Mean Value Method of Lateral Force Calibration

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Abstract

Lateral force microscopy is the primary means for the study of nanotribology: a rapidly growing field of research. A new method for lateral force calibration was derived and investigated. The technique utilizes mathematics of mean values and directionality, greatly simplifying the process of LFM calibration. Experimental analysis produced convincing data proving the validity of the concept. This in-situ method offers potential advantages over current methods, such as reduced tip wear, limited reliance on unproven assumptions, and ease of use.
Acknowledgements

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Chapter 1

Introduction

The study of friction at the nano-scale (nanotribology) is an important subset of nanoscience. Nanotribology offers many complex challenges to overcome if it is to be fully understood; however, the field also offers the potential for substantial scientific advancement. Advancement of nanotribology is an important element of nanoscience and atomic force microscopy is the primary means of study.

Lateral force microscopy (LFM) is an essential tool in the study of friction phenomenon at the nanometer scale, a fundamental area of research in the field of nanoscience. For valuable data to be acquired the cantilevers lateral stiffness must first be calibrated. Several methods exist to perform lateral force calibration; however, they all suffer from a few significant shortcomings. Currently accepted LFM calibration methods suffer from excessive tip wear, the need for special equipment, use potentially fallacious assumptions with regard to friction, and are very complicated. These traits make calibration very difficult perform and prohibit widespread adoption.

Inspired by these shortcomings we set out to derive an accurate in-situ method with emphasis on limited tip wear and a widely adoptable methodology. Using mean values and directionality we were able to cancel out many of the unknown variables in our force balance equations. This allowed us to derive a simple mean-value method of calibration that overcomes the challenges faced by currently accepted methods.
Chapter 2

Background

2.1 Atomic Force Microscopy

2.1.1 Workings of an Atomic Force Microscope

Atomic force microscopes (AFM) are important tools in nano-scale research. Initially AFM was used primarily for analyzing nano-scale topography; however, it now has the ability to perform a wide variety of analysis. Unlike a traditional microscope the AFM does not use optics to view objects, but instead measures forces through the use of a cantilever. The cantilever is very small and flexible "finger" with a very sharp tip protruding perpendicularly at the end. Generally made of a material such as monolithic silicon nitride, cantilevers come in both rectangular and triangular shapes. The top surface of an AFM cantilever is highly reflective often coated with aluminum or gold. A laser is reflected off the surface of the cantilever and then picked up by a four way photodiode. The diode detects the position of the laser and thus the bending of the cantilever can be interpolated from the diodes response. Figure 2.1 illustrates a cantilever scanning a sample and the effect on the lasers position on the photo diode. The basic AFM mode works just fine when determining simple topography; however, determining actual forces involved with tip sample interactions requires calibration.

The AFM can be used in many other ways as well. Besides the standard AFM mode the device has many other modes to measure friction forces, magnetic forces, adhesion
forces and many other nano-scale interactions. The study of nanotribology has greatly increased the interest in lateral force microscopy in particular. The study of friction at the nano-scale is becoming increasingly important and lateral force microscopy is the primary means to pursue such analysis.

2.2 Current State of Lateral Force Calibration

2.2.1 1996 Ogletree et al. Wedge Calibration Technique

The 1996 wedge calibration technique put forward by Ogletree et al. [1] has long been regarded as the standard for lateral force calibration. Many subsequent researchers have modified the method; however, the initial methodology remains primarily intact. The technique is based on comparing lateral force signals on surfaces with different slopes. When scanning a slope of known angle the geometric contribution of the lateral force can be determined in relation to the normal force. To accomplish this experimentally the researchers employed a sample of \(SrTiO_3\) shown in figure 2.2, which forms a series of ‘wedges’ on its surface with two known and consistent slopes. Since the slope is known the ratio between the normal and lateral force constants can be determined.
This method has been shown to produce accurate results; however, the technique has several major drawbacks. The commercial unavailability of the \( \text{SrTiO}_3 \) sample makes widespread adoption of the wedge calibration method infeasible. Additionally, many scans are required for accurate calibration. As a result the cantilever is subject to excessive tip wear which can have negative effects when acquiring the desired data.

\subsection*{2.2.2 2003 Varenberg et al. Improved Wedge Calibration Method}

The 2003 Varenberg et al. wedge calibration method \cite{varenberg2003} addresses many of the shortcomings of the original Ogletree et al. method \cite{ogletree1996}. Most significantly the technique uses a commercially available TGF11 silicone calibration grating, shown in figure 2.3, as opposed to the obscure \( \text{SrTiO}_3 \) sample. The silicone grating has exposed (100) and (111)
faces, forming a surface with angles of $0^\circ$ and $\pm 54.44^\circ$. The TGF11 sample allows for the use of wider tips and the flat facets serve to greatly simplify calculation. These properties make the Varenberg method far more accessible allowing for widespread adoption.

2.2.3 2006 Colloidal Torsional Lateral Force Calibration

One of the more recent developments in lateral force microscopy was put forward by Carpick et al. in 2006 [3]. Based on the 2004 torsional Sader method developed by C.P. Green et al. [4] this method makes use of colloidal sphere and a test cantilever for a zero wear lateral force calibration. The method begins by fabricating a test cantilever of the same width as the cantilever to be calibrated. Then a colloidal sphere is attached to the test cantilever and the colloid is pushed up against a freshly cleaved gallium arsenide sample, as seen in figure 2.4. This allows for a force vs. distance curve to be recorded and the lateral deflection sensitivity to be extrapolated from the slope.

![Figure 2.4: Diagram of modified colloidal cantilever pushing against a gallium arsenide sample [3].](image)

The calibration method is notable because it does not require the tip to come into contact with any surface at all; however, this is also the source of its greatest flaw. By
calibrating the test cantilever instead of the target cantilever to be used the method is susceptible to inconsistencies in individual cantilever parameters. Such inconstancies could invalidate the assumption that the test cantilever and the target cantilever will behave in the same way. Another issue with the Carpick et al. method is the complexity of its setup. It requires the fabrication of a colloidal tipped cantilever with the same width, thickness and reflective characteristics of the target cantilever. Additionally it is imperative that the laser be centered on the end of the cantilever and on the photodiode to limit coupling of the lateral and normal force signals. These drawbacks make the method inconvenient and ill suited to widespread usage.

2.3 Summary of Methods

The state of lateral force calibration has made significant progress. Despite the complex nature of the problem great strides have been made in improving and simplifying the process. However, there still exist many drawbacks to even the best current methods. Most significantly the problem of tip wear has never been successfully addressed without making major sacrifices. In addition many calibration methods make questionable assumptions about the nature of friction at the nano-scale. Ease of use is also lacking in nearly all accepted calibration methods, making it a challenge to carry out even simple experiments without spending exorbitant amounts of time calibrating equipment. If progress is to be made these issues need to be addressed. The mean-value method of lateral force calibration, if successful, may hold the key to eliminate or minimize all of these shortcomings.
Chapter 3
Methodology

As we have seen there exists a myriad of lateral force calibration techniques. However, all currently accepted methods suffer from at least one of two major shortcomings; they require the development of special apparatus or evoke excessive wear on the tip. The method published by Asay and Kim [5] attempted to solve the problem of excessive tip wear; unfortunately, many of their assumptions were physically flawed and ultimately the approach was invalid. Despite the methods failure the goal of a calibration method with limited tip wear inspired our own independent research. Driven by the shortcomings inherent in previous calibration methods we set out to develop an alternative method of lateral force calibration.

Research began at a fundamental level, with the development of an accurate free-body diagram, as seen in figure 3.1. Analysis of the free-body diagram allowed for the development of equilibrium equations.

\[
\sum F_z = 0 = (N - A) |\cos(\theta)| - (\hat{v}_x \cdot \hat{i}) f \sin(\theta) - F_{load} \tag{3.1a}
\]

\[
\sum F_x = 0 = - \left( \frac{\cos(\theta)}{|\cos(\theta)|} \right) (N - A) \sin(\theta) - (\hat{v}_x \cdot \hat{i}) f |\cos(\theta)| + F_{lat} \tag{3.1b}
\]

Variables are all as defined in figure 3.1. The equilibrium equations (3.1) present us with a problem as there are too many unknown variables. We can determine \( \theta \) by fitting a polynomial to the topography. Load force (\( F_{load} \)) can be determined by any accepted
normal force calibration technique and the set-point value. In this experiment we will employ the thermal calibration method developed by Erik Thoreson and Dr. Nancy Burnham [6]. Unfortunately, we cannot accurately determine the normal force \(N\), adhesion force \(A\), or friction force \(f\); therefore, to solve for \(F_{lat}\) we shall have to employ some simplification.

First we consider equation (3.1a). Manipulating the equation to solve for \((N - A)\), arriving at:

\[
(N - A) = \frac{F_{load} + (\hat{v}_x \cdot \hat{i}) f \sin(\theta)}{\cos(\theta)}.
\] (3.2)

Plugging this into equation (3.1b) we arrive at an equation for \(F_{lat}\).

\[
F_{lat} = \left( \frac{F_{load}}{\cos(\theta)} \right) \sin(\theta) + (\hat{v}_x \cdot \hat{i}) f \left( \frac{\sin(\theta)^2}{\cos(\theta)} \right) + (\hat{v}_x \cdot \hat{i}) f |\cos(\theta)|
\]

\[
F_{lat} = F_{load} \tan(\theta) + (\hat{v}_x \cdot \hat{i}) f \cos(\theta)(1 + \tan(\theta)^2).
\] (3.3)
Equation (3.3) shall form the basis for our calibration method. However, we are still presented with the problem of accurately determining the friction force \( f \). To overcome this problem we will make use of some emergent properties that can be gleamed from a thorough analysis of equation (3.3). Analysis of the terms, specifically the sign changes, reveals some interesting properties that will be vital to our derivation. Looking at \( F_{load} \tan(\theta) \), we observe that \( F_{load} \) is of constant sign, therefore, the sign is dependant only on \( \tan(\theta) \) which changes at \( \theta = \frac{\pi}{2} \). In other words the sign of the term is dependent on the slope of the surface being analyzed. In the other term in equation (3.3), \((\hat{v}_x \cdot \hat{i})f|\cos(\theta)|(1 + \tan(\theta)^2)\) |\), the sign is dependant only on \((\hat{v}_x \cdot \hat{i})\), which is dependant only on the direction of motion of the cantilever.

We can utilize these observations to effectively eliminate the friction force from the equation. The friction force lies in the second term of equation (3.3) which changes sign depending on the direction of the cantilever. We make use of this fact by finding the mean value of the lateral force \( (F_{lat}) \) scanning a surface in each direction. By taking the mean we can effectively eliminate the second term from our equation resulting in.

\[
F_{lat-L} = F_{load} \tan(\theta) + (\hat{v}_x \cdot \hat{i})f \cos(\theta)(1 + \tan(\theta)^2) \tag{3.4a}
\]

\[
F_{lat-R} = F_{load} \tan(\theta) - (\hat{v}_x \cdot \hat{i})f \cos(\theta)(1 + \tan(\theta)^2) \tag{3.4b}
\]

\[
F_{lat-avg} = \left(\frac{1}{2}\right) F_{lat-L} + F_{lat-R} \tag{3.4c}
\]

\[
F_{lat-avg} = F_{load} \tan(\theta). \tag{3.4d}
\]

We now have a solvable equation for lateral force; however, for successful calibration we need to relate the lateral force to the LFM signal \( (V_{LFM-avg}) \), in volts, displayed by the device. This will require the addition of a calibration constant, we shall call \( \alpha \). We are left with the equation,

\[
V_{LFM-avg} = \alpha F_{lat-avg} = \alpha F_{load} \tan(\theta). \tag{3.5}
\]
Equation (3.5) provides us with a solvable relation on which we shall base our calibration method. $\alpha$ serves as an experimental constant relation between the lateral force and the LFM signal; thus, finding its value shall be the primary goal and the focus of our experimental method.

To find the value for $\alpha$ we shall first require a smooth curved surface to take lateral force measurements moving in each direction. For example consider the surface presented

Figure 3.2: Graphs of example topography and expected lateral force signal (V) vs. x position (\(\mu\text{m}\)).
in figure 3.2a. The figure represents a simplified example and it is not necessary that the sample used be of this shape; however, the surface used must be smooth and free from asperities. From such a surface we might expect our LFM signal ($V_{LFM}$) plotted against horizontal cantilever position ($x$) to look like those presented in figure 3.2b. Given force curves such as these it is a simple matter to fit a polynomial to each of the curves and find the mean. Equation (3.5) tells us that our, experimentally derived, mean value curve is equal to $\alpha F_{load} \tan(\theta)$. We find an equation for $\tan(\theta)$ with respect to horizontal position ($x$) by returning to the plot of our topography, figure 3.2a. If we fit a polynomial to the topography we are presented with an equation for the surface height ($y$) in terms of horizontal position ($x$). Noting the fact that, 

$$\frac{\partial y}{\partial x} = \tan(\theta), \quad (3.6)$$

it becomes clear that the derivative with respect to $x$ of our experimentally derived equation for $y$ is indeed an equation for $\tan(\theta(x))$. $F_{load}$ was found using normal force calibration and the set-point value and, for our purposes, should be considered constant.

To solve for $\alpha$ in equation (3.5) we will re-plot our lateral signal ($V_{LFM-avg}$) in an attempt to linearize the curve. In this case we redefine the x axis as $F_{load} \tan(\theta(x))$ and find the slope of the resulting line, which should be $\alpha$; starting with figure 3.2b we would expect linearization to result in figure 3.3.

The procedure is reproduced experimentally on the Autoprobe M5 AFM system using the X-Y trace mode with the x detector as our driving force. By collecting lateral force signal ($V_{LFM}$), and topographical data from the Z detector for the cantilever moving in each direction we can perform the same procedure and determine the calibration constant $\alpha$. 

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Figure 3.3: Graph of $LFM_{avg}$ vs. $F_{load} \tan(\theta)$. We expect the slope of the line ($\alpha$) to be the calibration constant.
Chapter 4

Results

4.1 Searching for Samples

Proceeding experimentally with our methodology a suitable sample had to be identified. We required a sample containing a range of smoothly transitioning slopes, have sufficient curvature to see a noticeable change, and be very smooth on the nano-scale.

4.1.1 Piano String

Steel piano string was readily available in the lab and became our first candidate for testing. Piano wire is round, small, and seemingly smooth to the eye. The wire used was steel with a diameter of .011in. Despite the samples desirable properties it became clear that piano wire would not be a suitable sample for our experiment. The extrusion process used to manufacture the steel string left long horizontal ridges lengthwise along the sample. This interfered with the lateral force signal and resulted in highly erratic data.

4.1.2 Steel Rod

Sample testing continued with a small steel rod. The rod was chosen in the hope that it might not have the same problems as the piano wire since it had been machined on a lathe which would leave grooves in the radial direction. Radial grooves would be less
likely to affect our data and the steel rod presented a convenient sample.

Following the methodology the normal force was held constant and data was acquired for the topography (\(z\) position), lateral force, and the horizontal position (\(x\) position). We then calculated the average LFM value and fit a 3rd order polynomial to the topographic data. Plotting the topography (\(\mu m\)) and LFM (V) vs. horizontal position (\(\mu m\)), shown in figure 4.1, we found a much smoother surface then the previous samples. Unfortunately, the LFM signal was still fairly erratic.

![Figure 4.1: Plot of steel rod topography (\(\mu m\)) and LFM (V) vs. x detector (\(\mu m\)).](image1.png)

**Figure 4.1:** Plot of steel rod topography (\(\mu m\)) and LFM (V) vs. x detector (\(\mu m\)).

![Figure 4.2: Plot of \(LFM_{avg}\) vs \(F_{load}\tan(\theta)\) taken on a steel rod.](image2.png)

**Figure 4.2:** Plot of \(LFM_{avg}\) vs \(F_{load}\tan(\theta)\) taken on a steel rod.
Re-plotting the $LFM_{avg}$ vs. $\tan(\theta)$, shown in figure 4.2, we see that the plot displays a low degree of linearity with $R^2 = 0.363$.

### 4.1.3 Solder Spheres

Having exhausted readily available samples we attempted to create our own using solder. Using the hotplate we attempted to melt solder onto a metal sample disk. It was hoped that the surface tension would cause the liquid solder to form a smooth spherical ball that would then solidify and could be used for testing. The liquid solder did indeed form smooth shiny spheres on the sample disk. Unfortunately a problem arose during the cooling phase producing a rough dull surface entirely unsuitable for our needs. Theorizing that the solder might be cooling unevenly, an attempt was made to cool the solder slowly. This method did not produce improved results. Despite the clear failure of this sample attempt, data was collected and analyzed. Figure 4.3 shows the topography ($\mu m$) and LFM (V) vs. horizontal position ($\mu m$).

![Figure 4.3: Plot of solder sphere topography ($\mu m$) and LFM (V) vs. x detector ($\mu m$).](image)

Both the topography and the LFM signals show a high degree of erratic behavior and the results get no smoother when re-plotted. Re-plotting the $LFM_{avg}$ vs. $\tan(\theta)$, shown in figure 4.4, we see absolutely no semblance of linearity, with an $R^2$ value of 0.035.
Figure 4.4: Plot of $LFM_{avg}$ vs $F_{load} \tan(\theta)$ taken on a solder sphere.

4.1.4 $Si_3N_4$ Ball Bearings

The next sample analyzed was a $\frac{1}{16}$ th in (1.588 mm) diameter silicon nitride($Si_3N_4$) loose ball bearing. $Si_3N_4$ is an extremely hard substance and high quality ball bearings are extremely smooth. The manufacturer’s specifications listed the ball bearings to be spherical to within 5 millionth of an inch.

Figure 4.5: Plot of $Si_3N_4$ ball bearing topography ($\mu m$) and LFM (V) vs. x detector ($\mu m$).
Figure 4.6: Plot of $LFM_{avg}$ vs $F_{load}\tan(\theta)$ taken on a $Si_3N_4$ ball bearing.

Data was collected and the topography ($\mu m$) and LFM (V) vs. horizontal position ($\mu m$) are shown in figure 4.5. The figure shows a very smooth topography, as we expected, and a much smoother LFM signal. Figure 4.6 shows the re-plot of the data $LFM_{avg}$ vs $F_{load}\tan(\theta)$. Despite the extremely smooth surface the re-plot still did not show the degree of linearity expected, with an $R^2$ of only 0.434. These findings lead to an investigation of possible sources of error.

### 4.2 Crosstalk Between Normal and Lateral Signals

Seeking to improve our data we began a thorough investigation of possible problems. Crosstalk between the normal and lateral force signals was investigated as a possible source of the non-linearity in our data. Crosstalk occurs when the photo diode is not perfectly aligned with the lasers path of motion. This misalignment causes some portion of the normal force change to be detected as a lateral force, as shown in figure 4.7. Even a small misalignment in the photo detector can hugely interfere with the relatively small LFM signal. Ogletree et al. found that a rotation as small as $2^\circ$ was enough to cause the lateral force due to cross talk to be much greater than the lateral force being detected under standard experimental conditions [1].
In order to test the alignment of our photo detector we setup the cantilever and performed a driven oscillation in free space. With the cantilever oscillating we recorded both the normal force signal and the lateral force signal and compared the two. We collected data at a variety of driving forces all displayed in appendix A. Figure 4.8 represents our findings and shows a graph of both the normal and lateral force signals vs. time at 100% driving force. From the figure we observe a clear and strong correlation between the normal and lateral force signals.
4.3 Averageing Values From a Full Image

In an attempt to overcome the nonlinearities in the single scan method we attempted to take the mean values of topography and LFM over a 256 X 256 point scan. Scans of the $Si_3N_4$ ball bearing were taken over a range of 27 $\mu m$ X 27 $\mu m$ and the data points averages along the y-axis. The normal force was maintained at a constant value recorded by the photo diode as $\approx -2.0 V$. The normal force of the NSC16 cantilevers used were calibrated using the thermal technique described in the appendix of Erik Thoreson’s dissertation [6]. The normal force constant of the cantilever used in the acquisition of the data set presented was found to be 33.396 $N/m$. A force curve was taken to determine the signal response vs. cantilever movement and was found to be $19.11 \times 10^7 V/m$; however, it should be noted that the reaction of the cantilever is variable between setups and data must be acquired without altering the apparatus. Plugging this data into equation (4.1) we see that $F_{load} = 3.49 \times 10^{-6} N$.

$$F_{load} = (2.0V)(3.396 \frac{N}{V/m})(1.11 \times 10^7 \frac{V}{m})^{-1} = 3.49 \times 10^{-6} N.$$  (4.1)

![Figure 4.9: Plots of mean topographic and LFM data from a 256 X 256 point scan of a 397$\mu m$ Si$_3$N$_4$ ball bearing. Data was taken over a range of 27 $\mu m$ X 27 $\mu m$ with a calibrated NSC-16 cantilever](image)

The average topographic and LFM data with respect to the x detector is shown in
figure 4.9. Ten data points on each side were dropped to allow the cantilever to reach equilibrium as it changed directions. Then a third degree polynomial was fit to the topographic data in figure 4.9a resulting in an equation for the topography of:

$$Z = -2 \times 10^{-6}x^3 + 1 \times 10^{-4}x^2 + 0.065x - 3.562.$$  \hspace{1cm} (4.2)

Taking the derivative with respect to x we arrive at,

$$\tan(\theta) = -6 \times 10^{-6}x^2 + 2 \times 10^{-4}x + 0.065.$$  \hspace{1cm} (4.3)

Using this data we re-plot the $LFM_{avg}$ with respect to $F_{load}\tan(\theta)$ as shown in figure

Figure 4.10: Plot of $LFM_{avg}$ vs $F_{load}\tan(\theta)$ from a 256 X 256 point scan of a 397µm $Si_3N_4$ ball bearing. Data was taken over a range of 27 µm X 27 µm with a calibrated NSC-16 cantilever
4.10. Ploting a least squares fit line to the data we see an $R^2$ value of .961 indicating a high degree of linearity. We expect the slope of this line to be the lateral force calibration constant $\alpha$. Calculations show the slope to be $47396\frac{V}{N}$. We take the inverse of this value to put the calibration constant in standard form, arriving at $21.1\frac{\mu N}{V}$. 

Chapter 5

Conclusions

The experimental phase of the undertaking faced a myriad of unpredicted difficulties and delays, as is the nature of experimental work. Looking into the cause of these difficulties we derived many potential causes some of which may require further analysis in the future.

5.1 Sources of Error

While our experiment demonstrated a successful proof of concept there are still several sources of error to consider.

Crosstalk

Crosstalk between the lateral and normal force signals was investigated as a possible source of error. As shown in figure 4.8 crosstalk is occurring in our Autoprobe M5 microscope and this could be affecting our data. It is unclear exactly what affect this crosstalk might be having on our data or the extent to which it is affecting our results. Additional study is required in this area.

Curvature of Ball Bearing Insufficient

AFM cantilevers are very stiff in the lateral direction; therefore, the LFM signal is generally much smaller than the normal force signal. If the sample is not producing a sufficient LFM response than our data will be far more susceptible to other forms of error such
as crosstalk and interference. A potential solution to this problem is to acquire smaller diameter ball bearings; this will increase the curvature and provide a wider range of LFM response per area.

**Approximations Made in Full Scan Analysis**

Likely the biggest source of error is the approximations made in the full scan analysis. By taking the topography and LFM signal from a 27 X 27 (µm) scan and averaging it into one data set we are making an assumption that the topography and LFM of these areas are nearly identical. This assumption may or may not be valid, depending on the situation, thus this is a major potential source of error.

**5.2 Accomplishments**

Despite the experimental difficulties encountered significant accomplishments were made in the development of the mean-value lateral force calibration method. We succeeded in showing a myriad of samples and settings that do not work. However, after much trial we were successful in identifying the Si₃N₄ lose ball bearings as a suitable sample for the technique.

We were also successful in collecting data for the improved Asay and Kim method proposed in the Major Qualifying Project (MQP) of Keeley Stevens and Colin DeGraf [7]. Their MQP proved inconclusive due to wild oscillations in their data. It was determined that the data acquisition software’s auto correction feature (Scanmaster) was causing their erratic findings. The data collected is displayed in appendix B.3.

The greatest success came from collecting a data set displaying the strong linear relation we expected, figure 4.10. Through experimentation the original method was adapted to include an entire 256 X 256 point scan. Taking the average data over this entire scan range resulted in very clear data in support of our methodology. While we did not have
sufficient time to complete analysis of the calibration method a proof of concept has been established as a foundation for further development.

5.3 Further Research

While we were successful in completing a solid proof of concept there is still much room for further analysis. Due to the unexpected complications intrinsic in a project of this nature time constraints were the limiting factor in the pursuit of additional study.

In order to test the accuracy of our results a comparison needs to be performed with one, or more, of the many accepted means of lateral force calibration. This will give us a better idea of the extent to which the sources of error are affecting the results. Along these lines the problem of normal and lateral signal crosstalk could be investigated more thoroughly as this may be a significant source of error.

Another area for improvement is the way in which the scans are averaged. Currently we are simply averaging the topographic and LFM values along the y axis and then re-plotting the $LFM_{avg vs. F_{load}} \tan(\theta)$. This may be an acceptable method given the right conditions and sufficiently small scan range. However, a more accurate method may be re-plotting each line of topographic and LFM data, then combining all of this data to calculate a least squares fit. This method would require a script to be programmed and this was not possible to pursue given time limitations.

There also exists the possibility to pursue the mean value analysis described in our methodology to other physical problems. The fundamental principals should be applicable to any force balance system. Therefore, it is quite possible our simplification could aid in the development of many other physical fields.

The results for the technique we have shown here hold great promise. The method has the potential to greatly improve the process of lateral force calibration and could aid
future researchers advance the field of nano-science.
Bibliography


Appendix A

Supplementary Data

Figure A.1: Graph of vertical and lateral voltage signal vs. time from an oscillating NSC-16 cantilever.

(a) 40% driving force.

(b) 80% driving force.
Figure A.2: A complete data set from an un-calibrated full image analysis of a 27 X 27 (µm) scan of an Si₃N₄ ball bearing.
Figure A.3: A complete data set from an un-calibrated full image analysis of a 27 X 27 (µm) scan of an Si₃N₄ ball bearing.
Figure A.4: A complete data set from a calibrated full image analysis of a 27 X 27 (µm) scan of an $Si_3N_4$ ball bearing.
Appendix B

Grant Proposal

In order to secure funding for the project we applied for a grant from Sigma-Xi, through the Grants-in-Aid of Research Program. The highly competitive program offers grant money to undergraduate researchers based entirely on scientific merit and potential of the applicant. We were awarded the entire $482 dollar sum requested.

B.1 Mean-Value Method

Lateral force microscopy (LFM) is an essential tool in the study of friction phenomenon at the nanometer scale, a fundamental area of research in the field of nanoscience. For valuable data to be acquired the cantilevers lateral stiffness must first be calibrated. Several methods of LFM calibration have been derived; however, all of these methods suffer from excessive tip wear or the need for special equipment. In 2006 Asay and Kim published a new LFM calibration method with the goal of reducing tip wear. Unfortunately the method suffered from serious physical errors and in 2006 a team at WPI produced a corrected model of the Asay and Kim method. With the corrected model problems with the fundamental assumptions in the method became apparent. Taking the goal of the Asay and Kim model, limited wear to the tip, we began our own original research.

Motivated by the flawed method proposed by Asay and Kim, we hope to develop an accurate, inexpensive, and reliable method of LFM calibration with limited tip wear.
Using a proper free-body diagram for the tip sample interactions we arrive at equilibrium equations for the vertical and lateral directions. The vertical load, normal, and adhesion forces are found by calibrating the spring constant of the cantilever, leaving only the kinetic friction and the lateral force unknown. If we are sampling a surface with a small angle and sufficiently low friction coefficient then the vertical component of the friction becomes insignificant. Solving the system then for lateral force yields a fairly simple equation, although it still involves friction force. To resolve this issue we use a clever trick; observing that the friction force changes signs depending on the direction of the scan, we find that the mean lateral force of a scan in each direction produces an equation for lateral force that does not involve friction force.

To take advantage of this simplification experimentally we will need to scan a smooth sample with small angles. After calibrating the normal spring constant of the tip we will take a constant force scan in both lateral directions and graph both the topography and the Lateral force signal (in volts) vs. the lateral position of the scanner. We will then take the mean value of the two lateral force signals which should be equal the vertical load times the tangent of the slope angle times a conversion constant (volts to newtons). A curve can then be fit to the topography, and we can determine the angle of the surface leaving only the conversion constant unknown. It is then a simple matter to linearize the equation and determine the conversion constant. This methodology, if successful, would greatly simplify the process of lateral force calibration and minimally wear the cantilever tip. Sigma Xi’s support would enable this project, which we plan to publish in a peer-reviewed journal.

B.2 Literature Citations


B.3 Project Budget

VXB Ball Bearings
10 X 1.588mm diameter-Ceramic $Si_3N_4$-Grade 5-Loose Balls $14.00
It is hoped that these samples will provide a smooth sample for scanning and collecting LFM data.

Advert Research Materials
1.0m - 0.25mm diameter - 99.99% pure - Indium Wire $168
By melting the wire into a smooth puddle it is hoped that we can produce a very smooth curved sample.

Mikro Masch 15 X NSC 16 rectangular cantilevers with AL backside coating $300
Prolonged testing will result in the wear on cantilevers thus we will require new cantilevers for continued data acquisition.
Appendix C

Corrected Asay and Kim Model

Here is presented a proof of concept for data acquisition of the corrected Asay and Kim model proposed in Keeley Stevens and Colin DeGraf’s Major Qualifying project [7]. They were unsuccessful in acquiring data because they did not turn off the Scanmaster function in the acquisition software.

Figure C.1: Graph of LFM (V) vs. Z Detector (µm) on flat portion of TGF11 sample.