Doctoral Thesis

Combined low-temperature scanning probe microscopy and magneto-transport experiments for the local investigation of mesoscopic systems

Author(s):
Rychen, Jörg

Publication Date:
2001

Permanent Link:
https://doi.org/10.3929/ethz-a-004157885

Rights / License:
In Copyright - Non-Commercial Use Permitted
Combined Low-Temperature Scanning Probe Microscopy and Magneto-Transport Experiments for the Local Investigation of Mesoscopic Systems

A dissertation submitted to the
SWISS FEDERAL INSTITUTE OF TECHNOLOGY ZÜRICH

for the degree of
Doctor of Natural Sciences

presented by

Jörg Rychen
Dipl. Phys. ETH

born December 28, 1971
citizen of Wilderswil (BE)

accepted on the recommendation of

Prof. Dr. K. Ensslin
Prof. Dr. H.-J. Güntherodt
Dr. T. Ihn
# Contents

Abstract iii

Zusammenfassung v

1 Introduction 1
   1.1 Overview of the field of low temperature scanning probe microscopy 1
   1.2 The scope of this work 3

2 Principles of Scanning Probe Microscopy 5
   2.1 Introduction 5
   2.2 Scanning Tunneling Microscopy 6
      2.2.1 Tunneling Spectroscopy 6
      2.2.2 Other STM related techniques 6
   2.3 Scanning Force Microscopy 7
      2.3.1 Different Forces in Scanning Force Microscopy 7
      2.3.2 Deflection Detection Methods 8
   2.4 Dynamic Force Detection 10
      2.4.1 The Oscillator in the Interaction Potential 10
      2.4.2 Monitoring the Oscillator Properties 13
      2.4.3 Controlling the Tip Sample Distance 15
      2.4.4 Fundamental Limits in Dynamic Force Detection 16
   2.5 Kelvin Probe 17

3 Construction of the Scanning Probe Microscope 21
   3.1 Requirements 21
   3.2 The Oxford Cryo-SXM 23
      3.2.1 Overview 23
      3.2.2 Modifications 23
      3.2.3 Problems 25
   3.3 Experimental Platform 27
      3.3.1 Cryostat, Crane and Vibration Isolation 27
      3.3.2 Vacuum Chamber 28
   3.4 Microscope Head 31
      3.4.1 Microscope Casing 31
      3.4.2 XY-Table 32
3.4.3 Sample Holder ............................................ 32

4 Piezoelectric Quartz Tuning Forks ............................................ 35
  4.1 Introduction .................................................. 35
  4.2 Theory of tuning forks .......................................... 36
    4.2.1 Electrical model .......................................... 37
    4.2.2 Electromechanical Coupling .............................. 38
    4.2.3 Mechanical Model ......................................... 39
    4.2.4 Fundamental Limits ...................................... 43
  4.3 Experimental Implementation of Tuning Fork Sensors ......... 44
    4.3.1 Preparation of Tuning Fork Sensors .................... 44
    4.3.2 The Phase Locked Loop .................................. 45
    4.3.3 The Low Temperature Preamplifier ...................... 47

5 Application Examples .................................................. 49
  5.1 Tip Sample Interactions ....................................... 49
    5.1.1 Force versus Distance Curves ........................... 49
    5.1.2 Electrostatic Interactions ............................... 52
  5.2 Topographical Imaging ......................................... 56
    5.2.1 Evaporated Gold Films .................................. 56
    5.2.2 Semiconductor Nanostructures .......................... 58
    5.2.3 Nuclear Pore Complex ................................... 60
  5.3 Scanning Gate Microscopy of a Quantum Wire ................. 61
    5.3.1 Quasi-Ballistic Transport ............................... 61
    5.3.2 Scanning Gate Microscopy on AlGaAs-GaAs Heterostructures .. 63
    5.3.3 Experiment ................................................. 66
    5.3.4 Conclusions and Outlook .................................. 70
  5.4 Imaging the Inter Edge State Tunneling ...................... 71
    5.4.1 Quantum Hall Effect and Edge States ................... 71
    5.4.2 Sample ..................................................... 72
    5.4.3 Finding the Working Point ............................... 73
    5.4.4 Scanning Gate Microscopy of the Inter Edge State Tunneling .. 74
    5.4.5 Conclusions and Outlook .................................. 75

List of Abbreviations .................................................. 76

Publications ......................................................... 78

Bibliography .......................................................... 78

Acknowledgments .......................................................... 89

Curriculum Vitae .......................................................... 91
Abstract

Mesoscopic systems have successfully been realized in semiconductor nanostructures by various technological means. Over the last ten years there has been a wealth of new results in this novel regime, where electron transport can be described by ballistic and phase coherent phenomena. Practically all experiments so far have been performed by measuring currents and voltages via macroscopic contacts attached to the mesoscopic device. The understanding of local properties such as potential landscapes, quantum mechanical wave functions or electron motion is primarily based on indirect experimental evidence. This is were the focus of this PhD thesis sets in. The goal is to have a local experimental probe for semiconductor nanostructures and to develop experimental skills that exploit the potential of scanning probe techniques for the better local understanding of mesoscopic devices and quantum Hall systems.

The two main techniques in nanotcchnology, the scanning probe microscopy and the semiconductor processing technology shall be combined with low temperature techniques to study quantum mechanical devices.

First, an introductory chapter will give a historical overview of the field of low temperature scanning probe microscopy. The dynamic force detection, widely used to control the probe-sample spacing is explained in the second chapter. It is an example of a measurement of a physical quantity by conversion to a frequency. Of all measurement quantities, the frequency represents the one that can be determined with the highest degree of accuracy. This allows to detect the tiny interaction forces on the atomic level between the tip and the sample surface.

The difficulties for the construction of the low temperature scanning probe microscope are mainly the thermal contraction of the materials, the vibrations in the cryogenic environment, and the inaccessibility of the microscope in the cryostat. A solution to these problems is given in the third chapter where the construction of the microscope and the experimental setup are presented. The microscope is placed in a small vacuum chamber which can be cooled in a $^4$He cryostat down to 1.7 K and exposed to a magnetic fields of up to 8 T. Coarse positioning is implemented with piezo actuated slip-stick mechanisms and allows to position the probe in all three directions on the sample even at low temperatures.

The sensors used in this work for dynamic force detection are based on piezoelectric quartz tuning forks which stand out for the high and robust quality factors. The drop of the quality factor due to any asymmetries of the two prongs of a tuning fork is explained with a mechanical model consisting of three masses coupled by springs. The electrical properties of the quartz resonator can be modeled with high accuracy with an electrical equivalent circuit. With a digital phase locked loop the resonance frequency can be monitored with high resolution. The piezo-electromechanical coupling has been calibrated with an optical
interferometer. The fundamental limits of the dynamic force detection with quartz tuning forks are given and their experimental feasibility is discussed.

Force-distance curves of a metallic tip on a graphite sample, show the advantages of the tuning fork sensor. The stiffness is high enough to avoid a snap into contact, allowing the operation with oscillation amplitudes below 1 nm. Since the purpose of the developed microscope is to investigate electronic structures that are typically buried a few tens of nanometer below the surface, the electrostatic interaction of the tip and the sample is of special interest. The tip-sample capacitance, which determines the electrostatic interaction, is measured in various ways, and the electrostatic force is then used to calibrate the tuning fork for the force detection. Then, on the basis of topographical imaging of various surfaces, the operation of the sensor, the phase locked loop and the tip-sample distance feedback controller is discussed.

Finally, on the basis of two application examples the possibilities of scanning gate techniques on semiconductor heterostructures is demonstrated: first measurements on a ballistic quantum wire show that the electron transport in mesoscopic systems can be investigated be using the tip as a local gate. Further, the edge channels of the quantum Hall effect were visualized with a special sample and the further investigation is discussed.
Zusammenfassung


Die zwei wichtigsten Techniken der Nanotechnologie, die Rastersensormikroskopie und die Halbleitertechnologie, zusammen mit der Tieftemperaturtechnologie, erlauben es, quantenmechanische Bauelemente zu studieren.


Die Schwierigkeiten ein Tieftemperatur-Rastersensormikroskop zu bauen sind hauptsächlich die thermische Kontraktion der Materialien, die Vibrationen der Tieftemperaturumgebung und die Unzugänglichkeit des Mikroskops im Kryostaten. Eine Lösung zu diesen Problemen wird im dritten Kapitel gegeben, wo die Konstruktion des Mikroskops und der experimentelle Aufbau beschrieben werden. Das Mikroskop befindet sich in einer kleinen Vakuumkammer, die in einem ⁴He-Kryostaten auf bis zu 1.7K abgekühlt und einem Magnetfeld bis 8T ausgesetzt werden kann. Die Grobpositionierung des Sensors wird mit piezo getriebenen „slip-stick“ Mechanismen implementiert, die eine Positionierung in allen drei Raumrichtungen sogar bei tiefen Temperaturen erlauben.

Die in dieser Arbeit verwendeten Sensoren für die dynamische Kraftdetektion basieren auf piezoelektrischen Quartzstimmgabeln, die sich durch einen hohen und robusten Gütefaktor auszeichnen. Die Abnahme des Gütefaktors auf Grund einer Asymmetrie der zwei Stimmgabelarme wird anhand eines mechanischen Modells erklärt, das aus drei Massen besteht, die mit Federn miteinander gekoppelt sind. Die elektrischen Eigenschaften des Quartzresonators lassen sich sehr genau mit einem elektronischen Ersatzschaltbild
modellieren. Mit einer digitalen „Phase-Locked-Loop“ kann die Resonanzfrequenz mit ho- 
her Auflösung verfolgt werden. Die piezo-elektrische Kopplung wurde mit einem 
optischen Interferometer kalibriert. Die fundamentalen Grenzen der dynamischen Kraftde-
tektion mit Quartzstimmgabeln werden berechnet und deren experimentelle Erreichbarkeit 
will diskutiert.

Kraft-Abstands Kurven einer metallischen Spitze auf Graphit, zeigen die Vorteile des 
Stimmgabelsensors, der sich als steif genug erweist, um ein Einschnappen der Spitze zu 
verhindern. Es ist daher möglich, die Stimmgabel mit Amplituden unter 1 nm zu betreiben.

Von speziellem Interesse ist die elektrostatische Wechselwirkung der Spitze mit der 
Probe, weil mit dem entwickelten Mikroskop elektronische Strukturen untersucht werden 
sollen, die einige zehn Nanometer unter der Oberfläche liegen. Die Kapazität zwischen 
Spitze und Probe, welche diese elektrostatische Wechselwirkung bestimmt, wird mit ver-
schiedenen Methoden gemessen und die elektrostatische Kraft wird dann benutzt, um 
die Stimmgabel für die Kraftdetektion zu eichen. Anhand der topographischen Abbildung 
von verschiedenen Oberflächen wird dann der Betrieb der Stimmgabel, der „Phase-Locked-
Loop“ und der Abstandsregelung diskutiert.

Schließlich wird anhand zweier Anwendungsbeispiele die Möglichkeiten der „Scanning 
Gate“-Technik auf Halbleiter Heterostrukturen gezeigt: Erste Messungen an einem balli-
stischen Quantendraht zeigen, dass der Elektronentransport in mesoskopischen Systemen 
untersucht werden kann, indem man die Spitze als lokales Gate benutzt. Weiter wurden 
mit einer speziellen Probe, die Randkanäle im Quantenhalleffekt sichtbar gemacht was 
deren weitere Untersuchung in Aussicht stellt.
Chapter 1

Introduction

Binnig and Rohrer’s Nobel prize awarded invention of the scanning tunneling microscope (STM) [1] represents a milestone in condensed matter physics: tunneling microscopy was the first technique to allow us to directly probe and image systems on the scale of individual atoms. The intellectual excitement surrounding this new instrument quickly inspired a wide array of new experimental techniques, collectively known as scanned probe microscopes (SPM). Among the most widely applied of these new local probes is the technique of atomic force microscopy (AFM), developed by Binnig and Quate [2]. As an eye and a hand to the nanometer sized world, the scanning probe microscopes became the central tool in nanotechnology, one of today’s most important field of research.

Operation of these microscopes at low temperatures enhances both the resolution in tunneling- and force-spectroscopy and the general stability of the microscope. The realization of a low temperature scanning probe microscope (LTSPM) is still a challenge and some examples will be given in the following.

1.1 Overview of the field of low temperature scanning probe microscopy

The first successfully working low temperature scanning force microscope (LTSFM) built by Giessibl et al. [3], was equipped with a tunneling tip on the backside of a cantilever as deflection sensor.

One of the technically most impressive implementations of a LTSFM is the one built by Hug et al. [4]. It works in ultra high vacuum (UHV) at temperatures down to 6K, employs a fiber optical interferometer and shows very high resolution for atomic imaging [5]. Using this microscope for magnetic force microscopy [6], it was possible to image and study single vortices in superconductors [7].

A LTSFM with similar capabilities has been built in the group of Wiesendanger by Allers et al. [8, 9]. They presented scanning tunneling spectroscopy experiments on InAs, for example the scattering states of ionized dopants in InAs [10], or the local density of states of the three-dimensional conductor in the extreme quantum limit [11] and the tunneling spectroscopy of quantum dots, that were electrostatically induced by the tip [12].
While such machines demonstrate what is possible on the microscopic scale, a great deal of current interest centers on physics of so-called mesoscopic devices. These devices are on a length scale where the quantum mechanical description replaces the classical description.

Carbon nanotubes are very interesting objects in mesoscopic physics. Their transport properties have been investigated by SPM in ref. [13, 14].

Karrai et al. developed a scanning near field optical microscope (SNOM) for optical spectroscopy investigation of semiconductor nanostructures at low temperatures. The force detection is based on quartz tuning forks [15]. In ref. [16] they report on the investigation and manipulation of single quantum dots. In another group, the electron wave function in self assembled quantum dots could be imaged at low temperature with a scanning tunneling microscope [17].

Mesoscopic devices based on AlGaAs-GaAs heterostructures are ideal candidates to investigate the quantum mechanical electron system. The electron system is confined to the atomically sharp and clean interface of the two alloys where the electrons have a long mean free path and behave two-dimensional. The two-dimensional electron system (2DES) has a low density ($10^{15} \text{m}^{-2}$) and therefore the Fermi wave length is up to 50 nm. Furthermore the effective mass in GaAs ($m^* = 0.068 m_e$) is very low and therefore the electrons have a high mobility (up to $\mu = 100 \text{m}^2/\text{Vs}$). This leads to interesting devices like quantum point contacts, quantum wires, antidot lattices, Aharanov-Bohm rings and much more [18, 19].

The group around Westervelt developed a GaAs/AlGaAs self sensing cantilever for the low temperature scanning probe microscopy [20]. In ref. [21], the scanning gate microscopy of the electron transport through a ballistic point contact has been published. Later, in ref. [22], the coherent electron flow in a quantum point contact with tunable number of transversal modes could be imaged for the first time.

Quantum wires defined on AlGaAs heterostructures by wet chemical etching were investigated with the ballistic electron emission microscope (BEEM) by Smoliner et al. [23, 24]. The microscope operates in He-gas at temperatures down to 1.5 K.

Whenever single energy levels are resolved by the experiment, their shift in magnetic field is an important way to proof the theoretical models. The properties of the two-dimensional electron gas (2DEG) in high magnetic field is of great interest since the discovery of the quantum Hall effect (QHE) by Klitzing et al. [25], who received the Nobel prize for this work. According to the theory the current flows along the so-called edge channels [26, 27]. A vast number of electrical transport experiments concerning the QHE have been performed but only few experiments address the spatial current distribution. This is a field predominant for the use of LTSPM's:

The subsurface charge accumulation technique developed by Ashoori et al. was used to image the incompressible strips in the quantum Hall liquid [28]. The microscope operates in liquid $^3\text{He}$ and does not have any force sensor but a tunneling tip with a ultra sensitive charge detector. In another work they mapped the potential topography of a quantum Hall liquid using a few electron bubble induced by the tip [29].

One of the most impressing work on semiconductor structures with LTSPM is the scanning single electron transistor experiments on a AlGaAs heterostructure [30]. The SET is fabricated at the end of a sharp glass tip and it detects the electrostatic interactions...
1.2. THE SCOPE OF THIS WORK

between the tip and the sample. Individual ionized charge sites and fluctuations in the dopant and surface-charge could be imaged with a resolution of 100nm. In ref. [31] the quantum Hall liquid was imaged with this technique.

The group around McEuen developed a LTSPM employing piezoresistive cantilevers to investigate the edge and bulk currents in the quantum Hall regime [32]. In an unpublished paper they also present the imaging of single inter-edge state scattering centers in the quantum hall regime [33].

Weitz et al. investigated the Hall-potential profile of a two dimensional electron system under quantum Hall conditions with submicron resolution [34]. They developed a scanning force microscope operating at 1.4K and used piezoresistive cantilevers [35, 36].

The two dimensional electron system (2DES) itself is a fertile topic of research, as e.g. the metal-insulator-transition observed at low temperatures is still an open question. The transport properties of 2DES at ultra low temperatures (< 1K) demonstrate unusual effects arising from quantum mechanical phase coherence of the carriers, e.g., the universal conductance fluctuations, the weak localization and Aharonov-Bohm oscillations observed in small wires and rings. Phase coherent electronics is a new and very exciting field due to the prospect of the realization of quantum computers. However, for the phase coherence of the electrons very low temperatures are needed and only a few LTSPM direct to temperatures below liquid $^4$He (1.5K):

Nunes et al. [37, 38] report on a LTSPM for the investigation of mesoscopic devices at millikelvin temperatures. The microscope is mounted in $^3$He-$^4$He dilution refrigerator and employs a fiber optical interferometer for the cantilever deflection detection.

Pan et al. reported in a paper on a STM that operates in a sorption-pumped $^3$He cryostat [39]. Tunneling microscopy is most useful if both the tip and sample are prepared in UHV. The task of combining millikelvin refrigeration techniques with UHV needs a UHV transfer path from room temperature of the preparation of the sample and tip in the low temperature environment which is intrinsically UHV. Another tunneling microscope for the operation in a dilution refrigerator is described in [40].

1.2 The scope of this work

The main purpose of the microscope, the construction of which is reported in this work, is to investigate semiconductor nanostructures. Usually they are investigated by transport experiments at low temperatures and high magnetic fields but with a scanning probe microscope these transport experiments can be complemented to gain local information about the electronic processes inside the sample. The microscope will work at liquid $^4$He temperatures down to 1.7 K and in high magnetic field up to 8T and allows for advanced in situ transport experiments. At this temperature phase coherence phenomena are not very pronounced but the mean free path is only limited by impurities, allowing experiments addressing ballistic transport. However, the quantum Hall effect is easily achieved at these temperatures and the investigation of the edge channels, which show phase coherence over macroscopic length scales, will be a main purpose of the instrument.

A quartz tuning fork with a metallic tip will be used as the force sensor since it works well at low temperatures and in high magnetic fields. Furthermore, the metallic tip is suitable for the electrical interactions with the sample. On one side the tip can locally
influence the electron transport in the sample; key phrases are:

- Scanning gate microscopy
- Permanent electrostatic charge of the surface
- Polarization of a ferroelectric layer
- Mechanical modifications: scratching
- Deposition of material from the tip onto the surface
- Local anodic oxidation of the surface

On the other side the local properties of the sample and the response to electron transport can be investigated locally:

- Scanning Kelvin Probe Microscopy
- Magnetic Force Microscopy
- Scanning Capacitance Spectroscopy
- Subsurface Charge Accumulation Microscopy
- Scanning Dissipation Microscopy
- Scanning FET, SET, SQUID, etc.

Figure 1.1: Scanning probe techniques to complement the traditional transport experiments.
Chapter 2

Principles of Scanning Probe Microscopy

2.1 Introduction

For a long time the technique of sensing a surface has been used for the playback of music. The gramophone has a small sharp needle mounted on a spring and senses the surface topography of tracks on the vinyl discs. The topography is a direct mapping of the pressure variations which account for the music. Also blind people use their sensitive fingers to scan papers with small humps, they read the embossed printing. The techniques of sensing a surface is also important in the industry where it is used for profilometry for example in the quality control of balls for ball bearings. In 1981, Binnig and Rohrer and their colleagues at the Zürich Research Laboratory of IBM developed a Scanning Tunneling Microscope (STM) which pushed this technique to the limits: it became possible to observe individual atoms. Two key elements are necessary for this technique: A precise and fine positioning facility, which is usually implemented by piezoelectric actuators, and a fine sensing method to feel the surface, which was the measurement of the tunneling current in this pioneering work. There were also technical problems they had to solve as for example the vibration isolation, the production of sharp, eventually atomically sharp tips, the feedback controlling electronics and also the ultra high vacuum (UHV) environment which makes the experiment to a "remote" experiment. G. Binnig and H. Rohrer were awarded the Noble Prize in physics in 1986 for their outstanding contribution to science. The successful achievement initiated a surge of research and engineering activity. While the positioning facility remained the same except for a few special applications, the sensing method was quickly developed into a manifold of different techniques. One major advance was the ability to sense the forces between the tip and the surface: the scanning force microscope (SFM). The restriction to conductive samples dropped out and in recent years atomic resolution imaging and manipulation became possible on all kinds of surfaces. Even though the atomic topography of a surface is a matter of particular interest, it is the ability to sense also other local properties which makes the scanning probe microscopes so attractive. Here are first the properties that are directly related to the sensing method as for example the local density of states in STM or the elastic and adhesion properties of the surface in SFM. The magnetic and electrical forces as well as chemical forces are also
directly accessible by SFM. There is also a vast activity in trying to bring other sensors into play. One prominent example is the Scanning Near Field Optical Microscope (SNOM) in which the near field of an illuminated surface is collected by a sharp tapered waveguide. Other examples employ Superconducting Quantum Interference Devices (SQUIDs), Hall Bar Magnetometers or Single Electron Transistors (SETs).

2.2 Scanning Tunneling Microscopy

As mentioned above, scanning tunneling microscopy is the first of the scanning probe techniques that was realized and is in many aspects also the simplest. It employs the fact that the electrons can tunnel from the tip to sample even before there is a mechanical contact. This tunneling current is the quantity that is controlled by the distance controller. Experimentally the sensor consists of a sharp tip, mainly etched Tungsten- or Platinum-Iridium wires and a current measurement device. Since the tunneling current occurs when the tip is closer then approximately 0.5nm and rises exponentially with decreasing gap, STM is very sensitive to vibrations. The tunneling currents are typically of the order of pA to nA and the feedback controlling should have a bandwidth of about 1 - 100kHz. STM has the advantage that the shape of the tip does not play such an important role because the tunneling current will automatically flow through the foremost atom as a result of the exponential law for the tunneling current. The major disadvantage of STM is that it is limited to conductive samples.

2.2.1 Tunneling Spectroscopy

To have a model for the tunneling current a planar tunneling geometry between tip and sample is assumed. An external voltage $V_t$ is applied between tip and sample separated by a vacuum gap of width $d$. For zero applied bias the height of the barrier $\phi$ is taken as the average of the work functions of tip and sample. In a one dimensional WKB-approximation the current from the tip to the sample would depend on the density of states of both materials and the transmission probability through the barrier:

\[
I_t \approx \int_{E_F}^{E_F+eV_t} dE \rho(E + eV_t) \rho_s(E) \exp\left(-\frac{\hbar}{m} \sqrt{\frac{\phi - eV_t}{2} - (E - E_F)d}\right)
\]  (2.1)

The energy $E$ is measured with respect to the sample Fermi-level $E_F$ and the temperature was chosen to be $T = 0K$. The dispersion relation of the electron in the vacuum gap was assumed to be free-electron-like and characterized by a mass $m$. Equation (2.1) says that for small tip bias the tunneling current is proportional to the integral of the local density of states (LDOS) $\rho_k(r_0, E_F)$ of the sample at the position of the tip. Thus the local density of states can be measured with a differentiation of the current-voltage curve. An important application example is the investigation of superconductors where the gap in the density of states can be measured and the vortices can be imaged.

2.2.2 Other STM related techniques

With some modifications the classical STM can be extended to give access to other surface properties. The scanning Josephson junction microscope is able to image the local mag-
2.3. SCANNING FORCE MICROSCOPY

Netic field of a surface by the modulation of the critical current of a Josephson junction scanned over the sample with a STM feedback [41]. Tunneling of spin polarized electrons gives also insight into magnetic properties of the sample [42]. Quantitative imaging of the dielectric permittivity was demonstrated with a scanning near field microwave microscope [43] and nonlinear microwave frequency mixing was used for the distance control with atomic resolution [44]. The ballistic electron emission microscopy (BEEM) has been introduced for the investigation of buried interfaces [45]. An overview of different techniques based on the interaction of the tunneling current with phonons and photons was presented in [46].

2.3 Scanning Force Microscopy

One alternative to sense the surface via the tunneling current is to detect the forces between the tip and the surface. Technically this is much harder to realize than the detection of the current.

For a small distance \( d \), this force is repulsive \( (d < 1 \text{ nm}) \) and its intensity is about \( 10^{-9} \text{ N} \). For larger distances, the force is attractive and is weaker \( (10^{-12} \text{ N}) \). The corresponding pressures are of the order of mega Pascal depending on the geometry of the tip. The forces are either volume forces or surface forces and only in the case of the chemical forces they are very local. Therefore the forces depend strongly on the tip shape and atomic resolution is much harder than with STM. In the literature the tip-sample interactions are well described and in the following an overview of the different forces is given.

2.3.1 Different Forces in Scanning Force Microscopy

Van der Waals Forces

Van der Waals forces are short range attractive forces attributed to the dipolar interaction between the atoms or molecules of both materials constituting the tip and the surface. The van der Waals dipolar interaction \( U_{vdW} \) between two atoms or molecules can be written as

\[
U_{vdW}(d) = -\frac{A}{d^6},
\]

(2.2)

where \( A \) is a constant and \( d \) is the distance between the two considered atoms. An integration over the volumes of the two interacting bodies gives the total interaction potential \([47, 48, 49, 50, 51]\).... potential with bond Energy \( E_{\text{bond}} \) and equilibrium distance \( \sigma \) \([47, 48, 49, 51, 52]\).

Chemical Forces

When the tip is close enough to the surface that the valence electrons of the closed shells overlap, a repulsive force due to the Pauli exclusion principle arises. When the atoms do not have closed shells at their surfaces chemical bindings between the tip and the surface can occur at close distances. For tip sample spacings smaller then the interatomic distances \( d < 0.5 \text{ nm} \) the interaction potential is modeled by a Lennard-Jones potential with bond Energy \( E_{\text{bond}} \) and equilibrium distance \( \sigma \) \([47, 48, 49, 51, 52]\).

\[
U_{1,4}(d) = -E_{\text{bond}} \left[ 2 \left( \frac{\sigma}{d} \right)^6 - \left( \frac{\sigma}{d} \right)^{12} \right]
\]

(2.3)
CHAPTER 2. PRINCIPLES OF SCANNING PROBE MICROSCOPY

Capillary Forces

Except for low temperature -, ultra high vacuum - or in liquid environments, surfaces are always covered by a water film as long as they are not hydrophobic and a liquid bridge will be formed between the tip and the surface causing a strong attractive force [47, 48]. In addition to the attractive force the water film causes a damping for any oscillating probes [53] which can be advantageous for safe distance control. The waterlayer on the surface is also important for the technique of local oxidation by a scanning probe [54].

Electrostatic Forces

Electrostatic forces are given in principle by the Coulomb law. Coulomb forces generally can be attractive and repulsive but because in most cases at least the probe or the sample is conductive, the forces are usually attractive since an image charge with opposite sign is build up on the conductive side. In this case the forces are given by the geometrical arrangement of the probe and the sample which is reflected in the capacitance $C(d)$ as a function of the spacing $d$ [55, 49].

$$U_{\text{elec}}(d) = \frac{1}{2} C(d) V^2$$  \hspace{1cm} (2.4)

Here $V$ is the electrostatic potential. In sec. 2.5 a detailed analysis of the electrostatic interactions is given.

Magnetic Forces

With a magnetic probe it is possible to sense the magnetic interactions with the sample. These are namely the magnetic stray fields due to magnetization [6] or currents and for the case of oscillating probes eddy currents which can cause a damping. Magnetic Force Microscopy (MFM) became an own branch of the SPM techniques [56] since it is important for the investigation of magnetic thin films for magnetic recording [57], for the investigation of vortices in superconductors [7] and possibly for single spin resonance experiments which are promising for quantum computer applications.

Dissipative Forces

Additional to the forces acting statically on the probe there is also friction when the probe is moved, i.e. scanned over the surface. The origin of friction on a nanometer scale became an own field of interest, nanotribology, because of its technical importance. With a laser deflection detection mechanism the lateral forces are easily recordable and with any oscillating probe the damping of the oscillation is a measure for the friction [58, 59]. The viscosity of the liquid covering the surface, the direct emission of phonons, and an imaginary part of the dielectric constant are origins of the dissipation.

2.3.2 Deflection Detection Methods

Micromechanical techniques made it possible to detect the small forces discussed in the previous section (2.3.1). The first principle (static mode) is based on the conversion of the
force into a deflection of an appropriate spring and to measure then the deflection. The second principle (dynamic mode) is based on the influence of the tip sample interaction forces on the resonance characteristics of an appropriate oscillator. Both principles need the detection of the deflection of a lever or generally an oscillator. Some different methods for the deflection detection are discussed in the following.

Detection with a Tunneling Junction

Historically this was the first idea to measure the deflection of a cantilever. A tunneling tip on the conductive backside of a cantilever is used to measure its deflection. Although very accurate this technique lacks of friends due to some technical problems: changes of the tunneling tip and contamination of the cantilever backside during operation which shifts the zero position, resonances of the cantilever which are usually higher than the feedback bandwidth of the tunneling system, attractive van der Waals forces can lead to a "snap into contact"of the cantilever and tunneling tip and finally there is also the problem of the coarse positioning of the tunneling tip.

Laserbeam deflection

The deflection of a laserbeam on the backside of a cantilever allows to measure its deflection very precisely. Most of the commercially available SFM are equipped with such a detection. However, the focusing and alignment of the laserbeam has to be controlled optically and its implementation in a cryogenic environment is therefore cumbersome.

Interferometric Detection

The interference of the light reflected from the cantilever with that reflected from the end of a fiber, which is mounted very close to the backside of the cantilever, is used to detect the motion of the cantilever [60]. This technique is very precise and is successfully applied in several LTSPM's [61, 38, 8]. Because light can scatter onto the sample under investigation, this method is not suitable for the investigation of semiconductor heterostructures.

Capacitive Detection

The motion of the cantilever is driven and sensed electrostatically via the capacitance of the cantilever-backside and a fixed electrode. The equivalent electrical circuit is the same as for an piezoelectric resonator and is shown in figure 4.2. In ref. [62] a detailed analysis of this technique is given. This method is principally handsome for use in a cryogenic environment but the signal of the cantilever is so small compared to the capacitive background signal, that the long distance between the cantilever and the detection electronics becomes a problem. In conjunction with integrated low temperature electronics, this technique could be very powerful.

Piezoresistive Detection

The change of the resistance of a resistive path on the backside of the cantilever is a measure of its deflection [20]. Such cantilevers are commercially available and this technique is also
successively implemented in LTSPM's [63, 64, 56, 35, 21]. However, the relative changes in the resistance are of the order of $10^{-6}$ which demands for good detection electronics and the heat production due to dissipation in the cantilever requires low measurement currents. For well defined electrostatic interactions this cantilevers have to be metallized for example with Iridium.

**Piezoelectric Detection**

The piezoelectric effect transforms mechanical stress into electrical charge separation and vice versa. This detection method is well suited for SFM because the oscillator can be driven and detected by electrical contacts and it is very accurate. There are only a few applications of the piezoelectric detection method [65, 66, 67, 68] because the optically detected cantilevers are well established. Crystalline Quartz, the material with low internal mechanical losses, GaAs, the material for which advanced technology exists, and ZnO a material with a high piezoelectric constant, are the main candidates for such applications. An additional advantage of the piezoelectric detection method compared to the others is the possibility to measure the dissipation power very easily and accurately. In the future, as soon as such sensors are commercially available, many SFM's and specially LTSPM's will be equipped with such a detection. Piezoelectric quartz tuning forks are the choice for the presented work and will be described in detail in section 4.

**2.4 Dynamic Force Detection**

As mentioned above there is an alternative to the static mode where the deflection of a cantilever is measured to detect the force between the probe and the sample: In the dynamic force detection the influence of the probe sample interactions on the dynamics of an oscillating probe is monitored. In contrast to the literature, where the term "dynamic mode" is used for a frequency modulation technique, in this work it is referred to all techniques where an oscillating probe is employed.

In dynamic force detection very sensitive Lock-in techniques can be applied and the problems with dc-drifts are eliminated. Of all measurement quantities, the frequency represents the one that can be determined with the highest degree of accuracy. This also allows precise measurement of other physical and technical quantities whenever they could be put down to a frequency measurement.

**2.4.1 The Oscillator in the Interaction Potential**

Any mechanical oscillator can be used in principle to detect the interaction forces of a tip attached to the oscillator and the surface. The following analysis is restricted to the case where the tip oscillates perpendicular to the surface. The motion of the tip $z(t)$ is described by a harmonic oscillator with an effective mass $m$ and an effective spring constant $k$:

$$m \ddot{z} = -k(z - z_0) - \gamma \dot{z} + F_{\text{int}}(z) + F_{\text{exc}}(t)$$

(2.5)

Where $z_0$ is the equilibrium position and $\gamma$ is the damping constant. The force due to the interactions with the sample is denoted by $F_{\text{int}}$ and the force due to the external excitation
by $F_{\text{exc}}$. For a first analysis the assumption is made that the excitation compensates the damping exactly at every time and the system is therefore a conservative system that can be analyzed by the intuitive Hamilton formalism:

$$H = \frac{p^2}{2m} + \frac{m\omega^2}{2} (x - z_0)^2 + U_{\text{int}}(z)$$ (2.6)

Here, $p$ is the momentum of the oscillator, $\omega = \sqrt{k/m}$ is the resonance frequency of the undisturbed oscillator and $U_{\text{int}}(z) = -\int_{z_0}^z F_{\text{int}}(z)dz$ is the interaction potential. For the case that a constant external force $f$ acts on the oscillator ($U_{\text{int}} = f z$), only its mean position is deflected. If the external force varies linearly with the position ($U_{\text{int}} = k_{\text{int}} z^2$), the resonance frequency is altered to

$$\omega_0 = \sqrt{\frac{k + k_{\text{int}}}{m}}$$ (2.7)

and for small $k_{\text{int}}$, an expansion of (2.7) results in

$$\Delta \omega = \frac{1}{2k} \omega_0 k_{\text{int}}.$$ (2.8)

The application of equation (2.8) principally allows to determine the force gradient from the measured frequency shift as long as the force gradient is constant over the orbit of the probe.

Figure 2.1 shows a contour line map of the Hamilton function (2.6) for a typical cantilever and a tuning fork interacting with a Lennard-Jones potential. The trajectories in phase space are equal to the contour lines of $H$ and the period of a closed trajectory is given by

$$T(E) = \frac{\sqrt{2m}}{\omega_0} \int_{z_1(E)}^{z_2(E)} \frac{dx}{\sqrt{E - H(0, x)}},$$ (2.9)

with $z_{1,2}(E)$ being the turning points of the trajectory with energy $E$ [69]. The frequency is then $\omega_0 = 2\pi/T(E)$ and is easily calculated by numerical integration of equation (2.9).

Important in this context is how the dynamics of the oscillator is altered when it is, for example with a fixed energy or fixed amplitude, approached to the surface as it can be done experimentally (see 5.1.1). The inverse problem, to extract the interaction potential from the oscillator properties as a function of the tip sample distance, is more complicated and is addressed in [69] and for SFM specially in [70, 50, 58].

In Figure 2.1(c),(d) the trajectories are increasingly deformed from the ellipse with increasing energy. It is clearly shown that the motion of the tip becomes anharmonic as soon as the probe enters the interaction potential of the surface. An important point to note is that the amplitude doesn’t rise with $\sqrt{E}$ as expected for a harmonic oscillator but is limited by the hard repulsive potential of the surface. This is experimentally relevant since the amplitude is detected and any change is generally but not correctly attributed to a change in dissipation.

A second point to note is that the motion of the oscillator is usually detected with a lock-in amplifier and thus only the amplitude of the first harmonic of the motion is measured. Higher harmonics are just neglected with this method but play a role for
the excitation of higher flexural modes which are important for the dynamics of a real cantilever [71]. Since the higher harmonics and higher flexural modes are not detected, not all the motional energy is visible and an increased dissipation can be feign.

The trajectories in phase space show that there is an instability for the cantilever when for a given energy two closed contours exist. This problem is called "snap into contact" and prohibits the operation of cantilever oscillators close to the surface and with small amplitudes. For the tuning fork the spring constant is always large compared to the force gradient of the surface potential and the tip won't snap into contact. It can therefore be operated at small amplitudes and close to the surface.

For the legitimation of this easy Hamilton formalism we made the assumption that the excitation compensates exactly the damping at every time as it is possible for the free damped harmonic oscillator with a harmonic excitation. But since the dissipation will

\[
\begin{align*}
\text{Tuning Fork} & : & k &= 28132 \text{ N/m} \\
& & m &= 663.8 \mu g \\
& & \omega_0/2\pi &= 32.766 \text{ kHz} \\
& & z_0 &= 1 \text{ nm} \\
\text{Cantilever} & : & k &= 50 \text{ N/m} \\
& & m &= 31.7 \text{ ng} \\
& & \omega_0/2\pi &= 200 \text{ kHz} \\
& & z_0 &= 10 \text{ nm}
\end{align*}
\]

\begin{figure}
\centering
\begin{tabular}{cc}
\includegraphics[width=0.4\textwidth]{figure2_1a} & \includegraphics[width=0.4\textwidth]{figure2_1b} \\
\includegraphics[width=0.4\textwidth]{figure2_1c} & \includegraphics[width=0.4\textwidth]{figure2_1d}
\end{tabular}
\caption{(a),(b) Lennard Jones interaction potential (2.3) with $E_{\text{bond}} = 3.12$ keV and $\sigma = 0.5$ nm and the harmonic oscillator potential for a tuning fork and a cantilever respectively. (c),(d): Trajectories in the phase space for different energies.}
\end{figure}
2.4. **DYNAMIC FORCE DETECTION**

be highly anharmonic in the interaction, this assumption can't be fulfilled anymore and a numerical integration of the equation of motion (2.5) is necessary and the system may show chaotic behavior.

### 2.4.2 Monitoring the Oscillator Properties

In the last section the theoretical modeling of a mechanical oscillator interacting with a sample surface was given. Now the experimental detection of the varying oscillator properties are discussed.

Experimentally, the most complete information is obtained by recording the full transfer function in the frequency domain. This is either done with a Fast Fourier Transform (FFT) of the time domain response, or by a sequential measurement of the response at the different frequencies with a lock-in amplifier. For a harmonic oscillator the transfer function is a Lorentzian resonance curve as shown in figure 2.2. As already discussed in the previous section the anharmonic interaction leads to higher harmonics which will show up in the transfer function as additional Lorentzian shaped peaks at integer multiples of the resonance frequency. However, experimentally only the first peak is taken into account for the sake of simplicity.

The Lorentz curve is sufficiently described by the resonance frequency $f_0$ and the quality factor $Q$. These two parameters can be influenced by a tip sample interaction: an alteration of the quality factor $Q$ is attributed to a dissipative interaction and lowers and broadens the resonance curve, and an alteration of the resonance frequency $f_0$ is attributed to a conservative interaction and shifts the peak position of the resonance curve.

In addition to the parameters for the Lorentz curve one more parameter is needed to determine the energy of the system: either the mass or the spring constant. In a typical SPM application the mass of the oscillator will remain constant and is not altered by an interaction with the sample\(^1\).

Technically it is difficult to record the full response in the frequency domain with a high enough repetition rate in order to allow for a z-distance controller to control the probe sample spacing. Therefore the response is detected at one frequency for example with a lock-in amplifier. Three detection schemes that are commonly distinguished are discussed in the following: The "Amplitude Detection" where the amplitude of the oscillator response at a fixed frequency is monitored, the "Phase Detection" where the phase of the oscillator signal relative to the fixed driving signal is monitored and the "Frequency-Detection" where the excitation frequency follows the momentary resonance frequency and the frequency shift is monitored.

**Amplitude Detection**

In this detection scheme the oscillator is driven at a fixed frequency and its amplitude is detected and used as a signal for the varying oscillator properties. As shown in Figure 2.2 three different cases have to be distinguished: the driving frequency can be below, at or above the free resonance frequency. This configuration determines how the shift in the resonance frequency $\Delta f$ and the additional dissipation $\Delta P$ is combined in the resulting

\(^{1}\)In some special applications of micromechanical oscillators, as for example in the artificial nose or in micro balances, the mass is changes and its effect is to be detected.
amplitude. For the case that the driving frequency is at the free resonance frequency, this detection is more sensitive to alterations of the damping and, in first order, insensitive to frequency shifts. In this scheme, the thermodynamical limited signal-to-noise ratio \( S/N \) of the cantilever can be increased by increasing the \( Q \) of the oscillator resonance (sec. 2.4.4). However, increasing \( Q \) also decreases the maximum available bandwidth of the system [72].

**Phase Detection**

In the phase detection scheme, the driving frequency is also kept fixed, usually advantage exactly at the free resonance frequency. Instead of the amplitude, the phase of the signal from the deflection detection relative to the the excitation signal is used as a signal for the varying oscillator properties. Mainly the conservative forces which cause a shift in the resonance frequency are detected here. Since the slope in the phase is very steep at the resonance, this method is very sensitive to small frequency shifts \( \Delta f \). The amplitude may simultaneously be recorded as a signal that gives additional information. For frequency shifts smaller than the resonance width \( (\Delta f \ll f/Q) \) the amplitude signal can be at-

![Figure 2.2: Illustration for detection schemes with a fixed frequency (black lines). The three different resonance curves are for the free oscillation (blue)\((k = 50N/m, m = 31.7ng, Q = 1000)\), with increased damping (green)\((Q = 500)\), or with an attractive force gradient (red)\((k_{at} = 0.05N/m)\). Depending on the driving frequency (black lines) relative to the free resonance frequency, a conservative interaction and a dissipative interaction combine in different ways to the detected amplitude or phase.](image)
tributed to dissipative interactions. The interpretation of the signals in phase detection as well as in amplitude detection is difficult due to the unknown combination of dissipative and conservative signals.

**Frequency Detection**

In the frequency modulation scheme [62], the cantilever is driven always at its momentary resonance frequency. This is realized by feeding the response signal back with the correct phase shift or by generating a driving signal that is phase locked to the response signal. In the first case the signal has to be demodulated in order to get a signal for the frequency shift. This is done either by a heterodyne demodulation or by a Phase Locked Loop (PLL). In any case the amplitude of the oscillator can additionally be kept constant by controlling the amplitude of the driving signal. The amplitude of the excitation signal serves then as the measure of the dissipation. The big advantage of the frequency detection compared to the phase- or amplitude detection is that both parameters, the resonance frequency $f_0$ and the quality factor $Q$ can separately and simultaneously be detected. In this way all the parameters of the harmonic oscillator are determined since the effective mass is assumed to be constant.

**2.4.3 Controlling the Tip Sample Distance**

Generally the forces between the tip and the sample are attractive at large distances and repulsive at small distances. To keep the tip at a certain distance to the sample, a feedback controller has to adjust its $z$-position to maintain a given deviation of the oscillator properties. This is usually performed by a PID-Controller [73, 74]. Generally it is distinguished whether the set point is in the repulsive regime or in the attractive regime.

**Repulsive Forces: Tapping Mode**

The setpoint for the $z$-distance controller is set such that the tip laps into the repulsive part of the interaction. The tip will bounce from the surface in each period which abbreviates its cycle (fig. 2.1) and enhances therefore the frequency. The amplitude and thus the energy of the oscillator should be high enough to allow the tip to escape the attractive forces near the surface, i.e., to avoid a "snap into contact".

**Attractive Forces: Non Contact Mode**

The setpoint for the $z$-distance controller is chosen such that the tip sample interactions are dominated by the attractive forces and the tip does not reach significantly the repulsive part of the interaction potential. Only in this mode the atomic resolution was achieved [75]. Since the slope of the frequency or phase vs. distance curve changes the sign, this mode is difficult [76]: a sudden external perturbation may bring the tip into the repulsive part of the interaction potential and then the feedback controller reacts with a further approach to the sample and the tip will be fully forwarded towards the sample. A combination of the frequency shift signal and the dissipation signal may avoid this problem [77]. However if the sample surface is very flat and the external vibrations are minimized this operation mode can yield atomic resolution [51]. If a voltage between the tip and the sample is
applied, the attractive forces range much longer, up to hundreds of nanometers, and the tip can safely be kept at such a distance.

### 2.4.4 Fundamental Limits in Dynamic Force Detection

With the achievement of atomic resolution it has been proven that using a macroscopic oscillator it is possible to detect forces between two single atoms. The question then arises what magnitude of the forces can be detected with the dynamic force detection. In the following the fundamental thermodynamic limitations for a mechanical oscillator are given.

According to the fluctuation dissipation theorem each dissipating element produces also fluctuations. The thermal white noise drive of an oscillator is

\[ F_{\text{th}}(\omega) = \sqrt{4\pi m\omega_0 k_B T/Q} \quad [\text{N/Hz}] \]  \hspace{1cm} (2.10)

Multiplied with the transfer function of the harmonic oscillator

\[ G(\omega) = \frac{1}{m\sqrt{(\omega_0^2 - \omega^2) + (\omega_0\omega/Q)^2}} \quad [\text{m/N}] \]  \hspace{1cm} (2.11)

the spectral noise density for deflections results in

\[ z_{\text{th}}^2(\omega) = \frac{4k_B T\omega_0}{mQ \left( \omega^2 - \omega_0^2 + (\omega_0\omega/Q)^2 \right)} \quad [\text{m}^2/\text{Hz}] \]  \hspace{1cm} (2.12)

In accordance with the equipartition theorem the mean square deflection results to

\[ \frac{1}{2} k z_{\text{th}}^2(\omega) = \frac{1}{2} k_B T \]  \hspace{1cm} (2.13)

The thermal energy in the cantilever is given only by the temperature, and via the spring constant \( k \) the thermal mean square deflection of the oscillator is also given. Equation (2.12) determines how this energy is distributed in the frequency domain. The higher the \( Q \)-value the more concentrated is this energy around the resonance frequency \( \omega_0 \). This implies that the thermally activated motion at the resonance frequency is enhanced with increasing quality factor; but a few linewidths off the resonance the thermally activated motion is diminished. Frequency modulation noise at a modulation frequency \( \omega_{\text{mod}} = \omega - \omega_0 \) arises from thermal noise in the sidebands. A detailed analysis of the signal to noise ratio (S/N) is given in ref. [72]. The minimum force gradient that can be detected by frequency shifts is

\[ F_{\text{min}}' = \sqrt{\frac{4k_B T B}{\omega_0 Q z_{\text{osc}}} \left( z_{\text{osc}}^2 \right)} \]  \hspace{1cm} (2.14)

with \( B \) being the detection bandwidth and \( z_{\text{osc}} \) the driven oscillation amplitude (signal). To reach his fundamental thermodynamic limit several conditions have to be fulfilled: the thermal white noise drive (2.10) has to be dominant over other forces. External vibrations and the noise produced by piezos connected to high voltage amplifiers with a certain electrical noise have to be taken into account. Other limitations arise from the deflection
2.5. KELVIN PROBE

Detection equipment. For very high Q-values an oscillator shows a very low thermal activated motion off the resonance (eq. 2.12) which is easily superseded by the noise of the deflection detection system. A similar formula as (2.14) can be derived in this case. The thermodynamic limit has been reached experimentally [78], even at low temperatures [61].

This extreme sensitivity to small force gradients is not only used for dynamic force microscopy but also for any kind of force detection as for example for magnetometry and for gyroscopes. Since micro-oscillators are versatile tools many special designs have been reported [79, 80, 81, 82]

2.5 Kelvin Probe

In 1898 Lord Kelvin developed a method to measure the contact potential difference (CPD) in reference to a known standard [83]. The CPD is defined as the difference of the chemical potentials $\mu_{\text{ch}}$ of the two neutral solids. By an electrical connection the electrochemical potentials tend to equilibrate resulting in a build up of an electrostatic potential. The electrochemical potential $U$, as it is measured and applied by laboratory devices, is the sum of the chemical and the electrostatic potential.

For a capacitance the change in charge $\Delta Q$ as function of the change in the capacitance $\Delta C$ is given by

$$\Delta Q = (U - U_{\text{CPD}})\Delta C$$

and hence periodical variations in $C$ will cause a periodic charge or current. The signal (2.15) can be nulled by adjusting the external electrochemical potential difference $U$ such that the electrostatic potential difference $U - U_{\text{CPD}}$ vanishes. The CPD is very sensitive to surface properties and treatments and is generally a function of the position on the surface due to changes in composition and adsorption. The scanning dynamic force microscope is ideally suited for a local measurement of the CPD since the probe and therefore the capacitance are oscillating [84]. The tip sample capacitance as a function of tip position is also of interest and can be measured simultaneously.

Experimentally either the electric field is nulled by a feedback controlling of the tip sample bias voltage which signals the local CPD [85], or the tip sample voltage is kept fixed and the variations of the electrostatic potential as a function of tip position are recorded. The nulling of the electric field allows the scanning probe to be electrically non-invasive and is of importance even if the CPD is not of interest at all. Several operation modes will be discussed in the following.

**Displacement Charge or Current**

A periodic modulation of the distance between the two electrodes at frequency $\omega$ results in a current $I(t)$ given by

$$I(t) = \dot{Q}(t) = (U - U_{\text{CPD}})\omega\dot{C}\cos\omega t$$

where $\dot{C}$ is the amplitude of the capacitance modulation. For the actual measurement of the $U_{\text{CPD}}$, the external bias voltage $U$ between sample and probe is adjusted until the space in between is field free and the current goes to zero [86, 87, 30]. Care has to be taken to discriminate the capacitive coupling of the excitation signal for the oscillator.
via parasitic capacitances to the tip. As a variant, the voltage induced on the tip can be measured with high impedance instead of the current measured with low input impedance.

Resonant Electrostatic Force

The electrostatic force $F_z$ acting on the two parts of a capacitor is given by

$$F_z = \frac{dE}{dz} = \frac{1}{2} \frac{dC(z)}{dz} (U - U_{CPD})^2,$$  \hspace{1cm} (2.17)

where $E$ is the electrostatic energy. Applying a small ac voltage in addition to a dc voltage $U(t) = U_{dc} + U_{ac} \cos(\omega t)$ results in an ac force proportional to the electrostatic potential difference.

$$F_\omega = \frac{dC}{dz} (U_{dc} - U_{CPD}) U_{ac}$$  \hspace{1cm} (2.18)

A force at twice the frequency, $2\omega$, also arises and is determined by the derivative of the capacitance

$$F_{2\omega} = \frac{1}{2} \frac{dC}{dz} U_{ac}^2.$$  \hspace{1cm} (2.19)

Detecting periodic forces is very sensitive with high $Q$ oscillators as described in section 2.4.4. The frequency $\omega$ is therefore chosen to match either the resonance frequency of the cantilever or its half frequency in order to have the double frequency signal on the resonance. Because the first resonance of the cantilever is usually used to perform the distance regulation, often the second flexural mode of the cantilever is used for the Kelvin probe.

Low Frequency alternating Force Gradient

According to equation (2.8), the frequency shift is proportional to the force gradient which in this case is given by

$$\frac{dF_z}{dz} = \frac{d^2E}{dz^2} = \frac{d^2C}{dz^2} (U - U_{CPD})^2.$$ \hspace{1cm} (2.20)

Thus again applying a dc voltage in addition to a small ac voltage with a low frequency $\Omega$ compared to the resonance frequency of the cantilever $\omega_0$, $U(t) = U_{dc} + U_{ac} \cos(\Omega t)$, an ac frequency shift $\Delta \omega$ with frequency $\Omega$ and amplitude proportional to the electrostatic potential difference results:

$$(\Delta \omega)_\Omega \propto \frac{d^2C}{dz^2} (U_{dc} - U_{CPD}) U_{ac}.$$ \hspace{1cm} (2.21)

At the double frequency, $2\Omega$, the frequency shift is given by the second derivative of the capacitance and is independent of the dc-voltage.

$$(\Delta \omega)_{2\Omega} \propto \frac{d^2C}{dz^2} U_{ac}^2.$$ \hspace{1cm} (2.22)

The frequency $\Omega$ of the ac-voltage has to be well below the frequency demodulation bandwidth. Again, to allow for simultaneous topographical feedback a higher flexural mode of the oscillator can be used.
Potentiometry of Conductors

An interesting application of the Kelvin probe is the mapping of the potential distribution of a conducting sample in response to a flowing current. In this case the local variation of the CPD due to contaminations of the surface has to be distinguished by a reference measurement in which a voltage is applied to all contacts of the sample simultaneously. Cleaved surfaces of GaAs devices have been measured in ref. [88] and the investigation of a quantum Hall conductor is reported in ref. [32, 36].
Chapter 3

Construction of the Scanning Probe Microscope

In this chapter the experimental setup and the engineering work for the construction of the scanning probe microscope operating at low temperatures and in high magnetic fields and capable of in situ transport measurements is presented. The first section defines the requirements for the microscope to be set up. The second section reports on a commercially available microscope with which the work has been started for exploring and learning the techniques of scanning probe microscopy at low temperatures. In the third section the setup of an experimental platform for sensitive low temperature experiments in vacuum is presented followed by a section where the new microscope head fitting to that platform is described.

3.1 Requirements

The demands for the resolution are not too high since the electrostatic interaction between the probe and the electronic structure which is usually buried a few 10's of nm below the surface is limited. The Fermi wavelength of the electrons in AlGaAs heterostructures is about 30nm and thus a lateral resolution of about 1nm is good enough and atomic resolution on the surface is not necessary.

The scan range on the other hand should be large enough to allow the imaging of photo-lithographically defined structures. At low temperatures the piezo effect of typical piezo ceramics is reduced by about a factor of 5 and therefore a large piezo tube has to be employed for scanning. This has the disadvantage that the low resonance frequency of the large tube will limit the maximum bandwidth for the feedback controlling and therefore limiting the scan speed. A scan range of about 5 - 10μm at low temperatures is a good compromise. The stability of the piezos is a great benefit of the operation at low temperatures and creep compensation [4] can be omitted.

The demands for the probe sample interaction detection depends on the experiments that are to be performed. Tunneling detection is not appropriate for the distance control because the semiconductor surfaces are generally insulating at low temperatures and a local current would disturb the electronic structures. Nevertheless it is a good option for some spectroscopy applications (sec. 2.2.1). For the actual experiment, the local probe
interacts with the sample electrically or eventually magnetically. Therefore an appropriate sensor for the electro-magnetic interactions has to be applied. A separately contacted tip in conjunction with a current to voltage converter or a sensitive charge amplifier realized with a field effect transistor (FET) are two simple examples. However, as mentioned in section 2.5, electro-magnetic interactions can also be probed via the forces acting on the probe and a dynamic force detection with a high quality factor is of advantage.

Optical deflection detection systems cannot be used here because the heterostructures under investigations show the persistent photoeffect. Piezoresistive detection is proven to work for this purposes but the problems with magnetic fields and power dissipation favors the piezoelectric detection.

The ability of coarse positioning the probe relative to the sample even at low temperatures is a crucial point for the purpose of this microscope. Thermal drifts usually let the probe drift away from the position of interest and the structure has to be searched at low temperatures. This positioning cannot be monitored with an optical microscope and a capacitive position detection simplifies the task. Markers on the sample are also an option for the orientation on the sample. A symmetrical design of the microscope and a careful choice of the materials helps to minimize the drifts during cooldown.

The materials have also to be compatible with high magnetic fields excluding superconducting materials like aluminum and magnetic materials like stainless steel. The materials should have a good thermal conductivity but the electrical conductivity may cause problems due to eddy currents. The thermal expansion coefficients have to be considered when joining two different materials in order to minimize strain and avoid cracks.

The cabling is a big challenge for the construction of a low temperature microscope. Depending on the design up to fifty or more electrical connections are needed which have to cope with higher frequencies (up to MHz) for oscillator operation and capacitance measurements and with higher voltages (a few 100V) for the piezos. A compromise between the thermal load and a good electrical conductivity has to be found. The cables should be shielded to minimize the stray capacitances and thermally anchored to ensure the thermal homogeneity.

The handling of the system is a crucial point because both the sample and the probe are very fragile and their replacement should be as easy and safe as possible although the limited space inside the bore of the magnet demands for a compact design. The electrical connectors for the sample and the probe have to be reliable and robust without affecting the mechanical stability. An important point is also the maintenance, that means that parts that can fail can easily be replaced. Specially the piezos and the plug connectors are candidates for signs of wear. Furthermore it has to be taken into account that future experiments may need additional equipment. Optical fibers, microwave transmission lines, additional cables or even a 3He system should be installable.

To operate the microscope in vacuum has several advantages: during cooldown the condensation onto the sample is prevented and the water layer on the sample can be minimized by pumping the vacuum for several hours at elevated temperatures. Furthermore the dynamic force detection profits of the higher quality factor and frequency stability of the oscillator. The problems due to the formation of superfluid He films are also eliminated. A sample heater that allows to heat the sample isolated from the rest of the microscope gives additional possibilities to overcome the problem of contaminations.
3.2. THE OXFORD CRYO-SXM

The vibration isolation is another big challenge in low temperature scanning probe microscopy. Since all materials get very stiff at low temperatures a passive damping with visco-elastic materials is not possible. The damping with eddy currents is also difficult because of the presence of high magnetic fields and the demands for its homogeneity. The cryogenic environment itself produces a lot of noise from the boiling of liquids and the vibrations of the pumps connected to it. A small, compact and rigid design helps to make the microscope less sensitive to vibrations.

3.2 The Oxford Cryo-SXM

3.2.1 Overview

The first version of the microscope was based on the commercially available "Cryo-SXM" from Oxford Instruments (fig. 3.1), which was delivered with a tunneling feedback system. The microscope head which is made of non magnetic material (mainly Ti and CuBe) is mounted at the end of a sample rod which can be suspended in the sample space of a 1.7K variable temperature insert (VTI). The VTI is part of a standard 4He cryostat with a superconducting magnet producing magnetic fields up to 8 Tesla. The microscope can be operated under ambient conditions or in the cryostat in 4He gas at temperatures between 200 K and 1.7 K and at pressures of typically a few millibars.

The scanning unit is a five electrode tube scanner [89] of 50.8 mm length and outer diameter of 12.7 mm. With a maximum (bipolar) voltage of 230 V it gives a lateral scan range of 52.2 μm in x-y direction and a z-range of 5 μm at a temperature of 290 K. At 4.2 K the lateral range is 8.8 μm and the z-range is 0.85 μm.

The TOPS3 control electronics by Oxford Instruments consist of a digital feedback loop which can be switched from logarithmic amplification for STM operation to linear amplification for the SPM mode. Tip approach and data acquisition with 8 simultaneous input channels is fully computer controlled.

The microscope head allows coarse probe-sample approach using a slip-stick drive [90, 91] moving the scan piezo up or down (fig. 3.2). A puck mounted at the end of the scan-piezo can be laterally positioned in a similar fashion.

3.2.2 Modifications

In contrast to the commercial design of the head, the puck serves here as the platform where the sensor is mounted (fig. 3.2d). Four semi-rigid coax cables were installed in addition to the three stainless steel coax cables of the standard configuration. They are used for the operation of the sensor for the dynamic force detection. For the transport experiments and the thermometry, additional twenty-four teflon insulated copper braid cables were installed. The original plug, connecting the microscope with the sample rod, was replaced by a self made plug connector due to problems with reliable contacts. A new sample holder unit (fig. 3.3) was constructed that can be used for standard magneto transport measurements independent of the SPM operation. Samples are mounted in chip carriers which can be easily plugged into a 32-pin chip socket. The socket is mounted on a copper block which incorporates the sample heater and the sample thermometer. The heater allows to evaporate the water film from the sample surface before cooldown.
It further allows to keep the sample warmer than its surroundings during the cooling process in order to avoid freezing contaminations on the sample surface. For detection of temperature gradients, additional thermometers were installed at the bottom of the VTI and at the top of the microscope head.

**Figure 3.1:** The "Cryo SXM" from Oxford Instruments was originally delivered with a tunneling feedback system. It has then been adapted with a sample holder and cabling for transport experiments and with sensors based on quartz tuning forks for the dynamic force detection.
3.2.3 Problems

Although some experiments could be performed with this microscope (sec. 5.1 - 5.3), we decided to redesign the system due to the insufficiencies of the system listed below:

- The system has no vibration isolation. Vibrations arise from the building vibrations, from the pumps connected to the cryostat, from the boiling of the liquid helium in the cryostat, from the turbulences of the gas jet escaping from the needle valve, from the mechanical 50Hz signal produced by all the transformers of the numerous

Figure 3.2: The coarse positioning mechanisms. For the z-positioning, the scan piezo is mounted in a CuBe-piece (a) which is clamped with springs on three quartz tubes and slides up and down (b) when they are kicked by piezo tubes within the quartz tubes (c). For the xy-positioning, a puck (d) is clamped at the end of the scan piezo (e) and can be moved by lateral kicks performed with the scan piezo.
devices in the lab as well as from other acoustic sources as for example the typing on the computer keyboard.

- The insertion in the cryostat is a very critical action. The probe has to be positioned at room temperature and then the microscope has to be inserted in the VTI as gently as possible. The long sample rod and the heavy cables connected to it makes this a risky task. The VTI can unfortunately not be heated up to room temperature so the operation has to be completed as fast as possible to avoid blocking of the needle valve, not to speak from contaminations condensing onto the sample.

- The cables do not have a thermal anchoring, they are just cooled by the He gas. The large amount of electrical connections, necessary to operate a scanning probe microscope, leads to thermal loads which cause the microscope head to have a very inhomogeneous temperature profile. A stable temperature gradient along the sample rod has to be maintained in order to avoid thermal drifts.

- Capacitive coupling between the cables for the piezos, the transport measurement and the sensor impede the experiments and can generate artifacts.

- The pressure in the VTI, which strongly affects the resonance frequency, has to be stabilized below the vapor pressure of $^4$He in order to prevent liquid helium to enter the VTI. Operation in normal liquid helium is possible but the $Q$-value is reduced by a factor of four. Below 2.2K the helium becomes superfluid and the operation of the tuning fork becomes difficult. In our cryostat the resonance frequency becomes unstable, presumably due to thermodynamic instabilities.
3.3 Experimental Platform

In this section the new designed experimental platform is described that allows to perform sensitive experiments at low temperatures, high magnetic fields and ultra high vacuum. First the laboratory installations and the system integration is presented followed by a description of the vacuum beaker and the connector as the interface to the experiment.

3.3.1 Cryostat, Crane and Vibration Isolation

The installations in the laboratory involve the cryostat with the gas handling system and the pumps necessary for its operation. Suspending the cryostat from bungee ropes eliminated the building vibrations very effectively due to the large mass of the cryostat (resonance frequency below 1 Hz). This is not necessary anymore since the setup is now located in the basement in a lab with its own decoupled foundation. The cryostat has large metallic containers that have bell like resonances of very high quality. This leads to an enhanced coupling of acoustics from the room to the microscope. The acoustic resonances of the outermost container can be damped by a caoutchouc mat tied around the cryostat body. Vibrations from the $^4$He pump are effectively damped by guiding the pumping line through a box of sand.

The important vibration isolation between cryostat and sample rod is implemented by the stone ring which is supported on special rubber foam (fig.3.5). The sample rod is mounted with a tripod on top of this stone. When the microscope is in the cryostat, a direct mechanical contact to the VTI tube can be prevented by slightly tilting the sample rod with three adjusting screws. The inner diameter of the VTI tube is 50mm and the

![Figure 3.4](image-url)

Figure 3.4: The microscope (a) is mounted on a stone plate (b) which can be moved vertically by a simple hand operated crane (c). After the experiment is prepared to cool down with the help of an optical microscope (d), the sample rod is lifted, the folding table (e) is turned down and the cryostat (f) which is movable on four steel balls (g) is placed under it. The sample rod with the vacuum beaker (h) is then gently lowered into the VTI until the stone plate sets down on the pillars (i) on top of the cryostat.
Figure 3.5: To minimize the vibrations, the microscope is mounted on a massive stone plate (c) (ca. 50 kg), which is mechanically isolated from the cryostat by special rubber foam (d). With three adjusting screws (b) the sample rod can be slightly tilted so that it hangs freely in the VTI tube. The brass ring (g) is movable in \( xy \)-direction to allow a precise centering. The VTI is connected by a neoprene bellow (not visible). The cables are embedded in a sand filled plastic tube (a). The cryostat is tightly packed into caoutchouc (f) to prevent acoustic resonances.

The outer diameter of the vacuum beaker is 44 mm. To allow also for a fine \( xy \)-positioning of the sample rod, a brass ring laying on teflon sheets can be moved by hand.

The preparation of the experiment and the maintenance of the microscope can be done on a table with a hole in the center, which allows to adjust the height of the microscope with the hand operated crane to an optimum working position. An optical microscope is useful for the coarse positioning of the probe onto the sample. When the experiment is prepared for cooldown, the stone plate and the sample rod can be lifted and the cryostat, which is movable on four steel balls, can be brought into position directly under the vacuum beaker. The crane is then lowered until the stone plate sets softly down on the three pillars on top of the cryostat.

3.3.2 Vacuum Chamber

As material for the vacuum beaker, oxygen free high conductivity (OFHC) copper is used to ensure a good thermal homogeneity. The inner diameter is 40 mm and the wall thickness is 2 mm resulting in an outer diameter of 44 mm, fitting well in the VTI tube of the cryostat which has a diameter of 50 mm. The disadvantage of the high conductivity are the eddy currents in response to a changing magnetic field which will produce some amount of heat. For scanning probe microscopy applications that won't be an obstacle, since the scanning of an image last from several minutes up to hours and the magnetic field will be kept constant. To control the temperature of the copper beaker two calibrated thermometers are incorporated inside the copper cone. This is namely a PT100 resistor for temperatures from 150°C down to liquid nitrogen temperatures of -200°C and an Allen Bradley resistor...
3.3. EXPERIMENTAL PLATFORM

Figure 3.6: The vacuum chamber as an experimental platform for low temperature experiments. (a) Cone with flange and steel tubes for the feed through. (b) Stainless steel tube with flange. (c) Sealing of the feed through with Stycast epoxy. (d) The connector as the interface to the experiment. (e) Self-made semi-rigid coax cables for the connection to the outside. (f) The copper beaker attached to the cone which forms the sealing.

for the lower temperatures. A heater allows to control the temperature with an appropriate temperature controller. Due to the excellent thermal conductivity of the OFHC copper, the temperature of the beaker is very homogeneous and is controllable with a stability of 1 mK.

Due to the limited space in the cryostat (diam. 50 mm), the beaker is closed with a conical sealing. The angle of the cone is 3 degrees and determines the pressure between the two surfaces and has to be in a range that the beaker holds even if the vacuum is vented but can be taken off without big effort. Vacuum grease is used to prevent a permanent joint. The vacuum beaker is held by a long stainless steel tube (fig. 3.6b) which is flanged to the copper cone with an indium sealing. The upper end is sealed with an all-metal valve. The warm part of the vacuum space is fully UHV compatible, which is important when the beaker is cold and cryo-pumps the warmer part. Contamination of the sample due to outgasing parts in the warm part of the vacuum is avoided. This is the first reason to make the feed-through of the electrical connections at the low temperature part. The second reason is the better cooling of the cables by the cryogenic helium gas.

Figure 3.6d shows the layout of the connector to the experiment sitting inside the
beaker. All fifty four connections are separately fed through in a coaxial manner. An Epoxy filled with Al₂O₃ powder (Stycast 2850FT [92]) is used for the sealing of the feed-through's [93, 94, 95]. This epoxy is very suitable because of the thermal expansion coefficient that is close to that of copper and the good thermal conductivity compared to other insulators [96, 97]. The cables are also thermally anchored with this epoxy by guiding them along the neck of the copper cone (fig. 3.6a,c). Home built semi-rigid cables consisting of stainless steel tubes (outer diameter 0.85mm) and a teflon insulated copper braid wire are used for the connection to the outer world. The cables are 4.5m long and lead without interruption to the rack with the electronic devices. This circumvents an additional connector plug on the upper part of the the sample rod, but doesn't allow to place preamplifiers close to the experiment and one has to cope with the high cable capacitances of 400pF. The bunch of cables is guided in a sand filled plastic tube from the stone plate to the rack. This eliminates mechanical vibrations onto the stone plate and also the impact of acoustics to the cables. Figure 3.7 shows the connector box with the fifty four standard BNC-connectors for a versatile access to the experiment. The cables have a good conductivity (2.5Ω) and small capacitive coupling (< 10fF). Three of the cables are realized as a triax cable¹ as shown in figure 3.7b: the coax cable is insulated with a teflon tube and is then tread in another stainless steel tube. With triax cables the problems due to the cable capacitances can be circumvented by keeping the guard (the

---

¹only one of them works properly
inner steel tube) with a guard amplifier on the same potential as the signal cable.

3.4 Microscope Head

3.4.1 Microscope Casing

For the microscope constructed for the operation in the vacuum beaker, the same scan unit as in the "Cryo-SXM" is used. The casing is 38 mm in diameter, fitting in the vacuum beaker with a 1 mm gap on each side. It is made from a single piece of copper-beryllium (CuBe), an alloy that is very hard, non magnetic and of good thermal conductivity. A rigid design and a mechanical path from the sample to tip as short as possible are important in order to have the lowest resonance frequency as high as possible. The threefold

Figure 3.8: The microscope casing in which the scan unit and a sample holder can be mounted. (a,b): The piece is machined from a single piece of copper-beryllium (CuBe) alloy. (c,d,e): The connector consists of two parts, the outer ring for the connections to the sample and the inner ring for the scan unit and the sensor. The inner ring is detachable to allow the scan unit to be taken out for maintenance purposes. (f): fully assembled microscope.
symmetrical design minimizes the lateral drifts when the microscope is cooled down. Figure 3.8b shows this masterpiece of metalworking.\footnote{made in the machine shop of ETH Hönggerberg} Subfigures (c), (d) and (e) show the connector that is divided into two parts. The outer ring serves for the connections to the sample which lead via coax cables to a plug at the bottom of the casing where the sample holder can be mounted. The inner ring is for the scan unit and for the sensor. The scan unit can be taken out with the inner ring for maintenance purposes. The microscope is screwed tightly to the copper cone for a good thermal linkage.

3.4.2 XY-Table

The big advantage of the xy-coarse positioning mechanism implemented in the original "Cryo-SXM" system and shown in fig. 3.2 is the fact that no additional cables are needed for the operation. All the movements are performed by kicks of the scan piezo. Unfortunately the original system with the puck clamped in the titanium pocket didn't work very reliably, especially at low temperatures. Bringing electrical connections, preferably coax-cables onto the puck for the sensor made the reliability even more difficult. To overcome these problems the xy-coarse positioning mechanism was redesigned as shown in figure 3.9. The puck is pulled by the six coax cables to a alumina ring at the end of the scan piezo. On this ring the puck slides on three sapphire balls. The pulling force can be adjusted by a screw determining the load of the springs on top of the scan piezo.

![Figure 3.9: The redesigned xy-coarse positioning mechanism.](image)

3.4.3 Sample Holder

In the group where the presented work was performed all samples are mounted in a standard chip carrier with thirty two pins. The socket where the chip carrier can be plugged in is shown in figure 3.10a. It is wired with coax cables to keep the capacitive coupling as small as possible. This is important especially for a capacitive position detection of the
3.4. MICROSCOPE HEAD

Figure 3.10: The sample holder (a) that can be plugged into the connector (b) at the bottom of the microscope (c). It provides space for additional electronics used for transport experiments. A different material than CuBe can be chosen to compensate for the thermal expansion coefficient of the probe sample spacing.

tip via electrodes on the sample. Inside the body of the sample holder there is enough space to place additional electronics like capacitors, resistors, voltage dividers or even preamplifiers. A heater and two thermometers as described in sec. 3.3.2 are incorporated in a small piece of copper which is in good thermal contact to the sample and isolated from the metallic body of the sample holder. This allows to control the temperature of the sample to a higher temperature than the rest of the microscope. However, the sample and specially the electron gas in it is in good thermal contact via the electrical connections which are thermally anchored to the copper cone and an isolated heating of the sample is difficult. The sample holder is made again from CuBe, but other materials with other thermal expansion coefficients can be used here for thermal compensation of the probe sample spacing.
Chapter 4

Piezoelectric Quartz Tuning Forks

In this chapter the application of piezoelectric quartz tuning forks in dynamic force microscopy is described. For the motivation first a historical overview and a comparison with traditional cantilevers is given. In the second section the theories for tuning forks as oscillators for the dynamic force detection are introduced and in the third section the experimental implementation is described.

4.1 Introduction

The tuning fork is one of the best mechanical oscillators. It was invented in 1711 by the English trumpeter John Share. The important mode of the tuning fork is the one where the two prongs oscillate in a mirrored fashion. This has the unique advantage that the center of mass stays at rest and all forces are compensated inside the material connecting the two prongs. Since quartz is one of the materials with the lowest internal mechanical losses, quartz tuning forks have very high quality factors. Furthermore the piezoelectric effect of quartz allows to excite and detect the oscillation of the tuning fork fully electrically. Quartz resonators are widely used as frequency standards and tuning forks are just special examples with high quality factors and low resonance frequency. They are used for example in wrist watches where the insensitivity to accelerations is an additional advantage. Other applications are gyroscopes, micro balances, gas sensors and scanning probe microscopy. Due to the large industrial production they are available at very low cost.

Piezoelectric quartz tuning forks were introduced into scanning probe microscopy by

Figure 4.1: Piezoelectric quartz tuning forks are industrially produced in large numbers and serve as a frequency standard for example in wrist watches. They are packed in a evacuated steel casing and show quality factors of typical 30'000. The type shown is 4mm long and oscillates at \(2^{16}\)Hz = 32768Hz.
Günther, Fischer and Dransfeld [65] for use in scanning near field acoustic microscopy and later by Karrai and Grober [15] and others [98, 99, 100], as a distance control for a scanning near field optical microscope (SNOM). In this microscopes the optical fiber tip is oscillating parallel to the surface resulting in a shear force detection. Shear forces where then explicitly investigated using tuning forks by Karrai and Tiemann [59]. Quartz tuning forks with a magnetic tip were also used for magnetic force microscopy [101, 57]. Rensen et al. where able to resolve atomic steps with a cantilever and Si-tip attached to the tuning fork [102]. Recently Giessibl et al. demonstrated atomic resolution on the Si(111)-(7×7) surface using a tuning fork with one prong fixed (qPlus Sensor) [103].

Compared to micromachined Si cantilevers the tuning forks are very stiff. The problems discussed in section 2.4.1 concerning the nonlinearity of the oscillator motion in the interaction potential are reduced due to the high spring constant compared to the interaction forces. The stiffness avoids the snap into contact and thus allows to operate it with lower amplitudes. This simplifies the interpretation of the oscillator signals when the short range interactions are investigated. The high stiffness is also of advantage for nano manipulation applications as for example nano-lithography. However, it is a disadvantage for the detection of very small forces (eq. 2.14), and is a danger for the tip to be crashed since the force is not limited by a soft spring.

Since only two electrical contacts are necessary for the operation, piezoelectric tuning forks are simple to integrate in a scanning probe microscope even in a cryogenic environment [104]. They are insensitive to high magnetic fields and operate well at low temperatures. The fact that no light is needed for the deflection detection is important for the investigation of semiconductor heterostructures which show the persistent photo effect. Any light scattered onto the sample would alter its properties permanently.

Since the tuning forks are relatively large and massive, the quality factor is quite robust. In contrast to cantilevers, tuning forks have high quality factors even in ambient conditions ($Q \approx 10^6$) and dynamic force microscopy even in liquids is possible. The perspective to use them as a carrier for sensors like FETs, SETs, SQUIDs and hall sensors makes them attractive especially for the investigation of super- and semiconducting nanostructures in combination with transport experiments.

The direct electromechanical coupling also allows to measure the dissipation power very easily and accurately, a very powerful advantage of piezoelectric oscillators in dynamic force microscopy.

### 4.2 Theory of tuning forks

In this section the models used to deal with the tuning forks are introduced. First an electrical model, the Butterworth-Van Dyke circuit is presented and following the electromechanical coupling via the piezo effect is studied. The influence of asymmetries is then examined with a mechanical model and finally the fundamental limits concerning the dynamic force detection are calculated for the type of tuning fork used in this work.
4.2. THEOREY OF TUNING FORKS

4.2.1 Electrical model

Piezoelectric oscillators can be modeled by an electronic equivalent circuit called the Butterworth-Van Dyke circuit (fig. 4.2 and 4.3) [105, 106]. The LRC resonator models the mechanical resonance: the inductance stands for the size of the kinetic energy storage, i.e., the effective mass, the capacitance reflects the potential energy storage, i.e., the spring constant and the resistor models the dissipative processes [104]. The parallel capacitance is given by the contacts and cables. The transfer function \( Y(\omega) = \frac{I(\omega)}{U(\omega)} \), the so called admittance is

\[
Y(\omega) = \frac{1}{R + \frac{1}{\omega C} + i\omega L + i\omega C_0},
\]

and is experimentally measurable. In Figure 4.3 a Nyquist plot for the transfer function (4.1) is shown. As for any resonance the curve is a circle but due to the parallel capacitance \( C_0 \) its center is shifted along the imaginary axes by \( i\omega C_0 \). This leads to the typical minimum in the admittance shortly after the maximum in a Bode plot as shown in figure 4.4. On the resonance the current through the LRC branch flows in phase with the voltage. The current through the parallel capacitance has a phase shift of 90 degree and causes a small phase shift of the total current. However, the admittance of the capacitance \( C_0 \) is small compared to the admittance of the LRC branch and can be neglected. If it turns out to be a problem it can be compensated electronically with a bridge circuit.

![Figure 4.2: Butterworth-Van Dyke equivalent circuit for a piezoelectric resonator.](image)

![Figure 4.3: Nyquist plot for a Butterworth-Van Dyke equivalent circuit with parameters as in figure 4.4: \((f_0 = 3275\text{Hz}, Q = 62000, C_0 = 1.2\text{pF}, L = 8.1\text{kH}, R = 27.1\text{k\Omega}, C = 2.9\text{F})\). The frequency is increasing in clockwise direction and the spacing of the data points is 0.01 Hz.](image)
4.2.2 Electromechanical Coupling

A fit of equation (4.1) to the experimental data works extremely well. Out of the electrical data the parameters \( L, R, C \) and \( C_0 \) are obtained. This parameters are not sufficient to determine the mechanical oscillation amplitude. An additional parameter is needed: the piezo-electro-mechanical coupling constant \( \alpha \). It describes the charge separation \( Q \) on the electrodes on the piezo material per mechanical deflection \( x \): \( |\alpha| = C/m \). With a simultaneous measurement of the electrical response and the mechanical amplitude with an optical interferometer this constant can be determined \cite{104}. This constant is characteristic for one type of resonator and a modification, for example the attachment of a probe or the change of the environment will not alter this constant. The mechanical amplitude can always be determined by the current \( I \) through the resonator:

\[
Q = \alpha x \\
I = \alpha \dot{x} \\
I_{\text{rms}} = \alpha \omega x_{\text{rms}}
\]  

\( (4.2) \)

To model the mechanical resonance, an energetically equivalent mechanical model consisting of one mass and one spring is applied (inset fig. 4.4a). With the knowledge of the electro mechanical coupling constant \( \alpha \), the mechanical parameters can be determined from the electrical parameters by equating the potential energy \( Q^2/2C = kx^2/2 \) and the

\begin{figure}[h]
\centering
\includegraphics[width=0.8\textwidth]{figure4_4.png}
\caption{(a) Experimental measurement of the mechanical displacement of the front of a tuning fork prong at room temperature and a pressure of \( 10^{-6} \) mbar. The inset shows an energetically equivalent mechanical model (both prongs included). (b) The simultaneous experimental measurement of the electrical response. The inset shows the parameters for the electrical equivalent circuit.}
\end{figure}
the kinetic energy $LI^2/2 = mv^2/2$:

\[
\begin{align*}
L &= \frac{m}{\alpha^2} \\
\frac{1}{C} &= \frac{k}{\alpha^2} \\
R &= \frac{\gamma}{\alpha^2}
\end{align*}
\] (4.3)

From the electrical and mechanical correspondence the voltage can be identified as the driving force: $F = \alpha U$. Figure 4.5 shows the electric field in the crystal produced by the electrodes and how they are connected. This configuration detects and excites only movements of the prongs against each other. An interesting point to note is that there is a coupling of the two prongs via the piezoelectric effect. When one prong is deflected it produces a charge separation that in turn produces a voltage and thus deflects the other prong in the opposite direction. Assuming the tuning fork is not connected to any other electronics the charge is converted into a voltage over the capacitance $C_0$ of the electrodes and the coupling constant is then $\alpha^2/C_0 = 57 \text{ N/m}$. However, if the tuning fork is connected to cables, the capacitance is much larger and the coupling can be neglected. In the case of a fixed voltage (low impedance) the coupling is zero.

![Figure 4.5: Illustration of the electrical field lines in the cross section of the quartz tuning fork. The electric field along the horizontal direction causes a contraction or dilatation of the quartz in the direction perpendicular to the drawing plane. Only the movement of the two prongs in the mirrored fashion is electrically excitable or detectable.](image)

4.2.3 Mechanical Model

In the last section a mechanical model was introduced that energetically modeled the proper tuning fork mode and that is appropriate for the determination of the oscillation amplitude. However, questions concerning asymmetries of the tuning fork as they occur when preparing the tuning fork for the dynamic force detection, cannot be answered with this model. A model that takes the two prongs into account has to be applied and is shown in figure 4.6a. This system has two modes, a symmetric and an antisymmetric one, that are degenerate for vanishing coupling. The coupling splits the two frequencies and the two modes get mixed when the symmetry is broken. This model, however, cannot explain why the counter oscillating mode has a much higher quality factor than the synchronous mode. It is not the appropriate model for a tuning fork since it cannot explain its speciality. The model (b) in figure 4.6 has a third mass that models the movement of the base. In this model the counter oscillating mode still has a high quality factor because the center of mass stays at rest and all the forces are compensated inside the fork. The synchronous mode however, produces reaction forces in the support of the base and undergoes a much stronger damping. This model also explains the reduction of the quality factor when the
symmetry is broken for example by the attachment of a tip to one of the prongs. In the following this model is examined with the help of the Laplace transform and the influence of asymmetry is studied. The equations of motion are

\begin{align*}
    m_1 x_1''(t) &= \alpha U(t) - k_1 [x_1(t) - 0(t)] - k_c [x_1(t) - x_2(t)] - \\
                  &\quad - \gamma_1 [x_1'(t) - 0'(t)] - \gamma_c [x_1'(t) - x_2'(t)] \\
    m_2 x_2''(t) &= -\alpha U(t) - k_2 [x_2(t) - 0(t)] - k_c [x_2(t) - x_1(t)] - \\
                  &\quad - \gamma_2 [x_2'(t) - 0'(t)] - \gamma_c [x_2'(t) - x_1'(t)] \\
    m_0 x_0''(t) &= -k_0 x_0(t) - k_1 [x_0(t) - x_1(t)] - k_2 [x_0(t) - x_2(t)] - \\
                  &\quad - \gamma_0 x_0'(t) - \gamma_1 [x_0'(t) - x_1'(t)] - \gamma_2 [x_0'(t) - x_2'(t)],
\end{align*}

where \(\alpha\) is the piezoelectric coupling constant and \(U(t)\) is the applied voltage and the \(m\)'s \(k\)'s and \(\gamma\)'s are the masses, spring constants and damping constants respectively. Applying the Laplace transform, the system of differential equations (4.4) is transformed in a system of algebraic equations which can be solved analytically. The transfer function \(T_n(i\omega)\) for the motion of the mass \(m_n\) is given by

\[ T_n(i\omega) = X_n(i\omega)/U(i\omega), \quad l = 0, 1, 2 \]

where the capitals denote the Laplace transformed variables. The transfer function for the electrical response is given by the relative motion of the two arms:

\[ Y(i\omega) = \frac{i\omega C_0 + \alpha i\omega (X_1(i\omega) - X_2(i\omega))}{U(i\omega)} \]

Electrically only the relative motion of the two masses \(m_1\) and \(m_2\) are detected and excited, so that for a symmetric tuning fork only the proper mode is detectable.
4.2. THEORY OF TUNING FORKS

This model has too many parameters as to be determined from experimental data. However, the important values \((m_i, k_i, \gamma_i, i = 1, 2)\) are known from the model discussed in sec. 4.2.1 and the other parameters will just be guessed in order to get a qualitative understanding. For the mass \(m_0\) one has to decide which part of the base will come into motion when reaction forces act on it. This can be just the part of quartz that connects the two prongs, it can be the metallic ring that supports the tuning fork (fig. 4.1) or it can be all the sensor that is mounted on the piezo tube which itself can show resonances at the frequencies to be considered. When the tuning fork starts to oscillate in an asymmetric manner several different parts of the base and support will come into motion that are connected in a complicated manner. Here \(m_0\) just models the effective mass of all the oscillations that get excited. This effective mass will depend on the frequency and will also be altered at low temperature, but for the purpose of demonstration a fixed value of 5 mg (compared to the mass of 0.33 mg of one prong) is chosen. The spring constant \(\gamma_0\) is chosen to be 500 kN/m resulting in resonance frequency of around 50 kHz for the mass \(m_0\). The damping constant is now adjusted to have a quality factor of the order of ten for the oscillation of mass \(m_0\): \(\gamma_0 = 100 \text{mNs/m.}\) Finally the following values are used for an example:

\[
\begin{align*}
    m_1 &= m_2 = 0.33 \text{mg} \\
    k_1 &= k_2 = 13.79 \text{kN/m} \\
    \gamma_1 &= \gamma_2 = 1 \mu \text{Ns/m} \\
    C_0 &= 1.2 \text{pF} \\
    \alpha &= 4.2 \mu \text{C/m} \\
    k_c &= 100 \text{N/m} \\
    \gamma_c &= 0.1 \mu \text{Ns/m} \\
    m_0 &= 5.0 \text{mg} \\
    k_0 &= 500 \text{kN/m} \\
    \gamma_0 &= 100 \text{mNs/m}
\end{align*}
\]

A satisfactory agreement with the experimental data in figure 4.4 is achieved with the parameters (4.7) and (4.8) for the calculation of the transfer function (4.6) shown in figure 4.7. The poles of the transfer function can be calculated and the location in the \(\omega\)-plane determines the eigen frequencies and the damping of the three eigen modes. The model can now be applied to investigate the behavior of the tuning fork when an additional mass

---

**Figure 4.7:** The admittance (4.6) calculated with the parameters (4.7) & (4.8). The curve is in good agreement with the experiment of figure 4.4.
Table 4.1: The eigenmodes of the tuning fork model for increasing additional mass on one prong.

is brought onto one of the prongs. To illustrate the eigenmodes the following transfer functions will be examined:

\[ T_d(i\omega) = \frac{(X_1(i\omega) - X_2(i\omega))}{2U(i\omega)} \]

\[ T_s(i\omega) = \frac{(X_1(i\omega) + X_2(i\omega) - 2X_0(i\omega))}{2U(i\omega)} \]  

(4.9)

They correspond to the relative motion \( T_d \) and to the motion of the center of mass of the two prongs relative to mass \( m_0 \) \( T_s \). Table 4.1 lists the three eigenmodes and the transfer functions (4.9) for increasing additional mass \( \Delta m \) on prong 1. The remarkable reduction of the quality factor is in good agreement with the practical experience. Figure 4.8 shows the quality factor as function of the additional mass. The reduction of the frequency of the proper tuning fork mode is also observed experimentally. In conclusion it can be noted that the symmetry of the tuning fork is very important for a high quality factor. Any asymmetry lets reaction forces act on the base of the tuning fork which will cause an additional damping. Furthermore it was shown that the other modes of the tuning fork do generally not interfere with the proper mode or have a much lower quality factor. This is in contrast to the model of two coupled oscillators, where the degeneracy is only

![Figure 4.8: The quality factor is reduced significantly when an additional mass is brought onto one of the prongs \((m=0.33\,\text{mg})\).](image-url)
slightly resolved by the coupling and the quality factors of both modes are approximately the same.

4.2.4 Fundamental Limits

Applying the formalism introduced in section 2.4.4, the fundamental limits for the dynamic force detection with a quartz tuning fork [78] are discussed. As a mechanical model the simple spring mass model shown in figure 4.4a will be applied. While absolutely correct for the modeling of the proper tuning fork mode, the complications arising from having two prongs are avoided. For the parameters given in figure 4.4, the thermal white noise drive (eq. 2.10) is $192 fN/\sqrt{\text{Hz}}$ at 300 K and $11 fN/\sqrt{\text{Hz}}$ at 1 K. Multiplied with the transfer function for the mechanical model, the spectral thermal motion results and is shown in figure 4.9. Assuming that the deflection detection can detect such small motions, the minimum detectable force gradient calculated with equation (2.14) is $4.3 \text{mN/m}$ at 300 K for a detection bandwidth of 100 Hz and a amplitude of 1 nm. The value gets smaller for low temperature (1 K), smaller bandwidth (10 Hz) and larger amplitude (30 nm): $3 \mu \text{N/m}$. However, experimentally this will be hard to realize, since the frequency shift that has to be detected is as low as 3 $\mu \text{Hz}$ (for the second case). This corresponds to a relative frequency shift of $10^{-10}$, which demands for a stability of the reference frequency that exceeds the values of standard equipment. To reach the thermodynamic limit with tuning forks, the deflection detection has to be able to detect the thermal noise off the resonance. For a current to voltage converter with a noise of $100 fA/\sqrt{\text{Hz}}$ the thermodynamic limit at room temperature could be reached with a detection bandwidth of about 10 Hz. For

![Figure 4.9](image)

Figure 4.9: The thermal motion of a tuning fork at room temperature (300K) and at 1K. The motion is converted into a charge via the piezo-electro-mechanical coupling constant $\alpha$ and into a current by multiplying the charge with the frequency.
the low temperature case, this is not possible with such a current to voltage converter. With a sensitive charge detector with a noise of 0.01 e/√Hz, however, the thermal noise of the tuning fork at low temperature is dominant over a bandwidth of about 50 Hz. In conclusion it can be stated that to reach the thermodynamic limit for force detection, the detection bandwidth has to be narrowed or the quality factor has to be reduced to have the thermal noise of the tuning fork dominant over the deflection detection noise. Experimentally one has to worry also about other sources of noise that could exceed the thermal noise of the tuning fork. For example the noise of the excitation signal has to be smaller then 1.3 nV/√Hz which produces a force of U that corresponds to the thermal white noise drive of 11 fN/√Hz at low temperature.

### 4.3 Experimental Implementation of Tuning Fork Sensors

In this section the experimental implementation of the tuning fork sensors and the detection techniques are presented. First the assembly of the sensors is described followed by a description of the driving circuit with a Phase Locked Loop and a description of the low temperature preamplifier.

#### 4.3.1 Preparation of Tuning Fork Sensors

Tuning forks are produced industrially and are packed in an evacuated steel casing where they show quality factors of about 30'000. The casing is removed at the base and the quality factor drops to about 10'000 in ambient conditions [107]. The tuning fork is then mounted on a small circular plate with six holes for the connection to the puck (sec. 3.4.2). A thin metal wire is glued to one of the prongs and then electro-chemically etched to form a sharp tip (fig. 4.10a). The preparation of metallic tip for scanning probes is well described in the literature [108, 109, 110, 111], and in ref. [112] specially the etching of the thin wires used for the tuning fork tips. For the attachment of the tip it is very important to disturb the symmetry of the tuning fork as little as possible (sec. 4.2.3). The metal wire has a diameter of only 15 μm to minimize the additional weight on one prong. A small drop of glue on the other prong can help to compensate for the additional mass. The wire is either connected with one of the tuning fork electrodes causing an additional mass of about 1.5 μg, or is is separately contacted causing an additional weight of up to 50 μg. In the latter case the metal wire loop causes also an additional spring and damping for the one prong. To keep the wire loop as short as possible it is attached to a pillar mounted closely to the end of the tuning fork prong (fig. 4.10b). Tuning fork sensors with separately contacted tip show quality factors of about 50'000 in vacuum at 4.2K

With a separately contacted tip it is comfortable to perform tunneling or capacitance measurements. For the small signals as they occur in capacitance measurements and in Kelvin Probe Microscopy, a Field Effect Transistor (FET) that can be mounted very closely to the tip is advantageous (sec. 4.3.3). The FET has to be mounted parallel to the magnetic field to minimize its effects on the electron gas of the FET. Within this work, only the configuration where the tip oscillates perpendicular to the sample was tried out.

---

1 All quantitative data are related to the type shown in figure 4.1
4.3. EXPERIMENTAL IMPLEMENTATION OF TUNING FORK SENSORS

Figure 4.10: Quartz tuning fork sensors for the dynamic force detection. A thin metal wire is attached to one prong and etched or cut to form a sharp tip (a). The tip is separately contacted to a current to voltage converter (b) or to a field effect transistor acting as a low input capacitance preamplifier (c). The sensor plate is mounted on the puck of the xy-table with six screws which also serve for the electrical contacts (c).

but also the shear force configuration, where the tip oscillates parallel to the sample is well established [59].

4.3.2 The Phase Locked Loop

The frequency shift of the tuning fork is much smaller than that of traditional cantilevers when the tip interacts with the sample because the spring constant is much higher (eq. 2.8). This demands for a frequency demodulation with high resolution and stability. Since the tuning fork resonance is at a quite low frequency (33kHz), digital signal processing (DSP) can be applied. This is of great advantage because no analog devices can have a relative accuracy of $10^{-9}$ or better. As shown in figure 4.4 the phase between the excitation signal and the current through the fork as a function of the frequency is very steep at the resonance (about 180 degree/Hz). This allows to detect any shifts in the resonance frequency very sensitively. With a controller the excitation frequency is then automatically adjusted to maintain the phase at the value of the resonance. This is the idea of the PLL and is schematically shown in figure 4.11. This PLL is put together with
standard laboratory equipment and with two self made analog Proportional-Integral-(PI) controllers and a self made software for the integration and automation. This has the advantage that all the parameters like the detection bandwidth and PI parameters can easily and transparently be adjusted.

The deviation of the phase is detected with a digital two channel lock-in amplifier (SRS 830) which is synchronized by a digital signal from the frequency generator. The lock-in amplifier generates two orthogonal sinus signals as reference for the two channels. The phase of this reference signals with respect to the external synchronization signal can be shifted by an arbitrary value and is adjusted to have the x-reference signal in phase with the signal of the tuning fork at the resonance. Ideally this phase shift would be zero (fig. 4.3) but the current-to-voltage converters and the long coax cables cause an additional phase shift. The output of the y-channel which indicates any deviation from the resonance, is fed into a PI-controller to control the frequency to the resonance.

The frequency generator (Yokogawa FG300) employs the Direct Data Synthesis (DDS) and has a phase register of 48bit and a clock rate of 40MHz. The span of the frequency modulation range is typically configured to be 100mHz/V and the resolution for the frequency shift is then about 100μHz. For very sensitive force detection the parameters can be adjusted to achieve resolutions of the order of 1μHz. However, the stability of the reference frequency is specified to be 100μHz/°C and for the ultimate frequency shift detection an external reference frequency with a temperature controlled quartz oscillator (OCXO) or an atomic clock should be employed.

As an additional feature the oscillation amplitude of the tuning fork is detected with the x-channel of the lock-in amplifier and the output signal is kept constant by a second feedback loop which controls the amplitude of the excitation signal. This simplifies the interpretation of the different recorded signals, since the mechanical oscillation amplitude of the tuning fork can be assumed to be constant. Second, the transients are avoided that occur in response to a sudden change in the damping and could last up to seconds for high quality factors.

The PLL provides two signals that indicate the frequency shift and the excitation amplitude. Both signals can be used to control the probe sample distance by the z-

![Figure 4.11: A phase locked loop is employed to measure the frequency shift of the tuning fork. The driving signal is generated by an digital function generator employing the direct data synthesis method (DDS). The motion of the tuning fork is detected with a current to voltage amplifier and analyzed with a digital two channel Lock-in amplifier. Its output signals indicate the mechanical amplitude and the phase shift, which is used to control the frequency to the resonance. The mechanical amplitude is kept fix by controlling the amplitude of the excitation signal of the function generator.](image-url)
feedback controller.

The power dissipated in the tuning fork is the product of the current and the voltage multiplied by the cosine of the phase angle between the two signals. The phase angle is zero on the resonance and is locked by the PLL. The current is kept constant by the amplitude controller and therefore the amplitude of the excitation signal is a direct measure for the power dissipation. Any additional damping caused by probe sample interactions can be detected very sensitively in this manner. Additional power dissipations of the order of $1\mu$W have been detected. This corresponds to an energy loss of $0.2eV$ per cycle of the tuning fork with a typical oscillation energy of the order of $10^5eV$ (for $1nm$ amplitude).

4.3.3 The Low Temperature Preamplifier

In both, scanning capacitance and in Kelvin probe microscopy, very small amounts of charges are induced on the tip. This charge elevates the potential of the tip depending on the capacitance to the rest of the world. When the tip is connected via a coax cable to a current-to-voltage converter then the cable capacitance is of the order of $100 - 400pF$ depending on the length of the cable. The current-to-voltage converter detects the small rise in voltage and compensates it by letting the current flow over the feedback resistor. Only a small fraction of about $1\%$ of the charge that is induced on the tip is brought onto the gate of the first input transistor of the operational amplifier which has typical input capacitances of the order of $1pF$. The rest of the charge is used to fill the cable capacitance. To overcome this problem it is desirable to have the preamplifier as close to the experiment as possible eliminating the need of propagating very small signals over long coax cables. The low temperatures, the high magnetic fields, the limited space and the low cooling power are not forming the ideal environment for a preamplifier. However, GaAs-Field-Effect-Transistors (FETs) work even at low temperatures and when mounted parallel to the magnetic field, it is possible to operate them even at 10 Tesla. The power consumption can be reduced to reasonably low values but at the expense of the signal to

![Figure 4.12: The low temperature preamplifier allows to detect small signals very close to the experiment providing a small input capacitance. The conductance of the channel is measured with a current-to-voltage converter keeping the Source-Drain voltage fixed and thus allowing an operation at much lower dissipation power.](image-url)
noise ratio.

In figure 4.10c a tuning fork sensor with a GaAs FET connected to the tip is shown. For the detection of the tuning fork itself also a cryo-preamplifier can be used if very low oscillation amplitudes are desired.

Figure 4.12 shows the circuit used to drive the preamplifier. The conductivity of the channel is measured with a current-to-voltage converter keeping the source-drain voltage $U_{SD}$ fixed. Compared to the established source follower circuit, this has the advantage that the FET can also be operated in the ohmic regime and thus allowing an operation with smaller dissipation power. The circuit is powered by batteries and its potential $U_{tip}$ relative to common ground can be externally defined within ±100V. The output signal is optically coupled out allowing a completely isolated detection circuit. The whole circuit and with it the tip can also be modulated with frequencies up to 100kHz without affecting the output signal except for the capacitive coupling of the tip to its surrounding.

The working point of the FET can be adjusted by the Gate-Source voltage $U_{GS}$ and the Source-Drain voltage $U_{SD}$. To account for the changing properties of the FET with varying temperature and magnetic field, the characteristics can be measured in situ allowing the optimum operation point to be found by giving an additional condition like the maximum power dissipation or the minimum signal to noise ratio.

The dc-current through the channel of the FET flows over the $1k\Omega$ resistor, and the small ac-current in addition to the dc-current is taken by the current-to-voltage converter. The voltage $U_{ID}$ has to be adjusted so that no dc-current will cause the current to voltage converter to come into overload.

The application of the FET as a preamplifier close to the tip allows to detect very small signals. This is especially advantageous for the Kelvin probe experiments where any small signal has to be nulled by a bias voltage between the tip and sample. The calibration and the stability of the amplifier is not important for this application. However, for the scanning capacitance microscopy the small variations of the tip-sample capacitance have to be detected in a large background signal from stray capacitances. In this case the gain of the amplifier has to be stabilized with an accuracy better than $10^{-4} - 10^{-6}$. 
Chapter 5

Application Examples

In this chapter experimental results obtained with the two microscope setups described in chapter 3 are shown and discussed. The first section deals with the tip sample interactions in detail and in the second section the topographical imaging, the operation of the feedback loops and of the tuning fork sensor is discussed. In the third section a first glimpse on scanning gate images of a quantum wire are presented which were obtained with the Oxford microscope described in section 3.2. The forth section finally describes the first experiment obtained with the new setup (sec. 3.3 & 3.4) and is about the scanning gate microscopy of the edge channels in the quantum hall effect.

5.1 Tip Sample Interactions

The tuning fork sensor is very suitable for the investigation of the tip sample interaction forces [113]. Due to the high stiffness the tip doesn't snap into contact with the sample and the low oscillation amplitudes simplify the interpretation of the curves. Furthermore, the low temperature environment eliminates the capillary forces of the water layer and allows a very stable operation of the piczos. Force distance curves were obtained that do not show any hysteresis in the approach and retract experiments. The experiments are not only reversible but also reproducible, indicating that the tip and the sample do not undergo any modifications. This enabled us to take a series of force distance curves with different oscillation amplitudes. The metallic tip is also very suitable for the investigation of electrostatic interactions. The tip-sample capacitance as a function of the distance and the Kelvin probe response to a bias voltage are also examined in this section. These measurements lead to an experimental calibration of the tuning fork sensitivity to force gradients.

5.1.1 Force versus Distance Curves

To explore the tip sample interaction and the response of the tuning fork sensor the tip is approached very slowly to the sample surface while the frequency shift and the dissipation power are recorded. In figure 5.1 the results of four experiments are shown where an etched tungsten tip was approached to an HOPG sample. The tip was in different conditions for each experiment due to voltage pulses applied to the tip or due to controlled contact with
the sample. The four experiments presented in figure 5.1 are just a collection of different observations and not a systematic study. The tip is approached with a rate of 0.6nm/min. and at any point the direction of the movement could be turned and the tip is retracted with the same rate.

Subfigure (a) shows a force distance curve as it is expected from a van der Waals surface like graphite. The slow decrease in frequency on the first two nanometers is attributed to the electrostatic force (eq. 2.4). Both, sample and tip were grounded but the contact

![Figure 5.1: Force distance curves of an etched tungsten tip and a HOPG sample measured at 1.9K in a He gas atmosphere at 8 mbar. Different curves are obtained by modifying the tip with voltage pulses and lateral displacement on the sample. The approach and retract rate was 0.1Å/sec.](image_url)
5.1. TIP SAMPLE INTERACTIONS

potential difference causes this weak attractive force. Within the next nanometer the frequency drops about 5 Hz due to the attractive van der Waals and electrostatic forces. The frequency then starts to rise with an rate of up to 50 Hz/nm which indicates the repulsive part of the Lennard-Jones potential (eq. 2.3).

A point to note is that the noise in the experimental data is strongly enhanced when the tip interacts with the surface. This was identified to be a problem of vibrations, the noise is in fact a noise in the z-coordinate. This also limits the minimum oscillation amplitudes: an oscillation amplitude lower than the mechanical noise in z-direction doesn’t make much sense.

Subfigure (b) shows an example with a larger oscillation amplitude (6.5 nm rms). After a weak minimum the frequency starts to rise with a rate of about 3 Hz/nm. The dissipation shows interesting oscillations with a period of about 1 nm and its explanation is still an open question.

Subfigure (c) is a snapshot of a force distance curve where the tip seems to have two apexes. From the analysis of topographical pictures it is known that tips can have several apexes, see e.g. figure 5.14. It is also conceivable that a sheet of graphite sticks to the tip and causes this relatively weak interaction. Only a few fW additional dissipation power and a few 100 mHz frequency shift have been recorded.

Subfigure (d) shows an example of a force distance experiment where the curves for approach and retract do not match. This is probably due to an instability of the tip.

Figure 5.2: Force distance curves with different amplitudes measured at 1.9K. The dissipated power (top), the frequency shift (middle) are directly measured and the average force (bottom) is calculated by using a calibration of 0.8 Hz/(N/m) and integrating the curve of the frequency shift.
which undergoes mechanical deformations or due to a particle in between the tip and the sample. The strong negative frequency shift indicates that the tip must be very blunt. The large contact area causes then the strong attraction. Once the tip is too blunt no stable z-feedback controlling could be achieved anymore.

Figure 5.2 shows the results of the approach retract experiments that have been repeated with different oscillation amplitudes (peak) between 0.56nm and 2.0nm. At a given $z$, larger amplitudes lead to smaller frequency shifts in accordance with the theoretical results in ref. [50]. The minimum in $\Delta f$ shifts to larger $z$ and becomes more shallow when the amplitude is increased. With a calibration of $0.8\text{Hz/}(\text{N/m})$ [114] (sec. 5.1.2), the frequency shift can be converted into a force gradient and by integration the average force as a function of the distance is obtained (see fig. 5.2).

When the tip is approached to the surface the dissipation power starts to increase into the pW-range. For larger tip-oscillation amplitudes the onset of the dissipation is at larger tip-sample separations. Even at the smallest tip-sample amplitudes the onset of the power dissipation occurs at a tip-sample separation at which the frequency shift is negative and the tip experiences an overall attractive force. The foremost tip atoms start to come into intimate contact with the surface atoms and in addition to the conservative tip-sample interaction forces there occur dissipative phenomena. It is believed that these are mainly phonon emissions into the sample and the tip. It is interesting to notice that the power increase occurs at a tip sample spacing, where other authors have reported true atomic resolution [75].

5.1.2 Electrostatic Interactions

The electrostatic interactions of the tip with the sample are important for the investigation of buried electronic structures. First the capacitance as a function of the tip sample spacing is measured and then the Kelvin probe response is examined. The electrostatic forces are then used to calibrate the frequency shift of the tuning fork.

Capacitance versus Distance Curve

The capacitance as a function of the tip sample separation $C(z)$ determines completely the electrostatic interaction of the sample and the tip. This interaction manifests itself by the electrostatic forces and by induced currents or charges on the tip or the sample. The capacitance accounts for the geometrical aspects and the material properties. Theoretical models for different tip geometries have been calculated in ref. [49, 55].

As a very simple model three capacitors in parallel are assumed: $C_0$ is a stray capacitance that originates in cable crosstalk and does not depend on the tip-sample distance. $C_1(z)$ is a stray capacitance between the hole sample and the electrical connection of the tip. The mean spacing of the two electrodes is much larger than the variations by the approach experiment and the capacitance can be approximated by a first order expansion $C_1(z) = C_1(z_0) + C_1'(z_0)(z-z_0)$. The third capacitor is the interesting capacitance between the tip and the sample and will be modeled by an inverse power law $C_2(z) = P_2/(z-z_0)$, with $P_2$ being a parameter accounting for the effective area and $z_0$ is the position where the tip starts to touch the sample as verified for example by a tunneling current. Thus the
5.1. TIP SAMPLE INTERACTIONS

Figure 5.3: Tip-sample capacitance $C(z)$ as a function of the tip-sample spacing $z$ measured at 1.9K. An ac voltage of 100mV (rms) at a frequency of 95kHz has been applied to the sample and the current induced on the tip has been measured with a current to voltage converter. The calibration of the capacitance measurement was done at $z = 330$ nm where an offset capacitance of 231.5fF was compensated with a calibration capacitor in a bridge circuit. The contact between the tip and sample occurred at $z_0 = 351$ nm.

The experimental curve in figure 5.3 can be approximated by the three parameter expression

$$C(z) = P_0 + P_1(z - z_0) + P_2 \frac{1}{z - z_0}$$  \hspace{1cm} (5.1)

Of course much finer models as given in ref. [49] and [55] can be applied but here only an analytical curve is needed in order to allow a better determination of the second derivative for the purposes of the calibration procedure in the section after the next.

**Kelvin Probe**

For the experiment illustrated in figure 5.4 the tip is placed at a distance $d = 200$ nm from the sample surface. The tuning fork is driven on its resonance frequency by the PLL and the tip oscillation amplitude is controlled to be $A = 30$ nm. The bias voltage $U_{\text{Bias}}$ is varied slowly from -10V to +10V (at 10V/min.) while the frequency shift $\Delta f$ and the current induced on the tip $I_{\text{tip}}$ are recorded.

Since $A/d < 1$, the frequency shift is proportional to the force gradient (eq. 2.8). The experimental data is thus very precisely described by equation (2.20), which predicts a parabola with the vertex shifted by the contact potential difference. In our measurement we find $U_{\text{CPD}} = 64$ mV.

On the other side the complex amplitude of the tip current is plotted in the complex plane as a function of the bias voltage. The data produces a linear curve as expected from equation (2.16) but does not cross zero. This is due to a capacitive coupling of the tuning fork excitation signal to the tip. This capacitively induced current on the tip has a phase of 90 degree to the excitation signal. The Kelvin current (2.16) however, is in phase with the velocity of the tip which is further in phase with the tuning fork current (eq. 4.2). The tuning fork current on the resonance is again in phase with the excitation signal. Finally the Kelvin probe signal and the stray signal from the excitation are separable by the
phase shift of 90 degrees. In figure 5.4 the stray signal is indicated by the red line which has a phase shift given by the preamplifiers.\footnote{A phase shift of 180 degree is expected for an ideal current-to-voltage converter.}

**Calibration of the frequency shift**

Once the capacitance as a function of the distance $C(z)$ is known, all the electrostatic interactions of the tip and the sample are determined. The derivative of the capacitance allows to determine the electrostatic force acting on the tip (eq. (2.17): $F_z = \frac{1}{2} C''(z) U_e^2$) and the second derivative determines the force gradient (eq. (2.20): $dF_z/dz = C''(z) U_e^2$). Here $U_e$ denotes the electrostatic potential $U - U_{CPD}$. It is thus possible to calibrate the tuning fork by registering the frequency shift in response to a bias voltage producing a known electrostatic force gradient. The experiment in fig. 5.3 resulted in a value of about 1Hz/(N/m).

The second approach for determining the second derivative of the capacitance employs the measurement of the Kelvin current (fig. 5.4) which determines directly the derivative of the capacitance (eq. 2.16). The second derivative can be obtained by repeating this measurement as a function of $z$. The experiment of figure 5.4 resulted in a value of 0.8Hz/(N/m).

An accurate calibration of the frequency shift with the first method relies on the calibration of the $z$-movement of the scan piezo and on the calibration of the current...
5.1. **TIP SAMPLE INTERACTIONS**

measurement. For the second method the calibration of the tuning fork amplitude and the calibration of the current measurement is important.

With the simple mechanical spring-mass model introduced in section 4.2.2, the frequency shift can be calculated with equation 2.8. The value obtained in this way is $0.58\text{Hz}/(\text{N/m})$, in acceptable agreement with the values obtained with the electrostatic force.

The Kelvin method has the advantage that only the capacitance of the oscillating tip is measured. The parts of the electrical connections of the tip that do not oscillate will not contribute to the Kelvin current. The Kelvin current accounts exactly for that part of the electrostatic interaction that causes the frequency shift. For the direct capacitance measurement however, the model with the three capacitors has to be applied in order to discriminate the tip capacitance from other stray capacitances.

**Conclusion**

The electrostatic interactions of the tip and the sample was measured experimentally. The capacitance as a function of the tip sample spacing $C(z)$ fully determines the electrostatic interaction and was measured by a direct admittance measurement. The Kelvin current induced on the oscillating tip in response to a bias voltage was measured and the derivative of the capacitance was determined. Finally also the frequency shift in response to bias voltage producing a force gradient was measured and the second derivative of the capacitance could be determined. With the knowledge of the capacitance function the electrostatic forces are known and the tuning fork response could be calibrated.
5.2 Topographical Imaging

5.2.1 Evaporated Gold Films

Evaporated gold surfaces are popular test surfaces for scanning probe microscopy. They consist of granules with typical diameters of 10 to 100 nm depending on the conditions in the evaporation process. Figure 5.5 shows two examples of images recorded on such gold surfaces at 2K and 300K respectively. On the left side a sample inspected at low temperature and on the right side a sample inspected under ambient conditions is shown. The two experiments have been performed with two different samples and tips. The topography, the frequency shift and the excitation amplitude, which is proportional to the dissipation power (sec. 4.3.2) are recorded simultaneously. The z-feedback controller controlled the frequency shift to a positive value of 200 mHz and 50 mHz respectively. The oscillation amplitude was about 1 nm and the scan velocity was 300 nm/s for both cases.

An obvious difference is that on the left side the topography image is sharp whereas on the right side the topography looks blurred but the frequency shift image looks more plastic than the one on the left side. This is due to different feedback parameters for the

![Figure 5.5: Two different samples of gold evaporated on a Si chip which shows typical granules. Left: Image recorded at 2.0 Kelvin. Right: Image recorded under ambient conditions.](image)

...
two experiments: for the example on the left side the bandwidth for the frequency demodulation was much smaller but the bandwidth for the z-feedback controller was higher. This results in a lower noise of the frequency shift and a better imaging of the topography. In the experiment on the right the z-feedback is too slow to follow all the details of the topography but the frequency shift shows all the fine details. For a lower scan speed however, the topography would be depicted very well and the frequency shift image would become more flat.

The z-feedback controller is mainly an integrator and therefore the error signal, which is the frequency shift, is the derivative of the z-signal (topography) in scan direction. This leads to the three dimensional effect, as if the surface were illuminated from left. The faster the z-feedback controller can correct the frequency deviations the flatter the image of the frequency shift will turn out. A slow z-feedback controller causes larger deviations of the tip sample distance. Therefore the dissipation image on the right side shows an artefact because the dissipation power depends very strongly on the tip sample distance. The dissipation image on the left side however, shows bright structures at the grain boundaries. This could have two reasons: first, the dissipation could be enhanced when the tip is in a depression because the contact area is larger, or second, the attractive forces are increased when the tip is in a depression and therefore the z-controller has to bring the tip closer to the surface to maintain the setpoint frequency shift. In any case it seems to be a geometrical effect of the tip and the sample, not an effect due to varying material properties.

As discussed so far the bandwidth of the frequency demodulation should be as high as possible. The z-controller which uses the frequency shift as the error signal must have a smaller bandwidth than the frequency demodulation, but also as high as possible to allow for fast scanning. However, the signal-to-noise-ratio (SNR) increases with decreasing bandwidth and a compromise between speed and noise has to be found. Second the system may become unstable and starts to oscillate when the gain of the feedback controller is too high. Since in this example the three feedback loops are nested in a rather complicated manner it is difficult to give an explicit recipe for the feedback parameters for which no quantitative statements can be given here. The parameters have to be found for each experiment individually. It depends strongly on the tip sample interaction and therefore on the materials and the geometry of the tip and the sample. The environment (UHV, low temperature, air) also affect the tip sample interaction. It further depends on the scan piezo, its deflection constant and its resonance frequency. Furthermore it depends on the quality factor of the tuning fork which also defines a bandwidth of $\omega_0/Q$. The amplitude of the oscillation of the tuning fork and the noise of the electronic detection determines the signal to noise ratio of the deflection detection. The low pass filters and the PID-controllers determine then the signal to noise ratio of the frequency demodulation. A rigorous theoretical analysis of the feedback controllers could be applied or an automated self optimizing multi-input multi-output (mimo) controller could be employed which adapts the parameters to fit a given criterion, for example to minimize the phase deviations in first priority and the frequency deviations with a second priority for a given oscillation amplitude, scan speed and surface roughness.
5.2.2 Semiconductor Nanostructures

The example shown in figure 5.6 is an image of a typical sample to be investigated with the microscope that has been developed. The image was acquired under ambient conditions and the scan range was nominally 20 by 20 μm. It is strongly distorted due to creep and probably also due to a partly depolarization of the scan piezo. The fast scan direction was from bottom to top and the slow scan direction was from left to right. The vertical bright stripes arise from an overshooting of the z-controller when it has to climb the 80 nm high mesa edge. The dark spots on the mesa are roughly 0.5 nm deep depressions that arise from defects in the crystal. The white lines are oxid lines written by local anodic oxidation with another AFM [115].

At low temperatures the scan range is limited to 5 μm and the interesting places have to be found by coarse positioning the probe on the sample (sec. 3.4.2). Figure 5.7 shows an antidot lattice defined by AFM-lithography on top of a Hall bar. The search of the place of interest took more than two days.

The dissipation images in figure 5.7 indicate a reduced dissipation when the tip is on top of an oxide dot. This could be due to different material properties but is more likely again a geometrical effect as described in section 5.2.1. The close-up which was acquired at a slower scan speed of (300 nm/s) compared to the full scale image (2 μm/s), shows an instability of the tip, most clearly visible in the dissipation image. It may be explained by the pickup of a small grain of oxide. Furthermore, a periodic disturbance causes the diagonal stripes in the frequency shift and the topography image. It is not visible in the

Figure 5.6: A AlGaAs heterostructure has been processed with standard lithographical processes to define the mesa (gray). With a scanning probe microscope the two dimensional electron gas is then cut into several isolated areas by local anodic oxidation of the surface. The bright lines are oxid lines and underneath the 2DEG is depleted. With this sample, two quantum wires, each 5 μm long and respectively 300 nm and 500 nm wide, can be measured by four point transport experiments. The wire can be influenced in size and in position by the isolated areas forming side gates. The sample was fabricated by Silvia Lüscher.
5.2. TOPOGRAPHICAL IMAGING

Figure 5.7: Antidot lattice defined by AFM-lithography on top of a Hall bar and imaged at 1.7K. The sample was fabricated by Ryan Held.

The full scale image since the bandwidth was larger and the noise covers the small disturbance.
5.2.3 Nuclear Pore Complex

As the last example for topographical imaging, a membrane of the nucleus of a frog egg is imaged. This is not what the microscope is intended for, but it demonstrates the capability of the tuning fork sensor on soft matter. Nuclear pore complexes (NPCs) are the rate limiting barriers for the exchange of macromolecules (e.g. transcription factors or mRNA) between the nuclear and cytosolic compartments. The NPCs represent supramolecular structures with an estimated molecular mass of 124MDa per NPC. Each NPC has a central channel, about 30-50nm in length and highly variable in width through which macromolecules can enter and exit the nucleus. NPC conformation determines the movement of cargo in either direction and thus controls the gene expression. The topographical image in figure 5.8 shows clearly several NPCs which appear as rings of about 100nm diameter. In the image a NPC that seems to be closed, is marked with an arrow. It is known that in the presence of calcium the NPCs open and in the absence of calcium the NPCs close the channel [116, 117].
5.3 Scanning Gate Microscopy of a Quantum Wire

Quantum wires in the quasi-ballistic regime, where the mean free path of the carriers is longer than the width but shorter than the length of the wire, are of principal interest to mesoscopic transport physics [18]. In transport experiments universal conductance fluctuations [118] and the ballistic "wire-peak" phenomenon [115] could be observed. The ability to have an additional perturbation potential, that can be moved around in the wire, promises exciting new insight into the electron transport of a mesoscopic conductor [119]. This is exactly what can be done with scanning gate microscopy: a voltage is applied to the tip while scanning it over the sample and with it the induced screening charge in the sample.

First, the theoretical background of the quasi-ballistic electron transport in quantum wires is shortly reviewed, followed by a description of the experimental situation for scanning gate experiments on AlGaAs-GaAs heterostructures. Then the description of the quantum wire sample for a scanning gate experiment is given, which is reported and the data discussed in the following. Finally the conclusions and an outlook are given.

5.3.1 Quasi-Ballistic Transport

Drude theory

In the semi-classical Drude theory the transport of electrons is characterized by the mobility of the electrons, which relates the steady-state drift velocity

\[ v_D = \mu E \]  

with the applied electric field \( E \). The mobility is further related to the Drude momentum relaxation time \( \tau_D \)

\[ \mu = \frac{e\tau_D}{m^*} \]  

with \( m^* \) being the effective mass. The mean free path is then

\[ l_D = \tau_D v_F \]  

with \( v_F \) the Fermi velocity \( (v_F = \frac{\hbar}{m^*} \sqrt{2m^* n_s} \) in 2D). In a two dimensional conductor with an electron density \( n_s \), the resistivity is then given by

\[ \rho = \frac{1}{en_s \mu} \]  

and for a conductor of width \( W \) and length \( L \) the resistance \( R \) is

\[ R = \frac{L}{W \rho} \]  

At low temperatures phonons are frozen out and the mean free path \( l_D \) is determined by the impurities in the sample. The advances in molecular beam epitaxy led to electron systems with very long mean free path (up to 100 \( \mu m \)). If the width of a conductor is smaller than the mean free path, boundary scattering becomes important for the transport as indicated in figure 5.9. Specular boundary scattering will not contribute to a momentum
Figure 5.9: Characteristic electron trajectory for the case of specular boundary scattering in a quasi-ballistic wire. The mean free path \( l_D \) is longer than the width \( W \) but shorter than the length \( L \) of the wire.

relaxation in transport direction and has no influence on the resistivity. Diffusive boundary scattering, however, will contribute to the resistivity as worked out in [18]:

\[
\rho = \frac{\pi \rho_0}{2} \frac{l_D}{W \ln(l_D/W)}.
\]  

(5.7)

**Phase Coherence Length**

Beyond the Drude picture, the electrons are described by quantum mechanical wavefunctions which are subject to interference within a region where their phase is well defined. Phonon-electron and electron-electron scattering randomize the phase, so that the electrons "loose" their phase coherence over a characteristic length, the phase coherence length \( l_\phi \). Elastic electron-impurity scattering which defines \( l_D \), has no effect on the phase coherence length of the electrons. At low temperature (\( \approx 1 \) K) phonons freeze out, electron-electron scattering dominates the phase-randomizing process, and \( l_\phi \) can become very large (\( \gg l_D \)). As long as \( l_\phi \) is much bigger than \( l_D \), the electrons move along a diffusive two-dimensional path and the relation between \( l_\phi \) and the phase relaxation time is given by

\[
l_\phi = \sqrt{D \tau_\phi},
\]  

(5.8)

with the diffusion constant \( D = \frac{1}{2} l_D v_F \). At non-zero temperatures the Fermi function is smeared out meaning that electrons of up to a few \( k_B T \) away from \( E_F \) take part in transport. Interfering paths with an energy difference \( k_B T \) become uncorrelated after a distance

\[
l_T = \sqrt{\frac{hD}{k_B T}}.
\]  

(5.9)

the thermal length.

**Universal Conductance Fluctuations**

When the phase coherence length of the electrons exceeds the length of the wire shown in figure 5.9, its resistance will depend on the exact configuration of the elastic scatterers due to the interference of different paths (fig. 5.10). For an ensemble of different configurations the magnitude of the conductance fluctuations is characterized by the standard deviation of \( G \)

\[
\delta G = \sqrt{\langle (G - \langle G \rangle)^2 \rangle}
\]  

(5.10)

is of the order of \( \frac{e^2}{h} \), hence the name *Universal Conductance Fluctuation* (UCF). Experimentally the universality is disturbed by various effects. These fluctuations have been measured in mesoscopic conductors as a function of the magnetic field [120, 121].
5.3. SCANNING GATE MICROSCOPY OF A QUANTUM WIRE

Figure 5.10: Intuitive model for the coherence of electrons. Circular waves spreading out from \( N = 100 \) randomly placed centers are reflected by the walls. The scalar wave function is \( p(r) = \cos(\frac{2\pi r}{\lambda_F}) \exp\left(-\frac{r^2}{2\sigma^2}\right) \) for the distance \( r \) from a single scatterer. The scatterers are randomly placed in the wire of width \( W = 500 \text{nm} \) and \( L = 5 \mu\text{m} \). The "Fermi wavelength" is \( \lambda_F = 50 \text{nm} \), the "phase coherence length" is \( l_\phi = 2 \mu\text{m} \). The scatterers are mirrored at one boundary and the configuration then periodically continued crosswise to the wire up to the phase coherence length, and the superposition of all scatter wave functions then calculated.

Phase coherent conductance of a quasi-ballistic wire is highly sensitive to small changes of the position of one impurity, as calculated by Altshuler and Spivak [122] and experimentally confirmed by Feng et al. [123].

5.3.2 Scanning Gate Microscopy on AlGaAs-GaAs Heterostructures

Two-dimensional electron gases

An electron gas with a constriction in one of the three dimensions, which is strong enough to lead to an energy quantization in the range of the Fermi energy, is called a two-dimensional electron gas (2DEG). Such 2DEG's appear at the interface of Al\(_x\)Ga\(_{1-x}\)As and GaAs, \( x \) denoting the Al-concentration, on which the band-gap of AlGaAs depends. Figure 5.11a shows the semiconductor layer-structure (heterostructure) grown with molecular beam epitaxy (MBE). The self-consistent solution of the Poisson equation for the potential and the Schrödinger equation for the wave function is shown in figure 5.11b. Because the 2DEG within the almost perfectly crystalline GaAs is spatially separated form

Figure 5.11: (a) Semiconductor layer-structure containing a 2DEG grown with MBE. Si-atoms in a sheet 17 nm away from the interface form the donors. (b) Band structure and electron wave function in the semiconductor heterostructure calculated self-consistently.
the donor layer, very high electron mobilities are achieved. Due to the confinement in one direction, the continuous energy spectrum for the motion along that direction collapses into discrete levels (subbands). The density of states in such a subband does not depend on the energy, its value is given by

\[ D(E) = \frac{m^*}{\pi \hbar^2} \]  

leading to the Fermi wave vector

\[ k_F = \sqrt{2\pi n_s} \]  

where \( m^* \) is the effective mass of the electrons and \( n_s \) is the sheet-density of the 2DEG.

To fabricate mesoscopic systems an appropriate shape of 2DEG is cut out of the layers by optical lithography and a wet chemical etch process. Gates on top of the structure can be defined by a lithographical lift-off process. Additional structuring below the optical resolution is done by electron beam lithography, local anodic oxidation with an AFM [115] or focused ion-beam.

### Electrostatic Interaction with a Metallic Tip.

To investigate the mesoscopic systems defined on a heterojunction, the charged tip is scanned over the structure where it produces a small electrostatic disturbance which will, for example, influence the transport properties. Two problems have to be solved for the understanding of the results of such an experiment.

(i) How does the charged tip influence the electrostatics in the sample, how is the electric field screened and what is the electron density distribution?

(ii) How does the induced local electrostatic perturbation influence the transport properties? This is the scientifically important part of the problem, since the theories for transport in mesoscopic systems are still under development and such experiments promise new insights.

For the first problem (i) one can solve the three-dimensional Poisson and the Schrödinger equations self-consistently as it is done for the one-dimensional bandstructure calculation in figure 5.11b. The assumption of a cylinder-symmetrical tip reduces the problem to two dimensions. When the 2DEG is assumed to be fully metallic it will screen any electrical field. For this case the method of the virtual image tip replacing the 2DEG can be applied as indicated in figure 5.12. The induced screening charge is related to the electric field \( E \) by \( \Delta n_s = E/4\pi \). The high dielectric constant discontinuity of GaAs (\( \varepsilon \approx 13 \)) to vacuum reduces the apparent depth of the electron gas beneath the surface. Eriksson et al. calculated the density perturbation \( \Delta n(r) \) in the 2DEG and found an empirical formula[21],

\[ \Delta n(r) = \frac{n_s}{1 + (r/d)^2} \left( \frac{V}{V_d} \right) \]  

for \( r \) the radial distance to the tip, \( n_s \) the undisturbed sheet density, \( d \) the depth of the 2DEG below the surface, \( V \) the potential of the tip and \( V_d \) the potential of the tip where the

\(^2\)Spatial separation prevents the electrons in the 2DEG from being strongly scattered at ionized donors
5.3. SCANNING GATE MICROSCOPY OF A QUANTUM WIRE

The tip induces a screening charge in the two-dimensional electron gas. As long as the 2DEG is regarded as a metallic layer, the electric field can be calculated using a virtual image tip. The dielectric constant of GaAs is about 13 which causes the electric field lines to spread out inside the sample.

2DEG starts to deplete and depends on the radius of the spherical tip apex. However, when the tip is irregularly shaped the induced charge distribution will be irregularly shaped as well. Furthermore the influence of the donor layer has to be considered. Even the smallest conductivity in this layer would screen the electric field of the tip since it scans very slowly over the surface. The situation gets complicated when the 2DEG is depleted under the tip. This is the case when the induced positive screening charge exceeds the density of negative charge $n_a$. The hole in the 2DEG, also called an antidot, is transparent for electric field lines and the total screening charge is reduced. Similar effects occur when the tip is over an oxid line or near the mesa edge, where the 2DEG underneath is depleted and cannot screen the tip (fig. 5.12). Furthermore the density of states of the 2DEG is of importance. In the quantum Hall effect (sec. 5.4), the local density of states can be zero at the Fermi level and the 2DEG is transparent for the electric field.

A theory based on the convolution with a transfer function of the tip as it has been demonstrated by Hug et al. [6] for the magnetic force microscopy, is much more problematic in scanning gate microscopy since the distribution of the screening charge depends also on the properties of the sample. However, for an extended 2DEG and for small density perturbations the transfer function of the tip is well defined. Experimentally, one can measure the capacitance (sec. 5.1.2) which determines the total charge induced in the 2DEG.

For the second problem (ii), several approaches exist. Non-coherent, ballistic conduc-
tors can be modeled by solving the classical equation of motion for the electrons [119]. The statistics of an ensemble of orbits determines then the transport properties. More advanced models involve recursive Greens functions or scattering matrices and are beyond the scope of this work [19, 18].

5.3.3 Experiment

Sample

The two-dimensional electron gas (2DEG) in the GaAs-Al$_x$Ga$_{1-x}$As ($x = 0.3$) heterostructure is 37 nm below the surface. The 2DEG has a sheet density of $N_s = 5.5 \times 10^{15} \text{ m}^{-2}$ and a mobility of $\mu = 92 \text{ m}^2/\text{Vs}$ at a temperature of $T = 1.7 \text{ K}$. The mean free path is therefore $l_D = 11.2 \mu\text{m}$ and the Fermi wavelength is $\lambda_F = 33.8 \text{ nm}$. The thermal length is $l_T = 2.5 \mu\text{m}$ at $2 \text{ K}$ (eqn. 5.9). The phase coherence length at $2 \text{ K}$ is not known but assumed to be shorter than the thermal length due to electron-electron and electron-phonon scattering.

On top of the heterostructure a Hall bar shaped mesa, defined by photo lithographical techniques, a $L = 40 \mu\text{m}$ long and $W = 400 \text{ nm}$ wide quantum wire has been patterned by AFM-lithography (fig. 5.13). The structuring of semiconductor surfaces by local anodic oxidation, called AFM-lithography is described in ref. [54, 115] and section 5.2.2.

The four-terminal resistance of the wire was measured with standard lock-in technique at a frequency of about 400 Hz to be $R = 3.3 \text{ k}\Omega$ ($G = 1/R = 7.8 \times 10^{5} \text{ } \Omega^{-1}$). Applying a voltage to the two isolated regions of the 2DEG forming in-plane sidegates (IPG), the wire can be influenced in position and density [118].

The depletion length for oxide structures is typically $15 - 20 \text{ nm}$. The number of modes in the channel can therefore be estimated to be $N = 21$. The mean free path can be estimated using $R = h/(2e^2N)(1 + L/l_D)$ [19] to be $l_D = 9 \mu\text{m}$.

Assuming an unchanged 2D-density in the wire, the number of electrons in the wire is about 80'000. The density of ionized donors is higher then the density of electrons in the

Figure 5.13: A quantum wire defined by local anodic oxidation with a scanning probe microscope on a AlGaAs heterostructure. The wire is 40 µm long and nominally 400 nm wide and can be measured by four terminal transport experiments. This sample was fabricated by Ryan Held.
5.3. SCANNING GATE MICROSCOPY OF A QUANTUM WIRE

2DEG, because the surface is negatively charged (fig. 5.11) and therefore the number of remote ionized scatterers is about $10^5 - 10^6$.

Universal conductance fluctuations have been measured on a similar sample at temperatures of 100 mK as function of magnetic field and of the lateral shift induced by antisymmetric sidegate-voltages [118].

Measurement

The sample with the quantum wire was mounted in the microscope (sec. 3.2) and then cooled to 1.7 K. The metallic probe was positioned on the entrance of the quantum wire as indicated in figure 5.13. The top row of the images in figure 5.14 shows a repeated scan of the topography. The tip is an etched tungsten tip, that seems to be in a bad condition. It may be that frozen contaminations on the tip and probably also a mechanical deformation due to a tip crash caused the tip to be very blunt. The double image of the oxid lines is due to a double tip with a spacing of about 1.5 µm and is in the direction of the wire. A scratch resulting from a tip crash in a former experiment on this sample is visible as a diagonal structure in the lower left part of the images. However, the repeated scan of the topography shows nearly identical images, indicating a stable tip and stable piezos.

The resistance of the wire has been measured simultaneously by driving a current of 20 nA through the wire at a frequency of 421 Hz. The voltage drop over the wire is measured with a time constant of 10 ms, conform to the scan speed of 500 nm/s. The in-plane gate IPG2 did not shown any influence on the wire resistance, probably due to bad wire bonding whereas IPG1 showed a clear influence on the wire resistance and no leakage current for gate voltages of $U_G = \pm 200$ mV.

The images corresponding to the resistance of the wire as a function of the tip position are shown in the bottom row of the images in figure 5.14. The sidegate IPG1 were kept on a voltage $U_G = -200$ mV for the left image and $U_G = +200$ mV for the right image. The influence of the gate voltage on the wire is visible in the transport images. On the left side, when the gate voltage is negative and the density in the wire is reduced, the scanning gate image shows a much brighter and wider structure. Figure 5.15 shows a cross section of the images in figure 5.14 indicated by the blue line. For a negative gate voltage the background resistance is higher due to the decreased density.

In addition to the scanning gate mode, many other modes are feasible. For example the differential mode used to depict the image shown in figure 5.16. The tip height $z$ is oscillating and the induced screening charge $q$ in the sample also oscillates:

$$q(t) = q_0 + \frac{\partial C}{\partial z} z(t)V$$

(5.14)

Where the $C(z)$ is the tip sample capacitance determining the electrostatic interaction and $V$ is the tip potential. The oscillating screening charge will cause the resistance $R$ to do so as well. Driving a dc current through the wire and measuring the potential drop over the wire with a lock-in amplifier at the frequency of the tuning fork oscillation, yields the quantity $\frac{\partial R}{\partial z}(x, y)$, imaged in figure 5.16. Much finer structures than in the scanning gate pictures in figure 5.14 are resolved.

3The positioning with the xy-table (sec. 3.4.2) took over two day.
Figure 5.14: The wire resistance as a function of the tip position at 1.7K. The sidegate voltage influences the scanning gate pictures. Left: $U_\Omega = -200\text{mV}$, Right: $U_\Omega = +200\text{mV}$, $U_{tip} = 0\text{V}$.

Figure 5.15: Left: The resistance of the wire as a function of a line-scan of the tip across the wire indicated by the blue line in figure 5.14. Right: The same data but shown as conductance in units of $\frac{1}{e^2}$. 
5.3. SCANNING GATE MICROSCOPY OF A QUANTUM WIRE

These images show the first experimental results on a mesoscopic device obtained with the "Cryo-SXM" built-up and operated during this thesis. Unfortunately the sample was damaged after this first images and the exploration of the experimental techniques ended abruptly. For future experiments it is important to perform also magneto-transport measurements in order to allow a detailed characterization of the wire (density, mobility, phase coherence length). The dependence of the scanning gate images on the tip voltage and on the magnetic field will be important for the discussion of phase coherent transport.

Discussion

The displacement of the electrical image relative to the topographical image of about 400 nm is explained by two different apexes on the tip. The foremost apex of the tip which is responsible for the topographical imaging is not the same as the electrically active part. It is possible that the frozen contaminations on the tip are insulating but mechanically robust enough to allow sensing the surface. The metallic tip, which forms the scanning gate, is displaced by about 400 nm perpendicular to the wire. It may also be possible that a charged contamination causes the scanning gate image while the metallic tip is sensing the surface. However, the displacement of the topographical and electrical images proves, that the mechanical interaction of the surface sensing mechanism on the wire, does not produce a detectable change in the wire resistance, as it could be the case due to the piezoelectric effect of GaAs.

Contact potentials between the 2DEG and metallic gates of up to 0.8 V have been reported [31]. Here the tip was grounded and hence reduces the density. An electrostatic tip voltage of $-0.3$ V has been reported to deplete the 2DEG under the tip in a heterostructure with much deeper 2DEG. It is therefore assumed that the tip here locally depletes the 2DEG.

When the side-gate voltage is negative and the density in the wire is reduced, the
scanning gate image shows a much brighter and wider structure. This is plausible since the lower density reduces the ability of the electrons in the wire to screen an external potential and the tip will more easily deplete the wire. On the right side, when the gate voltage is positive, the density of electrons in the wire is higher and the protrusion of the screening potential through the Fermi energy will form a smaller antidot.

For a homogeneous and continuous wire one would expect to have a response in the resistivity independent of the tip position along the wire. In figure 5.14, however the signals varies considerably along the wire. The fact that the structures in the scanning gate images, show a repeated elliptic pattern with about 30 degree to the wire direction, indicates a folding process with the shape of the tip. It seems that hot spots, where the wire resistance is very sensitive to the tip potential, produce a repeated pattern of the tip. As already noted in the former section the tip is very blunt and in the direction of the wire about 1.5 μm extended. A multiple imaging of the hot spots in the wire has to be considered and the spacing of hot spots can therefore not be measured.

The scanning gate image on the left side in figure 5.14, where the gate voltage is negative ($V_G = -200 \text{ mV}$), shows "holes" inside the structure of the wire response. It is not clear, whether this is an experimental artifact due to an overload of the lock-in amplifiers or real.

The experimental results presented in figure 5.14 are hard to interpret in a quantitative manner because the tip is in a to bad condition and the experimental conditions are not well enough defined.

5.3.4 Conclusions and Outlook

The proof of concept could be brought for the scanning gate microscopy of a quantum wire and fascinating images of the quantum wire could be recorded at low temperatures. The first glimpse on the scanning gate images of quantum wire are motivating for further work in this direction. The results are promising, that as soon as the experiment can be performed with a clean and sharp tip on a clean sample, allowing imaging with the quality as shown in figure 5.6, the experiments will reveal many facts about the ballistic transport in quantum wires.

For the measurement modes discussed so far, the tip acts as an actuator that influences the transport properties of the quantum wire. Even more promising are techniques where the tip is used as a sensor. With the Kelvin probe, the potential drop along the current carrying wire, or the potential fluctuations due to disorder could be imaged. With capacitance spectroscopy one could try to investigate the local density of states. To investigate the local density of states along the wire, tunneling spectroscopy is the ultimate measurement mode. For that direct tunneling into the electron system is necessary and it could be realized on a quantum wire defined by anodic oxidation of InAs quantum wells that can be formed at the surface.
5.4 Imaging the Inter Edge State Tunneling

5.4.1 Quantum Hall Effect and Edge States

When a magnetic field is applied perpendicular to the plane of the two-dimensional electron gas, traveling electrons are confined to orbits because of the Lorentz force acting on them. The Hamiltonian for electrons in a magnetic field can be written as

\[ H = \frac{(p + eA)^2}{2m^*} + V(z), \]  

(5.15)

e neglecting the spin energy. For the vector potential it is useful to apply the Landau gauge \( A = (0, Bx, 0) \), \( B = \nabla \times A = Be_z \). The movement in \( z \)-direction is separated off and the Hamiltonian for the movement in the \( xy \)-plane can be written as

\[ H = \frac{p_x^2}{2m^*} + \frac{m^*\omega_c^2}{2}(y - y_0)^2, \]  

(5.16)

with

\[ \omega_c = \frac{eB}{m^*}, \]  

(5.17)

the cyclotron frequency and \( y_0 = l_B^2 \) with the magnetic length

\[ l = \sqrt{\frac{\hbar}{eB}}. \]  

(5.18)

The energy eigenvalues for the Hamiltonian (5.16) are

\[ E_n = \hbar\omega_c \left(n + \frac{1}{2}\right), \]  

(5.19)

and the wave functions associated with the eigen states are plane waves in \( x \)-direction with wave vector \( \frac{y_0}{l} \) and harmonic oscillator wavefunctions in the \( y \)-direction. Each of the levels is highly degenerate, per unit area there are

\[ N_L = \frac{e}{\hbar}B, \]  

(5.20)

\[ \text{Figure 5.17: Left: The density of states collapses to \( \delta \)-functions in the magnetic field. Due to scattering the Landau-levels are broadened. Right: At the edge, the Landau levels are bend up and where they cross the Fermi energy one dimensional unidirectional channels are formed.} \]
states per Landau level. The filling factor \( \nu \) is defined as the number of filled Landau levels

\[
\nu = \frac{n_e \hbar}{Be}
\]

and when ever this is an integer number, e.g. the Fermi energy is between two Landau levels, the interior of the sample is insulating and the current is carried by the one dimensional edge states with quantized conductivity

\[
\sigma = \nu \frac{e^2}{h}
\]

leading to the quantum Hall effect \cite{25}.

Figure 5.18: Left: Classical skipping orbits. Electrons are localized in the middle of the sample (A), but can travel from one end to the other at the edge, where the potential rises above the Fermi level (grey areas C), on skipping trajectories (B). Right: Quantum mechanical edge states are formed where the Landau levels intersect the Fermi-energy.

5.4.2 Sample

On a AlGaAs heterostructure, similar to that described in section 5.3.3, a mesa is defined by photo lithographical techniques as shown in figure 5.19. It is a circle of about 400 \( \mu \text{m} \) in diameter and has four U-shaped holes for inner contacts with a narrow link to the rest of the 2DEG. Six voltage probes lead to the terminals shown in the upper part of figure 5.19(P1-P6) and serve for the characterization of the sample by four terminal magneto transport measurements. All the contacts are then evaporated with an eutectic Gold-Germanium alloy and annealed to form ohmic contacts to the 2DEG. Finally the Gold gate, which goes U-shaped around the inner contacts, is brought onto the sample. Around the 20 \( \mu \text{m} \) diameter hole in the center, the edge channel to be investigated will be formed in the quantum Hall regime. The idea of the sample structure is that the ungated region is brought to an integer filling factor \( \nu \) by a high magnetic filed and the density of the gated region is then lowered, until a smaller integer filling factor is reached. In this situation, the inner contacts are isolated from each other, except for the tunneling current between the edge channels around the hole in the center. This situations is illustrated in figure 5.20, where the green lines correspond to the edge channel of the first Landau level and the yellow lines correspond to the second Landau level. The transport from contact A to contact B, measured in a two terminal configuration, involves then the tunneling from the yellow edge channel of contact A to the green edge channel around the hole and a further tunneling process to the yellow edge channel of contact B.
5.4. IMAGING THE INTER EDGE STATE TUNNELING

Figure 5.19: The sample with four inner contacts. The sample was processed by Stephan Lindemann.

Figure 5.20: Schematic drawing of the edge channels.

5.4.3 Finding the Working Point

In order to adjust the magnetic field and the gate voltage to have integer filling factors in both the gated and the ungated region, a magneto-transport measurement on the outer

Figure 5.21: Current from contact A to contact B as a function of the gate voltage and the magnetic field. The bias voltage was 10 μV. The Shubnikov-de Haas oscillations of the ungated region are superimposed on the Landau-fan, originating from the gated region.
contacts (P1-P6) was intended. Unfortunately not enough contacts worked for this kind of measurement and the working point had to be found by a conductance measurement from contact A to contact B. In figure 5.21, the conductance measured at 1.9K is shown as a function of the magnetic field and the gate voltage. Most pronounced is the Landau-fan originating from the gated region. It arises from Shubnikov-de Haas (SdH) oscillations along the B-axes, which show a decreasing frequency for decreasing density. At a gate voltage of about \(-80\) mV, the electron gas under the gate gets depleted. Superimposed on the Landau-fan from the gated region, the SdH oscillation of the ungated region can be found, which are independent of the gate voltage and much weaker. To define a working point, a point where both oscillations show a minimum has to be found. In figure 5.21, the point \(B = 5\) T, \(U_G = +20\) mV was chosen to perform the experiments presented in the next section. A fact to note is that under the gate with \(U_G = 0\) mV the density is much lower than in the ungated region. A positive gate voltage of about \(U_G = 80\) mV is needed for a equal density over the whole sample.

### 5.4.4 Scanning Gate Microscopy of the Inter Edge State Tunneling

The edge of the 2DEG formed by the hole in the center of the structure is investigated with the scanning probe microscope. Figure 5.22a shows the topographical image of the mesa edge of the ungated sector belonging to contact B. Figure 5.22b shows the simultaneously recorded current collected on contact A with a current-to-voltage converter. The contacts C and D where open and the contact B was grounded. A small offset voltage of the current-to-voltage amplifier of the order of 10 \(\mu\)V served as bias-voltage. The PtIr tip was kept on a potential of 0 V. The tunneling current between the edge channels is significantly enhanced when the tip is over the edge. The peak to peak current in the hole image is below 1 pA. Based on topographical imaging it has been assumed, that the tip is blunt with a lateral extent of about 500 nm. The pick up of small grains leads to a temporarily sharp depiction of the topography as in figure 5.22. The electronic picture, however, does not profit from the small grain on the tip and shows a structure along the edge that is about
1 μm in width. Nevertheless filament-like structures can be recognized that presumably originate from a folding of the complicated shape of the tip induced screening charge with the point like scattering centers. The single scattering sites, which are believed to be spaced $0.5 - 2 \mu m$ [124, 33], can not clearly be distinguished in this measurement. In Figure 5.23 a finer scan of the same area is shown and some hot spots are visible and can be attributed to single scattering sites. Further investigations of the dependence on the tip voltage and the magnetic field are needed in order to have conclusive data. However, this experiment is an impressive proof of the concept of the edge channels for the explanation of the quantum Hall effect.

5.4.5 Conclusions and Outlook

With this experiment the successfully setup of a versatile scanning probe microscope for in-situ magneto transport experiments is demonstrated. Sensitive transport measurements can now be complemented by a scanning probe at a temperature of 1.9K. The vacuum beaker allows to maintain stable experimental conditions over several weeks.

With this experiment, the proof of the concept of the inner contacts to separate edge channels is brought. The special sample introduced in section 5.4.2 allows to investigate a closed edge channel around a hole in the 2DEG by four tunneling connection to outer edge channels. The central hole in the structure can be reduced down to about 2 μm in diameter with photo lithographical techniques. For decreasing size of the hole, quantum effects like quantization of the charge (Coulomb blockade), quantization of the flux and quantization of the energy will come into play.

The ability to image and modify locally the tunneling processes inside the edge channels offers a new possibility to investigate the phase coherent transport in edge channels. The new concept of inner contacts together with the scanning probe microscopy opens the door for a variety of experiments with phase coherent edge channels.
List of Abbreviations

<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Full Text</th>
</tr>
</thead>
<tbody>
<tr>
<td>AFM</td>
<td>Atomic Force Microscopy</td>
</tr>
<tr>
<td>STM</td>
<td>Scanning Tunneling Microscopy</td>
</tr>
<tr>
<td>SFM</td>
<td>Scanning Force Microscopy</td>
</tr>
<tr>
<td>SNOM</td>
<td>Scanning Near Field Optical Microscopy</td>
</tr>
<tr>
<td>MFM</td>
<td>Magnetic Force Microscopy</td>
</tr>
<tr>
<td>BEEM</td>
<td>Ballistic Electron Emission Microscopy</td>
</tr>
<tr>
<td>LTSPM</td>
<td>Low Temperature Scanning Probe Microscope</td>
</tr>
<tr>
<td>SPM</td>
<td>Scanning Probe Microscopy</td>
</tr>
<tr>
<td>SXM</td>
<td>see SPM</td>
</tr>
<tr>
<td>UHV</td>
<td>Ultra High Vacuum</td>
</tr>
<tr>
<td>VTI</td>
<td>Variable Temperature Insert</td>
</tr>
<tr>
<td>SQUID</td>
<td>Superconducting Quantum Interference Device</td>
</tr>
<tr>
<td>SET</td>
<td>Single Electron Transistor</td>
</tr>
<tr>
<td>FET</td>
<td>Field Effect Transistor</td>
</tr>
<tr>
<td>LDOS</td>
<td>Local Density of States</td>
</tr>
<tr>
<td>HPOG</td>
<td>Highly Oriented Pyrolytic Graphite</td>
</tr>
<tr>
<td>Ti</td>
<td>Titanium</td>
</tr>
<tr>
<td>Ptlr</td>
<td>Platin Iridium Alloy</td>
</tr>
<tr>
<td>CuBe</td>
<td>Copper Beryllium Alloy</td>
</tr>
<tr>
<td>GaAs</td>
<td>Gallium Arsenide Alloy</td>
</tr>
<tr>
<td>InAs</td>
<td>Indium Arsenide Alloy</td>
</tr>
<tr>
<td>2DEG</td>
<td>Two-Dimensional Electron Gas</td>
</tr>
<tr>
<td>SdH</td>
<td>Shibnikov-de Haas</td>
</tr>
<tr>
<td>DSP</td>
<td>Digital Signal Processing</td>
</tr>
<tr>
<td>OCXO</td>
<td>Oven Controlled Quartz Crystal Oscillator</td>
</tr>
<tr>
<td>VCO</td>
<td>Voltage Controlled Oscillator</td>
</tr>
<tr>
<td>DCO</td>
<td>Digital Controlled Oscillator</td>
</tr>
<tr>
<td>DDS</td>
<td>Direct Data Synthesis</td>
</tr>
<tr>
<td>PID</td>
<td>Proportional Integral Derivative (Controller)</td>
</tr>
<tr>
<td>LP</td>
<td>Low Pass (Filter)</td>
</tr>
<tr>
<td>RMS</td>
<td>Root Mean Square</td>
</tr>
<tr>
<td>SNR</td>
<td>Signal to Noise Ratio</td>
</tr>
<tr>
<td>CPD</td>
<td>Contact Potential Difference</td>
</tr>
<tr>
<td></td>
<td>continued...</td>
</tr>
<tr>
<td>Abbreviation</td>
<td>Full Text</td>
</tr>
<tr>
<td>--------------</td>
<td>----------------------------</td>
</tr>
<tr>
<td>SISO</td>
<td>Single Input Single Output</td>
</tr>
<tr>
<td>MIMO</td>
<td>Multi Input Multi Output</td>
</tr>
<tr>
<td>FFT</td>
<td>Fast Fourier Transform</td>
</tr>
<tr>
<td>ac</td>
<td>Alternating Current</td>
</tr>
<tr>
<td>dc</td>
<td>Direct Current</td>
</tr>
<tr>
<td>NPC</td>
<td>Nuclear Pore Complex</td>
</tr>
<tr>
<td>mRNA</td>
<td>Messenger Ribonucleic Acid</td>
</tr>
<tr>
<td>ATP</td>
<td>Adenosin Triphosphate</td>
</tr>
<tr>
<td>MDa</td>
<td>Mega Dalton</td>
</tr>
</tbody>
</table>
Publications


Bibliography


At this point I would like to thank Prof. Klaus Ensslin for the possibility to realize such a complex project. It was a great pleasure to work in his group. I enjoyed a great freedom and his enthusiasm for mesoscopic physics was a big motivation to tackle the technical problems.

I would also like to express my thanks to Thomas Ihn for the guidance through this thesis. His endless patience and the many lively discussions helped me a lot. I enjoyed his didactic excellent explanations of complicated theories.

Paul Studerus, the electronics engineer was the one who taught me all the electronics matter. He is an expert in low noise measurements and without him the presented setup would not have been possible. He proved a big patience in listening to the problems of a physicist and did not get tired in building (black-) boxes for quickly changing demands. I would like to thank him for the excellent cooperation.

Andreas Herrmann who was the technician introduced me into the metalworking and all the handling of the laboratory equipment. His efficiency of getting the daily work done was a model to me. Later Cecil Barengo took over his job and showed a big enthusiasm for building tricky mechanisms. Both of them I have to thank especially since my work is considerably based on their help.

Furthermore I would like to thank Jean Pierre Stucki for his advises concerning the copper cone sealing and for the soldering and polishing of this part.

It was at the end the machine shop under the guidance of Peter Brühwiler where all the parts for the microscope were made. I would like to thank him for the patience for explaining me how to draw technical drawings and all his crew for the excellent work.

The microscope setup of Hans Hug at the University of Basel was a model for me from the beginning on and I would like to thank him for the possibility to calibrate the tuning fork sensor with his fiber optical interferometer.

Our Secretary, Brigitte Abt, was taking care of all the bills and the administration stuff for which I want to express her my thanks. Furthermore she brought a nice atmosphere into the group and mediated in difficult situations.

Special thanks go to Ryan Held and Silvia Lüscher for providing me with two AFM-patterned quantum wire samples. I would also like to thank Rainer Jäggi and Alfredo Franco for the biological sample and Stefan Lindemann for the processing of the samples for the edge-channel experiment.

Tobias Vancura was responsible for the computer network and the Linux server and he helped me a lot dealing with computer problems. Sharing with him the Laboratory was cheering up and I wish him success for the construction of the \(^9\)He-SPM.

I would also like to thank the other PhD-students Andreas Fuhrer, Volkmar Senz,
ACKNOWLEDGMENTS

August Dorn and the former PhD-students Sebastian Brosig and Gian Salis for the great atmosphere in our group.

Finally, I would like to express my best wishes to my successor Andreas Baumgartner who will now use the microscope to uncover the electronic properties of mesoscopic devices.
Curriculum Vitae

Bürger von Wilderswil (BE), Schweiz

Zivilstand: ledig
Eltern: Diethild und Hanspeter Rychen
Geschwister: Martina und Nora

1978 - 1982 Primarschule Neufeld Thun
1982 - 1986 Sekundarschule Länggasse Thun
1986 - 1990 Realgymnasium Kirchenfeld Bern
1991 - 1996 Studium der Mathematik und Physik an der ETH Zürich
1997 Diplom in Experimentalphysik
1997 - 2001 Doktorarbeit an der ETH Zürich, Institut für Festkörperphysik,
       Gruppe Prof. Klaus Ensslin