A Vertical Coarse Approach Scanning Tunneling Microscope

by

Benedict Drevniok

A thesis submitted to the Department of Physics, Engineering Physics, and Astronomy in conformity with the requirements for the degree of MSc

Queen’s University
Kingston, Ontario, Canada
June 2009

Copyright © Benedict Drevniok, 2009
Abstract

A Pan-style scanning tunneling microscope (STM), with a vertical coarse approach mechanism, was designed, built and tested. The microscope will be operated in ultra-high vacuum and also at cryogenic temperatures (8 K) inside a continuous flow cryostat. Fundamental differences in operating principle exist between the new microscope and the beetle-type inertial sliders [1] that have been the mainstay of the group for the last eight years. While Pan-style microscopes do already exist [2], they remain challenging to build, and an active area of research [3]. This system represents a bold departure from well-trodden paths, and will greatly expand the range of experiments that our group can perform.

The operating principles of inertial piezoelectric motors are detailed. Design guidelines for a piezoelectric motor are given, and used in the design of the vertical coarse approach motor. A simple, inexpensive implementation for creating waveforms with an extremely fast fall time is discussed. Motor performance is tested, and a minimum step size of 20 nm is found for frequencies ranging from 0 Hz to 3 kHz. The motor operates with high dynamic range: individual 20 nm steps can be taken, as well as being able to move at a velocity of 0.4 mm s$^{-1}$.

Little is known about the vibrational properties of Pan-style microscopes. Vibrational testing of the microscope revealed the expected scanner bending mode at
1.6 kHz (above the scanner bending mode of our beetles at 1.2 kHz), and a complicated response signal above this frequency. Custom extension springs for an eddy-current damping system are built and tested. A low resonant frequency of 1.8 Hz is found, which is ideal for the application.

Initial testing of the STM in ambient conditions is performed on two different surfaces. A moiré supermesh [4] with periodicity 3 nm is observed on a highly-oriented pyrolytic graphite (HOPG) surface, and agrees well with previously published results. Using a flame-annealed Gold on mica surface, a low drift rate of 0.6 nm s$^{-1}$ is observed over a period of 13 minutes. Single-height atomic steps are observed on both surfaces. Additionally, the microscope is shown to be capable of zooming into different features on a surface, and scanning at different length scales.
Acknowledgments

Alastair McLean. Your patience and belief in my abilities have been invaluable. Not everything has proceeded according to plan during the last three years, but you have remained positive the entire time. Thank you. Andrew Mark. Spending time with you in lab has made me a better scientist. You have been a major driver behind this work. Arguing the finer points of copyright law will not be same with anyone else. Thank you. Jay Weymouth. I throw bad ideas at you, and receive good ideas in return. Your enthusiasm and success have, and continue to, allow me to strive for bigger and better things. Thank you. Will Paul. Nothing in this thesis would move without your tireless assistance. The inventiveness of your work inspires me. Thank you. Gary Contant, for building the STM, and so many failed prototypes. Thank you.

Mom, Dad, Ulrika, Daly, Thomas, Sasha and John. The last three years have been very challenging for me. Nothing makes me feel better than a solid round of applause when I walk through the door at 28 des Mangliers. Thank you, and I love you all.
# Contents

Abstract .................................................. i  
Acknowledgments ........................................... iii  
Contents ...................................................... iv  
List of Tables ............................................... vi  
List of Figures .............................................. vii  

1 Introduction .............................................. 1  
1.1 Outline .................................................. 3  

2 Scanning Tunneling Microscopy .................................. 5  
2.1 Theory .................................................... 5  
2.2 Operation ............................................... 10  
2.3 Temperature ............................................. 12  
2.4 Vacuum ................................................... 14  
2.5 Vibration ............................................... 15  

3 Coarse approach in scanning probe microscopy ...................... 17  
3.1 Piezoelectric ceramics .................................. 18  
3.1.1 Shear Plate Piezo .................................... 21  
3.2 Horizontal Motors ...................................... 22  
3.3 Vertical motors ......................................... 27  

4 Design and testing of the Zmotor .................................. 37  
4.1 Design of Zmotor ........................................ 38  
4.1.1 Body .................................................. 40  
4.1.2 Piezoelectric shear plate stacks ...................... 41  
4.1.3 Slider .................................................. 47
4.1.4 Spring, back plate and ruby ball ............................. 48
4.1.5 Motor assembly .............................................. 50
4.2 Snap-down piezo driver ......................................... 51
4.3 Testing ............................................................ 54
4.3.1 Circuit and hardware ......................................... 55
4.3.2 Distance measurements ................................. 57

5 Design and Testing of the STM ............................. 65
5.1 Operation .......................................................... 65
5.2 Design ............................................................. 67
  5.2.1 Chassis ......................................................... 68
  5.2.2 Sample plate ................................................. 70
  5.2.3 Sample holder ............................................... 71
  5.2.4 Piezoelectric tube scanner .............................. 72
  5.2.5 Tip holder ...................................................... 74
  5.2.6 Tips ............................................................. 75
5.3 Vibrations .......................................................... 76
5.4 Initial results ....................................................... 81
  5.4.1 HOPG .......................................................... 82
  5.4.2 Au on Mica .................................................... 85

6 Conclusion ......................................................... 87
6.1 UHV Chamber ...................................................... 88
6.2 Cooling cans ....................................................... 89
6.3 Outlook ............................................................ 90

Bibliography .......................................................... 92
List of Tables

3.1 Piezoelectric strain coefficients ........................................... 20
4.1 Distribution of piezoelectric stack heights. ............................ 45
4.2 Time constants for different piezo amplification schemes. ......... 54
List of Figures

2.1 Simple 1D tunneling junction. ........................................ 6
2.2 Model used for Bardeen formulation. ............................... 8
2.3 Schematic of STM generating an image line. ..................... 11
2.4 Simple model of vibrations affecting STM performance. ....... 15
3.1 PZT unit cell. .......................................................... 19
3.2 Deformation of shear plate piezo. ................................ 21
3.3 Schematic of the operation of a horizontal inertial motor. .... 23
3.4 Free-body diagram for a horizontal inertial motor. .......... 24
3.5 Phase diagram for a horizontal motor. ............................ 26
3.6 Simple diagram of a vertical motor. .............................. 28
3.7 Friction motor producing a step. .................................. 29
3.8 Vertical inertial motor producing a step. ......................... 30
3.9 Free-body diagram of a vertical motor. ......................... 31
3.10 Phase diagram for a vertical motor. .............................. 33
3.11 Uneven loading on the slider. .................................... 35
4.1 Overhead view of Zmotor. .......................................... 39
4.2 Zmotor exterior body. ................................................ 41
4.3 Model and photograph of piezoelectric shear stack. .......... 42
4.4 Stack assembly process. ............................................. 43
4.5 Charging and discharging a piezoelectric shear plate. ....... 46
4.6 Labelled photograph of the slider. ............................... 48
4.7 Ruby ball, spring and backplate. .................................. 49
4.8 Schematic of snap-down circuit. .................................. 53
4.9 Illustration of distance sensor. ................................... 57
4.10 A calibration curve of the distance sensor. ...................... 58
4.11 Single voltage step size curves. ................................ 60
4.12 Phase-space plots of motor performance. ....................... 62
4.13 Distribution of step sizes. .......................................... 63
5.1 Schematic diagram of connections in an STM. ................ 66
Chapter 1

Introduction

The effect of a tool-driven revolution is to discover new things that have to be explained.


Experimentalists rely on good tools and great techniques. Advancements in many fields come about when a new experimental tool or technique is applied successfully to a system that has defied existing tools and techniques. Scanning tunneling microscopy (STM) proved its worth by helping sort out the atomic structure of Si(111)-7×7, a problem that had eluded the surface science community for nearly 30 years [5, 6]. Scanning probe microscopy (SPM) has matured and flourished for the last 25 years, with new tools and techniques enhancing the types of research available to SPM scientists. These tools include the scanning tunneling microscope [7], atomic force microscope [8], magnetic force microscope [9], and many others [10, 11]. Techniques like scanning tunneling spectroscopy (STS) [12], atomic manipulation [13], inelastic tunneling spectroscopy [14], and spin-polarized STM [15] have created exciting new
avenues for multiple branches of science.

The advent of STM enabled the study of the electronic structure of surfaces with unprecedented spatial resolution. In STM, a single channel of information, the tunneling current, is available. It is possible to extend standard tunneling microscopy to include another source of information. Using the tip of the STM as a source of low-energy electrons, one may excite photon emission from the sample [16, 17, 18]. Photons are a rich source of information; the intensity, spectral distribution, polarization, or angular distribution of the emitted light may be analyzed. This presents an extremely interesting situation, the extreme spatial resolution of tunnel microscopy may be coupled with the versatile photon channel to provide spatially-resolved spectroscopic measurements. The emission spectra of single molecule may be studied with sub-molecular resolution [19, 20, 21]. More established spectroscopic techniques must average over a comparably large area, and essentially, there is much lower (or no) spatial resolution. STM-induced light emission is unique in its abilities to spatially resolve the source the extremely localized photon emission.

Complications arise when one considers the feasibility of obtaining emission spectra with atomic resolution. The low currents (low electron flux) used in STM will not generate a high photon flux. This demands longer data collection times to obtain spectra with a good signal-to-noise ratio. Extended integration times at a single atomic site (or molecule) requires an extremely stable microscope, as well as low surface diffusion rates. Much care must be put into the instrumental design to ensure that photon-STM is a viable technique.

Ultimately, the goal of this project is to study light emission from individual molecules utilizing the spatial resolution offered by the STM [22, 21, 19, 23]. While
this is not a new technique, it is a relatively nascent field with much room for improvement. Additionally, this represents a new set of techniques and tools available to our laboratory.

Our research group has been focused on studying the electronic structure of semiconductor surfaces [24, 25, 26] using a home-built room-temperature ultra-high vacuum scanning tunneling microscope [1, 27]. The adsorption of small organic molecules or the growth of various atomic species on the surface may be investigated with atomic resolution. The tools and techniques provide a solid base on which to perform interesting science. Alas, the current technology has limited applicability. An expanded research scope demands a new set of tools.

The initial challenges of producing a microscope capable of atomic resolution light-emission scanning tunneling microscopy provides the motivation behind this work. Therefore, the goal of this thesis is:

\begin{quote}
\emph{to develop and characterize an isothermal vertical coarse approach STM designed for operation at cryogenic temperatures in ultra-high vacuum, including an in-depth study of the principles behind piezoelectric vertical coarse approach systems to ease future development.}
\end{quote}

\section{Outline}

This thesis details the design, construction, and initial testing of a low-temperature, ultra-high vacuum scanning tunneling microscope. Initial STM results in air, as well as the major challenges in STM design are presented.

The major theoretical background of STM is presented in Chapter 2 and discussed
in terms of the requirements it places upon high-resolution STM. The need for a low-
temperature, ultra-high vacuum system is placed in context of the operating principle
of a tunneling microscope.

Chapter 3 details the operating principles of different piezoelectric coarse approach
motors used in SPM. Simple physical models for each type of motor are presented,
along with the advantages and disadvantages of each. Chapter 4 deals with the design
of a vertical approach motor. Decisions made during the design process are discussed.
Assembly and testing of the vertical approach motor are presented at the end of the
chapter.

Chapter 5 presents the result of the design and testing of a scanning tunneling
microscope with a vertical coarse approach. Initial results, obtained in ambient con-
ditions are presented.
Chapter 2

Scanning Tunneling Microscopy

The following discussion provides a theoretical basis of the experimental techniques used throughout the thesis. While more exhaustive descriptions of STM can be found elsewhere [28, 29], this chapter is sufficient to put certain design decisions in context.

2.1 Theory

A schematic representation of electron tunneling is presented in Fig. 2.1. This system is typical of metal-insulator-metal tunneling junctions, which have been studied extensively in the past [30]. Many theoretical approaches to the description of these systems have been developed. Among the most successful, and most widely-used, is Bardeen’s time-dependent perturbation approach that was developed in 1960 [31].
Figure 2.1: The schematic in (a) represents a scanning tunneling microscope. A metallic tip is brought within close proximity of a conductive surface, and a bias \( V \) is applied across the tunneling gap. Tunneling (b), is the physical effect exploited in STM.

In a basic picture, the tunneling junction in STM consists of a conductive sample surface, with a metallic tip situated some distance \( z = s \) above it. An electron may tunnel through the classically forbidden region of width \( s \). The electron’s wavefunction, shown in Fig. 2.1(b) decays exponentially in the forbidden region, resulting in a non-zero probability of finding the electron at \( z > s \). With one electrode held at a potential \( V \) with respect to the other, a net current will flow. This is the physical effect exploited in tunneling microscopy.

Although Bardeen’s theoretical description of tunneling was developed for planar tunnel junctions, it can be extended to describe the non-planar tip-vacuum-sample geometry found in STM [32]. Here, a one-dimensional treatment based on Bardeen’s theory will be presented. The end result is to describe the rate of electron transfer from one metal to the other through a vacuum barrier. The density of states in the tip and sample are described by \( \rho_t \) and \( \rho_s \), respectively, and a bias voltage of \( V \) is applied between the tip and the sample (Fig. 2.2(b)). Chen [28] presents a form of
Bardeen’s solution for the tunneling current, flowing between tip and sample, where

\[ I = \frac{4\pi e}{\hbar} \int_{-\infty}^{+\infty} [f(E_F - eV + \epsilon) - f(E_F + \epsilon)] \rho_s(E_F - eV + \epsilon) \rho_T(E_F + \epsilon) |M|^2 d\epsilon, \]

(2.1)

\( f(E) \) is the Fermi distribution function, \( E_F \) is the Fermi level, and \( |M| \) is the tunneling matrix element. To arrive at this expression, the coupled system in Fig 2.2(a) is treated as two separate systems. The derivation of this equation can be found elsewhere [28]. The important elements of the tunneling integral include: the Fermi-Dirac distribution for both the tip and sample, the density of states of the tip and sample, and the tunneling matrix element.

The Fermi function, \( f(E) = \{1 + \exp (E - E_F/k_BT)\}^{-1} \), determines the occupancy of electronic states at temperature \( T \). As \( T \to 0 \), the Fermi function becomes a step function and the energy resolution of the STM, when it is used in spectroscopic modes (scanning tunneling spectroscopy, light-emission spectroscopy) is improved.
Figure 2.2: In the Bardeen formulation, tip and sample are treated as separate subsystems. Wavefunction overlap is calculated at a separation surface between the tip and sample. In (b) the different density of states of tip and sample are illustrated. In this example, elastic tunneling will only occur from the filled states of the sample, those below $E_F$, into the empty states of the tip (with energy shifted by $eV$).

A convolution of the density of states of the tip $\rho_t$ and sample $\rho_s$ also affects the tunneling current $I$. If the tip has a constant (free-electron metal) density of states, information about the sample’s density of states can be inferred from the magnitude of the tunneling current at a particular voltage. Tunneling current measurements, therefore, are affected by the electronic structure of the sample in question.

The final component of Eqn. 2.1 is the tunneling matrix ($M$). In Bardeen’s theory, the matrix element is calculated by performing an integral of the sample and tip states over a separation surface. $M$ describes the overlap of sample ($\psi$) and tip states ($\chi$), as shown in Fig. 2.2(a). Following Bardeen’s description,

$$M = \frac{\hbar^2}{2m} \int (\chi^* \nabla \psi - \psi^* \nabla \chi) dS.$$  

If tip and sample states were known, calculation of the matrix element $M$ would be
trivial and the tunneling current could be calculated. The issue is that the wavefunctions of the tip and sample are not always known. Determining the wavefunctions of tip and sample is not simple, and as such, determining the matrix element $M$ has been the subject of intense study for some time [29, 33, 32].

Here, a simple one-dimensional approach is sufficient and will be used to describe the parameters that are key to the design of an STM. Considering a square barrier, as in Fig. 2.2(a), the 1D wavefunctions are:

$$
\psi(z) = \psi_0 e^{-\gamma z}, \quad 0 \leq z \leq s, \text{ and }
$$

$$
\phi(z) = \chi_0 e^{\gamma(z-s)}, \quad 0 \leq z \leq s. \quad (2.3)
$$

The decay constant $\gamma = \sqrt{\frac{2m(eV - E)}{\hbar}}$, where $eV - E = \phi$, and the work function $\phi$ is assumed to be the same for both the tip and sample. We may then evaluate a much simpler form of $M$, at the separation surface $z = a$,

$$
M = \frac{\hbar^2}{2m} \left( \chi^* \frac{d\psi}{dz} - \psi^* \frac{d\chi}{dz} \right) = \frac{\hbar^2}{2m} \gamma e^{-\gamma s}. \quad (2.5)
$$

This results in an amplitude $|M|^2 \propto \gamma^2 \exp(-2\gamma s)$. The tunneling current (related to $|M|^2$) depends exponentially on the distance between tip and sample. A reasonable value for the decay constant $\gamma = 1 \text{Å}^{-1}$, which results in a current that changes nearly a decade per Ångström. A scanning tunneling microscope can be sensitive to changes in tunneling current that are related to changes in separation orders of magnitude less than an Ångström.

The tunneling current depends on: the thermal broadening of the electronic states, the density of states of both sample and tip, and the distance between sample and
tip. Interpretation of STM data, therefore, can be challenging at times as it is a convolution of both the electronic and structural effects.

### 2.2 Operation

Although a one-dimensional model has been developed in the previous section, the STM is intrinsically a local probe. The exponential behavior of the tunneling current is exploited to create a real-space image of a surface with atomic resolution. In the following section, the operation of a scanning tunneling microscope will be described.

In STM, a sharp conducting tip is placed in close proximity to a conducting surface, sufficiently close to create wavefunction overlap. When a bias voltage is applied between the tip and sample, a tunneling current will flow. The tip is then moved parallel to the surface while the magnitude of the tunnel current is measured and recorded. The magnitude of the current as a function of position is then used to create a map of the surface. Alternatively, the height of the tip can be adjusted, under feedback control, to keep the tunnel current constant. Then, the height that the tip is moved perpendicular to the surface is recorded at each point and used to generate a map of the surface.
Figure 2.3: Schematic of a single-line constant-current scan in STM. The vertical tip position ($z$) is recorded as a function of lateral tip position ($x$). In constant-current mode, tip height is controlled to keep the tunneling current constant. This mode is used to more often, as it protects the tip from crashing into the surface.

The two topographic modalities of the STM are constant-current and constant-height. As this document only features constant-current images, only this process will be discussed.

Fig. 2.3(a) demonstrates the generation of a single scan line in the constant-current topographic mode. The tip and surface are brought into close proximity ($s < 1.0$ nm), to create wavefunction overlap and generate a tunnel current. Tip position can be controlled in three dimensions in an extremely precise manner using a piezoelectric scanner. Over a single point, at a set height and fixed bias voltage, the tunneling current will remain constant. The tip is then scanned across the sample surface, while a feedback loop controls the tip-sample separation, keeping the tunneling current constant along the line. Changes in tip height are recorded and create a single line scan. Rastering across an area produces an entire image, built up line-by-line. The previously mentioned electronic and structural contributions can make an image
difficult to interpret. In Fig. 2.3(b) the topography line appears very similar to the atomic corrugation, essentially replicating the surface. This is not always the case as electronic effects that could be due to an impurity atom in the surface layer can alter the tunneling signal. Electronic effects are strongly influenced by the bias voltage setting. It is often possible to identify strictly electronic effects by scanning a similar area with many different bias voltages.

2.3 Temperature

Temperature is a very important parameter in scanning tunneling microscopy. If the microscope is operated isothermally and also at low temperature the effect of thermal drift of the tip relative to the sample can be dramatically reduced. The other benefit of temperature control is that it allows many processes on surfaces that depend critically on temperature to be studied. These include adatom and molecular diffusion [34] and the nature of surface-molecule adsorption, with physisorption being more common at low temperature [35].

The tunneling current’s extreme sensitivity to tip-sample separation is a benefit for high-resolution imaging. Unfortunately, one repercussion is that thermal gradients in a typical lab setting are no longer negligible. Considering the linear thermal expansion

\[ \Delta L = (\alpha \Delta T)L, \] (2.6)

of a material with coefficient of thermal expansion \( \alpha \), can reveal the effect of temperature on tunneling gap. Typical values for \( \alpha \) for various materials used in STM construction range from \( 10^{-6} \text{K}^{-1} \) – \( 10^{-5} \text{K}^{-1} \). Given a change in temperature \( \Delta T = \)
0.01 K, and a standard thermal path in a STM $L = 1\, \text{cm}$ results in a change in length $\Delta L = 1\, \text{nm}$. If operating without environmental controls, such a small change in temperature is plausible. This kind of temperature change would greatly alter the tunneling current.

The time scale involved in a STM scan typically eliminates some of the concerns related to linear thermal expansion. An image with a pixel density of $256 \times 256$ can be acquired in slightly under one minute. The feedback control performed by the electronics operates at a rate high enough to allow the tip to remain in position for constant current imaging. Spectroscopic measurements require that the tip is placed above an atom or molecule for an extended period of time. Typically this may be tens of minutes or an hour [13]. While steps can be taken to minimize the effects of thermal expansion, exact matching of coefficients of thermal expansion is very difficult and temperature variations will still occur.

A tried and tested way of eliminating differential thermal contraction is to place the microscope in an isothermal container. When operating in an isothermal mode, where the sample and microscope are held at the same temperature, the temperature can vary as little as 0.01 K/day. This allows a single atomic site to be imaged for over 24 hours [36, 13]. Vertical drift rates as low as 0.001 Å/min have been observed [37]. This level of performance is achieved by cooling the entire assembly with liquid nitrogen or liquid helium. When allowed to thermally equilibrate, no differential thermal expansions will occur on any reasonable time scale. These design concerns have been previously considered by other researchers [38, 39, 40].

The sample temperature also has direct consequences for the thermal motion of atoms and molecules adsorbed on the surface of the sample. As the temperature
is reduced so is the diffusion rate. At cryogenic temperatures most adsorbates are immobile and therefore they can be readily imaged and in some cases also manipulated [13]. To acquire the highest resolution images operation in the 4 K range is necessary. Moreover, studying molecules on a low-temperature surface allows for physisorption to the surface, which removes the perturbation of a strong chemical bond.

2.4 Vacuum

Being a probe with atomic resolution, STM is sensitive to low levels of contamination on a surface. Like most surface science techniques, STM is best when performed in an ultra-high vacuum (UHV) environment. Through the use of specialized pumps and stainless steel vacuum chambers, the operating pressure of a UHV STM can be around $3 \times 10^{-11}$ Torr. Lowering the pressure increases the amount of time that a sample will remain free of unwanted adsorbates. Pressure around $10^{-10}$ to $10^{-11}$ Torr will keep a sample clean for 12 to 24 hrs. While STM can be performed in ambient conditions, high resolution experiments that require long-term stability must typically be performed in UHV conditions.

By combining both low-temperature and ultra-high vacuum, extreme sample cleanliness may be achieved. Reports of samples with no noticeable contamination over a period of weeks are available [13]. This is a direct result of the cryopumping pumping by the elements in the system that are cooled.


2.5 Vibration

The exponential dependence of the tunneling current on tip-sample separation makes the STM extremely sensitive to vibrational noise that may perturb the tip-sample spacing. Many discussions of these problems exist in the literature, of note would be the treatments by Park [41], Chen [28], and more recently by Kern [42].

![Diagram of STM vibrational coupling](image)

**Figure 2.4:** A very simple model to study the effect of external vibrations on the tip-sample gap in STM. The body of the STM acts as a stiff spring $k_s$ with some internal damping $b_s$.

A simple model of STM vibrational coupling is shown in Fig. 2.4. Here the microscope consists of a sample and tip attached via a single metal body, which can be modelled as a spring with some internal damping. This system can be described by the following equation of motion:

$$m_s \ddot{x}_1 + b_s (\dot{x}_1 - \dot{x}_0) + k_s (x_1 - x_0) = 0, \quad (2.7)$$

where $m_s$ is the mass supported by the STM body, $b_s$ is a damping coefficient, and $k_s$ is the spring constant. External vibrations are coupled into the tip-sample spacing...
through the position of the sample body, $x_0$. Changes in the tunneling gap $z = x_1 - x_0$ will show up in the tunneling current signal, and are not \textit{a priori} distinguishable from true changes in electronic structure. The body must be connected to the outside world in some manner. The design challenge is thus to isolate the microscope from external disturbances as much as possible.

Performing the highest resolution, highest stability measurements requires a cryogenically cooled, ultra-high vacuum scanning probe system with proper vibration isolation.
Chapter 3

Coarse approach in scanning probe microscopy

Scanning probe microscopes bring a tip and sample to separations of less than a nanometer. This presents an interesting and challenging design problem: the system must be able to control the tip with sub-Angstrom resolution while translating it over millimeter distances. Typically, this is solved by decoupling the very fine tip control and coarse translation. A coarse approach motor is used to translate over millimeter distances with very small step sizes (a few nanometers), while a scanner is used for precise motion. The scanner is in many ways a solved problem. Most modern designs have converged on the use of a segmented piezoceramic tube which permits three-dimensional tip adjustments with the desired resolution. Coarse approach mechanisms are more challenging.

The need to operate microscopes in both ultra-high vacuum (UHV) and at cryogenic temperatures creates experimental challenges. These challenges have driven innovation along different paths and a number of different coarse approach systems
are now in use [43, 44, 45, 46, 34, 2, 47, 48]. While coarse approach in a scanning probe microscope may be achieved in many different ways, the focus here will be on piezoelectric-based motors, which have proven to be the most reliable.

Although a large number of publications can now be found in the peer-reviewed literature describing different coarse approach methodologies, there is a clear gap in the literature; few of these papers describe the principles of operation in detail [2, 49, 50, 46]. The intention here is to describe the operating principle of two families of approach motors, highlighting their differences. With an understanding of the key factors behind a properly operating motor, choices for a new design will become clear.

3.1 Piezoelectric ceramics

The direct piezoelectric effect refers to the generation of a electric potential by a material due to the material being mechanically stressed. Similarly, the reverse piezoelectric effect refers to the mechanical deformation of a piezoelectric material in response to an applied electric potential. Many natural materials, such as quartz, topaz, and Rochelle salt, as well as man-made ferroelectric ceramics such as lead zirconate titanate (PZT) exhibit such behavior. In the following, the focus will be on PZT.

Below the Curie temperature, the perovskite structure of a PZT unit cell (Fig. 3.1(a)) is deformed, as in Fig. 3.1(b). A consequence of the deformation is a polarization of the unit cell. Each PZT cell is then piezoelectric. Owing to the ceramic nature of the material, domains of individual cells are polarized in random directions, which results in no net polarization at length scales that can be considered large compared to the size of a unit cell. PZT is also ferroelectric; the polarization direction of the domains can be changed through application of an external electric field. With the
domains aligned as in Fig. 3.1(d) the ceramic may be used as an extremely reliable piezoelectric material.

![Figure 3.1: The structure of lead zirconium titanate; Lead in green, Oxygen in blue, Titanium and Zirconium in red. The perovskite structure in (a) is distorted (b) when the material is at a temperature that is below the Curie temperature, creating a polarized unit cell. Random orientation of polarization in domains (c) can be manipulated by an external field. Polarization remains aligned after the field is removed in (d).](image)

Piezoelectric ceramics provide a method to translate electrical energy into mechanical energy. For scanning probes, these are extremely useful as the deformation occurs in a continuous manner, and can be made to be arbitrarily small. Typically, this is only limited by the precision of the voltage source controlling the motion. Other effects, owing to internal heating and hysteric behavior of the material do affect the absolute positional stability of PZT. We ignore these for the most part, as they are typically small effects at voltages and frequencies used in scanning probes. Additionally, these elements contain no moving parts, they can be made in essentially any shape, and they feature fast response times. Finally, they are safe to use in ultra-high vacuum and can operate at cryogenic temperatures. The mechanical response
CHAPTER 3. COARSE APPROACH IN SCANNING PROBE MICROSCOPY

20

of a PZT ceramic to an applied voltage is, in a very simple form

\[ \Delta L = \alpha \Delta V, \]  

(3.1)

where \( \Delta L \) is the expansion in some direction, \( \Delta V \) is the change in applied potential, and \( \alpha \) is some constant term that depends on the geometry of the element and the alignment between the applied field and polarization of the element.

It follows, that the piezo acceleration is given by

\[ a_p = \frac{d^2 \Delta L}{dt^2} = \alpha \frac{d^2 V}{dt^2}, \]  

(3.2)

and that the acceleration of the piezo element is related to the second time derivative of the applied voltage. This description of the behavior of the piezo element is not entirely complete. However, it is accurate to first order and illustrates that the acceleration of the voltage determines the maximum possible acceleration of the piezoelement.

<table>
<thead>
<tr>
<th>piezo type</th>
<th>( d_{31} ) (293K) [pm V(^{-1})]</th>
<th>( d_{31} ) (4.2K) [pm V(^{-1})]</th>
<th>( d_{15} ) (293K)</th>
<th>( d_{15} ) (4.2K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>EBL 2</td>
<td>-170</td>
<td>31</td>
<td>580</td>
<td>105</td>
</tr>
<tr>
<td>EBL 3</td>
<td>-260</td>
<td>33</td>
<td>730</td>
<td>92</td>
</tr>
</tbody>
</table>

Table 3.1: Strain coefficients \( d_{ij} \) for two types of piezoelectric ceramics used in scanning probe designs at both room and LHe temperatures.

While many different types of piezoelectric elements exist, two types will be examined in detail: the shear-type element and a four quadrant tube. Differences in these elements arise from polarization direction and geometry.
3.1.1 Shear Plate Piezo

Figure 3.2 demonstrates the deformation produced by a shear plate when different voltages are applied. In the case of a shear plate, the deformation as a function of applied voltage is given by

\[ \Delta L = d_{15} \Delta V, \]  

(3.3)

where \( d_{15} \) is the appropriate strain tensor element for a field applied perpendicularly to the polarization direction. From Table 3.1, it is found that the deformation of an EBL3 shear piezo with an applied voltage of 100 V will be 73 nm. This is a precise level of control through rather simple means. One may induce repeatable deformation in a piezo through application of any arbitrary waveform, tailored specifically for the situation. Typical piezo material used in scanning probes can be cycled safely at high frequencies (100 KHz) and high voltages (300 V - 400 V).

![Figure 3.2: A shear plate piezo element with (a) no field applied and (b) with an electric field applied that causes a deformation of the plate. For illustrative purposes, the shearing of the plate has been exaggerated with respect to the size of the shear plate.](image-url)
Dynamic operation of a piezo introduces other concerns. If one wishes to induce motion in a repeated fashion, the capacitive load of the PZT results in a required current of

\[ i_{\text{min}} = C_{\text{PZT}} \frac{dV(t)}{dt}, \]  

(3.4)

where: \( i_{\text{min}} \) is the minimum required current, \( C_{\text{PZT}} \) is the capacitance of the piezo, and \( V(t) \) is the time-varying voltage. This sets a limit for the rate at which the piezo can expand. A large change in voltage in a short time (large \( dV(t)/dt \)) requires a high current. Slew rate and maximum current output of the amplifier are important factors, as they limit the maximum operating frequency of the piezo. Problems can arise when the high-voltage amplifier used to drive the piezo cannot supply the current required by Eqn. 3.4. This will be discussed in a later section.

An overview of the different types of coarse approach mechanisms will now be presented. Only those motors commonly used in a LT-UHV environment will be discussed. The different methods are divided into three main categories: horizontal inertial motors (HIM), friction motors (FM), and vertical inertial motors (VIM).

### 3.2 Horizontal Motors

A common method of coarse approach is the horizontal inertial motor. These come in many different forms, each relying on the same basic principle of operation. The motor consists of three important parts: a static base, piezoelectric elements, and a moveable mass. While many types of horizontal inertial motors exist, a few designs have become popular for use in LT-UHV STM. These are the Besocke and Wilms beetles, used with success by several different research groups as both homebuilt and
commercially made devices [44, 43, 37, 51].

Figure 3.3: The stick-slip operation of a horizontal inertial motor is shown schematically. (a) The mass and legs are static. (b) As a slow ramping voltage is applied to the piezoelectric legs, the mass and legs move with each other. (c) The voltage is then changed rapidly, the legs slip underneath the mass. This completes a step. The process is then repeated, as in d.

In Fig. 3.3, the operating principle of a horizontal inertial motor is presented. The motor moves a mass with a stationary base and piezos. Motion is achieved in steps, which is produced with a stick-slip motion. Initially, the moveable mass, piezo, and base are static. By applying a slowly rising voltage to the piezo element, the moveable mass and piezo move in unison, due to static friction (Fig. 3.3(b)). The piezo expands by an amount proportional to the input voltage. This is the stick phase. A rapid change in voltage then follows, causing the piezo to contract to its
original position, leaving the mass behind in its new position (Fig. 3.3(c)). This is the slip phase. Therefore, step size should be equal to the expansion of the piezo element. Repeating the process, as in Fig. 3.3(d), will produce macroscopic positioning, with high resolution.

\[ F_P = F_F^{\max} = \mu_s F_N = \mu_s m g, \]  

(3.5)

where \( F_F \) is the force due to friction, and \( F_P \) is the force on the shaft as a result of the accelerating piezo elements. This leads to an expression for the minimum possible piezo acceleration that will allow slipping between the piezo element and the mass to occur. This minimum piezo acceleration \( a_p \) is given by

\[ |a_P| \geq \mu_s g. \]  

(3.6)

Examining Eqn. 3.6 reveals that the HIM presents a simple constraint on motion.

**Figure 3.4:** Forces considered for a model of the horizontal inertial motor.
The piezo acceleration \( a_p \) must be just large enough to overcome static friction \( \mu_s g \). Then, as in Fig. 3.3, by using a suitable waveform, a step may be produced. More complex models of HIM-type motors have been presented \([52, 53, 54]\), and they are capable of reproducing dynamic behaviour, though the simple model presented here encapsulates the most important effects. The key factors in a horizontal motor are the acceleration of the piezos and the static friction between the piezo and mass.

Most horizontal motors used in scanning probes are designed as a tripod. The mass is supported by three legs that contact the surface with smooth hemispheres. The three point contact provides a well-defined plane and even loading on all three piezo actuators. In the simple model above, it is assumed that the loading is even, so that a one-dimensional treatment of the situation is valid. Additionally, it is assumed that the plane defined by the three points is the horizontal plane, and that the gravitational force is normal to this plane.
CHAPTER 3. COARSE APPROACH IN SCANNING PROBE MICROSCOPY

Figure 3.5: The restriction on piezo acceleration as it relates to sticking and slipping modes of the motor. The stick and slip regions are delineated by the critical acceleration $a_p = \mu s g$. The vertical axis is not important in this case, but will be used in the next phase diagram.

A motor that is supported only by gravity is particularly simple. The maximum acceleration required to create a slip is simple to describe. Also, loading the three actuators evenly is essentially guaranteed with careful design. This design approach clearly produces motion in the horizontal plane. But it can’t be used to provide motion in a plane other than the horizontal plane. Fig. 3.5 presents a useful phase diagram to visualize the performance of a stick-slip actuator. The requirement for motion relies only on the acceleration exceeding some critical value, and as such, divides the phase space into two regions. Both the stick and slip phase must be accessible by the motor. If the friction coefficient is too low, the slow ramping voltage will create a slip too easily. These diagrams become useful when used to illustrate the
more exacting requirements placed on other types of coarse approach mechanisms.

3.3 Vertical motors

Two types of motors will be discussed in this section, the friction motor (FM) and the vertical inertial motor (VIM). In design and physical appearance the motors are not dissimilar. Differences in actuation method create a distinction between the two. For that reason, they will be discussed in parallel, and differences will be indicated when necessary. Additionally, while these will at times be referred to as vertical motors, they can operate in any orientation.

In the most common form, the motors exist as a sliding element held on three sides by sets of piezo elements, providing both resistance to gravity, as well as a method of producing motion. Fig. 3.6 illustrates a vertical motor. No differences exist between a vertical inertial motor and a friction motor in terms of basic structure.
A vertical motor is based around using a stick-slip type motion where piezoelectric elements are used to move a sliding element. While designs such as Fig. 3.6 are most commonly used, the principle behind the motor can be more clearly seen in a simplified model of the motor (Fig. 3.7). Here, we see that a slider is held in place on three sides by piezo elements. Again, a simple model can be used to describe the behavior of a vertical motor.

The friction motor (FM) that is frequently used in scanning probes, was developed and popularized by S. H. Pan. Unlike the horizontal inertial motor [2], it can produce motion in planes other than the horizontal plane. To produce a single step in a friction motor requires multiple independent control signals. Initially, all piezo elements (Fig. 3.7(a)) are in an equilibrium state. Then, as in Fig. 3.7(b), a fast rising signal is used to accelerate a single piezo element to allow it to slip over the slider surface.
All other piezo elements are held in place such that the slider does not move. Once the slip has occurred, a second signal is used to cause another piezo element to slip over the slider, as in Fig. 3.7(c). This process is repeated until all piezo elements have slipped over the slider, though the slider has not translated. The final movement is a simultaneous slow return to the equilibrium positions by each piezo element. The elements do not slip, and thus create a step, with a size defined by Eqn. 3.3.

![Figure 3.7](image)

**Figure 3.7:** A schematic representation of a friction (or Pan) type motor producing one step.

Single steps in a vertical inertial motor follow a simpler workflow than a friction motor. The VIM begins in an equilibrium state, as in Fig. 3.8(a). In Fig. 3.8(b) a rapidly rising voltage deforms all of the piezo elements simultaneously, causing a slip. A slow return to equilibrium state creates a step (Fig. 3.8(b)), according to Eqn. 3.3.
Many different incarnations of such motors exist in scanning probe literature. The Pan-type motor is used by various research groups. These motors appear very similar to that shown in Fig. 3.6. This includes a middle slider with $D_{3h}$ symmetry, being held on three sides by sets of shear piezoelectric elements [2, 49]. There are also other motors that use the same method of approach, though in appearance are quite different [55, 47]. Despite their differences, these motors are based on the same physical principle.
Figure 3.9: The forces acting on the slider of a vertical motor. To create a slip event, the static friction must be overcome.

Fig. 3.9 illustrates a basic model for an inertial or friction motor held in place when oriented vertically. Using this as our model, the forces on the slider are

\[
F_s = F_N, \quad \text{(3.7)}
\]

\[
F_F - F_g = ma_p, \quad \text{(3.8)}
\]

where \( F_F \) is the force of friction, \( F_s \) is some controllable force provided by an element of the design, and \( a_p \) is the acceleration. As before, this model is used up to the point that static friction can be overcome. This leads to two important equations for our phase diagram. First of all, to produce a step, the piezo must be able to slip over the slider. The requirement for slipping is

\[
|a_p + g| > \frac{\mu_s F_s}{m}, \quad \text{(3.9)}
\]
where \( F_F = \mu_s F_N \) has been used in the static limit. One more limitation exists in a vertical motor. The slider must be held against gravity at all times, including times when the acceleration is zero. The slider is held against gravity when

\[
\frac{\mu_s F_s}{m} > g. \tag{3.10}
\]

Fig. 3.10 is a manifestation of the inequalities presented in Eqns. 3.9, 3.10. When compared to Fig. 3.5, the relative proportion of the area presented that enables slipping (bright green) in a vertical motor is much smaller. The slipping phase is required to create a step in either an inertial or friction motor. Accelerations required to create a slip in a vertical motor must then be higher than in a horizontal motor. A vertical motor must be able to overcome the acceleration due to gravity, as well as a higher frictional force that results from being held in place. The force that is applied to hold it in place cannot be made arbitrarily small, as it must be able to support the slider against gravity.
Figure 3.10: A graphical representation of the accelerations that are required for the operation of a vertical motor. Slipping will only occur small bright green region labelled “slip”. In comparison to the horizontal motor, the situation is much more restricted. Here, “not held” refers to the motor not being held enough by the spring to support it against gravity.

The preceding discussion treated the friction motor and vertical inertial motors as being the same. In some ways, the above model should treat the two approach methods exactly the same. To create a step, the piezo elements of either motor must be able to translate the slider, slip over the slider and hold the slider in place. Differences arise from the magnitude of the accelerations required to slip over the slider in the inertial and friction cases. For an inertial motor, all piezos must slip at the same time. This requires a greater acceleration than the case where only a single piezo element must slip at any one time. The slider in the friction case will resist movement more readily when it is being held fast by all other piezo elements.
By assuming that the slider takes a step of size $s$ at the frequency $f$ of the waveform applied to the piezo stacks, we can arrive at an expression for the distance $d$ travelled by the motor in time $t$:

$$d(t) = sft. \quad (3.11)$$

Eqn. 3.11 would hold for any type of motor that is taking discrete steps at a certain frequency. Relating $s$ to piezo extension and applied voltage from Eqn. 3.3, it is seen that the distance travelled should be proportional to step size, step rate, and time of travel. In a motor like the HIM, this would be true for either direction of motion. In a friction motor, the step size should generally be equivalent if the motor is moving up or down [2]. The slider is continually held in place, and slip events are performed one-at-a-time, so that the slider is never entirely slipping, only the stacks are slipping over the slider.

A difference arises for the vertical inertial motor. In a VIM, if a step is to occur, the slider is, at one point of the process, slipping. If the step is intended to move the slider against gravity, the inertia of the slider must overcome some negative acceleration due to gravity. When moving with gravity, the acceleration due to gravity is positive. This introduces some asymmetry between upwards $d_u$ and downwards $d_d$. As the time and frequency may be considered constant, it is step size up $s_u$ and down $s_d$ that would change. This is not really an issue, it only means that the step size is different.

When a simple model is presented, it is important to take stock of the various simplifying assumptions that are made to create the model. These assumptions will now be examined in more detail. More complex dynamical models of stick-slip actuators have been previously published [53]. There are ignored in this discussion; the dynamical behavior does not have a direct bearing on the operation of a course
CHAPTER 3. COARSE APPROACH IN SCANNING PROBE MICROSCOPY

An approach mechanism in scanning probes. The operating frequency of a coarse approach mechanism is far below the resonant frequency of the structure. Many of the effects occur at frequencies that are not used in normal operation. Additionally, positioning with a coarse approach motor is performed in a step-by-step manner, further reducing the operating frequency. Another complication can be introduced through more complex models of friction at the sliding interface. It is known that the static coefficient of friction will be greater than the kinetic coefficient of friction. To create a slip therefore, the only concern is overcoming static friction. The greatest concern is the manufacture of a motor that operates reliably.

![Diagram](image)

**Figure 3.11:** An uneven piezo element would induce a torque on the slider. This changes the model presented earlier, and the motor will not function properly, if at all.

Another assumption is that the forces on each of the piezo elements are equal. This asserts that the interface between the slider and each piezo element is always perfectly planar, so that no unbalanced force is applied. This is not always the case and it does prove to be an important factor in motor operation. Uneven stacks create the situation depicted in Fig. 3.11. In fact, this case is not so extreme. As the three points will define the position of the slider, if the fourth piezo is only slightly
misaligned in height, the forces will not be balanced. Here, the forces are no longer symmetrically distributed, and a torque is exerted on the slider. This results in extremely unreliable operation or no steps being taken at all.

Careful considerations in design and assembly must be taken to avoid these issues.
Chapter 4

Design and testing of the Zmotor

To maximize flexibility and modularity in the design of a new low-temperature scanning probe microscope, the choice was made to use a clamped inertial-type approach motor. Previous designs in our group have focused around the horizontal inertial (beetle) type of motor [27]. While these are useful instruments, and are capable of producing high-quality data, the limitations of a horizontal motor could no longer be neglected. This approach motor represents a departure for the group, and is a step towards more capable instrumentation.

The design goals for the approach motor were:

- Modularity, create it as a replaceable component.
- Compact design, with a size comparable to other published designs.
- Predictable vertical motion
- Mechanically rigid
- Compatible with liquid helium temperatures.
Modularity allows us to replace the motor or anything that interacts with the motor at a later time. Simplicity in design (the coarse approach has a single function) assists in creating a modular system. The compact design is also helpful in this sense; the motor may be used in many different situations.

Restricting motion to a single axis enables use of a wide range of sample sizes. With previous designs in our group, sample size has been fixed. The method of approach in a linear beetle places additional requirements on sample orientation and size. An approach along a line creates a situation where any sample that is centered on that line may be used. This will allow very small samples to be used which will include bead crystals that are typically only a few mm in diameter.

The basics of inertial motors were discussed in the previous section. Here, more detail regarding the construction, testing and performance of an actual coarse approach motor will be given. The design goals will be fulfilled by using the principles that were presented in the previous chapter. We begin by discussing the functional design choices for each of the major components of the approach motor.

### 4.1 Design of Zmotor

An attempt has been made to design a motor that incorporates many of the successful design choices of previous vertical inertial motors, while improving the design where possible. The components identified in Fig. 4.1 were presented above in Fig. 3.6. They are: the slider, exterior body, piezo elements, and a spring holding things in place.
Functionally, the motor is designed to work as a vertical inertial motor. The inner slider is held on three sides by sets of two shear piezo stacks. The interface consists of polished alumina inserts on the slider pressed against sapphire discs on the piezo stacks. This provides a smooth contact between two surfaces that are optically smooth and flat. Two sets of piezo shear stacks are glued onto the body, while a third set is glued to an independent back plate. A spring presses a ruby ball into the center of the back plate. This provides the force $F_s$ illustrated in Fig. 3.9. By allowing the spring to contact the floating back plate through the single ruby ball, the force of the spring is automatically adjusted to be evenly applied to the slider. An asymmetric sawtooth waveform is then applied to the shear stacks to create the stick-slip motion.

Each component of the motor will now be discussed. The hierarchy of parts is structured by how the actual motor is assembled. Some parts are very simple and
thus do not warrant extensive discussion.

4.1.1 Body

The exterior body of the coarse approach motor serves as an area to affix piezo elements, a base for wire routing, as well as an area to mount it to some superstructure, such as a sample holder. Design criteria dictated that the body be small in size, simple to assemble, and provide options for future alterations.

Overall, the shape of the body takes basic cues from a design which originates in the J.C. Davis lab by S.H. Pan [2]. The interior of the body (see Fig. 4.2) features two 60° walls that support the piezo shear stacks. The shape and size are constrained by the size of other components in the design. The width and depth depend on the size of the piezo shear stacks and slider. The slider must be able to wedge into the space between piezo stacks. Additionally, the body is mirror symmetric along the mid-plane of the translation direction, which creates a design well-suited for temperature insensitivity. The body is 26.5 mm wide, 20.8 mm deep, and 20 mm tall. This size is similar to other designs, and is satisfactorily small [56, 39, 2]. Entirely constructed from a single piece, the body provides a mechanically rigid base for the rest of the motor.
Figure 4.2: Zmotor exterior body. In (a) the location of (1) mounting holes, (2) 60° walls for piezo stacks and (3) area for wire routing pin blocks. The photograph in (b) is of the actual approach motor body. A scale bar is provided for reference.

Fig. 4.2 (a) notes some of the other features of the design. The mounting holes are included to allow the Zmotor to mount to a sample holder. Many motor designs keep the sample holder and approach motor as a single unit. For modularity, we have made the Zmotor independent of a sample holder, providing mounting points that allow it to couple to an appropriately designed receptacle. A flat backed design, which differs from many other designs, is a consequence of the desire for modularity. This provides the ability to completely change sample holder without altering the basic coarse approach.

4.1.2 Piezoelectric shear plate stacks

Shear stacks provide the acceleration required to create movement in an inertial motor. These multi-layer structures are commonly used in inertial motors, due to their
compact size and simple behaviour. They consist of multiple shear plates stacked on top of each other. Through an intelligent choice of stacking direction and electrode placement, the displacement of a stack will follow Eqn. 3.3 with each plate contributing to the total extension. Overall extension $L_s$ of a $n$ layer shear stack is given by

$$L_s = nd_{15} \Delta V.$$  (4.1)

The amplification in displacement is often necessary to produce steps of reasonable size, especially when operating at low temperature.

**Figure 4.3:** A model and photograph of a fully-assembled stack. Each stack is handmade, and consists of: three piezoelectric shear plates (yellow), two sapphire discs (transparent), and four metal electrodes (grey). Displacement occurs in the $\pm \hat{x}$ direction.

To create our shear stack, we followed a method used by the Nanoscience and SPM group of Prof. P. Grütter at McGill University\(^1\). An appendix of assembly instructions is included for completeness and does feature some changes from the original method.

\(^{1}\)Private communication
Fig. 4.3 presents a model of the shear stack along with a photograph of an assembled stack.

For our stack, we use three piezoelectric shear plates of type EBL#3\(^2\). They are assembled as in Fig. 4.4. Of greatest importance here is how the plates are stacked with the polarization always 180° relative to the plate above or below it. This is required to follow Eqn. 4.1. That is, all of the shear plates must shear in the same direction. As the faces that are in contact are at the same potential, the electric field across the bottom plate will be opposite to the field across the middle plate. Opposite polarization creates motion in the same direction when an opposite field is applied. Using Table 3.1, a 100V signal will produce displacement from a three plate stack of \(L_s = 219\) nm.

**Figure 4.4:** A step-by-step series of models representing the stack assembly process. Each numbered step is a unique part of assembling a functioning shear stack. Note the arrows, used to indicate polarization direction. In (1) and (3) \(\vec{P}_1 = -\vec{P}_3\), and similarity for (3) and (5). A thin layer of conductive epoxy is applied at each interface.

Polished sapphire discs\(^3\) are fixed to the top and bottom of each stack. The discs provide an electrically insulating, hard, parallel and smooth sliding surface. The

\(^2\)EBL products - http://www.eblproducts.com

\(^3\)W2.48 windows - http://www.swissjewel.com/sapphire_windows.htm
hardness of the sapphire ensures that no deformation will occur at the slider/stack interface. Optically polished surfaces allow for a sliding surface that will not inhibit slider motion. Stacks are assembled layer-by-layer, as in Fig. 4.4. Individual plates are glued together with conductive epoxy\(^4\), and a metal electrode is sandwiched between each plate, allowing the operator to applying a specific signal to each interface. Thermal expansion coefficients of sapphire, PZT, E4110 epoxy and stainless steel are well-matched. The stacks have been shown to survive exposure to liquid nitrogen.

Home-built stacks are advantageous for two reasons. The first is cost: commercial stacks can cost upwards at CAD 400 each, the stacks described here cost less than CAD 30 each. Secondly, stacks can be constructed in any size. The piezoelectric material is purchased as a large sheet, and diced into smaller pieces.

To ensure that each stack contacts the slider evenly, stacks of even height are very important. Simple in theory, creating stacks of even height can prove challenging. Careful steps in assembly, with quality assurance steps along the production pathway will produce stacks that are equivalent in height. If the stacks are not the same height, the forces on the slider will not balance. If a stack is not in contact with the slider, the normal force at that point will not exist. This will apply a torque on the slider, and it will not move. It has been possible to produce stacks that are similar in height. Additional corrections for the variations in height are made when assembling the motor.

\(^4\)Epotek E41100 (http://www.epotek.com)
### Table 4.1: Distribution of heights of the six stacks built for Zmotor. Each stack height was measured with a vernier caliper from the bottom of the insulating sapphire window to the top of the sapphire disc. The range in height values is $\Delta h = 0.127 \text{ mm}$. The mean stack height is $2.98 \pm 0.04 \text{ mm}$. Stacks were paired with the stack closest in height.

<table>
<thead>
<tr>
<th>stack number</th>
<th>height $h$ [mm]±0.013 mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>3.010</td>
</tr>
<tr>
<td>2</td>
<td>3.000</td>
</tr>
<tr>
<td>3</td>
<td>3.048</td>
</tr>
<tr>
<td>4</td>
<td>2.972</td>
</tr>
<tr>
<td>5</td>
<td>2.921</td>
</tr>
<tr>
<td>6</td>
<td>2.964</td>
</tr>
</tbody>
</table>

Ideally, each stack is also square, so that the polarization is indeed $180^\circ$ relative to each other. This is not as sensitive as the height of the stacks. If each stack’s polarization is nearly $180^\circ$ to the other stacks, the difference is extension will only be a $\cos(\theta)$ factor, as dictated by Eqn. 4.1. This only affects the total extension of the shear stack, it would not change the assumptions made in the models presented earlier.

Capacitance can be used to ensure that each layer has been properly connected. With the plates wired in parallel the capacitance of each plate adds to the total capacitance of each stack. Each plate was measured to have a capacitance of $C_{\text{plate}} = (0.46 \pm 0.05) \text{ nF}$, each stack of three plates was measured to have a capacitance of $C_{\text{stack}} = (1.45 \pm 0.05) \text{ nF}$. Additional capacitance is attributed to epoxy.
Figure 4.5: Piezo charging/discharging while using a standard operational amplifier. The input waveform in (a) is a asymmetric sawtooth, which would produce a high acceleration. After driving the capacitive load presented by the piezo stack with a standard operational amplifier (b) the piezo cannot be accelerated rapidly, and follows a waveform as in (c).

The capacitive load of the stack, along with the output resistance $R_{out}$ of the amplifier used to drive the stack form a $RC$ circuit. As the output resistance is limited to some minimum value, the capacitance determines the time required to discharge the stack, given by $\tau = R_{out}C_{stack}$ [57]. Figure 4.5 demonstrates how the discharge time affects the sawtooth signal used to drive the stack. Referring to Eqn. 3.2, the acceleration of the stack is determined by the waveform used to drive the stack. As the waveform becomes less sharp, the acceleration decreases. Six stacks are used by the motor, which results in a time constant $\tau \approx (5000 \Omega)(6)(1.45 \times 10^{-9} \text{ F}) = 43.5 \mu\text{s}$, using an output resistance of $R_{out} = 5000 \Omega$. It is not simple to tell if this $\tau$ will be able to produce an acceleration great enough to cause a slip. Experience has shown that it is not. This led to the development of an alternate method to accelerate the stacks. This is discussed in a later section in more detail.
4.1.3 Slider

A simple, yet integral part of the system is the slider. Like many other designs, our slider is a truncated triangular prism, clamped on three sides by the piezo shear plates. Other designs require that the slider be constructed from very specialized materials. This is done to ensure that the piezo elements are pressing against a very smooth, hard surface. Often, the prism is made completely of polished sapphire [49, 2], quartz [58], or specially-coated steel [47] which can be quite costly, or challenging to machine. Our design foregoes the need for a specialized slider. As illustrated in Fig. 4.6, polished alumina inserts\(^5\) have been fixed to the slider to provide a smooth, flat, and hard contact surface for the sapphire discs.

At the top of the slider is a fitting for a scanner piezo tube. The slider is hollow to reduce the mass, as well as provide a pathway for wiring used in STM. In the initial version the body is made from aluminum, though this can easily be changed to some other metal if this becomes an issue.

Maximum translation size of the motor depends on the spacing of the stacks on the body, as well as the length of the alumina inserts on the slider. For Zmotor, the maximum translation distance is 4 mm. This number could be adjusted by using a longer slider. In our case, 4 mm proves to be enough coarse translation distance.

Figure 4.6: A photograph of a complete slider. Important features noted are (1) the hollow for wire routing, (2) area to mount a scanner tube piezo, and (3) polished alumina sliding inserts. The slider was made to be very compact and light, without resorting to any extreme measures.

4.1.4 Spring, back plate and ruby ball

To overcome gravity the vertical motor must be held rigidly in place. The body is made so that the slider is held on three sides by piezo stacks. If the body were made completely rigid, any unmatched thermal contractions would lead to a motor that held the slider too loose, too tight, or not at all. For this reason, the third wall in the Pan design [2] is held in place with a spring providing a holding force that can compensate for any changes in the body size. Additionally, this provides a way to mediate the force that holds the slider in place.

Changing the force applied by the spring can be done in three ways. In Fig. 4.7 (a) it can be seen that the backplate that holds the slider in place is supported by a ruby ball that in turn is held by a spring. The spring force can be changed by
tightening the screws that hold it in place, changing the spring constant (by changing the spring dimensions or material), or by changing the size of the ruby ball.

![Figure 4.7: A photograph (a) of the Zmotor, where the spring and ruby ball are evident. Part (b) is a cut-away view, highlighting (1) the spring, (2) ruby ball, and (3) backplate.](image)

The spring is made from a thin (0.08 mm) piece of stainless steel. Other materials, typically Molybdenum [59] or Beryllium Copper [60], are also used as springs. Material choice is dictated by acceptable performance in the available temperature range of 8 - 300 K. The spring constant $k \propto E$, the elastic modulus, and $E$ changes with temperature in a complex way. To date, no problems have been encountered with the spring. Though operation at low temperature may demand a reexamination. There are systems known to operate with a simple stainless steel spring at 4 K with no known issues.

The backplate in Fig. 4.7(b2) is a somewhat uncommon feature. Other motors use a less wide backplate [2] that does not span the entire gap in the body. Our backplate is as wide as the opening. It can still move freely up and down, but it cannot twist.

---

6http://www.cryogenics.nist.gov/MPropsMAY/material%20properties.htm
The ease of alignment offered by the backplate that spans the whole gap is seen as an advantage.

### 4.1.5 Motor assembly

The final assembly of the Zmotor does not proceed in a step-by-step manner. As indicated earlier, a major concern in a functioning motor is a smooth, evenly loaded slider, with each stack contacting in the same manner. A detailed step-by-step instruction list of motor assembly is not the goal of this section. However, there are a few key points that should be considered when assembling the motor. Without great care in assembly, even a well-designed motor may not function.

To assemble the complete Zmotor requires a body, slider, ruby ball, backplate, spring, four 0-80 bolts, six piezoelectric shear stacks, and Torr Seal 7 epoxy. By assembling these parts in a single step, additional alignment between all of the parts can be obtained. The thick epoxy aids by filling in spaces created by stacks that are not exactly the same height. Essentially, the process is:

- Only use very new mixes of epoxy at each step. Have someone else make batches as you assemble.

- Put a small amount of epoxy on the backside of each piezo stack, and place them on the body or backplate.

- Put a small amount of epoxy on the backside of the alumina inserts on the slider, and place them on the slider.

---

• Place the slider into its place in the body. Make sure that the slider is sitting flat on all of the piezo stacks. Ensure that it has balanced well.

• Lower the backplate into place, and make sure that these stacks are also sitting flat.

• Put the ruby ball in place, and tighten down the spring. This does not need to be overly tight, but it does need to hold the slider against gravity.

• Allow the entire structure to cure for at least 24 hours.

The motor may then be disassembled, but it is important to recall that the slider must be used in the motor in the exact same orientation. As this is a custom fit, with many small differences in glue thickness, there is only one correct way to use the motor. Once this is complete the wiring may be run to the stacks.

Only two wires are required to control the Zmotor. The control signal is applied to one side of a plate, while ground is applied to the other. Each stack is controlled by the exact same signal, so any wiring plan that moves the stacks in the same absolute direction is useable.

4.2 Snap-down piezo driver

Standard high-voltage operational amplifiers used in scanning probe microscopy hardware prove to be incapable of producing the required acceleration to drive a vertical inertial motor. Attempts were made to use the RHK Technology SPM100 \(^8\), Nanonis HVA4 \(^9\), as well as a home-built amplifier similar to the one illustrated in

---

\(^8\)http://www.rhk-tech.com/
Fig. 4.5. Commercial piezo amplifier systems that are capable of driving the stacks with the proper acceleration are available, but are very expensive, often costing several thousand dollars each. A simple system that is capable of producing very high accelerations, at very low cost has been developed.

The capacitance of the stack determines the minimum current that must be supplied by a high voltage amplifier in order to drive it (see Eqn. 3.4). As all physical amplifiers are limited by some maximum current, this can cause a problem when moving a piezo quickly. The combination of the stack capacitance ($C_{\text{stack}}$) and the output resistance of the piezo amplifier ($R_{\text{out}}$) produces a RC circuit. The time constant of this circuit is $\tau = R_{\text{out}}C_{\text{stack}}$. The time constant $\tau$ is taken to be an important figure of merit for any loaded piezo driver system. While $R_{\text{out}}$ is also a factor in the time constant it must also be considered that Eqn. 3.4 sets a minimum current required to drive the piezo, and as such, removing $R_{\text{out}}$ altogether does not always improve the situation. Additionally, the output resistor protects the operational amplifier. Driving the piezo stacks with a true sawtooth wave would require a non-physical current. If we require $\tau = 2.5$ s, the minimum current required by Eqn. 3.4 would be $i_{\text{min}} = 0.35$ A, for a sawtooth of magnitude 100 V. The capacitance thereby sets the minimum required current, as well as the maximum piezo acceleration. Given a simple setup as in Fig. 4.5(b), it is not possible to generate high accelerations.

Much of the electronics design is attributed to W. M. P. Paul, a former student in this group. It is presented here as it is an integral part of the functioning motor. Called the piezo snap-down circuit (hereafter referred to as snap-down), the circuit provides a method to take a simple home-built amplifier as in Fig. 4.5(b) and produce accelerations that are much higher.
Specifics of the circuit are laid out in Fig. 4.8. There are two important parts of the snap-down. Signal path 1 Fig. 4.8(a) is an inverting amplifier that uses a PA83 operational amplifier\(^\text{10}\) with ±150 V supplies to amplify a small control signal to levels required to produce large enough translation in the piezo shear stacks. Signal path 2 is a more complicated and important part of the snap-down. There are two sections: the first consists of a set of comparators used to generate a logical HIGH or LOW, based on some threshold voltage. In our system, this threshold voltage is some value just below the peak voltage of the input signal. This produces a HIGH when above the threshold, and a LOW when below it. This HIGH and LOW is then used to control a MOSFET switch. When the switch receives a HIGH signal, it immediately drives the high voltage output of signal path 1 to ground. This is equivalent to

\(^{10}\text{http://www.cirrus.com/en/pubs/proDatasheet/PA83U_P.pdf}\)
having a op-amp circuit that has a much improved $\tau$ (i.e. smaller), and therefore able to produce higher accelerations in the piezo shear stacks. Table 4.2 summarizes the differences between the time constant for a simple op-amp piezo driver and our snap-down circuit. A performance enhancement of at least 18 times is gained. The accelerations produced are much higher, and this allows the slip phase to occur.

<table>
<thead>
<tr>
<th>circuit</th>
<th>time constant $\tau$ [\mu s]</th>
</tr>
</thead>
<tbody>
<tr>
<td>simple op-amp / RHK SPM100</td>
<td>45</td>
</tr>
<tr>
<td>snap-down</td>
<td>2.5</td>
</tr>
</tbody>
</table>

Table 4.2: Time constants for two different methods of driving piezo stacks in an inertial motor. The simple inverting amp scheme does not perform well. With the addition of a few simple parts, the performance is greatly enhanced.

4.3 Testing

Characterization of the motor is an important part of the process. Robustness and repeatability are key factors when considering any system that is to be used in an ultra-high vacuum system. The motor will be inaccessible to the user and any repairs would necessitate a chamber vent, wasting time and energy. A great deal of testing was performed to ensure that the motor operated as expected. There are several different approaches to testing the performance of a coarse approach motor. Some study the average translational performance using a traveling microscope [27], while others measure step-by-step performance using an interferometric setup [52, 47]. The interferometric approach has much greater time and space resolution than the traveling microscope. The microscope is simple to set up, and by knowing the number of steps taken per second, as well as the distance traveled in a set time, a reliable
estimate of step size can be found. Again, dynamics are often unimportant, as the approach speeds in SPM are often quite low.

Our measurement uses neither of these methods. A novel scheme inspired by an undergraduate experiment at McGill University (M. Orchard-Webb) is used. In some ways, it is a hybrid of the two techniques. An OPTEK OPB703 reflective object sensor to track the slider is utilized. The OPB703 consists of an infrared light emitting diode and a phototransistor mounted on converging optical axes. Any reflective object placed on axis will produce a current that is related to the intensity of reflected light. While the distance-current response is highly non-linear over the entire range, there are portions which are locally linear with steep slopes. This is taken advantage of by using only the linear portion of the response. Converting the output current to a voltage, and recording the voltage as a function of time, motor movement may be measured with a precision between that of an interferometer and a traveling microscope. The entire cost for the distance measurement circuit was less than CAD 50. It is an inexpensive, simple, and precise method to measure distances.

4.3.1 Circuit and hardware

The circuit used in the distance measurements consists of a transimpedance amplifier with a few additional parts to provide some adjustments to overall gain, leaving the option to work in an appropriate linear response range. By operating in the linear range, we can expect a distance-voltage relationship

\[ V = \alpha d + V_0, \quad (4.2) \]

\[ \text{www.optekinc.com/datasheets/OPB703-705_70A-70D-B.PDF} \]
where $\alpha$ is a constant related to sensor performance, reflectivity, and gain, $V$ is the voltage from the sensor, $d$ is the distance, and $V_0$ is some offset that is not relevant to the measurement.

The circuit and sensor is mounted to two linear micrometer stages, allowing for precise adjustment of the sensor relative to the motor. Being able to adjust the average spacing between sensor and reflective object is important for keeping the current-distance relationship in a linear range. A piece of reflective tape is added to the top of the slider to increase its reflectivity. In Fig. 4.9 the (1) micrometer stages, (2) distance sensor circuit board, (3) OPB703 and (4) Zmotor are noted. The entire device is placed in an opaque Faraday cage during operation to reduce pick up from external lights and other sources.
Figure 4.9: An illustration of the distance sensor and the distance sensor circuit. The distance between the sensor and the slider $d$ changes as the motor is made to walk. The sensor produces a current that is proportional to $d$, and the circuit converts this to a useable voltage. The circuit consists of three major portions: (1) a transimpedance amplifier $V_1 = -i_c(R_{1a} + R_{1b})$, (2) an offset and gain stage $V_2 = (R_{2f}/R_{2a})V_1 - (R_{2f}/R_{2b})V_{cc}$, (3) a unity-gain inverting amplifier, and a low pass filter $f_{3dB} = (2\pi R_f C_f)^{-1}$.

4.3.2 Distance measurements

Performance of the motor was tested as a function of the magnitude and frequency of the voltage applied to the stacks. All of these measurements were performed using LabVIEW 7.1 and a National Instruments DAQ6229 data acquisition card. The parameter $\alpha$ is determined for the system any time a change has been made. To measure $\alpha$, the fact the sensor is mounted to micrometer stages is used. The stage is moved in 10 $\mu$m steps and 100 voltage readings are taken and averaged at each distance. The data is then fit to the linear model in Eqn. 4.2, as seen in Fig. 4.10. A value for the change in voltage per change in distance, $\alpha$, is then extracted. The
distance sensor is then appropriately calibrated, and the conversion factor $\alpha$ is used for the remainder of measurements.

![Figure 4.10: A calibration curve of the distance sensor. The slope of this curve determines the conversion factor $\alpha$. Here $\alpha = 28.4 \pm 0.3 \text{mV(\mu m)^{-1}}$.](image)

When using the sensor to measure motor performance, the sensor is not moved. Changes in the sensor output are created as the slider moves, and the data is recorded as a series of voltage-time curves. A unique curve is created for each driving voltage $V$ and driving frequency $f$. Using $\alpha$ to convert each curve to a distance-time curve, the slider position as a function of time is then

$$x_{Vf}(t) = \alpha^{-1}(mt + V_0). \quad (4.3)$$

By taking the slope from Eqn. 4.3 and dividing by the driving frequency, an
expression for the step size \( s \) as a function of driving frequency \( f \) at each driving voltage \( U \) is obtained:

\[
sv(f) = \frac{m}{\alpha f}.
\]  \hspace{1cm} (4.4)

This is illustrated in Fig. 4.11. It is interesting to notice the difference in step size when moving against gravity (up) and when moving with gravity (down). Also note that the step size appears to be quite constant for wide range of frequencies. This is taken as a good measure of the robustness of the motor. Models presented earlier are taken as frequency independent phenomena when the motor operates below resonance modes of its structure. Outlier points are taken to result from various environmental effects; the experimental apparatus being disturbed mechanically, for instance. However, two changes related to the frequency do occur.

The slight decrease in step size as the frequency is increased is simplest to explain. This is related not to the motor itself, but to the control electronics used to produce the driving voltages. As \( f \) increases the op-amp behavior changes, and the peak driving voltage \( U \) decreases. The obvious result of a lowered peak driving voltage is a smaller step size. In true operation this is not a concern, as the motor will only operate at a single driving frequency.

More complex is the slightly oscillatory nature of the step size at \( f > 2.2 \text{kHz} \). One may arrive at the conclusion that the high frequency is beginning to excite a resonance in the motor, and this is altering the behavior. This explanation is possible, but with our current measurement device, it is not simple to verify. Another possible explanation is related to the number of points used to determine the step size at each frequency. As the velocity of the slider increases, the number of points in the sensor voltage/time plot decreases, and then each fit suffers in precision. In the future the
motor could be tested to much higher frequencies, using a sensor with greater time resolution.

![Graph showing step size vs. frequency](image)

**Figure 4.11:** Two step size curves. The red curve (lower) is for the slider moving up (against gravity) and the back curve is moving down (assisted by gravity). Notice the difference in average step sizes.

A series of curves to explore a large portion of frequency, applied voltage, step size phase space can be produced. A single trial produces a single point on a (frequency, applied voltage, step size) plot. Allowing the following workflow to be handled by a computer generates an ideal situation where the motor can be left for several days to automatically explore all of the available phase space. The basic backbone of the LabVIEW program is:

1. The initial are set as applied voltage $U = 20\text{ V}$ and initial frequency $f = 1\text{ Hz}$, with the initial sensor voltage $V = -3\text{ V}$.

2. Generate a sawtooth waveform with a specific peak voltage $U$ and frequency
3. Record the sensor voltage $V$ as a function of time. Allow the slider to reach a threshold sensor voltage $V_{up}$.

4. Stop the slider. Fit a line to the voltage-time data to extract step size $s_{Uf}$ at a specific $U$ and $f$. Save the $(s_{Uf}, U, f)$ triplet in a text file for upwards data.

5. Now generate the same waveform applied in reversed polarity to create downward motion.

6. Record the sensor voltage $V$ as a function of time. Allow the slider to reach a threshold sensor voltage $V_{down}$.

7. Stop the slider, record the new triplet $(s_{Uf}, U, f)$ in a downwards data text file.

8. Increment $f$ by some small amount $\Delta f = 10$ Hz and repeat steps (2) - (7) until $f = 3$ KHz.

9. Increment $U$ by some small amount $\Delta U = 3V$ until $U = 80V$. Reset $f = 1$ Hz, repeat steps (2) - (7).
**Figure 4.12:** Two (applied voltage, frequency, step size) phase-space plots of the Zmotor. Of note is the flat performance as a function of frequency, and the steady rise in step size as a function of applied voltage. One interesting portion is the low $U$, high $f$ region, where the step size drops to zero. The motor could never be made to walk in these regions.

The plot in Fig. 4.12 represents almost the entirety of what is helpful to know about the motor. It is evident that the step size increases linearly with applied voltage. Greater extension from the stacks produces a greater step size, as expected. Again, it is clear that very little change in step size results from a change in frequency. Additionally, all of this data was generated from one continuous set of measurements. No adjustments to the motor itself were made during the data collection. This is a solid indication of the long-term stability of the motor. Generating this set of data requires almost 10000 trips from $V_{\text{down}}$ to $V_{\text{up}}$, resulting in a total travel distance of nearly 3 m, one 50 nm step at a time.
A repeated set of measurements at a single $U$ and $f$ was also performed. Two thousand trials were performed in both the up and down direction. Step sizes at a set frequency and voltage are seen to be distributed normally in Fig. 4.13, with a small standard deviation. What does small standard deviation mean in this case? For the upslope $s_u = (74 \pm 2) \text{ nm}$, and for the downslope $s_d = (-97 \pm 4) \text{ nm}$. The absolute accuracy of the step sizes is limited by the calibration step. We will also see that compared to the overall extension of the piezo tube used in unison with the motor for coarse approach, the uncertainty is small enough to not interfere with an effective approach.

The Zmotor fulfills its original design goals. It has been shown to reliably take steps that are $(10 - 160) \text{ nm}$, depending on applied voltage. When reviewing scanning tunneling microscope results in a later chapter, it will be seen that these steps sizes are small enough to approach to within tunneling range. By limiting the two translational degrees of freedom and all rotational degrees of freedom, motion along a single axis
is guaranteed. While the motor has not yet been operated at low temperature, the
motor has been thoroughly tested at room temperature. Its repeatable and reliable
performance at room temperature gives us hope that it will perform similarly when
cooled.
Chapter 5

Design and Testing of the STM

A coarse approach motor is only one component of a scanning tunneling microscope. There must also be facilities to scan the surface, hold a sample and isolate the microscope from external vibrations. The design of each these components will now be described.

Performance testing done with the new isothermal microscope, including vibrational analysis, will be outlined. While the new microscope head will operate in a liquid-Helium cooled UHV system, only initial images taken in ambient conditions are presented.

5.1 Operation

In order to place elements of the design into context, the operation of this STM is outlined. Particulars regarding the operation of the system in vacuum will not be discussed, as they do not have a direct bearing on the operation of the microscope.
Figure 5.1: A schematic diagram of the components used for STM scanning in air, and a resulting image. The identified elements are: (1) eddy-current vibration isolation, (2) vertical approach microscope, (3) vibration isolation table, (4) amplified tunneling current, (5) bias voltage, (6) piezo control signals, (7) computer and electronics for STM feedback, (8) STM image.

With a sample in the sample holder, a tip installed in the tip holder, and a bias voltage applied to the sample, the approach process can begin. This is an algorithmic procedure handled under software control:

1. The piezoelectric scan tube is extended towards the surface. The tube can extend approximately 400 nm. The feedback loop waits to detect a tunneling current.

2. If a tunneling current is detected, the tip is in tunneling range, and the approach process is complete. If no current is detected, the approach is not complete, the tube is retracted, and the loop continues.

3. A step is then taken towards the sample by the vertical approach motor. This step is performed at a relatively low voltage, so that the step size does not exceed
the overall extension of the tube. The process is repeated until a tunneling current is detected.

With a stable tunneling signal, STM experiments may now be performed. The control electronics raster the tip over the surface by controlling the voltage applied to the PZT tube. If constant-current mode is used, the tip height is controlled to produce a constant tunneling current. The voltages applied to the tube are converted to a distance, and a series of line profiles are used to produce a single image.

## 5.2 Design

The design found in this thesis draws inspiration from other successful low-temperature, ultra-high vacuum scanning tunneling microscope designs [61, 2]. Considerably attention was given to the creation of a simple microscope design that cools efficiently, and is mechanically very rigid.

![Figure 5.2: The STM fully assembled showing: (1) sample holder, (2) sample plate, (3) tip holder, (4) PZT scan tube, (5) vertical approach motor, and (6) SmCo magnet.](image)

The most important constraint placed on the microscope is on the size; it must fit
within the copper radiation shield. The cooling system was designed by J. Visser [62], a former student in the lab. The design is similar to other isothermal microscopes [37, 56, 40].

The microscope design can be divided into key components: the chassis, sample holder, piezoelectric tube scanner, and the tip holder. The following section will outline the design and implementation of each of these parts.

5.2.1 Chassis

The chassis acts as a base for all other parts of the microscope. It consists of a single piece of machined OFHC copper, chosen for its high thermal conductivity. Care was taken to keep the chassis as simple as possible. Fig. 5.3 indicates the key features of the design.

The back wall of the chassis is almost completely solid, and does not feature any protruding parts. Two counter-sunk through holes on the back wall are used to mount the vertical motor. The microscope will be cooled by placing the back of the body in direct contact with a surface that is being held at liquid-Helium temperatures. By creating a small thermal mass body with a large surface area to contact a cooling surface, rapid, efficient cooling is ensured.
Figure 5.3: An image of the chassis, shown in a perspective view, with the main dimensions indicated. Other parts include: (1) area to mount the sample holder, (2) holes to mount the coarse motor, (3) an area to route wiring, and (4) inset areas for mounting SmCo magnets.

Along the bottom of the chassis are four circular holes, which are used to hold Samarium Cobalt (SmCo) magnets\(^1\). These are used in the vibration isolation system, providing eddy-current damping. The eddy-current damping will be described more fully in a later section.

As mentioned earlier, the cooling system constrains the size of the microscope. The chassis’ dimensions are illustrated in Fig. 5.3. While the cans constrain the width and depth, the height is defined by the magnets on the bottom, as well as the coarse approach motor, with the scanning piezo tube now attached to the top of the slider.

Finally, the chassis is made to be as open as possible. This allows for easy access for tip transfers, sample transfers, as well as dosing the sample with an adsorbate while it is in the sample holder. Additionally, visual access to the tip-sample gap is made much simpler.

\(^1\)1/2" SmCo disc #5768K22 – http://www.mcmastercarr.com
5.2.2 Sample plate

The external dimensions of the sample plate are identical to a commercial sample holder \(^2\). This affords the possibility of integrating this microscope into a commercial ultra-high vacuum system in the future. Key features of the sample plate are illustrated in Fig. 5.4. It consists of a flat plate made of a refractory metal, such as Molybdenum, that allows a range of samples to be mounted in a number of ways. The material choice is important as the sample plate is not only cooled while mounted in the microscope, but it can also be heated to over 1300 K during sample preparation. A refractory metal will not produce any sample contamination during heating, and it is also a good thermal conductor.

![Sample plate diagram](image)

**Figure 5.4:** The sample plate used in this microscope. The sample plate may be modified in many ways to accept samples of different sizes or preparation techniques.

A key tab protrudes from the front of the plate. This couples with a sample transfer tool, and allows the sample to be mounted/dismounted from the sample plate and carried within the vacuum chamber.

\(^2\)SPECS GmbH Berlin – [http://www.specs.de](http://www.specs.de)
5.2.3 Sample holder

The sample holder is a receptacle for the sample plate. When operating in a UHV system, sample transfer is a major concern. Direct access to the sample is not possible, so a robust sample manipulation system is highly desirable, and totally necessary. Fig. 5.5 illustrates the new sample holder, also made of OFHC copper.

![Sample holder](image)

**Figure 5.5:** The dimensions of the sample holder are indicated in (a). Note the beveled front edge of the sample holder. An exploded view of the sample holder (b) with extra parts required to electrically isolate the sample holder from the chassis. The labelled parts are: (1) a 0-80 bolt, (2) a stainless steel washer, (3) a ceramic washer, (4) a ceramic tube, (5) sapphire windows and (6) a 4-40 bolt.

The front edge of the holder is beveled to allow the sample plate to be pushed into position while sliding underneath springs designed to hold the plate in place. The sample plate is then held firmly on the sample holder, providing a strong thermal link between the sample and sample holder.

As discussed earlier, when performing STM, a bias voltage must be applied between the sample and tip. The sample holder and chassis must be electrically isolated from one another. At the same time, for cooling purposes, it is desirable to keep
the sample holder and chassis in thermal contact. Fig. 5.5(b) illustrates how this is accomplished in the design. Two sapphire windows\(^3\) are inserted at the sample holder/chassis interface. Sapphire is useful as it is both an excellent electrical insulator and an acceptable thermal conductor. Stock sizes of sapphire windows are used, which simplify the assembly process. Additionally, judicious use of ceramic sleeves and washers provide electrical isolation for each of the bolted connections.

Finally, three 4-40 bolts are mounted upside down on the sample holder. These are used as mounting points for the springs that the microscope will be suspended from.

### 5.2.4 Piezoelectric tube scanner

Creating a STM image requires very precise control over the location of the tip. Again, PZT ceramics are used to control the tip when actually scanning the surface. After the coarse approach motor has brought the tip close enough to the sample to establish a tunneling current, the piezoelectric tube scanner takes over control of the microscope. The piezo electric scan tube is attached to the top of the vertical motor slider.

The tube used here is sectioned into four separate electrodes on the outside, while the inside of the tube is a single electrode. Applying a voltage to the tube causes it to expand or contract. When a positive (negative) voltage is applied to an external electrode, only the quartered section will expand (contract), creating a torque, and causing the tube to bend. A signal applied to the inner electrode will cause the entire tube to expand or contract. Thus, the piezo electric tube scanner can be manipulated

\(^3\)Swiss Jewel SP103 – http://swissjewel.com/sapphire_rectangle_windows_stock_sizes.htm
in three dimensions.

\[ L = 12.7 \text{ mm} \]
\[ D = 3.8 \text{ mm} \]
\[ h = 0.64 \text{ mm} \]

**Figure 5.6:** In (a) dimensions of the piezo electric scanner tube are indicated on a drawing of the tube (D is the inner diameter, L in the length). Part (b) illustrates the signals applied to the piezoelectric scan tube.

A typical wiring arrangement is illustrated in Fig. 5.6(b). Quadrants which are \(180^\circ\) apart have the same signal applied with opposite polarity, controlling movement in the \(x\) and \(y\) directions. Another signal, applied to the inner electrode, acts as a \(z\) offset to the signal.

As in Sec. 3.1, the response of the tube with respect to the applied voltage can be described as a function of the piezo polarization and geometry. A voltage \(V_x\) applied to the \(x\) electrodes produces translation

\[ \Delta x = \left[ \frac{2\sqrt{2}d_{31}L^2}{\pi Dh} \right] V_x, \quad (5.1) \]

where \(d_{31}\) is the appropriate coefficient of the strain tensor, \(L\) is the tube length, \(D\) is the inner diameter of the tube, and \(h\) is the tube thickness. Any extension in the \(y\) direction is calculated in the exact same manner. Calculating the \(z\) extension is
simpler because of the geometry,

\[ \Delta z = \left[ \frac{d_{31} L}{h} \right] V_z. \]  

(5.2)

Here, \( d_{31} \) is once again used, as the orientation of the electric field and polarization remains the same. It is merely the geometry of the electrode that has changed.

It is useful to consider only the portions of Eqns. 5.1 and 5.2, that are in the square brackets. These quantities are an expansion coefficient for a specific piezo tube, called \( K_{xy} \) and \( K_z \), respectively. Given the dimensions shown in Fig. 5.6, as well \( d_{31} \) for an EBL#2 tube at 293 K from Table 3.1, suggests that \( K_{xy} = 104 \, \text{ÅV}^{-1} \), and \( K_z = 35 \, \text{ÅV}^{-1} \).

### 5.2.5 Tip holder

The tip is an integral part of a scanning tunneling microscope; a well-designed tip holder can make a huge difference in the day-to-day operation of the microscope. The intention was to design the microscope with an in-situ (inside of the vacuum chamber) exchangeable tip holder. There are many different exchangeable tip holder designs in the literature [56, 63, 39]. The design used here was based on the work of A.J. Weymouth, a PhD student in the group. Some additional alterations have been made to the design by the author, to enable use with the current microscope, and improve performance.
Figure 5.7: The tip holder is made up of: (1) a W tip, (2) magnetic stainless steel tip holder, (3) non-magnetic cup, (4) SmCo magnet, (5) grounded tip-shielding skirt, and (6) a ceramic washer used as electrical insulation.

The tip holder is illustrated in Fig 5.7. Of importance are the holder, cup and skirt. The holder is made of magnetic stainless steel, and the cup is made of normal stainless steel. In an effort to allow the holder to self-align, the cup is made non-magnetic. A SmCo magnet is glued to the underside of the cup. This provides the holding force to keep the holder in place. Various ceramics washers are used to electrically isolate the parts. The grounded skirt is used in a effort to shield the tip from electromagnetic interference from the voltages applied to the piezo tube.

Non-conductive epoxy is used at each step to connect the components of the tip holder. It is then glued to the top of the piezo tube.

5.2.6 Tips

Reliable tip preparation is important for atomic-resolved STM images [64]. In this microscope, most of the tips are made by electrochemically etching polycrystalline Tungsten wire. This produces high-quality tips, with a small radius. An exemplary tip
is demonstrated in a SEM\textsuperscript{4} image in Fig. 5.8(b). These tips are produced with nearly 75% efficiency, and quality can typically be confirmed with an optical microscope.

![SEM images of a single electrochemically etched Tungsten tip.](image)

**Figure 5.8:** SEM images of a single electrochemically etched Tungsten tip. In (a) a 18400x magnification image can be seen, with an estimated tip radius $\approx 40$nm. The radius was estimated by enlarging the image and drawing the white lines visible in the inset to part (a). The scale bar on the bottom of the image was used to determine a pixel-distance conversion. In (b) a zoomed-out view of the same tip is presented. The tip is a good example of tips created through electrochemical etching. SEM images were obtained with the FEI Phenom system.

## 5.3 Vibrations

Effective vibration isolation is essential for STM. A multi-stage vibration isolation approach is used to decouple the tip-sample junction from external disturbances [41]. Mechanical vibrations can affect the microscope in a variety of ways, at a range of frequencies [65]. The intention is to create a system with a series of unmatched transfer functions, creating a vibration filter with a large bandwidth.

The first stage of vibration isolation is provided by the microscope itself. Vibrational eigenfrequencies of the tip-sample junction are kept as high as possible by using a very rigid structure for the microscope. The chassis and sample holder are inherently stiff, making their vibrational characteristics irrelevant when looking at the lowest resonant frequency. The vertical approach motor, with a scan tube mounted on top, is the source of the lowest resonance frequencies. To determine the vibrational characteristics of the microscope, the self-excitation method was used [1]. This consists of exciting the one quadrant of the piezoelectric scanner with a sinusoidal signal $V_e = 4 \, V_{pp}$ at some known frequency. The pickup on a quadrant directly opposite is recorded with a lock-in amplifier\textsuperscript{5}, using the $V_e$ as a reference signal. From this the lowest resonance mode of the structure can be roughly determined. In Fig. 5.9 we see that the lowest mode is around $f_{res} = 1.6 \, \text{kHz}$.

\textsuperscript{5}Stanford research systems SR830 – http://www.thinksrs.com/products/SR810830.htm
Figure 5.9: Vibrational spectrum of the piezo scan tube when glued to the vertical coarse approach motor. The green curve is with the entire tip assembly placed on the scan tube, the red curve is with the tip holder removed. The extra mass of the tip holder moves the resonance frequency.

The vibrational spectra reveal that the microscope appears to be insensitive to low-frequency disturbances. The lowest vibrational mode will also limit the possible scan speed of the microscope. To scan a single line in 1 ms requires a fundamental above 1 kHz. Fig. 5.9 also suggests that a less-massive tip holder (item (2) in Fig. 5.7) should produce a system with a higher resonance frequency. This may be pursued as the project matures.

An eddy-current damped spring suspension provides the next stage of vibration isolation. The spring system is made to have a very low eigenfrequencies. Using a spring system with low resonance frequencies works well, as it does not transmit high frequency disturbances effectively, while the rigid microscope is very insensitive to
low frequency disturbances. The resonance frequency $f$ of a spring mass system is determined by

$$f = \frac{1}{2\pi} \sqrt{\frac{k}{m}},$$

(5.3)

where $k$ is the spring constant of the suspension springs, $m$ is the mass supported by the springs. By using custom-made springs, the resonance frequency of the microscope can be made $< 5$ Hz.

Figure 5.10: Illustration of an eddy-current damper. Indicated are (1) the custom coil extension springs, (2) SmCo magnet, and (3) the exterior copper wall of the cooling can. As the microscope oscillates at velocity $v$, the force, $F_{ec}$, which results from the induced eddy-currents counteracts the motion.

Custom coil extension springs were created through a simple process. A 6.35 mm diameter stainless steel rod was used as a mandril for the spring forming. Two holes drilled through the rod, placed 40 mm apart, were used to secure spring wire. The mandril was then placed on a lathe. For initial tests, music wire with diameter 0.254 mm was used as the spring wire. One end of the wire was attached to the rod, by clamping it between two nuts. Wire was then wrapped along the rod, simply by turning the lathe manually, while maintaining tension in the wire. Each wrap was
made as close as possible to the previous wrap. This results in a spring with a pitch determined by the wire diameter. The spring constant

$$k = \frac{Gd^4}{8nD^3}, \quad (5.4)$$

where $G$ is the shear modulus of the material, $d$ is the wire diameter, $n$ is the number of coils that are extending, and $D$ is the mean diameter of the coil. For the music wire used to create the springs used in this thesis, the shear modulus $G \approx 75 \text{ GPa}$. From Eqn. 5.4, using $d = 0.254 \text{ mm}$, $D = 7.62 \text{ mm}$, and $n = 12$ active coils results in $k = 7.4 \text{ Nm}^{-1}$. Using Eqn. 5.3 for a microscope of mass $m = 0.180 \text{ kg}$, hung by three individual springs (mass $m/3 = 0.060 \text{ kg}$), a resonance frequency of $f = 1.8 \text{ Hz}$ is obtained.

The spring constant for the custom spring was determined by measuring the extension of the spring as a function of mass. These measurements resulted in a spring constant of $k = 10.0 \pm 0.5 \text{ Nm}^{-1}$. From this, $f \approx 2 \text{ Hz}$.

By attaching magnets to the chassis, as described in Sec. 5.2.1, any oscillations induced in the microscope can be damped out to further the vibration isolation. Eddy-current damping results from the current induced in a conductor when a magnetic field is moved relative to it. As the force is proportional to the velocity of the magnet, large amplitude oscillations can be damped out quickly. The force induced $F_{ec}$ is directed opposite to the velocity $v$, and is given by

$$F_{ec} = -C_{ec}v = C_o \left[ \frac{\pi a^2 B^2}{\rho} \right] v, \quad (5.5)$$

where $\rho$ is the resistivity of the metal, $C_{ec}$ is the damping coefficient [in Nsm$^{-1}$], $C_o$
is a geometric factor taken as \( \approx 0.5 \) for a conductor much larger than the magnet (as it is here). Additionally, \( a, t \) and \( B \) are the radius, thickness and field strength of the magnet, respectively [65]. Using strong SmCo magnets in close proximity to a copper surface creates excellent damping. Eddy-current damping is a very effective method of vibrational isolation, and does not suffer any ill effects at low-temperatures.

The final stage of vibration isolation consists of a passive vibration isolation air table\(^6\). These tables are very effective at isolating the entire system from vibrations. The manufacturer places the isolation efficiency at 10 Hz or above at 90 - 97%.

The combination of very rigid microscope, a spring system with low eigenfrequency, and an air table with broadband isolation make for an effective vibration isolation system. Each component is temperature-independent, and expected to perform equally well when cooled.

### 5.4 Initial results

Early results from the microscope are now presented. Initial tests were performed in air. Performing STM in ambient conditions is often quite challenging, as surface and tip cleanliness cannot be controlled well. Ambient scans were performed with the microscope suspended by an eddy-current damped spring suspension system, and placed on a vibration isolation table (see Fig. 5.11). The RHK SPM100 controller with a IVP200 current preamplifier was used to control the STM and acquire scan data.

---

Figure 5.11: Photographs of the ambient STM setup. In (a) the (1) acoustic isolation, (2) air table and (3) electrical input are indicated. In (b) the (4) springs, (5) microscope, and (6) the eddy-current damping are indicated.

5.4.1 HOPG

Highly ordered pyrolytic graphite is a commonly used substrate in SPM studies, and for initial calibration of a new instrument. It is easily cleaved with scotch tape, leaving behind a chemically stable sample, with large atomically-flat domains. Atomic resolution in ambient conditions can typically be achieved. No atomic resolution scans will be presented in this thesis. For this reason, scale bars on the images are only approximate. An approximation to the scan size was made by choosing the maximal scan range equal to what was determined by Eqn. 5.1. This should place the estimated scan sizes within at most a factor of 2 of the true image size. To accurately obtain a measure of the size of an image in STM, a calibration based on atomic spacing is
required.

**Figure 5.12:** A series of ambient STM images of a freshly-cleaved HOPG surface using a mechanically formed Platinum-Iridium tip. Imaging conditions in each image are $+1.3\,\text{V}$ bias (sample to tip), $1\,\text{nA}$ tunneling current. The highlighted box in (a) makes up the entirety of (b), and the highlighted box in (b) makes up the image in (c).

While no atomic resolution images are presented, some information may be still be extracted from the STM presented below. In Fig. 5.12, a series of images of the same area on a freshly-cleaved HOPG sample are presented. Here, the ability of the microscope to selectively scan an area of the sample is demonstrated. From (a) to (b) to (c) not only is the areal scan size reduced by 25 times, but a specific location on the surface is imaged. Fig. 5.12 (b) is clearly identifiable in (a), by both the appearance of the steps, as well as an interesting feature in the bottom left corner of (b). The scan in Fig. 5.12 (c) consists of two very flat areas, with a single atomic step running across the image. The image appears quite flat, but there is no evidence of atomic corrugations. This is likely due to tip cleanliness, which is much improved when working in UHV.
During ambient scans of HOPG, a moiré supermesh was observed [27]. An extensive review on the subject of superlattices on graphite has been published by Pong and Durkan [4]. Supermeshes result from shifted single layers of graphite, which produce periodic structures that are not atomic corrugation. Fig. 5.13 demonstrates a moiré pattern induced by adding a small amount of organic solvent (CHCl₃, chloroform 99.9% purity) to the surface, and allowing it to dry. The features in Fig. 5.13 are very similar to previously published results. The main interest of observing such effects with the STM lies with their purely electronic nature. While the graphite may remain atomically flat, additional corrugations appear due to the increased (decreased) density of states where the carbon atoms in subsequent layers lie near (far) from one another.
5.4.2 Au on Mica

Gold(111) surfaces are another commonly used surface when scanning in ambient conditions, due to the noble-metal nature of Au. A flame-annealing procedure can result in large \((1 \mu m \times 1 \mu m)\) atomically-flat domains [66].

![Figure 5.14](image)

**Figure 5.14:** A series of ambient STM images of a flame-annealed Au on mica sample. Imaging conditions in each image are +0.3 V bias (sample to tip), 1 nA tunneling current. Image (a) features a Au on mica sample, with some flat terraces, though it is clearly quite dirty. The highlighted box in (a) is the area imaged in (b). In (b) and (c) the same area is imaged 737 s apart. Nine images were recorded in the series of images. This was used to obtain some idea of the inherent thermal stability of the design. A drift rate of 0.6 nm min\(^{-1}\) is observed here.

In Fig. 5.14 (a) a large scale image of a typical Au/mica surface is presented. The white rectangle denotes the area imaged to form Fig. 5.14 (b) and (c). Again, the ability of the microscope to image in a controlled fashion is demonstrated. The images in (b) and (c) were used to obtain some idea of the drift rate of the microscope. Starting with Fig. 5.14 (b) the microscope was set to obtain a image of the same area every minute. Fig. 5.14 (c) is an image of the same area 737 seconds after the first
image was recorded. The displacement of a single feature on the surface was measured
to obtain a drift rate of 0.6 nm min$^{-1}$. 
Chapter 6

Conclusion

The intention of this project was to design, build and test a scanning tunneling microscope with a vertical coarse approach that could be used at cryogenic temperature and in ultra-high vacuum. A simple and reliable coarse approach has been designed, built, and tested. The microscope has been shown to perform exceptionally well under ambient conditions. Using the simple model introduced in Chapter 3, to drive design decisions, the basic requirements of a piezoelectric vertical coarse approach motor have been described. These guidelines can be applied to many other positioning devices, and will be used in this lab for future instrument design.

Piezoelectric motors that utilize a vertical approach can be very challenging to design and build. In principle, these devices appear to be very straightforward. However, in practice, many factors influence their performance, and these factors are not fully documented or well understood. Heed should be taken when reviewing the literature on the subject. Overly simplistic descriptions of these motors are commonplace, and many prototypes never evolve into working instruments.

Positioning performance of the vertical motor was excellent. It not only allowed
tips to approach the surface safely, without crashing, but individual steps could be
taken once in range, to allow for optimization. The microscope performed satisfac-
torily and it is fully expected, with more time, to produce images with atomic
resolution. The dependence on tip structure can lead to some difficulties in obtaining
atomic resolution with only a few attempts.

A solid foundation has been laid out for exciting new experiments with a liquid-
helium cooled isothermal scanning tunneling microscope. The head of the microscope
was the focus of this thesis. Complete operation of the instrument relies on the
vacuum system and the cryostat, which are described briefly in the remainder of this
chapter.

6.1 UHV Chamber

The custom UHV chamber was designed by J. Visser and constructed by Hunting-
ton\textsuperscript{1} [62]. It consists of two separate chambers: an STM chamber that includes the
microscope and cold tip of the cryostat, and another chamber that is to be used for
sample and tip preparation. Sample preparation hardware has been installed and
tested. It has been pumped to UHV conditions, and is ready to be used. Figure 6.1
provides two views of the UHV system. The first is a photograph shows the system
as it is now. The second image has been rendered using a 3D modeling program
(Autodesk Inventor) and some parts are labelled.

\textsuperscript{1}Huntington Mechanical Laboratories – http://www.huntvac.com/
Figure 6.1: The ultra-high vacuum chamber to be used with the isothermal STM. The labelled parts are: (1) continuous-flow cryostat, (2) STM chamber, (3) ion gun, (4) sample preparation chamber, (5) replaceable evaporator ports, (6) ion pump, and (7) vibration isolation table.

### 6.2 Cooling cans

The method of cooling the STM was another focus of J. Visser’s MSc thesis [62]. A simple, effective, and proven method of cooling has been chosen for this system [37, 56]. A copper can is attached to a continuous flow cryostat, and the can is cooled directly by the cold finger of the cryostat. The copper radiation shield can be kept at the base temperature of the cryogen with ease. The STM is suspended by springs in the can, and may be rapidly cooled by forcing it into contact with a wall of the copper can. Another can, which acts as a radiation shield, is placed around the interior can. The exterior can is cooled by the exhaust gas from the cryostat. An image of the
cans, that once again was rendered with a 3D modelling program, can be found in Fig. 6.2. Releasing the STM head from the wall of the inner can, it will maintain a low temperature, while not being attached directly to the cryostat. This minimizes the possibility of vibrations propagating to the microscope.

![Figure 6.2](image_url)

**Figure 6.2:** The copper radiation shields (cooling cans) used to cool the STM. In (a) the (1) continuous flow cryostat cooling finger, (2) the outer radiation shield, (3) inner radiation shield, and (4) the STM are labelled. Part (b) is a view of the cooling cans when they are situated in the vacuum chamber (part of the chamber wall has been cut away in this view).

### 6.3 Outlook

The microscope, and all subsystems required for low-temperature UHV operation are very nearly complete. This new set of tools will allow for a new, exciting set of experiments to be performed.

For example, this apparatus will allow atomic-resolved images to be collected from surfaces in the temperature range extending from room temperature down to
approximately 8 K. In surface physics, temperature is one of the most important physical parameters. The ability to change temperature allows different structural and electronic phases of matter to be explored. Surface processes, like diffusion and chemisorption, are also critically dependent upon temperature and being able to set the temperature will provide the capability to tune surface processes and measure activation energies associated with these processes. Additionally, the sharpening of the Fermi-Dirac function at low temperature will allow scanning tunneling spectroscopy to be performed with high energy resolution and with low microscope drift. All of these experimental possibilities become accessible with the microscope described in this thesis.
Bibliography


