Development of radio-frequency scanning tunneling microscope for magnetic point contact measurements

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Development of Radio Frequency Scanning Tunneling Microscope for Magnetic Point Contact Measurements

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Abstract

In this thesis we develop an instrument for studying high frequency transport in magnetic point contacts. A scanning tunneling microscope is designed to perform two assignments: locating nanostructures on a sample by surface scanning as well as making a contact to a specific nanostructure and with its subsequent transport characterization. The instrument thus allows to significantly shorten test times for transport nanodevices by eliminating the need for their full circuit integration.

In conventional scanning tunneling microscopes the long ground path to the sample introduces an inductance which prevents high frequency signals. We address this problem and present a implementation incorporating a local, short-loop ground, designed to allow high frequency measurements.

The main focus of this work was on constructing, evaluating and improving the microscope. Electronic and mechanical components were studied in detail and significant effort was made to develop computer algorithms for approach, feedback loop and scanning. The result is a fully functional STM with the capability of scanning a $1 \times 1 \mu m$ area with a speed up to $500 \mu s$/pixel, successfully distinguishing nano-patterns on the surface. Future advances will come from optimizing the high frequency measurement-part of the instrument using matching resonance-circuits integral to the tip-surface impedance.
Sammanfattning

I den här avhandlingen utvecklar vi ett instrument för studier av högfrekvenstransport i magnetiska punktkontakter. Ett sVeptunnelmikroskop är anpassat för att utföra två uppgifter: Lokalisera nanostrukturer på ett prov genom ytscanning samt skapa kontakt med en specifik nanostruktur och med dess efterföljande transportkarakterisering.

I konventionella sVeptunnelmikroskop medför den långa jordvägen till provet en induktans som förhindrar högfrekvenssignaler. Vi konfronterar detta problem och presenterar en implementering som integrerar en lokal jord med kort slinga, designad för att möjliggöra högfrekvensmätningar.

Tylngdpunkten i detta arbete låg på att bygga, utvärdera och förbättra mikroskopet. Elektroniska och mekaniska komponenter studerades i detalj och betydande ansträngningar gjordes för att utveckla dataalgoritmier för approach, feedback och scanning. Resultatet är ett fullt fungerande STM med möjligheten att scanna en yta på $1 \times 1 \mu m$ med en hastighet upp till 500 $\mu s$/pixel, som framgångsrikt kan särskilja nanostrukturer på ytan. Framtida avancemang kommer att erhållas genom optimering av högfrekvensmätningssdelen av instrumentet med hjälp av resonansmatchningskretsar integrerade med prob-yt-impedansen.
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List of abbreviations

ADC          analog-to-digital converter
AFM          atomic force microscope
AI           analog input
AO           analog output
ASIC         application-specific integrated circuit
BNC          Bayonet Neill-Concelman
CPU          central processing unit
DAC          digital-to-analog converter
DC           direct current
DSP          digital signal processor
FFT          fast Fourier transform
FIFO         first in, first out
FPGA         field-programmable gate array
FXP          fixed-point
HV           high voltage
I/O          input/output
IRQ          interrupt request
MMCX         micro-miniature coaxial
MTJ          magnetic tunnel junction
op-amp       operational amplifier
PCI          peripheral component interconnect
PZT          lead zirconate titanate
RF           radio frequency
RF-STM       radio frequency scanning tunneling microscope
SEM          scanning electron microscope
SMA          subminiature version A
SPM          scanning probe microscope
STM          scanning tunneling microscope
TVS          transient voltage suppressor
VI           virtual instrument
Chapter 1

Introduction

Since the middle of the 20th century, when the transistor was invented, microelectronic devices have been based on a digital logic of electrons. Data is expressed in electronic circuits as binary digits - ones and zeros - represented by existence or absence of electric charge. The semiconductor industry has dominated the microelectronics market for more than 50 years keeping up with the trend of doubling the number of transistors on integrated circuits every 18 months, also known as Moore’s law. Even so, this trend is slowing down as we are reaching the limits of how small transistors can be made before quantum- and thermal effects become a problem. For this reason scientists and engineers have started to investigate another property of the electron, called spin, to carry digital information [1].

In the field of spin transport electronics, also known as "spintronics", the intrinsic spin of the electron and its associated magnetic moment is exploited for data representation. Manipulating spin can potentially be done faster and require less energy than moving around electrons to charge and discharge capacitors. It can also be done on a smaller scale, which is extremely appealing for information processing devices [2].

The potential of this technology has lead to a huge interest in the field of spintronics and a lot of research is currently being done on magnetic nanostructures functioning as storage elements. Of specific interest is their high frequency properties since they determine the write/read speed of the memory cells. Characterizing these properties is an important step when developing such applications and comes with the need of improving instrumentation and measurement techniques.

The standard method for measuring these properties is to fabricate nanopillars, e.g. magnetic tunnel junctions (MTJs) or spin-valves, complete with wires connected to top and bottom contacts. This is a multi-step process requiring lithography, etching and deposition - fabrication processes taking a considerable amount of time and effort. An alternative method which avoids many of these steps is to have a surface probe capable of locating the nanopillar and doing a point contact current transport measurement without the need for a patterned top electrode. This method allows fundamental research on the core of the device without spending valuable time and resources on unavoidable fabrication steps [3] [4].

Scanning tunneling microscopy (STM) is an indispensable tool in nano-technology for studying the surface topography of a sample and very high resolutions can be obtained. STMs can also be used for extremely localized transport measurements by first locating the nanostructure of interest and then go into contact with the tip. However, high
frequency measurements requires a short ground path which conventional STMs do not have and this calls for a new grounding method.

In this thesis we develop a radio frequency scanning tunneling microscope (RF-STM) for locating nanostructures on a sample, going into electrical contact and doing radio frequency transport measurements. A similar type of microscope has been proposed in the work done by U. Kemiktarak but where he fails to show a proper method for grounding [5]. We present a novel, localized grounding scheme with a small ground path inductance allowing characterization of magnetic nanostructures by high frequency magneto-transport measurements.
Chapter 2

Methods

2.1 The Scanning Tunneling Microscope

2.1.1 Overview

The STM is an instrument used for imaging surfaces at an atomic level. It was developed in 1981 by Gerd Binning and Heinrich Rohrer at IBM. Their invention earned them the Nobel prize in physics 1986 [6] and opened the door to understanding phenomena on a nano-scale. The STM has since then been an indispensable tool in nanotechnology and has lead to the development of many other types of scanning probe microscopes (SPMs), like the Atomic force microscope (AFM).

2.1.2 Electron tunneling

The STM relies on a purely quantum mechanical phenomenon called tunneling. This phenomenon has no classical explanation and can only be explained by the wave-like behaviour of particles. Conceptually, tunneling is when a particle travels through a potential barrier even if its energy is too small to overcome the barrier. One could make a more tangible analogy of a ball thrown against a wall and actually passing through it instead of bouncing back.

The wave function of a particle contains all measurable information that can be known about a physical system - properties like position, momentum, energy, etc. This is why quantum mechanical problems center around analysis of the wave function. By using mathematical formulations of quantum mechanics, e.g. solving the Schrödinger equation, the wave function for a system can be determined. One problem which has been studied thoroughly is the one-dimensional rectangular potential barrier problem. By solving the Schrödinger equation for a particle encountering a rectangular potential energy barrier it is shown that there is a non-zero probability of the particle penetrating the barrier and continue travelling on the other side. This probability is however exponentially decreasing and drops to zero fast.
2.1.3 Working principles of STM

As already mentioned the STM is based on quantum tunneling. When a conducting tip is brought very close to a surface electrons will start to tunnel through the gap between them, see figure 2.1. This is the same principle as in the rectangular potential barrier problem, but here the gap is the actual barrier. If a bias voltage is also applied across the tip and sample there will be more electrons tunneling in one direction than the other, giving rise to a non-zero net current. The probability of an electron tunneling through the gap is exponentially dependent on the barrier width, i.e. the distance between tip and sample. This exponential dependence is the key to why an STM is so sensitive and can image single atoms. The magnitude of the resulting current also depends on the material of tip and surface, tip radius and bias voltage and can be approximated as

\[ I \propto V_{\text{bias}} e^{-A z \sqrt{\phi}} \]  

(2.1)

where \( V_{\text{bias}} \) is the applied bias voltage, \( A \) is a constant equal to 1.025 Å\(^{-1}\)eV\(^{-\frac{1}{2}}\) , \( \phi \) is the barrier height, or more exact; the average work function of the two metals, and \( z \) is the gap spacing [6]. For a typical work function of 4 eV an increase in \( z \) of 0.1 nm would lead to a decrease of the current by a factor 10. A typical atomic diameter of 0.3 nm would consequently lead to a change in tunneling current by a factor 1000!
To image a surface the tip is brought into tunneling distance and then raster-scanned over a small area of the surface. The tip needs to be moved with very high precision and resolution in order to resolve small features in the sample. This is usually done by putting the tip on a piezoelectric actuator which changes shape when a voltage is applied. With this kind of actuator the tip can be moved with sub-Ångström resolution across the sample. As the scan is performed a computer collects the data and translates it into an image.

2.1.4 Modes of operation

There are two modes of operation when it comes to STM; constant current mode and constant height mode, see figure 2.2. In constant current mode a feedback loop keeps the current constant by adjusting the tip height continuously during the scan. The topographic image of the surface is generated by keeping track of the tip’s vertical position at all points in the scan lattice. This mode has the advantage of the tip "following" the surface profile making it possible to scan rougher samples without crashing the tip. The downside is the increase of scan time due to the feedback.

In constant height mode the tip’s vertical position is kept constant during scan and no feedback is used. As a consequence the tunneling current changes depending on the surface structure and is used for creating the image. This mode is only appropriate for very flat surfaces and would otherwise lead to crashing of the tip or getting out of tunneling range. The advantage with constant height mode is the high scan speed which can be obtained because there is no need for feedback.

Figure 2.2: STM modes of operation. Image from Attocube systems AG [7]
2.2 The Radio-Frequency STM

So what is the difference between an STM and an RF-STM? Conventional STMs have a limited bandwidth when it comes to carrying and detecting current. This makes it impossible to use the STM for measurements involving RF signals. There are essentially two reasons for this limited bandwidth - first we have the impedance mismatch between tunnel junction and current detector. This impedance mismatch is due to the high impedance of the STM tunnel junction, typically between 1 MΩ and 1 GΩ, and the characteristic impedance of 50 Ω in the electronic devices used for detecting current. The mismatch leads to a loss in the power, caused by reflection, delivered from source to load [8]. This problem can be solved, within a certain bandwidth, by using an impedance matching circuit to transform the tunnel impedance to 50 Ω [5]. With this said we will not focus more on impedance matching in the scope of this thesis.

The other reason for limited bandwidth is the high ground path inductance in the STM at higher frequencies. This introduces an attenuation of the propagating signal, reducing the bandwidth. As conventional STMs do not require high frequency compatibility (bias is DC voltage for example) there is really no need for a short ground path. This problem can be solved by implementing a shorter ground path. In chapter 3 we present a design for a localized ground with the potential of increasing the total bandwidth of the system.
Chapter 3

Instrumentation

This chapter describes how the STM system is built and the details about each individual component.

3.1 Overview

![Diagram of STM setup]

**Computer:** User interface to control the STM. Calculations and data analysis.

**FPGA:** Real time controller for STM. Digital to Analog Converters (DACs) as voltage sources for the piezo tube. Analog to Digital Converter (ADC) to measure tunnel current.
**HV amplifier:** Amplifies the output voltage from the FPGA to the piezo tube.

**Piezo:** Moves the tip in xyz with a sub-nanometer resolution.

**Tip holder:** A small metal tube where the tip is inserted.

**Current amplifier:** Amplifies the tunneling current to a measurable quantity for the FPGA.

**Coarse approach controller:** Controls the piezo motor used for coarse approach.

**Coarse translation xyz stage:** Provides coarse adjustment of the sample position. Manual for lateral motion and motorized for z motion.

**Sample holder:** Where the sample is placed. A small spring keeps the sample stable.

### 3.2 Piezo Tube

The piezo tube plays a central role in the microscope. It works as an actuator and makes it possible to move the tip across the sample with high resolution and hence perform a scan. The tube does not only move in the lateral direction but also in the axial direction. This is crucial for the height control of the tip during scan and also for the initial approach to the surface. Having the possibility to control 3D motion with just one component is convenient and saves space in the setup.

#### 3.2.1 Properties

A piezoceramic material, such as lead zirconium titanate (PZT), changes shape when an electrical field is applied. This is usually done by applying a voltage across electrodes attached to the material. Depending on how the electrodes are positioned different parts of the material will be subject to the emerged electric field, making it possible to elongate the piezoceramic in the desired direction. As high electric fields applied corresponds to tiny changes in shape, a high resolution can be obtained, sub-nanometer, which is essential for atomic imaging.

For the STM in this experiment we use a piezo tube with quartered electrodes on the outside, as can be seen in Fig. 3.2, and one large cylindrical electrode on the inside serving as ground plane.

The four separate electrodes on the outside enable control of motion in ±x and ±y but can also be used for axial displacement. By applying the same voltage on all four quadrants the tube will be displaced in z direction. In practise it is difficult to apply exactly the same voltage on all four electrodes since they are connected to separate amplifiers which are not identical to each other. This introduces errors both in lateral and axial movement of the tube which is not desirable. However, there is another way to control the z displacement more accurately. The inside of the tube, serving as ground for the outer electrodes, can also be used for the axial motion. Applying a voltage on the inner electrode is effectively the same as applying a voltage on all outer electrodes but with opposite sign. This way the axial motion can be controlled without having to use the outer electrodes, allowing separate control of deflection and displacement of the tube. When using this method it is important to keep track of the total voltage across
Figure 3.2: Electrode configuration on piezo tube.

The amount of displacement, for a given voltage, depends linearly on the dimensions and material of the tube and is according to theory

\[
\Delta z = d_{31} \frac{L}{h} V, \tag{3.1}
\]

where \(d_{31}\) is the piezoelectric coefficient \([\text{Å}/\text{V}]\), \(V\) - applied voltage, \(L\) - tube length and \(h\) - the wall thickness \([9]\). It is convenient to define a piezo constant \(K_z\) as

\[
K_z = d_{31} \frac{L}{h}, \tag{3.2}
\]

which lets us write Eq.(3.1) as

\[
\Delta z = K_z V. \tag{3.3}
\]

The lateral deflection of the tube is

\[
\Delta x = \frac{\sqrt{2}d_{31}L^2}{\pi Dh} V,
\]

where \(D\) is the mean diameter, \(\text{Outer diameter} + \text{Inner diameter} / 2\). This deflection is increased by a factor two if the opposite voltage is applied to the opposite quadrant, resulting in a lateral deflection of

\[
\Delta x = \frac{2\sqrt{2}d_{31}L^2}{\pi Dh} V = K_x V, \tag{3.4}
\]

where

\[
K_x = \frac{2\sqrt{2}d_{31}L^2}{\pi Dh}. \tag{3.5}
\]

The same formula holds for the \(y\)-deflection as well.
The tube used in this experiment is 1 inch long (2.54 cm), 0.5 inch outer diameter (1.27 cm), wall thickness of \( \frac{1}{16} \) inch (0.1588 cm), \( D = 0.4375 \) inch (1.1112 cm) and has a piezoelectric coefficient \( d_{31} \) of -2.15 Å/V. Plugging these numbers into equations (3.2) and (3.5) yields

\[
K_z = -34.4 \text{ Å/V} \tag{3.6}
\]

and

\[
K_{xy} = -70.79 \text{ Å/V} \tag{3.7}
\]

which are the theoretical piezo constants for our piezo tube.

### 3.3 Sample stage

The sample stage used in the experiment is a MicroBlock 3-Axis Flexure Stage from Thorlabs. It has manually adjustable x and y axes while the z axis is driven by a Squiggle piezoelectric step motor which we will discuss in more detail in the next section.

The stage has been slightly modified in the earlier work by Sergiy Cherepov who used it for CIPT measurements [4]. With some additional parts and adjustments the stage is adapted to support our STM.

The whole set-up is placed on a TS-150 stable table from Table Stable Ltd which actively reduces low frequency vibrations which might affect the performance of the STM and also the quality of the results. The stable table stands on an optical table which reduces vibrations from the building as a first safety measure.

#### 3.3.1 Squiggle piezo motor

The piezo tube only has an axial range of approximately 0.5 μm. Therefore, a coarse approach needs to be done to get the piezo tube within range from the sample surface. For this kind of approach a longer range is more important than high resolution.

What we use in the set-up for coarse approach is a piezoelectric motor called "SQUIGGLE" from New Scale Technologies’ SQ-100 Series [10]. It is designed for nanopositioning applications. The SQUIGGLE motor is non-magnetic and has a range of 50 mm and a resolution up to 20 nm, which meets our requirements. It comes with a usb motor controller which can be controlled with the supplied software or with ActiveX commands and is therefore easily incorporated in a LabVIEW program.
3.4 Scanner head

When building the scanner head of the STM the tip holder needs to be attached to the piezo tube which itself needs to be secured somewhere. Piezo electrodes must be wired to the HV amplifier and the tip requires a proper electrical contact to the pre-amp.

![Figure 3.3: 3D-view of complete probe mount. Purple is piezo tube.](image)

3.4.1 Tip holder

When designing the tip holder we have a few requirements: Ability to easily change the tip, has to fit on the piezo tube, needs to be non-conducting and to support our grounding method. We decide to make a plug-resembling piece which goes onto the bottom of the piezo tube, see Fig. 3.4. The piece is made out of Macor, a glass-ceramic material which can easily be machined into any shape [11]. It is electrically non-conducting and also a good thermal insulator with very little expansion due to temperature changes. The center hole is big enough to fit an MMCX female receptacle which will serve as the actual tip holder. The receptacle is a small metal tube with an inner wall in the middle, dividing it into two parts. This makes it easy to solder a cable in one end leaving the other for inserting a tip. The four other holes on the piece make our grounding method possible.

The Macor piece is glued to the piezo tube with epoxy, a two-component adhesive which forms a strong polymer with high temperature resistance.
3.4.2 Piezo tube holder

To fix the piezo rigidly we design a plate, also in Macor, with a hole in the center having the same diameter as the tube. In this hole the tube is glued with epoxy such that its top edge is in height with the plate surface. Around the center hole four smaller holes are made, large enough to pull through wires to the outer electrodes on the piezo tube. In the corners of the plate four screw holes are made so that the plate can be attached to the sample stage.

Figure 3.5: 3D-, side- and top view of piezo tube holder.
3.4.3 Cabling

**RF compatibility**

Since the aim with this microscope is to perform RF measurements it is important to use proper cables and connections which can support high frequency signals. The MMCX receptacle, functioning as the tip holder, is soldered to the inner core of a SUHNER Switzerland RG 178 B/U 50 Ohm coaxial cable. This cable can carry GHz signals with sufficiently low attenuation, yet being thin and flexible enough to not hinder the movement of the piezo tube. To implement the local ground method the shield of the coax is exposed and thin wires are soldered onto it. These wires are lead through the holes of the tip holder piece, as shown in Fig. 3.6, and will close the electrical circuit by contacting the surface of the sample during measurements. This way a very short ground path is achieved which should increase the bandwidth significantly compared to conventional STM grounding methods.

All solder joints are covered with heat shrink tube to insulate from surrounding conductors and fields. This is very important since the cable goes through the piezo tube and is very close to its inner electrode on which there will be high voltage. On the other end of the coaxial cable an SMA connector is soldered so that the current pre-amp can be connected. SMA or Sub-miniature, type A is a coaxial RF connector which is much smaller than BNC and works up to approximately 18 GHz [12].

![Figure 3.6: Photograph of the tip holder piece. A coaxial cable is soldered to the tip holder and ground wires are drawn out through the side holes. One of the holes has an MMCX receptacle for availability to change ground wire.](image)

**Connecting the piezo electrodes**

A multi-wired cable is used to make the connection between high voltage amplifier and piezo tube electrodes. Four wires, one for each outer electrode, are lead through the designated holes in the Macor plate and stripped at the end. The stripped part is flattened out so that the strands form the shape of a fan and is then attached to respective electrode
with silver paint. The silver paint forms a sturdy junction when it hardens and provides a good electrical connection between wire and electrode. It is not as robust as a solder joint but soldering on a piezoelectric material always comes with the risk of depolarizing it if the temperature exceeds the Curie temperature [13].

The inner electrode is connected in the same way using a fifth wire from the multi-wired cable. The cable is fixed to the Macor plate with a clamp such that the electrode wires are secured without tension. At the other end a D15 connector is soldered with the pins matching the corresponding outputs on the HV amplifier.

### 3.5 STM circuitry

#### 3.5.1 Current pre-amplifier

The tunneling current typically ranges from 10 pA to 10 nA and to be able to measure this current it needs to be amplified. A transimpedance amplifier circuit is a good choice for this task because it converts an input current to a voltage which can easily be measured by the controller device.

**Circuit theory**

The transimpedance amplifier circuit is an operational amplifier (op-amp) with negative feedback, as can be seen in Fig. 3.7(a). In an ideal op-amp no current goes through it because the resistance between the inputs is infinite. In reality this resistance is not infinite but still very high. The resistor $R$ in our circuit is a 100 MΩ resistor, which is small compared to the internal resistance of the op-amp, and hence all the current will go through it. As the current goes through the resistor a voltage drop will occur. What the op-amp does is adding a voltage to the input source to compensate for the drop over the resistor and therefore keeping the differential input voltage at zero. The closed loop gain is easily determined as $V_{OUT} = -I_{IN}R$. What we also need to consider in this circuit is

![Transimpedance Amplifier Circuit](image)

(a) Basic transimpedance amplifier circuit.  
(b) Transimpedance amplifier circuit with tunnel junction as current source. The voltage applied on the positive input will yield a bias voltage over the tunnel junction.

Figure 3.7: Schematic over transimpedance amplifier circuit.

the bias voltage which is going to be applied over the tunnel junction. By inserting a
voltage source, \( V_B \), at the positive input of the op-amp, instead of ground, the potential is raised (See Fig. 3.7(b)). Since the amplifier keeps the differential input voltage at zero the potential will also be raised at the negative input, which leads to a bias voltage over the tunnel junction. By introducing this voltage source we also affect the output of the amplifier. The voltage going out will have an offset equal to the bias voltage

\[
V_{OUT} = -I_{IN}R + V_B. \tag{3.8}
\]

**Building the amplifier**

In our circuit we use an LF411 operational amplifier from National Semiconductor Corporation. It has a fast response time, less than 1 \( \mu s \) for a 10 V output voltage swing [14]. The typical swing when scanning is much lower than this value and what actually limits the measuring speed in the STM is not the op-amp but the FPGA which has sampling period of 5 \( \mu s \).

The op-amp requires a DC supply voltage and the performance of the amplifier depends on how stable this supply voltage is. An ideal voltage source would lead to an output only governed by the input. By using batteries as power supply, two 9 volt batteries in this case, the voltage will remain very stable and therefore increase the quality of the amplifier output. To prevent the batteries from running out when the amplifier is not being used a switch is installed which can open and close the connection to the op-amp socket. The batteries are also grounded to the box and the last component installed is the 100 M\( \Omega \) feedback resistor.

### 3.5.2 High voltage amplification

As the piezo tube requires high voltages in order to achieve a large displacement or deflection, the output from the FPGA card needs to be amplified. The \( \pm 10 \) V analog output would only correspond to a lateral deflection, or scan range, of \( \pm 70.79 \) nm, which is not sufficient for our purposes.

In this project we use an amplifier made in earlier work by Sergiy Cherepov [4]. It has the capacity of amplifying five separate channels, which is what we need for the piezo tube electrodes. The amplifier input connector is a male D25 and the output is a female D15.

The amplification on the five separate channels is determined by applying different voltages to the amplifier and reading out the output voltage with a multimeter. The way we applied the voltages to the amplifier was by writing a small LabVIEW program which output a voltage corresponding to a certain bit-value. This way we obtain the output voltages from the amplifier as functions of bit-values in the FPGA, which in the end is necessary to know to control the piezo tube. By plotting the data we retrieved (see Fig.3.8) we could clearly see a linear region in every channel as expected. The nonlinear parts in the beginning and end of every curve is where the saturation limit of the amplifier is reached and will not be used during operation. A linear fit of the data from each channel gives the conversion coefficients between amplifier output and FPGA output, which are necessary for being able to apply the desired voltage to the piezo tube. Results are summarized in table 3.1.
Figure 3.8: FPGA output voltage dependence of the amplifier output.

<table>
<thead>
<tr>
<th>Channel</th>
<th>Conversion [V/bit]</th>
<th>Offset [V]</th>
</tr>
</thead>
<tbody>
<tr>
<td>AO0</td>
<td>-0.0092</td>
<td>-0.2743</td>
</tr>
<tr>
<td>AO1</td>
<td>-0.0046</td>
<td>-0.3793</td>
</tr>
<tr>
<td>AO2</td>
<td>-0.0046</td>
<td>-0.1331</td>
</tr>
<tr>
<td>AO3</td>
<td>-0.0046</td>
<td>-2.6368</td>
</tr>
<tr>
<td>AO4</td>
<td>-0.0046</td>
<td>-0.3863</td>
</tr>
</tbody>
</table>

Table 3.1: Coefficients for converting FPGA analog output to high voltage amplifier output.

3.5.3 Voltage spike protection

When using high voltages there is always the risk of damaging sensitive equipment which uses low voltages, like logic devices. The FPGA-card is directly connected to the high voltage amplifier and is therefore vulnerable to voltage spikes caused by malfunction. This potential problem is easily prevented by putting Transient-Voltage-Suppression diodes (TVS diodes) in the circuit [15].

A TVS diode is a clamping device that shunts the excess current when the voltage exceeds a certain level. Below this level the diode is in principle invisible and will not affect the circuit. By putting a diode in parallel with signal and ground, see Fig. 3.9, any voltage spike will result in a current going to ground, clamping the voltage at a safe level.

There are a few definitions (also shown in Fig. 3.10) used when specifying the characteristics of a TVS diode:

- **Stand-off voltage** ($V_{RM}$): The voltage below which no significant current is shunted.

- **Breakdown voltage** ($V_{BR}$): The voltage when some specified and significant
current is shunted.

- **Clamping voltage** ($V_{CL}$): The voltage at which the diode will conduct its fully rated current.

These definitions are important to know in order to choose the correct diode for your circuit. In our case we wanted the FPGA to unhindered be able to output its full range, i.e. voltages between +10 V and -10 V. Therefore the stand-off voltage should be 10 V minimum, otherwise the diode would reduce the output range of the FPGA. Voltages outside this range are essentially undesired and might harm the electronics. The FPGA card has a built-in protection system but it only works up to 42 V and a voltage spike from the high voltage amplifier could in principle be much higher. This suggests a maximal clamping voltage of 42 V. We chose a diode from MULTICOMP called P6KE12CA with $V_{RM} = 10.2 \text{ V}$ and $V_{CL} = 16.7 \text{ V}$ which suits our needs.

![Figure 3.9: How the TVS diode is put in the circuit to protect from voltage spikes.](image)

![Figure 3.10: Electrical characteristics of a bidirectional TVS diode. Image from STMicroelectronics [16].](image)
3.6 FPGA

A Field-Programmable Gate Array (FPGA) is a chip which can be programmed to desired application, e.g. Digital Signal Processing (DSP). Unlike "Application Specific Integrated Circuits" (ASICs) an FPGA can easily be reprogrammed after manufacturing and is not designed for one specific task [17] [18]. This flexibility is very useful for a developer who might need to change the functionality often.

FPGAs have several advantages compared to conventional CPU-based systems. An FPGA chip is only processing logic and do not have an operating system which can introduce delays and interruptions. This is crucial when it comes to real-time control of a sensitive system, which the STM is. Furthermore, the speed of an FPGA is not limited by number of processor cores like a CPU. Each independent processing task in the FPGA has its own dedicated section on the chip. Depending on the memory size of the FPGA many processes can be done in parallel [19]. The chip also contains I/O blocks which allows the circuit to interact with the outside world through DACs and ADCs.

The FPGA we use is a PCI-7833R card from National Instruments. It has: 8 analog inputs with independent sampling rates up to 200 kHz (5 µs sampling period), 16-bit resolution and ±10 V range. 8 analog outputs with independent update rates up to 1 MHz, 16-bit resolution, ±10 V range. 96 digital lines for inputs or outputs at rates up to 40 MHz. The FPGA is programmable with the LabVIEW FPGA module which we will discuss more in the next section.

3.7 Software

3.7.1 LabVIEW basics

As mentioned earlier the FPGA is programmed and compiled in LabVIEW which is a development platform with the purpose of programming and automating measuring instruments in a lab environment. LabVIEW is based on a graphical programming language which lets the user create programs without any knowledge about conventional code languages. A LabVIEW program, called virtual instrument (VI), consists of two components: a front panel and a block diagram. The front panel is the user interface where controls and indicators are placed for data input and output respectively. They will appear as terminals in the underlying block diagram which contains the graphical source code. The control terminals are wired to function-nodes in order to perform operations on the input data and then wired to indicator terminals to display the result.

3.7.2 LabVIEW FPGA module

Programming an FPGA is different from other LabVIEW applications in many aspects. In general you have one main VI which is in charge of linking all smaller building blocks together to form a program. Programming an FPGA requires two VIs: A target VI, which contains the code which is going to be executed on the FPGA, and a host VI which is the program on the computer that communicates with the FPGA. These, somewhat special, conditions require careful planning of what to put in which VI. Some tasks are more desirable, or even necessary, to have in one of the two. All critical processes which need real-time control without interruptions should be placed in target. In our case
this applies to approach, scan and feedback. Even so, it is important to not put any avoidable calculations or data storage on the FPGA since it has a limited memory size which is easily exceeded. The host takes care of everything that is not so critical, such as converting user input to complete instructions for the FPGA, data collecting and processing, presentation of results.

**Host VI**

The host VI is the interface to the user. It lets the user set parameters like piezo properties, measurements rates, step sizes, current setpoint, scan size etc. It also contains controls for running the different parts of the program, such as approach, scan and manual squiggle control. For example, pushing a button triggers an event structure in the block diagram which holds the code responsible for that task.

Within the host VI a reference to the target VI is made. This reference is used throughout to program to communicate with the FPGA. Connecting a Read/Write node to the reference gives access to the controls on the target’s front panel and is typically used when timing is not important, e.g. setting scan parameters.

**Target VI**

In this subsection we will highlight what is different when programming the FPGA. The target VI is essentially invisible to the user, it is the "machine room" of the program where user commands are translated to real action. Programming the target is different from programming a normal VI. This is because the code is executed on the FPGA which has more restrictions and fewer compatible functions to use, mostly due to the limited memory size.

FPGAs use binary coding and with that the 16-bit output/input voltages are converted from/to binary values. The 16-bit analog output/input range is [-10, 10] V and hence, the voltage bit-conversion is given by

\[
\text{Voltage} = \frac{\text{Output Code}}{32768} \times 10.0 \text{V}. \tag{3.9}
\]

FPGAs use integer math for calculations and cannot process floating-point numbers efficiently. To get around this problem fixed-point numbers (FXPs) and FXP functions can be used. FXP numbers are essentially represented by a number of bits which is very convenient for the FPGA logic to handle. They have a user-defined range and precision which allows less memory to be occupied on the FPGA than with floats.

A process, such as scanning, can generate a vast amount of data, in a short period of time, which might be too much for the FPGA to store. This problem is solved by transferring the data from the FPGA to the host, using a "First In, First Out" (FIFO) buffer memory. The FIFO memory gives the host time to fetch data without stalling the data storage process in the target, as long as the buffer does not get full.

Arrays should in general be avoided in the target since they require a lot of space. Also, they do not have the same flexibility as in normal computer VIs where arrays can be resized, appended etc. Target arrays must have predetermined lengths at the time of compilation and is therefore not very useful in dynamic processes.

Since the target has to be compiled after each change it is also advantageous to use variables instead of constants in the FPGA because they can be changed during execution.
3.7.3 Approach algorithm

The approach mechanism is a combination of coarse approach with the SQUIGGLE and fine approach with the piezo tube. First the piezo tube is stepwise swept from fully contracted to fully expanded. In between each step the tunneling current is measured and averaged, 10 samples with a sampling period of 5 \(\mu s\). If the mean value is above or equal to the current setpoint the approach is completed or stopped. If the piezo tube has expanded fully without getting into tunneling distance the SQUIGGLE motor needs to be stepped. However, the SQUIGGLE is controlled with an ActiveX component which can not be called from the target. A signal needs to be sent to the host telling it to step the SQUIGGLE. Before doing this the piezo is reset to a fully contracted state, prepared for a new "piezo sweep". The target VI sends an interrupt request (IRQ) to the host. This IRQ stops the execution in the target until the host has responded on this request. Meanwhile the host listens for this IRQ and is programmed to step the SQUIGGLE when it obtains the signal. After the SQUIGGLE is stepped a response is sent back to the target, telling it to continue approach with the piezo again. This closes the loop which is iterated until approach is complete.

To summarize the approach algorithm:

1. Step piezo
2. Measure current
3. Iterate until setpoint is reached or piezo is fully expanded
4. If fully expanded, reset piezo and take a coarse step with SQUIGGLE
5. Repeat these steps until current setpoint is reached

The approach also serves another important purpose - determining the exact exponential relation between distance and tunneling current. By recording data from the approach it is possible to see exactly how the current behaves as a function of z piezo voltage. Fitting of the model to this approach data yields the parameters and a mathematical expression for the current as a function of output voltage [bits], which we want to use in the feedback algorithm.

3.7.4 Feedback algorithm

As we discussed earlier it is desirable to scan in constant current mode when having a rough sample. To keep the current constant it is necessary to have a feedback loop that adjusts the height based on the error signal, i.e the deviation between measured current and setpoint. Since we already know from the approach how the current behaves as a function of distance (corresponding to piezo voltage)

\[
I = ae^{-bz}
\]

(3.10)

where \(a\) and \(b\) are unknown parameters. We can use this relation to make a very precise, model-based, controller. However, it is important to notice that the approach curve may not be static since the tip-sample separation depends on more things than piezo voltage only. Drifts and noise will alter the tunneling current even though the piezo voltage is
kept constant. These drifts will manifest themselves as a time-dependent shift of the approach curve. This is graphically depicted in figure 3.11 where the original approach curve (blue) has been shifted by a constant, \( c \), to the position of the green curve.

\[
I = ae^{-bz} \rightarrow I' = ae^{-b(z+c)}, \quad c \in \mathbb{R}.
\]

If a current setpoint is defined as \( I_0 \) we have \( I_0 = I(z_0) \). A drift would mean that the current at the same piezo voltage changes to \( I'(z_0) = I_1 \). To account for this drift the piezo voltage needs to be adjusted back to \( I_0 \) which is now located at \( z_1 \). The adjustment \( \Delta z \) can be expressed in known terms

\[
\Delta z = z_1 - z_0 \\
= -\frac{1}{b} \ln \left( \frac{I_0}{a} \right) - c - z_0 \quad (3.11)
\]

The constant \( c \) is solved for using the second current measurement

\[
I_1 = ae^{-b(z_0+c)} \Rightarrow c = -\frac{1}{b} \ln \frac{I_1}{a} - z_0 \quad (3.12)
\]

which then can be substituted into Eq. (3.11)

\[
\Delta z = z_1 - z_0 \\
= -\frac{1}{b} \ln \left( \frac{I_0}{a} \right) - c - z_0 \\
= -\frac{1}{b} \ln \left( \frac{I_0}{a} \right) - \left( -\frac{1}{b} \ln \frac{I_1}{a} - z_0 \right) - z_0 \\
= \frac{1}{b} \ln \left( \frac{a}{I_0} \right) + \frac{1}{b} \ln \frac{I_1}{a} \\
= \frac{1}{b} \ln \left( \frac{I_1}{I_0} \right).
\]

And using the relation between distance and piezo voltage (Eq. (3.3)) we can write this

\[
\Delta z = K_z \Delta V = \frac{1}{b} \ln \left( \frac{I_1}{I_0} \right).
\]

It turns out that the only parameter necessary for the feedback to work is \( b \).

In principle this feedback method should be very robust. However, there are limitations in the hardware which introduce sources of error. First of all there is an upper limit in the tunneling current that can be measured. The current pre-amplifier will not be able to output a voltage higher than its internal power supply, which in this case is 9 V and corresponds to 90 nA tunneling current. If this limit is reached the actual current can not be determined and the feedback might not be able to adjust the piezo sufficiently in one step. However, it will make its maximum adjustment and decrease the current towards the setpoint.

If the maximum output of the pre-amp is exceeded there is a chance that the tip will crash. To reduce the risk of this happening we used a fairly high bias voltage, 500 mV, which means that a tunneling current will appear further away from the surface than with a lower bias voltage (see Eq. (2.1)).
A similar problem arises if the current gets too low. Since the current is exponentially decreasing with distance it goes to zero rapidly. The FPGA has a finite resolution and the smallest voltage it can read out corresponds to 0.3 mV. If the current goes below this value the tip is not within tunneling distance any more but there is no way to know how far away it is. The largest step down might not be enough to get into tunneling again.

![Graphical description of how drift affects the tunneling current.](image)

**Figure 3.11:** Graphical description of how drift affects the tunneling current.

### 3.7.5 Scan algorithm

There are essentially three parameters for the user to set before making a scan: Scan size, step size (resolution) and measuring time per pixel. The host converts the size parameters to bit values and sends them to the target.

The scan pattern used is a raster pattern - a series of rows covering a rectangular area as can be seen in Fig. 3.12. Each row the probe is moved from left to right (trace) and then back (retrace), ending up where the row started. Since the feedback introduces different artefacts when the tip goes up or down a steep edge, more information is obtained when scanning in both directions. It also makes it possible to measure how much drift there is because the same pixel is measured twice with some time in between. The scan algorithm consists of two loops - one inner loop for the fast scan direction and one outer for the slow. It is programmed such that scanning a row is an independent function which is called several times to build up the whole scan. At each pixel the tunneling current is measured after which the feedback adjusts the tip-height accordingly, before the tip is moved to the next pixel. The height-data for each pixel is sent to the host through the FIFO buffer.

Now the fast scan loop is closed and is iterated as many times as there are pixels in a row. At this point the outer loop has performed one whole iteration and the process
is repeated as many times as there are rows. When scan is finished the target sends a signal to the host to notify the completion.

3.8 Tip preparation

In order to have a functioning STM a sharp, conducting tip is required. STM tips are usually made from tungsten or Pt/Ir alloy which are stiff, chemically stable and do not oxidize in air. Oxidization of the tip might lead to a deterioration in the tunneling capability of the microscope. However, at this stage of the project we stuck to simple, easy to make, copper wire tips which is enough for our purposes.

There are essentially two methods when it comes to preparing STM tips; Electrochemical etching and cutting [20]. The etching method produces sharper tips with higher aspect ratio but requires more work. Such tips might be necessary when imaging highly profiled surfaces. If there is no need for high aspect ratio the cutting method is easier to do and still yields atomically sharp probes. The cutting method is really simple and only requires a pair of wire-cutters. The wire is cut at a 45 degree angle while simultaneously pulling it. The stretching force will cause a plastic deformation of the wire and will produce a small extended apex at the tip, as can be seen in figure 3.13. This apex has a radius in the order of 100 nm, which is very good for such a primitive preparation method.
(a) Plastic deformation of the tip can be seen where the cut was made.

(b) Closer view of the tip.

(c) View of the outermost part of the tip.

(d) Tip apex.

Figure 3.13: SEM-images of cut tip.
Chapter 4

Experimental results and discussion

4.1 Actual Piezo Displacement

The piezo constants obtained earlier from equations (3.2) and (3.5) are only theoretical and based on an ideal model. To see how well theory and practice matches we measured the displacement of our tube as a function of an applied voltage using an atomic force microscope (AFM). We performed an AFM scan on the top of the piezo tube while applying a continuous 10 Hz sinusoidal voltage to all quadrants on the piezo tube, making it expand and contract in the axial direction. The motion of the tube was caught by the AFM and depicted as a height difference in the sample as can be seen in figure 4.1.

![AFM scan of piezo tube with sinusoidal voltage applied.](image)

Normally an AFM-scan gives the height of the sample as a function of position. In this case the height will also depend on time since we are oscillating it. The beauty is that we can separate the time-dependent behaviour from the position-dependent by doing a lock-in at the frequency of the applied voltage. This isolates the periodic motion of the piezo from any surface features that might be present on the tube and makes it very easy to obtain the amplitude of the piezo response.
Determining the piezo motion amplitude as a function of the voltage amplitude is done in a few steps: First the height profile of each line of the scan image is obtained. The periodic behaviour is clearly visible which can be seen in Fig. 4.2(a). When having the height data for a line a fast Fourier transform (FFT) is performed, yielding a spectrum similar to the one in Fig. 4.2(b). The peak is located at 10 Hz as expected since the drive frequency is 10 Hz and since there is a linear response. The height of the peak corresponds to the amplitude of the oscillation, which is the value of interest for us.

Doing an FFT on all lines of the scan, typically 256, and taking an average of all the collected amplitudes gives an estimation of the displacement as a function of voltage. We did this procedure with different voltage amplitudes to see the overall behaviour and to obtain a more accurate result in the end. A linear fit of the data was made, see figure 4.3, in order to obtain the slope, which corresponds to the axial piezo constant. Comparing this value to the one we obtained from the theoretical model, Eq. (3.6), shows a small difference. The experimentally determined value seems to be a little smaller than the theoretical one. This is most likely because of the way we fixed the piezo tube to the probe mount, reducing the effective length of the tube. Another cause could be the tip holder and cable adding a weight load to the the tube and therefore decrease the ability to expand.

Figure 4.2: Height profile from one line of the AFM-scan and the corresponding amplitude spectrum.
Figure 4.3: Linear fit of the experimentally obtained piezo displacement as a function of applied voltage.

Slope: 3.2918 nm/V
4.2 Approach

Before running the automated approach algorithm some manual preparations are done. A copper tip is made, using the cutting method described earlier, and placed in the tip holder. A gold sample is mounted on the stage and positioned below the tip. At this point the distance between tip and sample is in the order of centimetres and would take hours to approach automatically. With the use of an optical microscope the gap can be decreased manually, by stepping the SQUIGGLE, to a point where the separation is only a few tens of micrometer. From here the approach algorithm can do the rest of the approach within a few minutes. A plot of the approach data, see Fig. 4.4, shows

![Figure 4.4: Exponential fit of the tunneling current as a function of piezo voltage.](image)

the exponential behaviour of the current as a function of distance which can only be explained by the quantum nature of the electrons. This result shows that it is possible to get into tunneling distance with the STM, an important step towards scanning. As we see in the figure the current goes from 0 to 1000 bits in approximately 100 bits output voltage at 500 mV bias voltage. These values correspond to a tunneling current from 0 to 3.05 nA in approximately 3.16 nm change in distance, which is quite high compared to others work. The reason for this is most likely the relatively large tip radius, caused by the somewhat primitive tip preparation method, which increases the amount of tunneling electrons and therefore also the current.

Fitting equation (3.10) to the data obtained from the approach yields the value of the parameter $b$, which we need to have in order to make the model-based feedback to work, see Eq. (3.13). In this specific approach a $b$-value of approximately 0.04 was obtained, a
value which several other approaches also yielded.

4.3 Scan

When testing the scan it is necessary to know what results to expect in order to evaluate the performance. Therefore, all scans in this thesis are made on a reference sample with known characteristics - an SPM calibration pattern. It is a gold sample with a matrix of etched down squares with a size of $500 \times 500$ nm and periodicity of $1 \, \mu m$.

4.3.1 Lateral deflection

By scanning the calibration pattern the actual piezo movement can be determined and compared to the theoretically calculated deflection. Figure 4.5 shows a scan which according to theory should correspond to $1.2 \times 1.2 \, \mu m$. A lateral step size of 5 nm is used and the current is measured and averaged over 40 $\mu s$ per pixel. Based on the scan image the side of a square is approximately 450 nm. This value is within 10% of the actual value (500 nm) and implies that the estimated deflection is quite accurate.

Figure 4.5: STM scan of reference sample. Scale is in nm.
4.3.2 Trace-Retrace comparison

Comparing trace and retrace image, Fig. 4.6, can reveal a lot of information. As we discussed earlier it makes it possible to determine how much drift there is during scan. Furthermore, parachuting effects due to slow feedback are scan direction dependent and can "smear out" the imaging of sharp drops in the sample. By looking at the scan image from the opposite direction this smearing will not be visible since the tip was going up the steep edge instead.

On a more basic level, comparing the two images gives a good perception on how reliable the scan is. If the two images match each other it is less likely that random errors have corrupted the result. Even so, systematic errors may still exist.

![Figure 4.6: Comparison of trace and retrace images taken with the STM.](image)

4.3.3 Impact of different measuring times

We also want to know how the image quality is affected by different time of measuring of the tunneling current per pixel. It is desirable to have short time-per-pixel since it would decrease the total scan time. However, the quality must be high enough to be able to distinguish nanopillars. The results of scans with different time-per-pixel are summarized in figure 4.7. All five scans are made consecutively at the same conditions. The lateral step size is 5 nm and the scan size is $1 \times 1 \mu m$. It seems like the reduction in time-per-pixel leads to the walls (between the squares) to appear wider. This might be a parachuting effect due to less accurate feedback, caused by a shorter measuring time, when the tip moves over the wall edge. However, this is only a guess and further investigations are necessary for determining the actual cause.
Figure 4.7: STM scan images of the reference sample with different times per pixel.
4.3.4 Impact of different resolution and measuring times

Scan resolution is another factor which affects the total scan time. Decreasing the lateral step size by a factor of two should certainly result in a more detailed image but would also lead to a four times longer scan duration. This prolongation can however be compensated for by reducing the time-per-pixel by a factor of four and still yield a higher quality than before. In figure 4.8 we see images from three different scans where these two parameters are varied. Judging by the images a higher quality is obtained when the 25 Å step size is used compared to 50 Å. As we discussed earlier this has the cost of a four times longer scan duration. Remarkably, when also decreasing the time-per-pixel a factor of four the result is an image with higher quality than the first one, even though the total scan time is the same.

Figure 4.8: STM scan images of the reference sample with different times per pixel.
Chapter 5

Conclusions

In this thesis we have constructed a fully functional STM, capable of locating nano-sized features on a surface. We have also presented a design for a local grounding method allowing high frequency measurements, which remains to be evaluated. The work done is the first step in the development of an RF-STM with the ability to accomplish nanometer-localized high frequency transport measurements.

In this work much time was devoted to understanding and evaluating the electronic and mechanical components of the STM, in order to motivate the choice of specific implementations, understand their limitations, and utilize them properly. The beauty of applied physics is the opportunity to combine theoretical and practical work. For example, circuit analysis had to be used when designing the amplifier for the tunneling current and soldering skills were required to build it. Furthermore, the piezoelectricity theory was used to determine the properties of the actuator responsible for moving the tip, so that accurate scanning could be done. These properties were also measured experimentally and showed good agreement with the theoretical models. The initial use of the four outer electrodes of the piezo tube for the axial movement had the advantage of reducing the number of high voltage channels but introduced a source of error when having to mix axial and lateral voltages. This configuration made it very difficult to apply the same voltage at the same time on all four electrodes and resulted in a malfunctioning scan, with distortions in the image. The problem was solved by using the inner electrode as an adjustable ground (at the cost of one more HV channel), effectively changing the voltage on all four quadrants. Hence, the control of the axial and lateral motion was separated, which improved scanning.

Although a significant amount of time has been spent on building and testing the hardware the main focus has been on developing computer algorithms for operating the STM. It was necessary to learn how to program in LabVIEW and especially how to make FPGA-based applications. Besides programming functioning approach and scanning algorithms we have implemented a model-based feedback system which uses the known relationship between the tunneling current and tip-sample separation to keep the current stable. This proved to work well as long as the lateral step size was not too big, causing the tip to lose the tunneling range - a known problem that arises with any kind of a feedback system. We found that the quality of the scans got worse if the step size was increased above 50 Å. Moreover, the scanning time was drastically shortened by improving the procedure for gathering the data and reducing the measurement time of the tunneling current per pixel, without deterioration in image quality. A long measuring time does not
lead to better results necessarily. It makes longer-term drifts to have a more pronounced effect and might actually worsen the image quality. At the same time, a short measuring time enhances the effect of high frequency noise and decreases the signal-to-noise ratio, so a trade-off had to be found. We managed to obtain images with sufficient quality with measuring times down to 250 $\mu$s per pixel.

There is always room for improvement and a few suggestions for future work would be: Use a different (thinner) piezo tube to increase the maximum scan size. The current size of $1 \times 1 \mu m$ is relatively small and it would be beneficial to have more room for manoeuvre in cases where a particular feature on the surfaces needs to be found and investigated. Furthermore, using an MMCX receptacle as the tip holder is not very flexible. It requires the tips to have the exact correct diameter to fit and remain solidly inside. The copper wire we used for making tips had a larger diameter and had to be filed off in order to fit in the holder, a tedious process which did not always work. A holder with some kind of a clip would solve this problem and would simplify tip preparation significantly.

On the whole, the experimental results confirm the functionality of the STM and show that we have a good basis for a continuation of the project. The fact that we have obtained scan images reproducing the appearance of known calibration samples is the ultimate proof of success.
Chapter 6
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