current Injection Tip**). In this experiment the current is withdrawn through the sample, alternatively a second Current Injection Tip could be used. A voltage must be applied between the Current Injection Tip and the sample to initiate the current flow. In this experiment the flowing current is measured on the sample side as well as on the STM side. To increase the sensitivity of transport measurements, the additional resistance in the current path has to be minimized.

To measure the voltage between two STM tips (called **Voltage Prober**), on the

other hand, the input resistance has to be infinite ($10\text{ T}\Omega$ in this experiment), to cancel out additional current paths.

These functions have been combined into the so called "black box" as described in [34]. Each tip and the sample are equipped with such a black box. To maintain flexibility, each tip is capable of measuring currents as well as voltages. During a measurement, each tip is in one of two modes (current measurement mode and voltage measurement mode). For a different measurement the tip configuration can easily be changed.

In current measurement mode, the potential of each tip can be adjusted between $\pm 10\text{ V}$ with respect to the common ground, independently of the other tips. In voltage measurement mode the tips are floating.

In both modes the output of the black box is a voltage signal proportional to its input. These up to five voltage signals are fed into further devices for recording and analysis. To measure potential at an STM tip without additional load, the voltage source is decoupled inside the black box by the $10\text{ T}\Omega$ input resistor and a voltage amplifier. The gain of this amplifier is typically 1, but can also be raised to 100.

For current measurements a low noise current to voltage amplifier (Femto DLPCA-200) is installed in the black box. With a variable gain between $\frac{10^3\text{ V}}{\text{A}}$ to $\frac{10^{11}\text{ V}}{\text{A}}$, an input current is transformed into an output voltage signal.

Chapter 5

Resistance measurements of p-doped GaAs nanowires on n-doped GaP and GaAs

The multi-tip room-temperature scanning tunneling microscope (RT-MT-STM) is used as a nanoprobe to measure the resistance profiles of as-grown p-GaAs nanowires on n-GaP or n-GaAs substrates in order to investigate the influence of the temperature during the growth of nanowires.

Several preliminary tasks are necessary before four-point resistance measurements on “as-grown” GaAs nanowires can be performed. Once the sample is prepared, it has to be mounted on an appropriate sample holder. One of the advantages of working with as-grown nanowires is that one of the four contacts is already present via the sample and only three additional contacts have to be realized by STM tips. During a measurement the positions of these three STM tips have to be controlled using the SEM. In order to be able to measure the position of a tip on a nanowire, the nanowire has to be visible in the SEM not from the top, but from an angle as shown in [Figure 5.1](#). The sample is mounted in 45° to the SEM plane. The three STM tips are mounted in a 45° angle to be able to contact the entire nanowire without shading the sample from the SEM (also see [Figure 4.2a](#)). The first tip is used to inject a current into the nanowire. It contacts the nanowire at the gold particle at its top, where the best ohmic contact can be established. In the following, this tip will be referred to as **Current Injection Tip**. The other two tips are used to measure the local potential along the nanowire. One Voltage Prober (tip 2) is positioned stationary near the Current Injection Tip. It will be called **Stationary Voltage Prober** henceforward. While the third STM tip (**Voltage Scanner**) sweeps along the nanowire step by step in voltage measurement mode.

Once the sample is installed inside the multi-tip STM, the area in which the mea-

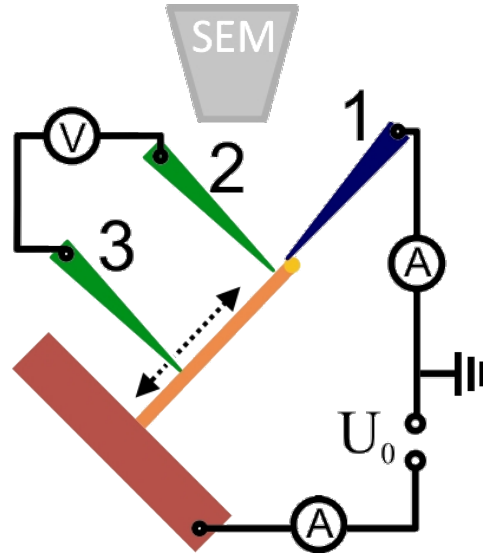


Figure 5.1: A nanowire is contacted at four points. Three contacts are realized by STM tips while the fourth is the contact on the back of the substrate. A current is injected into the nanowire through tip 1 (dark blue) and flows out through the substrate or vice versa. Tips 2 and 3 (green) measure the potential difference between two points on the nanowire.

measurements shall take place has to be identified. This area has to be reachable by all three tips and has to contain straight nanowires spaced wide enough to be accessible by all tips. Subsequently the tips can be moved to that area using coarse movement and be approached to the sample using the conventional STM feedback based auto-approach. This is the state shown in Figure 5.2. From an SEM image overlooking the designated area, a nanowire can be chosen for four-point resistance measurements. This nanowire can then be contacted by the three STM tips. While the lateral position of the STM tips can directly be displayed in the SEM image, the height over the substrate is not known. To adjust the height of a tip the 45° angle between the sample and the SEM plane can be used. For each point on the nanowire, there is a line on the sample that has the same height. Each tip can be approached on a point on the sample with the correct height, simply by mirroring the designated contact point on the base of the nanowire.

In the direct neighborhood of a nanowire only fine movement (offset) must be used, since uncontrolled movement and vibrations during coarse motion can easily break a nanowire. First the Current Injection Tip shall be brought into contact. Since the SEM image cannot show if a contact is established or not, another way of detecting a contact is needed. Therefore, a sufficient voltage has to be applied to the tip, while it is in current measurement mode section 4.4. As soon as a contact is established, a current will flow through the tip, the nanowire and the sample. This current can be monitored using an oscilloscope, for example. In principle, the two voltage measurement tips could also be brought into contact in this way. But since (at first)

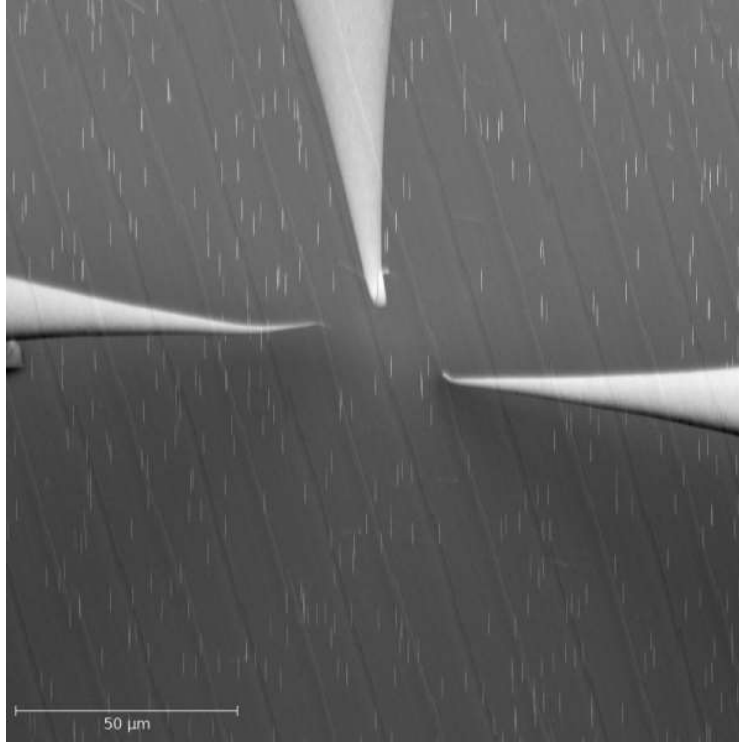
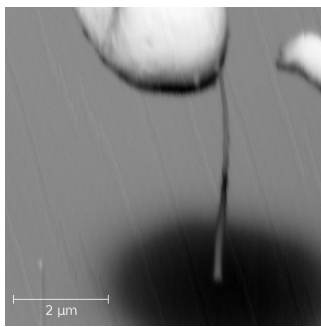
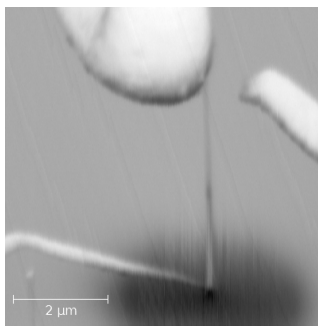


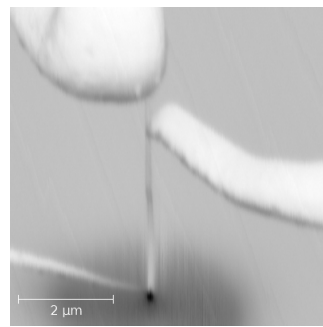
Figure 5.2: Before a nanowire can be contacted, all tips have to be brought close to that nanowire. Here an overview is shown. All tips are close to the sample and each other. Starting from such a configuration a nanowire can be chosen for measurement.



(a) first contact



(b) second contact



(c) third contact

Figure 5.3: A nanowire is contacted subsequently by three STM tips. In principle the order of contacting does not matter, but the order shown minimizes the probability of breaking the nanowire. First the nanowire is contacted at the gold particle with the Current Injection Tip. Afterwards the two Voltage Probers tips are contacted, starting with the one closer to the substrate.

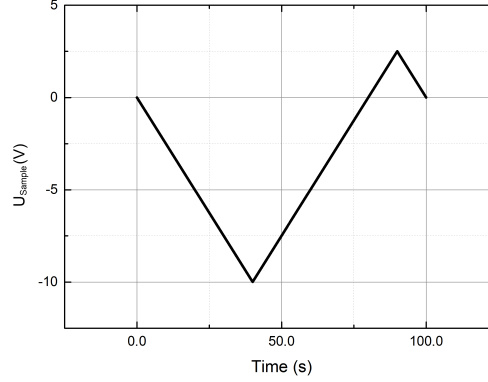
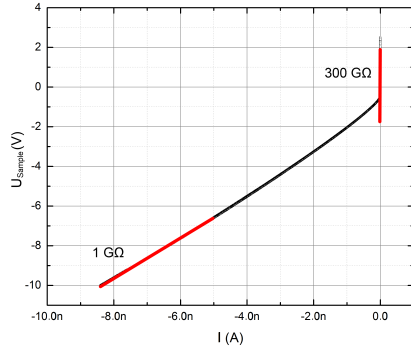


Figure 5.4: Voltage pattern applied to the sample during a four-point resistance measurement on the Gradient Sample. The voltage pattern for the other two samples is similar, but fine tuned to the two-point resistance.

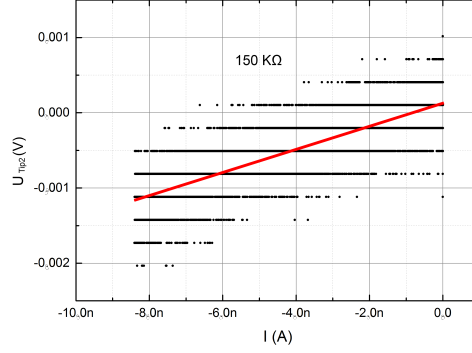
the resistances between the tips and the nanowire are unknown, the current path is unknown too. If, for example, the contact resistance of the additional tip is very high, only a very weak current may flow into that tip and a contact may not be recognized. In order to create a sufficient contact, the two remaining tips can be contacted in voltage measurement mode, as they will be used later on. Therefore the voltage output of the Black-Box (see [section 4.4](#)) has to be monitored while the tip approaches the nanowire. The potential of a tip in voltage mode is undefined unless it is in contact with a defined potential. As soon as tip and nanowire are in contact, the potential of the tips gets pinned to the potential of the nanowire at the contact point. In [Figures 5.3a to 5.3c](#) a sequence of pictures shows the procedure of contacting a nanowire seen through the SEM in Sample Current mode (see [subsection 4.2.1](#)).

A four-point resistance measurement is carried out by driving a current through the nanowire with the Current Injection Tip and measure the voltage drop between the two other tips. In order to generate the current a voltage is applied between Current Injection Tip and sample. In principle, a constant voltage can be used to perform a four-point resistance measurement. Higher precision, however, can be achieved by applying a voltage pattern. In these experiments the Current Injection Tip is grounded while the potential of the sample is changed. In [Figure 5.4](#) the pattern applied to perform four-point resistance measurements on the Gradient Sample is shown.

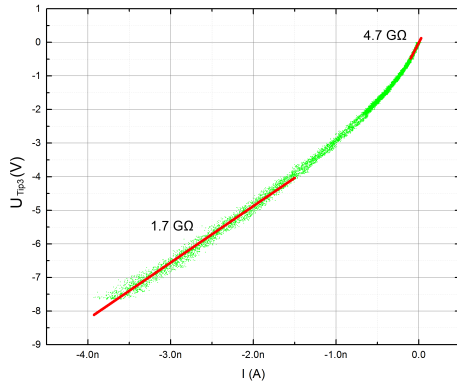
The measured two-point resistance is the sum of the resistances of the wires to the Current Injection Tip, the tip itself, the tip nanowire contact, the sample and the wire to the sample. It can be calculated from the two-point IV, shown in [Figure 5.5a](#). An IV shows the voltage drop over a resistance as a function of the current flowing through this resistor. The slope of a linear fit on an IV equals the resistance ($R = \frac{V}{I}$). For the two-point IV the voltage applied to the sample is plotted as a



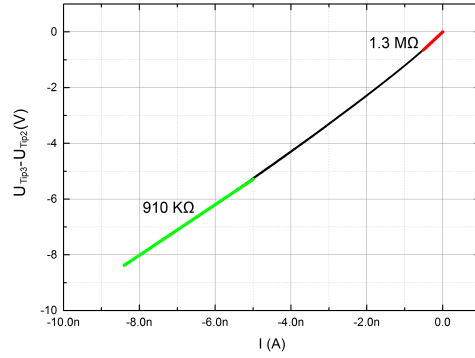
(a) Two-point IV



(b) Voltage at the upper tip



(c) Voltage at the lower tip



(d) Four-point IV

Figure 5.5: The different signals of a four-point resistance. A voltage pattern (Figure 5.4) applied between tip 1 and the sample (see Figure 5.1) typically results in a current flowing through the nanowire as shown in (a). The voltage measured by tip 2 as a function of the current is plotted in (b). The linear fit can be used as a measure to monitor the contact resistance of tip 1. Likewise, (c) displays the voltage at tip 3. In (d) the voltage difference between tip 2 and tip 3 is plotted as a function of the flowing current. The two linear fits represent the resistance of the nanowire section between the two tips in two different bias regimes.

function of the measured current through the nanowire (or the sample, since these two currents are identical). The form of the IV contains a first piece of information on the electronic structure. The IV of a perfect ohmic resistor, for example, is a straight line emerging symmetrically from 0 A @ 0 V, while a diode shows an asymmetrical voltage current relation.

In order to reduce the parts contributing to the measured resistance and to gain information on specific parts of the nanowire, several three-point IV can be calculated from the measured data. The voltage difference between either of the two voltage tips and the sample or Current Injection Tip (grounded) can be plotted against the current flowing through the Current Injection Tip and the sample. For all measured IV the I is always the same, since the same current flows through the entire system, according to Kirchhoff's current law [38]. In [Figure 5.5b](#) the voltage difference between the (grounded) Current Injection Tip (grounded) and the Stationary Voltage Prober (tips 1 and 2 in [Figure 5.1](#)) is plotted.

The resistance obtained by the fit in the I/V curve is the sum of three convoluted resistances. Beside the small nanowire part between the Current Injection Tip and the Static Voltage Prober, the nanowire tip contact and the wire from the Injection Tip to the Black Box also contribute to the three-point resistance. While the resistance of the wire is small compared to the other contributions, the main resistance arises due to the contact and the little segment of the wire. Since the resistance of the nanowire is constant, this IV can be used to monitor the contact between the Current Injection Tip and the nanowire and estimate an upper limit for the contact resistance.

In order to measure the resistance of a part of a nanowire without any other additional resistances involved, a four-point measurement is needed, as described in [section 3.5](#). A corresponding IV is shown in [Figure 5.5d](#). The difference between the signals of the two voltage probes ([Figure 5.5b](#) and [Figure 5.5c](#)) is plotted as a function of the measured current. Since the input resistance of a Black Box in voltage measurement mode is $\sim 10\text{ T}\Omega$, the voltage measurement is nearly completely decoupled from any current flow, so no contact resistances play any role. The IV shown in [Figure 5.5d](#) shows a nearly linear behavior. It has been fitted at both ends of the graph with a linear function to get the slope (=resistance) in these bias regimes.

Such IVs are measured along the nanowire to generate a resistance profile. One Voltage Prober is positioned stationary near the Current Injection Tip, while the other Voltage Prober sweeps along the nanowire step by step. At each point at least three IVs are measured. If the quality of the IVs is not sufficient, or they show different behaviors, additional IV are measured until a coherent picture of the resistance at that position can be made. Step by step, a resistance profile of a nanowire can be created. At each point the resistance shown in such a profile ([Figure 5.6](#)) is a measure for the segment of the nanowire between the two Voltage Probers tips. In the resistance profile, the result of a fit is shown at the position of the Voltage Scanner. The resistance near the base of a nanowire, for example, has to be interpreted as the resistance of nearly the entire nanowire, since the Stationary Voltage Prober is

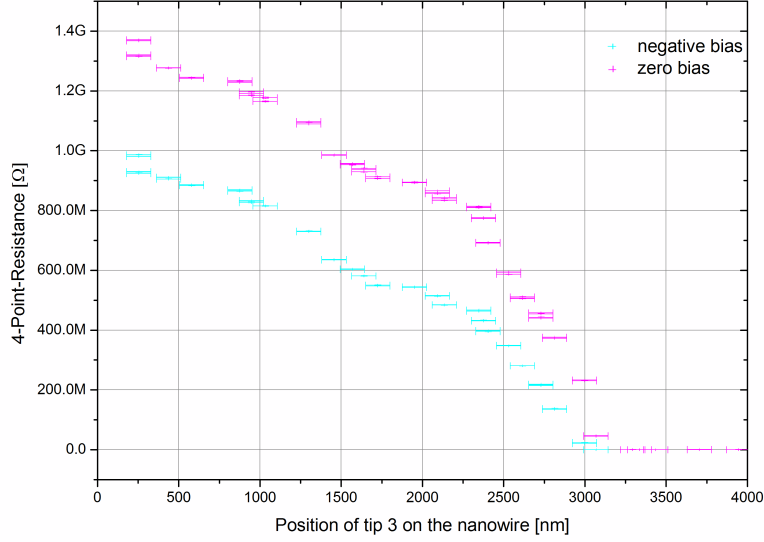


Figure 5.6: The resistances profiles at 0 A and -5 nA of a nanowire from the Gradient Sample is shown. According to two fits in [Figure 5.5d](#), two resistance profiles can be generated.

always positioned at the top of the nanowire.

Since the diameter of the nanowire is known, the resistance profile can be translated into a doping profile, using the transport model presented in [\[33\]](#). Additionally, the space charge layer thickness, the charge carrier mobility and the specific resistance can be calculated. Before the results of the measurements are presented, a closer look at the samples used is presented.

5.1 Analyzed samples

Three samples were prepared at the university of Duisburg-Essen in the course of these experiments. p-doped GaAs nanowires were grown on all three of them. As substrates n-GaAs and n-GaP were used.

The three different samples are sketched in [Figure 5.7](#). These icons and names will be used in the following to distinguish the different samples. The nanowires were grown according to a pattern based on the publication by Joyce et al [\[35\]](#). A key aspect of this pattern is to reduce the temperature once a sufficient base is grown, to obtain straight, single phase nanowires. The electrical characteristics were not studied in detail in [\[35\]](#).

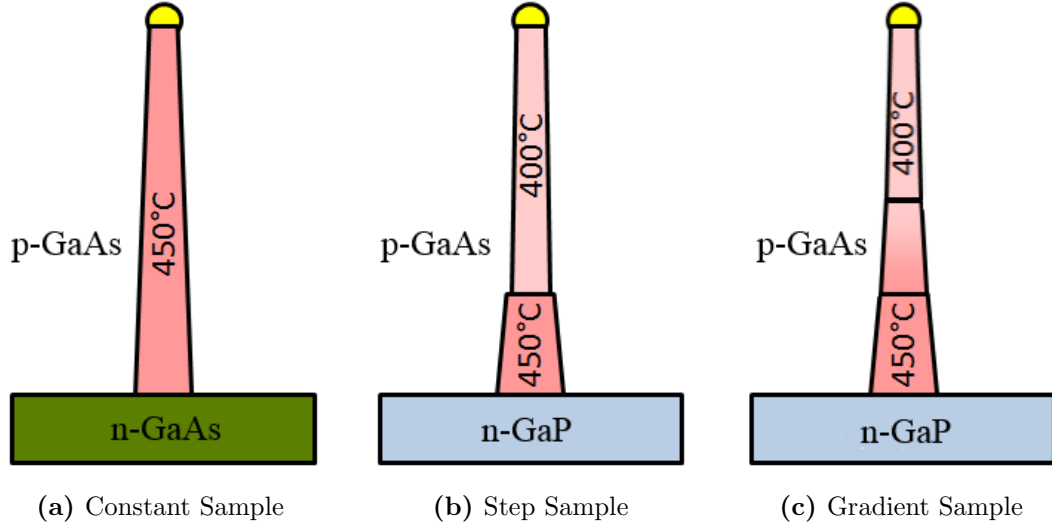


Figure 5.7: Shown here are the icons of the three different types of nanowires that are discussed in the following chapter. Each sample contains p-GaAs nanowires grown on n-GaP or n-GaAs. The different temperature regimes are indicated for in the corresponding parts of the nanowire as well as indicated by the color. At 450°C tapering is supposed to occur. This is also indicated in the icons.

5.1.1 Growth process

Semiconductor nanowires made of a variety of materials can be grown in different ways on different substrates. The nanowires measured in this work are made from a gallium and arsenide compound (GaAs). Combined, these two elements build a III/V-semiconductor. Zinc is added in a II/III ratio of 0.002 as positive dopant to control the conductance of the otherwise very pure conducting GaAs nanowire. The nanowires are grown on either a Gallium-Phosphide (GaP) or Gallium-Arsine substrate, two III/V semiconductors with very similar properties. The substrates are highly doped with phosphor. In [section 3.3](#) the preparation of the used samples is described in more detail. For the growth of the nanowire and the incorporation of dopants the temperature plays a crucial role. Starting nanowire growth at a high temperature ($\gtrsim 450^\circ\text{C}$) results in straight nanowire bases normal to the substrate, while nanowire bases grown at 400°C tend to grow in different directions. To reduce tapering (horizontal growth of the nanowire) the temperature should be reduced ($\lesssim 400^\circ\text{C}$) after a sufficient base for the nanowire has been grown.

For further applications not only the form and crystalline structure of the nanowires have to be perfectly tuned, but especially the doping profile. In order to learn more about the influence of the reactor temperature on the incorporation of dopants into the nanowire, three different samples were produced with different temperature settings.

The first sample was grown at 450°C entirely ([Figure 5.7a](#)). For the other two samples, the temperature was reduced from 450°C to 400°C . The nanowire growth was

switched off during the cool down on the second sample, so it can be treated as a sudden temperature step (Figure 5.7b). During the temperature change while preparing the third sample, the nanowire kept growing. The temperature was gradually decreased over 5 minutes, corresponding to a nanowire length of approximately 1800 nm (Figure 5.7c).

As nuclei for nanowire growth, gold particles were deposited on the sample. Before it has been annealed at 600°C for 10 minutes in the reactor to assure a clear and flat surface. After annealing, the gold particles were supposed to be exposed to zinc at 450°C to presaturate them with the dopant. Unfortunately, the line which supplies diethyl zinc to the reactor was not filled properly before the procedure. Consequently, the gold particles were insufficiently saturated with the dopant.

The growth process is started by supplying trimethyl gallium and tertiary butyl arsine. These gases decompose at the surface. While gallium and arsine stay on the surface, the rest of the molecules evaporates. Afterwards, the components diffuse over the surface until they are absorbed into a gold particle. In the gold particle, Ga and As are combined to GaAs and added to the nanowire crystal. Additionally, diethyl zinc should constantly be present in the reactor. As trimethyl gallium, it decomposes on the surface, offering Zn to the reaction. Since the supply line for diethyl zinc was not filled before the growth procedure started, no zinc was present at the beginning of the nanowire growth. During the time, the diethyl zinc needed to travel through the supply line, build up the designated partial pressure and saturate the gold seeds only low to non doping can be achieved.

In order to get straight nanowires, it is recommended [35] to start at a temperature of $\geq 450^\circ\text{C}$. However, at that temperature, nanowires grow not only vertically, but horizontally too. This so called tapering effect is sketched in Figure 5.7. The nanowire bases are wider than their top parts due to longer widening.

For the Constant Sample the temperature was kept at 450°C, and tapering was accepted. For the other two, the temperature was reduced to 400°C for the rest of the procedure, after a proper base (ca. 1 μm) was grown.

During the temperature change from 450°C to 400°C, the nanowires on the Gradient Sample were growing normally, while the trimethyl gallium and tertiary butyl arsine supply was cut off for the Step Sample. As a consequence, the nanowire on that sample could not grow further during the temperature change. A temperature profile along a nanowire will show a sudden step, hence the name.

Due to the insufficient presaturation of the gold particles at the beginning of the growth process, the doping profile of the nanowire base is unknown. The doping of the other nanowire parts is assumed not to suffer from this fabrication error.

5.1.2 First overviews (SEM)

Prior to transferring each sample into the RT-MT-STM, high resolution SEM images were taken at the Forschungszentrum Jülich by Dr. Elmar Neumann (Step Sample and Constant Sample) or at the university of Duisburg-Essen by Matthias Steidl (Gradient Sample), in order to get an idea of the dimensions of the nanowires, such as length and diameter. Such images can be used to perform measurements on

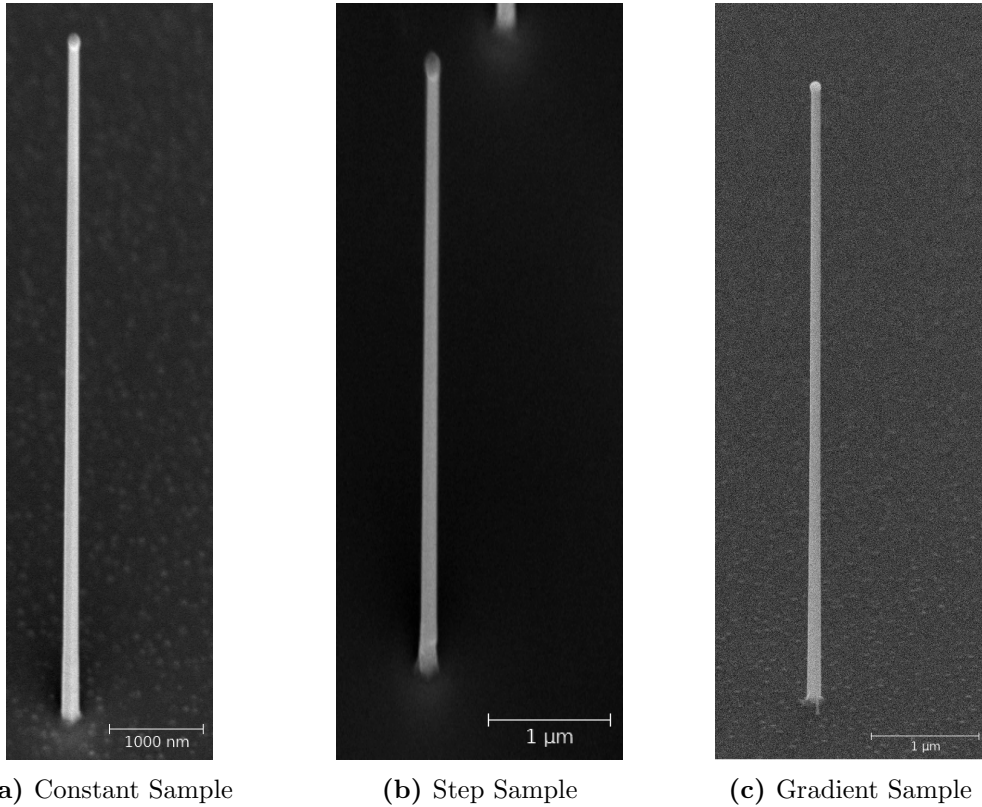


Figure 5.8: High resolution SEM images of the three samples before resistance measurement. A single nanowire is shown in each picture. Due to previous calibrating of the SEM these pictures can be used to measure the size, density and quality of the nanowires on the sample.

	constant Temp. 450°C	Temp. Step 450°C $\rightarrow$ 400°C	Temp. Gradient 450°C $\searrow$ 400°C
Sample ID	M4586	M4509	M4580
Length [μm]	7-7.5	5-7.5	6-8.5
$\varnothing_{Top}$ [nm]	90-105	90-95	90-100
$\varnothing_{Middle}$ [nm]	110-130	95-105	95-115
$\varnothing_{Base}$ [nm]	160-195	120-125	125-140

Table 5.1: For all three samples high resolution SEM images were taken, and the dimensions of the nanowires were measured. The length as well as the diameter at three positions are presented together with the sample ID.

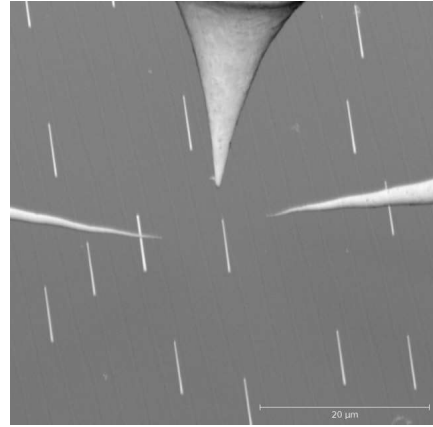
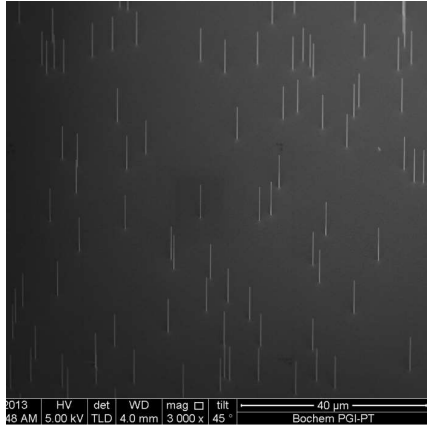
single nanowires (size, quality, etc) as well as to get a first impression of the sample (density on nanowires, defect, size variations throughout the sample). In [Table 5.1](#) some of the findings are displayed.

5.1.2.1 Nanowire sizes

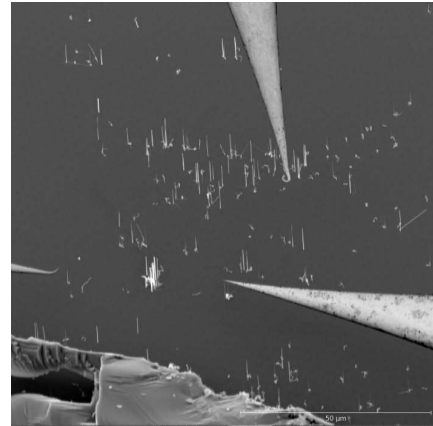
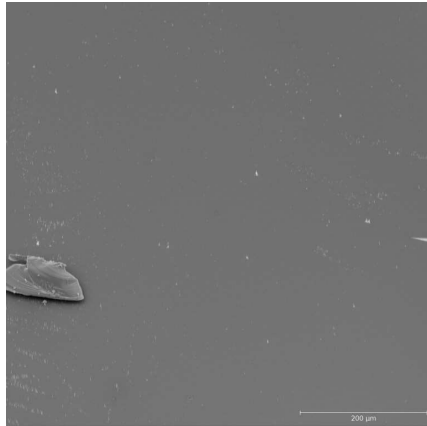
In [Table 5.1](#) to some typical values for diameter and length of the nanowires, are presented. The length of the nanowires are between $\sim 5 \mu m$ (Step Sample) and $\sim 8.5 \mu m$ (Gradient Sample). While the nanowires have comparable top diameters, the diameters at the bottom differ a lot. If the form of the nanowires on the Step Sample is compared to the corresponding results of the Constant Sample, it is striking that the difference in the diameter is growing from top to bottom. At the base the nanowires on the Step Sample are 25 % to 35 % smaller than the ones on the Constant Sample. The expected tapering is the explanation for this characteristic. The longer a nanowire part is exposed to high temperature while free gallium and arsine are available, the more widening of the nanowire occurs. Consequently the middle part of the Gradient Sample nanowire lies between the two other samples.

5.1.2.2 Sample structure

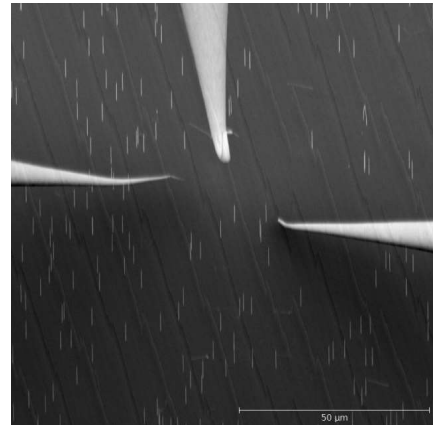
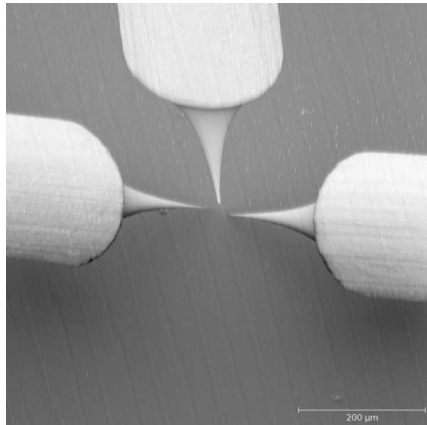
Once the samples were investigated with a high resolution SEM, they were transferred into the RT-MT-STM. First, an overview over the working area is gained with the SEM. To perform four-point measurements, first of all a sufficient amount of nanowires has to be situated in the area reachable by all three tips. In order to be reachable by all tips, the nanowires not only have to be on the correct spot of sample, but also no other nanowire must block the tips to reach their designated



(a) Constant Sample



(b) Step Sample



(c) Gradient Sample

Figure 5.9: Two different SEM images are presented for each sample. Such images are used to gain a first impression of the sample, as well as to target a specific nanowire for measurements

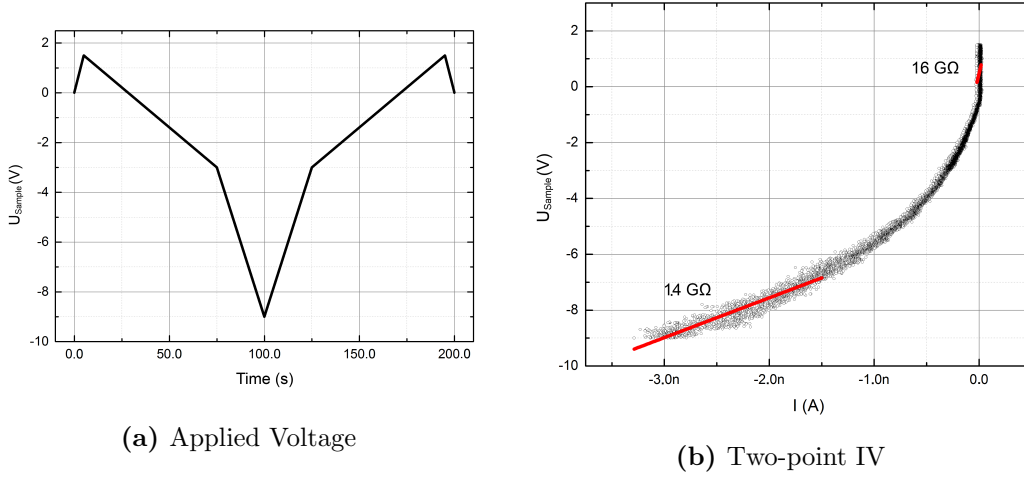


Figure 5.10: Constant Sample: A nanowire on the Constant Sample is contacted with an STM tip at the gold particle at its top. The voltage pattern shown in (a) is applied to the sample, while the Current Injection Tip is permanently grounded. The resulting current is shown in (b).

position on the nanowire. Furthermore, the nanowires have to be straight to be suitable for measurements.

The first SEM images (Figure 5.9b) of the Step Sample mounted inside the RT-MT-STM showed that the sample is only partly covered with nanowires in the area reachable by all tips. The nanowires on the sample were packed into small islands. Even on these islands the nanowires often clustered into groups, growing very close to each other. These densely packed nanowires could not be used for four-point resistance measurements. Furthermore, many nanowires show kinks or extra branches as can be seen in Figure 5.9b. These nanowires had to be excluded from the measurements.

However, on all three samples enough suitable nanowires could be used to carry out the measurements described in the next section. On the other two samples, the nanowires grew mostly straight with very little defects. Also, the nanowire density allowed fast and uncomplicated access to many nanowires.

5.2 Two-point resistance measurements

The first electrical measurements can be performed by contacting a nanowire with only one tip, like shown in Figure 5.3a. The contact is established by simply touching the gold particle at the top of each nanowire with the Current Injection Tip. The resistance of the entire system can be measured in a two-point measurement. While the Current Injection Tip is grounded, the voltage pattern shown in Figure 5.10a, is applied to the sample, in this case the Constant Sample. In this configuration the potential at the top of the nanowire is closer to ground than to the applied voltage.

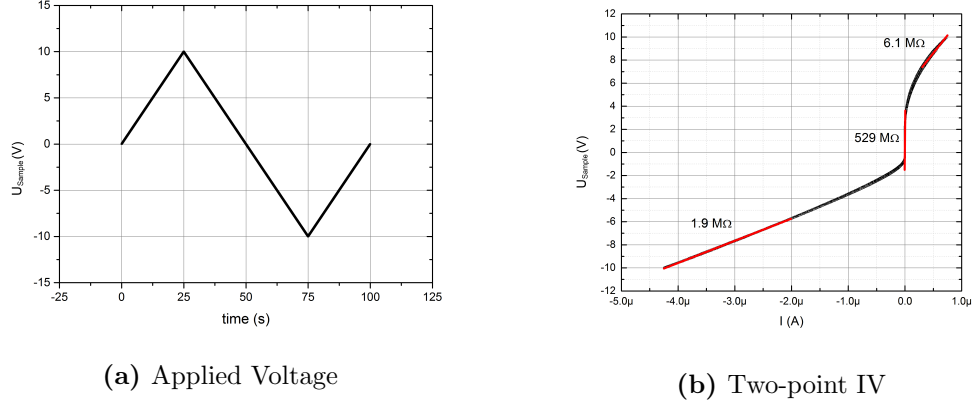


Figure 5.11: Step Sample: A two-point IV, typical for a nanowire on the Step Sample, is shown with three fits and the corresponding resistances. Due to the pn-junction the IV is asymmetric. Current flow in both directions is measurable.

Which will turn out to be advantageous in four-point resistance measurements. The resulting current through the nanowire is measured at the sample side as well as at the Current Injection Tip side. If the current is not flowing through the nanowire, due to leakage for example, the two measured currents differ. For all presented measurements the current flowed entirely through the nanowire.

5.2.1 Constant Sample (M4586)

The current flowing through the nanowire is shown in Figure 5.10b as a function of the applied voltage. The applied voltage is not measured during a resistance measurement, but the measurement electronics have been calibrated beforehand, so the applied voltage is known with sufficient precision.

The measured current shows a strong diode like non-linearity. For negative voltages applied to the sample a current, corresponding to a resistance of 1.4 G Ω , is flowing through the nanowire according to the linear fit between -4 A and -1.5 A. For positive voltages applied to the sample on the other hand nearly no current is flowing through the nanowire. A linear fit around 0 nA in Figure 5.11b shows a resistance of 16 G Ω . This asymmetry is to be expected since the p doped nanowire form a diode together with the n doped sample.

5.2.2 Step Sample (M4509)

The two-point IV shows a nonlinear behavior. If negative voltages smaller than -2 V were applied this nanowire had a resistance of 1.9 M Ω . For positive biases (≥ 7 V) the resistance of the same nanowire was 6.1 M Ω . In between these two voltage regimes the resistance is up to 529 M Ω around 0 A of current. Compared to the measurements of the Constant Sample (subsection 5.2.1), the two-point resistance of the nanowires on the Step Sample is up to 1000 times smaller.

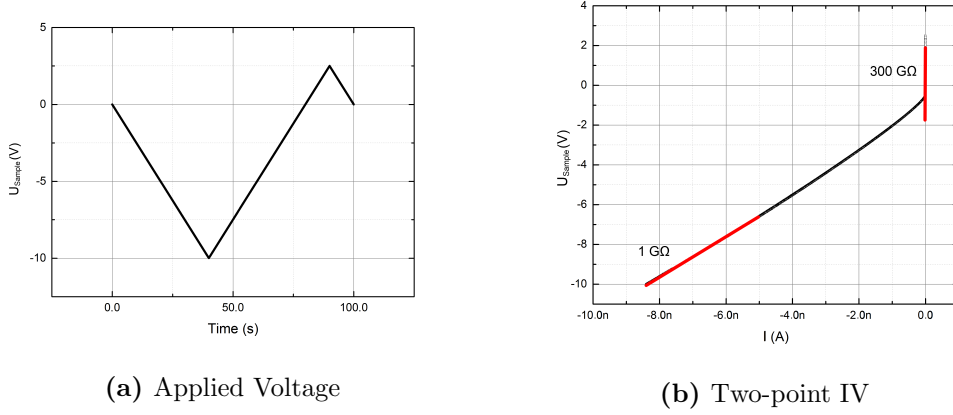


Figure 5.12: Gradient Sample: For the Gradient Sample, the applied voltage signal (a) and the resulting current (b) are presented. The two-point IV shows current flow for negative voltages.

Due to the small(er) resistance, more current flows through the nanowire at the same bias. If too much current is driven through a nanowire, it can be burned. So to perform resistance measurements on this sample, the current is limited to $\approx 1 \mu\text{A}$.

5.2.3 Gradient Sample (M4580)

The resulting current flowing through the nanowire is shown in Figure 5.12b as a function of the applied voltage (Figure 5.12a). Due to the pn-junction, the measured current shows the same diode-like behavior as the other two samples. For negative voltages a current, according to a resistance of $1.2 \text{ G}\Omega$, is flowing through the nanowire. The resistance in reverse bias is so high ($300 \text{ G}\Omega$), that almost no current can be driven through the nanowire within the bias range to $+10 \text{ V}$.

5.2.4 Comparison of the Two-Point Results

The three samples all show the expected diode like characteristic in their two-point IV. The total resistance, however, differs quite a lot. The Step Sample is by far the most conductive. At -10 V over $4 \mu\text{A}$ of current flows through the nanowire. Additionally, currents can flow in both directions. At $+10 \text{ V}$ nearly $1 \mu\text{A}$ flows.

The other two samples (Constant Sample and Gradient Sample) show similar two-point IVs. The available voltage range of 10 V is not sufficient to cause a substantial current flow in reverse bias. Also, the resistance at negative voltages is similar ($1 \text{ G}\Omega$ to $1.4 \text{ G}\Omega$). The overall characteristics of the two-point IV, on the other hand, is quite different. While the Gradient Sample shows a very pronounced kink between reverse and forward bias, the Constant Sample has a soft transition between the two parts.

Due to the soft transition, the fit around 0 A is very sensitive to the choice of the

lower fit limit. Also the noise, which is more present on the Constant Sample IV, influences this fit heavily.

The voltage patterns applied to the samples are customized to each sample. For example, the current shall not exceed $1\ \mu A$, consequently only $\pm 2\ V$ could be used for the Step Sample. Also, the capacity of the entire system has to be taken into account. At the Constant Sample, for example, a time difference between a voltage change and the corresponding change in the current was present, due to a bad Current Injection Tip nanowire contact. To spot such bad contact, the voltage pattern for the Constant Sample has regimes with different slopes.

5.3 Three-point resistance measurements

To learn more about the contact between the nanowire and the measurement equipment, the three-point resistance measurements can be analyzed. Therefore, the voltage difference between the Static Voltage Prober and the grounded Current Injection Tip is plotted as a function of the flowing current, as shown in [Figure 5.13a](#). From the resulting graph, the resistance between the Static Voltage Prober and the Black-Box at the end of the Current Injection Tip can be calculated (like in the two-point case). While the cabling and the tip itself can be neglected, only the short segment of the nanowire and the contact contributes to this resistance. Since the resistance of the nanowire does not change during the measurement, this three-point resistance can be used to monitor the contact resistance between the Current Injection Tip and the nanowire. Compared to the total resistance (typically in the range of several hundred $M\Omega$ to several $G\Omega$), the upper three-point resistance is nearly negligible (10 to 100 $k\Omega$). Accordingly, the voltage drop between the measurement electronics and the Static Voltage prober is very small (typically only a few mV). In the upper three-point IV, single digital bits of the measurement electronics will be visible. Nevertheless a linear fit with sufficient precision ($\pm 10\%$) can be performed.

Similarly, the resistance through the sample can be measured ([Figure 5.13b](#)). With the Voltage Scanner in a position close to the substrate, the difference between the voltage signal applied to the sample and the potential at the Voltage Scanner displays the resistance from the tip through the nanowire base and the sample to the Black Box.

5.3.1 Constant Sample (M4586)

In [Figure 5.13](#), characteristic voltage signals at the two Voltage Probers are presented. While the amplitude at the Voltage Scanner (positioned at the base of the nanowire) has several volts, the signal at the Static Voltage Prober is nearly flat at ground level. In the shown case, the resistance between the Black-Box at the injection side and the Static Voltage Prober is 12 $K\Omega$. The measured voltage signal is close to the resolution limit of the ADC (analog digital converter). So, instead of a smooth curve, single digital state (bits) are displayed as horizontal lines. Here the

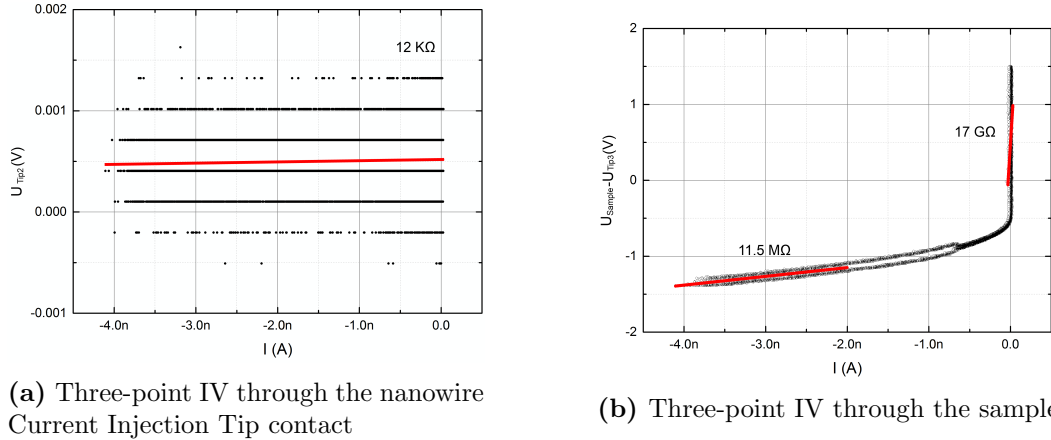


Figure 5.13: Constant Sample: Two typical three-point IVs for a nanowire of the Constant Sample are shown. In (a) the voltage difference between the Current Injection Tip and the Stationary Voltage Prober is plotted as a function of the current flowing through the nanowire. The contact resistance can be obtained from this plot by fitting the graph with a linear function. Here the contact resistance is so low that single digital bits of the voltage measurement electronics can be seen as horizontal lines. The difference between the Voltage Scanner positioned near the base of the nanowire and the applied voltage signal is plotted in (b).

nanowire was contacted at the gold particle. If the nanowire is contacted at another part, this resistance rises to the $10\text{ M}\Omega$ regime. In both cases the contact resistance is small compared to the total two-point resistance ($1.4\text{ G}\Omega$ to $16\text{ G}\Omega$).

The three-point IV from the Voltage Scanner through the nanowire base and the sample shows the characteristic shape of a diode. In forward bias (negative voltage applied) this path has a resistance of only $11.5\text{ M}\Omega$. In reverse bias, on the other hand, no substantial current flows at all. The corresponding fit results in a resistance of $17\text{ G}\Omega$. The pn-junction between the nanowire and the sample clearly dominates this IV. In addition to the diode like shape it is striking that the IV in forward bias splits into two branches. This is caused by a parasitic capacity between a tip and the nanowire. By adjusting the contact of this tip, the capacity can be reduced or removed. If it is present also in the four-point IV, this measurement has to be disregarded for the resistance profile of the nanowire.

5.3.2 Step Sample (M4509)

While the two-point resistance of the Step Sample was much smaller than the corresponding resistances on the other two samples, the contact resistance of the Step Sample is at least larger by a factor of 20, as the three-point resistance measurement in Figure 5.14 shows. While the three-point resistance at the top of the Constant Sample appears as a flat line, more details are detectable for the Step Sample. One factor for this might be that the resistance resolution depends on the total current

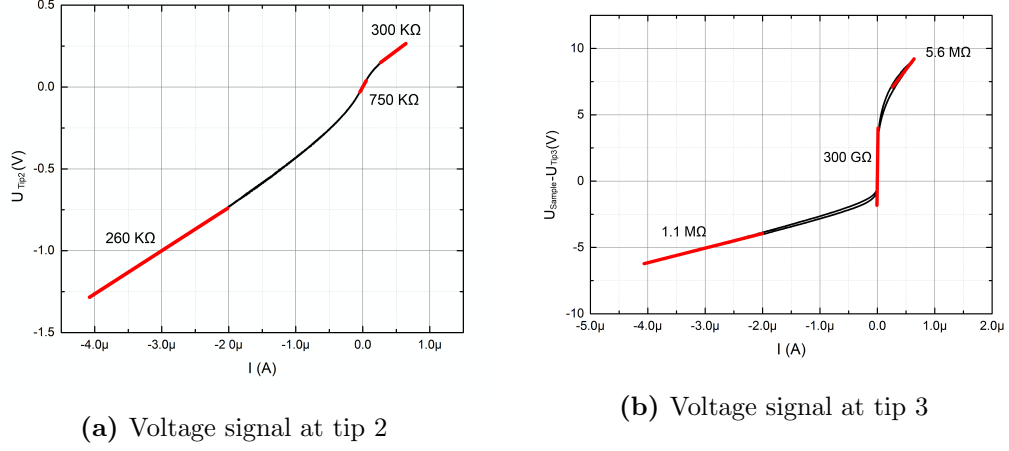


Figure 5.14: Step Sample: From the three-point IV shown in (a), the contact resistance between the Current Injection Tip and the nanowire can be extracted. Therefore, it is fitted in three different current regimes. The corresponding resistances are presented. Due to the Schottky barrier the IV is asymmetric. In (b) the result of a three-point resistance measurement is shown.

flowing through the nanowire. More current results in higher voltage drops over resistances. For this IV a small kink around 0 A can be seen. For currents higher than ~ 250 nA in both directions the resistance is ~ 280 K Ω , around 0 A it is 750 K Ω . This nonlinearity cannot be explained with a pn-junction. However, a semiconductor metal interface is known to form a Schottky-Barrier [39]. This barrier acts like a diode and contributes to the resistance depending on the bias. It is also present in the other contacts, but may not be detectable due to a lack of resolution. In any bias regime the contribution of the contact resistance is small compared to the total (Two-Point) resistance, so it can be neglected for the following measurements.

The three-point resistance through the sample shows the same characteristics as the corresponding one from the Constant Sample, except that the current also flows in reverse bias. The measured resistances are one order of magnitude smaller than on the Constant Sample. In forward bias 1.1 M Ω is fitted, while in reverse bias the result is 5.6 Ω . Around 0 A the resistance rises up to 300 M Ω .

5.3.3 Gradient Sample (M4580)

With 150 k Ω , the upper three-point resistance in this measurement turned out to be only a very small fraction of the total ≈ 1 G Ω two-point resistance. It shows no diode like behavior.

For the lower three-point IV Figure 5.15b the same features as for the Constant Sample can be found, except for the splitting. No current flows in reverse bias. In this regime the fit resulted in a resistance of 300 G Ω . In forward bias the resistance is 930 M Ω .

During all measurements the contact resistance was monitored in order to restore

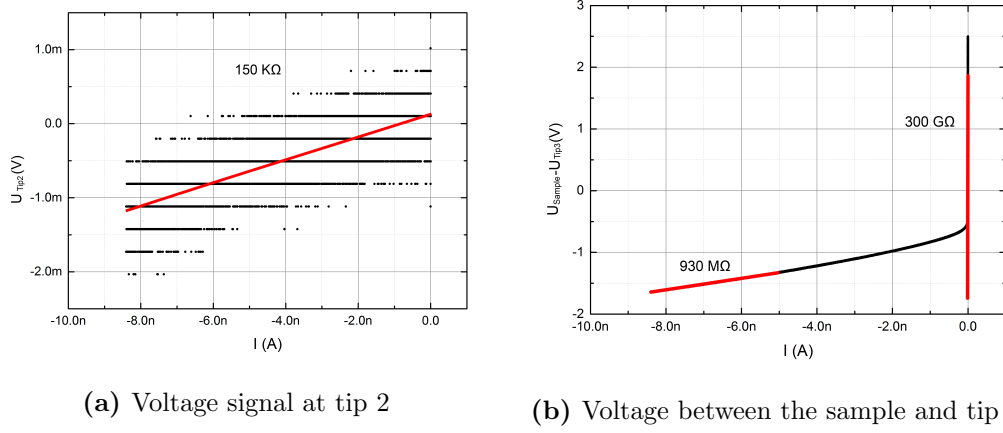


Figure 5.15: Gradient Sample: The upper and lower three-point IV of a nanowire on the Gradient Sample are presented.

proper contacting as fast as possible. From the difference between the two three-point resistances also first assumptions about the resistance of the nanowire itself can be made.

5.4 Four-point measurements

The main focus of the resistance measurements of p-GaAs Nanowires on n-GaP and n-GaAs substrate lies on four point measurements. As described, four point measurements have the advantage of being independent of additional (contact) resistances in the current path.

In order to measure how the different parts of a nanowire contribute to the total resistance, several four point measurements were performed along nanowires of each type. While one tip (Voltage Scanner) was moved step by step along the nanowire, the other tip (Static Voltage Prober) was positioned constantly near the top of the nanowire. The third tip (Current Injection Tip) was used to contact the nanowires at their top. The procedure is shown in [Figure 5.16](#).

At each position of the Voltage Scanner, at least three four point resistance measurements, were performed. For each of these measurements the voltage between the two voltage measurement tips was plotted as a function of the flowing current as shown in [Figure 5.17](#).

As for the two point measurements, the curve is fitted in two different regimes of current (three for the Step Sample) with a linear function. In the case shown in [Figure 5.17a](#), the resistance drops from 1.27 GΩ to 900 MΩ for stronger (negative) biases and higher currents. For a perfect ohmic resistor these two should be the same. In [Figure 5.17b](#) another four-point IV of the same nanowire is shown. It was recorded in the middle of the nanowire, while [Figure 5.17a](#) was measured at the very bottom of the nanowire close to the substrate.

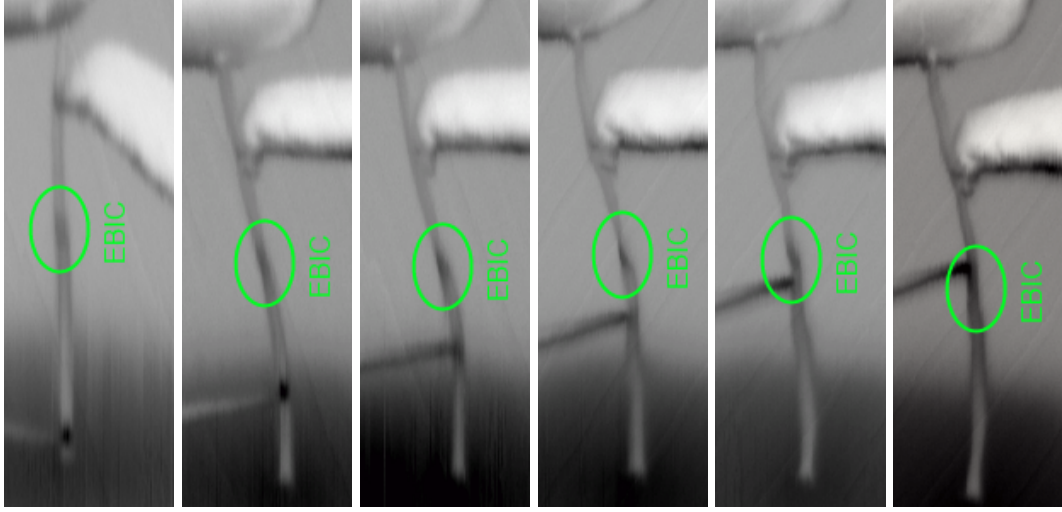
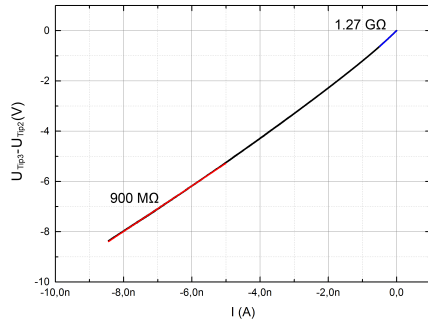
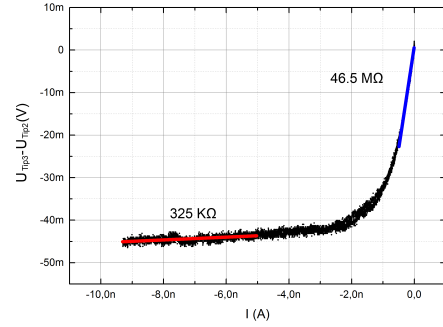


Figure 5.16: Four-point resistance measurement along a nanowire. Each image shows an area of $1.65 \mu m \times 5 \mu m$. The two upper tips stay at their positions during the measurement of an entire resistance profile. The third tip is moved up along the nanowire. At each point three four-point resistance measurements are performed and one SEM image is taken to measure the position of each tip. The point density of the resistance profiling can easily be in- or decreased if the resistance profile shows interesting features.



(a) Four-point IV with the Voltage Scanner near the substrate



(b) Four-point IV with the Voltage Scanner in the middle of the nanowire

Figure 5.17: Gradient Sample: Two 4 Point IVs of a nanowire grown with a temperature gradient are shown. Nearly the entire negative voltage drop (-8.5 V of -10 V) occurs between the two voltage probing tips in (a). In (b) the distance between the two Voltage Probers is reduced. Consequently, the voltage drop decreases. The data in both figures are fitted for very low 0 to -0.5 nA and for maximal ($\leq -5 \text{ nA}$) currents with a linear function. The results of these fits represent the resistance in their bias regime.

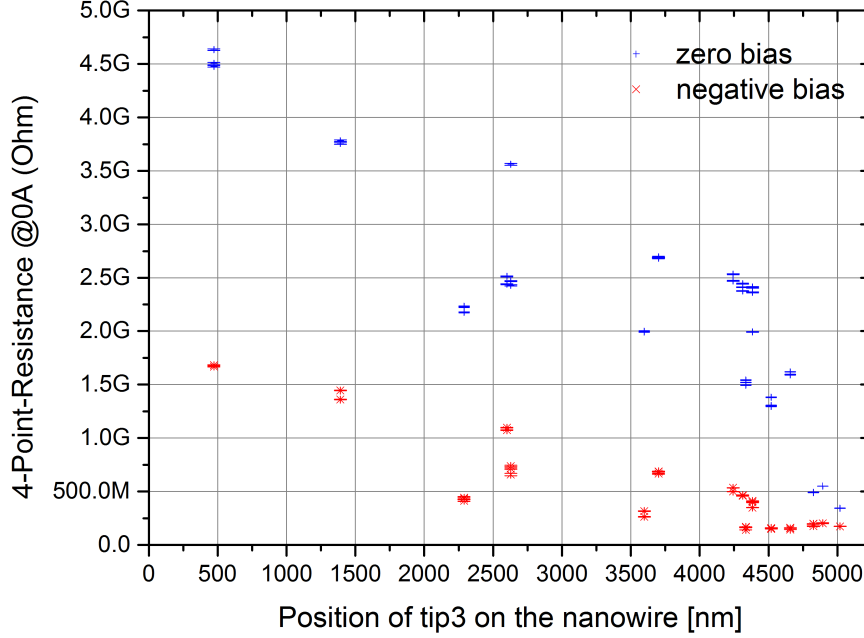


Figure 5.18: Constant Sample: For the Constant Sample, the evolution of the two fits shown in Figure 5.17 are plotted as a function of the position of the Voltage Scanner measured from the substrate surface.

If now the Voltage Scanner is moved along the nanowire as described, a resistance profile, as shown in Figure 5.18, is recorded. The two resistance profiles shown in Figure 5.18 correspond to the two fits in Figure 5.17. The $+$ show the resistances around 0 nA, while the $\times$ show the results for currents higher than 2 nA in forward current (negative voltages in the experiment). The limits of the fits in the four-point IVs differs from sample to sample depending on the total current flowing through a nanowire. In general the IVs show linear behavior for high currents as well as around zero. In these areas the fits are performed. For each sample the limits of the two or three fits are constant.

In general, the results of a four-point IV are derived from the entire nanowire resistance between the two Voltage Probers. Thus, to gain information on a specific part of the nanowire, the two Voltage Probers have to be positioned at both ends of this part. In this study the position of the Static Voltage Prober is constant. In this configuration, two measurements have to be performed to gain information about a specific part of the nanowire, one at each end. By comparing (subtracting) the two IVs, the contribution of the designated nanowire part can be deduced. From the two IVs in Figure 5.17, the resistance of the nanowire part between these measurements can be computed. For a more detailed analysis the resistance profile can be fitted as shown in Figure 5.19. By choosing a linear fit, it is assumed that each part of the

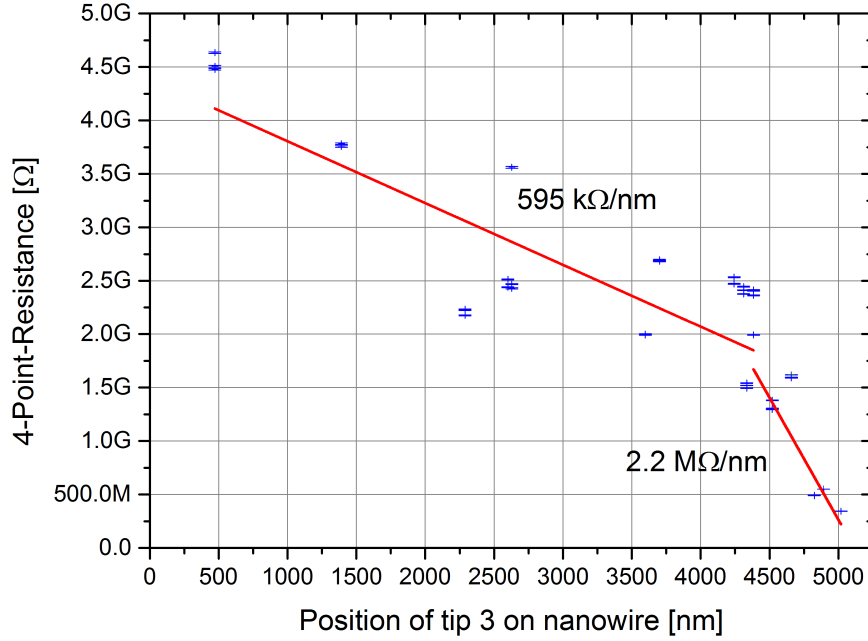


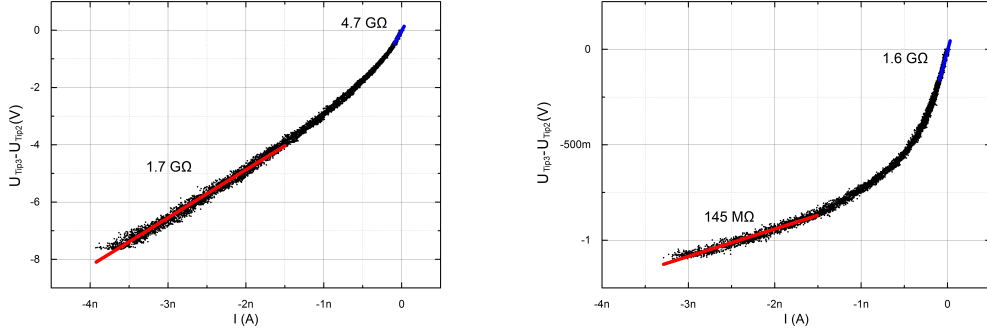
Figure 5.19: Constant Sample: The Resistance Profile for the fit around 0 A of the Constant Sample can be fitted with two linear fits. The lower part of the nanowire has a resistance gradient of $595 \frac{K\Omega}{nm}$, while the upper part shows a resistance gradient of $2.2 \frac{M\Omega}{nm}$. The constant resistance gradients are displayed in the figure.

nanowire in this regime has the same resistivity.

5.4.1 Constant Sample (M4586)

Two typical four-point IVs for a nanowire on the Constant Sample are shown in [Figure 5.20](#). One was measured with the Voltage Scanner close to the substrate, while for the other IV it was in the middle of the nanowire. From [Figure 5.20a](#) information on the entire nanowire can be gained, since it lies between the two Voltage Probers. First of all, a quarter of the total two-point resistance of the entire system originates from the nanowire around 0 A. For high currents it is only 10%. The two fitted resistances ($4.7 G\Omega$ at 0 A and $1.6 G\Omega$ for high currents) differ by a factor of 3. So, the nanowire is not an ohmic resistor, but has a bias dependent resistance component, too. Also the upper part of the nanowire, from the IV in [Figure 5.20b](#) upwards, shows an asymmetry ($1.6 G\Omega$ at 0 A to $140 M\Omega$ for high currents). At least a part of the bias dependent resistance must be located above the position where IV has been recorded. With several such IVs a resistance profile, as shown in [Figure 5.18](#), can be plotted.

The two profiles show very similar features along the nanowire. Two segments with



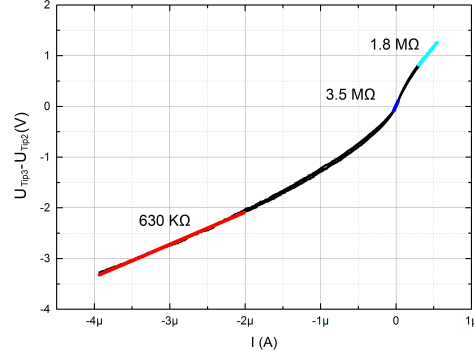
(a) Four-point IV with the Voltage Scanner near the substrate (b) Four-point IV with the Voltage Scanner in the middle of the nanowire

Figure 5.20: Constant Sample: The development of the four-point IV along the Constant Sample can be estimated from the two four-point IVs. They were recorded at opposite ends of a nanowire.

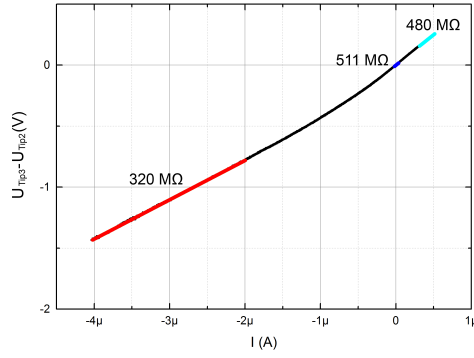
constant resistance gradients are present in both current regimes. The high current regime shows an additional segment at the top of the nanowire, where the resistance is constant and close to the resolution minimum of the experiment. The difference between the two profiles is a direct consequence of the asymmetry of each IV. In order to obtain the resistance gradient the two regimes can be fitted with a linear function each (Figure 5.19). At the kink, the resistance gradient jumps from $595 \frac{k\Omega}{nm}$ to $2.2 \frac{M\Omega}{nm}$.

5.4.2 Step Sample(M4509)

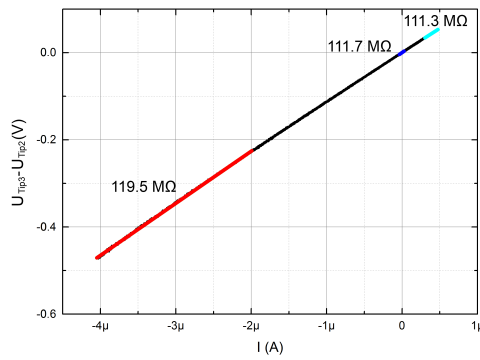
Due to the lower resistance and the resulting increased current flow in both bias directions, the three four-point IVs shown in Figure 5.21 are fitted in three different regions. In addition to the fits in forward bias and around 0 A, which were performed for the Constant Sample, a fit for reverse bias was made. While the asymmetry in the IV close to the substrate is distinctive, the one in the middle is nearly flat. The IV close to the top of the nanowire is completely straight. This can also be seen in Figure 5.22a, where the evolution of the three fits is shown in more detail. For most of the nanowire (1250 nm till the end) the resistance profile is flat. This area contains nearly no additional resistance. In this area the nanowire behaves like a pure ohmic resistor with a resistance of $34 \frac{\Omega}{nm}$. The major part of the resistance arises from the first $1.2 \mu m$ of the nanowire over the substrate. Two different parts can be identified in the area. While the first 500 nm drops very steep ($7580 \frac{\Omega}{nm}$), an intermediate area between the steep base and the flat top part exists with $400 \frac{\Omega}{nm}$ from 500 nm to 1200 nm. In order to assure that the measured results are not specific for a single nanowire, resistance profiles of several nanowires were measured. In Figure 5.23 three of them are compared to each other. While the nanowire 02 (discussed before) and 06 are comparable with respect to shape as well as to height,



(a) Four-point IV with the Voltage Scanner near the substrate

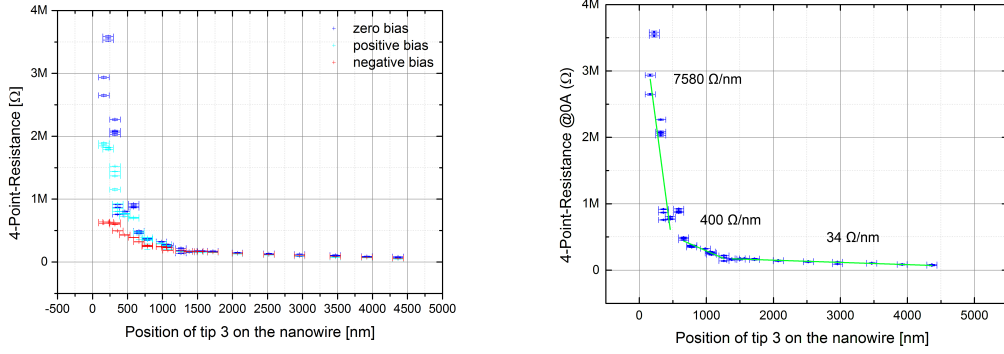


(b) Four-point IV with the Voltage Scanner in the middle of the nanowire



(c) Four-point IV with the Voltage Scanner close to the top of the nanowire

Figure 5.21: Step Sample: Three IV along the Step Sample are shown. From top to bottom the shape of the IV changes. While the three fits show a non linearity in (a), the IV is nearly straight in (c)



(a) The resistance profiles for the three analyzed bias regimes on a single nanowire are shown. The different bias regimes show a very similar form.

(b) The resistances profile at 0 A (corresponding to the fit in Figure 5.21) of nanowire 02 is presented. The profile has been fitted in three different sectors.

Figure 5.22: Step Sample: The resistances profile at 0 A (corresponding to the fits in Figure 5.21) of nanowire 02 is presented. The profile has been fitted in three different sectors.

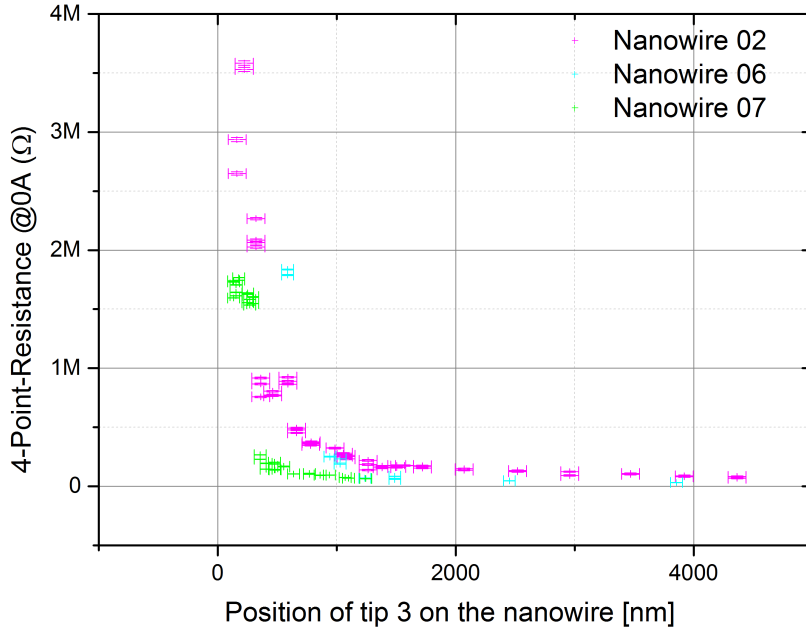


Figure 5.23: Step Sample: The four-point resistance profiles of the three nanowire 02, 06 and 07 show the same shape in (a). In (b) One of the three profiles is fitted in three regimes.

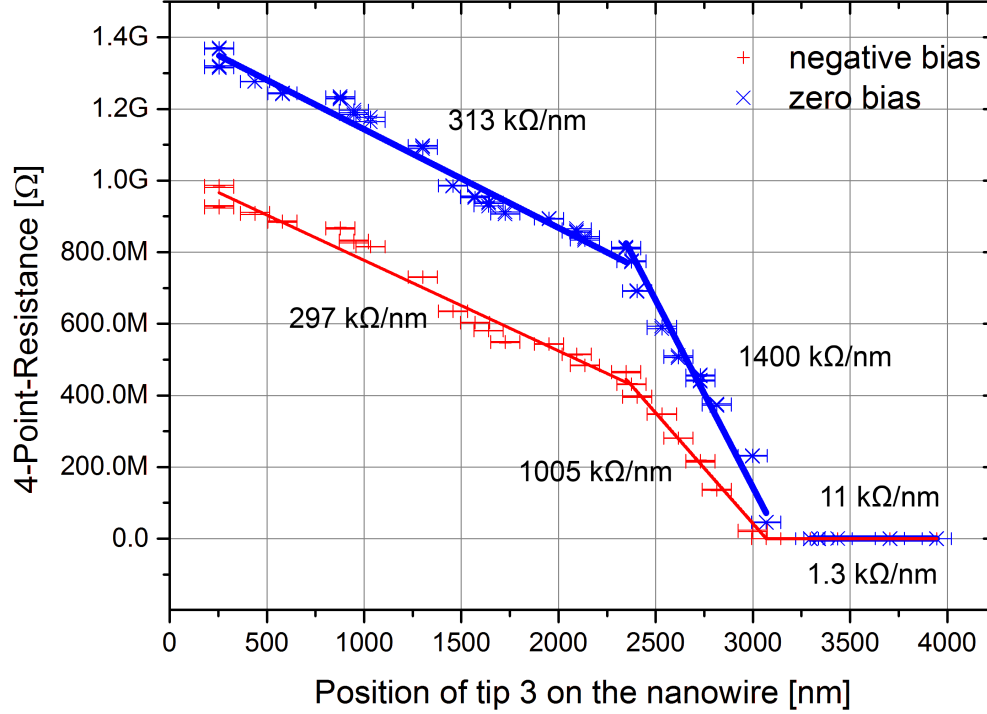


Figure 5.24: Gradient Sample: 4 Point resistance profiles of nanowire 02 at low and high currents. The two profiles show the same shape. Each profile can be divided into three parts with a constant resistance gradient. An intermediate section near the substrate is followed by a very steep slope. The upper part of the nanowire shows only very little resistance.

nanowire 07 shows a different profile. The form of the resistance profile of nanowire 07 is similar to the other two, but position and size of the steep part as well as the intermediate part differ. Several reasons are possible for such a difference. However, since most nanowires support the profile of nanowire 02, and the qualitative shape is very similar, nanowire 02 is used as a prototype in the analysis.

5.4.3 Gradient Sample(M4580)

The two resistance profiles in Figure 5.24 correspond to the two fits in the IVs in Figure 5.17. They show a very similar form. Three different regimes with constant resistance gradients can be identified in both cases. In each segment, the resistances are comparable, but the resistances measured at high current densities are always lower than the resistances for low current densities. Each of the three segments can be fitted linearly to get the resistance change per nanometer. Also, the resistance gradients are in comparable orders but lower for high current densities. The only striking difference between the two resistance profiles is the position of the transition between the second and the third segment. While the difference between the two

profiles is nearly constant in the first sector, it starts decreasing in the second. At the point where the second IV in Figure 5.17 was measured, the high current fit is already flat. In the resistance profile, the third starts earlier for the high current fit. The second segment stops ≈ 3000 nm over the sample for the high current densities, it continues to ≈ 3250 nm over the sample for the low current densities. The IV in Figure 5.17b was taken between the 3000 nm and 3250 nm above the substrate. While the high current density fit is already flat, the fit around 0 A still shows a considerable slope.

The resistance profile shown starts 1250 nm above the substrate (0 nm in Figure 5.24 corresponds to substrate level). There the four-point resistances drops with $313 \frac{k\Omega}{nm}$. The first segment ends ~ 2500 nm above the sample. In the following section the resistance grows with $1400 \frac{k\Omega}{nm}$ for roughly 800 nm. The transition from the second to the third segment is at 3150 nm. For the rest of the nanowire the resistance increases with only $11 \frac{k\Omega}{nm}$. At that point the potential difference between the two voltage measurement tips has nearly vanished and the resistance measurement is at its limit. So, $11 \frac{k\Omega}{nm}$ is only an upper limit for the resistance gradient of the remaining 3200 nm.

Such resistance profiles have been measured for three different nanowires (02, 08 and 13). In Figure 5.25 all measurements are compared. The three nanowires show very similar resistance profiles. Each of the three nanowires has three different resistance regimes. For the first ≈ 2400 nm (Nanowire 13: 2281 nm; Nanowire 08: 2352 nm; Nanowire 02: 2534 nm) the resistance increases with $\approx 300 \frac{k\Omega}{nm}$ (Nanowire 13: $315 \frac{k\Omega}{nm}$; Nanowire 08: $313 \frac{k\Omega}{nm}$; Nanowire 02: $275 \frac{k\Omega}{nm}$). Even though the first transitions between the first two section were scattered over 250 nm, the next transitions are well located around 3275 nm (± 75 nm) for all three nanowires.

5.5 Discussion

In the previous section, the results of the 4-Point resistance measurements for the three nanowires sketched in Figure 5.26 have been introduced. In this section these result will be further analyzed and the findings for the different samples will be compared to each other. A model will be presented that includes the findings from all three sample.

The temperature during the growth of a nanowire is known to influence the growth and resulting properties of the nanowire in various ways [40]. The decomposition of precursor gases depends on the temperature as well as the growth mode and direction. Effects like tapering occur only above a certain temperature threshold.

In the paper by Korte et al [33], the resistance profiles of nanowires (similar to the Step Sample) showed two segments with constant resistance gradients each. These two segments could be associated with the two temperature regimes. Based on these results, a one-dimensional conduction model was applied to the low current resistance profile (blue profiles in Figure 5.18, Figure 5.22a and Figure 5.24). It calculated the charge carrier density as a function of the resistance gradient and the radius of the nanowire. Subsequently, the charge carrier mobility, the size of the

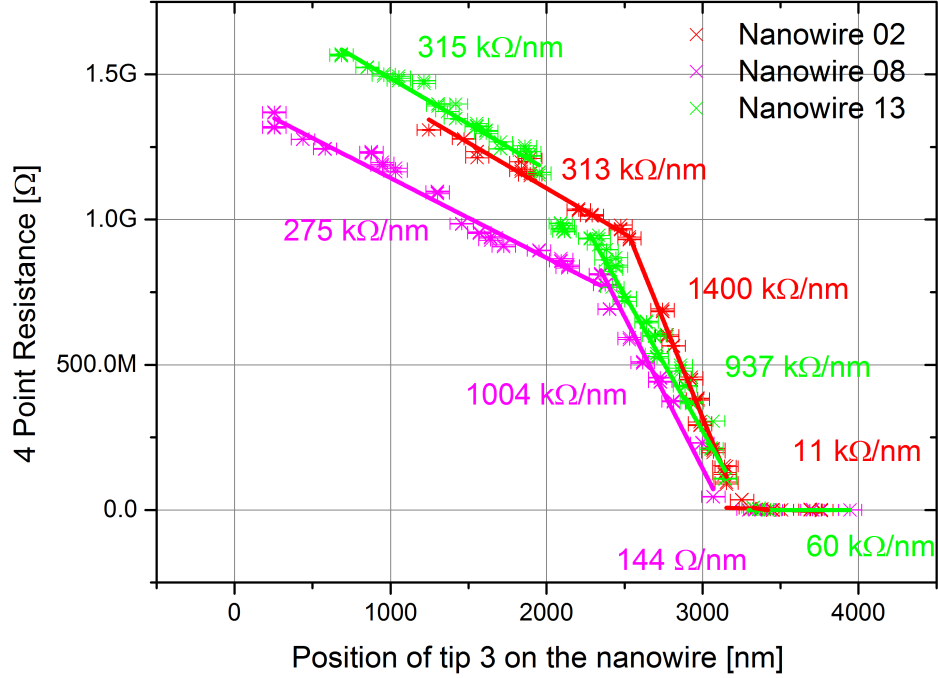


Figure 5.25: Gradient Sample: The 4 Point resistance profile of all the three measured nanowires (02, 08, 13) are compared. The profiles show all the same characteristics. Also the resistance gradients in the three sectors are of comparable size.

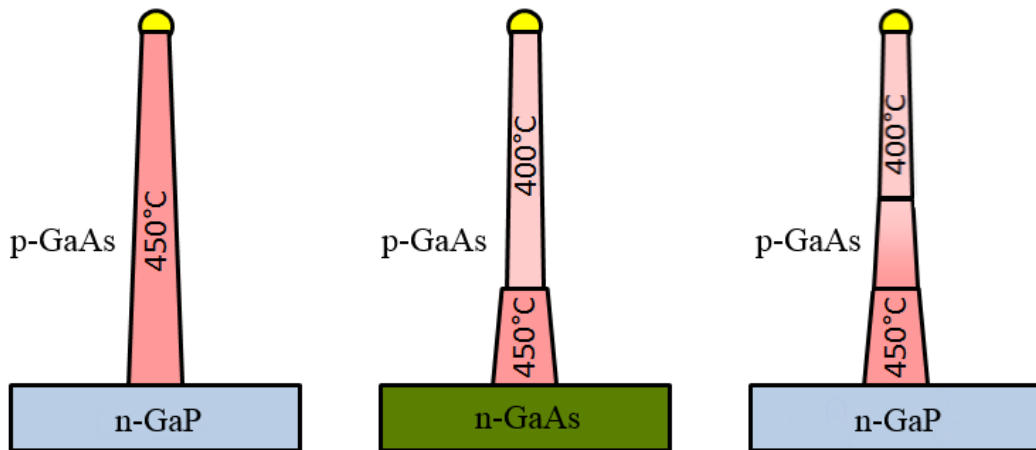


Figure 5.26: Shown are the icons of the three different types of nanowires that are discussed in the following chapter. Each sample contains p-GaAs nanowire grown on n-GaP.

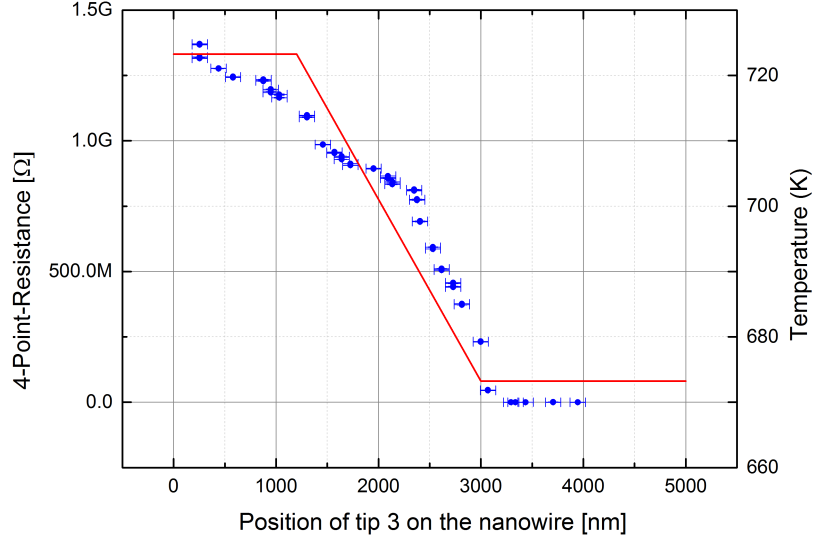


Figure 5.27: Gradient Sample: The resistance profile of a nanowire from the Gradient Sample is compared to the temperature during its growth. While the low temperature regime corresponds well with the nanowire part with the low resistance gradient, the other two regimes do not match.

depletion zone and the resistivity could be calculated. The model fitted the result of the measurements well.

To find a conclusive interpretation for the samples in this thesis, an analysis of the low resistance regime alone does not suffice, as will be shown latter. Other effects and conduction channels like surface conductance and changes in the band structure due to the application of external voltages will be taken into account.

5.5.1 Temperature dependence of the resistance profiles

It appears reasonable to compare the resistance profiles with the temperature profiles as a first attempt. The reactor in which the nanowires were grown, and the growth process itself are known with enough precision to assign each piece of the nanowire to the temperature during its growth. In Figure 5.27 the resistance profile (blue) of a nanowire from the Gradient Sample is compared to the temperature (red) during the growth. The low resistance part of the resistance profile matches the low temperature regime very well (above 3250 nm). The transition from the high temperature regime to the gradient area (at ~1200 nm) does not match the kink between the first two resistance gradient regimes. The temperature of the sample is not measured directly, but the heating device was calibrated with sufficient precision. The cooling from high to low temperature (450°C to 400°C) takes 5.5 minutes. In this timespan, the cooling is a dynamic process and the real temperature of the

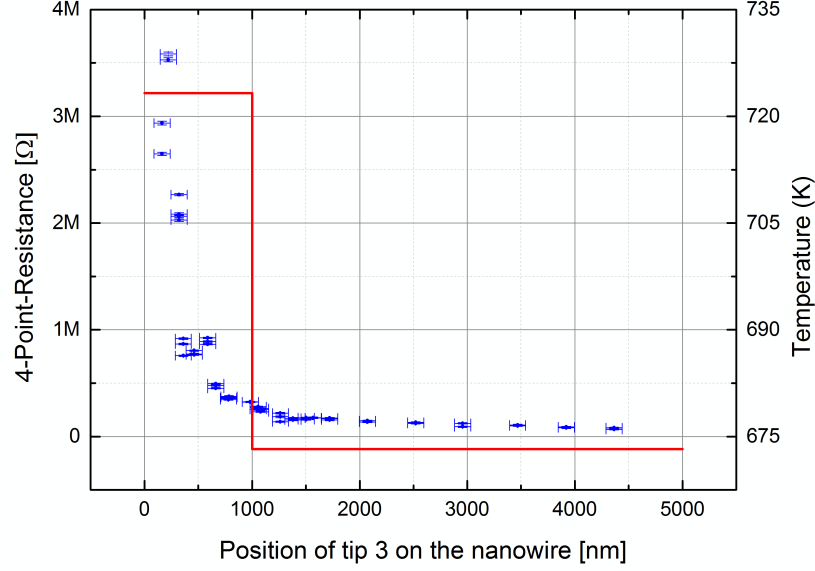


Figure 5.28: Step Sample: The resistance profile at 0 A (blue) is compared to the temperature (red) during the growth of the nanowires on the Step Sample.

sample can only be assumed. When thresholds for growth modes or tapering are crossed cannot be predicted with precision. The growth rate and the over all length of the nanowire, however, is a good indicator that the temperature of the sample is accurately adjusted.

Higher temperature during the growth of a nanowire segment should result in a higher resistance gradient of that segment. This relation is based on the fact that the efficiency with which the precursor gases decompose is strongly temperature dependent. At low temperatures ($\sim 400^\circ\text{C}$) only a fraction of the trimethyl gallium and the tertiary butyl arsine decomposes to supply gallium and arsine for nanowire growth, while nearly all zinc from the diethyl zinc is available. If the temperature is higher, more GaAs becomes available, while the amount of zinc stays constant. Consequently, less zinc per volume is incorporated into the nanowire and lower doping is achieved. During the growth of the second segment of the Gradient Sample nanowire however, the temperature is dropping, while the resistance gradient is growing. Here an additional effect other than temperature, must come into play.

The resistance profile of the Step Sample correlates better with the temperature pattern (Figure 5.28). The segments of the nanowire with high resistance gradients are situated in the high temperature area, while the flat top part grew completely at low temperature. A transition area (750 nm to 1250 nm) connects the two gradient regimes. The transition segment reaches into both temperature regimes.

For the Constant Sample nanowire, finally, no feature at all could be expected in the resistance profile, since the temperature was kept constant during the entire growth process. But a distinct can of the resistance gradient is present in the resistance

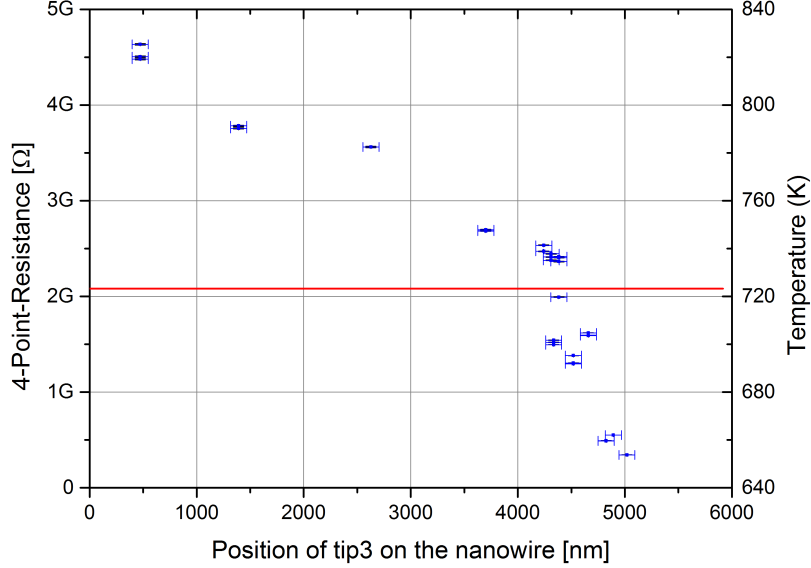


Figure 5.29: Constant Sample: For the Constant Sample, no temperature (red) dependence can be expected. The four-point resistance profile (blue data) on the other hand shows two different segments.

profile, as can be seen in [Figure 5.29](#)

One could argue, that the actual nanowire growth takes place at the interface between the gold particle and the nanowire. And that due to the constant flow of cooler gases and the growing distance from the sample, the temperature at that interface may differ from the temperature of the substrate. But, since the nanowires are only a few μm long, a temperature difference cannot be expected. And especially, a sudden change in the resistance gradient, however, cannot be explained in this way. Additionally, the change in the resistance profile shows a higher resistance gradient for lower temperatures, which does not match the expected temperature resistance described above.

In general, no direct correlation between the temperature and the resistance can be found for these samples. At low temperatures, some features match the temperature profiles, while at high and intermediate temperatures no correlation could be found. For a conclusive interpretation of the results additional parameters have to be taken into account. In the following, a closer look on the shape of each nanowire will be given and the one-dimensional transport model mentioned above will be applied to the findings of the three samples.

5.5.2 HR-SEM

Besides the temperature, the nanowire radius is also a very important parameter. Before the samples were introduced into the RT-MT-STM, high resolution SEM

images were made of some random nanowires. In order to be able to interpret the measured resistance profiles correctly, high resolution SEM images were taken after the resistance measurements were finished. The same HR-SEM was used to take the images before the samples were transferred into the RT-MT-STM. In contrast to the previous HR-SEM, images could now be taken from the very same nanowires that were used for the four-point resistance measurements. The three HR-SEM images can be found in [Figure 5.30](#). The structure of the nanowire basis can be seen. Especially for the Constant Sample, phase changes (horizontal lines) and irregularities can be seen. Such phase transitions ([\[41\]](#)) in GaAs nanowires are known to have no influence on the conductivity through the wire [\[40\]](#).

At the borders between the regions, the resistivity of the nanowire changes drastically. In the high resolution SEM image no reason (like a kink, a narrowing or a damage) can be identified. These detailed images could be used to measure the diameter of each nanowire with very high precision (~ 2 nm). The resulting measured diameter of the nanowires used for four-point resistance measurements are shown in [Figure 5.31](#).

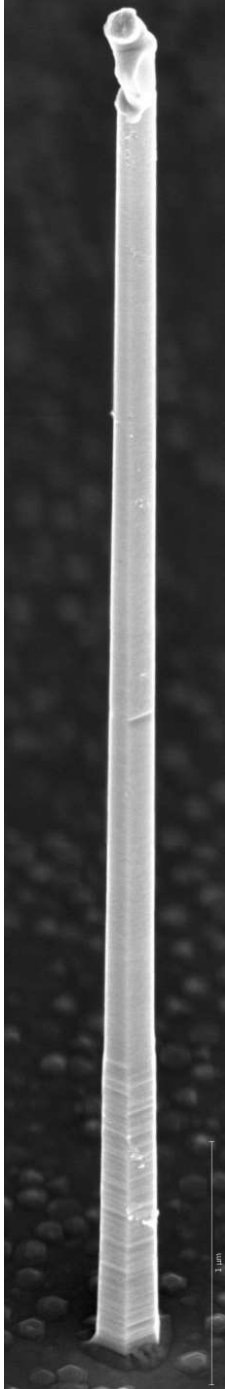
In these profiles, the gold particle at the top of each of the three nanowires can be made out as the peak at the end. Additionally, the nanowires on the Constant and the one on the Step Sample show a distinctive base. These features – gold particle and base – are also visible in the HR-SEM. Additional features can also be found in the profiles. Most importantly, the diameter of the three nanowires can be compared quantitatively. The diameter of the Gradient Sample nanowire is the smallest, followed by the nanowire on the Constant Sample, and finally the one on the Step Sample.

The fact that the nanowire with the largest radius has the smallest resistance makes sense. In order to find out if the small difference in the width between the Step Sample and Constant Sample nanowire can make such a difference, and why the resistivities of the nanowires on the Constant and the Gradient Sample are similar in the beginning, the one-dimensional transport model will be used in the next section.

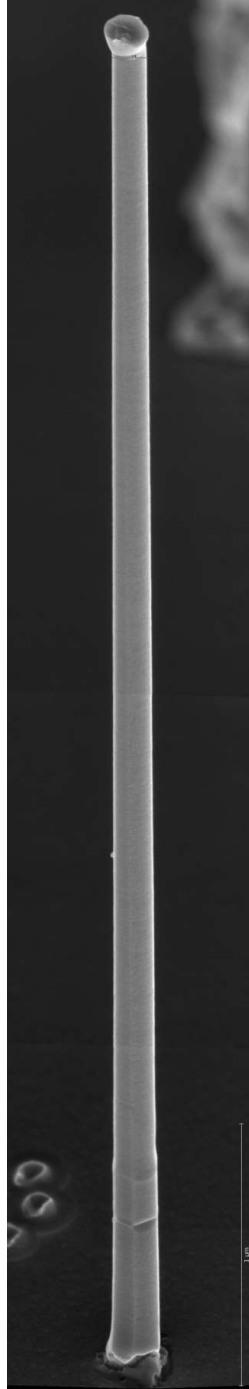
5.5.3 One-dimensional transport model

The one-dimensional transport model applied here calculates the size of the depleted area at the surface of the nanowire ([Figure 5.32a](#)) based on an estimation of charge carrier mobility against their concentration. Another very important parameter is the total radius of the nanowire. This model also takes into account how much area of the nanowire participates in the charge transport.

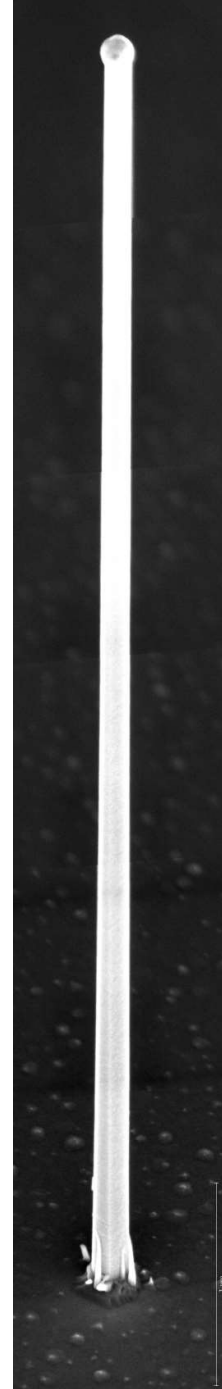
In [Figure 5.32](#) the electrical structure of a p-doped GaAs nanowire is shown. The band structure in the center of a thick nanowire is unperturbed by the surface ([Figure 5.32b](#)). A 1.42 eV band gap lies between the valence and the conduction band. The additional states due to the p doping with Zn are situated close to the valence band within this band gap. The Fermi level adjusts between the acceptor level and the valence band. By thermal excitation, electrons leave the valence band into an acceptor state, leaving holes in the valence band. The excited electrons as



(a) Constant Sample
compare [Figure 5.29](#)



(b) Step Sample
compare [Figure 5.28](#)



(c) Gradient Sample
compare [Figure 5.27](#)

Figure 5.30: HR-SEM images of the three nanowires used for four-point measurements are shown in these pictures. The resistance profiles from each nanowire can be found in the Figures listed below each picture. From these images, structure and radius at along the nanowire can be derived.

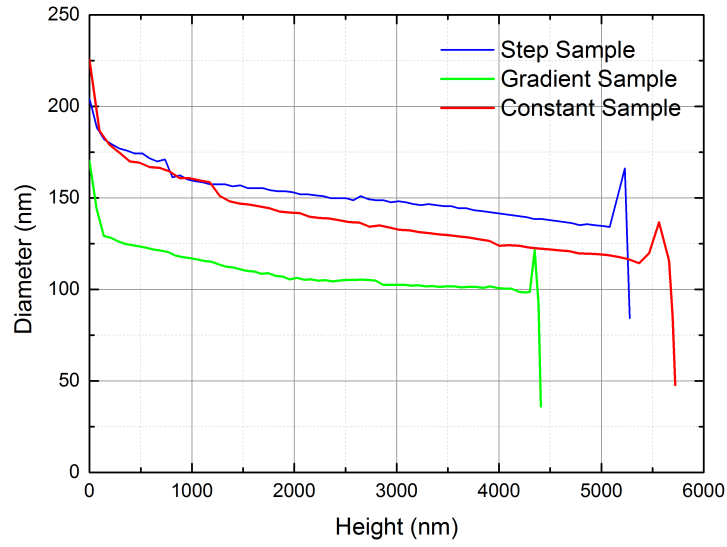


Figure 5.31: From the HR-SEM images shown in [Figure 5.30](#), the diameters of the nanowires used for four-point resistance Measurements could be measured. For each sample a typical profile is shown. The diameter of all nanowires drops very fast during the first few nanometers. The Constant Sample and Step Sample nanowires show an additional base of 800 nm and 1200 nm. The gold particles correspond to the peak at the end of each profile, respectively.

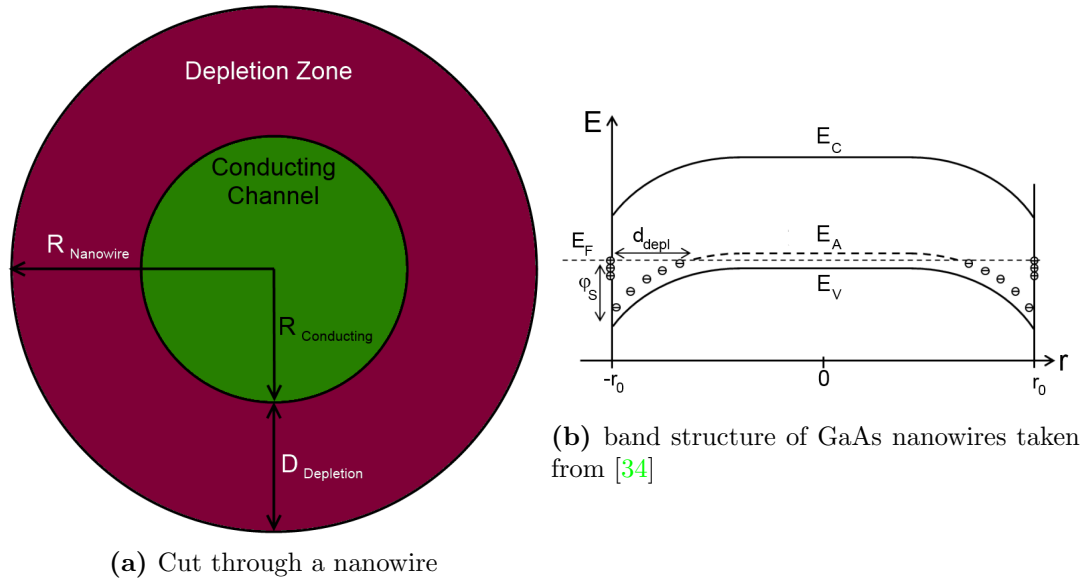


Figure 5.32: (a) A cut through a nanowire shows the conducting channel in the center of the nanowire, surrounded by the depletion zone at the surface of the nanowire. In (b), a band structure through the nanowire is plotted. In the center, the band structure of a nanowire is identical to the band structure of an infinite crystal. At the vacuum-nanowire interface, surface states bend the band structure down by 0.5 eV. A depletion zone forms.

well as the holes participate in the charge transport through the nanowire. The bands are continuous through the entire nanowire. On the surface of the nanowire, new states arise due to the end of the crystal lattices. These extra states bend the conduction and valence band down in case of a p-doped semiconductor. On the surface, a depletion zone forms. Due to the absence of free charge carriers, this region cannot participate in the charge transport. The size of the depleted area d_{depl} can be calculated with the dielectric coefficient of the vacuum ϵ_0 and of the nanowire ϵ_r , the surface potential of the nanowire ϕ_s and the charge carrier concentration N_A :

$$d_{depl} = \sqrt{\frac{2 \cdot \epsilon_0 \cdot \epsilon_r \cdot \phi_s}{e \cdot N_A}} \quad (5.1)$$

While the constants are known, the incorporation of acceptors into the nanowire (= charge carrier concentration N_A) has to be determined. The transport model can translate a resistance profile, together with the thickness of the nanowire, into a profile of the charge carrier density.

The resistance R of a nanowire can be expressed as:

$$R = \int \frac{\rho(z)}{A(z)} \cdot dz \quad (5.2)$$

with the resistivity $\rho(z)$ and the cross section conducting area $A(z)$.

If the area and resistivity are constant over a length L of a nanowire, [Equation 5.2](#) can be simplified to:

$$R = \rho \cdot \frac{L}{A}. \quad (5.3)$$

If not the entire nanowire, but only a small part with the length L is investigated, these requirements are fulfilled since the diameter of the nanowire, as well as its resistance, do not change rapidly on small scales, as can be seen in [Figure 5.31](#) and [Figure 5.27](#), for example. For a cylindric or cone shaped nanowire with the radius r_0 , the conducting area A is:

$$A = \pi \cdot (r_0^Z - d_{depl}^Z)^2 \quad (5.4)$$

The resistivity ρ can be expressed as:

$$\rho = \frac{1}{\sigma} = \frac{1}{N_A \cdot e \cdot \mu} \quad (5.5)$$

with the conductivity σ , the elementary charge e and the charge carrier mobility μ .

Finally, also the charge carrier mobility can be expressed as a function of the charge

carrier density N_A ([23], [42], [43]):

$$\mu(N_A) = \frac{450 \frac{cm^2}{Vs}}{1 + \sqrt{\frac{N_A}{10^{18} cm^{-3}}}} \quad (5.6)$$

If now Equation 5.4, Equation 5.5 and Equation 5.6 are inserted into Equation 5.2, an equation for the resistance R results:

$$R = \int_{Substrate}^{Nanowire\ top=L} \frac{1}{e \cdot N_A^Z \cdot \frac{450 \frac{cm^2}{Vs}}{1 + \sqrt{\frac{N_A^Z}{10^{18} cm^{-3}}}}} \cdot \frac{1}{\pi \cdot (r_0^Z - \sqrt{\frac{2 \cdot \epsilon_0 \cdot \epsilon_r \cdot \phi_s}{e \cdot N_A^Z}})^2} dz \quad (5.7)$$

With this equation the measured four-point resistance R can now be transferred into a charge carrier density N_A^Z , as long as the radius r_0^Z is known.

For the proper usage of this equation a reminder of the measurement technique is necessary. One of the two voltage measurement tips is located at the top of the nanowire close to the Current Injection Tip. The other is moved along the nanowire. The result is always the resistance between the two tips. If the charge carrier density is calculated for each point in a resistance profile, Equation 5.7 treats the resistance as homogeneously distributed along the entire length between the two voltage tips. Furthermore the changes of the radius of a nanowire are not taken into account. Since Equation 5.7 is neither a linear function of the radius, nor the charge carrier density, such averaging is far from beneficial.

In order to derive the charge carrier density with higher accuracy, Equation 5.7 can be modified to:

$$\frac{dR}{dL} = \frac{1}{e \cdot N_A^Z \cdot \pi \cdot \frac{450 \frac{cm^2}{Vs}}{1 + \sqrt{\frac{N_A^Z}{10^{18} cm^{-3}}}}} \cdot (r_0^Z - \sqrt{\frac{2 \cdot \epsilon_0 \cdot \epsilon_r \cdot \phi_s}{e \cdot N_A^Z}})^2 \quad (5.8)$$

The resistance gradient is known from the four-point resistance profiles (Figure 5.19, Figure 5.24 and Figure 5.22b), while the radii have been measured with the high resolution SEM (Figure 5.30) and are shown in Figure 5.31. Now all data are known to use Equation 5.8 to derive charge carrier concentration and the radius of the conduction channel from the resistance gradient $\frac{dR}{dL}$ and the radius of the nanowire at each point.

While the charge carrier densities are in the order of 1E17 to 3E18 per cm^3 (Constant Sample and 1E17 to 2E17 per cm^3 , Step Sample 1E17 to 3E18 per cm^3 , Gradient Sample 2E17 to 2E18 per cm^3), the resulting conduction channel radii for the Constant and Gradient Sample are tiny. For most of the Gradient Sample its conduction channel is smaller the 2 nm . With less then 1 nm the Constant Sample radius is even smaller. Only the radius for the Step Sample (10 nm to 60 nm) has a consider-

able size. Especially at the bases of the nanowires, where the doping was executed insufficiently, the conduction channel sizes are so small, that the results have to be doubted. In conclusion, the one-dimensional transport model does not provide feasible results for the three samples analyzed in this study! A different model has to be applied.

5.5.4 Common features of all samples

Even though the electric properties of the nanowires from the three samples differ, they can be included into a single model describing the properties of all three samples. Each nanowire can be divided into three regions (Figure 5.33a). From the one-dimensional transport model (subsection 5.5.3) it could be conducted, that the upper parts close to the gold seeds (region 1) are highly p-doped, while the nanowire bases (region 3) are very low or even undoped. Between these two regions, a transition region (region 2) exists. For the three different samples, only the sizes of each region and the doping level in these regions differ. The basic characteristics of the regions are the same for all three samples.

In Figure 5.33c and Figure 5.33d the band structure along the nanowire as well as the band structures across the nanowires in the three regions are depicted. Due to the surface state at $\Phi_{SS} \approx 0.5 \text{ eV}$ above the valence band, the surface Fermi level is pinned in the middle of the band gap. Thus the bands are bent and a depletion zone forms at the surface of each nanowire. Only the nondepleted channel in the center of the nanowire participates in the charge conduction. Its radius r_{el} depends on the dopant concentration as shown in Figure 5.33b. The bands at the surface (CB^{surf} and VB^{surf}) on the other hand are flat along the entire nanowire due to the surface Fermi level pinning.

A low dopant concentration reduces the conductivity in two ways. The resistivity is higher for lower dopant concentrations. Additionally, the radius of the conducting channel inside a nanowire is reduced, if the charge carrier concentration is low. After the channel opens in region 2, it is fully open in region 1. In region 3 the channel is closed (almost) completely. There, the electrical radius is reduced to 0. With further decreasing charge carrier concentration, the band becomes flatter as the valence band edge move away from the Fermi level.

In region 3 no current is conducted through the center of the nanowire. The conductance that has been measured in the previous described experiments must originate from another channel, such as surface conductance.

The different regions in the resistance profiles are shown in Figure 5.34. Not all three samples show all three regions, as will be discussed later.

In Figure 5.35 the resistance profile of the Gradient Sample nanowire is compared to a high resolution SEM image of the same nanowire. Additionally an IV from each region is presented to demonstrate the evolution of the four-point IVs along the nanowire.

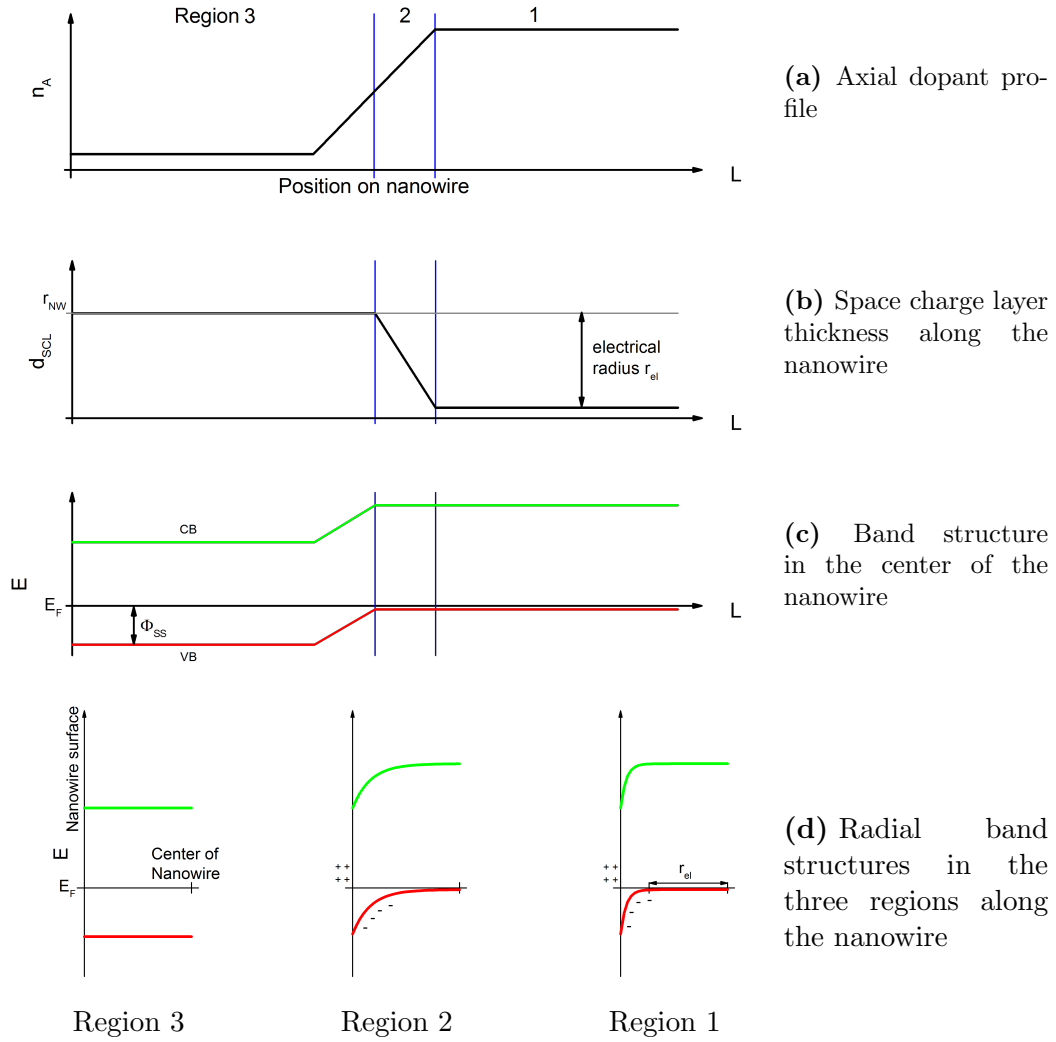


Figure 5.33: The nanowire can be divided into three regions along its axis. Idealized dopant profile (a), space charge layer thickness (b) and band structure are shown in this figure together with radial band structures (d) from each region.

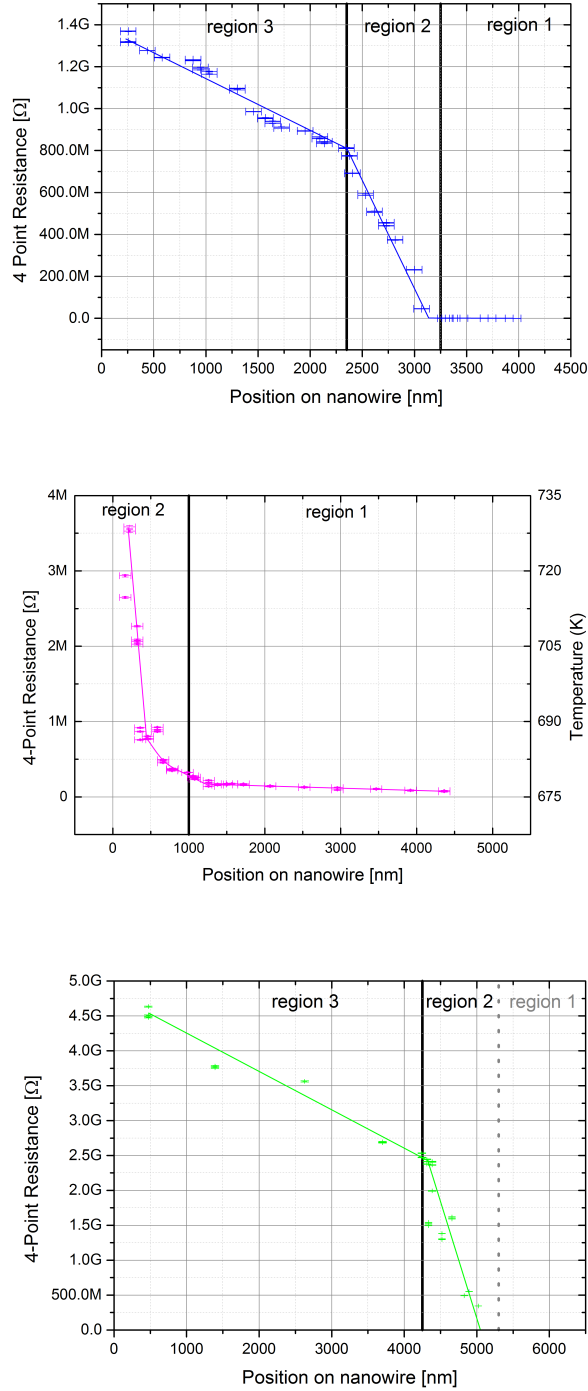


Figure 5.34: Based on the electrical features presented in Figure 5.33, the resistance profiles of the three samples can be split into regions with common features. While the Gradient Sample (a) shows all three regions, the Step Sample (b) and the Constant Sample (c) show only two regions.

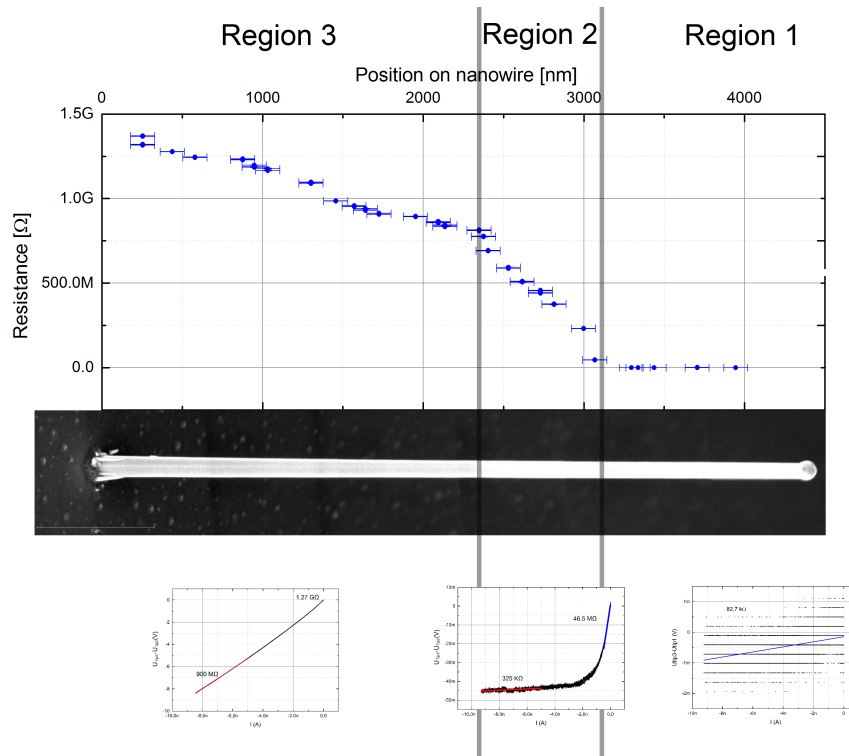


Figure 5.35: For a nanowire from the **Gradient Sample** the resistance profile (top) is compared to a high resolution SEM image (middle) of the same nanowire. Additionally an IV from each region is present.

5.5.5 Surface conductance

From the very high measured resistances at the nanowire bases it is concluded that the GaAs in region 3 is intrinsic or very low doped. For a flat band structure the valence band lies $\Phi_{SS} \approx 0.5 \text{ eV}$ below the Fermi level. In bulk GaAs a 0.5 eV gap between valence band and Fermi level occurs at a dopant concentration of about 10^{11} cm^{-3} . In this case the resulting resistance exceeds the resistances measured in the nanowire bases by far. While the conduction channel inside the nanowires is closed, the surface states which pin the Fermi level are available for charge transport.

In their paper [44] J.J. Mares et al published results for the electrical conductivity of semi-insulating GaAs surfaces at different temperatures. At room-temperature, the measured surface conductivity of semi-insulating GaAs is in the order of $\sim 10^{-9} \text{ S}/\square$. With a diameter between 150 nm and 200 nm, the surface conductivity can be transformed to a resistance gradient roughly between $300 \frac{\text{M}\Omega}{\mu\text{m}}$ and $600 \frac{\text{M}\Omega}{\mu\text{m}}$.

The resistance gradients of the completely depleted region 3 are expected to lie within this range for each sample. The measured resistance gradient of the Step Sample however is much smaller implying that a conduction channel is present from the gold particle down to the substrate. For the other two samples, the measured resistance gradients close to the substrate lie within the regime expected for surface conductance.

In Figure 5.36a a simplified equivalent circuit diagram is shown. The nanowire is split in three segments based on Figure 5.33. The current paths through the core (red) and the surface (blue) are displayed as resistors (R_X^C and R_X^{SS}). When a conducting channel is present in the nanowire, it can be assumed, that its resistance is much smaller than the one through the surface $R^C \ll R^{SS}$. Thus, if present, the path through the core is preferable for the current. The measured current voltage relations are not linear. To take this into account, a diode represents the non ohmic transition in the current path from the core of the nanowire to its surface. The expected IVs according to each segment in the equivalent circuit diagram are schematically shown in Figure 5.36b to Figure 5.36d.

In region 3 only surface conductance is available, a pure ohmic IV results. Both current pathes are available in region 1. Since the conductance through the core is much better than on the surface, this path is preferred by the current. In this region the expected IV is pure ohmic as well, but with a much smaller resistance.

The conductance in the transition zone (Figure 5.33c), however, is not ohmic, as the IVs in this area are not linear (Figure 5.17, Figure 5.20, Figure 5.21). Thus it can be modeled by an element with a non-linear IV. Additional current can flow over the surface of the nanowire. The nature of this non-linear element will be discussed later. If a certain threshold voltage is exceeded the current can flow through an additional channel, the nanowire core, with low resistance. Below the threshold voltage, the only current path left is via the surface.

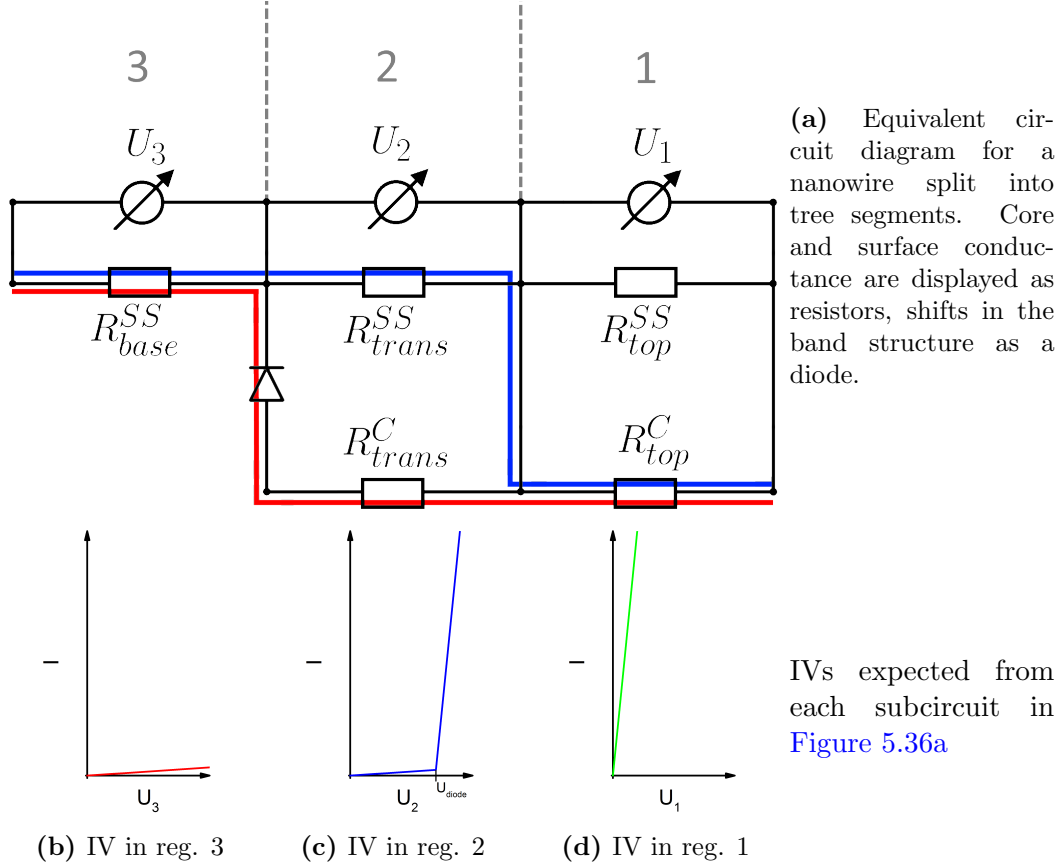


Figure 5.36: Equivalent circuit diagram for a nanowire. Core and surface conductance are displayed as resistors, the transition through the depletion zone as a diode. Here it is assumed, that the surface conductance is nearly constant on the entire nanowire, while the characteristics of the transition diodes and the core resistances changes drastically. For the Constant and the Gradient Sample, the core conduction channel is closing at some point ($R_{core}^X \rightarrow \infty$), and only surface conductance remains.

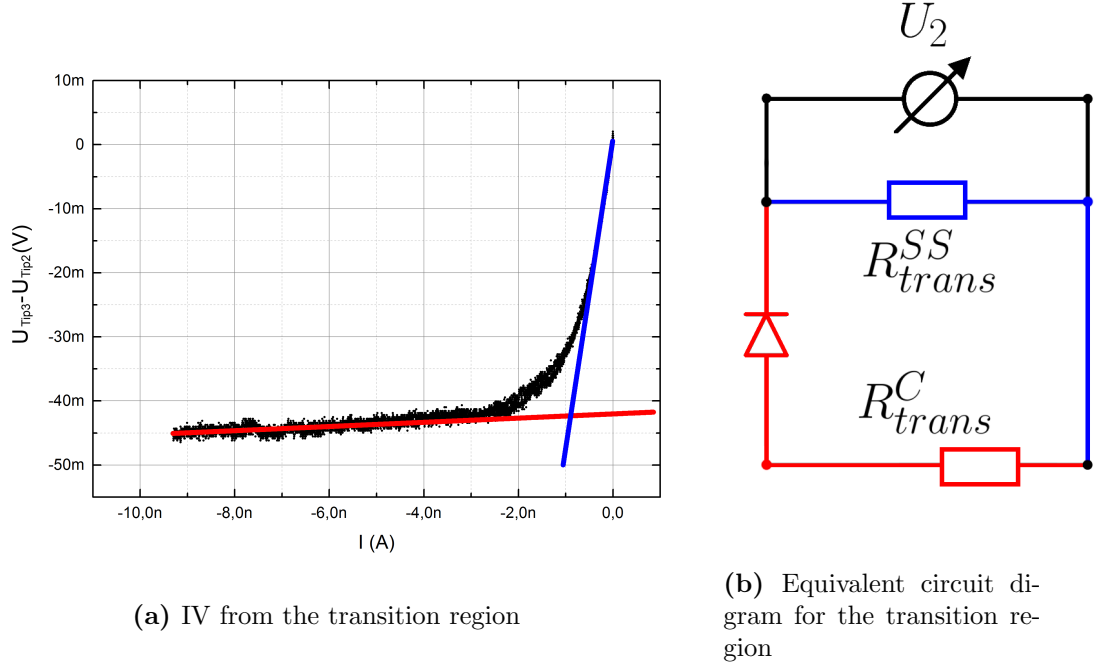


Figure 5.37: In (a), a IV from the transition region of a Gradient Sample nanowire is fitted between 0 A and -0.5 nA as well as between -5 nA and 9.5 nA. The fits represent the two resistors shown in the equivalent circuit diagram in (b).

5.5.6 Offset voltage

From the form of the four-point IVs additional information on the current path through the nanowire can be gathered. R_{top}^C , R_{trans}^{SS} , R_{trans}^C , R_{base}^{SS} and U_{diode} can be extracted from the IVs shown in Figure 5.17, Figure 5.20 and Figure 5.21. The resistances of the current paths through the core (red in Figure 5.36b) and via the surface (blue in Figure 5.36b) in the transition region have been obtained from the IV as shown in Figure 5.37, by fitting different current regimes. For the low current fit (blue) the path through the core is closed and the current has to flow through the surface channel. For the high current fit (red), on the other hand, this path is available and the current can flow through the nanowire core with lower resistance.

While the low current fit crosses 0 V at 0 A, the high current fit has a voltage offset. In the picture of the equivalent circuit diagram, this offset is the voltage threshold, from which the current path through the core in the transition region becomes available.

In regions 1, no offset is expected, due to the pure ohmic conductance. In the transition region 2, the offset has to grow with increasing resistance. No further increase of the offset is expected on the nanowire base, since according to the model, the base is pure ohmic too.

For the Gradient Sample (Figure 5.38), the Step (Figure 5.39) and the Constant

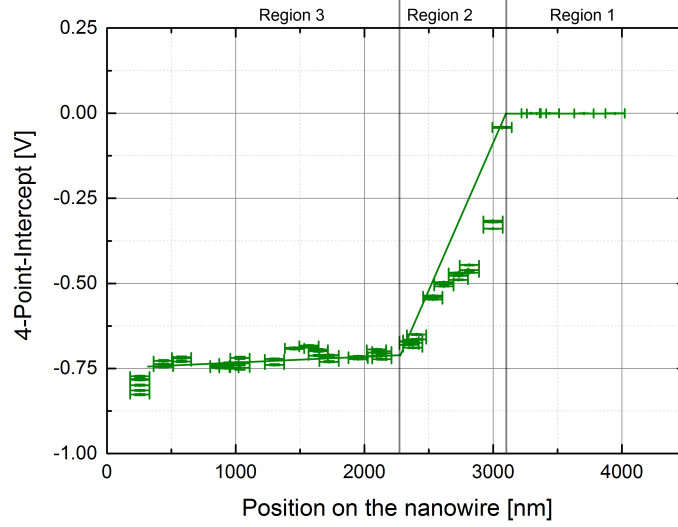


Figure 5.38: The offset of the high current fits in the four-point IVs are plotted vs. their position along a Gradient Sample nanowire. These offset voltages are based on the analysis of the 4-point IVs shown in [Figure 5.17](#)

Samples ([Figure 5.40](#)), offset profiles are shown. The Gradient Sample nanowire shows the expected profile. The two flat plateaus around 0 V and -0.75 V can be assigned to the regions 1 and 3. In between these two regions, the offset grows in the transition region 2.

For the other two Samples, the measurements do not show all three regions, as outlined before. For the Step Sample, region 1 is extended and clearly present. From the top of the nanowire a long flat plateau spreads at 0 V. The transition region however, following this plateau covers the 1000 nm above the base of the nanowire. The Step Sample nanowire does not have a region 3.

For the Constant Sample, the profile begins with the transition region. Since the first point of the offset profile is close to 0 V and the top most nanowire part was not included in the measurement, it is highly probable, that region 1 follows the measured profile. The measured offset voltages for region 3 are scattered between -0.5 V and -1 V.

5.5.7 Resistance profiles

Based on the equivalent circuit diagram ([Figure 5.36a](#)) not only the IVs in each region can be modeled, but the entire resistance profile along the nanowire, as shown in [Figure 5.41a](#).

If it is assumed, that the resistivity of the conducting channel in the nanowire core as well as the resistivity of the surface are constant along the nanowire, resistance profiles with two constant slopes result. The shape however differs for low (blue in [Figure 5.41a](#)) or high (red) currents.

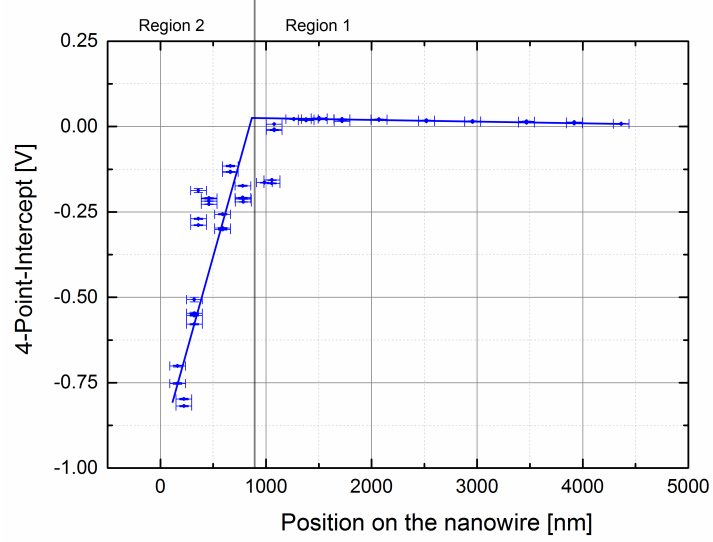


Figure 5.39: For the Step Sample, the high current offset is plotted as a function of the position of the corresponding four-point IV. These offset voltages are based on the analysis of the 4-point IVs shown in [Figure 5.21](#)

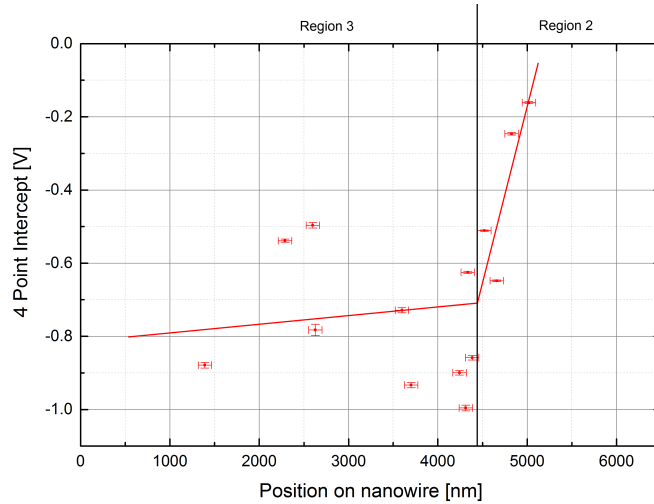


Figure 5.40: For the Constant Sample, the high current offset are plotted as a function of the position of the corresponding four-point IV. These offset voltages are based on the analysis of the 4-point IVs shown in [Figure 5.20](#)

For low voltages (or low voltages respectively) the path to the nanowire core in the transition region is closed and the current flows through the surface state until it can reach the nanowire core in region 1. For high voltages on the other hand, the this path is available and the currents can flow longer (region 1 and 2) through the conducting core.

The offset voltage and the resistance difference between the two current paths is generated only in the transition region, where the current is flowing through different paths. In region 1 and 3 the resistances and thus the difference between high and low current are constant.

If the prediction of the model (Figure 5.41a) is compared to the measured resistance profiles (Figure 5.41b to d), some features can be recognized. The profile of the Gradient Sample nanowire (Figure 5.41b) shows all three regions. In region 1 and 3 the two profiles are identical with respect to the precision of the measurement. The difference between the two fits ΔR arises completely from the transition regions.

In the transition region however, the resistance gradients differ from the one in the other two regions. Here the resistance gradient is highest. In subsection 5.5.9 this change of resistivity in the transition region will be discussed.

The Constant Sample and Step Sample profiles show only two of the three regions, as shown in Figure 5.34. On the Step Sample only the regions 1 and 2 were measured. In region 1 the three fits for positive bias, no bias and negative bias are identical, while difference is growing for the rest of the nanowire.

The Constant Sample resistance profile shows only the regions 2 and 3. The difference between the two fits is almost constant in region 3. In the transition regions this difference is growing with increasing distance from the nanowire top. While the high current profile is nearly flat, as expected from the model, the low current gradient is higher than in region 3, as for the Gradient Sample.

5.5.8 Interpretation of the non-linear voltage current relation

The four-point IVs along each nanowire showed a nonlinearity (which is represented by the diode in the equivalent circuit diagram) up to a certain point along the nanowire. This nonlinearity implies a change in band structure along the current path.

The pn-junction between the nanowire and the substrate could be identified as the source for the diode like characteristic in the two-point IVs (Figure 5.12). Also, the three-point IV of the Step Sample showed a nonlinear characteristic (Figure 5.14). There, the origin was a Schottky barrier between the tip and the nanowire. For the nonlinearities in the four-point IVs, however, no explanation was presented so far.

In Figure 5.42, the difference between the two fits along the nanowire on the Gradient Sample is shown. This difference can be used to locate regions in the nanowire where the IV shows a diode like behavior. Additionally the intercept of the high current fits of the Gradient Sample were plotted along the nanowire axis in Figure 5.38.

Two flat plateaus are present in this plot. In these flat areas no bias dependent resistance is generated. Only the step between the plateaus contribute to the non-linear characteristics of the entire nanowire. With other words, the origins of the

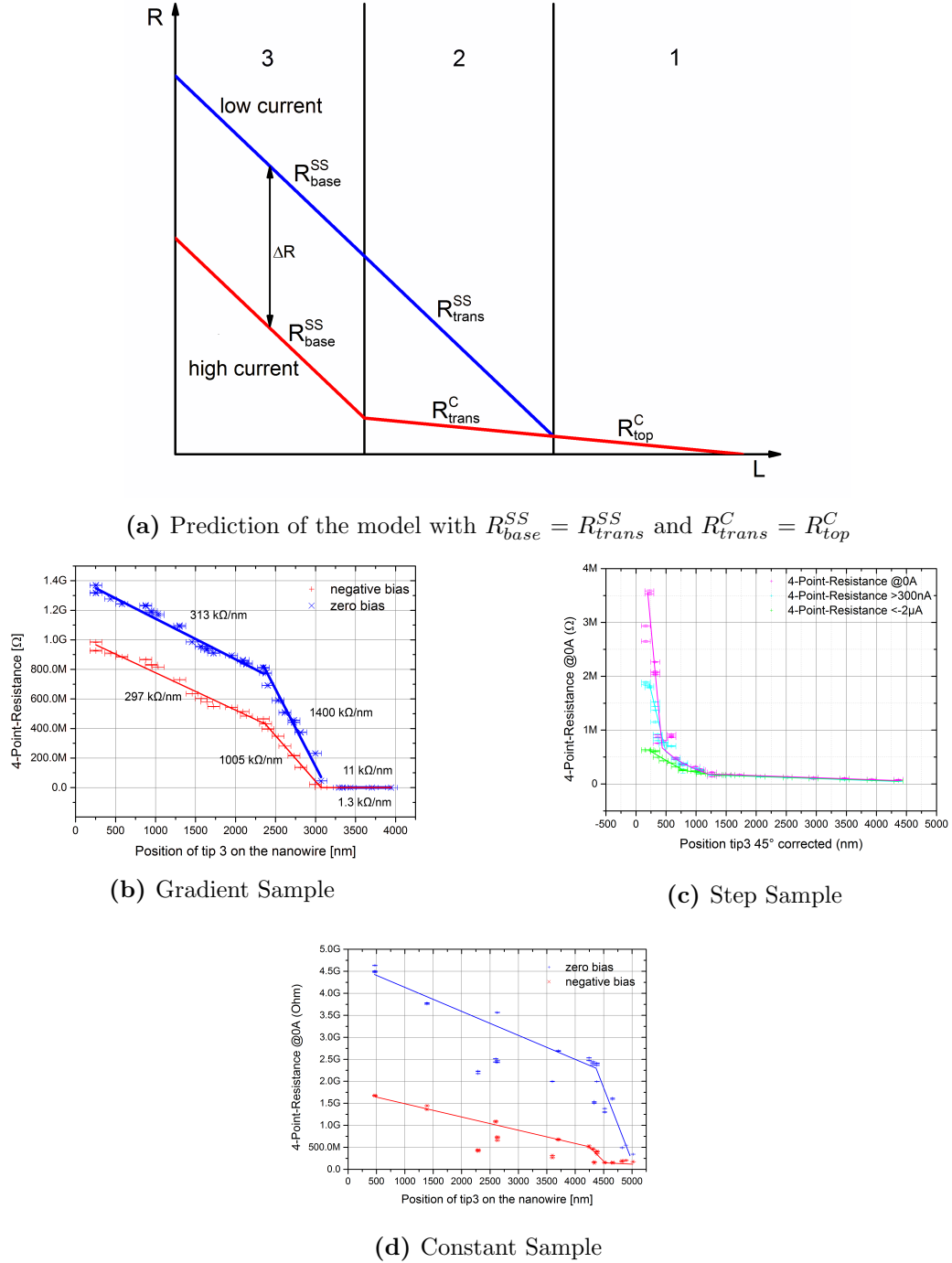


Figure 5.41: (a) A resistance profile along a nanowire is modeled according to the equivalent circuit diagram shown in Figure 5.36a. When the path through the core is available, the resistance is R^C otherwise R^{SS} . In the measured resistance profiles (b) to (d) several features can be recognized from the prediction.

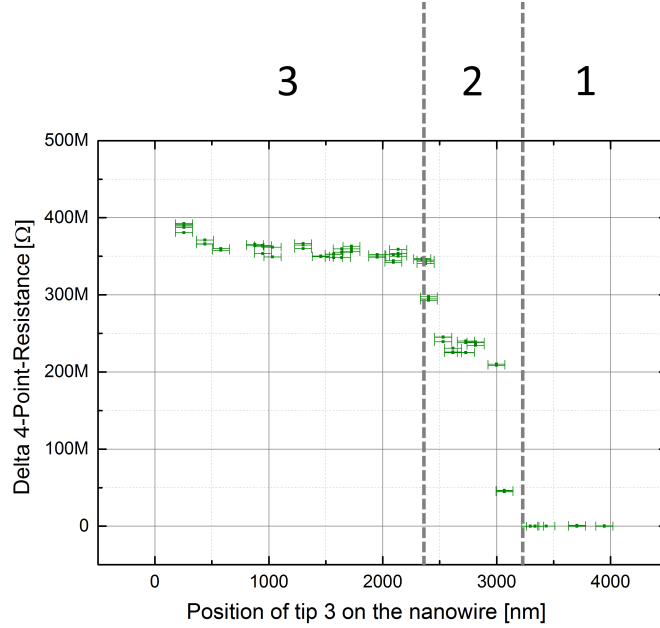


Figure 5.42: The difference between the fits at 0 A and at high currents for the Gradient Sample. Three flat segments are separated by two large steps.

nonlinear voltage current relation are well located in this step.

Another look at the current path is necessary to find the origin of the nonlinearity of the four-point IVs. The current is flowing from the gold seed through the core of the nanowire onto its surface and from there via the substrate out of the sample. The different available current paths are displayed in [Figure 5.43a](#) together with the band structure along the nanowire in [Figure 5.43b](#). Additionally the band structure across the nanowire at the interface between region 2 and 3 is shown in [Figure 5.44a](#).

Due to the small depletion zone in region 1 the surface state is well coupled to the conduction channel in the core, as the low resistance gradients measured in this region indicate. The wider depletion zone in region 2 and 3 disconnects the two channels. If a voltage is applied to the substrate, the surface states move away from the Fermi level of the nanowire core (E_F^{NW}), as shown in [Figure 5.44b](#).

Due to the Fermi level pinning valence and conduction band flatten, when a negative voltage is applied to the substrate. When the bands are completely flat, conduction through the depletion zone is enabled and the current path through the core is available.

At the interface between regions 2 and 3 valence and conductance bands are flat. More in the center of region 2 the band bending is weaker and a larger voltage is needed. In the center of region 2, for example, twice as much voltage ($U_2 = 2 \times \Phi^{SS}$) is needed as at the interface to enable charge transport through the nanowire core. Thus the transition from the surface states to the conduction channel in the nanowire is localized at the interface between region 2 and 3 and not spread over a larger area.

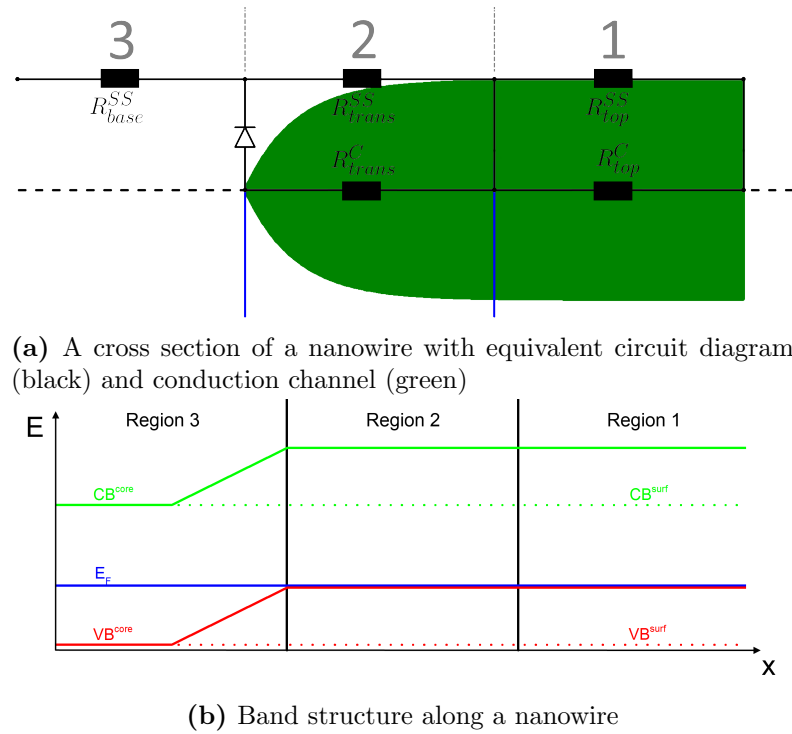


Figure 5.43: A cross section of a nanowire is presented in (a). In this cross section additionally the equivalent circuit diagram is shown. The conduction channel is presented too. In (b) the band structure in the center of the nanowire without applied voltage is presented. When the valence band reaches undepleted bulk level, the conduction channel starts to grow in diameter.

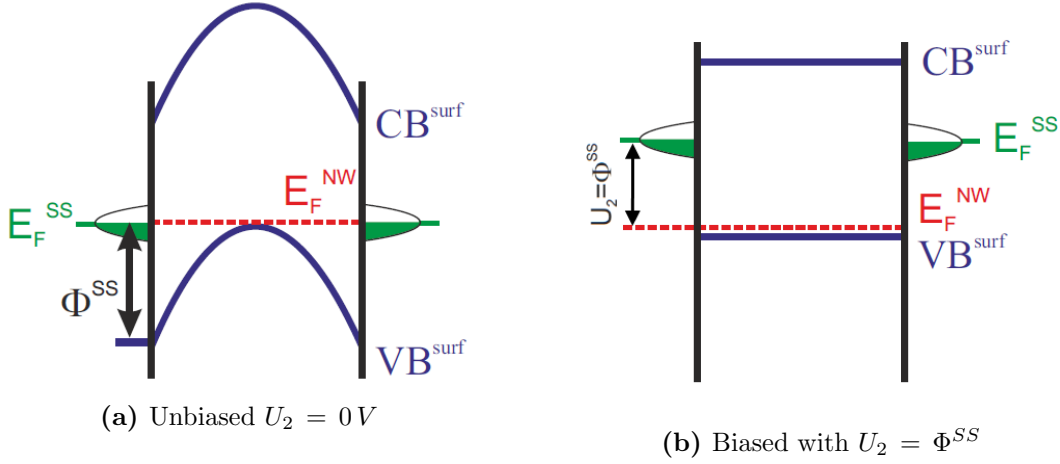


Figure 5.44: The band structure across a nanowire at the border between region 2 and 3 is presented. In (a) no bias is applied. The surface states pin the Fermi level (Φ^{SS}) above the valence band at the surface. Band bending in the bulk of the nanowire result. If a voltage $U_2 = \Phi^{SS}$ is applied to the surface, the bands flatten and a current can flow from the surface into the bulk. [?]

5.5.9 Peaking Resistance

In each region the current can flow through the surface channel. When other channels become additionally available and the surface channel does not change, the resistance gradient can only decrease, but never increase beyond the one of the surface resistance. Thus the maximal measured resistance gradient in these experiments should be $R_X^{SS}/L = 600 M\Omega/\mu m$. But in the Constant Sample as well as in the Gradient Sample profiles the resistance gradient is increasing in the middle of the nanowire. Here resistance gradients over $\frac{R_X^{SS}}{L} = \frac{1000 M\Omega}{\mu m}$ were measured. In subsection 5.5.7 it has been assumed, that the surface resistance per length is constant in regions 1, 2 and 3. This assumption may not hold. And the surface channel may change along the nanowire. While the total radius of the nanowire is changing very little in the transition region (Figure 5.31), the charge carrier concentration does.

In region 3, where the nanowire is undoped (or very low doped), the bands are flat and the depletion zone is spread out over the entire nanowire. This situation is shown in Figure 5.45a. The surface states are located at the Fermi level and current can be conducted with a resistance of $0.6 \frac{G\Omega}{\mu m}$. The surface is uncharged.

If now additional states (acceptors) between surface states and valence band are introduced into the bulk by doping, charge is flowing from the surface state back into the bulk. Thus the charge neutrality level (E^{CNL}) is separated from the Fermi level. As the depletion zone becomes thinner, the surface states become less populated and the density of states at the Fermi level is decreased. As can be seen in Figure 5.45b. The conductivity of the surface depends on the number of available states. Thus a

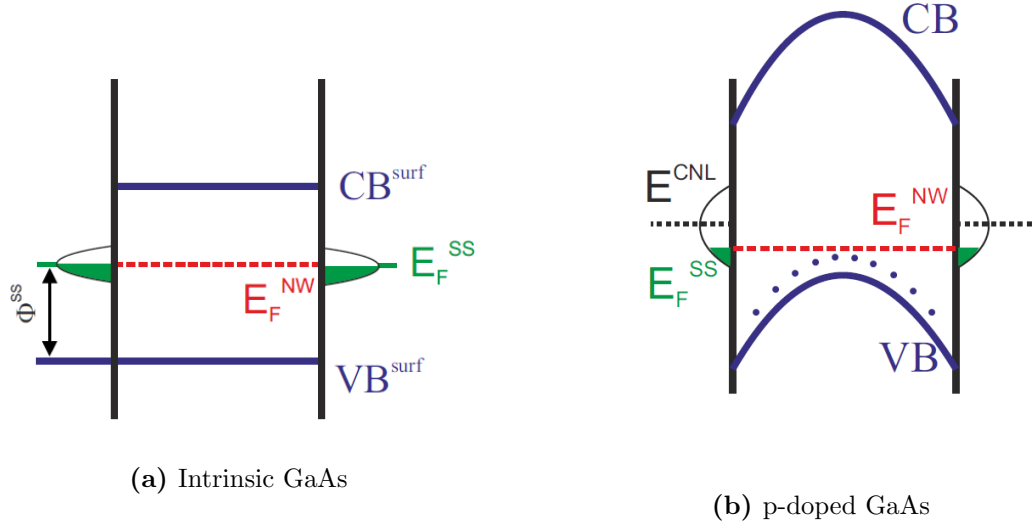


Figure 5.45: In (a) the band structure of intrinsic GaAs is shown. The Fermi level is pinned by the surface states Φ^{SS} above the valance band. The charge neutrality level (E^{CNL}) of the surface is identical to the Fermi level of the bulk (E_F^{NW}) and the surface (E_F^{SS}). If the GaAs is p-doped, charge is transferred from the surface states onto the acceptor states and a depletion zone forms. The surface states are less populated and the charge neutrality level of the surface is above the Fermi levels.[?]

lower density of states at the Fermi level increases the surface resistance considerably.

5.6 Electron Beam Induced Current (EBIC)

During the charge-transport-measurements, an interesting difference in the two available SEM-modes was noticed. In images taken with Sample-Current as source for contrast, a substructure on all nanowires could be seen, but only when the nanowire was contacted with at least one tip in current measurement mode. So, not only the topology of the sample, but also some electrical features could be investigated with the SEM.

In [Figure 5.46](#), two images of the same setup are shown. These two images were taken directly one after another, no changes were performed in the meantime and no bias was applied. The only difference between them is the contrast mode they were taken in. The two contrast modes – Sample Current and secondary electron multiplication – are presented in [subsection 3.2.3](#). The picture on the right was taken with the secondary electron detector (SED), while the left one was taken using a FEMTO to amplify the sample current and convert it to a voltage signal. The two modes have different properties and different features can be seen. During the four-point resistance measurements, the following characteristics of the additional Current Image signals were found:

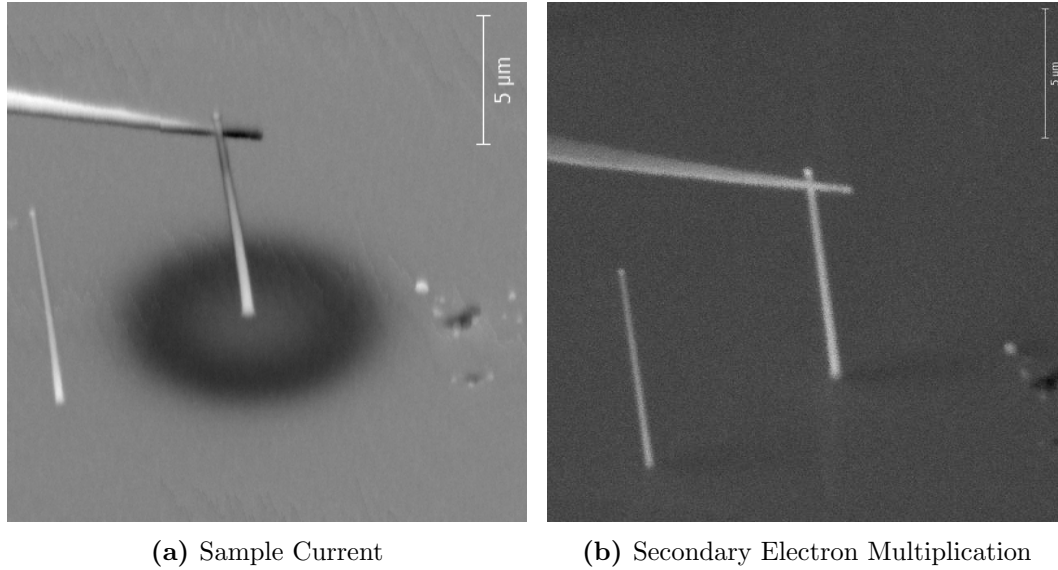


Figure 5.46: The two images show the same picture taken with two different contrast modes, Sample Current and SED. No bias was applied while these images were taken. Different features differ in these two images. In the Sample Current image, for example, a dark halo around the nanowire base is present, which is missing in the SED image. A darker color in the picture corresponds to a larger current flowing into the substrate.

1. Only when contacted with tip in current measurement mode
2. Only in Sample Current mode
3. Located in areas with high resistance gradient
4. Stronger for nanowires with higher two-point resistance

In voltage measurement mode, an STM tip has no defined potential and a input resistance of $\sim 1 \text{ T}\Omega$. By contacting a nanowire with a tip in current measurement mode ([item 1](#)), on the other hand, the nanowire is contacted with a defined potential and a low input resistance. Current can flow through the sample and the nanowire. In the SED image, no difference between a contacted or non-contacted tip is present. Hence, the additional features do not display an effect on the emission and absorption of charged particle due to an electron bombardment. But the amount of current flowing through the sample depends on the position where the SEM electron beam hits the sample, since it is visible only in the Current Image ([item 2](#)). The contrast differences between the two pictures display an Electron Beam Induced Current (EBIC).

In [Figure 5.46a](#), a dark halo around the base of the nanowire is very striking. Additionally, a shadow on the nanowire beneath the Current Injection Tip is visible. It has been shown in the charge transport measurements that the later is a location with high resistance gradients.

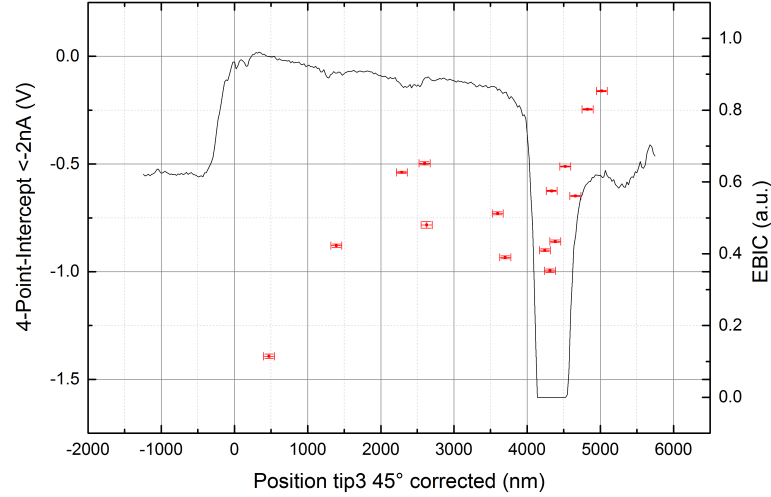


Figure 5.47: For the **Constant Sample**, the EBIC signal is compared to the voltage offset of the high current fit. The steep end of the intercept profile corresponds very well with the dip in the EBIC signal.

By bombarding the regions with high resistance gradients with electrons, the current flow through the sample is altered (item 3). A darker area in the sample current image corresponds to a larger current flowing through the sample. Either one of the two conduction channels through the nanowire (surface or core) is opened further or an additional low resistance path between the two channels is available. This results in a higher overall conductance (item 4).

Between the nanowire and the substrate a dark halo is present in the Current Images. If this area is hit with the electrons from the SEM, new free charge carriers are produced. These electrons and holes are available for charge transport and lower the resistance considerably. In this way, information on the electronic structure, even beyond the nanowire where four-point resistance measurements are very difficult to interpret, can be gathered.

The interface between the nanowire and the substrate is not the only electrical feature, which can be seen by comparing the two SEM images. Also, on the nanowire an EBIC signal is present. In Figure 5.47, the contrast along the nanowire is compared to the offset voltage of the high current fit from subsection 5.5.6. The position of the EBIC signal matches the position where the resistance difference between the low and the high current fit and the offset voltage are generated. At this point, the injection of additional free charge carriers decreases the two-point resistance considerably.

For the Gradient Sample such images are also available (Figure 5.48). The EBIC signal along the nanowire has several features in common with the four-point offset profile of the this nanowire.

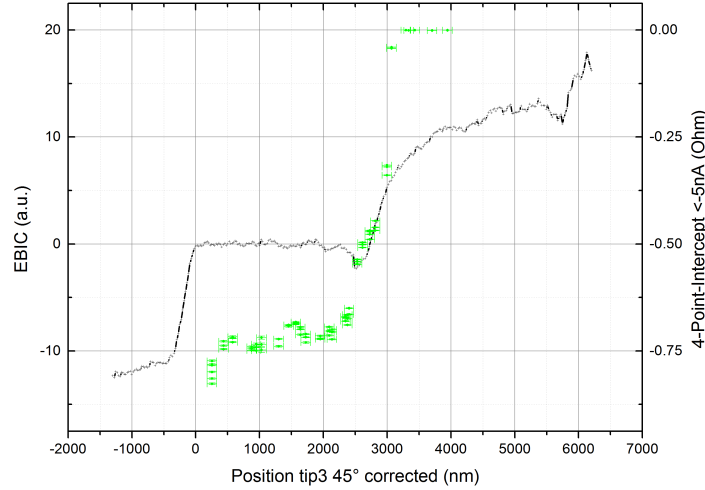


Figure 5.48: The intercept profile (green) and the EBIC signal (black) of the match very well for the **Gradient Sample**. In both signals a large step is present in the middle. Before that step both show a flat part.

EBIC signals can be used as an easy way to image electric features on a nanowire and on a nanowire substrate interface. The four-point resistance measurements and the EBIC signals showing similar features and support each other.

To understand the the dark halo around the base of the nanowire, more attention to the fourth contact to the nanowire is needed. The substrate is highly doped and thus conducting very well. When the nanowires are grown on it, a good contact is realized between the substrate and the nanowire. At the interface between the nanowire and the substrate, two different doping concentrations meet, which could in principal explain a non-linearity in the IV and thus an EBIC-signal, but not with a size of several micrometers.

Chapter 6

A closer look on Resistance measurements using a Multi-Tip Scanning Tunneling Microscope as Nanoprobe

In order to investigate the charge transport through GaAs nanowires, the RT-MT-STM is used as a nanoprobe. A nanowire is contacted with one to three scanning tunneling microscope tips.

A different approach to realize four-point resistance measurements along nanowires is to use lithography to produce four or more contacts ([Figure 6.1](#)). In this case, the nanowires have to be detached from the substrate and spread out flat on a non-conducting sample [\[45\]](#). In order to contact a nanowire it has to be located on the sample using an SEM. After that, leads can be fabricated lithographically with high precision to contact the nanowire ([\[46\]](#)).

In the top part of [Figure 6.1](#), an SEM image of such a sample is shown. The nanowire is contacted by six leads. The outer ones are used as source and drain for the current, while the inner ones measure the voltage drop on the nanowire. The result of such a measurement is shown in the lower part of the figure.

Due to the limited number of contacts, the spatial resolution of the measurement is quite poor. Also for each nanowire, individual contacts have to be fabricated after it has been localized in an SEM. In order to increase the resolution of the measurement, more contacts have to be fabricated, leading to more effort.

The RT-MT-STM used as nanoprobe provides an easy and fast way to measure four-point resistance profiles along several nanowires on a sample. Additionally, the nanowires do not have to be detached from the substrate they were grown on. The

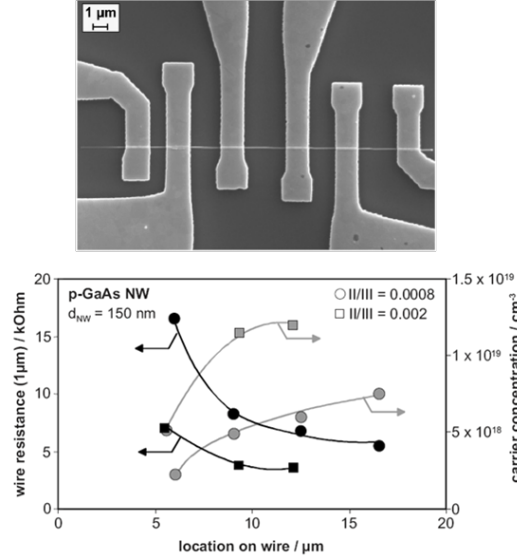


Figure 6.1: The lithographically fabricated sample used by Gutsche et. al. ([23]) for four-point resistance measurements is shown in the top. Six leads contact a nanowire which was removed from the substrate it was grown on. The results of the measurements performed with this sample are shown in the bottom figure. Due to the limited number of leads, the spatial resolution is much smaller than for the RT-MT-STM.

interface between nanowire and substrate can only be analyzed; it is still intact during the measurement.

The RT-MT-STM offers a superior new method of measuring four-point resistance profiles along as-grown nanowires, but it has to be validated first to demonstrate that it is useful for its task. The important points are that the method has to stable and deliver reproducible results. At the same time, this measurement technique does not introduce systematic error to the measurement. In order to test the use of a RT-MT-STM as a nanoprobe for resistance measurements on GaAs nanowires, several measurements have been performed. A closer look on the influence of the measurement on the result shall be taken. In section 6.1 the stability of the measurement demonstrated. After this, the influence of a tip on the measurement due to its electrical field is shown in section 6.2.

6.1 Stability Measurements

Two measurements were performed to validate the stability of the measurement. First, a constant voltage is applied to a nanowire for a long time, compared to a standard four-point measurement. In case the current has an influence on the resistance, by heating the nanowire, for example, a change in the resistance would be expected. In a second measurement, identical measurements were performed



Figure 6.2: SEM image of the long run measurement configuration. A nanowire is contacted by a single STM tip. The picture was taken using the current flowing through the sample for contrast.

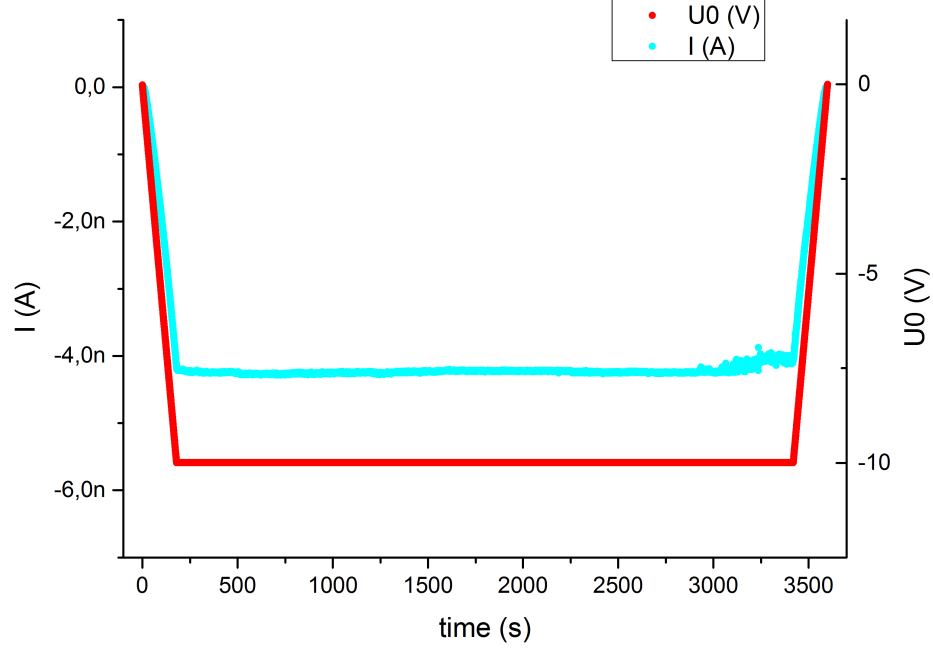


Figure 6.3: The voltage pattern applied to the nanowire (red) during a one hour long test run on a GaAs nanowire and the resulting current (cyan) are plotted over time. At the end of the measurement, the contact got unstable and the current starts to get noisy.

several times in a row to estimate the variation of the result.

6.1.1 Long time run

If a voltage V is applied to an object with a resistance R , a current I flows through the object according to $V = R \cdot I$. On its way through the object, however, the current can change the properties of the object.

In order to be able to measure changes in the entire nanowire, the test is carried out in a two-point configuration (Figure 6.2). A tip contacts the nanowire at the gold particle to provide a good and stable contact. A voltage pattern is applied to the sample while the tip is at ground potential. In Figure 6.3, the voltage pattern (red) is shown together with the resulting current flowing through the nanowire (cyan).

During four-point measurements, a voltage pattern has a duration of 100 or 200 seconds. In order to show the stability of the system, a one hour measurement was performed. At the beginning the voltage was ramped up to 10 V over a period of 3 minutes. Then, the voltage was kept constant for 54 minutes, before it was ramped down over 3 minutes again.

The current flowing through the nanowire shows the same pattern as the voltage

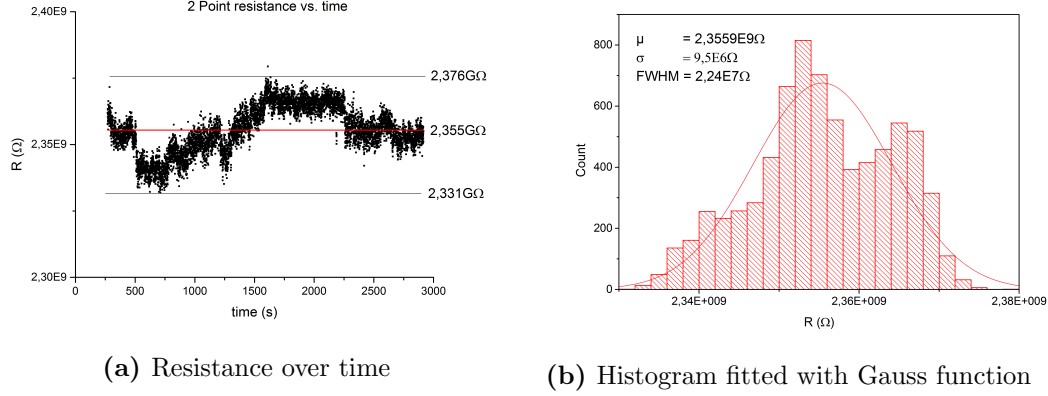


Figure 6.4: (a) The two-point resistance is shown as a function of time. Only the stable time interval between the two voltage ramps is shown (220s - 2700s). (b) shows a histogram of the distribution of the measured two-point resistances. The distribution is fitted using a Gauss function.

signal, as was to be expected. But after about 45 minutes, the noise level of the current signal increases and the current decreases a little. Most likely the quality of the contact between tip and nanowire decreased due to the tip drifting away from the nanowire. After reconnecting, however, the nanowire showed the same properties as in the first 45 minutes of the long run measurement, so the nanowire has not been permanently changed.

Knowing the applied voltage and the flowing current, the resistance of the nanowire can be calculated for each point. The evolution of the resistance is shown in [Figure 6.4a](#) for the time between the end of the starting ramp and the moment when the noise starts increasing. The two-point resistances during this period are between $2.331 G\Omega$ and $2.376 G\Omega$ with an average of $2.355 G\Omega$. With a very large period of ~ 2000 seconds, the resistance seems to fluctuate around its average. Such long periods are not typical for nano systems. The amplitude of the resistance change is about 2% of its average. Except for the oscillation, no trend over time is visible, and the following two-point resistance measurements showed similar results ($2.397 G\Omega$). Moreover, a typical time scale for a single measurement is 100 seconds, so the resistance change during a single measurement is even smaller.

In conclusion, one can say that the changes in the two-point resistance are small relative to its average, and do not disturb a typical resistance measurement. Mechanically, the system was stable for 45 minutes. This is more than enough time to perform several resistance measurements. Once contact is lost, it can easily be reestablished, and a new measurement can be performed.

6.1.2 Repetitive Measurements

In the last section the mechanical and electrical stability of two-point resistance measurements has been discussed. Four-point resistance measurements will be less

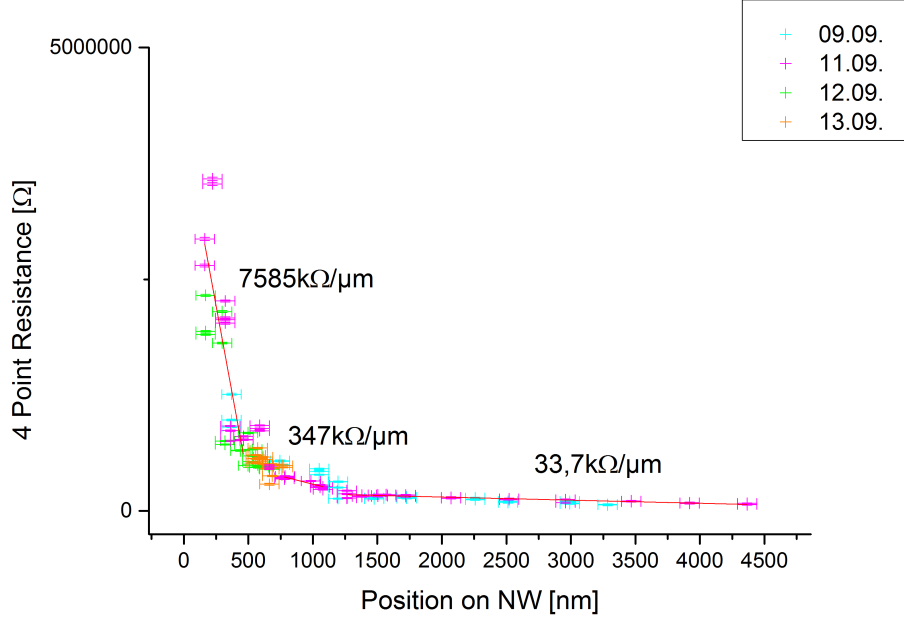


Figure 6.5: The shown resistance profile of a specific nanowire has been measured on four days. The results are all combined in this graph. All measurements follow the same profile. The measurement was stable over these four days.

stable mechanically, due to the higher number of drifting parts. However, the system is stable enough to perform several four-point resistance measurements before tip repositioning is needed. During a typical four-point measurement, only one tip is moved. But do four-point measurements show similar results when their tip configurations are similar?

In order to analyze the variance of four-point resistance measurements, such measurements were performed on the same nanowire over several days. This test was performed on the Step Sample.

In order to demonstrate the stability of the measurement, the resistance profile of a nanowire was measured on several days using four-point measurements as described in [section 5.4](#). At the end of each day, all tips were retracted from the nanowire to prevent damage to the nanowire by a drifting tip. By applying a constant voltage to the scan piezos of the STM tips, a spacing of ca. $\sim 5 \mu\text{m}$ was realized. The next day, the tips were placed at nearly the same spots, and four-point resistance measurements were carried out. In [Figure 6.5](#), each point is colored according to the date it was measured. The resistance profile measured on the 11.09. covers the entire measurement span. The resistances measured on the other days lie close to these results.

The results of all four days match each other well and show the same resistance profile of the nanowire. The measurement shows that the method gives reproducible results over several days. Moreover, it shows that no significant changes in the transport

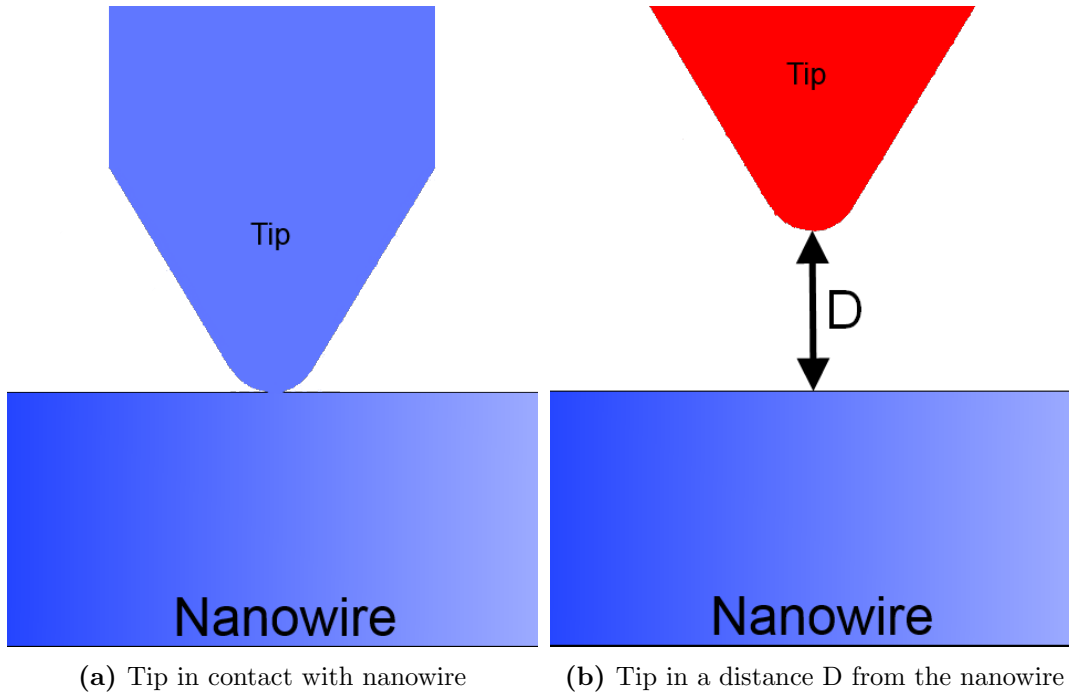


Figure 6.6: The local potential on a nanowire surface changes along the flowing current, as is depicted by color. The electric potential is the same on the entire tip, since it is made of a very conducting metal. In (a) the tip, in voltage measurement mode, is in contact with the nanowire. As a consequence, the tip has the same potential as the contact point. In (b) the tip is at a distance D from the nanowire and in current measurement mode. A Voltage is applied to the nanowire.

properties of the nanowire are generated by the measurement. Neither the electron bombardment of the SEM, nor the mechanical and electrical manipulation of the nanowire during a measurement altered the nanowire permanently.

6.2 Gate Measurement

When a nanowire is connected by an STM tip in voltage measurement mode, the potential of the tip is defined by the local potential at the contact point between nanowire and tip. The potential at the surface of the nanowire changes along the wire. A voltage difference is present between the other parts of the nanowire, and, the tip and as a consequence, an electric field is present.

This electric field may influence [8\[47\]](#) the nanowire next to the contact point. In order to be able to estimate the influence on a four-point resistance measurement, so called gate measurements were performed. The nanowire is contacted like for a four-point measurement. Next, one of the Voltage Probers (called Gate Tip from now on) is retracted a little and its black box switched to current measurement mode. Now a voltage can be applied between Gate Tip and nanowire. The three-point

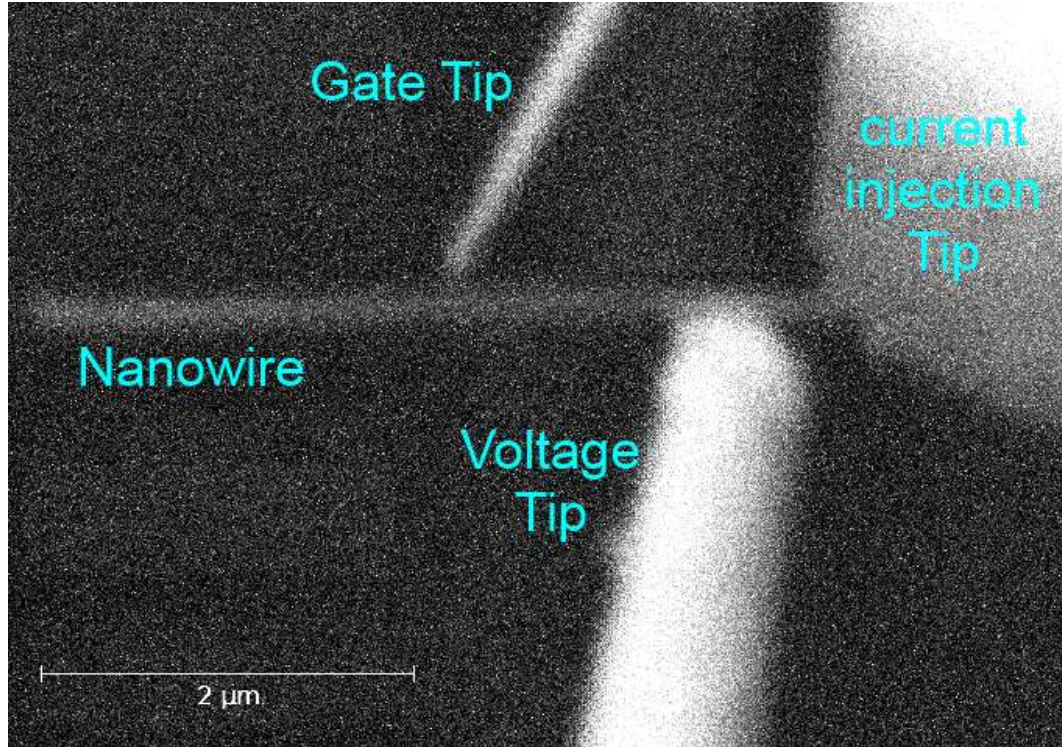


Figure 6.7: Shown is an SEM image of the tip configuration for gate measurements. Two tips contact the nanowire. One (shown on the right) to inject current into the nanowire and one (shown at the bottom) to measure the voltage difference between the sample and the contacted position on the nanowire.

resistance can be measured as a function of the distance between tip and nanowire and the voltage applied between the Gate Tip and nanowire.

The first measurements were performed on the Gradient Sample (M4580). An SEM image of the tip configuration is shown in [Figure 6.7](#). A constant voltage is applied to the Gate Tip. Subsequently, a voltage pattern is performed on the sample, while the Current Injection Tip was grounded. The current through the nanowire, as well as, the voltage at the residual Voltage Prober, was recorded. The three point resistance can be extracted from the measured data by fitting the voltage difference between the substrate and the voltage measurement tip as a function of the flowing current to the Current Injection Tip, like for a four-point measurement (see [Figure 5.5](#)). This is repeated for three different gate voltages +10 V, 0 V and -10 V. Subsequently, the distance between the Gate Tip and the nanowire is changed and another measurement is performed. The distance D in [Figure 6.6b](#) can be measured using the SEM with a precision of ~ 75 nm.

In [Figure 6.8](#), the result of this measurement is shown. The three-point resistance has been measured for three different gate voltages displayed as different colors (-10 V (magenta), 0 V (cyan), +10 V (green)) and for different gate distances from 0 nm to 1400 nm. The point at 0 nm corresponds to a measurement that has been performed

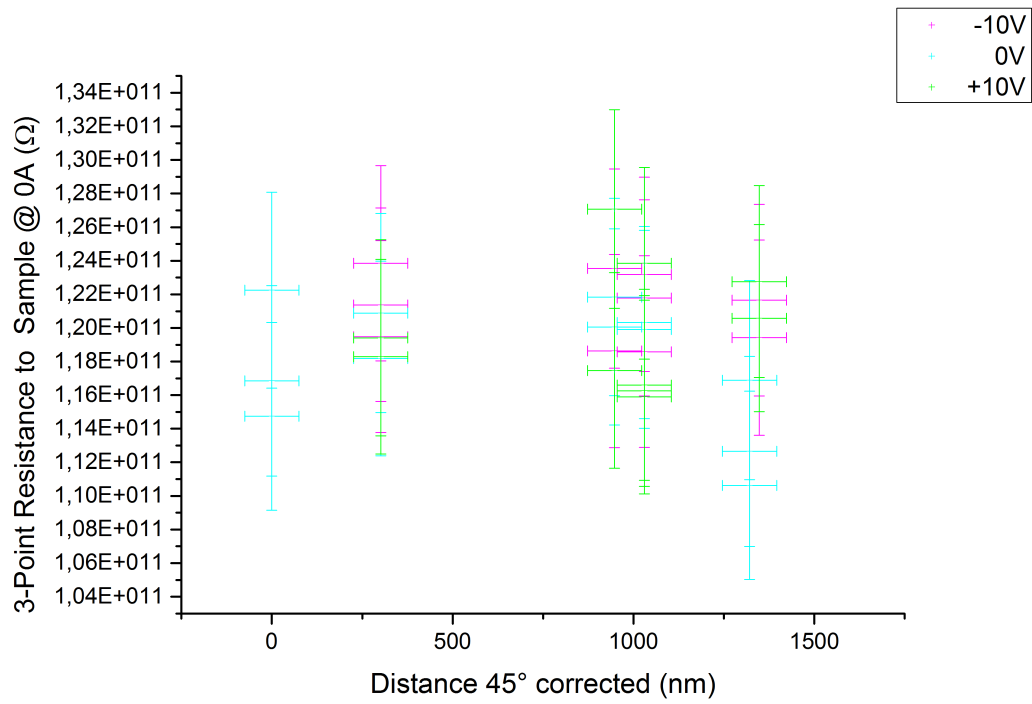


Figure 6.8: Results of the 3 Point gate measurement on a nanowire grown with a temperature gradient. For three different gate voltages (-10V (magenta), 0V (cyan), +10V(green)) the distance dependence of the three-point resistance is displayed.

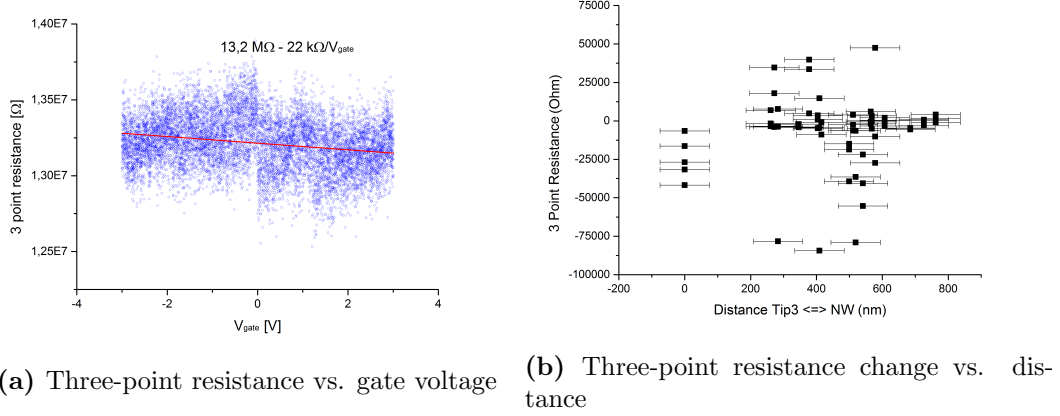


Figure 6.9: Results of the gate measurements on the Step Sample. In (a) the result of a single measurement is shown. The resistance is calculated for each point. Subsequently, the resistances are plotted as a function of the gate voltage fitted with a linear function. In (b) the results of these fits for different measurements are plotted as a function of the distance between Gate Tip and nanowire.

with the tip in contact to the nanowire. During this measurement the tip was in voltage measurement mode. The measured 3 Point resistances differ from 112 G Ω to 127 G Ω . Moreover, a field effect due to different potentials on the nanowire and the Gate Tip should have a strong (at least quadratic) dependence, but no distance dependence is present. The maximal influence of a gate voltage close to a nanowire can be estimated to be less than 5 % of the measured three-point resistance.

A similar measurement was performed on the Step Sample (M4509), with the same tip configuration as shown in Figure 6.7, but with constant voltage applied to the Current Injection Tip, and a voltage ramp of ± 3 V was applied to the Gate Tip. In this configuration, the three-point resistance of the nanowire cannot be calculated by fitting the applied voltage as function of the flowing current, as has been done in the measurement before. The resistance has to be calculated point by point, by dividing the applied voltage by the flowing current for each point. With this method, the accuracy of the resistance measurement is reduced, but the fast changing gate voltage can directly show changes in the flowing current.

In Figure 6.9a, a measured three-point resistance is shown as a function of the applied gate voltage. The resistance change with gate voltage is fitted with a linear function. The result of the fit is shown in the figure. The average three-point resistance for this particular measurement point is 13.2 M Ω . For each volt applied to the Gate Tip, the measured three-point resistance is reduced by 22 k Ω .

These results for different Gate Tip to nanowire distances combined are shown in Figure 6.9b. In contrast to the measurement before, the point at 0 nm is not measured with the Gate Tip in contact with the nanowire, but so close that no gap could be identified in the corresponding SEM image. Even though tip and nanowire were very close, they were not in contact, since no current was flowing between them.

The resistance gradients spread from $-85 \text{ k} \frac{\Omega}{V_{gate}}$ to $+47 \text{ k} \frac{\Omega}{V_{gate}}$ at an average three-point resistance of $9 \text{ M}\Omega$. Like for the previous sample, no distance dependence is present. On average, the resistance is reduced by $8 \text{ k}\Omega$ per volt applied to the Gate Tip. Since the resistance of this sample is much smaller than the one before, the precision of the gate measurement is much higher. For this sample an upper limit of the influence of a gate voltage near a nanowire on the resistance of the nanowire can be given as $8 \frac{\text{k}\Omega}{V_{gate}}$ @ $9 \text{ M}\Omega$ or $\frac{1}{V_{gate}}$.

In conclusion, no gate effect could be observed in either measurements. The results are distributed accordingly to the accuracy of the three-point measurements on the specific sample. More important, the resistances of the nanowires do not change when the distance between the nanowire and the tip changes, as should be expected for a field induced gate effect. For the performed multi tip resistance measurements, such an effect does not play a role.

Chapter 7

Nano Manipulation

In addition to the measurements presented in the previous chapters, the RT-MT-STM can also be used to manipulate nanostructures. One very simple example occurs regularly during four-point measurements. When a nanowire cannot be contacted by all tips due other nanowires blocking the path, the interfering nanowires can simply be disattached from the substrate using one of the STM tips.

7.1 Snap to contact

When a relatively large voltage (± 5 V) is applied between an STM tip and a nanowire, an attractive (electrostatic) force between the two is built up. If nanowire and tip get close to each other, the nanowire will bend towards the tip, when the attractive force is strong enough. During the operation of the RT-MT-STM, this “snapping to contact” occurred several times, especially for the nanowires with higher resistances.

7.2 Harvesting

Not only groups of nanowires, but also single ones can be disattached; not only to clear the way for four-point charge transport measurements, but also to perform measurements on single nanowires.

In the previous section ([section 7.1](#)), the effect of snapping to contact was introduced. It can be used to select single nanowires, contact it with one tip (the contact is strongest on the gold particle) and dislocate it with an other tip. This way, the nanowire will stick to the STM tip at its gold particle.

It is quite difficult to contact a nanowire with additional tips without disattaching it again. It was possible to attach an nanowire to an STM tip and contact it with another one. The electrical measurements, performed this way, unfortunately suffered from the bad nanowire tip contacts. While the contact at the gold particle

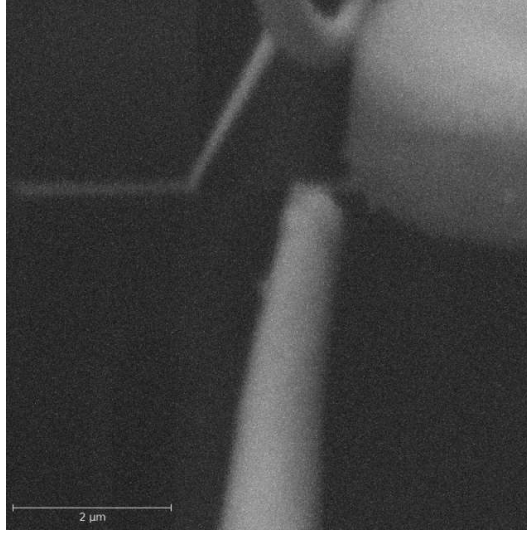


Figure 7.1: A nanowire is cut into two parts by sending a high current through a designated part of it. The middle part of the nanowire is burned due to the high current, while the upper and lower part remain unharmed.

allows good conductance, the other contacts are unstable, since no pressure can be applied.

This method offers several new applications for STM tips. For scanning tunneling microscopy, for example, a nanowire at the tip could be used to scan with a very small apex, or delicate structures could be contacted without the risk of damaging these structures, since the nanowire is softer than the tip. For different applications also larger areas can be cleaned from nanowires by scratching the tip over the surface of the sample. This is also handy if a large number of selected and free nanowires is needed in an experiment.

7.3 Precise cutting

The MT-RT-STM can be used to cut nanowires with very high precision. A high current ($>5\mu A$) can be pushed through a small part of the nanowire. Therefore, the nanowire has to be contacted by two STM tips from the side very close to each other. As a consequence, the small part between the STM tips will burn. The rest of the nanowire will not suffer any damage due to the high current. In [Figure 7.1](#), an image of such a procedure is shown. A nanowire is contacted like for a four-point measurement. Then, the two Voltage Probers are switched from voltage to current measuring mode, and a high ($\gtrsim 5V$) voltage is applied to the Scanner. A high current can flow through the nanowire part between the two Voltage Probers. If the current gets too high, this nanowire part is burned. The two remaining parts of the nanowire can be used for further measurements.

Chapter 8

Conclusion part II

In order to determine the influence of the growth temperature during the MOVPE growth of p-GaAs nanowires on their electronic structure, three samples with different temperature scheme were analyzed using the room-temperature multi-tip scanning tunneling microscope as a nanoprobe. It was possible to perform four-point resistance measurements on freestanding "as-grown" nanowires without detaching them from the substrate after growth.

The results of multiple four-point resistance measurements along single as-grown nanowires could be combined into resistance profiles for a nanowire. The spatial information about the tip positioning during each measurement was generated with a scanning electron microscope.

After the four-point resistance measurements were finished, the samples were transferred into a high resolution SEM. With some characteristic features present on the surface of each sample, it was possible to identify the exact same nanowires used for four-point measurements. so, the diameter of the nanowires could be measured with high precision.

A calculation of the conduction channel diameter along the different nanowires showed, that the bases of Gradient and Constant Sample are completely depleted. The only remaining channel for current flow is the surface. Literature values for the electric conductance of oxidized GaAs surfaces are in good agreement with the measured resistance gradients.

After the measurements were performed it turned out, that the supply with diethyl zinc to the reactor was delayed by several minutes when the nanowire growth started. Thus the bases of the nanowires are not doped at all. Unfortunately, this is the interesting area where the different temperature pattern were used during the nanowire growth. The influence of the reactor temperature on the core conductance of a nanowire could not be analyzed in this way.

The resistance profiles of the three samples could be divided into three regions with identical properties, even though not all nanowire showed all regions. While at the top the nanowires (region 1) the current can flow through the nanowire core with low resistance, the base (region 3) is not doped and thus the current has to take

the high resistive path via the surface. Interestingly the transition region (region 2) between region 1 and 3, has a higher resistance than region 3. Here the current has to cross the depletion zone from the surface into the core. If the applied bias is high enough to flatten the radial band structure, the current can cross at the border between region 2 and 3. If the bias is lower, the current has to stay on the surface until the boarder between regions 1 and 2. The four-point IVs measured in the transition region showed a non linear characteristic.

The results of the four-point resistance measurements were backed up by Electron Beam Induced Current images. When contacted with an STM tip in current measurement mode, all nanowires show extra features in SEM images taken with sample current. These features are localized exactly at the axial position along the nanowires at which the four-point resistance measurements showed the highest resistance gradients, i.e. in the transition region.

Long time and repetitive measurements have shown that the influences during four-point resistance measurements, like bending or changes due to current flow through the nanowire, do not change the conductive properties of the nanowire. Additionally the influence of the presents of an STM tip on the charge transport through the nanowire have been studied. Biased and unbiased tips have been brought close to an charge transporting nanowire. The three-point resistance did not change. The method used provides stable and reproducible results. Finally, additional applications for the RT-MT-STM, like cutting nanowires or utilizing STM tips, were presented, that could also be used for further measurements.

Part III

The Low-Temperature Multi-Tip Scanning Tunneling Microscope (LT-MT-STM)

Temperature plays a major role in most reactions and processes. As a (chaotic) form of energy, temperature contributes to most processes. In semiconductors, for example, the population of states around the Fermi level with charge carriers depends on their thermal energy. A change of this population has a major impact on the charge transport through the semiconductor.

In order to be able to perform measurements on low-energy states or systems or to find out how temperature influences a system or a process, it is necessary to be able to adjust the temperature of an experiment. Heating up a sample is fairly easy. With electrical heating a wide temperature range (room temperature to several thousands Kelvin) can be covered. Cooling down an experiment, on the other hand, is more complicated.

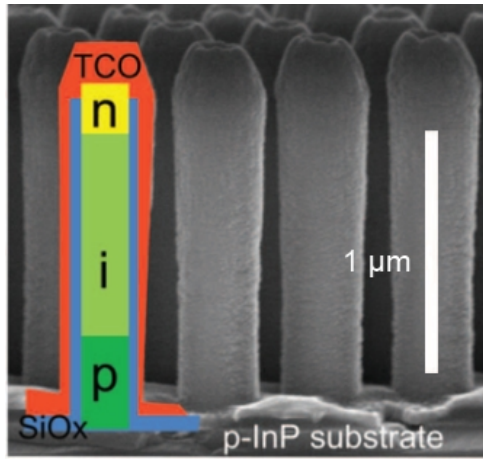
Based on the room-temperature multi-tip scanning tunneling microscope, a new experiment has been designed and built in the research center Jülich. As for the RT-MT-STM, four individual scanning tunneling microscopes are combined in a single device, while an SEM provides high resolution on the STM tips and the sample. Two additional reservoirs (cryostats) for liquid gases are installed next to the STMs.

Liquid gases are widely used for cooling processes, especially liquid Nitrogen N_{lq}^2 (77 K) and liquid helium He_{lq} (4.2 K). Those two liquid gases are also used in this experiment. To provide a homogeneous temperature and to reduce the needed amount of cooling gases, the thermal shielding of such an experiment is crucial and requires great effort.

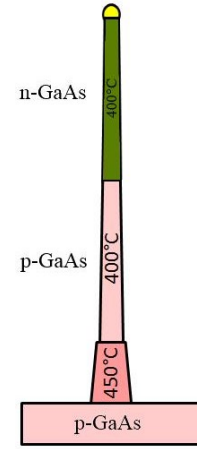
The room-temperature multi-tip scanning tunneling microscope (RT-MT-STM), as described in [Part II](#), was used as a nanoprobe to measure the resistance profile along as-grown p-GaAs nanowires. From these resistance profiles, the charge carrier density and mobility as well as the resistivity of the nanowire could be derived.

In order to be able to utilize such nanowires in integrated circuits ([\[48\]](#)) or as advanced solar cells ([\[49\]](#)), complicated electronic structures, as shown in [Figure 9.1a](#), must be realized. To analyze such structures, especially the interfaces between differently doped areas, measurements at different temperatures are necessary.

The interfaces between the differently doped sections are of most importance for the efficiency of such a device. The pn-junction of the samples used in the RT-MT-STM is located between the nanowires and the substrate. Four-point resistance measurements are complicated to perform on the substrate next to a nanowire. To study the pn-junction in more detail, a new sample with the junction in the middle of each nanowire was prepared ([Figure 9.1b](#)).



(a) Design for a solar cell utilizing InP nanowires.



(b) Structure of the used nanowires

Figure 9.1: In Figure 9.1a, an SEM image from a publication of Wallentin et al. is shown [49]. InP nanowires with an p-i-n doping structure were grown on a p-InP substrate. Afterwards, the sample was coated with layers of SiOx and TCO. Such a structure is designed for next generation solar cells.

In Figure 9.1b, the structure of the nanowires analyzed in this part is shown. On a p-GaAs substrate, $\sim 16 \mu\text{m}$ long GaAs nanowire were grown. Along the axis of these nanowires, a pn junction was realized. While the lower part is doped with zinc (p-doped) like the substrate, the upper part is doped with tin (n-doped). The color of each part of the nanowire depicts the temperature during its growth as well as their doping. At the very top, the gold particle used for the growth process is depicted.

Chapter 9

Experimental Setup

In [chapter 3](#), the experimental methods were introduced in a theoretical way. In this chapter, the focus will be on technical aspects and the concrete configuration. The low-temperature scanning tunneling microscope is based on the room-temperature version described in the previous part of this thesis. Two additional components are present: a cryostat to cool down the measurement area, and an 8 T superconducting magnet. With these two components, a variety of new possible applications arise.

The low-temperature multi-tip STM (LT-MT-STM) has been designed to perform charge transport measurements on nanostructures at liquid helium temperature (4.2 K). Four scanning tunneling microscopes (STMs), a scanning electron microscope (SEM) and a superconducting magnet are combined within a small volume. An additional two stage bath cryostat cools the experiment down to liquid helium temperature (4.2 K)

In [Figure 9.2](#) a technical drawing of the experiment is shown. The UHV-chamber can be divided into two sub-chambers. In the first one (preparation chamber), samples can be produced and their quality checked. The second one (measurement chamber) hosts the LT-MT-STM and the parts necessary for charge and magneto transport measurements at low-temperature. Beside the LT-MT-STM itself, the measurement chamber is equipped with an SEM (see [section 9.2](#)) to gain high resolution onto STM and the sample, and a liquid helium cryostat (see [section 9.3](#)) to cool down the STM and the sample to ~ 6 K. Inside the cryostat, the superconducting magnet is installed.

In the center of [Figure 9.2](#), the preparation chamber can be seen. It can be equipped with up to 7 evaporators to deposit different materials onto a specimen. The deposition rate can be measured and calibrated using a quartz balance. Additionally, a loadlock is mounted to the preparation chamber to transfer new samples and STM tips into the system, as well as to get old ones out. The transfer inside the UHV chamber is performed using two manipulators. As described in [section 3.3](#) the temperature of a sample can be crucial during preparation. This manipulator provides thermal and electrical contact to the sample holder in order to either heat it up or cool it down. The sample can be cooled down to liquid Nitrogen temperature (77 K)

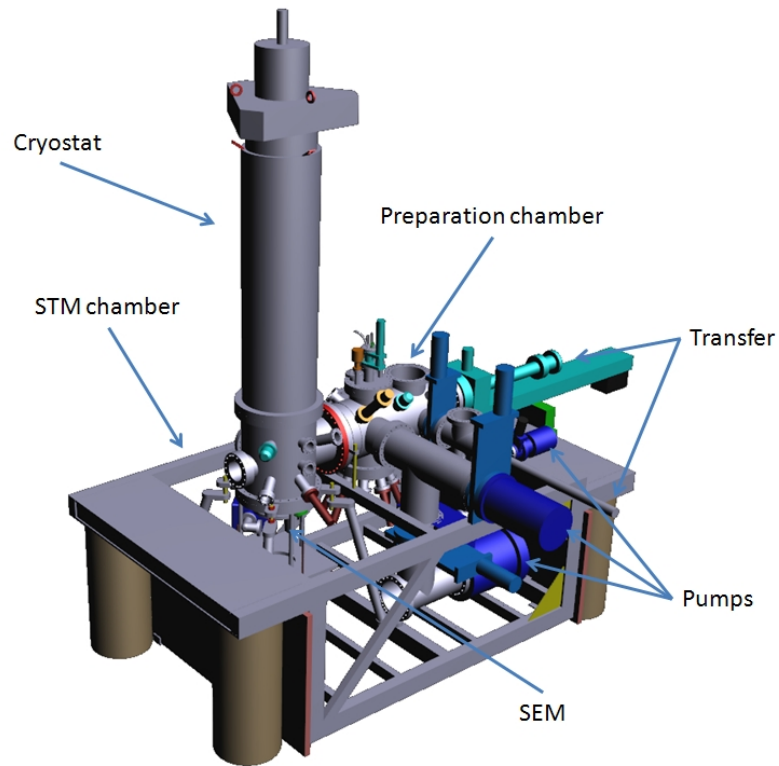


Figure 9.2: A technical drawing of the UHV chamber is presented. The UHV system consists of two parts. Sample preparation, structural and chemical analysis of the sample, as well as sample transfer, can be performed in situ in the preparation chamber. The measurement chamber hosts a liquid helium cryostat and, additionally, an SEM is mounted to the LT-MT-STM chamber.

or heated up while it is attached to the manipulator in the preparation chamber. To achieve and maintain a UHV, different types of pumps are used. Starting from ambient condition a scroll pump reduces the pressure inside the chamber to a range where the turbo pump can start ($\sim 5 \cdot 10^{-3}$ mbar). This turbo pump alone can evacuate the system to $\sim 5 \cdot 10^{-8}$ mbar. Finally, an ion pump evacuates the system to $\lesssim 1 \cdot 10^{-9}$ mbar. A titanium sublimation pump is installed to refresh the ion pump. During measurements, only the ion pumps are running, because the other pumps generate too many vibrations.

9.1 Scanning Tunneling Microscope(s)

The heart of the experiment is the four-tip scanning tunneling microscope. Two different four-tip STMs have been installed in this experiment. The first multi-tip STM was designed to be capable of magneto-transport measurements ([subsection 9.1.1](#)). The second one was designed as compact as possible ([subsection 9.1.2](#)), to reduce the impact of vibrations to the four-tip STM. The ultra compact STM is capable of performing measurements in ambient conditions as well as at low temperatures in a UHV.

9.1.1 Magneto-Low-Temperature Multi-Tip Scanning Tunneling Microscope (magneto-LT-MT-STM)

Four individual scanning tunneling microscopes (STM) are combined in a single device. Each of these STMs is based on the Besocke beetle-type STM [[37](#)]. As for the magneto-LT-MT-STM, for each STM, three piezo tubes are glued to a common baseplate, carrying a ring for each STM. To this ring a KoalaDrive ([subsection 9.1.3](#)) with attached tip is fixed as shown in [Figure 9.3](#).

The ring form was sacrificed to interlace them in order to bring the four tips together as close as possible, while maintaining a large scanning area. Small springs are used to attach the rings to the according piezo tubes, and, since magnets must not be used in high magnetic fields, a ruby half hemisphere is glued between the rings and each piezo to reduce friction. For coarse motion as well as for fine scanning, the three outer piezos holding each ring are used. Via slip-stick motion (see [[7](#)]), the ring can move several millimeters in the XY-plane with comparably low precision. While scanning, on the other hand, several μm^2 can be covered with subatomic precision. The four rings are placed on top of each other to gain more mobility, and to prevent contact between two or more rings. The KoalaDrives, on the other hand, are installed all at the same height to easily cover the same height regime with all four tips.

In the traditional beetle-type design, a so called rampring is used to approach the tip to the sample. The ring is build with three identical ramps along the ring, one for each piezo. If the ring is rotated, each piezo glides along the ramps, resulting in a z-motion. Such a coupling of XY- and Z-motion is a big disadvantage when

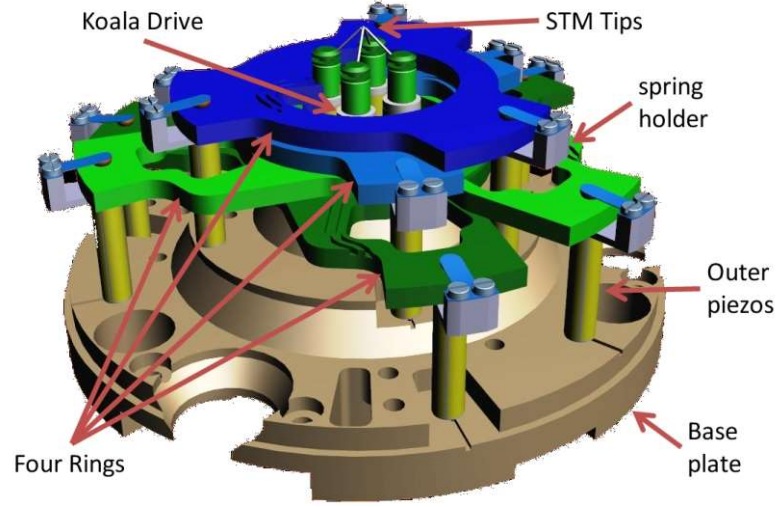


Figure 9.3: As shown in this technical drawing of the magneto-LT-MT-STM, the 4 STM tips are mounted on top of a KoalaDrive each. The KoalaDrives themselves are installed on 4 rings. While the tips can be moved in z-direction with the KoalaDrives, the rings are used for XY-motion, for scanning as well as for coarse motion via slip stick.

several of these devices are combined into one system. The magneto-LT-MT-STM used z-drives based on slip and stick motion to decouple XY and Z motion from each other. For the magneto-LT-MT-STM, this z-drive has been further developed into the KoalaDrive. The KoalaDrive will be introduced in detail in [subsection 9.1.3](#).

In a further stage, magneto transport measurements can be performed at low temperature with this setup. An 8 T superconducting magnet is installed inside the helium cryostat. Such a high magnetic field requires material without any magnetic moment. Even alloys with small magnetic moments, like stainless steel for example, must not be used.

Furthermore, the experiments are carried out at low temperatures. So the four-tip STM not only has to withstand liquid helium temperature (4.2 K), but also conduct it, to bring all the STM to the same temperature. Additionally, it had to be taken care of the fact that the different materials have different expansion coefficients. Over the entire temperature range from 300 K down to 4.2 K, the unequal expansion has to be taken into account.

To meet all the resulting requirements, the used materials had to be chosen with great care. Piezo elements are capable of working in high magnetic fields as well as at low temperatures. The performance of the piezos changes with the environmental conditions. The piezo constant changes due to the temperature for example ([50]), the overall behavior of the material is left unaltered. An applied voltage will result in a expansion or contraction of the piezo, but the size of the expansion (contraction) depends on the temperature.

The base plate, as well as the rings carrying the KoalaDrive, are made of a gold

covered copper-beryllium alloy. It has a negligible magnetic moment [51] and a low thermal expansion coefficient [52], so it is perfectly suitable for the task. The additional gold coating improves the thermal conductance and ensures homogeneous cooling.

9.1.1.1 Performance of the low-temperature magneto multi-tip STM

A graphite sample was used to test the performance of the magneto-LT-MT-STM. These first tests were performed in ambient conditions without external or internal vibration isolation. In Figure 9.4a, an STM scan can be seen. An atomic step is present. Such a step could be used to gauge the piezo constants of the scanning piezos.

According to [53], the graphite sample was cleaned with the scotch-tape method. First, scotch-tape (or any other adhesive tape) is pressed on top of a block of graphite. When the tape is removed, several layers of graphite (or graphene) stick to the tape. With the necessary care, a flat and clean graphite surface remains.

The cleaned graphite sample is stable in ambient conditions and conductive. In Figure 9.4a, an STM scan of the graphite sample is shown. An atomic step crosses the surface. A line scan across the step is plotted in Figure 9.4b. The Z-piezo constant could be calibrated with such scans.

9.1.2 Ultra compact Low-Temperature Multi-Tip Scanning Tunneling Microscope (LT-MT-STM)

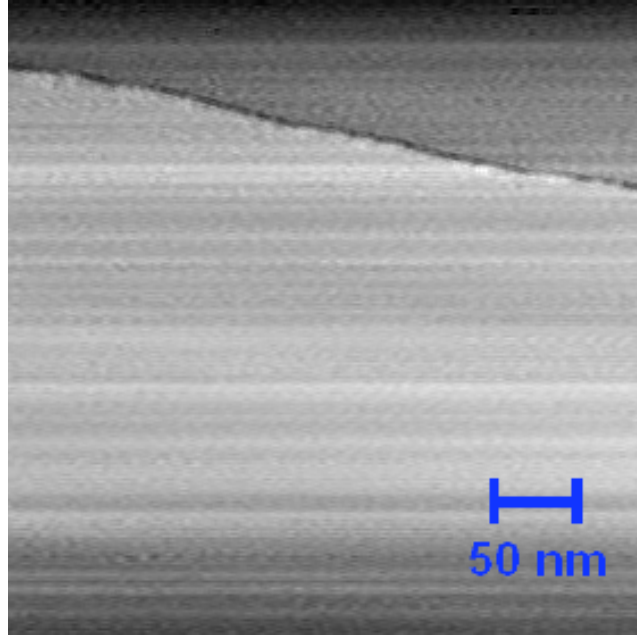
A new advanced design made it possible to build an even smaller multi-tip STM. Four individual scanning tunneling microscopes (STM) were combined in a single device of only **50 mm** in diameter, as can be seen in Figure 9.5a.

An overview over the four-tip scanning tunneling microscope has been published in Review of Scientific Instruments ([54]), as well as in [18]. The four rings are replaced by four plates, one for each STM. These plates are placed next to each other instead of above each other.

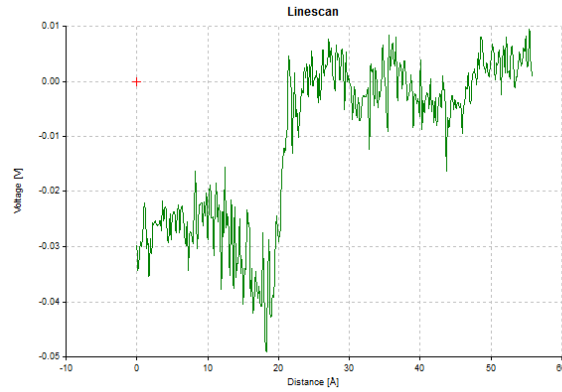
In Figure 9.5b, the LT-MT-STM is shown without the top cover. The four STMs are mounted on a common baseplate and resemble four leaf clover. Three segmented tube piezos carry a black plate for each STM. A sketch of the LT-MT-STM is shown in Figure 9.6. The plates are attached to the piezo tubes by a magnet each. By slip stick motion, the plate can be moved over the base plate in X and Y direction. To scan a surface, the piezo tubes bend slowly in the same direction.

This design uses the KoalaDrive instead of traditional beetle-type ramp rings to decouple XY and Z motion from each other, too. The sample is mounted above the four STM units, while the SEM takes images from below through the opening between the different STM units. Two of these units are shown in the sketch.

The ultra compact LT-MT-STM was designed to work in various conditions. Dr. Marcus Blab used it in ambient conditions for several measurements [18], before it was installed into the low-temperature UHV setup.



(a) A scan of a graphite surface



(b) A line scan across the step in (a)

Figure 9.4: In (a), a scan of tape cleaned graphite sample performed with the Magneto-LT-MT-STM is shown. An atomic step is running through the picture. It was used to calibrate the z piezo constant of the scanning piezos. In (b), a line scan over the step edge is shown.

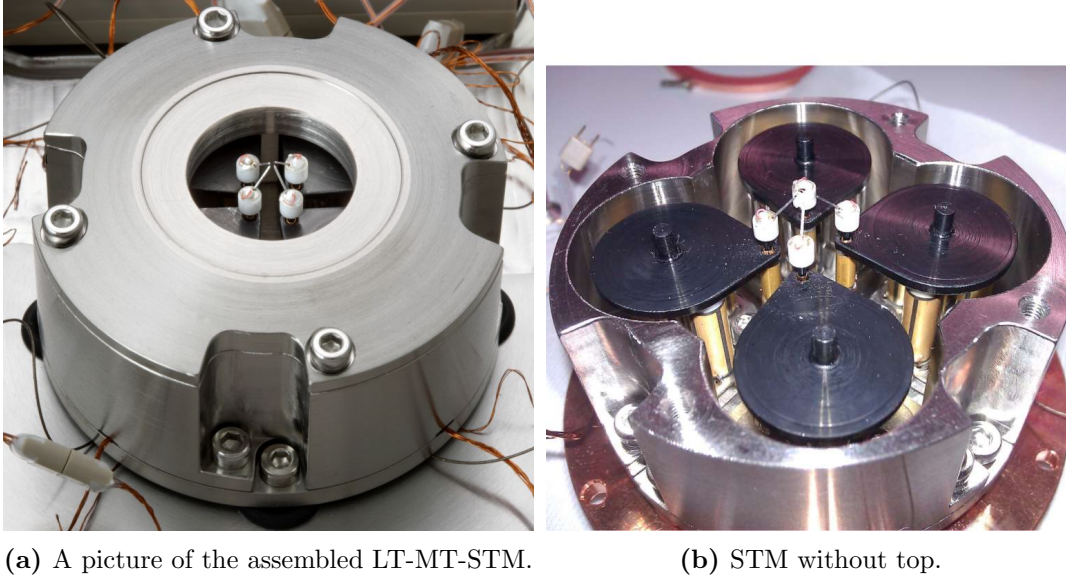


Figure 9.5: The second generation four-tip STM. In (a), the LT-MT-STM is completely assembled. To get a better view of the set up of the LT-MT-STM, the top plate is removed in (b). The four STMs are assembled in a clover-like order. Three piezo tubes carry a black plate. To this plate, a KoalaDrive with an STM tip is attached. The three tube piezos move the tip in X and Y direction, while the KoalaDrive approaches the tip towards the sample or retracts it.

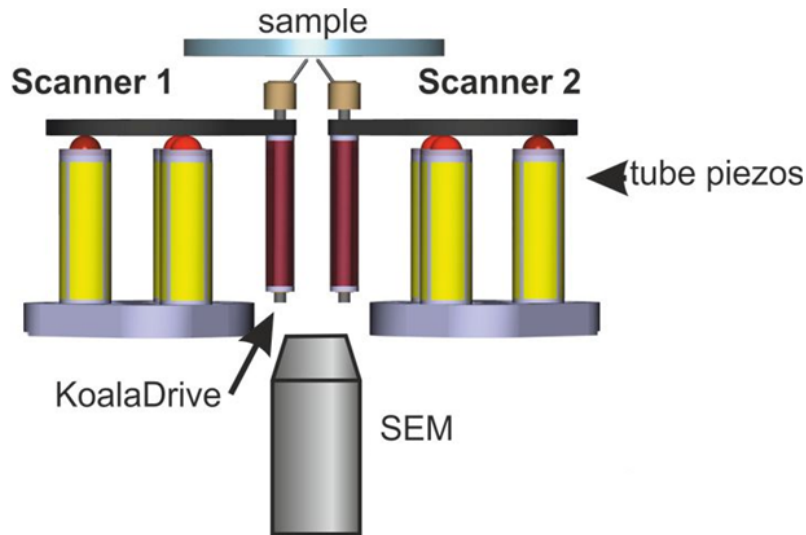


Figure 9.6: A sketch of the assembled LT-MT-STM. Two STM units are shown. Three segmented tube piezos (yellow) carry the base plate (black) to which the KoalaDrive (dark red) is attached. The SEM (gray) images the sample (blue) and the STM tips from below [55].

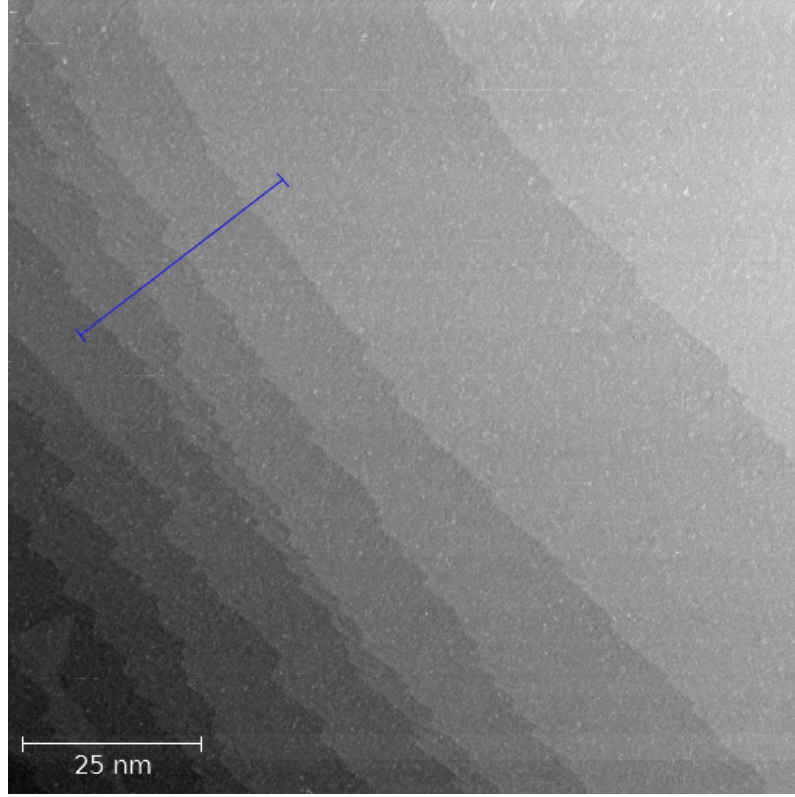


Figure 9.7: STM images of a Pt(100) crystal scanned at liquid nitrogen temperature. Several step edges can be seen. It was used to calibrate the z-piezo constant of the for piezo scanner.

For use in ambient conditions, the STM is embedded in a stainless steel shield. The sample can be attached to the STM in several ways. Originally, the sample was held by three magnets. With shear piezos between the sample and the shield, the sample could be moved. In a later step, the sample was attached to the steel shield by four screws to provide more mechanical stability and to reduce the magnetic disturbance for the SEM beam. In both configurations, no tip or sample exchange is possible in the low-temperature setup. In future upgrades, a tip and sample exchange mechanism is planned. Furthermore, the choice of stainless steel prohibits the performance of magneto transport measurements.

9.1.2.1 Characteristics of the LT-MT-STM

Due to the lack of a tip or sample exchange, only a limited selection of materials and reconstructions can be investigated with the LT-MT-STM at the moment. To demonstrate the performance of the ultra compact LT-MT-STM, and to calibrate the piezo constants, a Pt(100) bead crystal ([27]) prepared in the Research Center Jülich was used (subsection 3.3.2).

Pt(100) can be cleaned easily by heating, while it oxidizes were slowly ([56],[57]).

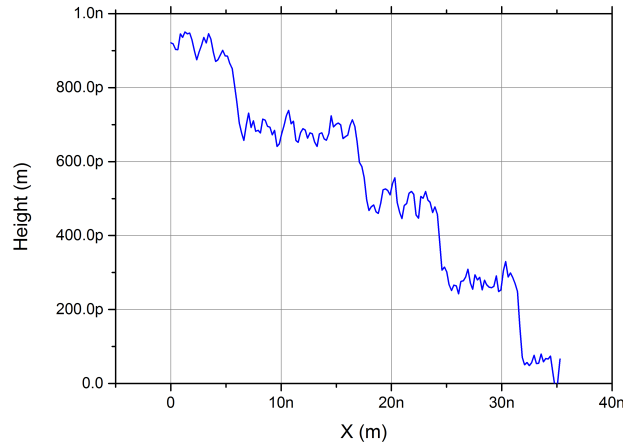


Figure 9.8: In Figure 9.7, a line scan (blue line) over several steps was performed. The height profile along this line is shown in this graph. Five atomic steps can be seen, each with a height of $1.96 \text{ \AA}$.

After cleaning enough time remains to install it in the chamber, close the chamber and create a sufficient vacuum -about 1h in total- before the Pt(100) surface is degraded.

In Figure 9.7, an STM scan of the Pt(100) is shown. Several atomic steps are present in the scanned area. The height of these steps is known from the literature to be $1.96 \text{ \AA}$ ([58]) and was used to calibrate the z-piezo constant. Additionally, Pt(100) is known to have a reconstructed surface, if prepared correctly.

The Pt(100) surface reconstruction has been described in several publications ([59]). To reduce the free binding energy the surfaces typically forms a (5×25) reconstruction ([60]). The first layer forms a hexagonal surface over the square bulk lattice ([56]). In a high resolution STM, rows with 6 atoms each can be resolved crossing the surface in $[01\bar{1}]$ -direction. These rows have a pitch of 1.4 nm .

In the LT-MT-STM scans, atomic resolution could not be achieved. For such resolutions, tips and samples have to be prepared in situ, to be free of contaminations. Only a few fragments of the Pt(100) reconstruction could be found in the scans. The timespan between cleaning the bead crystal in nitrogen atmosphere and evacuating the chamber seemed to be on the edge.

9.1.2.2 Drift measurement

Once the piezo constants are known, also the drift of the entire system can be measured. Over several hours, a designated area on the sample is scanned over and over again. Due to temperature changes at different points of the device, the position between the sample and the tips changes over time. This so called drift is an important characteristic of an STM system.

In this time span, the "movement" of a characteristic feature was observed. The mea-

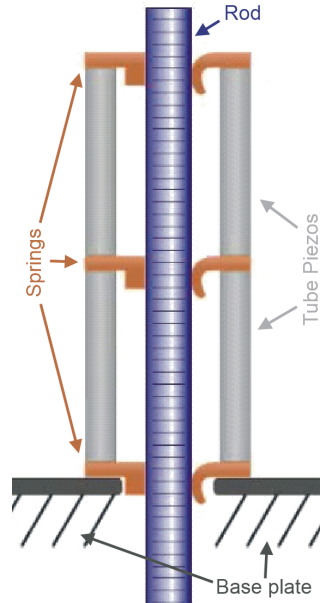


Figure 9.9: The KoalaDrive is built of two tube piezos glued on top of each other. At each end of the drive, and between the two piezos, springs to hold a rod in the center. By contracting and expanding, a rod or tube can be moved with high precision.[55]

surement was repeated for three different temperatures. At liquid nitrogen (77 K) temperature a drift of 4 nm per hour in the XY-plane, and of 2.5 nm in Z-direction was measured. When the temperature is further reduced to liquid helium temperature (4.3 K), no drift at all could be measured.

The same LT-MT-STM was used in a different setup for room-temperature measurements. At room-temperature, the drift of this LT-MT-STM was measured by Dr. Marcus Blab. A drift of 1.25 nm per hour in Z-direction, and less than 20 nm per hour in the XY-plane was found. The complete characterization of the LT-MT-STM can be found in [18] and [61].

9.1.3 The KoalaDrive

The KoalaDrive ([62]) is an ultra compact nanopositioner consisting of two tube piezos. The drive can move a tube or rod along its symmetry axis with a variable step size down to the nanometer regime. The step size of a drive depends on the temperature, the amplitude of the voltage signal and the size of the piezo tubes. A drive like the one shown in Figure 9.9 was tested down to liquid helium temperature (4.2 K). It also works in magnetic fields.

The KoalaDrive is based on the simple principle: “*Two are stronger than one*”. Two piezo tubes are glued on top of each other and on a base plate, as can be seen in Figure 9.9. At the base plate, as well as between the two tubes and at the end of the drive, springs are attached. These three copper-beryllium springs are aligned in

the center of the piezo tubes, so the rod or tube will fit in all three at the same time. During operation, one spring is moved relative to the two other springs. All three springs apply the same force to the rod, so the rod will follow the two springs instead of the single one. The operation of the KoalaDrive follows the following steps:

1. First, the upper piezo contracts. The top most spring moves downwards (in [Figure 9.9](#)). The rod will not move relative to the baseplate, since the spring at the baseplate, as well as the spring between the two piezos, do not move and hold the rod in position. All springs in the drive are identical. As a result, the rod will always follow the movement of two springs.
2. In the next step, the lower piezo will contract the same length as the upper piezo did during the first step. At the same time, the upper piezo will extend back to its initial size. The upper spring will not move since the two piezos movements will compensate each other. Also, the spring at the baseplate does not move. Again, the rod will not move due to two springs holding it, while only one moves.
3. Now the lower piezo expands to its initial size. Both upper springs will move, while the lower piezo expands. The rod will follow the movement of the two springs and move one step up (in [Figure 9.9](#)).

When the expansion is complete, the drive is in the same state as it was before the movement started. The rod was moved one step (upwards in [Figure 9.9](#)). An animation of the KoalaDrive during operation, as well as more details, can be found on the [web page of mprobes](#).

The KoalaDrive was tested in various conditions. For a signal amplitude (0-400 V) 1000 to 20000 steps with the KoalaDrive were performed. The distance the rod had traveled was measured with an optical microscope. The results for different temperatures in vacuum can be found in [Figure 9.10](#). The step size is shown as function of the signal amplitude of three different temperatures. The KoalaDrive starts operating at a certain temperature dependent voltage threshold. For voltages higher than this threshold, the step size grows linearly with the signal height. It is possible to use the drive from 60 V up to 400 V.

The piezo constant decreases with decreasing temperature [63]. As a consequence, the step size of the KoalaDrive is smaller at lower temperatures. At liquid helium temperature, more than 300 V are needed to move the rod in the drive. By choosing the amplitude of the voltage signal controlling the KoalaDrive, the step size can be adjusted to the designated size. The smallest measured step size was 50 nm at 4.2 K, the largest nearly 3 μm . The upper step size limit arises due to the size of the piezo tubes, their piezo constant and the maximal voltage that can be applied to them. Some part of the movement of each piezo tube is consumed by the springs. The springs can expand or contract along the movement of the rod. This reduces the maximal step size a little, but is mainly responsible for the lower limit of the step size. For movements smaller than 50 nm (at low temperatures), the piezo tubes contract or expand, but the friction between spring and tube holds all three springs

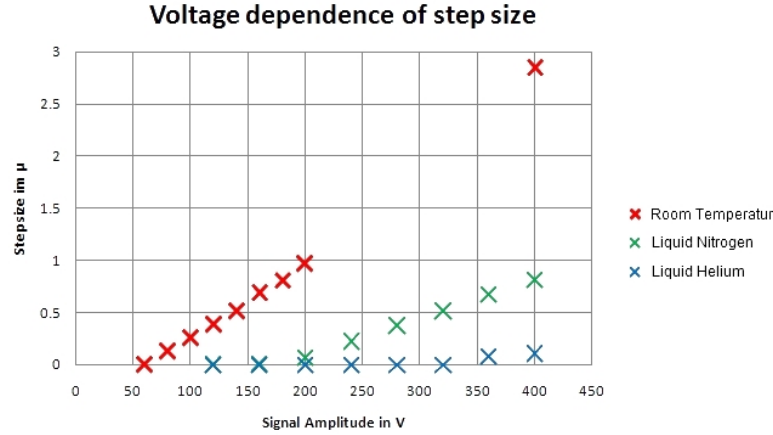


Figure 9.10: For three different temperatures (room-temperature 300 K, liquid Nitrogen temperature 77 K and liquid helium temperature 4.2 K), the KoalaDrive step size vs. signal amplitude was measured. In each temperature regime the step size is proportional to the signal amplitude. The step size can be adjusted between 50 nm and 2.8 μm .

in their position, and no movement is exhibited. The spring constant of these springs depends on their temperature.

For applications without a magnetic field, stainless steel tubes are used in KoalaDrives. Due to the high magnetic field possible in this machine, the tube has to be nonmagnetic. So titanium was chosen. In ambient conditions, the drive worked perfectly, but in vacuum, and especially at low temperatures, the tube tends to get stuck during operation. The surface of a rod used in a KoalaDrive is never perfectly flat. Scratches and bumps are always present on its surface. Especially titanium has quite rough surfaces. When a side of a spring is caught in a scratch, more force is needed to move it out. At some point, the “*Two are stronger than one*”-principle does not apply anymore and the drive stops to work. The stiffness of the springs at low temperatures additionally enhances this problem.

The first attempt to overcome this problem was to vary the diameter of the rod in order to reduce the overall force. In a next step, the titanium rod was polished. Mechanical, electrochemically and chemical etching were tested subsequently to smoothen the surface of the titanium tube, none of which lead to a sufficient result. Finally, the rod was coated, resulting in a satisfactory performance.

9.2 Scanning Electron Microscope

For multi-tip STM measurements it is essential to know the positions of all tips. For this purpose the LT-MT-STM is equipped with an SEM. The same SEM column (*FEI BDS 200 2LE*) as for the RT-MT-STM is used, while the SEM electronics are of a newer generation.

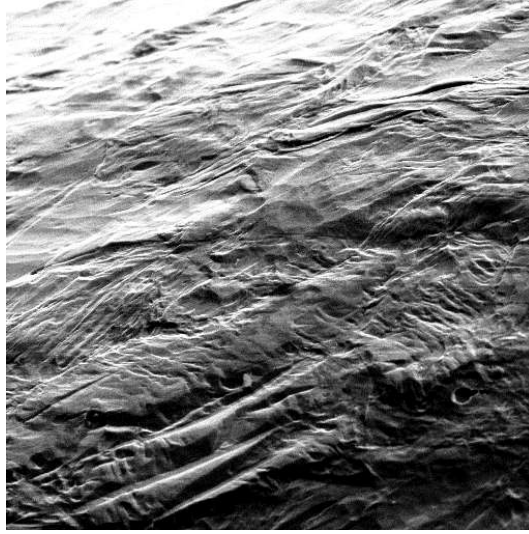


Figure 9.11: The surface of a titan tube is shown with a $1000\times$ magnification.

The SEM is installed beneath the cryostat, as described in [section 9.3](#). In order to enhance the visible area, and to compensate for a possible misalignment of the holes in the cryostat cups, the SEM is mounted on a custom made flange. This flange allows it to tilt the SEM a few degrees. So, it is possible to take an image not only the center of the sample with maximum resolution. An SEM picture can be taken within several seconds. At maximum magnification, several minutes are needed to get a good picture.

The contrast in an SEM has been discussed in [subsection 4.2.1](#) and [subsection 3.2.3](#). In principle, the same contrast methods are applied for the LT-MT-STM. Either the current flowing in or out of the sample as a response to electron bombardment by the SEM can be measured with a low noise current to voltage amplifier (Femto DLPCA-200)(Current Picture), or the secondary electrons (or ions) emitted by the sample due to electron bombardment can be collected and multiplied (SED).

In [Figure 9.12](#), the sample used to test the performance of the SEM is shown. The sample consists of a silicon substrate with gold structures of different sizes deposited on it. The largest feature on the sample is a $3000\text{ }\mu\text{m}$ long and $100\text{ }\mu\text{m}$ wide line. It is visible with the bare eye. The smallest is a square of $0.5\text{ }\mu\text{m} \times 0.5\text{ }\mu\text{m}$. For measurements of the bare SEM performance, the cryostat was removed from the chamber, and the sample was installed rigidly, directly above the SEM column.

In [Figure 9.13](#), two SEM images of the same sample are shown. The two images show different details of the sample. The left picture shows an overview over the entire sample, while the right one shows only the smallest structures in the middle. The two images were taken with different contrast methods and show inverted coloring. For the RT-MT-STM, a standard electron multiplier with a continuous dynode was used to collect the secondary electrons and amplify this signal. The electrons are sucked into the detector with a high electric field. At low temperatures, the dynode becomes superconductive and the field across the dynode collapses. To be able to

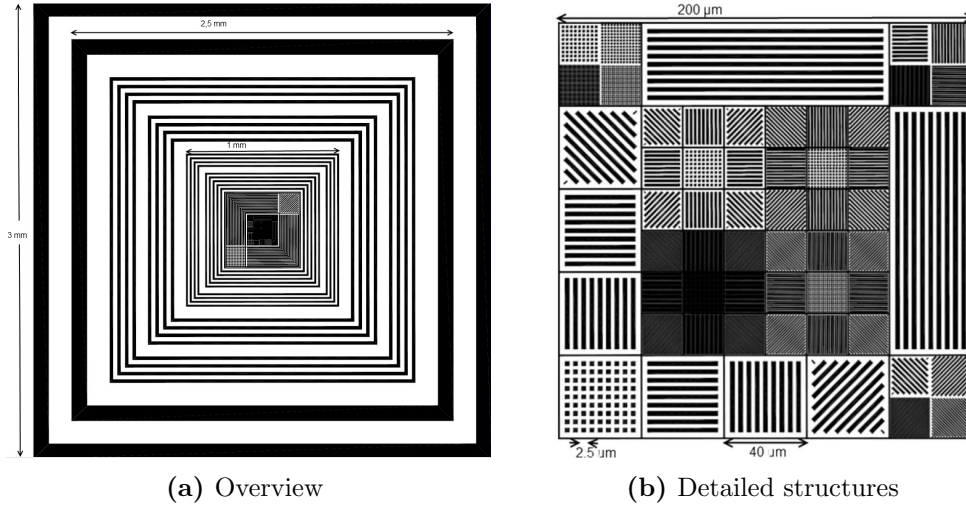


Figure 9.12: Structured sample used during the SEM test. On the left side, an overview is shown. The structured part of the sample is 3mm by 3 mm. The gold lines and squares on the sample have sizes from 100 μm down to 0.5 μm . On the right side, the most fine structures are shown.

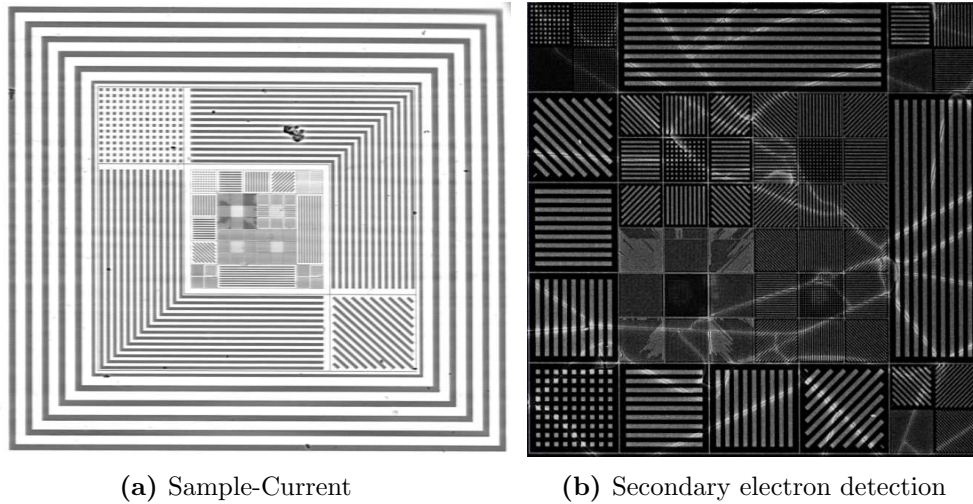


Figure 9.13: Image of the test sample shown in [Figure 9.12](#), taken with the SEM. On the right side, a large scan shows most of the structured part of the sample, while on the left side the smallest structures (pitch 0.5 μm) are resolved.

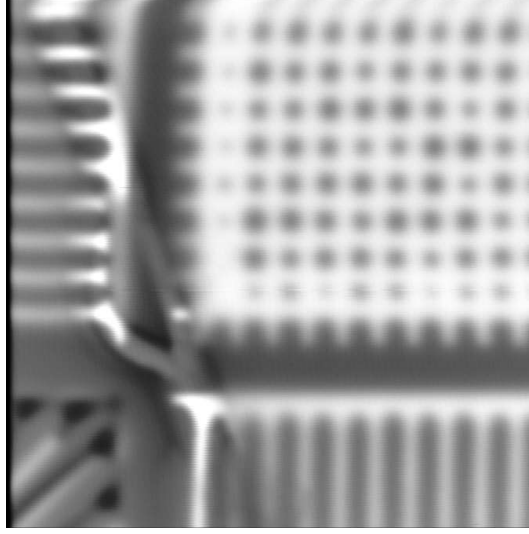


Figure 9.14: $7\mu\text{m} \times 7\mu\text{m}$ image of the smallest structures on the test sample (Figure 9.12) taken with the SEM. The smallest structures (pitch $0.5\mu\text{m}$) are clearly resolved and can be used to measure the best resolution of the SEM.

take SED images at low temperatures either the dynode must be heated or another design is needed. In this experiment the *HAMAMAZSU Electron Multiplier R5150-10* with discrete dynodes was used to collect and amplify the secondary electrons by a factor of 10^5 .

Two images recorded with the SEM are shown in Figure 9.13. The structures of the entire sample can clearly be recognized in the left image. The smaller structures are shown on the right side.

In order to measure the resolution of the SEM, the test sample was installed on the SEM flange without a cryostat. The distance between the SEM and the sample was adjusted between 0 and $\sim 5\text{ cm}$, while sample and SEM were rigidly fixed to each other.

In Figure 9.14, an SEM image of the smallest structures of the sample is shown. The $500\text{ nm} \times 500\text{ nm}$ squares can clearly be seen with the SEM. To find the resolution, of the SEM a line scan across an edge was analyzed. The space in which the signal changes from 80% to 20% of the total high is defined to be the resolution of the SEM. The final resolution of the SEM was measured to be **60 nm**. This sample was further used to measure the vibration isolation of the entire system in subsection 9.5.1.

9.3 Cryostat

Heat is a considerable source of noise for measurement at low energies. It is advantageous to cool an experiment down as much as possible. In [64] and [65], an insight into low-temperature physics is given. Nowadays, temperatures of only a few micro-Kelvin can be realized, but require a gigantic effort ([66]).

In this experiment, the LT-MT-STM was cooled down to liquid helium temperature (4.2 K) by a two stage bathycostat filled with liquid Nitrogen and liquid helium manufactured by the company *Cryovac* citeCryovac. A technical drawing of the cryostat can be found in [Figure 9.15](#). The cryostat consists of two thermally isolated reservoirs. 19.8 l of liquid helium (4.2 K) can be filled into the inner cylindric reservoir, the helium cryostat. The helium cryostat is surrounded by another reservoir holding up to 32 l of liquid Nitrogen (77 K), the Nitrogen cryostat. Inside the helium cryostat an 8 T superconducting magnet is installed.

On the one hand, each cryostat has to be thermally isolated from any higher temperature to prevent heat transfer. On the other hand, they have to be rigidly fixed to a supporting structure to provide mechanical stability. The helium cryostat is held inside the Nitrogen cryostat by three very thin tubes to reduce heat transfer from the Nitrogen to the helium reservoir. But since heat cannot only be transferred via materials, but via radiation, too, a stainless steel shield is installed between the two cryostats to shield the colder helium cryostat from the radiation of the hotter Nitrogen cryostat. The shield is cooled by the helium gas leaving the helium cryostat. The LT-MT-STM is installed hanging beneath the helium cryostat. To shield it from radiation an additional shield, called cup is installed on the bottom of each cryostat.

The LT-MT-STM can be moved 150 mm in vertical direction. In order to guarantee effective cooling, the LT-MT-STM has to be pressed onto the helium cryostat or its cup. For sample and tip transfer in and out of the LT-MT-STM via the door in the side of the cups unobstructed view through the windows in the two cups on the LT-MT-STM is needed from two sides. Therefore, the LT-MT-STM has to be brought into a middle position. In the current stage, tip or sample exchange is not yet realized. This will follow in an upgrade.

The magnet installed inside the helium cryostat is a superconducting solenoid and generates a magnetic field of up to 8 T parallel to the sample normal. In [Figure 9.15](#), the magnet is shown as two hatched areas. The magnetic field is most homogeneous and strong in the center of the solenoid. Towards the outside, the field of the solenoid is comparatively weak. To perform STM measurements in this magnetic field, it is necessary to position the STM inside the solenoid. To do so a cylindric void of 72 mm in diameter and 167 mm in height is fabricated in the bottom of the helium cryostat.

The LT-MT-STM itself is mounted below the reservoirs hanging on three tubes. These tubes provide stability for the LT-MT-STM. At the other end, these three tubes are fixed to a manipulator, so the STM can be moved up and down 150 mm. In the uppermost position, the STM is pressed to the bottom of the helium cryostat for cooling, and the sample is in the center of the magnetic field for magneto-transport measurements. In the lowest position, the STM is pressed to the helium cup for cooling and the distance between the LT-MT-STM and the SEM is smallest. As a consequence, the SEM resolution is best in the lowest STM position. For sample and tip exchange the STM has to be brought into a middle position, in line with the doors and the manipulator.

9.3.1 Temperature Measurement

Liquid helium has a temperature of 4.2 K. Due to unavoidable heat transfer from hotter sources, the temperature of the LT-MT-STM will be higher. In order to measure the actual temperature of the LT-MT-STM, the superconducting magnet and the helium cryostat, and to be aware of temperature gradients, four temperature sensors are installed. Three temperature diodes of type PT100 ([\[67\]](#)) are placed at different positions of the helium cryostat and its cup, and a calibrated cernox resistor is attached directly to the baseplate of the LT-MT-STM. The conductivity of the resistor is a direct measure for its temperature. To measure this resistance without the contribution of the cables, the four point resistance measurement method described in [section 3.5](#) is used.

9.3.2 Wiring

Each of the four STMs requires 9 wires. Additional wires are needed for the Secondary Electron Detector (SED) of the SEM, the magnet and temperature measurement. These -in total- 60 wires have to be fed into the UHV-chamber, 54 of those directly to the LT-MT-STM. 9 different feedthroughs are installed at the top of the cryostat as transition from ambient conditions to UHV for these wires.

The wires for the superconducting magnet and the two temperature diodes in the helium cryostat enter the vacuum at the very top of the cryostat, and are routed through the tubes holding the helium cryostat, as shown in [Figure 9.16](#).

The four tunneling and the two sample currents are very sensitive signals. In order to shield them, coaxial cables with stainless steel leads and shielding for low thermal conductance are used. The capacity of these coaxial wires can be heavily reduced by putting the shield on the same potential as the lead. To do so, the shields are not connected to the common ground and floating BNC are used at the cryostat. Inside the cryostat, the four cables are routed through holes in the two radiation shields. On their way through the shields, the cables are cooled down as described in [subsection 9.3.3](#). In one of the three small tubes holding the STM, the six cables are mounted. To avoid vibrational noise originating from the cables, they are wound around a smaller tube inside the tube holding the STM, and are fixed at three points. They end at the plug connecting the LT-MT-STM unit with its support.

The X, Y and Z signals for the scanner of the four STMs, as well as their ground, are mounted in the remaining two tubes. They end at the same plug as the tunneling wires. To double the maximum traveling distance of the scanning piezos, inverse voltages are applied at opposite sides of each piezo. Thus, the signals come in pairs of X^+ and X^- as well as Y^+ and Y^- , with inverted signals. To cancel the resulting fields, each wire is twisted with its counterpart. The Z-signals for the inner electrodes of the piezo tubes, as well as the grounds, have no inverted signal, so these two are coupled. To minimize the heat transfer, manganin wires with a radius of only 100 μm are used. Triple teflon coating isolates the wires from their environment.

The wires for the SED, as well as the wires for the temperature diode on the helium cup, go through the two radiation shields, as well. These wires pass between the

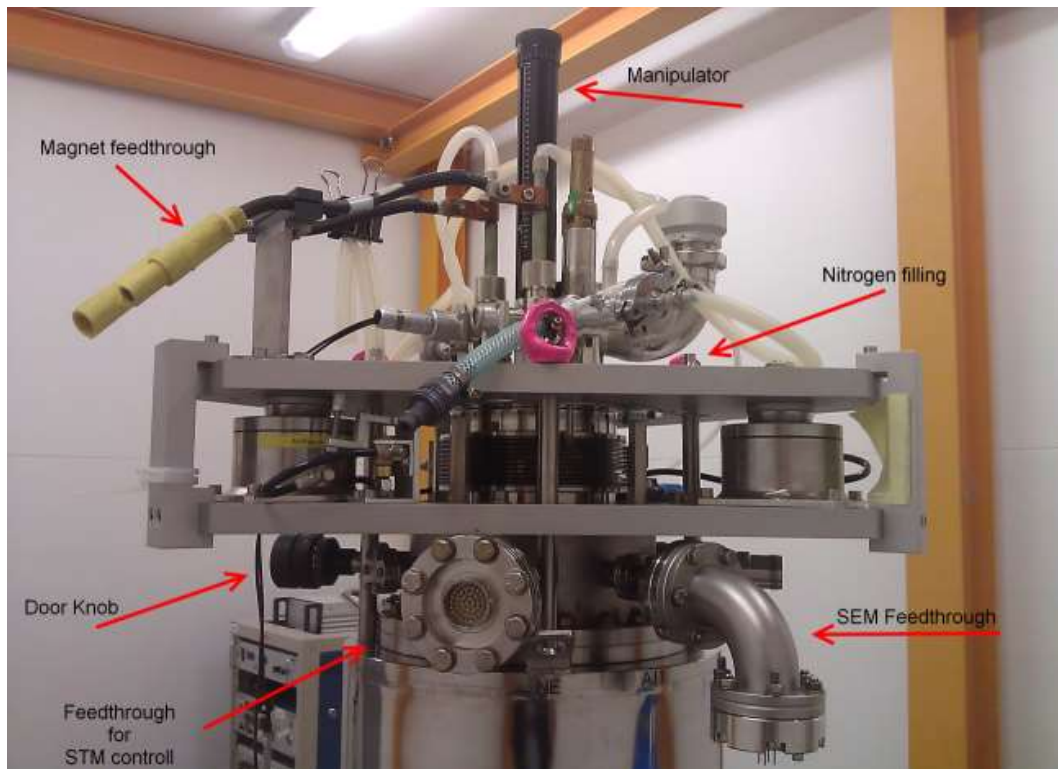


Figure 9.16: Picture of the top of the cryostat. The linear positioner, the current leads for the superconducting magnet, the liquid gas filling and exhaust, and the air-damping feed can be identified (from [Figure 9.15](#)). The visible feedthroughs are used for the wires to control the STM units, the wires and cables for the SEM detector and a knob to operate the door in the cryostat cups.

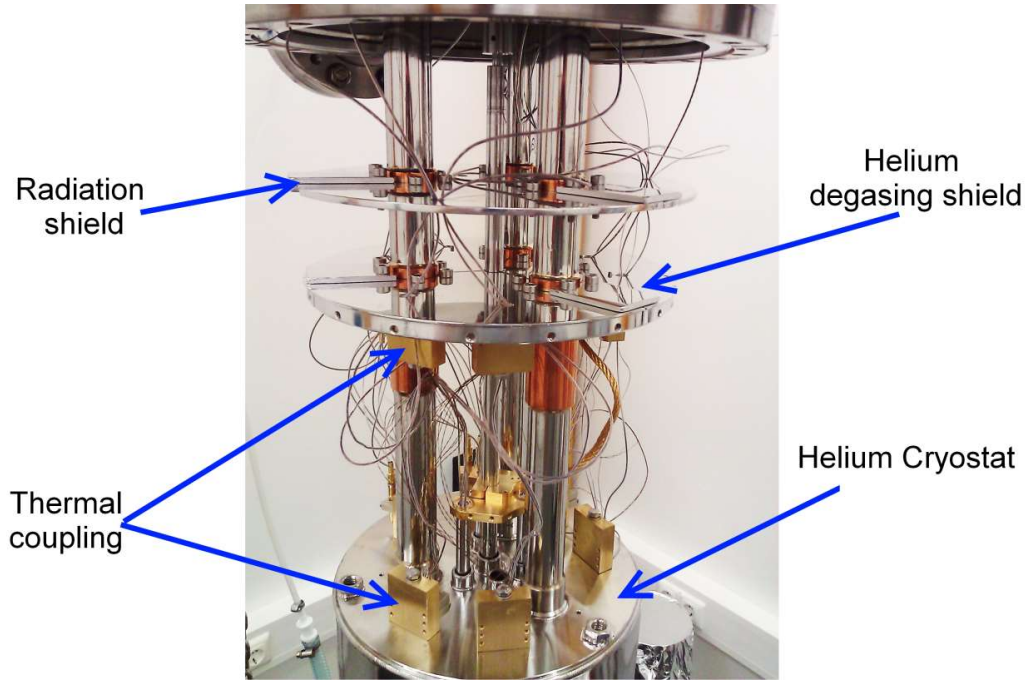


Figure 9.17: Before the wiring is fed into the cold zone of the experiment, they have to be cooled down. Each wire is glued into two gold covered copper blocks. The first block is pressed against the helium degassing shield, the second one on the helium cryostat itself. After the second block, the wires have the same temperature as the helium cryostat.

degassing cooled radiation shield and the helium cryostat. They are fixed by three springs spot welded to the helium cryostat, so they will not connect the shield with the cryostat.

9.3.3 Thermalization

To minimize heat transfer, the wires have to be cooled down on their way into the cold area of the cryostat. They are glued onto gold plated copper blocks, as can be seen in [Figure 9.17](#). Glue with high thermal and negligible electrical conductivity is used to fix each cable or wire/twisted pair to two blocks. The first block is pressed to the radiation shield, that is cooled by the helium gas leaving the cryostat (called helium degassing shield). The helium gas leaving the cryostat has to pass one of the tubes holding the helium cryostat, thus cooling it down. The helium degassing shield attached to this tube is cooled likewise. The second block is pressed on top of the helium reservoir. After the second block each wire has nearly the same temperature as the helium cryostat. To be able to service the cables and wires, a connector at each block is installed.

9.4 Measurement electronics

Each unit of the LT-MT-STM is a complete STM. Each tip is able to move individually and independently of the other tips in all three directions, scanning surfaces, performing spectroscopy measurements and so on. Each STM is equipped with a full set of measurement electronics. For the RT-MT-STM, the control signals for the four STMs are generated by two PCs and are amplified up to 200 V (maximum). For the LT-MT-STM, the control for the STMs has been "outsourced". The measurement PC uploads the specific tasks to an FPGA based control box. In the FPGA box the necessary voltage signals to fulfill this task are computed and generated. The generated signals can then be amplified up to a 400 V maximum.

To perform resistance measurements using the LT-MT-STM as nanoprobe, the same Femto-Boxes as for the RT-MT-STM ([section 4.4](#)) are used. Each STM unit can be switched to one of two modes. Either a current through the STM tip, or the potential at the tip can be measured. A current is generated by contacting a nanostructure with two tips at different potentials. It is measured with the two Femto current to voltage amplifiers at the end of the tips (mode 1). The two inner tips measure the potential (mode 2).

9.5 Vibration isolation

STM ([section 9.1](#)) and SEM ([section 9.2](#)) are not rigidly fixed to each other. The SEM is attached to the UHV chamber below the cryostat. The STM, on the other hand, hangs on thin stainless steel tubes passing through the helium cryostat. The entire arrangement of tubes has a length of 107 centimeters, as can be seen in [section 9.3](#). As a consequence, the STM swings relative to SEM.

SEM pictures will show this shaking. Structures cannot be clearly resolved when the shaking exceeds the size of that structure, and the period of the shaking is much smaller than the time to take an SEM picture. Also, the resolution of an STM is limited by mechanical vibrations. So, vibration isolation plays an important role in scanning tunneling microscopy.

Background vibrations are omnipresent. Originating from a variety of sources (cars, mining, industry, walking people etc.), vibrations travel through the ground for long distances. In general, these background vibrations are present in all frequency regimes, but the transfer function of soil is more transmissive for lower frequencies. The vibrations originating from the day-mining facilities around Jülich for example can easily be measured in the research center, even though they have to travel for approximately 10 km. Typical amplitudes of such vibration in the research center are in the $0.1\ \mu\text{m}$ regime.

It is advantageous to reduce the size of an STM itself and build it as rigid as possible. The lowest resonance will shift to higher frequencies for smaller and more rigid objects. The buildup of the STM is described in [section 9.1](#).

Due to the limited available space in the cold area, and the effort to reduce the size and mass of the devices to cool, no vibration isolation could be installed in the

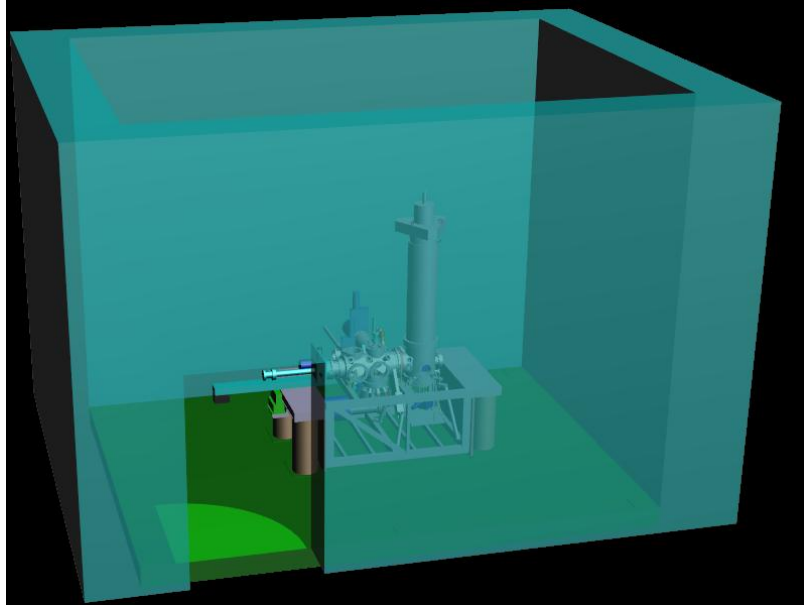


Figure 9.18: A picture of the room hosting the UHV chamber. It is situated in a second room to be shielded from external influences.

UHV chamber. Instead, a room-in-room concept was realized to isolate the entire experiment from external vibrations. In [Figure 9.18](#), a sketch of this room-in-room is presented. The UHV chamber is situated inside the inner room. The inner room itself rests on four air damping feet. It is surrounded by a second room to shield it from sound and other disturbances in the air. The entire construction was built inside a bigger laboratory. The inner room is equipped with an electro-magnetic shield. To reduce the coupling introduced by the necessary cables coming from the controls and supplies, 100 t of lead were added to the foundation of the inner room. The room-in-room concept shields the experiment from external vibrations. To prevent the generation of vibrations inside the inner room while an experiment is running, all devices that produce vibrations are located outside the inner room. In the inner room, only the devices essential for the experiment or necessary for its preparation are installed.

To prepare samples, the power supplies for the evaporators, the control of the manipulator and its heating, a mass spectrometer, and a quartz balance with their electronics are installed. Furthermore, one differential and two turbo pumps are present to evacuate the UHV chamber before the ion pump can start, as well as a liquid nitrogen reservoir to refill the outer cryostat. All these devices are switched off before an experiment starts. When the doors to the two rooms are shut, the power line to the entire room is cut, and devices are switched off, even the lights. During a measurement only the essential parts are running; ion pump, LT-MT-STM, SEM and, if necessary, the magnet.

The workspace for scientists is shown in [Figure 9.19](#), it is situated outside the outer room. From this terminal, the experiments inside the inner room can be controlled.

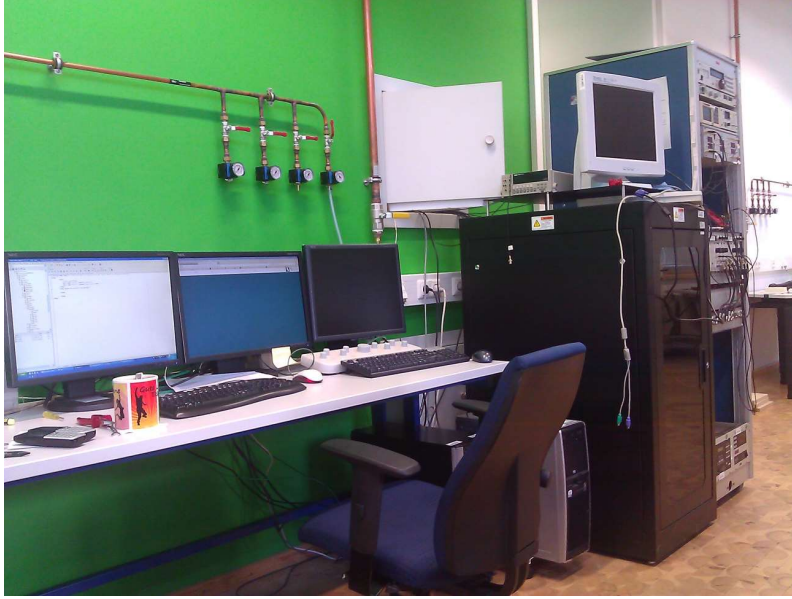


Figure 9.19: Workplace at the LT-MT-STM

Control and power lines are fed into the chamber via two hatches, from this terminal. Each hatch is equipped with doors on each side. These doors are soundproofing when closed. The cabling is pressed against the wall at the outer, as well as the inner door to reduce the coupling of vibrations transited through the cabling into the experiment.

On the left side of [Figure 9.19](#), the workstation with the two PCs controlling the LT-MT-STM and the SEM is shown; from here the measurements can be controlled and analyzed. Above the three monitors, the compressed air supplies are installed. To its right, the helium recovery line and the hatch into the inner room are situated. Below the hatch, the SEM rack is placed. Finally, the blue rack for STM control is shown in the picture. It hosts the supplies and control electronics for the four STMs as well as for the two ion pumps and the magnet.

9.5.1 Vibration Measurements

Two microscopes are combined in this experiment, the scanning electron microscope and the low-temperature multi-tip scanning tunneling microscope. The performances of the STMs are independent of the SEM, and the performance of the SEM is independent of the performances of the STMs. For charge transport measurements, single structures have to be contacted with several STM tips. To position the STM tips, the SEM is needed. In this experiment, the STM tips are positioned exclusively with the SEM in the sample plane. The resolution of the SEM on the LT-MT-STM limits the precision of the measurements.

To reduce the thermal coupling between the SEM at room-temperature, and the LT-MT-STM at liquid helium temperature, these two are not fixed rigidly to each other.

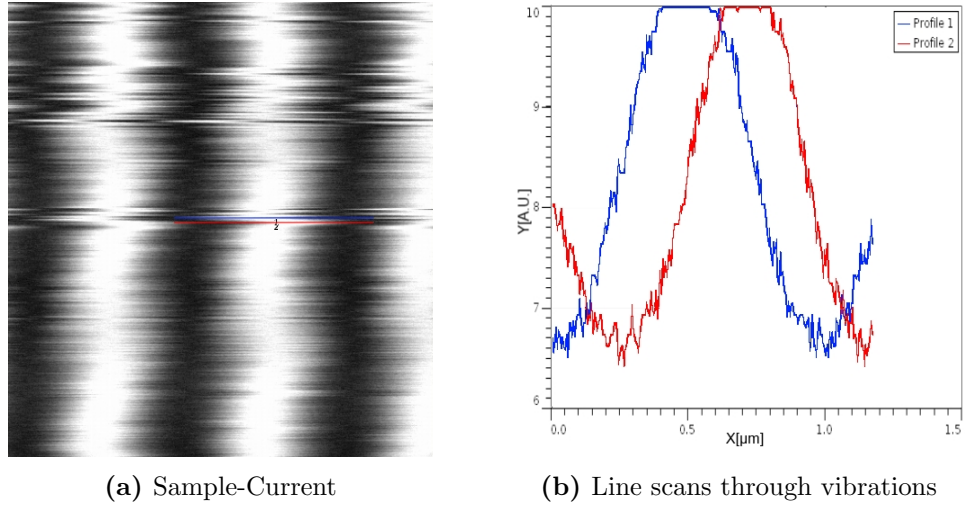


Figure 9.20: Noise in an SEM picture. On the left, a $2.5\ \mu\text{m}$ by $2.5\ \mu\text{m}$ picture taken with the SEM is shown. The straight lines are disturbed by vibration. On the right side, two line scans through the SEM-picture are shown. The position of these profiles are marked in the left picture. Due to vibrations, the profiles are shifted with respect to each other. In this example, the shift is $232\ \text{nm}$ large. In case the lines of the sample are not upright, more than 2 profiles are considered to determine the size of the vibration.

The resolution of the measurements depends on the vibration isolation capacity to limit the vibrations between the two microscopes.

The sample shown in [Figure 9.12](#) was used to measure the resolution of the SEM, as described in [section 9.2](#). Gold structures with sizes from $3\ \text{mm}$ by $0.1\ \text{mm}$ to $500\ \text{nm}$ by $500\ \text{nm}$ are deposited on a silicon substrate. These structures provide a good contrast in the SEM and were used to measure the resolution of the SEM in different configurations. The sample was subsequently installed in two different places. First, the pure SEM performance was tested with the sample attached rigidly to the SEM flange. Later, the sample was installed inside the helium cryostat, at the same position of a sample for multi tip measurements.

Furthermore, three different cryostat configurations were tested. In the first measurement, the standard configuration with a free helium cryostat hanging inside the Nitrogen cryostat was used. In a next step, the helium cryostat was blocked inside the nitrogen cryostat with Teflon wedges. In this configuration, the helium cryostat cannot swing inside the Nitrogen cryostat. A stiffer connection between the SEM and the sample is realized in this configuration. Finally, a thin nylon line was used to fix the helium cryostat inside the Nitrogen cryostat. To test the performance of the vibration isolation, each of the three measurements was executed twice, with and without vibration isolation. To disable the vibration isolation, the air damping feet were disconnected from compressed air.

The vibrations orthogonal to the electron beam can be seen directly in the SEM picture. Knowing the size of an image, the amplitude can be measured. In [Fig-](#)

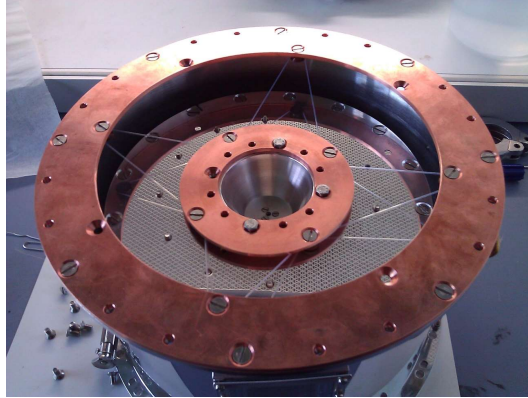


Figure 9.21: Damping of the He_{lq} cryostat inside the N_{lq} cryostat. At the bottom of each cup, an additional ring is installed. By winding a Teflon wire alternating through the two rings, their relative movement is dampened.

Figure 9.20 an example of such a measurement is shown. Three parallel lines on the test sample are disturbed by vibrations. To measure the size of them, two line scans orthogonal to the structures were generated. The resulting profile is not a representation of the height, but of the contrast in the picture. The two lines are placed on a structure that is disturbed due to vibrations. By moving the lines perpendicularly to the scanning direction, two neighboring extreme points of the vibration can be found. At this point, the shift of the peaks in the two line profiles is maximal and corresponds to the amplitude of the vibration. In order to assure that a tilt of the used structure in the SEM image does not contribute to the vibration measurement, not two but three line scans in neighboring maximums are used.

In Table 9.1, the results of these measurements are presented. The resolution of the SEM with active vibration isolation is compared to the resolution with disabled vibration isolation. It can be seen that the resolution is increased by a factor of ~ 8 . Also, it is very advantageous to install an additional damping between the helium and the Nitrogen cryostat.

The Teflon wedges used have to be pressed firmly between the two cryostats to prevent the helium cryostat from swinging. Consequently, the heat transfer through the wedges is considerable even with the small thermal conductance of Teflon. To reduce the heat transfer, the motion of the helium cryostat inside the Nitrogen cryostat is damped using a Nylon wire, as shown in Figure 9.21. The wire is wound around posts on two rings, one for each cryostat cup. By tightening the wire, the movement of the inner cup is limited and damped.

Not only can the amplitude of the vibrations be measured, but the frequency spectrum of noise, too. A line scan in a featureless region shows only electronic noise. When it is placed directly next to the gold structure, electronic and vibrational noise results. For different scan sizes, speeds and directions, electronic and vibrational noise can be distinguished. Since the scan speed is known, the line profile can be transferred into a time signal. A Fourier analysis of this signal results in the frequency spectrum of noise.

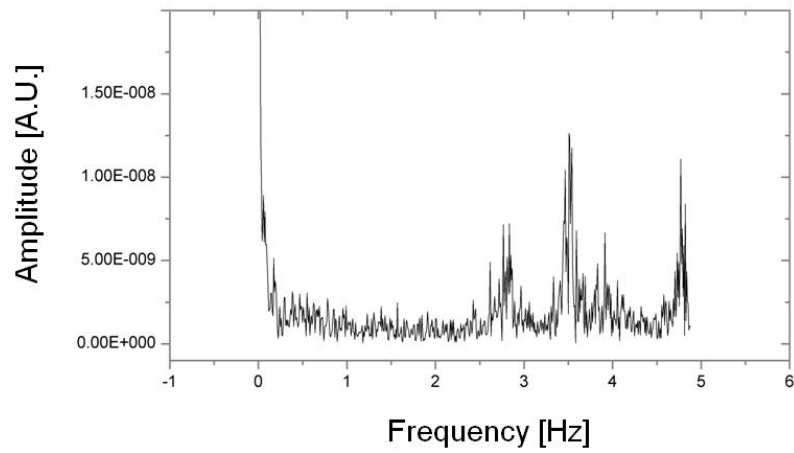


Figure 9.22: The low frequency part of the noise spectrum of the SEM signal shows several peaks between 2.5 Hz and 5 Hz. The peak at 3.5 Hz is in the same regime as the resonance frequency of the cryostat, and disturbs the measurements most.

Vibration isolation	ON	OFF
No cryostat	60 nm	220 nm
Free cryostat	815 nm	6510 nm
Blocked cryostat (Wedges)	200 nm	1558 nm
Blocked cryostat (Wire)	240 nm	n.m.

Table 9.1: Resolution of the SEM for five different setups was measured: without a cryostat, with a free cryostat, with a cryostat blocked with wedges, and with wires. For the first three, the measurement was carried out with enabled as well as disabled vibration isolation. The vibration isolation enhances the resolution by a factor of ~ 8 .

It turned out that the vibration spectrum (Figure 9.22) has three to four distinctive peaks between 2.5 Hz and 5 Hz. Mathematically, the period of oscillation of the cryostat as a ballistic pendulum is ~ 3 Hz. Unfortunately, the vibration isolation is transmissive in this frequency regime. Since the characteristics of the cryostat cannot be changed, further improvement of the vibration isolation was necessary.

9.5.2 Additional measurements and final configuration

To evaluate the results of the vibration measurements, and to measure the vibration spectrum inside the room-in-room without convolution with the motion of the cryostat, further measurements were performed. Three seismographs were placed in the inner room, while an additional seismograph was positioned outside. By comparing the vibrations outside of the room-in-room with the measurement inside the inner

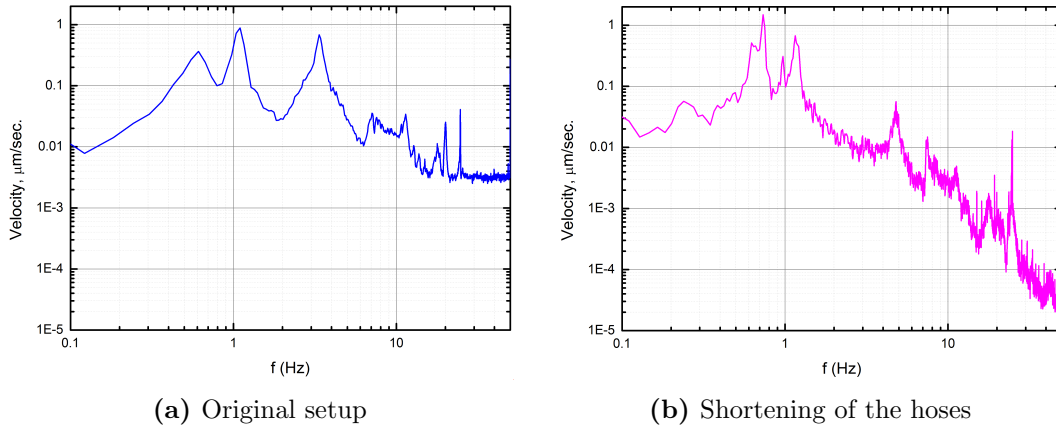


Figure 9.23: In the original vibration spectrum (a), several peaks are present. The peak at 3.35 Hz overlaps with the resonance frequency of the cryostat. By shortening the balance lines between the damping feet and the compressed air tank, this peak could be eliminated (b).

room, the performance of the system was tested. The outer seismograph measured the vibrations on the concrete directly next to one of the four air damping feet. The three inner ones were positioned at different points in the inner room, with one of them positioned at the spot where the cryostat would later be installed.

The measurements were carried out in two phases. In the first phase, the environmental background vibrations (cars, day mining, working people, geological activities, etc.) were measured, as they occur on a normal day. In the second stage, vibrations were generated on the concrete of the foundation of the laboratory, and their coupling into the room was measured.

These measurements (Figure 9.23a) supported the results of the previous measurements. The room-in-room shows a resonance frequency at ~ 3.35 Hz, among others. Unfortunately, the peak at 3.35 Hz in the frequencies spectrum of the room-in-room, and the resonance frequency of the cryostat overlap, and vibrations within this frequency overlap can pass into the LT-MT-STM nearly undamped.

One of the two resonance frequencies had to be shifted in order to optimize the vibration isolation. It was decided to change the room-in-room. The inner room is resting on four Schiller air damping feet. These feet are driven by compressed air. Between the feet and the supply of compressed air, a large reservoir was installed to compensate variations in the supply, and to reduce the frequency of the lowest resonance.

The vibrations that are transmitted through the feet into the inner room, are also present in the balancing line to the reservoir. Standing waves are formed inside the balancing line. By shortening the balancing lines, the geometry of the standing waves was changed. The resulting vibration spectrum inside the inner room is shown in Figure 9.23b. The resonance peak at 3.35 Hz has shifted to a higher frequency, as intended. Instead, a peak now occurs at 4.7 Hz. Additionally, the two peaks around 1 Hz are shifted to higher frequencies. The lowest resonance grew to $1.5 \frac{\mu m}{sec}$.

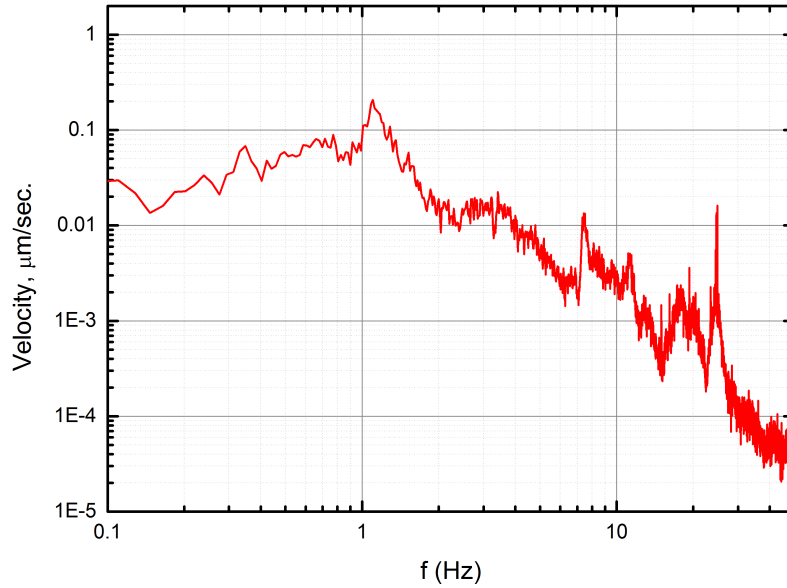


Figure 9.24: Frequency spectrum of the vibration after the vibration isolation was optimized. The peaks at 0.6 Hz and 3.35 Hz could be eliminated. The peak at 1 Hz and the vibrations for higher frequencies are damped more strongly.

To break the standing wave responsible for the 4.7 Hz resonance, an additional $\frac{\lambda}{4}$ -pipe was installed by Dr. Duden. This pipe leaves the balancing line in a 90°-angle and has a length of a quarter of the wavelength of this standing wave. At its end, the pipe is closed with a hard end. A wave traveling into this line will be reflected at its end and superimpose with itself. The phase of the wave leaving the balancing line is shifted by $2 \times \frac{\lambda}{4}$. Destructive superimposition between the waves entering and leaving the $\frac{\lambda}{4}$ pipe takes place, and the two waves cancel each other out.

The final result (Figure 9.24) shows the expected form. The peaks around 0.6 Hz, 1 Hz and 3 Hz in the original vibration spectrum have been strongly reduced. The peak around 1 Hz was reduced by $\sim 80\%$ and is still prominent in the spectrum. The two other peaks are nearly reduced to base level. By optimizing the vibration isolation, the performance needed to perform high resolution charge transport measurements was reached.

Finally, a resolution of 110 nm could be achieved with the SEM.

Chapter 10

PN Sample (M4523)

The sample for charge transport measurements with the LT-MT-STM was fabricated in the same process as the nanowire samples for the RT-MT-STM ([subsection 5.1.1](#)). The GaAs substrate is highly doped with zinc ($1.0E19/cm^3$).

The nanowires on this sample are made from GaAs as well. They have three different segments. The approximately $1\mu m$ long nanowire base grown at high temperatures ($450^\circ C$) is p-doped with zinc (Zn). The doping is expected to be in the order of $2.0E17/cm^3$. The second nanowire segment is p-doped with zinc too, but much higher $8.0E18/cm^3$ (expected), it has a length of $7\mu m$. The final (and top) part of the nanowire is n-doped with tin (Sn). According to the growth parameters, the doping is expected to be between $7.0E17/cm^3$ and $2.0E18/cm^3$. In [Figure 10.1](#), a plot of the expected doping profile is shown. Finally, at the top of each nanowire, a gold particle is present due to the growth process.

In [Figure 10.2](#), an SEM image of a few nanowires on the sample is presented. In the middle of the nanowire ($8\mu m$ above the substrate), a pn-junction has been realized. In total, the length of the nanowires on this sample is $16\mu m$, according to the growth parameters.

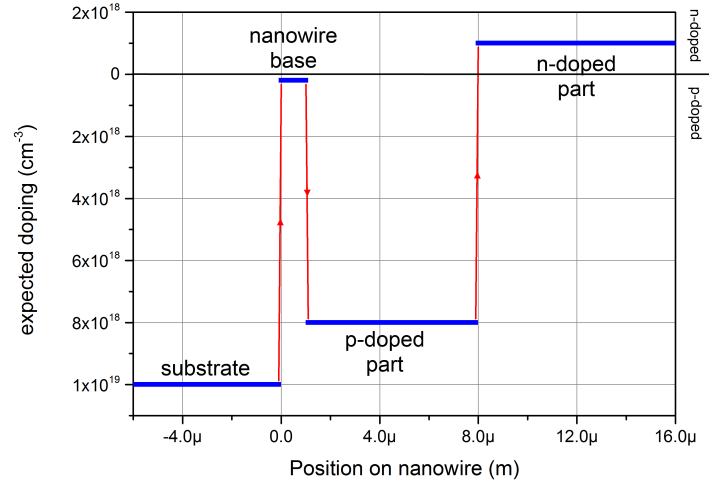


Figure 10.1: The expected doping profile along a nanowire of the PN Sample. While the substrate, the nanowire base and the first part of the nanowire are p-doped with zinc, the upper part of the nanowire is n-doped with tin.

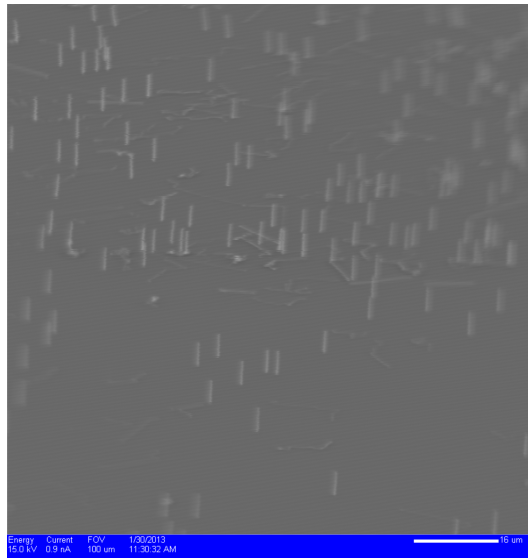


Figure 10.2: This SEM image of GaAs nanowires was taken with the SEM mounted on the LT-MT-STM system. Several nanowires are present in the $50\,\mu\text{m} \times 50\,\mu\text{m}$ image. Most of the nanowires show kinks or are disattached from the substrate, but two straight nanowires are present in the depicted area.

Chapter 11

Measurements

The first task in performing measurements on nanowires is to contact them. Many different approaches are used in today's science([Figure 6.1](#)). The experiments described in this part of the thesis were carried out like the measurements described in [chapter 5](#).

The nanowires are contacted standing upright as grown by up to three scanning tunneling microscope tips, while the fourth contact is realized by the interface between the nanowire and the substrate. The LT-MT-STM is used as a nanoprobe. To perform a four point measurement ([section 3.5](#)), one tip has to inject the current, while the two other tips measure the voltage difference along the nanowire. In [Figure 11.1](#), an SEM image of a nanowire contacted with a single tip is shown.

Under SEM surveillance, the first tip can be positioned above the nanowire. Using autoapproach nanowire and tip can be brought into contact. Due to imperfect alignment of the KoalaDrive with the SEM line of sight, the tip changes its position above the sample. The occurring tip movement has to be corrected.

Once contact is established, current can flow from tip 1 through the nanowire into the sample. The gold particle at the top of the nanowire attaches firmly to the tip, so not only can charge transport measurement be performed stably, but also the nanowire can be bent by moving the tip.

11.1 Electron Beam Induced Current (EBIC)

When a nanowire is contacted with an STM tip in current measurement mode, the contrast in SEM Current Images changes, as described in [section 5.6](#).

In [section 5.6](#) the EBIC signal could be identified as a change in the conductance of a nanowire due to the generation of electron hole pairs in the depletion zone between nanowire surface and its core by the electron beam of the SEM. The region in which such a signal occurred, could be identified as the regions with strong bent bands.

In [Figure 11.2](#) the contrast along the nanowire in [Figure 11.1](#) is shown. At 0 nm the interface between nanowire and substrate is positioned. Above 0 nm, the contrast

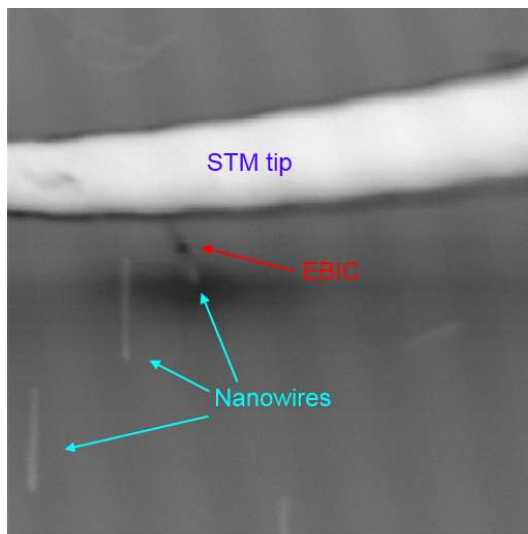


Figure 11.1: A nanowire contacted by an STM tip shows an EBIC signal when the tip is in current measurement mode, and a bias is applied. In this case, the bias is -3V. The substrate also shows an EBIC signal. The two unconnected nanowires do not show such a signal.

along the nanowire is shown, below 0 nm, the contrast on the substrate around the nanowire. The EBIC signal on the nanowires is positioned exactly $8\ \mu\text{m}$ above the surface and has a size of $1.6\ \mu\text{m}$.

In Figure 11.3, a series of SEM images is shown. Each image was taken with a different bias applied. The contrast in SEM images in Figure 11.3 is dynamically adjusted, which makes a quantitative analysis impossible, but qualitatively, several different features can be identified.

The EBIC signal changes with changing bias. For biases between -1000 mV and 4000 mV, no EBIC signal is present in the SEM images. For higher (more positive) biases, two changes occur. The base starts to glow white, the brighter the higher the applied bias is. Additionally, a dark band in the middle of the nanowire is present. For biases below (more negative) -1000 mV, the dark band also occurs in the middle of the nanowire, but much more pronounced. For biases below -2000 mV the substrate around the nanowire base is darkening.

The positions where the different EBIC signals occur are identical to the position where the interfaces between differently doped segments of the nanowires are expected. The interface between the p-doped substrate and the p-doped base of the nanowire ($1E19/\text{cm}^3 \Rightarrow 2E17/\text{cm}^3$) shows a dark shadow for biases below -2500 mV. The interface between the p-doped and the n-doped part of the nanowire ($8E18/\text{cm}^3 \Rightarrow 1E18/\text{cm}^3$) shows a shadow already from -1500 mV below. This interface shows a similar contrast also for positive biases above 5000 mV.

A second change occurs in the contrast along the nanowire. Above 5000 mV, the interface between the base of the nanowire and the p-doped part starts to glow white

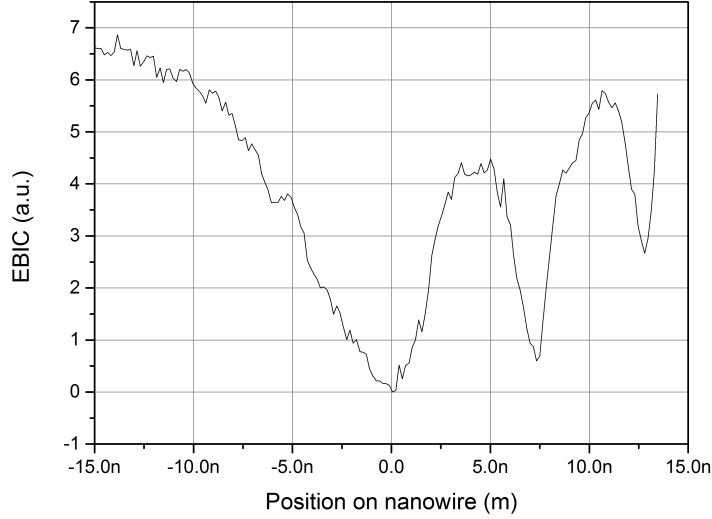


Figure 11.2: EBIC signal measured with the LT-MT-STM setup. Line scan along the nanowire shown in [Figure 11.3](#). At 0 nm, the interface between nanowire and substrate is located. The nanowire is located above 0, while the substrate is located below. The EBIC shadow on the nanowire is located at 8 nm with a size of 1.6 nm.

$$2E17/cm^3 \Rightarrow 1E18/cm^3.$$

In [Figure 10.1](#), the expected doping along each nanowire and in the substrate is plotted. The EBIC signal of a transitions from lower to higher doping level (in [Figure 10.1](#)), generates a dark shadow, while a downward transition glows white. The EBIC signals supports the expected doping profile very accurately. In areas with a positive doping gradient (in [Figure 10.1](#)), electron bombardment results in an increasing current flow through the nanowire. Negative gradients result in a decreasing current flow.

By injecting electrons or holes into regions with high doping gradients, the diode like characteristic of such regions is either in- or decreased, thus in- or decreasing the resistance of this area.

From the EBIC-signal the first conclusions on the electronic structure can be drawn. The signal in the center of the nanowire implies a diode like transition in this area. Either from the n-doped part to the p-doped part (or vice versa) or from core to surface conductance. The dark halo around the nanowire base indicates a similar transition on the substrate. Since the halo only occurs for biases below -3 V, the electronic structure in this area differs from the structures of the Gradient and the Constant Sample (see [chapter 5](#)).

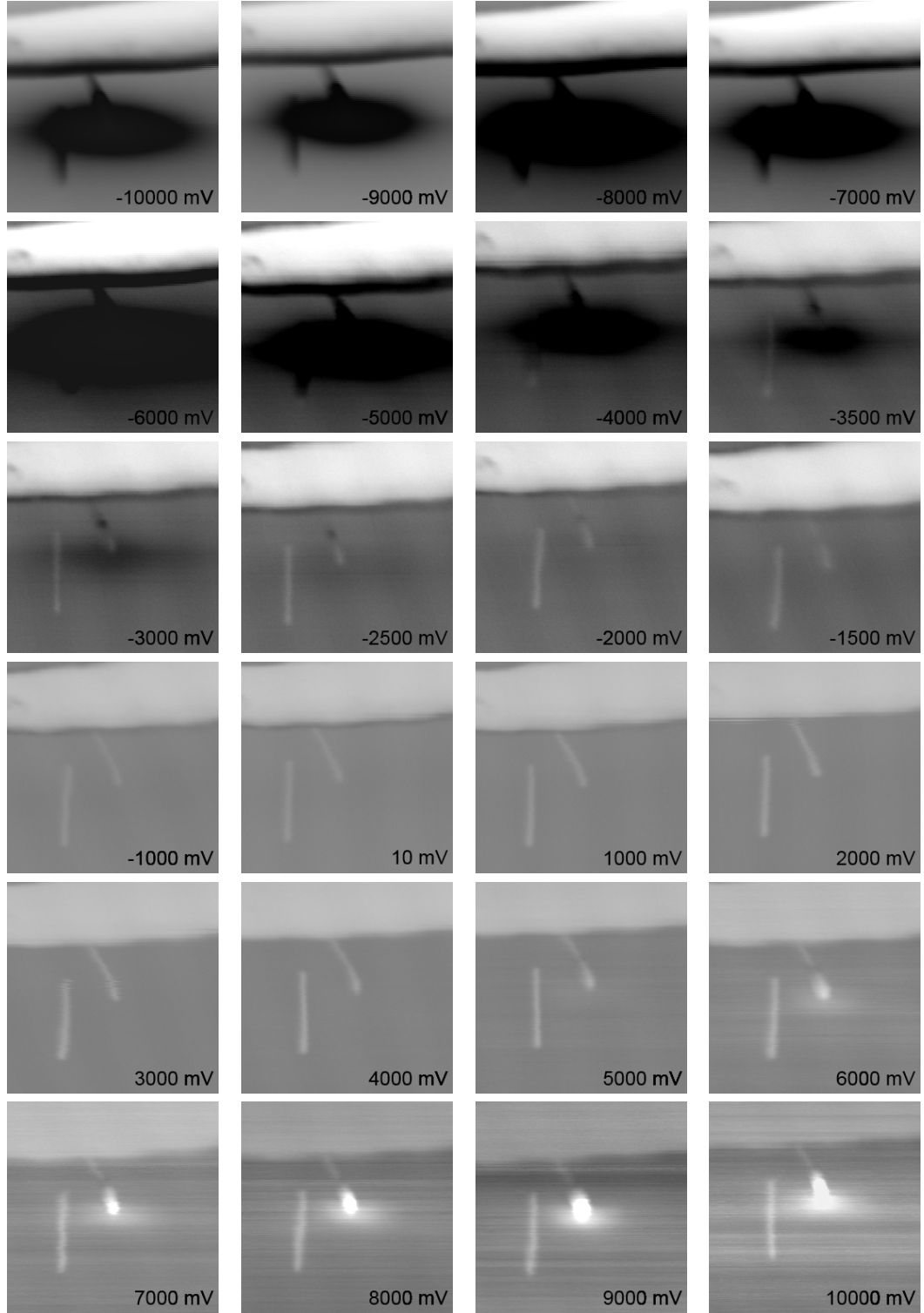


Figure 11.3: SEM pictures of a contacted nanowire. The nanowire is contacted by an STM tip. Different voltages are applied between nanowire and sample for each subpicutre. The current through the nanowire is altered by the electron beam of the SEM.

11.2 Multi-Tip measurements

For EBIC measurements, the nanowire was contacted with one tip. For charge transport measurements, like described in [chapter 5](#), it was necessary to contact a nanowire with all three tips.

When more than one tip was approached to the sample, the SEM images became distorted. In [Figure 11.4](#), a series of SEM images is shown. In [Figure 11.4a](#), two tips are visible, in front of the sample in the background. On the sample, nanowires are visible as fine lines. While the right tip is displayed clearly, the left tip seems to get wider to the left. The top half, especially on the right side, also seems stretched to a point above the image.

After the first image was taken, the third tip was moved down from above the image. The SEM image [Figure 11.4b](#) gets more distorted. The left tip is not fully depicted, and the lines of nanowires are blurred. In total, the distortion moved down with the third tip. Also the image of the right tip is stretched.

Before the next image ([Figure 11.4c](#)) was taken, the third tip was moved down approximately the same distance as between the first two pictures. As a result, the distortion of the images increased. The left tip seems to have completely vanished, but the third tip is now visible. It enters the image from the top left. The right tip and the third tip in this picture seem to touch each other, while the form of the two tips is barely recognizable. Only a small area in the lower right corner of the sample is left unaltered. In the last image, [Figure 11.4d](#), the two tips form a continuous band, while also the sample is (nearly) completely distorted. The left tip is not visible.

By moving the third tip into the picture, the electron beam of the SEM is distorted. Either an electric or a magnetic field is in the path of the beam changing its direction, as well as the form of the beam. The electric and magnetic fields in the SEM column can be adjusted to compensate such deformations, but only to a small extent. Deformations in the order, as shown in [Figure 11.4](#), cannot be compensated.

This effect is not limited to tip three. For each configuration with more than one tip, these distortions occur in the SEM images. The origin of these fields can be in various parts of the LT-MT-STM. By design, most surfaces of the LT-MT-STM are pinned to a defined electrical potential. The housing and the supports of the LT-MT-STM are grounded. The tips of the three STM units are connected to their individual grounds, while the scanning piezo are connected to their control voltage. All grounded elements can be excluded as sources for unintended electric fields.

Only the STM tips and the scanning piezos are not grounded by design. While the images in [Figure 11.4](#) were taken, all additional potentials were pinned to the ground level of the UHV chamber. Only a few parts in the LT-MT-STM need to be fabricated from isolating materials to separate different potentials from each other. The tip holder on top of the KoalaDrives, the glue to keep the different parts together, and the part of the piezos between the contacts are nonconducting. These parts can be charged due to the electron beam. When the electrons are transported off the isolators, an electric field is created.

In principle, a charged isolator could be responsible for the disturbance in the SEM

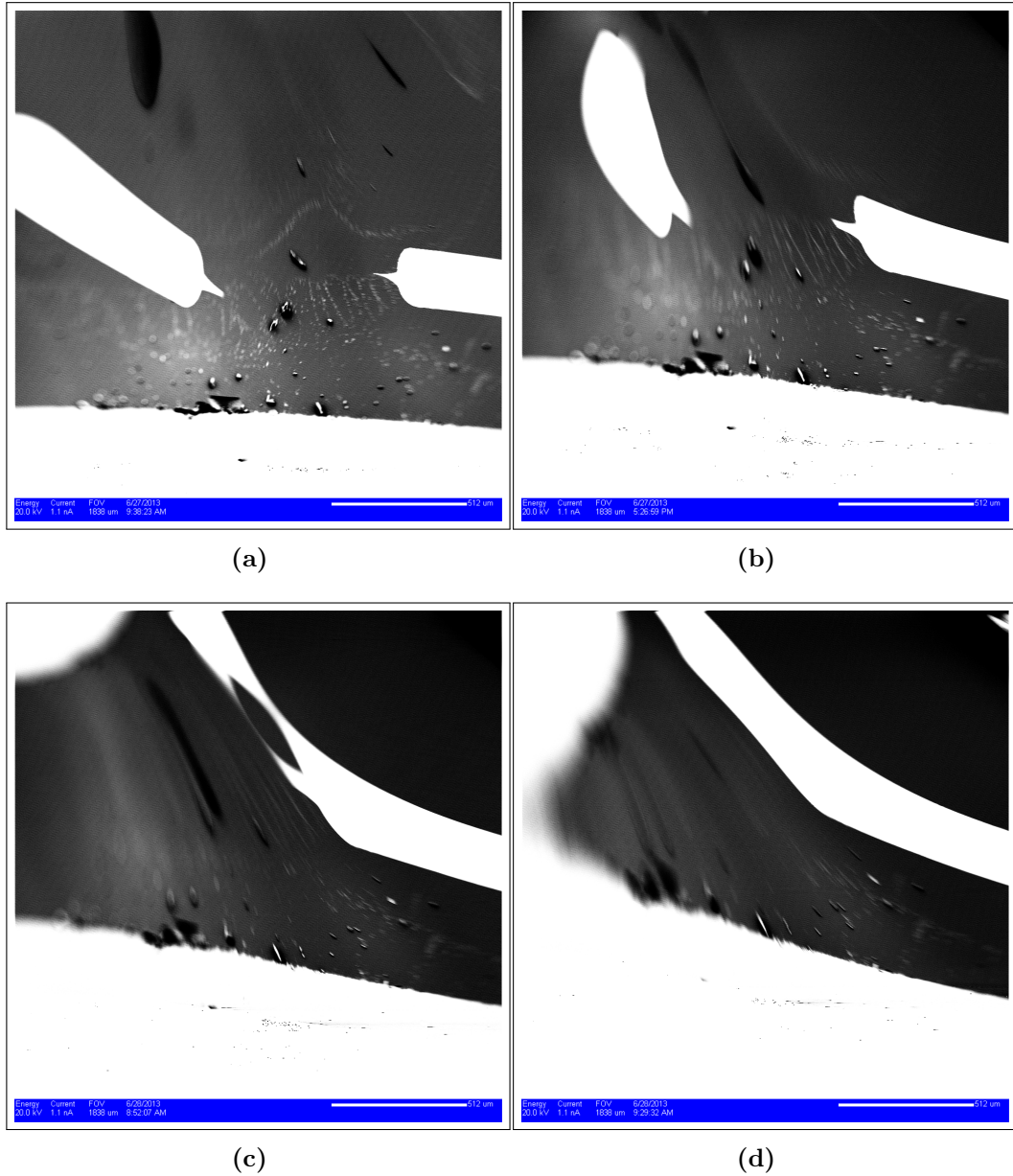


Figure 11.4: In the first image (a), two STM tips are visible as white shadows in front of the sample. While the right tip is depicted correctly, the shadow of the left tip is deformed. From (b) to (d), a third tip is moved down from the top. By moving the third tip from the top of the image down, the image becomes entirely distorted.

images, but the amount and size of isolating surfaces in the inner of the LT-MT-STM are reduced as much as possible. Furthermore, these (or similar) parts are also used in other experiments, without creating such problems.

Magnets are only used at two different points in the LT-MT-STM. The black plates (Figure 9.5) that carry the KoalaDrive are attached by magnets. Also, the sample was fixed to the LT-MT-STM with magnets. While the black plates are considerably far from the electron beam, the other set of magnets is close to the sample and the beam. The design of the LT-MT-STM was changed to get rid of these magnets. Three screws replaced the magnets and hold the sample in its position.

The distortion could not be eliminated by the design change. At the time this thesis was written, the problem could not be resolved. In this state measurements could be executed with only a single tip. Multi-tip measurements under these SEM conditions are not possible.

Chapter 12

Conclusion and Outlook part III

Based on the room-temperature multi-tip STM, a new customized multi-tip scanning tunneling has been build to carry out charge transport measurements at low temperature (down to liquid Helium). Additional an 8 T superconducting magnet was installed to enable magneto transport measurements. An SEM is used to surveillance the STM tips during operation. To shield the STM from vibrations, the experiment is placed in a room-in-room. The inner room is equipped with a 100 T foundation and resting on airdampening feed to reduce the coupling to the outer world. To optimize the vibration isolation and find the resonance frequencies of the experiment, SEM images taken with the build in SEM of a test sample were used. A newly developed nanopositioner was used to approach the STM tips to the samples. At low temperatures the KoalaDrive operates with high precision. It is also operating in magnetic fields. Measurements on three different samples were performed with the LT-MT-STM, Graphite, a Pt(100) bead crystal and a GaAs sample with GaAs nanowires. On the Graphite sample and the bead crystal atomic steps could be resolved. Additionally the Pt(100) 5×25 reconstruction was resolved in STM pictures. The STM resolution is better than 1.4 nm. Single nanowires from the GaAs sample were contacted with single tips and two-point transport measurements were performed. Charging of a tipholder led to an unintended electric field inside the STM. Thus the SEMs electron beam got distorted and no multi-tip measurements were possible.

The SEM, as well as each STM unit show good performance when used as stand alone devices. Also, the combination of one STM unit with the SEM is capable of performing precise measurements. But when two or more STM tips are brought close together, the SEM does not provide clear images of the measurement. The SEM images showed a heavily distorted picture of the STM tips and the sample.

In this state, only single tip measurements are possible. The EBIC measurements gave a first hint about the electronic structure along the nanowire. To get detailed information about the development of the electronic structure and the interfaces between differently doped nanowire parts, multi-tip measurements are needed. To perform multi-tip measurements, the surveillance with an SEM (or another micro-

scope) is essential.

For the further operation of the system, the highest priority is to eliminate the source of the disturbance, that has been identified as charging of insulated part. But, additionally, some other tasks remain. To use the full potential of the LT-MT-STM, tip and sample exchange have to be realized. A variety of different, self-assembled nanostructures can be produced in situ, that do not sustain the transport in ambient conditions. Additionally, the surfaces of in situ produced or cleaned samples are not unintentionally oxidized and thus better suited for charge transport measurements. Also the oxide layer that forms when tungsten is exposed to oxygen can only be removed in situ, when a working tip and sample exchange is established in the LT-MT-STM.

Once tip and sample exchange are established, and the SEM problems are resolved, the LT-MT-STM is ready to use its full potential. It then will be capable of performing STM and SPM measurements with high spacial resolution in a temperature range from 4.2 K to 350 K. Additionally charge and magneto transport measurements with up to six (two stationary contacts at the sample plus four movable STM tips that can be positioned with SEM precision) contacts will be possible.

Part IV

Appendix

Acknowledgments

This thesis would not have been possible without the support and help of many people. At this point I would like to articulate my thanks to all the people who supported me during this time.

First of all my special thanks goes to my advising Professor **Bert Voigtländer**, who's tremendous effort in encouraging and guiding me, made this thesis possible in the first place. Also I would like to thank **Markus Morgenstern** who agreed to be my second assessor.

Stefan Korte's introduction into the operation of the room-temperature multi-tip STM and the countless discussions over the interpretation of the measured data were an important key to the understanding of the resistance profiles. Also **Marcus Blab**, **Evgeny Zubkov** and **Felix Lüpke** were always a great help in scientific and technical issues.

For the realization of the low-temperature multi-tip STM **Vasily Cherepanov** was a great help in all technical and scientific questions. When ever technical problems occurred, also the support of **Franz-Peter Coenen**, **Helmut Stollwerk** and **Claudia Steufmehl** were a keystone to the experiments.

Without the samples fabricated by **Udo Linke** and **Matthias Steidl** the measurements and thus this thesis would have been impossible. But also all the technical personal in the Research Center who fabricated all the needed parts and electronic components were essential for the success of the experiments. During the time in the Research Center **Claudia Klamandt** and **Sonja Lexen** were always a great help with the necessary bureaucracy.

Besides the professional input, the work atmosphere created by all the colleagues from the **PGI-3** as well as the many friends from the entire Research Center Jülich made my time in Jülich an important part not only in my career but in my entire life.

Proper english the text **Timo Klein** has helped make.

Last but not least, unconditional support and love of my **family** and **friends**, especial my parents **Dorothee Junker-Schnütgen** and **Hartmut Junker**, my girlfriend **Nicole T. Haider** and my son **Magnus Felix Laser** helped me through the entire process.

Acknowledgments

List of Figures

2.1	Electron distribution in k-space	8
3.1	Sizes in comparison from a human being to an atom	12
3.2	Instruments by their Resolution	13
3.3	Diagram of 1-Dimensional tunneling	14
3.4	One-dimentional tunneling classical vs. quantum mechanics	15
3.5	Thermal Field Emitter (TFE)	18
3.6	SEM-beam acceleration and focusing	19
3.7	SEM image of a nanowire sample	21
3.8	Shape of STM tips for multi tip measurement.	22
3.9	Difference between two- and four- point resistance measurements	24
4.1	Side view of the RT-MT-STM	30
4.2	RT-MT-STM UHV chamber	31
4.3	Different SEM contrasts	32
4.4	Vibration isolation of the RT-MT-STM	34
5.1	Concept of four point measurements on nanowires	38
5.2	Over view SEM image of Probe M4580	39
5.3	Contacting of a nanowire	39
5.4	Applied Voltage during 4 point measurements	40
5.5	Signals of a 4 point resistance measurement	41
5.6	Resistance profile a nanowires on sample M4580	43
5.7	An overview over the different types of Nanowires	44
5.8	HRSEM images of the samples before resistance measurement	46
5.9	Overview images of all there samples	48
5.10	Signals of a 2 point resistance measurement	49
5.11	2 Point IV of sample M4586	50
5.12	Signals of a two-point resistance measurement	51
5.13	Three point resistance measurement	53
5.14	3 Point IV of sample M4586	54
5.15	Three point resistance measurement	55
5.16	Four-point resistance measurement along a nanowire	56
5.17	4 Point IV of sample M 4580	56

List of Figures

5.18	Constant Sample four-point resistance profile for negative and zero bias	57
5.19	Fitted Constant Sample four-point resistance profile	58
5.20	Evolution of four-point IVs along the Constant Sample	59
5.21	IVs along the Step Sample	60
5.22	Resistance profile of nanowires 02 on sample M4509 with fits	61
5.23	Resistance profiles for three nanowires on sample M4586	61
5.24	4 Point resistance profile of nanowire 02	62
5.25	4 Point resistance profiles around 0 A of all measured nanowires	64
5.26	An overview over the different types of Nanowires	64
5.27	Resistance profile compared to the temperature during growth for NW08 M4580	65
5.28	Resistance profile compared to the temperature during growth for NW06 M4509	66
5.29	Resistance profile compared to the temperature during growth for NW09 M4586	67
5.30	HR-SEM images of nanowires of all samples	69
5.31	Diameters the three analyzed nanowires	70
5.32	Cut through a nanowire showing the electrical structure	71
5.33	Model of NW doping profile, space charge laxer and Band structure along the nanowire. Radial Band structures along nanowire	75
5.34	Common regions in nanowire resistance profiles	76
5.35	High resolution SEM image with resistance profile and IVs from the three regions	77
5.36	Equivalent circuit diagram	79
5.37	Measurement of the high current fit offset	80
5.38	High current fit offset progression through the Gradient Sample nanowire	81
5.39	High current fit offset progression through the Step Sample nanowire	82
5.40	High current fit offset progression through the Constant Sample nanowire	82
5.41	Comparison between the predicted resistance profiles and the measured	84
5.42	Difference between the fit at 0 A and the one at high currents for the Gradient Sample	85
5.43	Cross section and band structur along the nanowire	86
5.44	Band structure between region 2 and 3 with and without bias	87
5.45	Band structure for an intrinsic and a p-doped nanowire	88
5.46	Comparison of two SEM images taken with an SED and Sample Current	89
5.47	EBIC vs. high current offset for the Constant Sample	90
5.48	EBIC vs. Band structure for the Gradient Sample	91
6.1	Example for a four-point resistance measurement by lithography	94
6.2	SEM image of the long run measurement configuration	95
6.3	Applied Voltage and resulting current during long run	96
6.4	Long run measurement results	97
6.5	Resistance profile measured over several days	98
6.6	Tip-Nanowire interaction	99
6.7	Tip configuration for gate measurements	100

List of Figures

6.8	Result of three-point gate measurements on M4580	101
6.9	Result gate measurement on constant temperature	102
7.1	Cutting a nanowire	106
9.1	Icon for pn and solar cell based on nanowires	112
9.2	An overview over the UHV system	114
9.3	Technical drawing of the LT-MT-STM	116
9.4	An STM image of an atomic step on a graphite sample	118
9.5	Top view on LT-MT-STM	119
9.6	Side view drawing of LT-MT-STM	119
9.7	STM image of PT (100)	120
9.8	Line scan across five steps in the STM image of PT (100)	121
9.9	Technical drawing of the KoalaDrive	122
9.10	KoalaDrive drive step size	124
9.11	Surface of a Ti tube used in the KoalaDrive	125
9.12	Drawing of the SEM testsample	126
9.13	SEM image of the testsample	126
9.14	SEM image of smallest structures on the SEM testsample	127
9.15	Technical drawing of the cryostat	129
9.16	Connections at the top of the cryostat	131
9.17	Thermalization of the wires and cables inside the cryostat	132
9.18	Room in room concept	134
9.19	Workplace at the LT-MT-STM	135
9.20	Vibration measurement principal	136
9.21	Damping of the He_{lq} cryostat inside the N_{lq} cryostat	137
9.22	Frequency spectrum of SEM noise	138
9.23	vibration measurements of the room-in-room concept	139
9.24	Frequency spectrum of vibration after the vibration isolation has been optimized	140
10.1	Expected doping PN Sample	142
10.2	SEM image of GaAs nanowires for the LT-MT-STM	142
11.1	Concept of EBIC measurements	144
11.2	EBIC signal measured with the LT-MT-STM setup	145
11.3	EBIC Overview	146
11.4	SEM disturbance by tip movement	148

List of Figures

List of Tables

5.1	An overview over the different types of nanowires and their typical dimensions	47
9.1	SEM resolution depending on the vibration isolation	138

List of Tables

Bibliography

- [1] C. Gutsche, A. Lysov, K. Blekker I. Regolin, W. Prost, and F.-J. Tegude. n-type doping of vapor-liquid-solid-growth gaas nanowires. *Nanoscale Research Letters*, 6:65, 2011. [1](#)
- [2] J. D. Jackson. *Classical Electrodynamics*, volume 1. John Wiley & Sons, Inc, 1962. [2](#)
- [3] Neil W. Ashcroft and N. David Mermin. *Solid State Physics*. Harcourt College Publisher, 1976. [2.1.1](#), [3](#)
- [4] J.P. Srivastava. *Elements of Solid State Physics*. Prentice-Hall of India Pvt.Ltd, 2006. [2.1.2](#), [3](#)
- [5] Thomas Heinzel. *Mesoscopic Electronics in Solid State Nanostructures*, volume 1. WILEY-VCH Verlag GmbH & Co. KGaA, 2007. [2.1.2](#), [2.1.2.1](#), [2.1](#)
- [6] David K. Ferry, Stephen M. Goodnick, and Jonathan Bird. *Transport in Nanostructures*, volume 2. Cambridge University Press, 2009. [2.1.2.1](#)
- [7] Bert Voigtländer. *Scanning Probe Microscopy*. Springer, 2015. [3](#), [3.1](#), [3.2](#), [3.3](#), [9.1.1](#)
- [8] Harald Ibach. *Physics of Surfaces and Interfaces*, volume N.N. Springer, 2006. [3](#)
- [9] Harald Ibach und Hans Lüth. *Festkörperphysik: Einführung in die Grundlagen*. Springer, 2009. [3](#)
- [10] Ch. Kittel. *Einführung in die Festkörperphysik*. Oldenbourg Wissenschaftsverlag GmbH, 2006. [3](#)
- [11] Charles P. Poole Jr and Frank J. Owens. *Introduction to Nanotechnology*. John Willey & Sons, 2000. [3](#)
- [12] Edward L. Wolf. *Nanophysics and Nanotechnology*. Wiley-VCH Verlag GmbH, 2004. [3](#)
- [13] Bharat Bhushan. *Springer Handbook of Nanotechnology*. Springer, 2004. [3](#)

- [14] G. Binnig, H. Rohrer, Ch. Gerber, and E. Weibel. Tunneling through a controllable vacuum gap. *Applied Physics Letters*, 40:178–180, 1982. [3.1](#)
- [15] R. Shankar. *Principles of Quantum Mechanics*. Springer, 2011. [3.1.1](#)
- [16] J. Tersoff and D. R. Hamann. Theory and application for the scanning tunneling microscope. *Physical Review L*, 50:1998, 1983. [3.1.1](#)
- [17] Wikipedia. [3.4](#)
- [18] Markus Blab. *Ladungstransport an Si(111) Oberflächen mit einem Multispitzen-rastertunnelmikroskop*. PhD thesis, RWTH Aachen, 2014. [3.2](#), [4.2](#), [9.1.2](#), [9.1.2](#), [9.1.2.2](#)
- [19] Richard P. Feynman. *QED - The Strange Theory of Light and Matter*, volume 1. Princeton Science Library, 1983. [3.2](#)
- [20] FEI. *Two Lens Electron Column with BDS-200 User's Guide*, FEI Company PN 4035 273 0251-X2. [3.2.1](#), [3.6](#), [9.15](#)
- [21] Merijntje Bronsgeest. *Physics of Schottky Electron Sources: Theory and Optimum Operation*. Pan Stanford, 2013. [3.5](#)
- [22] R. S. Wagner and W. C. Ellis. Vapor-liquid-solid mechanism of single crystal growth. *Applied Physics Letters*, 4:89, 1964. [3.3.1](#)
- [23] C. Gutsche, I. Regolin, K. Blekker, A. Lysov, W. Prostand, and F. J. Tegude. Controllable p-type doping of gaas nanowires during vapor-liquid-solid growth. *Journal of Applied Physics*, 105:024305, 2009. [3.3.1](#), [II](#), [5.5.3](#), [6.1](#)
- [24] T. K. Sharma, A. P. Shah, M. R. Gokhale, C. J. Panchal, and B. M. Arora. Short interval open tube diffusion of zn in gaas at low temperatures. *Semiconductor Science and Technology*, 14:327, 1999. [3.3.1](#)
- [25] C. Thiandoume, V. Sallet, R. Triboulet, and O. Gorochov. Decomposition kinetics of tertiarybutanol and diethylzinc used as precursor sources for the growth of zno. *Journal of Crystal Growth*, 311:1411 to 1415, 2009. [3.3.1](#)
- [26] M. Pristovsek. *Fundamental Growth Processes on Different Gallium Arsenide Surfaces in Metal-Organic Vapor Phase Epitaxy*. PhD thesis, Technische Universität Berlin, 2001. [3.3.1](#)
- [27] Udo Linke, Bert Voigtländer, and H. Stollwerk. Preparation of bead metal singlecrystals by electron beam heating. *American Vacuum Society*, 23(6):1535, 2005. [3.3.2](#), [9.1.2.1](#)
- [28] Kilian Flöhr, Kamil Sladek, H. Yusuf Günel, Mihail Ion Lepsa, Hilde Hardt-degen, Marcus Liebmann, Thomas Schäpers, and Markus Morgenstern. Scanning tunneling microscopy with inas nanowire tips. *Applied Physics Letters*, 101:243101, 2012. [3.4](#)

- [29] Allan J. Melmed. The art and science and other aspects of making sharp tips. *Journal of Vacuum Science & Technology B*, 9:601, 1991. [3.4](#)
- [30] Takhee Lee. *Electronic properties of Au nanoclusters/semiconductor structures with low resistance interfaces*. PhD thesis, Purdue University, 2000. [3.8a](#), [3.4](#)
- [31] J. P. Ibe, P. P. Bey, S. L. Brandow, R. A. Brizzolara, N. A. Burnham, D. P. DiLella, K. P. Lee, C. R. K. Marrian, and R. J. Colton. On the electrochemical etching of tips for scanning tunneling microscopy. *Journal of Vacuum Science and Technology*, A 8:3570–3575, 1990. [3.4](#)
- [32] S.M. Sze and K. N. Kwok. *Physics of Semiconductor Devices*. John Wiley & Sons, 1981. [II](#)
- [33] Stefan Korte, Matthias Steidl, Werner Prost, Vasily Cherepanov, Bert Voigtländer, Weihong Zhao, Peter Kleinschmidt, and Thomas Hannappel. Resistance and dopant profiling along freestanding gaas nanowires. *Applied Physics Letters*, 103:143104, 2013. [II](#), [4](#), [5](#), [5.5](#)
- [34] Stefan Korte. *Ladungstransport durch Graphenschichten und GaAs Nanodrähte untersucht mit einem Multispitzen-Rastertunnelmikroskop*. PhD thesis, Forschungszentrum Jülich & RWTH Aachen, 2013. [II](#), [4](#), [4.2b](#), [4.4](#), [5.32b](#)
- [35] Hannah J. Joyce, Qiang Gao, H. Hoe Tan, Chennupati Jagadish, Yong Kim, Xin Zhang, Yanan Guo, and Jin Zou. Twin-free uniform epitaxial gaas nanowires grown by a two-temperature process. *Nano Letters*, 7:921–926, 2007. [II](#), [5.1](#), [5.1.1](#)
- [36] Evgeniy Zubkov. *Aufbau einer Vierspitzen-Rastertunnelmikroskop/Rasterelektronenmikroskop-Kombination und Leitfähigkeitsmessungen an Silizid Nanodrähten*. PhD thesis, Forschungszentrum Jülich & RWTH Aachen, 2012. [4](#), [4.1](#), [4.4](#), [4.4](#)
- [37] Karl-Heinz Besocke. An easily operable scanning tunneling microscope. *Surface Science Letters*, 181:145–153, 1987. [4.1](#), [9.1.1](#)
- [38] Horst Haensel and Werner Neumann. *Physik*. Spektrum Akademischer Verlag, 1996. [5](#)
- [39] J.R. Waldrop and R.W. Grant. Interface chemistry of metal-gaas schottky-barrier contacts. *Applied Physics Letters*, 34:630, 1979. [5.3.2](#)
- [40] Shadi A. Dayeh, Edward T. Yu, and Deli Wang. Iii-v nanowire growth mechanism: V/iii ratio and temperature effects. *Nano Letters*, 7 (8):2489–2490, 2007. [5.5](#), [5.5.2](#)
- [41] Hannah J. Joyce, Jennifer Wong-Leung, Qiang Gao, H. Hoe Tan, and Chennupati Jagadish. Phase perfection in zinc blende and wurtzite iii-v nanowires using basic growth parameters. *Nano Letters*, 10:908–915, 2010. [5.5.2](#)

- [42] C. Hilsum. Simple empirical relationship between mobility and carrier concentration. *Electronics Letters*, 10:908–915, 1974. [5.5.3](#)
- [43] L. G. Meiners. Surface potential of anodized p-gaas mos capacitors. *Applied Physics Letters*, 33:747, 1978. [5.5.3](#)
- [44] J.J. Mares, J. Kristofik, V. Smid, and J. Zeman. On the d.c. conductivity in semi-insulating gaas. *Solid State Communications*, 60:275–276, 1986. [5.5.5](#)
- [45] W. Prost, K. Blekker, Q. T. Do, I. Regolin, S. Müller, K. Wegener D. Stichtenoth, C. Ronning, and F. J. Tegude. Modeling the carrier mobility in nanowire channel fet. *MRS Proceedings*, 1017, 2007. [6](#)
- [46] Kilian Flöhr, Marcus Liebmann, Kamil Sladek, H. Yusuf Günel, Robert Frielinghaus, Fabian Haas, Carola Meyer, Hilde Hardtdegen, Thomas Schäpers, Detlev Grützmacher, and Markus Morgenstern. Manipulating inas nanowires with submicrometer precision. *Review of Scientific Instruments*, 82:113705, 2011. [6](#)
- [47] R. Dombrowski, Chr. Steinebach, Chr. Wittneven, M. Morgenstern, and R. Wiesendanger. Tip-induced band bending by scanning tunneling spectroscopy of the states of the tip-induced quantum dot on inas(110). *Physical Review B*, 59:8043, 1999. [6.2](#)
- [48] Tomas Bryllert, Lars-Erik Wernersson, Truls Löwgren, and Lars Samuelson. Vertical wrap-gated nanowire transistors. *Nanotechnology*, 17:227, 2006. [III](#)
- [49] Jesper Wallentin, Nicklas Anttu, Damir Asoli, Maria Huffman, Ingvar Aberg, Martin H. Magnusson, Gerald Siefer, Peter Fuss-Kailuweit, Frank Dimroth, Bernd Witzigmann, H. Q. Xu, Lars Samuelson, Knut Deppert, and Magnus T. Borgström. Inp nanowire array solar cells achieving 13.8exceeding the ray optics limit. *Science*, 339:1057–1060, 2013. [III](#), [9.1](#)
- [50] R. A. Wolf and S. Trolier-McKinstry. Temperature dependence of the piezoelectric response in lead zirconate titanate films. *Journal of Applied Physics*, 95(3):1397–1406, 2004. [9.1.1](#)
- [51] F. Kajzar and G. Parette. Magnetic-moment distribution and environmental effects around titanium impurities in nickel and iron matrix. *Physical Review B*, 20, 1979. [9.1.1](#)
- [52] Peter Hidnert. Thermal expansion of titanium. [9.1.1](#)
- [53] G. Binnig, H. Fuchs, Ch. Gerber, H. Rohrer, E. Stoll, and E. Tosatti. Energy-dependent state-density corrugation of a graphite surface as seen by scanning tunneling microscopy. *Europhysics Letters*, Volume 1(Number 1):31, 1986. [9.1.1.1](#)

- [54] V. Cherepanov, E. Zubkov, H. Junker, S. Korte, M. Blab, P. Coenen, and B. Voigtländer. Ultra compact multitip scanning tunneling microscope with a diameter of 50 nm. *Review of Scientific Instruments*, 83:6, 2012. [9.1.2](#)
- [55] <http://www.mprobes.com/koalamulti.html>. <http://www.mprobes.com/koalamulti.html>, 05 2015. [9.6](#), [9.9](#)
- [56] Stefania Carmen Bobaru. *High-Pressure STM Studies of Oxidation Catalysis*. PhD thesis, Leiden University, 2006. [9.1.2.1](#), [9.1.2.1](#)
- [57] M.A. Van Hove, R.J. Koestner, P.C. Stair, Jean-Paul Biberian, L.L. Kesmodel, I. BartoÅi, and G.A. Somorjai. The surface reconstructions of the (100) crystal faces of iridium, platinum and gold: I. experimental observations and possible structural models. *Surface Science Letters*, 103:189–217, 1981. [9.1.2.1](#)
- [58] Alexander M Bittner. The growth of the second underpotentially deposited silver layer on pt(100). *Journal of Electroanalytical Chemistry*, 431:51–56, 1997. [9.1.2.1](#)
- [59] Asawin Sinsarp, Yoichi Yamada, Masahiro Sasaki, and Shigehiko Yamamoto. Local tunneling barrier height imaging of a reconstructed pt(100) surface. *Applied Surface Science*, 237:583–588, 2004. [9.1.2.1](#)
- [60] Paula Havu, Volker Blum, Ville Havu, Patrick Rinke, and Matthias Scheffler. Large-scale surface reconstruction energetics of pt(100) and au(100) by all-electron densityfunctional theory. *PHYSICAL REVIEW B*, 82:161418–1 – 161418–4, 2010. [9.1.2.1](#)
- [61] Martin Scheufens. Eigenschaften eines ultrakompakten rastertunnelmikroskops für messungen im uhv. masters thesis. Master’s thesis, RWTH Aachen, 2013. [9.1.2.2](#)
- [62] Vasily Cherepanov, Peter Coenen, and Bert Voigtländer. A nanopositioner for scanning probe microscopy: The koaladrive. *Review of Scientific Instruments*, 83:023703, 2012. [9.1.3](#)
- [63] K. Uchino. *Advanced Piezoelectric Materials*. Elsevier, 2010. [9.1.3](#)
- [64] Guy K. White. *Experimental Techniques in Low-Temperature Physics*, volume 3. Clarendon Press, 1979. [9.3](#)
- [65] Jack W. Ekin. *Experimental Techniques for low-temperature Measurements*. Oxford University Press, 2006. [9.3](#)
- [66] Hans-Christian Stahl and Siegfried Hunklinger. *Low-Temperature Physics*. Springer Science & Business Media, 2005. [9.3](#)
- [67] *Thermal Management Handbook*. maxim integrated, 2014. [9.3.1](#)