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Characterization and evaluation of the microcantilever radiation detector

Andrew Curtis Stephan

To the Graduate Council:

I am submitting herewith a thesis written by Andrew Curtis Stephan entitled "Characterization and evaluation of the microcantilever radiation detector." I have examined the final electronic copy of this thesis for form and content and recommend that it be accepted in partial fulfillment of the requirements for the degree of Master of Science, with a major in Nuclear Engineering.

Laurence Miller, Major Professor

We have read this thesis and recommend its acceptance:

Lawrence Townsend, Ronald Pevey, Thomas Thundat

Accepted for the Council:
Carolyn R. Hodges

Vice Provost and Dean of the Graduate School

(Original signatures are on file with official student records.)

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Anne Mayhew

Associate Vice Chancellor and
Dean of The Graduate School

CHARACTERIZATION AND EVALUATION OF A MICROCANTILEVER RADIATION DETECTOR

A Thesis
Presented for the
Master of Science
Degree
The University of Tennessee, Knoxville

Andrew Curtis Stephan
December 1999

DEDICATION

This thesis is dedicated to my parents,

Gerald and Nancy Stephan,

who wanted the best education for me,

and worked hard to see that it happened.

ACKNOWLEDGEMENTS

This thesis would not have been possible without the help of many people and I owe a debt of gratitude to all of them. The members of my thesis committee, Laurence Miller, Lawrence Townsend, Ronald Pevey, and Thomas Thundat, have been very helpful with their suggestions, encouragement, and patience and I appreciate it very much. I am very thankful for the people who have helped me move this thesis along, whether by working with me on experiments and problems, or by helping me to better understand microcantilevers. Among them are Thomas Thundat, Ari-David Brown, Travis Gaulden, Bruce Warmack, Eric Finot, and others. My parents have alternately encouraged and needled me along, something I definitely needed and am very thankful for. I have received generous financial support from the US Department of Energy through the Applied Health Physics Fellowship program. Without that support, I would not have been able to undertake this topic of study. I greatly appreciate the prayers of my church family at Bible Presbyterian Church. Only eternity will tell how much they have helped. Most of all, I would like to thank the Lord Jesus Christ, my Maker, Redeemer, and Friend. This would not have been possible without You.

ABSTRACT

In this study, a microcantilever charged-particle detector was characterized and evaluated. The method employed involved sensing an electric field emanating from a small collector plate on which charge built up to charged-particle flux. Sensitivity to alpha particles was determined using frequency and damping rate parameter shifts. Alpha particle detection experiments were compared to experiments using a charged plate of fixed voltage in order to characterize response more fully and to identify differences between expected charge accumulation and actual accumulation. Changes in cantilever behavior resulting from changes in ambient environmental conditions were also studied in order to determine to what extent they would impact charged-particle detection. In particular, microcantilever tip-surface adhesion force and jump-to-contact distance were studied as a function of relative humidity, and the dynamics of the liquid neck extending between the microcantilever tip and the surface at small separation distances were investigated. In addition, a relationship between the angle of the microcantilever tip relative to the surface and the excitation of multiple resonance modes was identified and described.

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Chapter 1

Introduction

The field of MEMS (micro electromechanical sensors) has become an important area of research in recent years. The microcantilever has generated considerable interest as a MEMS device, having been demonstrated to possess sensitivity far beyond current devices for detection and measurement. However, no investigation of microcantilevers as ionizing radiation detectors has been reported in the literature. The purpose of this work is to evaluate and characterize the microcantilever platform as a basis for radiation detection and measurement. The primary focus of this investigation is firstly the detection of charged particles using a method whereby charged particles are collected on an insulated, conducting plate thereby producing an electric field that can be measured and secondly the evaluation of the possible usefulness of this method. While this method is by no means the only one possible for detecting radiation, it was felt that it would be one of the more feasible methods given the instrumentation available and that the information gathered would have wide application in future work on microcantilever radiation detectors.

Investigations relating to the microcantilever radiation detector fell into several distinct areas. The first was to determine an appropriate design for the radiation detector. This involved testing different types of collector plates, determining how best to mount and electrically isolate the collector plate, how to accomplish charge drain without disturbing the microcantilever, and identification and mitigation of untoward effects that inhibited or prevented microcantilever response. One such problem was electron showers causing electrons to become trapped in materials adjacent to the collector plate, affecting charge distribution on the plate and diminishing cantilever response. Coming up with a properly working detector proved to be quite a challenge. Some of the factors considered and the design decisions made are detailed in Chapters 4 and 5.

The results of the charged particle detection experiments (specifically, alpha detection), describing changes in parameters such as cantilever frequency and damping rates, are discussed in Chapter 6. Included are other experimental results, in particular those relating to a series of experiments in which a voltage was put on a collector plate in order to simulate the accumulation of charge due to alpha irradiation. This allowed the rate of actual charge accumulation from alpha particles to be compared with the expected rate of accumulation. It also provided a controlled way in which to examine how cantilever behavior in response to an electric field (such was would be emitted from a collector plate) changed with variation in parameters such as relative humidity and cantilever to plate distance. These experiments also showed that some effects were taking place during the charged particle detection experiments that were initially unexpected but readily explainable. Analysis of the data from these experiments allowed a detailed prediction of expected cantilever response for different detector configurations and shed light on other possible detector designs not specifically studied in this work.

Finally, investigations were conducted on other factors capable of affecting detector response, particularly those that were dependent on ambient environmental conditions. Several areas were singled out. The effect of relative humidity level on adhesion forces between a cantilever tip and an adjacent surface was characterized as this substantially affected the breakaway curves generated during, and necessary to, a charged particle detection experiment. The dynamic behavior of the water bridge existing between the cantilever tip and the adjacent surface during breakaway was investigated in detail as a critical influence on the breakaway curve. Finally, the positioning of the cantilever relative to the collector plate, in the form of the tip-surface angle, was shown to have a profound effect on the breakaway curve. The causes of this were found and an idea of acceptable positioning given. Discussion of such modifying factors is in Chapter 7.

Chapter 2

Overview of Microcantilevers

The past decade has seen the beginnings of an exciting new field: that of micro-electromechanical sensors (MEMS). These devices promise to revolutionize many aspects of science and scientific applications. For example, researchers are developing microsensors for detecting drugs and explosives that will render canine detection obsolete. Microcantilevers are in many ways at the forefront of this emerging area of technology.

In its most basic form, a microcantilever is a micron-scale miniature diving board used for sensing and measurement applications. (See Figure II-1 for an image of a microcantilever.) Variances in microcantilever characteristics and responses can be measured and used as indicators of changes in environmental conditions. The most common responses of interest are changes in the deflection (that is, the bending), the resonance frequency, the Q-factor (damping rate), and the oscillation amplitude of a cantilever. There are some simple examples of how each of these responses works. Suppose a microcantilever is coated with gold on its upper surface and then exposed to mercury vapor. Gold is excellent at absorbing mercury. As mercury diffuses into the

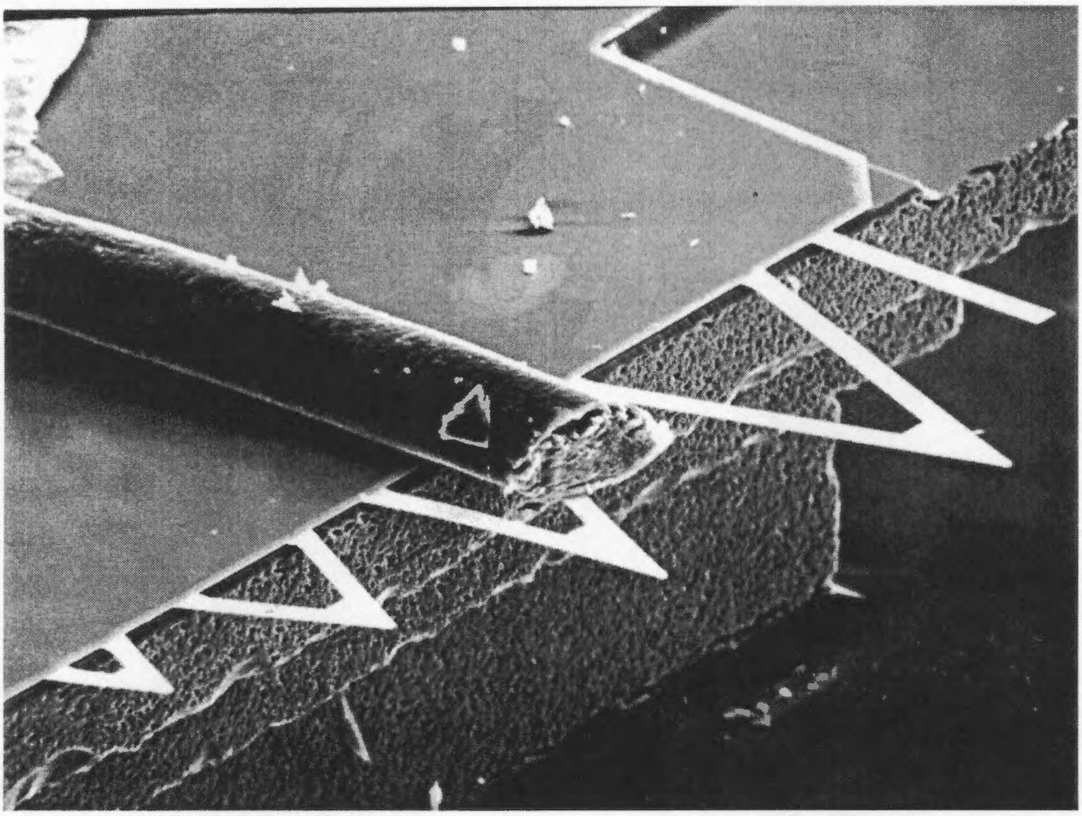

Figure II-1. Microcantilevers and a Human Hair

A group of microcantilevers attached to a substrate wafer shown with a human hair for size comparison. The image was taken with an SEM (Scanning Electron Microscope). Photo courtesy of Dr. Bruce Warmack, Oak Ridge National Laboratory.

gold and forms an amalgam, the surface stress on the upper surface of the cantilever changes and the cantilever bends. This bending can be used as an indicator of the mercury vapor concentration in the air. Frequency can change for a number of different reasons, one of the most common being a change in the mass of the cantilever system caused by adsorption of a target molecule onto a substrate coating the cantilever surface (such as in the gold/mercury example). Generally this occurs in conjunction with changes in the Young's modulus of a material in the cantilever as molecules diffuse into it (this also acts to change the natural harmonic frequency). One reason for a change in the cantilever's Q-factor might be the presence of an electric field. As cantilevers are made of semiconductor materials, they will, in this case, act rather like a conductor and become more negative on one side and more positive on the other. This has the effect of reducing the effective spring constant of the cantilever, causing it to damp out at a different rate. Lastly, a cantilever's amplitude will change if the viscosity of the surrounding medium changes.

The sensitivity of microcantilevers is extraordinary. Originally microcantilevers were used almost exclusively in Atomic Force Microscopes (AFMs) for surface scanning. As a microcantilever rose and fell with a surface, deflections of an Angstrom could be easily measured using a laser or other position detection system, enabling imaging of individual atoms. In 1993, Drs. T. G. Thundat and E. A. Wachter observed changes in cantilever behavior, specifically frequency, with humidity. They realized that microcantilevers could be used for sensing and measurement in a whole host of applications.[1,2,3] Since then, microcantilever measurement and sensor systems have been demonstrated in many physical, biological, and chemical areas. Sensitivities of parts-per-trillion for chemicals in air, picograms for cantilever mass change due to molecular adsorption, and femtoJoules in calorimetry have all been reported.[4]

The number of people working in the microcantilever field has been steadily increasing and several companies have been formed specifically to develop micro-cantilever-based products. The first microcantilever-based sensors (infrared imagers) will likely appear over the next few years. The ultimate potential of the microcantilever sensor is as yet unknown, but the future looks promising.

Chapter 3

Microcantilever Theory

3.1. Microcantilever Composition and Geometry

Microcantilevers usually come in one of two distinct shapes. The first is a bar or diving board shape, while the second is a hollow centered isosceles triangle shape in which the mounting base forms the third and shortest side. (See Figure III-1 for an image of a bar cantilever and Figure III-2 for an image of triangular cantilevers.) Each shape has distinct advantages and disadvantages. Triangular cantilevers have a larger surface area than the bar version. They can be easily heated by passing a current through the legs (the base of one leg is positive, the base of the other, negative). The primary disadvantage of triangular cantilevers is the presence of torsional vibrational modes (side to side, rather than up and down) which can complicate cantilever resonance at certain frequencies. The advantages and disadvantages of bar cantilevers are primarily that they lack the disadvantages and advantages (especially easy heating capabilities) of triangular cantilevers.

Microcantilevers generally usually have a tip, a narrow cone-like structure

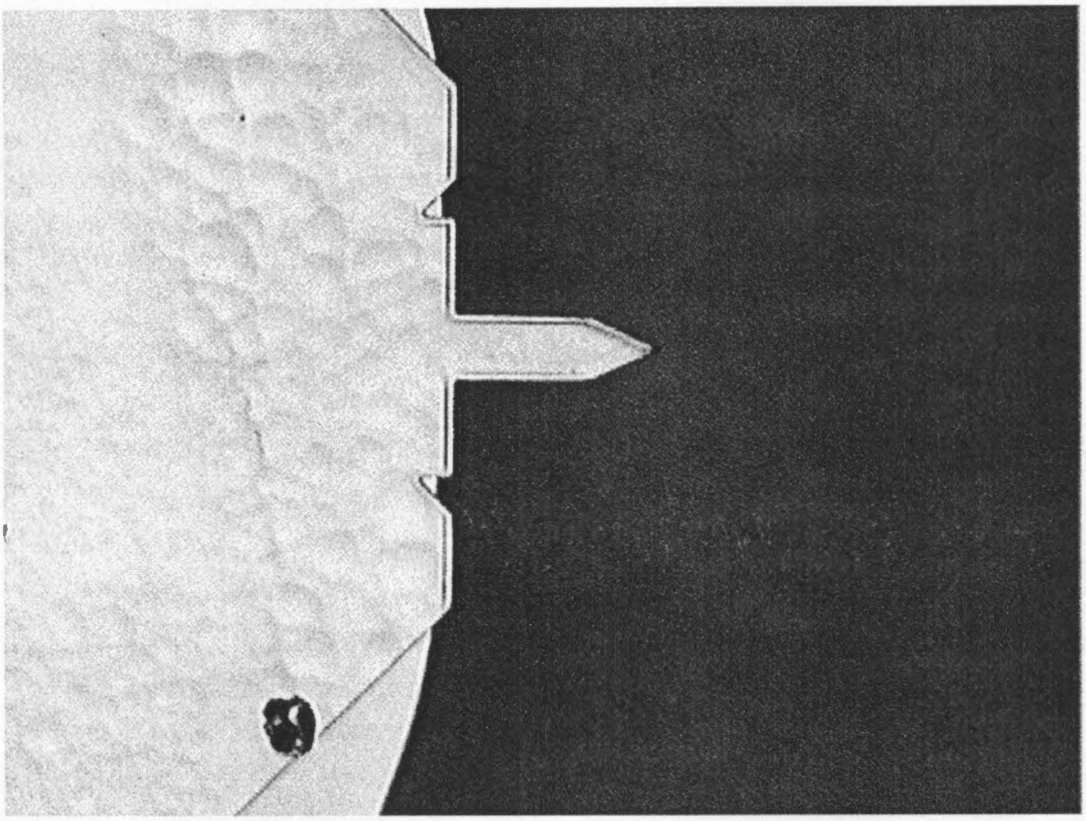

Figure III-1. Bar Cantilever

Bar cantilevers can come as purely rectangular shapes, or, as in this case, with pointed ends.

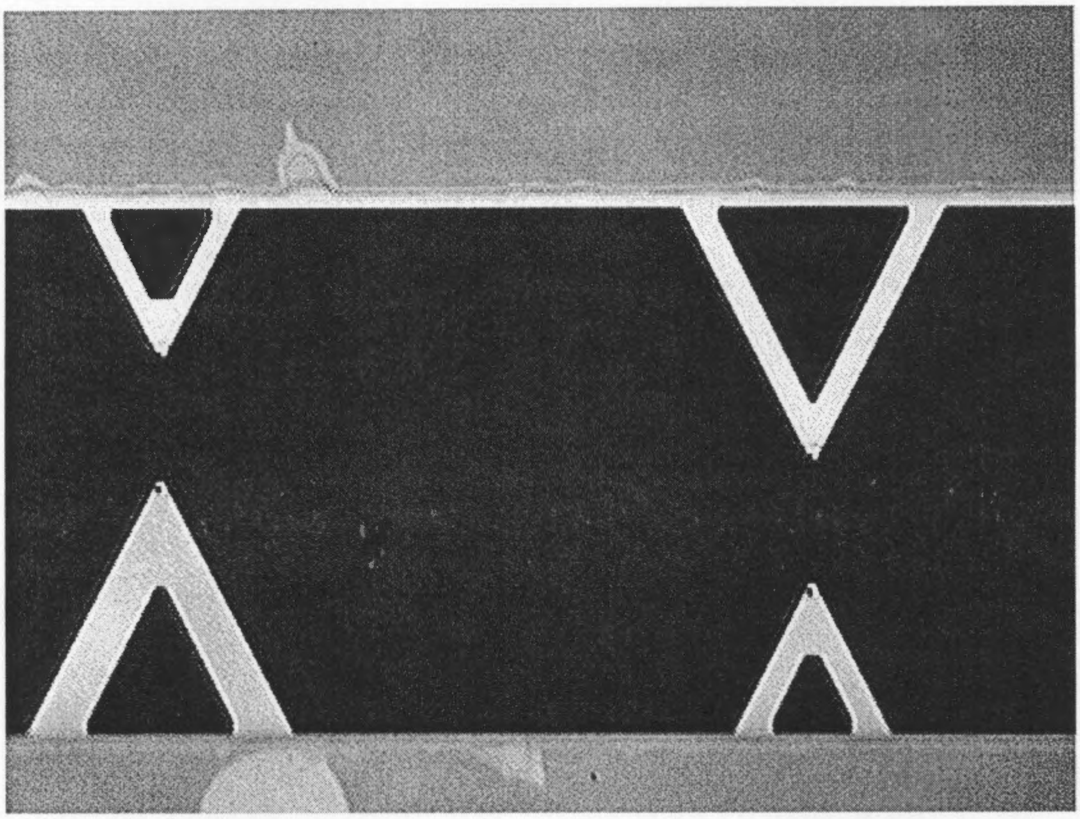

Figure III-2. Triangular Cantilevers

Cantilevers of different lengths and widths are shown. The areas that appear to be holes are actually indentations in the upper surface of the cantilever above the tip.

projecting from the underside of the cantilever near its end. (See Figure III-3 for a picture of a tip.) The tip is typically very sharp, although manufacturing limitations dictate that the apex of the tip be spherical in shape. The radius of curvature of the apex of a tip is generally on the order of 15 to 50 nm and the total tip length two or three microns. The presence of a tip is critical for microscopy applications as it allows for a very small area of contact between the microcantilever and the surface being probed, thus maximizing resolution. The main portion of the cantilever is generally referred to as the beam when it is necessary to avoid confusing it with the tip.

Commercially available microcantilevers come in a variety of sizes with typical lengths of 100 – 200 μm, widths (per leg) of 20 – 40 μm, and thicknesses of 0.3 – 4 μm. With these large variations in dimension, cantilevers vary substantially in harmonic frequency, stiffness, damping rates, and other characteristics.

In theory, microcantilevers can be constructed from a vast range of materials. In practice, they are usually made from semiconductors as existing semiconductor manufacturing technology makes it relatively easy to make such small devices cheaply and precisely compared to construction out of other materials. Specific materials used to date are silicon (Si), silicon nitride (Si_3N_4), and gallium arsenide (GaAs). All cantilevers used in this work were silicon nitride.

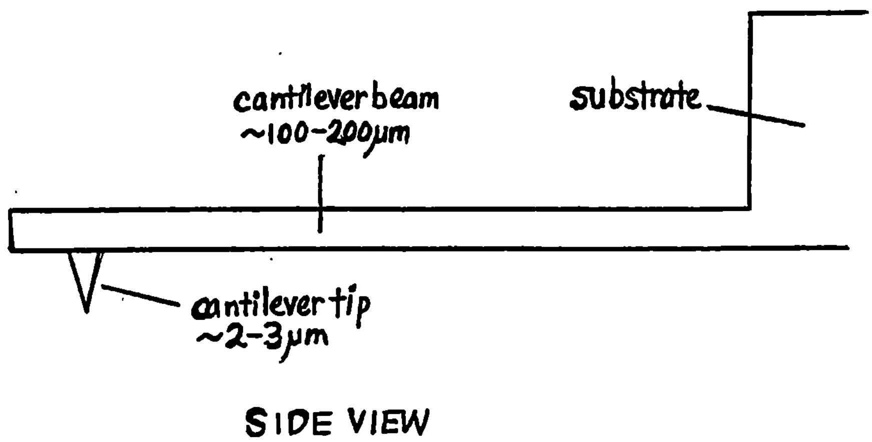

Figure III - 3. Microcantilever Tip

The arrangement of a cantilever tip relative to a microcantilever beam.

3.2. Basic Mechanical Characteristics of Microcantilevers

As alluded to earlier, microcantilevers, being rather like miniature diving boards, exhibit similar kinds of mechanical characteristics; they can be bent if a force is applied to them and, if excited, they tend to vibrate at particular frequencies. (See Figure III-4 for an illustration of the basic mechanical characteristics of microcantilevers.) The parameters for these kinds of motions (spring constant and resonance frequency) can be calculated if basic structural information is known. This will now be discussed and explained. The following equations for f, k, and I and the accompanying discussions are based on those Dror Sarid's book.[5]

The motion of a cantilever tip approximates a simple spring quite well. Cantilevers have distinct frequencies of vibration, spring constants, and damping rates. The resonance frequency, *f*, of an oscillating cantilever can be expressed as

$$f = \frac{1}{2\pi}\sqrt{\frac{k}{m^*}}.$$

Where k is the spring constant and m^* is the effective mass of the cantilever. The effective mass of a cantilever is related to the actual mass of the bar, m_b, by a geometric parameter n where $m^* = n\, m_b$. The value of n for a bar cantilever is 0.24, while triangular cantilevers typically have values between 0.14 and 0.18, depending

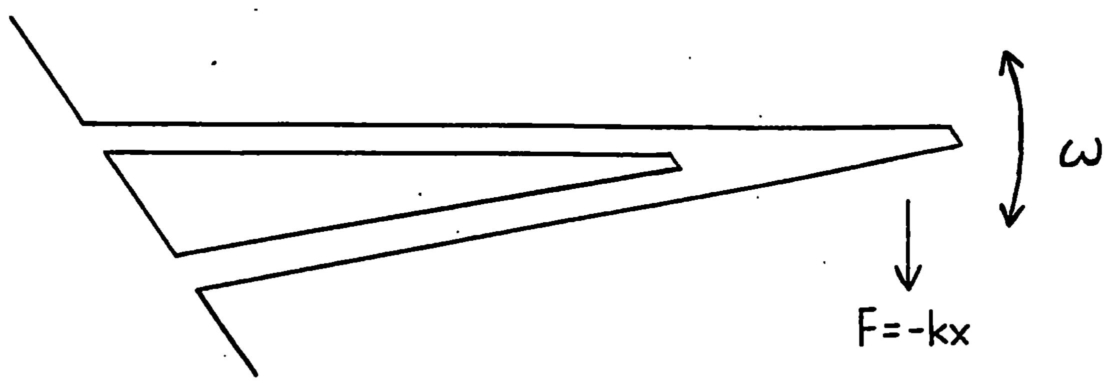

Figure III-4. Basic Mechanical Characteristics of Microcantilevers

A cantilever has some spring constant k that determines how much it bends when a force is applied to it. It also has some natural rate of oscillation, ω, when excited.

on their exact geometry. (The coefficient for bar cantilevers can be easily mathematically derived, while triangular cantilevers, depending on their exact geometry, more often need to be experimentally measured.) Frequency can shift as a result of changes in either mass or spring constant. Shifts in spring constant are generally the result of changes in either the surface stress or the Young's modulus of a cantilever. This can be problematic as, in some cases, changes in mass and spring constant upon exposure to the influence of interest can offset each other producing no net effect. If this problem occurs, it can usually be mitigated in one of several ways. One such method is to confine the adsorption area to the terminal end of the cantilever (end loading), thus minimizing differential stress and ensuring that changes in resonance frequency can be wholly attributed to changes in mass loading.

Determining k for a bar cantilever of uniform composition is quite simple:

$$k = \frac{3EI}{l^3}$$

Where E is Young's modulus of elasticity for the cantilever material, I is the moment of inertia, and l is the length of the lever.

Since the inertia for a rectangular lever is

$$I = \frac{wt^3}{12},$$

where w is the width and t is the thickness of the cantilever, then

$$k = \frac{Ewt^3}{4l^3}.$$

Unfortunately calculating k becomes rather complex for all but this most simple case and so approximations are frequently used. One common approximation for a triangular cantilever of uniform composition consists of two cantilevers lying side by side, where each cantilever represents one leg of the triangular cantilever. The lengths are taken as the maximum length of a leg in the original cantilever. The two cantilevers side by side are then treated as a single bar cantilever. This method is generally accurate to within 10%. An actual calculation of k (non-approximation) involves integrating over the area of the cantilever to find the moment of inertia. There is no simple equation as geometries vary substantially. Similarly, when cantilevers are inhomogeneous, i.e. have multiple layers, calculating k becomes increasingly difficult. In such cases the spring constant can be determined experimentally by applying a force to a cantilever and measuring the bending response.

Having shown how to calculate frequency and spring constant for a bar cantilever of uniform composition, other geometries and cases involving multiple layers will not be discussed. As mentioned earlier, these cases quickly get very complex. In the experiments discussed in this thesis, values were experimentally determined as needed or taken from the manufacturer's data if deemed sufficiently accurate.

3.3. Force Fields and Microcantilevers

When in the presence of a force with a non-zero derivative over space, a microcantilever will exhibit a change in resonance frequency. Forces with derivatives equal to zero will simply deflect the cantilever and the lever will resonate about this new position with an unchanged frequency. (This assumes that the amount of deflection is not sufficient to move it out of the area of purely elastic materials response.) This effect is very important to microcantilever sensors and Atomic Force Microscopes in general and to our application (radiation detection by charge sensing) in particular. Forces with non-zero derivatives are found near surfaces and in most electric fields, to give two pertinent examples. The following derivation follows that of Dror Sarid.[5]

Fields with non-zero derivatives cause frequency shifts in microcantilevers by changing the effective spring constant. It is simple to demonstrate this mathematically. Spring constant can be defined in several possible ways. Besides the usual force per distance method, spring constant can be written as a function of the potential energy, W, of a deflected cantilever, which is

$$W = \frac{1}{2}kx^2.$$

Taking the second derivative of the energy W with respect to x (the deflection distance),

$$k = -\frac{\partial^2 W}{\partial x^2}.$$

If a force F(x) is present with a derivative in the direction of the deflection of the cantilever, we can add in this second force to the restoring force to get a total force,

$$F(x) = F(x_0) + \frac{\partial F(x_0)}{\partial x}dx.$$

Applying the definition of k to the definition of W (which now includes the second force term), gives us the effective spring constant,

$$k_{eff} = k - F_1, \text{ where } F_1 = -\partial F / \partial x.$$

Thus, a tip-sample attractive force with a positive derivative will decrease the resonance frequency of the cantilever. For tip-sample attractive forces, the derivative will normally be equal to or greater than zero. If the derivative is negative, the tip-sample attractive force will decrease as the tip moves closer to the sample. Such a situation does occur very close to surfaces. It arises when the repulsive (contact) force begins to rise more quickly (in nominal terms) than the (attractive) van der Waals force. This occurs at approximately one angstrom and thus is essentially irrelevant to us. Tip-sample repulsive forces will normally increase the resonance frequency of the cantilever. As in the case of the attractive force, a reversal of the sign of the force derivative (in this case from negative to positive) is unusual and is not important to us. Note that a reversal of the sign will cause the cantilever's frequency to decrease instead of increase for repulsive forces, or increase instead of decrease for attractive forces.

It will be remembered that the frequency can be written as a function of the spring constant, k, and the effective mass, m^*. In the fields of microcantilever sensors and AFMs (Atomic Force Microscopes), the more common convention is to use ω, as in

$$\omega = \sqrt{\frac{k}{m^*}}.$$

Having found the effective spring constant under the conditions of a force with a non-zero derivative, we can now write the new equation for oscillation rate, namely,

$$f = \frac{1}{2\Pi}\sqrt{\frac{k - F_1}{m^*}} \text{ or } \omega = \sqrt{\frac{k - F_1}{m^*}}.$$

Or, alternatively, we can substitute k_{eff} for the term $k - F_1$, this, like the use of f or ω, is purely a matter of personal preference.

3.4. Electric Fields and Microcantilevers

If a microcantilever is near a surface, a difference in potential between the cantilever and the surface will result in electrostatic force acting between the cantilever and the surface. Derivation of theoretical models for force has been done using approximations to the shape of the tip.[6,7] Empirical results support these models.[7]

The electrostatic force between the cantilever and the surface is dependent on a number of different parameters, in particular: d, the tip-to-sample separation; R, the tip radius; Θ, the tip cone angle; L, the tip length; and U, the potential difference between the tip and the surface (in volts). For a tipped cantilever in the near vicinity of a surface, it is sufficient to just consider the tip because the distance between the surface and the rest of cantilever results in the surface-beam force being small, the electrostatic force being a $1/r^2$ force. Using this fact, several different geometrical approximations have been made for cantilever tips: the plane surface model (a circular area), the sphere model, and the charged line model (the equipotential surface from a uniform charged line is a good approximation to a conical tip). It has been found that the sphere model works well for $d<R$, while the charged-line model is superior when $R<d<L$.[7,8]

Given below are the formulae for F, the tip force, and F', the tip force gradient, for the plane surface model, the sphere model, and the van der Waals force model. Note that the electrostatic force equations are valid for d<R, zero contact potential, and an electrically grounded tip. As mentioned above, the charged-line model works reasonably well for R<d, even though the equation is technically not valid in that case. The van der Waals force is dominant at low voltages, while the electrostatic force is most important above several volts.[9]

Electrostatic - sphere[6]

$$F = -\frac{\Pi \varepsilon_0 R U^2}{d} \qquad F' = \frac{\Pi \varepsilon_0 R U^2}{d^2}$$

Electrostatic – charged line[7]

$$F = -\frac{\alpha^2}{4\Pi\varepsilon_0}\ln\left(\frac{L}{4d}\right)U^2 \qquad F' = \frac{\alpha^2 U^2}{4\Pi\varepsilon_0 d}$$

Where $\alpha = 2\Pi\varepsilon_0 / arc.\sinh[1/\tan(\Theta/2)]$

van der Waals (sphere)[10]

$$F = -\frac{HR}{6d^2} \qquad F' = \frac{HR}{3d^3}$$

Calculating the deflection of a microcantilever exposed to a force field is straightforward. Recalling that a cantilever behaves in a highly similar manner to a spring, one can simply use and rearrange Hooke's Law, which yields

$$x = -\frac{F}{k},$$

where x is the distance of deflection. The minus sign is an artifact of the mathematics and can essentially be ignored. Note that for fields with non-zero gradients, deflection of a cantilever may cause that cantilever to move into an area of different field strength, reducing the bend below what was expected the motion is into an area of lesser field strength. Likewise, motion into an area of greater field strength will make the bending greater than what was initially predicted. This effect can complicate matters. Most of the time though, for small deflections, this is a minor issue.

Besides causing deflection of the cantilever, a tip-sample force will cause a shift in resonance frequency. If the force gradient is approximately constant over the range of tip motion, the relationship between the force gradient $F = \partial F / \partial d$ and the shift in resonance frequency Δf is

$$F = -\frac{2k\Delta f}{f_{res}},$$

where k is the spring constant of the cantilever and f_{res} is the resonance frequency of the cantilever when no external force is acting upon it.[11]

Based on the information presented in this section, it can be seen that cantilevers with low spring constants are rather more sensitive to forces in general, and electrostatic forces in particular, when compared with cantilevers of higher spring constants. The lower a cantilever's spring constant, the more it will deflect if a given force is applied to it. Likewise, it can be seen from the last equation that for a given force, the change in resonance frequency will increase with a decreasing spring constant. Both of these observations make sense intuitively.

Cantilever damping rates are more complex than the other parameters discussed, in large measure because of their tendency to drift substantially over time (this will be explained and discussed later) due to various natural processes such as changes in relative humidity. These natural drifts are quite large relative to the shifts that they undergo due to the effect of interest. This means that while they may be useful for experimentation, but do not make a very practical basis for a field detector. Some general statements can be made without entering into the complex details. For a cantilever that shows an increase in frequency as a result of exposure to a field, damping will decrease. Conversely, a cantilever that shows a decrease in frequency will damp out more quickly.

Chapter 4

Cantilever Control Systems

4.1. Introduction

By itself, a microcantilever is a very small object. Under good conditions it may be possible for someone with sharp eyesight to barely make it out. Dealing with a microcantilever – moving it around, measuring its response – is not a very straightforward process. Certain things can be done to facilitate handling and control of a microcantilever. As an example, cantilevers are attached to a small substrate wafer that can be moved around by hand with tweezers. (See Figure IV-1.)

In this section, some important practical aspects of using microcantilevers will be discussed. The way in which a cantilever is physically dealt with (being so small) and how its mechanical response is measured will be covered. Several different methods for detection of deflection will be discussed as will the use of a cantilever when a surface is present, as well as when one is not present. Control electronics, being either an AFM (Atomic Force Microscope) controller or a direct interface using an oscilloscope and, if desired, a driving function generator, will be

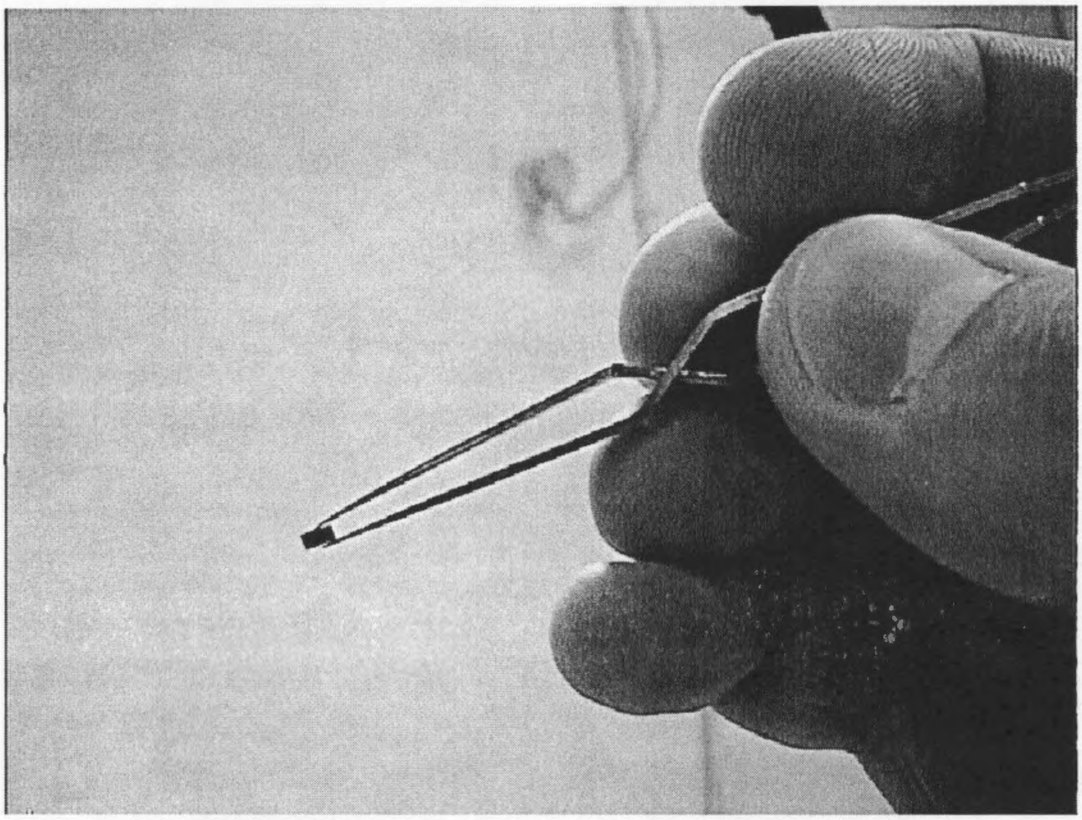

Figure IV-1. Handling Microcantilevers

One or more cantilevers are attached to a substrate that is large enough to be manipulated by hand, usually with tweezers. A typical substrate is shown above held in the tweezers.

explained, as will different issues and problems that can arise, particularly with respect to different methods of control.

4.2. Optical Cantilever Control Using a Head

There are several different ways of controlling a cantilever. Typically, cantilevers are mounted in a head that can either be part of a complete system assembly or run independently with some simple equipment. A head is essentially a large mounting bracket that contains equipment for moving and exciting cantilevers and for measuring cantilever deflection. (Other information, such as the frequency of vibration, is extracted from the deflection data.) The cantilever itself sits in a cantilever mount that in turn fits into the head. (See Figure IV-2 for a diagram of a cantilever mount.) The cantilever mount serves several functions such as holding the cantilever in approximately the correct position relative to the rest of the head. (Some variation in position does occur; thus the laser used for deflection detection can be shifted slightly to allow correct focusing on the cantilever, as will be discussed shortly.) Applying an AC signal of variable frequency to piezoelectric crystals residing within the cantilever mount produces excitation of the cantilever. These crystals allow the microcantilever to be vibrated at a particular frequency and amplitude.

Cantilever Mount

Mount From Above

Mount From Front

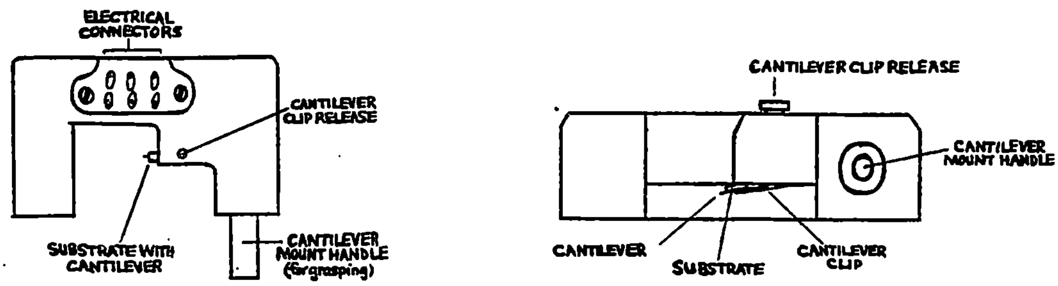

Figure IV-2. Cantilever Mount

A substrate with attached cantilevers is inserted into a cantilever mount which holds the substrate and cantilevers steady and can vibrate them at a particular frequency and amplitude if desired, using a built-in piezo crystal. The cantilever mount is inserted into an AFM head.

While deflection data can be generated using piezo crystals, the deflection measurement system that is typically used is a bit different. A laser beam is aimed at the end of a cantilever and bounces off. A Position Sensitive Detector (PSD) is positioned in the path of the deflected beam. When a cantilever bends, the angle between the surface and the incoming laser beam changes causing the angle of deflection of the beam to shift. This change in angle results in the shifting of the laser across the PSD. This movement is detected by the PSD and the output signal is adjusted accordingly (the level of the output signal varies approximately linearly with the deflection of the cantilever). (See Figure IV-3 for the layout of the optical detection system.) In order for this system to work, the laser must be aimed sufficiently well such that enough light is reflected off into the PSD and the PSD must be positioned such that approximately half of the laser light is incident on an upper detector and half on a lower detector. (If too much laser light strikes one side of the PSD relative to the other, the relationship between deflection and change in signal breaks down.) A digital voltmeter on the AFM head registers the sum of the light intensity falling on both sides of the PSD - A+B - and a measure of the difference in light intensity on the two sides - (A-B)/(A+B) - that is used for determining cantilever deflection. (See Figure IV-4 for a diagram of a head and Figure IV-5 for a picture of one.) In the preceding equations, A and B are the

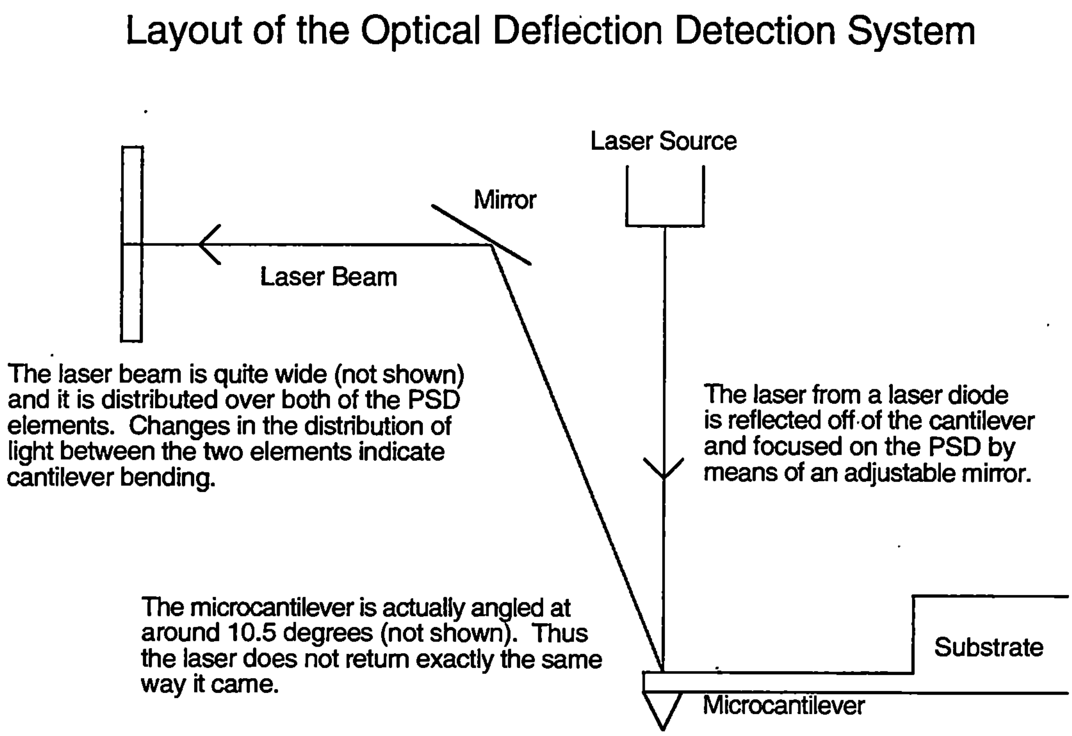

Figure IV-3. Optical Deflection Detection System Layout

The most common method of detecting cantilever motion is the optical system, which involves bouncing a laser off the end of a microcantilever. This is a convenient method because an AFM head already contains most of the electronics and control hardware that is needed.

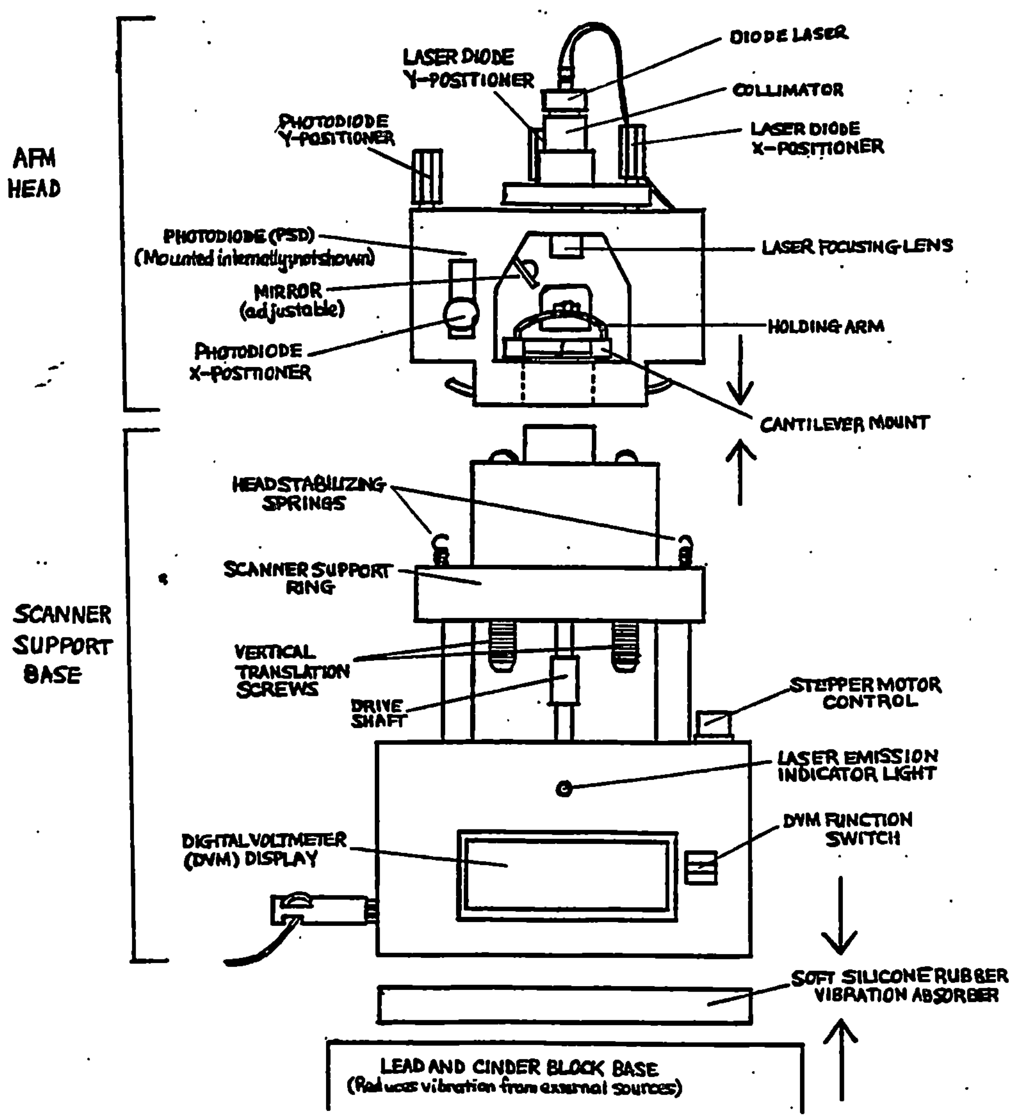

Figure IV-4. AFM Head Diagram

The positioners on top allow that laser to be aimed at the cantilever tip and the reflected beam to be aimed at the center of the PSD (Position Sensitive Detector). The Digital Voltmeter (DVM) display switches between two readings, one being the sum of the intensity of the beam falling on the two plates and the other being a measure of the skewness of the laser beam towards one side of the PSD or the other.

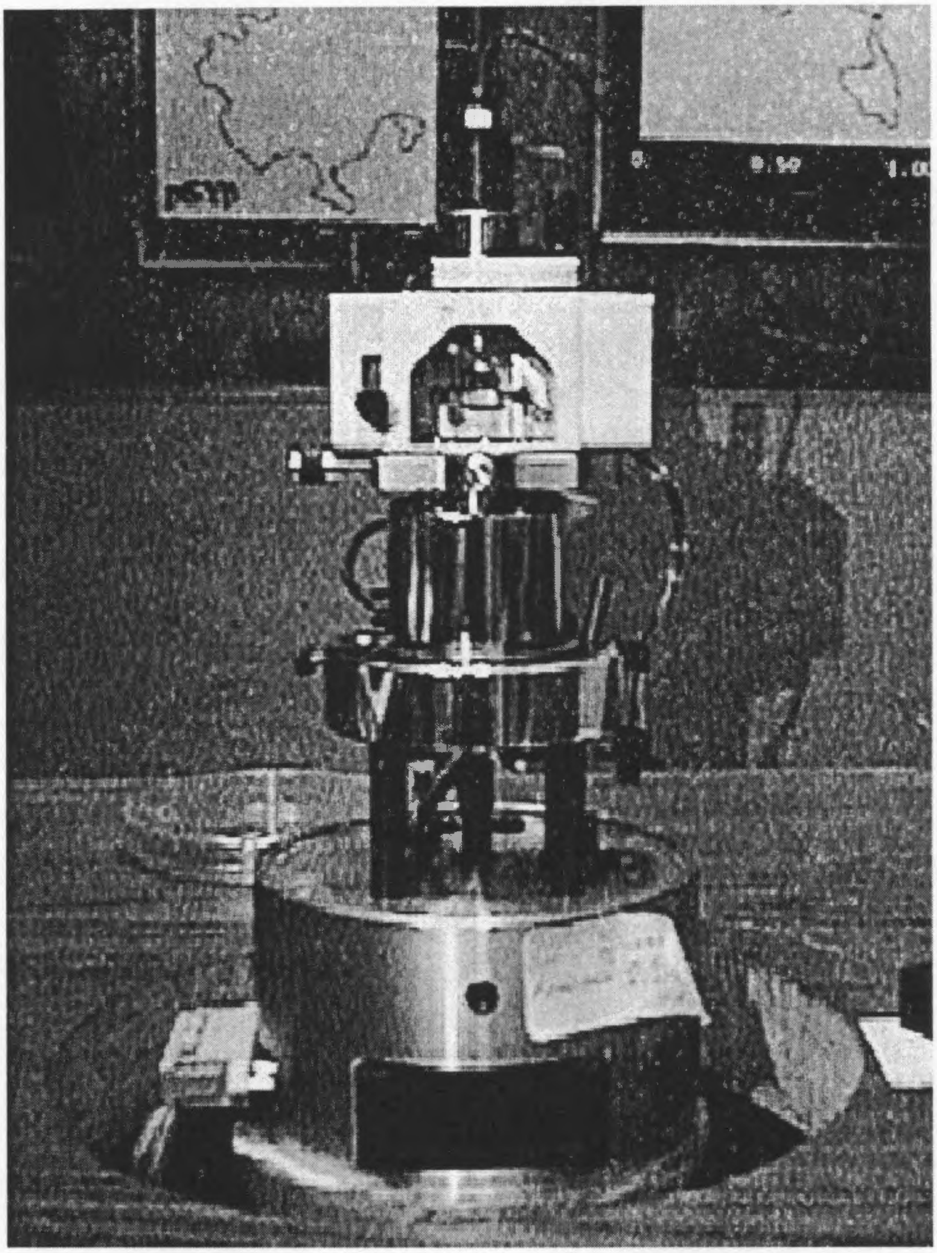

Figure IV-5. Photograph of an AFM Head

Most of the items shown in the diagram (Figure IV-4) can be distinguished in this photograph.

intensities of the light falling on the two PSD plates. The two signals are used during setup respectively for positioning the laser beam on the cantilever beam and aiming it at the center of the PSD.

This deflection measurement system is not perfect. One limitation is that the change in the angle of the cantilever is measured at the point where the laser strikes it and it is assumed that the change in angle is proportional to the change in deflection. In general this is true, although the proportionality constant can change depending on the geometry of the cantilever, the angle at which the cantilever is mounted, and the point on the cantilever where the laser beam strikes it. In order to get an accurate determination of the value of dx/dV (that is, the change in cantilever deflection per unit change in the output signal) it is necessary to perform a force calibration using a force calibration curve. This will be discussed later in greater detail (see pp. 51-52).

4.2 Other Methods of Deflection Detection

There are other methods available for measuring cantilever deflection. The second most common technique is the piezoresistive in which a cantilever is made in part or in whole of a piezoresistive material. The deflection of a cantilever compresses or stretches the piezo crystal producing a voltage across it which can be measured. For fairly small deflections the voltage produced is proportional to the bending of the lever. One disadvantage is that such cantilevers must be at least 10 μm thick and thus have rather high spring constants. Another is that thick cantilevers tend to be relatively insensitive. Beyond the optical and piezoresistive methods, most deflection measurement methods are fairly obscure. The capacitive method was investigated by several different groups but has been largely discarded, although the Molecular Imaging Group (MIG) at Oak Ridge National Laboratory (ORNL) is working on arrays of cantilevers that will use the capacitive method. This technique works by measuring the capacitance between two plates, one being the cantilever itself and the other being a small conductive area set a short distance below the cantilever. As the distance between the plates changes due to the bending of the cantilever, the capacitance of the system changes. A big disadvantage of this system is that the charge on the cantilever can change due to some external effect (such as a radiation interaction) and the system can be fooled into reporting a deflection that did not in fact occur. There are a couple of other methods (interference, STM) that are not widely used at this time.

4.3. AFM Systems

AFM controllers are often used in cantilever sensing experiments. (See Figure IV-6 for a simplified diagram of an AFM control system layout.) AFM systems incorporate hardware and software for all kinds of imaging applications.

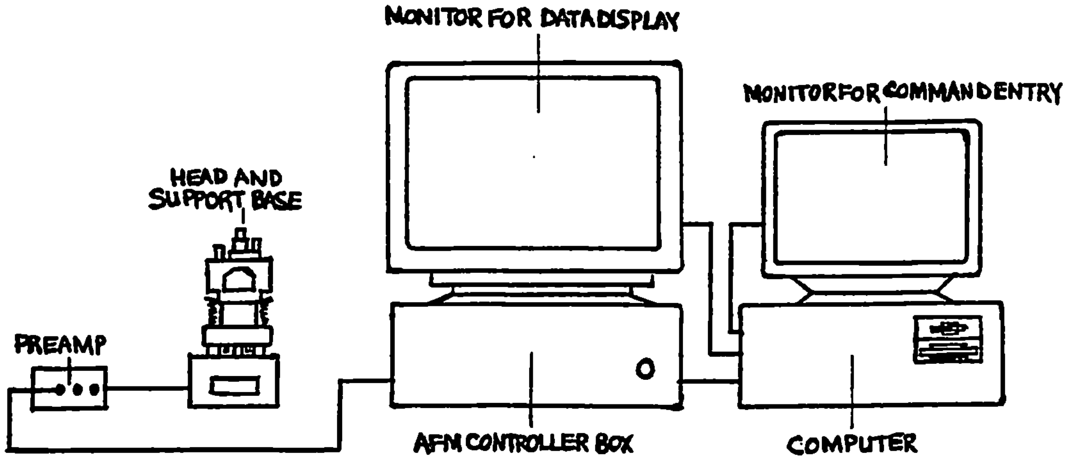

Figure IV-6. Simplified AFM Control System Layout

Many of these features are also useful for experiments in cantilever physics and in sensing and detection. For example, the Nanoscope series of AFM systems (the type I used in my research) are set up to easily "tune" cantilevers, that is to determine their harmonic frequencies by exciting them at different frequencies and finding the frequency at which the greatest amplitude occurs. This feature is very useful in experiments in which a frequency shift is used to detect or measure something.

AFM control systems are fairly complex pieces of equipment, but their basic principles of operation are fairly straightforward. A cantilever head contains a cantilever held in place above a sample surface. The system can move the sample surface closer or farther away from the cantilever tip and can vibrate the cantilever at a frequency and strength specified by using piezoelectric crystals mounted under the sample and in the cantilever mount. Large motions can be accomplished using a combination of computer controlled and user operated screws attached to a vertical translator. As discussed earlier, deflection is most often detected and measured using a laser-based optical system. Normally, the item or surface of interest is placed on the sample mount and the cantilever is brought into contact with it. The sample being investigated does not necessarily have to be a solid; the user might want to investigate cantilever behavior in a liquid. In this case, the cantilever might be lowered into a small puddle (formed by several drops of water) or an open-topped container of liquid sitting on the top of the sample mount. Cantilevers do not necessarily have to be operated near a surface using a controller. They may be operated free in the air. This permits investigation of cantilever physics and the effects of different gases on cantilever behavior. When no surface is present, the controller can do all the things that it can normally do to the cantilever except for those requiring the presence of a surface (such as a force calibration curve).

Controllers provide an interface between the AFM software on a computer and the AFM head (containing the microcantilever). Controllers take the commands from the software and the information coming in from the head, process it, and send out the necessary signals to the head to accomplish the desired result (such as moving the cantilever or vibrating it at a particular frequency and amplitude). Some functions, such as exciting the cantilever via the piezo in the sample mount, can be accomplished without the use of the controller box. Other functions, such as moving the sample mount relative to the cantilever, cannot be done without the controller and software (i.e. a complete AFM system). Of course, the cantilever mount can be moved up and down relative to the sample (surface) mount in coarse increments (microns), but it cannot approach the angstrom-level movements possible when using the controller and software. This makes surface or near-surface work impossible without the controller.

Generally, controllers are used for surface or near-surface work. The user manually positions the cantilever close to the surface (the surface mount can be raised and lowered by hand) and the system controls the final approach by the cantilever to the surface. Contact is assumed when the cantilever deflection signal increases beyond a certain level. The deflection comes as a result of the cantilever being pushed against the surface; the tip either no longer moves (for rigid samples) or does not move very much (for softer samples) when the base is moved (as the tip is now held in place by pressure and friction). This determination is made by setting a specific amount of cantilever deflection as indicated by the PSD to be a trigger to indicate that tip motion has stopped or almost stopped and the movement of the base of the cantilever is producing deflection. Upon making contact with a surface, a cantilever can then be retracted to a user-specified distance from the surface if desired. This is useful for studying electric fields around an object, for example.

False contacts can be a major problem when using particularly reflective samples, especially if they are flat. The laser beam in the Nanoscope's optical detection and measurement system is not very well focused. Invariably, a significant portion of the laser footprint misses the cantilever and strikes the surface below. Generally this secondary beam is reflected in approximately the same direction as the main beam from the cantilever. (In some circumstances this beam can go directly into the PSD.) As the cantilever is moved, the signal beam from the cantilever and the secondary beam from the surface can interact, going back and forth between constructive and destructive interference, causing the optical signal intensity to fluctuate. Sufficiently large reductions in the intensity can fool the system into registering large deflections (substantial deflection of the cantilever leads to a reduction in the signal as the laser's footprint begins to move off the PSD) and thus reporting contact with the surface. This can be a very serious problem in certain situations. Since the system ceases to move the cantilever closer to the sample when it registers contact, this can lead to the necessity of repeatedly attempting to engage the surface and resetting the deflection measurement system each time. Several options for mitigating this effect exist. If the surface is flat, it can be placed at a shallow angle so that the surface-deflected beam will not interact with the signal beam as much. Another possibility is to coat the surface with a non-reflective paint-on or spray-on polymer such as Aerodag G. If one desires to study the surface itself, the second option is obviously not viable. The second method is useful, however, for our application - charge-based radiation detection -if the coating in question is sufficiently conductive. This method was employed in some of the experiments described herein and will be discussed again later.

4.4. Direct Interface

Cantilevers can be controlled by direct interface with the head. For example, one method is to use an output from a signal generator to drive the piezo crystal, allowing the cantilever to be vibrated. The signal output representing deflection might be fed into a digitizing oscilloscope to monitor the microcantilever's motion over time or a lock-in amplifier to find the frequency and measure any shifts. (See Figure IV-7 for a direct of direct head interface.) Positioning a cantilever accurately near a surface is not really possible with this system, so a controller must be used to put the cantilever in place. Once it is in place, control of the system can be switched from the AFM controller to the signal generator or other source of a driving function. It is not really possible to move the cantilever closer to or farther away from the surface at this point unless control is switched back to the controller which can be a problem if an experiment is running. This system does have several important advantages over using an AFM controller in many situations, however. One is the ability to apply a wide variety of driving functions to a cantilever that an AFM controller is not designed to provide. Another is that the direct readout can give a clearer picture of what is actually going on as

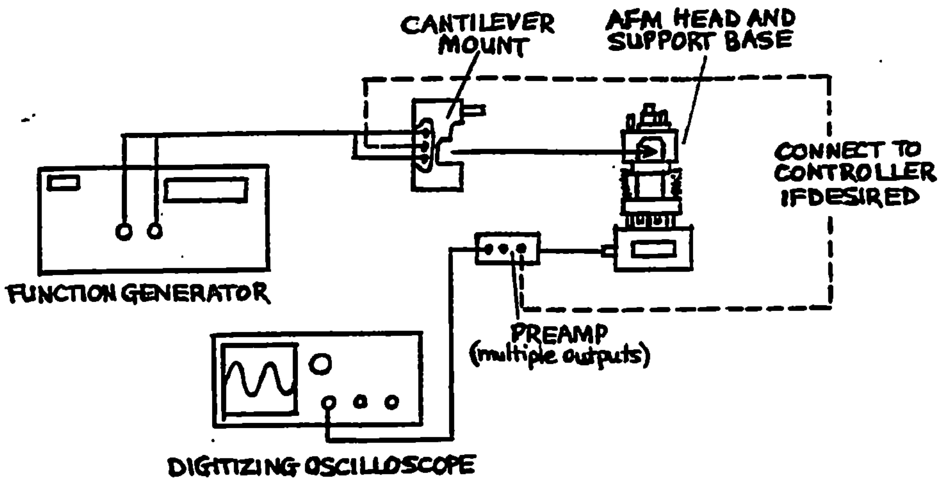

Figure IV-7. Direct AFM Head Interface

This is carried out using a driving function source (such as a function generator) and some kind of readout electronics (such as a digitizing oscilloscope). Depending on what is connected directly and how, the AFM controller box and associated equipment can usually be connected if desired in order to accomplish such things as changing the separation distance between the cantilever and an adjacent surface.

AFM systems typically present data in a processed form that is dependent on certain assumptions that are made being true.

Chapter 5

Experimental Methods and Procedures

5.1. Introduction

This chapter discusses the methods and procedures used in the alpha particle detection experiments. The first issue covered will be the type of alpha source used and the design of the detector system used to detect it. Issues related to detector design will be considered (although the results of experiments designed to determine the degree to which these issues influence detector capability will be left until later) as will the methods employed for cantilever control and response analysis. The means used for environmental control, a necessity, will be detailed. Finally, experiments done apart from the alpha detection experiments in order to characterize cantilever response and its sensitivity to various factors will be described.

5.2. Alpha Detection: Methods, Setup, and Considerations

5.2.1. Introduction

It was decided to attempt charged particle (specifically, alpha) detection by detecting and measuring the accumulation of charge on a collecting plate resulting from exposure to a charged particle beam. The principle is that an electric field acting on a microcantilever will affect the behavior of that microcantilever in ways that can be easily measured. An uncharged semiconducting or insulating cantilever will tend to become more positive on one side and more negative on the other. When the field is non-uniform, that is that the strength varies with position, the effective spring constant of the cantilever will change. This change will cause the cantilever to oscillate at a different frequency and damp out at a different rate[12]. These changes are dependent on the degree of variation in the electric field, which in turn is related to the amount of charge producing the field and its distribution in space. The idea, then, is that the degree of variation in the cantilever's behavior can be related back to the charge accumulated on the collecting plate, the charged particle flux rate at the collecting plate, and ultimately the strength of the sources in the immediate vicinity.

5.2.2. Detector Design

Several different experiments were conducted using a couple of different sources. Initially a 4.92 μCi (2Π surface activity) Am-241 alpha source purloined from a smoke detector provided the charged particle stream. The source was mounted on a small circular metal plate glued to the bottom of a short piece of metal piping. At the top of the metal pipe another circular metal plate was affixed (the upper or top plate), perpendicular to the direction of the metal pipe and parallel to the plate at the bottom.

As will be discussed later, the alpha beam was incident on a 1/16" diameter steel ball bearing. The target area of this ball bearing for incoming alpha particles was approximately 0.025 cm^2. The 4.92 μCi alpha source produced approximately 1.82×10^5 alphas per second. The source to ball bearing distance was approximately 0.9 cm. This meant that, without taking into account any other considerations, approximately 0.49% of the alphas given off or 890 alphas per second hit the collector plate. In actual fact, the number of alphas striking the collector plate was lower than this. The reason is that not the entire target zone was in fact exposed to the alpha beam. The mica holder plate, while thin, did take up some area around the middle of the ball bearing and covered up a small portion of the lower hemisphere of the ball bearing. Of greater significance than this was the glue used around the edges of the hole in the mica holder piece where it contacted the ball bearing. The layer of dried glue, while thin, was in fact thick enough to stop alphas impinging on it from passing through and reaching the collector plate. For these reasons, it is probably more reasonable to estimate the number of alphas entering the collector plate at 750 per second. For all intents and purposes, all alphas entering the collector plate can be assumed to have stayed there.

Two very strong rare earth magnets were mounted inside the pipe section facing each other, and generating a magnetic field of strength approximately 1 Tesla. (For a diagram of the setup of this experiment, see Figure V-1.) Their purpose was to deflect the electron beam coming from the Am-241 source. (Am-241 is primarily an alpha emitter but it also produces some beta particles.) Electrons are more easily deflected than alpha particles because of their much lighter mass. It was desirable that no electrons reach the target as their negative charge would partially cancel out the expected accumulating positive charge from the alphas and reduce the effect on the microcantilever.

The top plate had a small hole drilled in the center to allow the alphas to pass through and reach the collector plate. The purpose of the upper plate was to provide a solid base for items above it and to allow easy calculation of the number of charged particles incident on the detecting element by using a filter (the hole in the center of the plate) of known size.

The collecting plate sat directly above the hole in the upper plate. Initially, when Ari-David Brown ran the experiment,[13] he had a roughened piece of gold mounted above the hole. A piece of mica with a hole in it to go over the hole in the upper plate sat between the gold and the upper plate to prevent the gold from discharging. I later mounted a 1/16" ball bearing in a hole drilled in a piece of mica

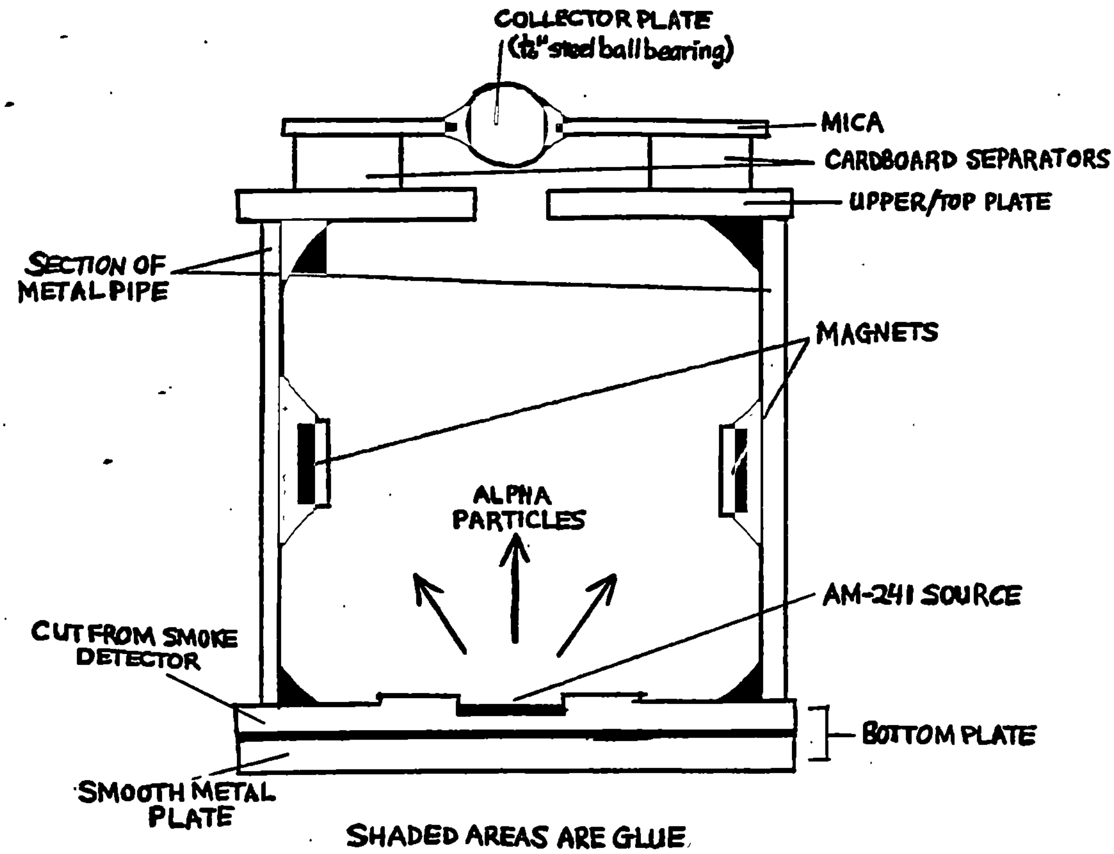

Figure V-1. Diagram of Source-Detector Arrangement

A cutaway diagram of the source-detector arrangement used in the alpha particle detection experiments. In an experiment, a microcantilever would be positioned a short distance (nanometers) above the collector plate.

and elevated it a short distance above the upper plate using some cardboard. Although I replaced the ball bearing a couple of times, this remained the basic system for both alpha detection and the later voltage experiments.

5.2.3. Special Considerations

There are several important considerations, besides those previously mentioned, when constructing such a system. One critical aspect is charge collection. The possibility of the collector plate becoming charged is not the only pitfall to be avoided. Charge often does not evenly distribute itself over the surface of a collecting plate; instead it tends to concentrate at places where the radius of curvature of the plate's surface is small, such as points and rough areas. This can be problematic for charge detection. The electric field will be strongest around the areas of greatest charge concentration. Points also tend to have steeper field gradients around them than straight sections for reasons related to simple geometry. Given these facts, it can be seen that the greatest response will be obtained around rough areas of surface or points. Before running actual experiments, the extent to which possible unevenness of charge distribution would affect microcantilever response was unknown and considered a potentially serious problem.

Using a ball bearing as a collector plate was appealing in that it ought to have an even distribution of charge over its surface, eliminating questions about the significance of roughness and point effects. In practice an examination of the surface of the ball bearing showed that it did have areas where it was especially rough and especially smooth. The effect of the variation of roughness in this case was probably not very great, however.

Another complicating factor is the electron sprays induced by incoming alphas. These can cause electrons to stream out of the collector plate and into the insulating material used to hold the plate in place. These electrons can become stuck in traps and, once there, cause positive charges in the collection plate to migrate to areas of the plate immediately adjacent to areas of trapped electrons in the collection plate. (This possibility was originally suggested by Dr. R. H. Ritchie.[14]) (These areas are by definition inaccessible to a microcantilever, as they are unexposed.) This, of course, would mean that less charge would be present elsewhere on the collector plate, the electric field would be weaker, and the cantilever response would be diminished. (See Figure V-2 for an illustration of this effect.) It was a good idea to keep a removable shield of some sort between the source and the collector plate when not using the system in order to prevent weeks and weeks of continuous alpha bombardment from filling all of the electron traps.

A method for achieving charge drain was necessary. Exposure of the collector plate to an alpha beam would result in the build up of charge on the plate

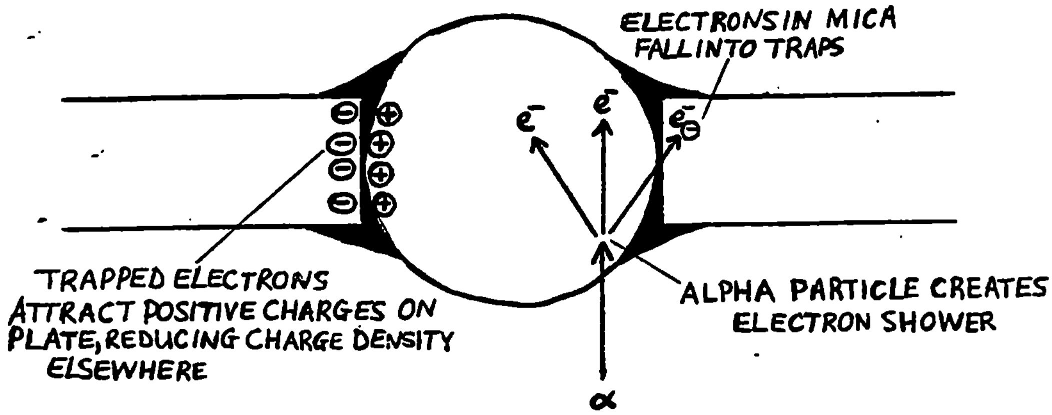

Figure V-2. Electron Spray Effects

An illustration of how constant radiation bombardment was believed to cause the filling of electron traps in the areas of the mica adjacent to the collector plate. Electron sprays produced by slowing down alpha particles resulted in some electrons travelling into the mica and becoming caught in traps. Positive charges on the collector plate resulting from alpha irradiation of the plate tended to concentrate in areas adjacent to the negatively charged mica, reducing the charge concentration at the top of the collector plate, and thus the microcantilever detector response.

until equilibrium was achieved when loss mechanisms cancelled out the gain from incoming alphas. At what point this would occur was unknown. If the experiment were to be halted, it might take a considerable period of time for the built up charge to be lost. A method for quick removal of any charge on the collector plate was required. There were numerous possibilities for grounding methods. One was a direct connection wherein a wire would be run from the collecting plate to ground with the switch opening and closing the connection. A disadvantage inherent to this was that some portion of the wire must lie between the collector plate and the switch. This section would act as part of the collector plate and the concentration of charge on the plate itself would be diluted. Another possibility was to use a retractable conductive prod, such as a wire, that could temporarily make mechanical contact with the collection plate. A potential problem with this was the possibility of shaking the collector plate and thus affecting the cantilever. Although cantilevers themselves are very rugged, they can be highly sensitive to mechanical vibration in certain situations such as in the system described. Since the cantilever and the collector plate were mounted on different parts of the head, vibrations induced directly into the collector plate would not produce vibrations of any significance in the cantilever. The result would be that rather than the cantilever and surface vibrating in sync with each other the cantilever would tend to be beaten against the surface. Use of the prod method requires that contact to be made very gently.

5.3. Measurement of Detector Response

The actual method for alpha detection used in this set of experiments was to look for changes in the damping rate and resonance frequency of a vibrating microcantilever resulting from the presence of an electric field produced by the build up of charge on a collector plate. It will be recalled that an uncharged cantilever placed near the surface of the collector plate ought in theory to damp out more quickly and exhibit a drop in resonance frequency as the charge on the plate grows. The first alpha detection experiments used a piece of roughened gold as a collector plate. When repeating the experiment with a ball bearing, the force calibration curve method originally suggested by Ari-David Brown[12] was used to excite the cantilever momentarily while in close proximity to the surface. The oscillations of the microcantilever were recorded instrumentally while the cantilever's oscillatory motions died down and the frequency and damping data extracted.

Force calibration curves were used to calibrate dx/dV, the distance of deflection of the cantilever per unit change in output signal amplitude. As discussed earlier, this value can change dramatically from case to case, requiring an accurate determination of it whenever an experimental setup undergoes substantial changes. The basic principle behind a force calibration curve is that a cantilever can be deflected a known distance, the change in the output signal measured, and the change in output signal per change in deflection determined. Bringing the sample mount (or sample, such as a sheet of cleaved mica) into contact with the cantilever (by applying a voltage to the piezo that the mount sits on) and then pushing it a set distance accomplishes this. Once this has been done, the sample mount is pulled away until it is no longer in contact with the cantilever.

In theory this is very simple. In practice it is anything but. The cantilever is sufficiently small that many effects that are essentially unobservable under normal circumstances by virtue of being too weak or too short-range in extent for us to see suddenly become important. One of the most important is that water on the surface of the sample and on the tip of the cantilever forms a bridge between the cantilever tip and the sample surface (in this case the collector plate) when the tip is in very close proximity to the surface. (Once the bridge has been formed it is capable of being stretched out and persisting even when the tip and sample have been separated by a couple of hundred nanometers. See Figure V-3 for a diagram of this.) As the cantilever is pulled away, the water acts a bit like glue and holds the

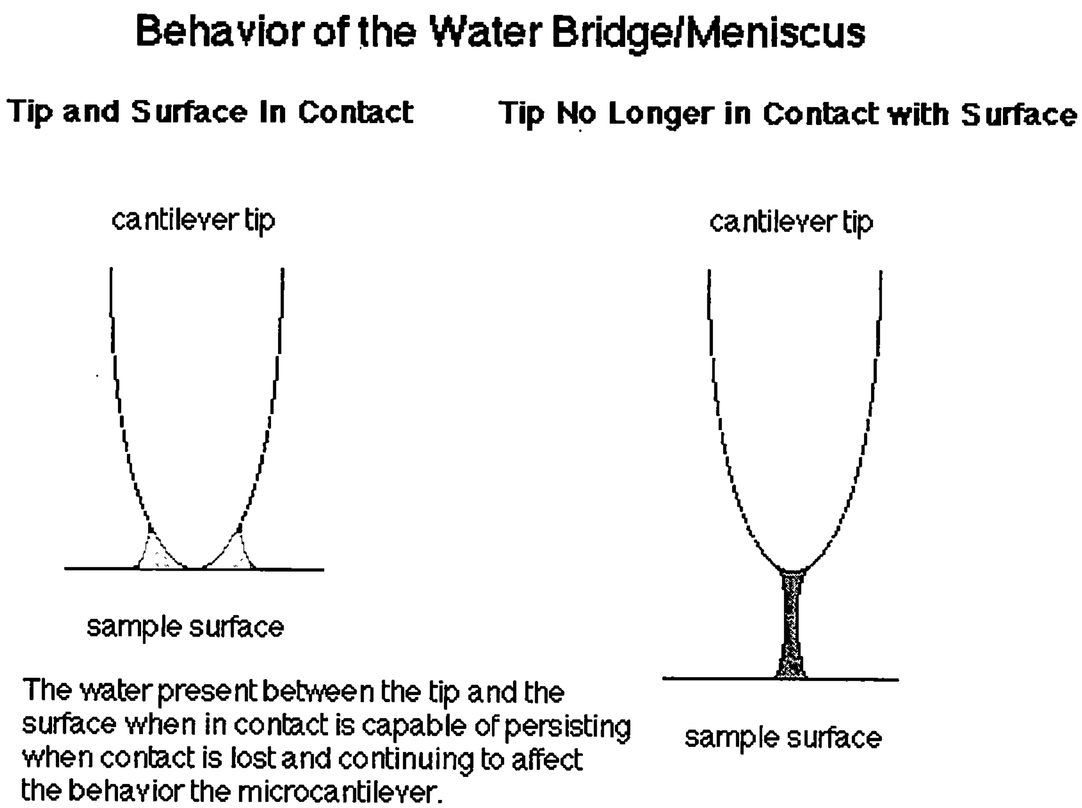

Figure V-3. Water Bridge Behavior when Stretched

two surfaces together. Of necessity, the cantilever bends as the mount is being pulled away while the tip stays still. Eventually the tip breaks free of the surface. This happens when the restoring force of the cantilever (the cantilever acts like a spring and as it bends its restoring force increases in linear proportion to its deflection) becomes stronger than the forces holding the cantilever tip to the surface (primarily capillary and van der Waals). When the tip breaks free, it snaps away suddenly and is excited, going into harmonic motion. (See Figure V-4 for a diagram of this.) This motion is very transient, damping out within a fraction of a second. (See Figure V-5 for an example of such a breakaway curve.) As will be seen, there are several important effects influencing the cantilever's behavior immediately preceding and subsequent to breakaway.

The transient produced by breakaway during a force calibration curve is extremely important to us. The method of detection used requires the excitation of the microcantilever in close proximity to the surface. Different methods exist for doing this. The force calibration curve method proved to be straightforward and yielded the desired results; thus it was used to produce transients from which frequency and damping data could be extracted. The primary purpose of this project is to explain how these transients are affected by radiation and by environmental factors, with a view to determining the potential capabilities of

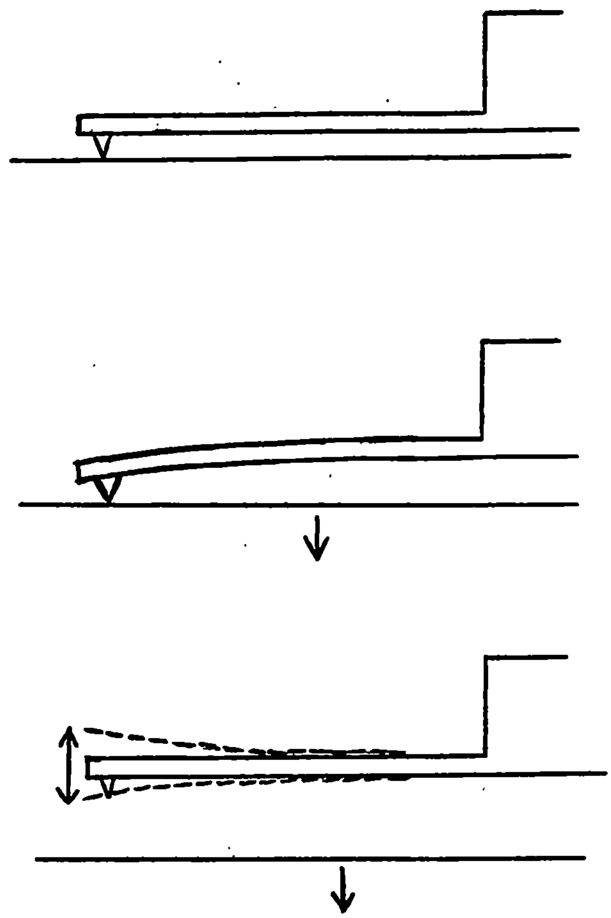

Figure V-4. Cantilever Motion Before, During, and After Breakaway

Usually a cantilever in contact with a surface is initially in an undeflected state. As the surface is withdrawn, the cantilever tip bends with the surface until the restoring force of the cantilever exceeds the tip-surface attractive force, causing the cantilever tip to break free of the surface. After breakaway, the cantilever oscillates until it damps out.

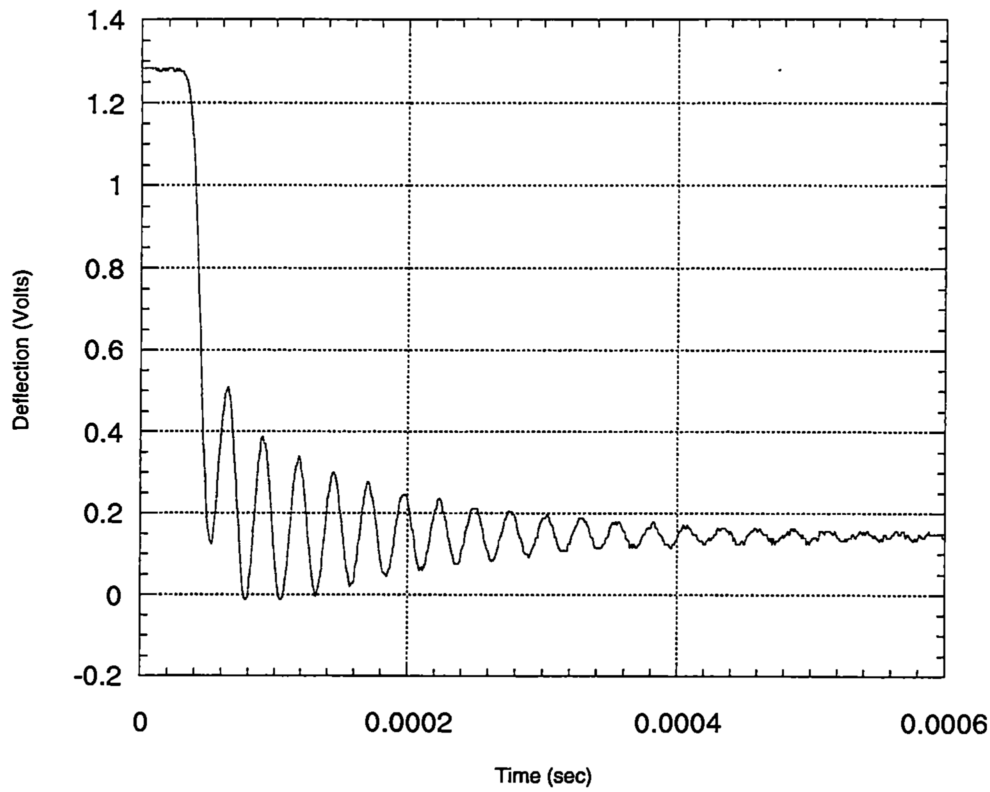

Figure V-5. Typical Breakaway Curve

At the top left the cantilever was in contact with the surface (essentially glued to it by the tip-surface attractive force) and was slowly deflecting as the surface was moved (making it still for all intents and purposes over this time scale). The spot where the trace suddenly began to drop represents the point at which the restoring force of the cantilever exceeded the tip-sample attractive force and breakaway occurred. Immediately following this, the cantilever became excited as a result of being released from an extended position and went into harmonic oscillation that gradually damped out.

microcantilever-based radiation detectors.

5.4. Practical Aspects of Alpha Detection

5.4.1. Practical Aspects of Detector Construction

The very first alpha detection experiments performed by Ari-David Brown and myself used a piece of roughened gold as the collector plate.[15] The plate was neutralized using a grounding wire and switch arrangement. The roughness of the gold was sufficient to prevent charge migration to the wire (when ungrounded) from becoming too much of a problem.

When I set out later to repeat the experiment and determine what physical principles were at work, I found it necessary to replace the gold with some other type of collector plate, as I could not align the cantilever correctly on the gold. The problem was a result of the cantilever being fixed in one place by the cantilever mount and the collector plate having a small range over which it could be positioned due to being surrounded by the head. What this meant was that the cantilever could only be positioned in a very few number of ways relative to the collector plate. Since part of the collector plate was excessively sloped, part was inaccessible due to the grounding wire coming out the top, and part of the gold was covered over with the solder used to hold the wire in place, there were only very limited areas suitable as cantilever target zones. Positioning the cantilever against one of these areas of surface turned out to be difficult. Due to the small range of motion possible for repositioning the collector plate, it was difficult to align the cantilever above an area of suitable surface. Making contact with such a surface was even worse as various surface features, such as elevated areas, confounded attempts to lower the cantilever to the desired position. After wrecking a number of cantilevers, it was determined that it would be necessary to replace the collector plate with one that did not have such problems. (This turned out to be a blessing in disguise because it eventually led to the use of a small ball bearing, an object possessing very predictable properties and being very consistent in its characteristics over its surface.)

Initially a square piece of silver foil with a grounding wire/switch arrangement was used. The silver foil was highly reflective and it was not possible to make contact with the surface despite repeated attempts. Another attempt was made to solve this problem by applying Aerodag G, a spray-on graphite-based conductive and non-reflective coating. The expectation was that this would solve the reflection problem without impeding charge accumulation in the targeted area. In the end this design was abandoned for several reasons. It was realized that three potentially major problems existed. Firstly, the charge would tend to concentrate at the edge of the foil (although it was not known at the time how significant this effect would or would not be). Secondly, the grounding wire was by necessity quite large and would collect a significant amount of charge. (These first two problems would work to reduce detector response.) Finally, the surface reflectivity was still problematic and causing much noise in the signal (although substantially reduced).

After thinking this through, it was determined that the best method given what equipment was immediately available was to use a ball bearing as a collector plate. In theory, charge would be evenly distributed over the surface eliminating guesses about the degree to which edges and other areas with small radii of curvatures would affect charge distribution. A spherical collector plate would be less susceptible to reflectivity problems; the cantilever could be positioned on a shallow-angled area of the surface and the secondary beam would be reflected in a different direction from the signal beam, largely avoiding interaction with the signal beam and missing the PSD. (Depending on the geometry it is quite possible for the secondary beam to be reflected into the PSD, producing a false signal.) It would also be relatively easy to set up a prod-based charge drainage system for this setup.

A ball bearing-based charge collection system (discussed earlier) was constructed with these ideas in mind. To hold the ball bearing in place a tiny hole was punched in a small piece of mica. The hole was made to be such a size that the ball bearing would be held in it fairly tightly by friction (half the ball bearing extruded from one side and half from the other). To make sure the ball bearing would not move a small amount of glue was put around the ball bearing where it stuck out of the mica on the upper side of the piece of mica (although some glue inevitably flowed through to the lower side). As mentioned earlier, the mica was mounted on small pieces of cardboard that in turn sat on top of the top plate. This kept the ball bearing from making contact with the (metal) top plate and thus grounding itself. The mica was positioned such that the ball bearing target was directly above the hole in the top plate through which the alpha beam streamed.

Charge drainage was accomplished using a prod-based system. Several designs were tried before settling on one specific method. The first design used a long sliver of sheet metal bent such that it formed a kind of spring that could be manually pushed from above down onto the ball bearing. The contact end was cut to form a pointed end and thus minimize contact area. There were several problems with this system. One was that the spring had to sit above the ball bearing, which was a problem given the limited space within the head (the cantilever, sample, and any attached equipment sit in a hollow space inside the head). Another was that when contact was obtained, the ball bearing was made to vibrate excessively (in part this was because the spring constant of the spring was quite high and considerable force had to applied, making it difficult to not apply too much by accident).

The second method used a solenoid to extend and retract a grounding wire. Applying a voltage to the solenoid would cause it to extend a metal bar to the end of which was attached to a grounding wire that continued out from the bar for several inches. The wire would be pushed into making contact with the ball bearing, grounding it. Removing the voltage from the solenoid would cause the metal bar, and thus the grounding wire, to retract. Because of the speed at which the wire hit the ball bearing (the solenoid would extend and retract it very quickly), substantial vibrations would result, making the design impractical.

It was finally settled that a grounding wire attached to a horizontal translator would be used. Turning a screw on the translator would have the effect of moving it towards or away from the ball bearing and, of course, bringing the grounding wire into or out of contact with the ball bearing. (See Figure V-6.) By turning the translator's screw slowly, the wire could make or break contact gently without creating excessive vibrations. In addition, very thin wire was used that gently curved along its length. This meant that it would act as a spring and easily bend at the end. As a result, if it were to hit the ball bearing fairly hard it could absorb a lot of the energy and vibration would be reduced. This scheme was found to be fairly effective.

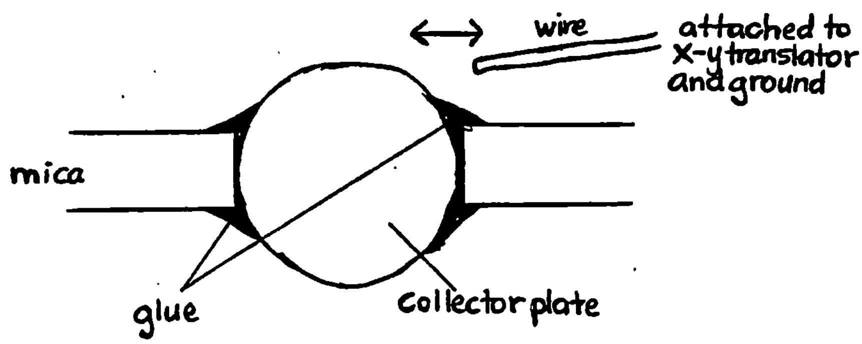

Figure V-6. Grounding Wire Operation

A thin, flexible wire connected to an x-y translator and ground was gently moved back and forth into and out of contact with the collector plate, alternatively grounding and electrically isolating it.

5.4.2. Cantilever Control and Measurement

For most experiments, Digital Instruments triangular silicon nitride cantilevers of length 100 μm, width 40 μm, thickness 0.7 μm, spring constant 0.24 N/m, and fundamental frequency approximately 16 kHz were used. Some cantilevers of slightly different thickness and frequency around 40 kHz were also used. Cantilevers of this design had been used for other experiments and it was felt that it would make sense to use that with which the experimenter was most familiar. In addition, said cantilevers are quite wide as far as cantilevers go and would be less likely to suffer damage if accidentally struck by the grounding wire, dropped, etc.

Cantilever control and measurement was accomplished using both the Nanoscope II control system and the direct interface of a digitizing oscilloscope (Tektronix TDS 430A XL Two Channel Digitizing Oscilloscope) with the head. The Nanoscope II was used to position the cantilever on the surface and to generate force calibration curves. The oscilloscope was used to record the motions of a particular cantilever as it broke contact with the surface and oscillated. Oscillations were generally recorded at a sample rate of 1 or 2.5 Megasamples/sec. A trigger level allowed recording to start immediately prior to breakaway and thus avoid collecting many unnecessary data points. Generally 500 or 1000 points were recorded; usually 500 when operating at 1 Megasample/sec and 1000 when at 2.5

Megasamples/sec. After a data set was recorded, it could be easily transferred by floppy disk to a computer for analysis.

5.4.3. Experimental Procedures and Data Analysis

The procedure for carrying out an experiment was as follows. The grounding wire was initially put in contact with the ball bearing to ensure that no charge was present on the collector plate. The cantilever would be brought into contact with the surface and the machine made to indefinitely perform force calibrations (by running force calibration curves). The settings (sample rate, record length, trigger value, etc) on the digitizing oscilloscope would be adjusted to focus in on the breakaway curve. The grounding wire would be withdrawn from the ball bearing by turning the screw on the translator. This would be done as gently as possible. Vibrating the ball bearing would result in the breakaway curve shifting on the oscilloscope screen. Typically, the trigger value would have to be readjusted. Attempts were made to avoid this as much as possible since readjustment took time and could result in not being able to stop the experiment (due to it not being ready) when the time came to do so. After the specified time of exposure (perhaps 10 seconds or a minute), the oscilloscope would be stopped, meaning that the most recent force calibration curve would be displayed frozen (i.e. not be updated anymore) on the screen. This curve would be saved to disk, eventually to be transferred to a computer for analysis. The grounding wire would be reapplied and the experiment would be repeated as many times as desired.

Raw data, in the form of Force Calibration Curves, was processed using several different software packages. Unfortunately there was no simple method for doing this. Even rudimentary analysis required considerable effort for just a single data point. Analysis was performed using three software packages for the Macintosh: KaleidaGraph (Synergy Software, Reading, PA), Excel (Microsoft, Redmond, Washington), and SigmaPlot (SPSS, Chicago, IL). Of these, KaleidaGraph was the most useful.

In analyzing the data, two important parameters needed to be determined for each data point: the damping rate and the frequency. It was expected that both would vary in the presence of an electric field and that the variation in one ought to be predictable from the variation in the other as the same factors cause both to change. Determining the correct value for each parameter was not a simple process given the tools available. In theory, it ought to be simple to fit an equation to a force calibration curve using a curve fit in SigmaPlot. Indeed, this is pretty straightforward to do. The problem lies in the fact that, while the fit obtained is very good, there is considerable uncertainty in the values of the individual parameters, in fact unacceptably so in the case of the damping rate. The frequency value was also more uncertain than would be wished but as a superior method for determining the frequency was not found, frequency data was taken from SigmaPlot curve fits.

The most accurate method for determining damping rates was to find the coordinates of individual peaks on the force calibration curve (i.e. where the cantilever was maximally extended during its oscillations) and then fit an exponential decay curve to them. This required some discretion in the choice of points. To start with, it was necessary to select the individual sample points that made up the upper part of an individual peak. Around the end of a peak the curve takes on an approximately parabolic shape. A parabolic fit would be made and the point of maximal extension (both magnitude of displacement from the zero deflection point and the time at which this occurred) would be found. This method worked quite well for peaks that were clearly defined. As noise in the signal became appreciable in size relative to the signal, the accuracy of results deteriorated. Once fits were made for as many peaks as could be accurately evaluated, an exponential decay curve was fit to the points. Of the peak points that could be distinguished, not all could be used; the first few points had to be ignored (see Chapters 6 and 7). Another effect was at work besides damping that shifted the points, making them unsuitable for inclusion in the exponential decay curve.

5.5. Atmospheric Control

Atmospheric control was critical to a number of experiments, especially those that required specific relative humidities or immersion in helium, nitrogen, or other gases. A bell jar with holes for control wires to and from the head and gasflow through a pipe was used. (See Figure V-7 for a diagram of the bell jar system.) The bell jar was not completely airtight; a limited airflow through the cracks in the wire holes was possible. This was actually desirable in that inflowing air needed some way to escape so that pressure would not build up.

Several different sources of gas were used. Helium and nitrogen were run directly from gas cylinders. Dry air of approximately 0% RH (relative humidity) was run from an outlet in the laboratory. Splitting a dry air stream in two and running one stream through a bubbler allowed air to be produced at a desired humidity level by adjusting the relative proportion of air going through each channel. Air from the bubbler was at almost 100% RH. Humidity levels in the chamber (i.e. the inside of the bell jar) were monitored via a hygrometer whose probe was inserted into said chamber through a hole for that purpose in the top of the bell jar. The cracks between the probe and the inside of the hole were lightly covered over with putty. As stated earlier, it was desirable that some air be able to escape. At the same time, it was important that the potential leakage in and out of

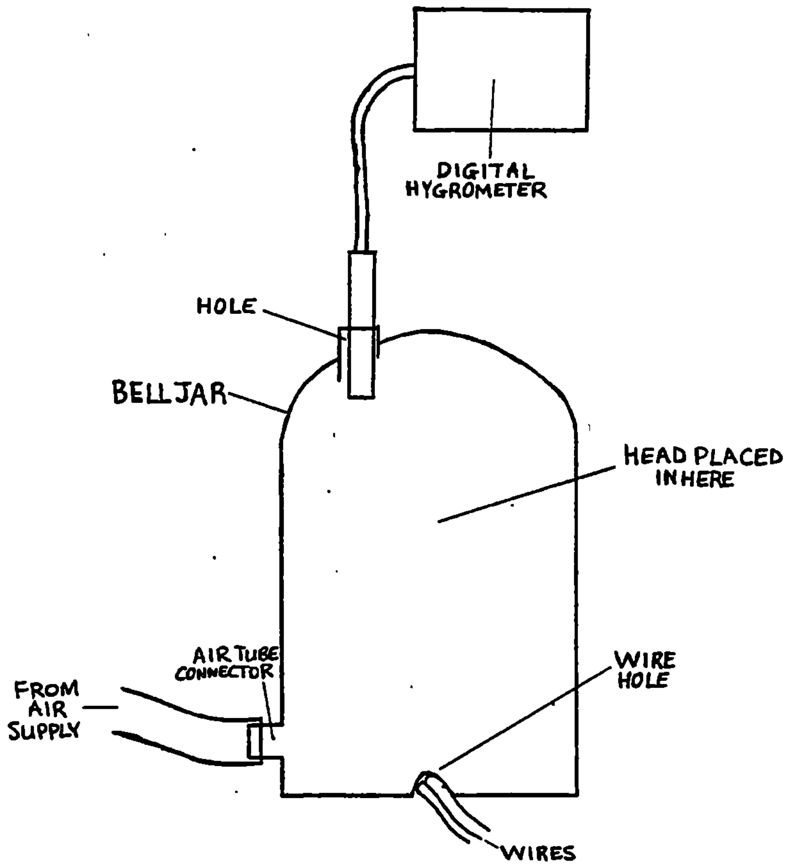

Figure V-7. Bell Jar for Environmental Control

The arrangement of the bell jar for controlling environmental conditions during experiments. Several holes and holes with connectors existed for the purpose of running in wires, air, and the probe from a digital hygrometer. The hole used for the hygrometer could also be used for inserting an instrument used for turning the Photodiode Y-Positioner in the event that cantilever deflection caused the reflected laser beam to stray too far from the center of the PSD/Photodiode.

the bell jar did not excessively exceed the incoming airflow. If this were the case, air leaking in would swamp the control air and it would be difficult to achieve humidities substantially lower or greater than the humidity in the laboratory.

The hygrometer (VWR Digital Hygrometer) employed was accurate for humidity levels between 5% and 95%. (VWR Scientific Products, West Chester, PA.) In some experiments humidity levels that were very low (almost 0%) were desired. In these cases, pains were taken to ensure that the leakage of humid air into the chamber from the laboratory environment would be kept to as minimal levels as possible. Purging the chamber with dry air for half an hour or so would result in the hygrometer reading 0.00% RH. Precisely how accurate this reading was is unknown, but it is highly likely that relative humidity was in fact very close to 0%. On the upside, this setup was able to boost humidity levels to slightly over 90%.

The reason for the apparent discrepancy, namely that approximately 0% RH could be easily achieved but that humidity could not be boosted beyond the lower 90s, is that the bubbler system had limited flow relative to the dry channel as its valves were not as commodious. The valves that needed to be negotiated in order to run dry air into the bell jar were much larger than those for the moist air and thus allowed a much greater flow of air to be pumped in. Successfully letting off the larger volumes of air when applying dry air did not prove to be a problem for two reasons. Firstly, the air went out the cracks with greater rapidity due to the higher pressure, and secondly, some of the wires entered the bell jar by going underneath the lower rim. This meant that the bell jar in fact sat at a slight angle with some space open at the bottom for air to flow out through. If the pressure were to build up sufficiently in the chamber, it would be possible for the jar to tilt up more and allow more air to escape.

5.6. Function Generator Experiments

In some experiments not involving alpha detection direct control of the cantilever a function generator (Stanford Research Systems Model DS345 30 MHz Synthesized Function Generator) was used to apply specific driving functions to the piezo controlling the cantilever. Besides varying the frequency and amplitude of the driving force, the function generator could apply bursts of pulses of specified duration to the cantilever and then cease producing any signal. It was also possible to apply different waveforms (sinusoidal, square, and triangular) to the cantilever, although there was no necessity for doing this.

The function generator was used in experiments for several reasons. It was considered desirable to probe cantilever response to driving functions of different amplitudes and frequencies in part to get a feel for cantilever behavior and to look for any unusual kinds of behavior that might have an effect on radiation detection experiments. Exciting that cantilever at different frequencies and comparing the amplitude of oscillation as found with a digitizing oscilloscope was a convenient way of measuring the harmonic resonance frequency and any shifts that it underwent. Applying tone bursts (brief periods of excitation of specified duration) permitted controlled study of cantilever damping characteristics.

Chapter 6

Results and Discussion

6.1. Introduction

The alpha detection experiments did indeed lead to the detection of alpha particles via changes in cantilever dynamic response. In this chapter we will discuss these responses, how they compared with responses produced using a charged plate with a controlled voltage using a DC power supply, and what this indicates about the detector system. Other topics will include further experiments with modified detector designs, what was learned from said modifications, and possible directions for future work.

6.2. Considerations in Detection and Data Analysis

6.2.1. General Comments

As expected, changes in both damping and frequency were observed in the alpha detection experiments. Getting to the point of having useful data, however, was not easy. It is instructive to consider the problems encountered and discuss them at some length. While many points are discussed in detail elsewhere, it is worthwhile describing how they specifically impacted the alpha detection experiments and what kinds of conclusions we can draw from those effects.

Actually observing a definite change in the cantilever's behavior as a result of alpha irradiation was initially very difficult. There were several reasons for this, among them the collector plate design, problems in the data analysis methods (particularly those used early on), and the fact that charge build-up on the plate was not as great as had been anticipated.

6.2.2. Comments on Detector Design

The first collector plate design posed problems for several reasons. The plate itself was a small section of approximately square silver foil. Silver is, of course, highly reflective and great trouble was encountered in bringing the tip down to the surface due to the numerous false contacts that were registered. As mentioned earlier, application of some Aerodag G to the target zone substantially alleviated this problem. The foil had long, sharp edges as well as a large grounding wire (a grounding wire with a switch arrangement was employed) and deep suspicions existed regarding charge concentration at the center of the plate versus concentration at the edges. Another geometry-related problem was that, by using a flat area of the collector plate, the gradient in the electric field was not as great as would be the case for a curved surface. In general, the more the surface is curved, the steeper the gradient in the electric field. So it was known that both of these geometry-related problems would probably work to reduce cantilever response, although precisely by how much was unknown.

The collector plate design finally settled upon was, of course, a very small ball bearing. This overcame many of the problems inherent to earlier detector designs such as reflectivity. The ball bearing collector plate was placed above a window of known size to control the number of alphas striking the collector plate.

6.2.3. Transient Production and Analysis

Breakaway curves were used to generate transient excitations of the microcantilever. This permitted simultaneous measurement of the frequency and the damping rate of the cantilever. We will now quickly review the characteristics of a breakaway curve and examine how they related to alpha detection.

The characteristics of breakaway curves (used for alpha detection) are important for understanding how and why the environmental and other modifying effects were significant in relation to alpha detection using the method described herein. See Figure V-5 for a typical breakaway curve that occurs within a force calibration curve. At the top left of the curve, the cantilever was in contact with the surface and because the surface had been steadily pulled away from the cantilever and was some distance from the zero displacement line of the cantilever. The cantilever was at this point very close to breaking free of the surface. This happened when the restoring force of the cantilever exceeded the tip-surface attractive force. After breakaway, as can be seen, the cantilever oscillated about its zero displacement line some distance from the surface, with some initial displacement from zero displacement resulting from the lingering effects of the water bridge between the cantilever and the surface. The cantilever was excited at its fundamental frequency and vibrated at it. The oscillations died down and the cantilever gradually came to rest. The frequency parameter used for detection and measurement of charged particles was the frequency of these oscillations. The damping rate was the rate at which the oscillations died down, determined by fitting an exponential decay curve to the peaks on the upper side of the series of oscillations.

Note that voltage was a fairly arbitrary measurement of displacement. Some voltage to displacement conversion ratio existed, but as discussed elsewhere (pp. 32, 51-52), it was dependent on a number of factors such as cantilever geometry and angle. When a new cantilever was used or the setup for a cantilever in use was substantially altered, remeasurement of the displacement per voltage conversion ratio was desirable. For the cantilevers used in these experiments, conversion ratios of 150-600 nm/Volt were typical.

Successfully isolating and measuring changes in cantilever behavior was not always straightforward. As discussed later in Section 7.5, the water bridge between the cantilever tip and the surface affected the behavior of the cantilever immediately after breakaway from the surface. After the tip broke free of the surface, the water bridge did not immediately break but instead stretched out some distance. (The bridge can be stretched a long way, relatively speaking, before it reaches the critical distance at which it becomes unstable and breaks.) When the bridge is in place, the cantilever can still vibrate but the bridge may affect the behavior of the cantilever. An example of this is the skewness evident in the initial vibrations of the cantilever (the bridge is unstable at this point and will break, but it has not yet done so). Since the distance between the center of cantilever vibration and the true zero-displacement line falls off as an exponential decay function, the first peaks produced by vibration are not useful for calculating damping rates. The exponential decay displacement mode is so large relative to the magnitude of the damping signal that it will overwhelm it. The solution to this problem is to ignore some of the first peaks. Whether this is a viable alternative or not depends on how long the skewness effects persist relative to the length of time that vibrational peaks remain of sufficient amplitude to be measured accurately. Conditions at the surface as well as the characteristics of a cantilever affect these things. So far, a case has not been encountered for which it has not been possible to compute damping rates for an alpha experiment. However, there have been wide fluctuations in the damping rate due to a number of factors that are at work. Large shifts in the damping parameter typically correspond with very small changes in the actual damping curve, generally not being evident by simple visual inspection. Some damping rates have been quite noisy, primarily as a result of only being able to use a small number of vibrational peaks to compute damping.

As an example of how the choice of points affects the noise level in a plot of damping rate versus time of exposure to alphas, see Figure VI-1 below. Note that all of the different sets of points were generated from the same set of data. Each set of points indicates a general trend of a drop in the absolute value of the damping parameter with increasing alpha exposure, but some sets are much noisier or less noisy than others are. The difference between these different sets is which peaks and how many of them were used to compute the damping rate. Many of the early peaks were dramatically affected by the skewness of the breakaway curve. Some of the final peaks became quite small relative to the noise level in the signal coming from the cantilever and as a result could be rather inaccurate. Only a limited number of peaks were available for analysis and disregarding too many at the beginning and end of the period over which the cantilever was excited could result

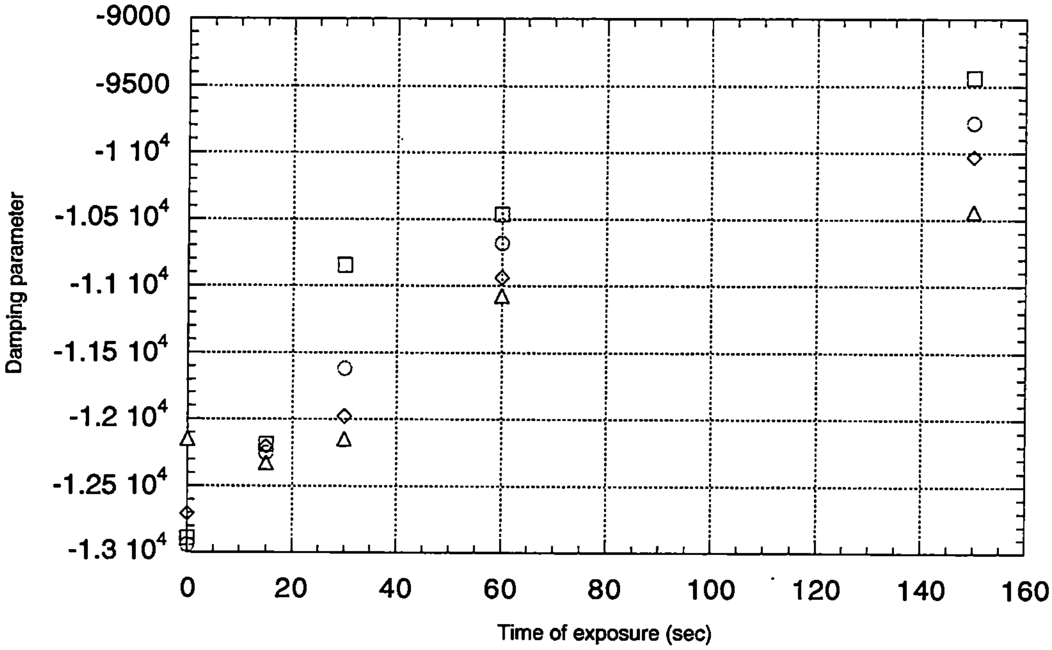

Figure VI-1. Variation of Data Quality with Choice of Peaks

The effect of choosing different peaks for calculation of the damping rate - each type of symbol represents a different choice for peak set used in computing damping rate. The trend in the damping rate resulting from exposure to alphas is evident in each case, but picking the wrong points (such as noisy points from the end or skewed points from the beginning) results in very noisy data.

in a noisy signal due to generating a curve fit from a small number of points. The optimum choice of points varied from experiment to experiment depending on the circumstances of each experiment (for example, in some experiments the water bridge would cause more skewness than in other experiments, necessitating throwing out a greater number of peaks at the beginning of the excited period). In this way, analysis of data in order to generate damping information was as much an art as a science. This was one definite drawback of damping as opposed to frequency analysis.

Curve fitting was accomplished in a couple of ways. As discussed earlier (p.76), damping rates were computed by finding the tips of individual peaks in a breakaway curve (time and deflection position) and then fitting an exponential decay curve to that set of peaks. General curve fitting for the breakaway curves was accomplished using the mathematical software package SigmaPlot. SigmaPlot fit given mathematical functions to breakaway curves (corresponding to cantilever motion) by altering given parameters in those functions such as time constants. It was found that the breakaway curves were best fitted using three modes: a fast mode, being an exponential decay mode; an intermediate mode, being another exponential decay mode; and a slow mode, being a damped oscillatory mode. An explanation of these modes is in order. The first two modes, the fast and the intermediate, are caused by the same effect, namely the water bridge that exists between the tip and the surface (this is elaborated on in Chapter 7). The slow mode (the damped oscillatory mode) is produced by vibrations of the excited cantilever; these steadily damp out due to internal friction and energy transmission to the air and the substrate to which the cantilever is attached. The presence of an electric field in an alpha detection experiment changes the damping rate, of course. The frequency of the damped oscillatory mode is the frequency of the cantilever. Shifts in the frequency, as measured using SigmaPlot, turned out to be a good basis for alpha detection.

6.3. Alpha Detection Results

6.3.1. Introduction

The alpha detection experiments were successful and repeatable changes in both damping and frequency were shown with definite dependencies on the length of time the collector plate was exposed to an alpha particle beam. It should be noted that many things affect the frequency and damping rates of a microcantilever. Under the conditions encountered in this set of experiments, the damping rate was less stable than the frequency and more difficult to measure, making the frequency generally a better choice for detection. Before discussing the factors that impact charged particle detection, it would be good to review the results of the detection experiments. Doing this will give an idea of the magnitude of changes observed in the parameters of interest due to alpha exposure and thus the degree of significance of changes in said parameters induced by changes in environmental and other conditions.

Results from detection experiments conducted during October 1998 will be discussed. Earlier data acquired in the spring of 1998 in by Ari-David Brown in collaboration with myself[15] will not be discussed as it was acquired with a rather oddly shaped collector plate (see previous discussions of collector plate geometry). As a result it will not tend to give as accurate a picture of general cantilever response as data taken with a spherical collector plate does. Data was acquired at other times in different sets of experiments than those mentioned above. Due to the extremely long periods of time required to analyze the data in detail (almost entirely because of the time required to find damping rates due to the relatively primitive methods used), only these two experiments were analyzed in full detail.

6.3.2. Frequency Response

Cantilever resonance frequency showed definite shifting when exposed to the electric field produced by the build-up of charged particles on the collector plate. As can be seen in Figure VI-2, significant frequency shifts were measured. A frequency shift of around 450 Hz took place after 620 seconds of alpha exposure

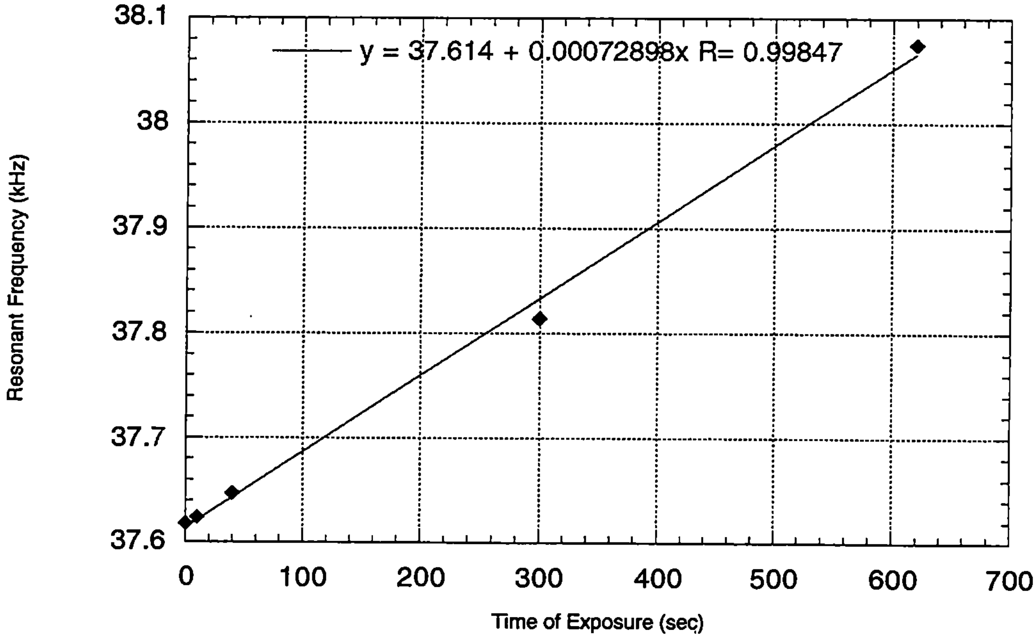

Figure VI-2. Variation of Resonance Frequency with Alpha Exposure

Cantilever resonance frequency as a function of the time the collector plate was exposed to alpha particles.

(the frequency versus exposure time line was quite linear out to this point). This is about 1.4 seconds/Hz of shift. Estimates are that a shift of 1 Hz can be detected using a good lock-in amplifier. Shifts of a fraction of a hertz can probably be detected under the right conditions and using the right methods. For example, the lock-in is more sensitive to frequency shifts if an indirect method – phase shift measurement – is used instead. Some noise is present in the frequency due to such things as pressure effects, however. The methods used in this experiment were, unfortunately, somewhat limited compared to phase shift measurement and minimum detectable frequency shifts were substantially higher. (One reason a lock-in was not used is because they require direct driving of the cantilever in order to plot amplitude and phase as a function of frequency rather than performing a spectrum analysis of the signal.) Taking the 1 Hz minimum as an indicator of what could be achieved using a detection device such as this one would indicate approximately 1050 alphas could be detected without any optimization or changes in the design of the cantilever and the charge collector plate. Although this might seem like a large number, a couple of things ought to be considered. The first is that this sensitivity could likely be radically improved with optimization of the system. The second point is that detectors with relatively low sensitivity are not necessarily without uses. Detectors with very high sensitivity exist, as do detectors with very low sensitivity, but there are large dynamic ranges for which good detectors largely do not exist.

6.3.3. Damping Response

In detection experiments based on changes in the damping coefficient, the damping coefficient k was calculated using an Ae^{kx} fit for the damping of the cantilever's vibrations. As in the frequency plot, the shifts were very linear over the time period examined. (See Figure VI-3.) The damping coefficient shifted by about 1200 between 20 seconds and 620 seconds of exposure, a decrease of about 16% over that time period. This works out to a coefficient shift of around 2 $second^{-1}$. No specific data on minimum detectable damping rate shifts exist (due to the fact that damping rates are apparently much less stable than frequency) but it is highly unlikely that a level of sensitivity comparable to that of the frequency method could be achieved.

The second damping experiment (see Figure VI-4) showed roughly similar results in that the damping coefficient shifted in the same general kind of way. Several things stand out, however: the damping occurred rather more quickly (i.e. the shift in the damping rate per unit time was much larger) and the curve was non-linear. It will be noticed that the curve was in fact quite linear over the first 15% or so shift in the damping rate. In general, such curves will be quite linear over fairly

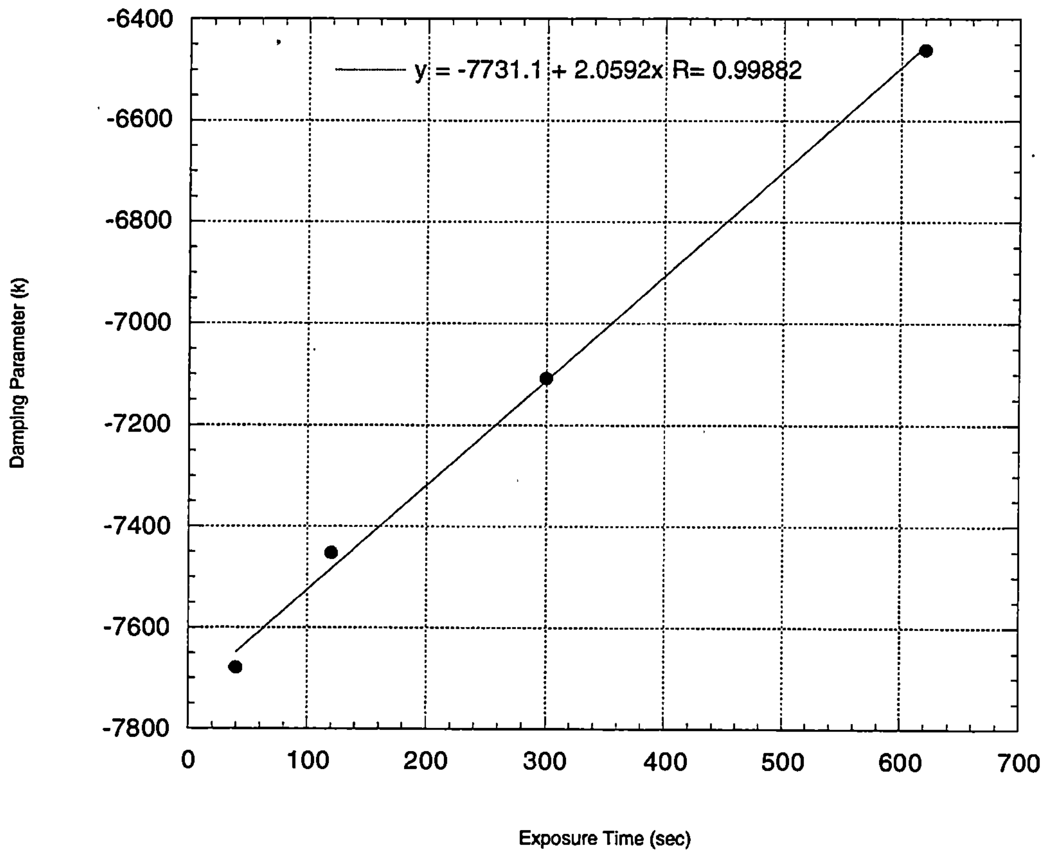

Figure VI-3. Variation of Damping Rate with Alpha Exposure

Cantilever damping rate as a function of the exposure of the collector plate to alpha particles.

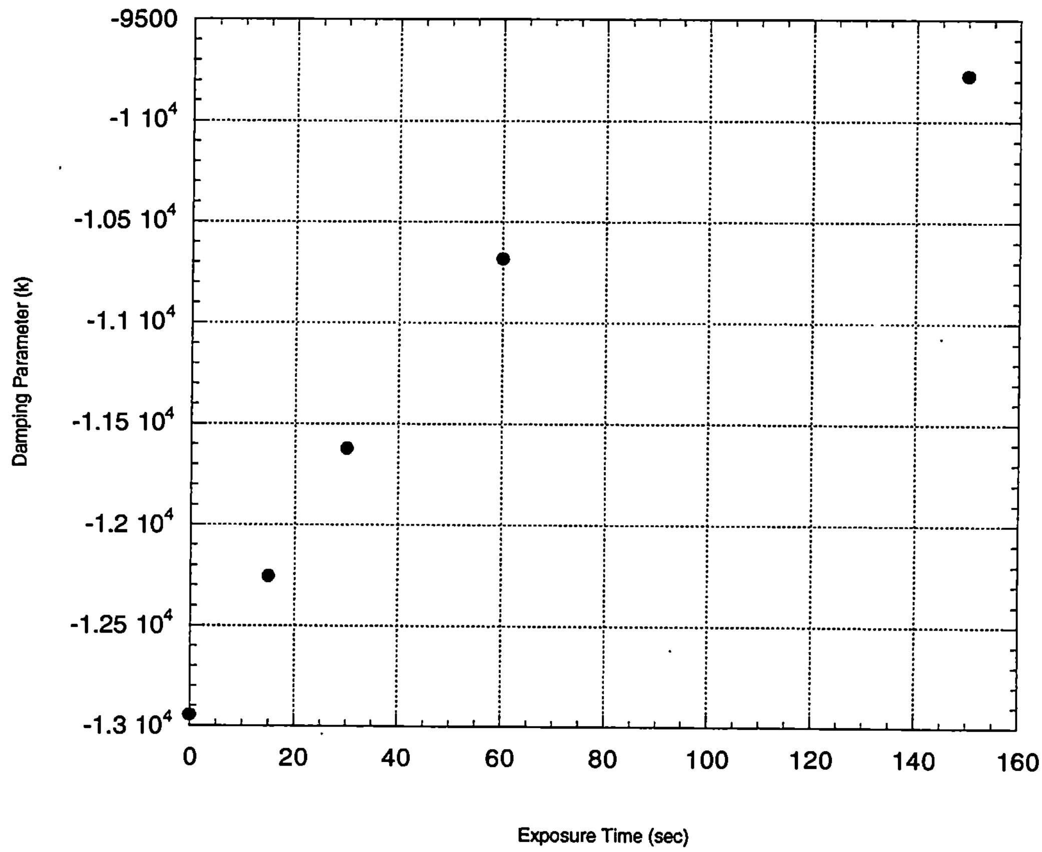

Figure VI-4. More Variation of Damping Rate with Alpha Exposure

Damping rate as a function of collector plate exposure to alpha particles, this time for a different run of the experiment.

small ranges of shift. Seen over longer periods of time, the curves lose their appearance of linearity. The reason is that the damping rate will move towards an asymptote when the collector plate is exposed to a charged particle beam. The asymptote is the result of the collector plate reaching a point at which charging by incoming alphas is completely cancelled by charge loss. Changes in the damping rate appear approximately linear over short distances, but over longer distances the true nature is revealed. Returning to the specifics of this curve, a shift of around 2200 in k occurred during the first 60 seconds of exposure. This is about 37 second^{-1}, a vastly larger figure than in the previous experiment. A further shift of around 900 occurred over the next 90 seconds, or about 10 second^{-1}. Looking at the general shape of the curve, it may be that this last point is an aberration and perhaps should have shown more change than it did. In any event, a decrease in damping rate shift per unit time is clearly occurring and an asymptote is being approached.

6.3.4. Summary of Results

To sum up detectable shifts were observed in both frequency and damping rates as a result of alpha exposure. As could be seen between the two experiments, damping rates can vary substantially between experiments which, along with the difficulty in measuring them accurately, makes them not very well suited to radiation detection and measurement. Frequency rates are much more stable (experiments with one cantilever showed not more than 5 Hz drift in either direction over several months) and are the most logical parameter to use for this purpose. We will consider the factors that affect these parameters and the impact they have on this method of radiation detection and measurement in the chapter on modifying factors.

6.4. Voltage Experiments

6.4.1. Introduction and Overview

The method for radiation detection investigated in this work was charged-based, that is, it involved measuring charges produced by incident charged particles. This being the case, putting a charge on the collector plate could simulate the effects of actual exposure. This allowed for much more controlled investigation of the various effects of interest by eliminating many elements of uncertainty from the experiment. If the relationship between charge and voltage for a collector plate is known, extrapolations can be made from the simulated results in order to determine what would happen in cases that cannot be measured easily, such as very high exposure rates and very brief exposure times. This approach has its limitations, of course, one of which being that the actual voltage that builds up on the plate as a result of charged particle irradiation is not simply a function of just the number of incoming particles and the time of exposure (as discussed in Section 6.5.3).

Travis Gaulden (the other summer student besides Ari-David Brown) and I simulated alpha fluence in this way using a power supply connected to the collector plate.[16] (All data in this section is included under this citation.) The effect of modifications to and the charging of a cantilever were also probed. The aim was to find the dependence of frequency shifts and deflection on the distance to and voltage on the collector plate. Cantilever damping rates were not investigated as it was felt that they did not represent a very practicable method for detection in the long run due to their instability (large drifts) over time.

Modification of cantilevers was suggested by published work from other researchers. It has previously been shown that putting a conducting layer and/or a voltage on a cantilever can increase the sensitivity of that cantilever to electric fields.[8] The modifications made in this set of experiments usually consisted of putting down a layer of gold on the lower side of a cantilever using an evaporator (homemade, various manufacturers). In some cases, tip-less cantilevers were used. These can be purchased, so was not necessary for us to remove the tip ourselves.

As expected, a difference in voltage between the cantilever tip and an adjacent conducting surface produced cantilever deflection. This deflection, besides being highly sensitive to changes in voltage, was strongly affected by the presence of a conducting layer of gold on the cantilever tip, the level of relative humidity in the surrounding atmosphere, and the tip-surface distance. The effects could be quantified as follows. The presence of a 30 nm layer of gold on the underside of the cantilever (including the tip) resulted in a tripling of deflection as compared with an uncoated cantilever. Reducing the humidity from ambient laboratory conditions (50 – 60% RH) down to approximately zero resulted in about two and a half times as much deflection for both regular cantilevers and cantilevers with gold-coated tips. Deflection was proportional to the square of the voltage difference between the cantilever and the surface, at least over moderate deflection distances. (Over larger distances, the electric field can change appreciably from position to position and result in the deflection being significantly different from the expected value.) Several different cantilevers were used in these experiments, all of the same design and with very similar tip radii. Very little difference was observed in response between these different cantilevers.

The dependence on distance was not really what one would intuitively expect. When one thinks of electrostatic forces, one thinks of $1/r^2$ dependence. This kind of dependence is true for point charges and things that approximate point charges. However, there are several fundamental differences between the voltage experiment and a point charge situation. The (induced) charge on a tip in a voltage experiment is not confined to the very apex of the tip but instead is spread along it.

In a sense this could be seen as a kind of averaging of forces for some distance over the chip. If the tip-surface distance were large relative to the length of the tip, the situation would somewhat approximate a charge above a charged plane. (Although the induced charge on the cantilever tip would not be fixed, but would instead vary with the tip-surface distance and the charge on the surface.) If the tip-charged surface distance were small, it can be seen that force would change less rapidly with distance than for pure point charges. At very small separation distances, force dependence on distance would likely change again as the force due to charges at the very end of the tip would far outstrip the force resulting from charges farther along the tip. Other differences besides the two already mentioned would play a lesser but significant role. Referring back to the chapter on theory, we find that indeed the function relating force to distance varies with distance. When $R<d<L$, where R is the tip radius, d is the tip-surface distance, and L is the tip length, the best model is a charged-line model. In this case, force falls off with distance as $1/\ln(d)$. When $d<R$, a spherical model works best (the charges on the end of the tip becoming dominant) and the force falls off as $1/d$.[7,8] Deflection data in the voltage experiments was generally taken when $R<d$. It was always the case that $d<L$. This being the case, we would expect to see relatively small drops in deflection with distance (deflection being proportional to force) that were most pronounced at small

d and became less significant as d increased. Of course, this discussion applies quite well to many other changes in cantilever behavior because the physical mechanisms that bring about changes in those parameters work in the same sort of fashion and would be expected to demonstrate very similar changes with distance. Cantilever frequency shifts, for example, ought to show similar regimes with distance. However, the rate of change with distance would fall off at a 1/r rate rather than as 1/ln(r) since frequency shifts are dependent on the derivative of the tip-surface electrostatic force rather than the force itself.

6.4.2. Experimental Results and Extrapolation to other Configurations

Using a cantilever with a gold-coated tip set 100 nm from the surface in a dry environment, a potential difference of 2 Volts yielded a deflection of 16.9 nm. (See Figure VI-5.) Recalling that deflection is proportional to the square of the potential difference[6,7], if we assume a minimum detectable deflection of 1 nm (it is considerably less than this but thermal and electrical noise can be substantial) and a change in potential voltage from zero, a minimum change in voltage of 0.5 Volts is detectable. There are ways to improve this, however. The effect a change in voltage is much enhanced if the voltage from which that change occurred is increased. Assuming that V (Voltage difference between the tip and surface) is large relative to ΔV (change in this value), the change in deflection resulting from

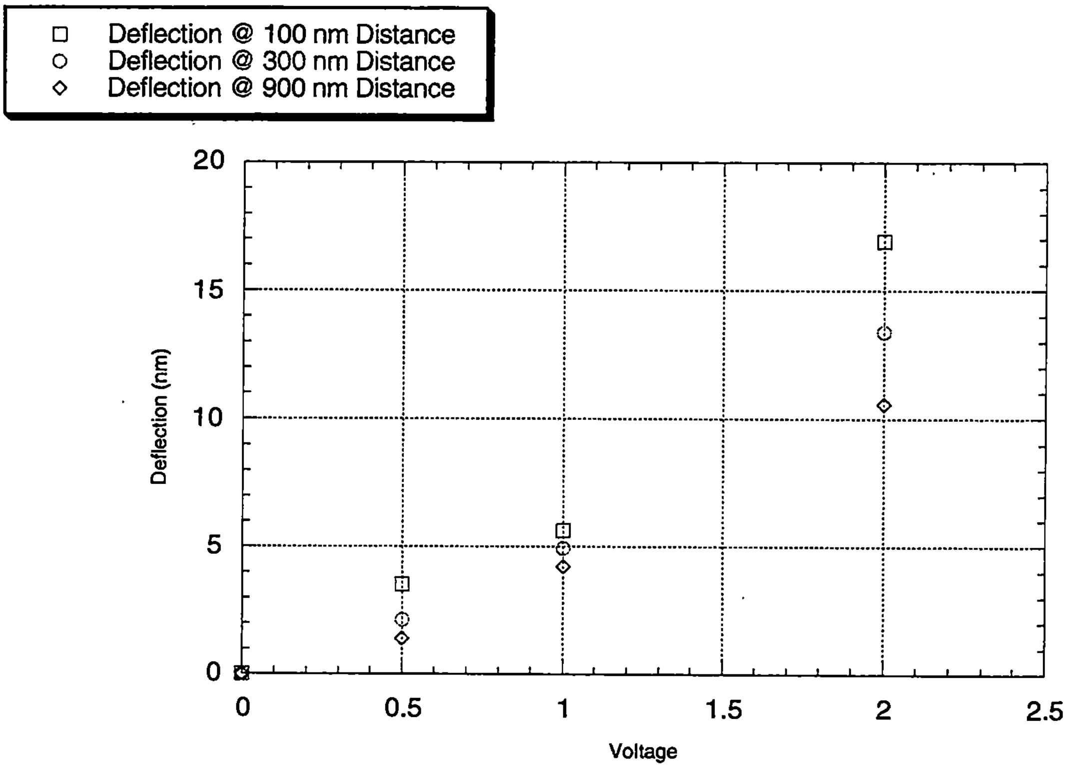

Figure VI-5. Deflection of a Coated Cantilever in Dry Conditions

Deflection of a gold-coated cantilever in dry atmospheric conditions when in the vicinity of a charged plate.

ΔV will be proportional to V^2. This being the case, for V = 2 Volts, the minimum detectable ΔV using the previous assumptions will be about 30 mV. Larger values of V would improve this figure even more, but two things need to be remembered. There is a limit as to how large V can be before the tip jumps to contact with the surface and noise will get larger with larger values of V (although it will increase much more slowly than V).

It is worth looking at some specific data sets acquired under different conditions in order to get a sense of the relationship between potential difference, tip-surface distance, environmental conditions, and deflection distance. Besides the data already shown, several data sets are instructive for this purpose. Figure VI-6 shows the deflection of an uncoated cantilever under conditions of normal humidity at different distances from the charged surface. Figure VI-7 shows deflection for an uncoated cantilever in dry air. Note the much larger deflection for this second case as opposed to the first case for the same voltage and approximately the same distance. Finally, Figure VI-8 shows deflection as a function of distance and voltage for a gold-coated cantilever (the coat being on the underside of the cantilever, including the tip) under conditions of normal humidity. Note that the deflection in this case is substantially less than in the first case (shown in Figure VI-5), which was identical except for the use of dry air.

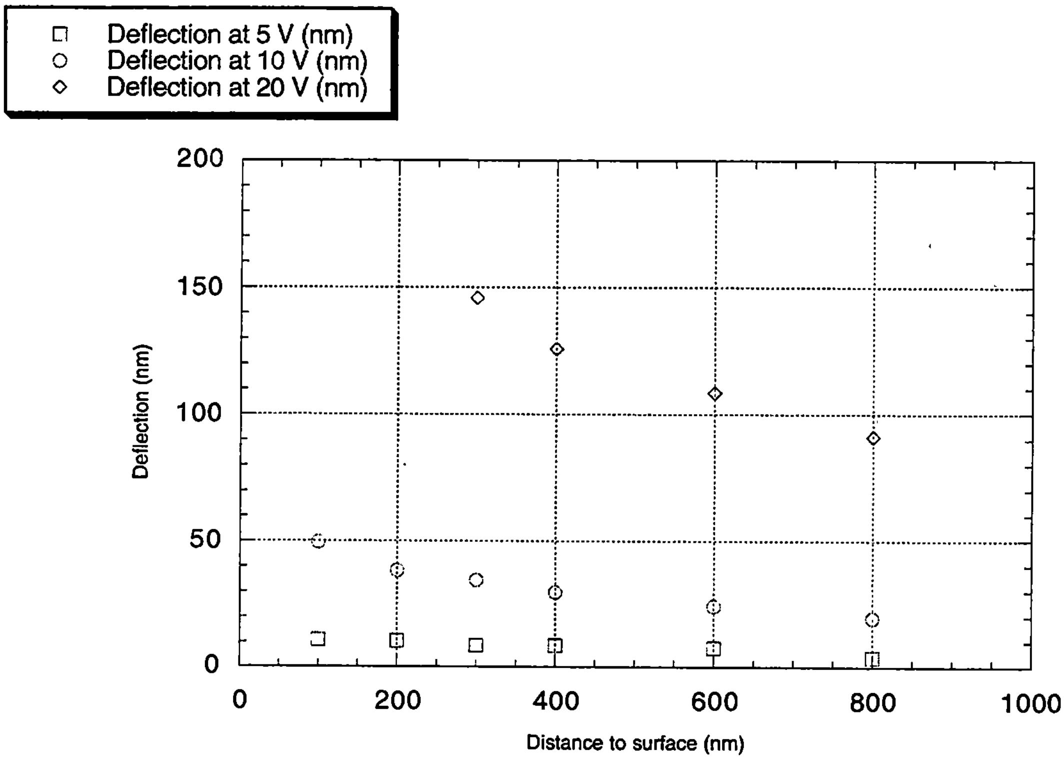

Figure VI-6. Deflection of an Uncoated Cantilever in Humid Conditions

Deflection of an uncoated cantilever under normal atmospheric (humid) conditions for different plate voltages and tip-plate distances.

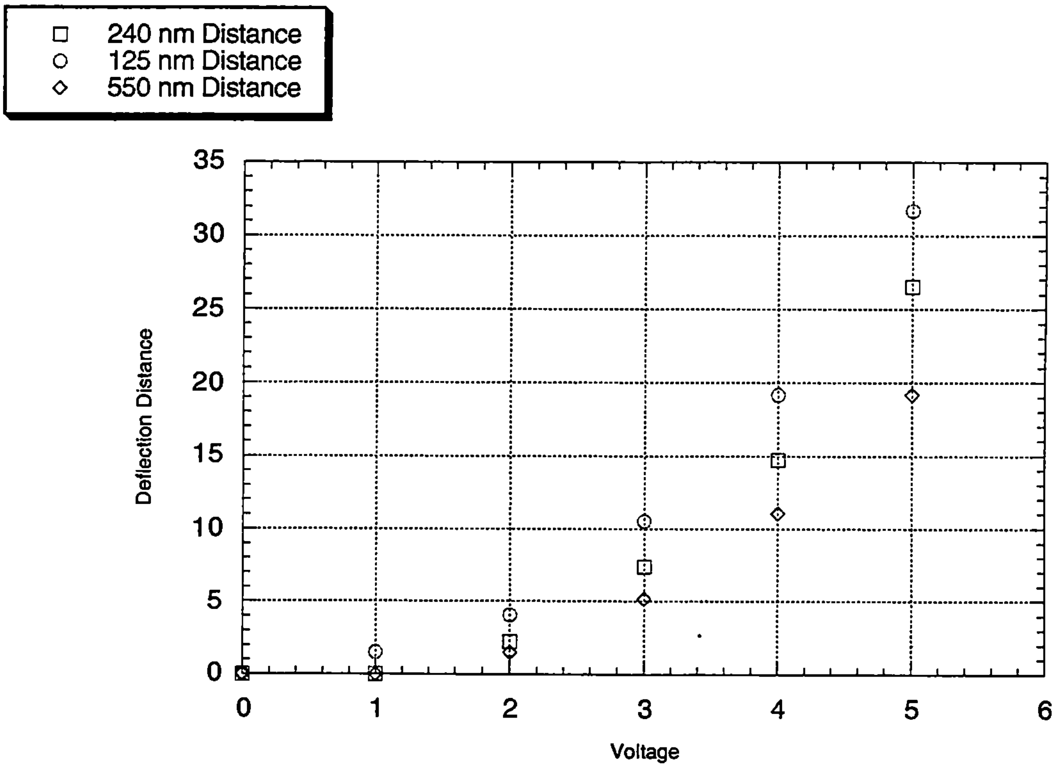

Figure VI-7. Deflection of an Uncoated Cantilever in Dry Conditions

Deflection of an uncoated cantilever in dry air as a function of tip-plate distance and plate voltage.

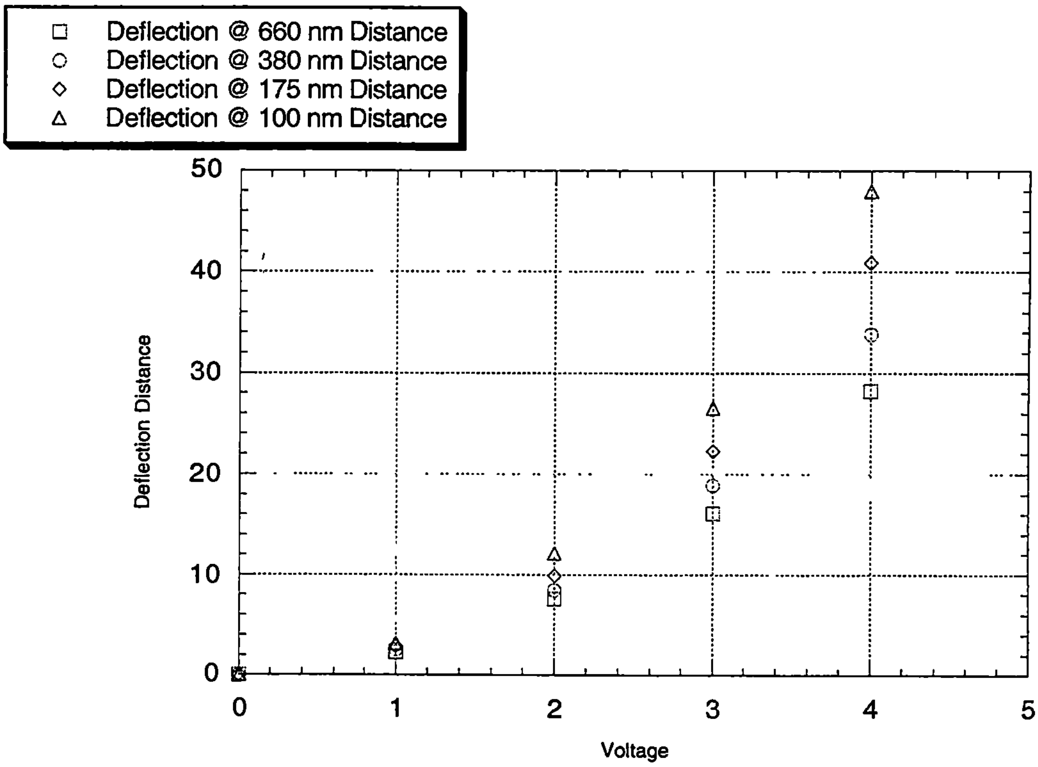

Figure VI-8. Deflection of a Coated Cantilever in Humid Conditions

The deflection of a gold-coated cantilever under normal (humid) atmospheric conditions as a function of different tip-plate distances and plate voltages.

The large difference between dry and humid air is explained in this way. Objects in electric fields, especially charged ones, will cause the fields to drop off much quickly than they otherwise would. Water is a highly polar molecule and, although it is neutral in total, the humidity in the air greatly affected the electric field, causing it to drop off much more quickly than in dry air and thus resulting in large reductions in deflection and frequency shift.

Cantilever resonance frequency underwent a substantial shift in response to the presence of an electric field, just as deflection did. Many of the characteristics were the same, such as shifts being proportional to the square of the voltage on the surface (assuming an uncharged cantilever). This was to be expected, as the causes of deflection and resonance frequency shifts are highly similar; deflection being a function of the strength of the electric field and frequency shift a function of its derivative. For a fixed physical layout with no varying voltage and other factors constant, the derivative of the electrostatic force will be proportional to that force at a particular spot, no matter how the voltage changes. Over ranges of distances, the force and the derivative of the force will change at different rates. However, for fairly constant distances from the surface or cases where the force and its derivative vary only slowly with distance, there will be a rough ratio between the shift in frequency and the deflection distance, as there was in this instance. Thus, it was not felt necessary to characterize frequency shifts under different conditions such as dry and humid air and gold-coated or regular tips since they ought to be predictable for a given design given the deflection data. In these experiments, a conversion factor of approximately 4 Hz of frequency shift per 1 nm deflection was found. (This would vary a little with tip-surface distance but would be pretty close to this value over the range of distances investigated.) Again, a very precise figure is not given due to uncertainties in tip-sample distance, noise, and the fact that this factor changes somewhat with distance.

Using a coated cantilever under dry conditions at 100 nm distance from the surface, the application of a potential of 2 Volts to the surface produced a frequency shift of around 75 Hz - a drop from an initial resonance frequency of around 15.31 kHz. (See Figure VI-9.) Other experiments with frequency shift were done under different conditions and the effects of distance, humidity, and gold-coated tips on frequency were very similar to the effects of those things on deflection. If we assume that a shift of 1 Hz in the main resonance frequency could be detected (the easiest way to achieve this is by measuring higher order resonance peaks, especially if using phase shift), then a change in voltage of less than 0.25 Volts (starting from zero) is detectable. Again, if the potential difference is initially 2 Volts, a change of less than 15 mV is detectable. This fact – that starting out with a significant

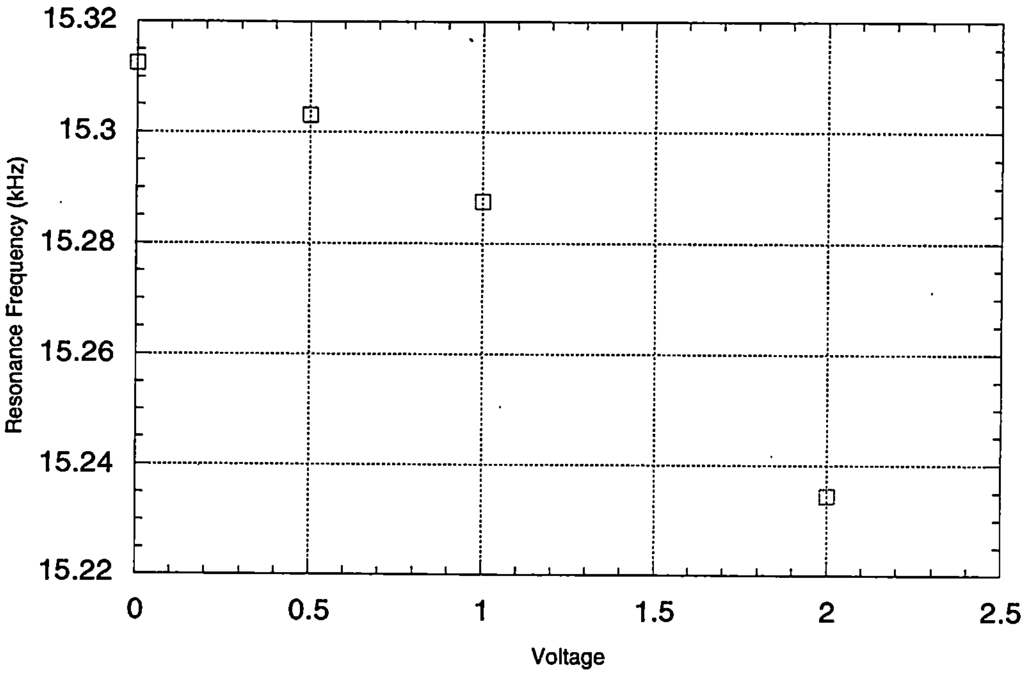

Figure VI-9. Variation of Frequency in Voltage Experiment

Cantilever frequency as a function of voltage on the collector plate for a cantilever positioned 100 nm from the plate.

potential difference already in place before an exposure to charged particles will greatly enhance sensitivity – is very important, as will be seen later. While it was not possible to modify the current detector to take advantage of this, if a slightly different principle were to be used as the basis of detection, good use could be made of this effect, as will be seen later.

6.5. Comparison of Alpha Experiments with Voltage Experiments

6.5.1. Differences between Alpha and Voltage Experiments

Definite differences in response between the alpha experiments and the voltage experiments were observed. It was expected that alphas impinging on the collector plate would cause its voltage to rise at a constant rate since the alpha flux rate was constant and the voltage on an object is proportional to its charge. (The voltage of an object comes from the equation $q = C V$ where q is the charge, C is the capacitance, and V is the voltage, namely $V = q / C$.) It was further expected that changes in cantilever response (primarily frequency shifts and deflection) would occur in both voltage and alpha detection experiments and in the same ratio to one another. In actual fact, this did not happen and the responses in the two types of experiments were quite different.

The differences between the alpha detection and voltage experiments were these. The alpha detection experiments showed substantial changes in frequency with little or no deflection. The voltage experiments, by contrast, showed both frequency shifts and deflection. Had the ratio of deflection to frequency shift in the voltage experiments held true for the alpha detection experiments, deflection in the alpha detection experiments ought to have been easily measurable. Furthermore, frequency rose in the alpha detection experiments, whereas it dropped in the voltage experiments.

6.5.2. Differences Resulting from Charge Transfer to Tip

Why such a difference in behavior? The difference is related to contact between the tip and the surface. In the voltage experiments, charge was present on the (collector plate) surface. The microcantilever tip never came into contact with the surface, apart from an initial contact needed to establish the position of the surface. (When this was made, the surface was grounded.) By contrast, the alpha detection experiment involved the tip coming into contact with the surface during a force calibration curve when measuring changes in resonance frequency. Measurement of deflection in the alpha experiment did not require contact between the tip and the surface other than an initial calibration of surface location relative to the cantilever tip, again when no charge was present on the surface. The drop in frequency during the voltage experiments was indicative of a tip-surface attractive force as would be expected, whereas the rise in frequency observed in the alpha detection experiments suggested a repulsive tip-surface force. As there was no difference in the collector plate (except perhaps the quantity of charge present on it), it follows that the difference in behavior must be as a result of some difference in the state of the cantilever. The dividing line seems to be whether or not the cantilever tip made contact with the collector plate, suggesting that the difference in state was related to the contact, or lack thereof, between the cantilever tip and the surface.

The substantial frequency shifts recorded in the alpha experiments when contact was made between the tip and the surface and the lack of measurable deflection when no such contact was made indicates that tip-surface contact resulted in much larger forces acting between the tip and the surface. Clearly, this is not explainable in terms of any persistent changes in the induction of charge on the tip caused by tip-surface contact. Other forces (capillary, van der Waals) never demonstrated any substantial changes in their magnitude as a result of contact during other experiments (such as tip-surface attractive force measurement experiments), essentially ruling them out as candidates for this difference in force. A major clue is provided in the fact that the frequency shift in the alpha detection experiments was positive, instead of negative as in the voltage experiments, strongly suggesting a tip-surface repulsive force. This could be easily accounted for by the transfer of charge from the surface to the tip during tip-surface contact. This is in fact what was actually happening.

Charge transfer can be accounted for in an unexpected way. Silicon nitride, the constituent material of the cantilever is an insulator and not prone to picking up charge. In the actual alpha detection experiments, the cantilever used did not have a deposited layer of gold on its underside, precluding charge transfer to the gold. It did, however, have a small drop of water at the tip as a result of the condensation of water from the ambient humid air.

With the microcantilever tip being as small as it is (15 to 20 nm tip radius), it acts as an excellent nuclei for water droplet growth. Water has a particular affinity for very small particles. A graphic illustration of this is the formation of water droplets around microscopic dust particles in the atmosphere which then fall as rain. In general, the smaller the particle, the greater the affinity of the water for it. It has been suspected for some time that under normal (ambient) levels of relative humidity a fairly large water drop is present at the tip of the cantilever[17,18]. Our experimental results showed this to be the case. In dry conditions (approximately 0% RH), the drop is for all intents and purposes no longer present. (A very thin layer of water does remain tightly bound to the surface.) When the drop is present, it acts as a wonderful conductor and readily soaks up charge when in contact with a charged surface.

In the detection experiment, the water on the tip picked up charge when in contact with the charged collector plate. For a collector plate of fixed voltage the precise amount of charge picked up by the cantilever varied according to the volume of the water droplet, but the voltage on the drop itself remained the same. (Note that as the drop was so small, fluctuations in its size had a significant impact on cantilever response in detection experiments.) In the detection experiment then, the cantilever had charge of the same sign as the collector plate. As a result, a repulsive tip-plate force was introduced and a rise in cantilever frequency was observed with increasing charge on the plate. By contrast, in the voltage experiments contact between the tip and the plate was not normally made while the plate was charged, thus the tip-plate force remained attractive and frequency decreased with increasing voltage.

The existence of a substantial water droplet under normal laboratory conditions and the absence of one under dry conditions was proved in this way. Under normal conditions a cantilever was placed near a charged collector plate (some minimum amount of charge was required for this to work) and the cantilever jumped-to-contact with the plate, in part as a result of the attractive electrostatic force. (Jump-to-contact and other cantilever phenomena involving tip-surface forces are explained in greater detail in the chapter on modifying factors.) The cantilever jumped in as expected and charge was transferred to it. Charge of the same sign was now present on the cantilever tip and the collector plate, causing a repulsive force between them. The cantilever immediately broke away from the surface and moved well out into the surrounding air. As can be readily deduced, some maximum initial separation distance existed beyond which the initial jump-to-contact would not occur. Some minimum separation distance also existed because in most cases the cantilever restoring force as well as the repulsive electrostatic force were required to break the cantilever free of the surface.

When the cantilever was placed between these two positions - the minimum and maximum separation distances - a particular kind of behavior was observed. The cantilever initially jumped into contact with the surface and then broke free due to the electrostatic tip-surface repulsive force as described above. After a period of time, the cantilever began to inch towards the surface, eventually jumping into contact again, only to immediately break away. This process repeated itself indefinitely. Under dry conditions, the cantilever jumped into the surface and stayed stuck to it. The explanation is that under normal conditions substantial quantities of charge were transferred to the water droplet on the cantilever's tip, while under dry conditions insufficient water was present on the tip to pick up enough charge to result in breakaway. (Probably practically no charge was transferred and besides the regular forces gluing the tip to the surface, an induced charge on the cantilever also acted as a strong bonding force.) When charging of the tip was sufficient for breakaway to occur, the charge would be gradually lost to the surrounding atmosphere after separation from the surface and the cantilever would eventually become electrically neutral, allowing the process to repeat itself.

6.5.3. The Rate of Charging of the Collector Plate

The absence of significant cantilever deflection and the presence of substantial frequency shifts in the alpha detection experiments, was of course due to the presence of substantial charge on the tip in the frequency shift experiments and the lack thereof in the deflection experiments, as outlined above. The presence of charge on the tip vastly increased the magnitude of the tip-surface force. Several things are worth noting here. For a constant tip-surface distance and a case in which the cantilever tip and the surface have the same voltage on them (such as when the tip and surface were just in contact), the repulsive force between them will be proportional to the square of the voltage. (This comes from the force between charges being proportional to the magnitude of the charges being multiplied with each other.) What is surprising, though, is that the alpha detection experiments using both frequency and damping rate changes did not show the rate of change in the parameters increasing over time, but rather holding constant (linear or near-linear changes with time at first, then a gradual drop-off). This indicated that charge build-up on the collector plate was not linear with time, but was instead decreasing such that the change in the square of the voltage was staying approximately constant, before finally stopping completely as some sort of equilibrium of charge on the plate was reached. This kind of charge build-up could be accomplished by a case wherein the rate of change in voltage was exponentially decreasing over time.

That voltage change per unit time was not constant presents us with a question: namely, why? Prior to running the voltage experiments, the assumption had been that only those alphas actually striking the collector would have any effect on its net charge. In actual fact, as the alpha particles streamed through the air on their way to the collector plate, they gave up energy in the form of ionizations. Even though the atmosphere was, and is, very rarified in comparison with a liquid or solid and is thus not especially effective at slowing down alpha particles, large numbers of ionizations were created in the immediate vicinity of the collector plate. These ionizations were able to migrate through the air if propelled by some force (in this case an electric field resulting from the build-up of charge on the plate). Of course, as they moved they were subject to some probability per path length of being stopped. The positively charged collector plate attracted electrons from the surrounding air that tended to neutralize the charge on it. As the charge on the plate increased, the effective volume for electron collection around the collector plate increased and neutralization became more pronounced. At some point, the voltage became sufficiently large that any further increases were completely negated by attracted electrons. It is easy to see, intuitively, that the charge on the collector plate initially rose at a relatively quick pace but that the rate of increase dropped off and eventually went to zero. The voltage on the plate rose asymptotically, approaching some final value. It was estimated that in the case of the ball bearing collector plate, maximum charging was around a tenth of a volt.

6.6. Analysis of Results

6.6.1. Operation with no Charge Transfer or Ion Migration

The effect on charge accumulation that results from the movement of ions in air made things rather different from what was initially expected. While this did result in a reduced detector response for the setup in ambient conditions, it is possible to use this effect in other ways to construct a detector. Given the right design, it is also possible to avoid this effect altogether.

To prevent migration of ionized particles to the charge collector, the cantilever-collector plate system could be placed in a vacuum. In this case it was not possible to do this because the equipment was air-cooled and would become overheated and damaged if subjected to a vacuum environment for any substantial length of time. Of course it would still be possible to do this if appropriate equipment were to be used. If this were to be done, the cantilever detector would essentially behave as predicted by the electrostatic force equations. (This would be in good agreement with the results obtained in zero relative humidity environments during the voltage experiments.) For all intents and purposes, water would no longer be present on the surface of the collector plate or on the tip of the cantilever. Rather than using force calibration curves to excite the cantilever, a piezo could be mounted at the base of the cantilever or some other method of excitation could be employed in order to track cantilever frequency shifts by finding phase shift and amplitude as a function of frequency. Deflection measurements would not require excitation of the cantilever and would thus be easier to accomplish.

Were a cantilever-collector system thus placed in a vacuum environment, it is straightforward to calculate the rate of voltage build-up on the charge collector plate. If we assume a spherical collector plate as was used in the alpha detection experiments, we find that the capacitance is related to the radius of the collector plate by

$C = r$, where r is in cm and C is in abstatfarads[19], which gives

$C = 1.11 * 10^{-6} r$, where r is in cm and C is in microfarads.

Knowing that q = C V, it is then a simple matter to calculate the number of alphas required to obtain any particular level of voltage on the collector plate.

The size of an individual charge collector plate (in a practical device a large number of individual charge collection elements and cantilever detectors would likely be present) can be determined on the basis of its intended purpose. Specific applications might call for counting small numbers of particles or for measuring particles incident over a large area. It is straightforward to derive an expression for the response of the microcantilever detector given different collector plate sizes and numbers of incoming charged particles.

Given,

$C = 1.11 * 10^{-6} r$, where C is in microfarads and r is in cm,

then

$C = 1.11 * 10^{-12} r$, where C is in farads and r is in cm.

Since

$$q = CV,$$

then

$$V = q / C.$$

Substituting in for C, we get:

$$V = 9.01 * 10^{11} q / r.$$

Alternatively, if we wanted q for a given V,

$$q = 1.11 * 10^{-12} rV.$$

To transform both of these expressions to a charge carrier basis, we would use the conversion ratio of one single charge equals $1.602*10^{-19}$ Coulombs. Doing this gives

$$V = 1.44 * 10^{-7} q / r \text{ and}$$

$$q = 6.93 * 10^{6} rV,$$

where q is the number of electronic charges.

6.6.2. Optimization of Response by Varying Collector Plate Size

A couple of things are evident from the equations just presented. The smaller the collector plate, the fewer the minimum number of charges that must accumulate in order to be detectable (i.e. to reach some minimum voltage), as would be expected. It will also be noted that the cross sectional area of the collector plate increases as the square of the radius while the capacitance increases in proportion to the radius. What this means is that if one desires to measure the strength of a flux going through an area, the larger the collector plate, the greater the sensitivity to that flux. (This assumes that the area through which the flux is travelling is as great or greater in extent than the cross sectional area of the collector plate.) Thus we see that the optimal design of a detector based on the principles outlined herein is strongly dependent on the desired application.

To get a feel for what kind of charge-voltage relationship these equations indicate and what limits on detection capabilities result, let us consider a couple of cases. It has already been shown that for a system with a pre-existing voltage difference of 2 volts between the microcantilever and the collector plate, a change in the potential difference of less than 15 millivolts is sufficient to produce detectable changes in resonance frequency. (For pre-existing potential differences greater than 2 volts, this minimum necessary voltage change would be even smaller.) The first case we will consider uses a spherical collector plate of radius 50 microns. This would require the accumulation of 460 charges on the collector plate, or about 230 alphas. A collector plate with a radius of 0.5 cm would require 46,000 charges, or about 23,000 alphas.

These numbers may at first seem high, and indeed they are if one is thinking in terms of counting single alphas. Before reaching any conclusions, several things should be considered. The first consideration is that the sensitivity of the setup can be improved in a number of ways, of which we will discuss three here.

It will be recalled that the force acting between the tip and the collector plate is proportional to V^2, where V is the voltage on the collector plate, the cantilever being uncharged. These calculations assumed a V of 2 volts. It was mentioned earlier that increasing V would increase sensitivity and the large difference made by using 2 volts instead of 0 volts (initially) was shown. This increase is by no means the most that could be achieved. Much higher values of V are possible, although the increasing jump-to-contact distances that result from larger values of V are a limiting factor. Increasing V to 10 volts would result in twenty-five times the level of response although such an increase would probably necessitate moving the cantilever substantially farther from the collector plate such that the reduced strength of the field at this distance might cut this increase in half. Nevertheless, this is more than an order of magnitude increase. Note that although the volts per meter field strength indicated in this case would be very high, breakdown would not be a problem due to the fact that the breakdown value for air undergoes a very rapid increase with decreasing distance at very small separation distances, becoming very high at the distances we are discussing (nanometers).[20]

6.6.3. Other Methods for Boosting Detector Response

Another useful method of attack for improving detector response would be to boost the charging produced by an incoming energetic charged particle by designing the collector such that electrons in the electron spray produced as the charged particle slowed down could easily exit the material. (This could be done, for example, by using a thin circular collector plate. This would have the additional advantage of having a lower capacitance than the spherical collector plate.) The number of electrons produced in an electron spray is considerable. The average energy loss per ionization in a conductor is very roughly 1 eV. For an alpha particle of approximately 5 MeV, this corresponds to an electron spray consisting of some five million electrons. Most of these electrons are still bound to the material, even though they are mobile within it. Although only a small fraction of these electrons are capable of exiting the material, a small fraction of such a large number of electrons is still a large number. A collector capable of expelling just a small fraction of these electrons would exhibit a much greater response than one that did not take advantage of this possibility. For example, if an alpha particle were to cause the expulsion of 60 electrons, the detector response would increase by one-and-a-half orders of magnitude. Once electrons were expelled an electric field could be used to prevent them from returning to the collector plate.

Another method for increasing detector response is to put a voltage on the cantilever itself. The case in which both the cantilever and the collector plate are charged is a bit more complex than the charge induction case and the theory will not be covered in depth here as it is beyond the scope of this work. (The increase in complexity is in part due to geometric considerations - the whole cantilever is an irregular object and in such a case it would have charge all over it, rather than have charge concentrated at the tip as in the charge induction case. However, for very small tip-surface distances, the force on the apex of the tip will dominate.) Limited experimentation[21] was done in which both plate and cantilever were charged. Sensitivity was greatly increased. The limit to which charging of the cantilever could be taken has not been investigated. (There is some limit to the strength of the field between the tip and surface beyond which problems such as breakdown will arise. In addition, there is a limit to the amount of force a cantilever can be subjected to without causing adverse effects to its mechanical characteristics.) However, it is known that considerable gains in sensitivity can be readily achieved using this method. An order of magnitude should not be readily achievable if the collector-detector system is designed properly.

It ought to be pointed out at this time that charges on collector plates tend to be slow to dissipate. The actual dissipation rate depends on the size of the collector plate, the level of relative humidity, the presence of ions in adjacent areas capable of travelling to the plate, and how well the collector plate is isolated from adjacent conducting materials. In experiments in ambient conditions, charges of several volts on a 1/16" ball bearing took approximately 45 minutes to dissipate.[21] Under vacuum conditions this time would be greatly extended. The implication of this is that the collector plate can act as an integrator of incident charged particles. Measurement of accumulated charge could be done at any desired time without the loss of any accumulated charge. Grounding, essentially a resetting of the detector, would also be similarly flexible in its schedule. A microcantilever charged particle detector using this design could measure the incident charged particle flux over some period of time without necessarily having to detect individual charged particles. Such a device could also act as a short-term dosimeter, and potentially as a longer-term dosimeter as well if charge retention were sufficient for it.

6.6.4. Comments on Usefulness of Detector

A microcantilever charged particle detector constructed around the principles described herein would probably not possess as high a sensitivity as is possible for a microcantilever radiation detector (certain other detection principles would likely produce greater responses), but this would not necessarily be a bad thing. A detection system following the principles outlined in this work would a very wide dynamic range. This would result from being able to put a voltage on the collector plate and/or the cantilever prior to exposure (or at reset during exposure), as well as to vary the time of each cycle (between resets of the detector). There are distinct cases in which charged-particle detectors with wide dynamic ranges are very desirable. As an example, current charged particle detector technologies are good at detecting very low and very high fluxes of particles, but a large dynamic range between these two extremes is left uncovered. Specifically, commercially available detectors are lacking for the region between 10^3 and 10^9 particles/second, which poses significant problems for researchers working with particle beams. Microcantilever charged particle detectors based on the detection of accumulated charge would be viable over this range.

6.7. Other Experiments and Application to Possible Successor Devices

As mentioned earlier, numerous other methods exist for using microcantilevers as radiation detectors and dosimeters. Considerable thought has been put into what other physical principles would be good to use for detection. A comprehensive review of different possibilities is beyond the scope of what we are trying to cover here, but mention will be made of a couple of methods that are related to the method described in this thesis. Some of the data gathered during the investigation of this method sheds light on other methods of detection.

As will be recalled, it was found that the charge on a plate exposed to an alpha beam did not rise above some particular value at which further charging was entirely prevented by ionizations attracted by the charge on the plate striking the plate and canceling out its charge. This led to experiments that worked in an opposite way to the detection experiments that are discussed herein. Specifically, a substantial charge (around ten volts, very much beyond the equilibrium charge due to alpha beam exposure) was placed on the collector plate. Exposure to an alpha beam resulted in a cascade of ionizations being formed in the air around the collector plate. Many of the ionizations migrated to the plate which then settled down to the equilibrium charge under alpha beam exposure (a small fraction of the original charge) in a matter of several seconds.

When the collector plate was kept permanently connected to a DC power supply, it was found that some critical distance between the source and the collector plate existed on either side of which the cantilever would either be fully deflected or completely undeflected.[16] (It was found that the behavior of the cantilever, and thus by implication the charge on the plate, could be a bit unpredictable right around this critical distance. The assumption is that the rate at which the power supply supplied current to the collector plate, or perhaps the rate at which the current flowed through the wires to the collector plate – the wires being small and the quality of the contacts perhaps changing over time – was not constant from one instant to the next.) The explanation for this phenomenon is that when the source was sufficiently close to the collector plate, enough alphas passed through the air in the vicinity of the plate, creating ionizations, to prevent any significant buildup of charge on the plate. At greater distances, the power supply kept the voltage on the collector plate at the set level, despite incoming ionizations canceling the charge; in other words, the rate of current flow from the power supply exceeded that to the collector plate from the ionizations in the air.

The upshot of these observations is that a microcantilever-based detector can be constructed using very similar principles to a gas-filled detector, such as a Geiger counter or proportional counter. In this case a collector plate would be periodically recharged to some particular voltage level. Ionizing radiation entering a chamber or region of gas containing a charged plate would produce ionizations that would migrate to the plate and cancel out some of its charge. The change in charge on the plate would be measured by an adjacent microcantilever operating in either deflection or frequency variation detection mode. Alternatively, the charge to the plate could be supplied at some constant rate rather than in discrete units. This second possibility would work best if the flux was more or less constant and the rate of charge replenishment was variable as this would give a much wider range than would be possible using constant rates of current flow. (Of course, current would only flow if the voltage on the plate had dropped as a result of exposure to ionizing radiation. If the plate had the charge it was intended to have, no current would flow.) Numerous possibilities for variations on this system exist. Certain advantages would result from using this kind of system. Since a large voltage would already be present on the plate, changes in deflection and frequency resulting from changes in the voltage on the plate would be maximized. (Remember that shifts are proportional to V^2. Since the voltage on the plate would be falling under exposure to radiation, the initial voltage could be set at whatever maximum the system could handle, thus maximizing the shift resulting from a change in the voltage on the plate.) The most sensitive region of the detector's response characteristics would be used for lower levels of ionizing radiation flux, while the less sensitive regions would be used when fluxes were higher. Overall detector sensitivity would be much higher than that of a straight charge collection detector because the detector would be collecting many charges from one incident radiation (by collecting ionizations from the gas) rather than collecting only a couple of charges in the case of an alpha. The effective volume of space used for charge collection would also be larger, which would be advantageous for some applications.

There are other interesting variations on these themes that can be pursued when designing a radiation detector. One is to have a cantilever that resonates at a frequency proportional to the charge buildup or cancellation on a plate. This can be done in one of several ways. One example would be to set up the cantilever such that once a certain amount of charge built up on the plate, the cantilever would jump to contact with the surface. A cantilever coated with conductive material would ground the plate and break away from it by virtue of the strength of its restoring force once the electrostatic attractive force were gone. Once a sufficient charge was built up again to cause it to come into contact with the surface, the process would repeat itself. The rate at which the cantilever performed this jump/breakaway cycle would be indicative of the rate of charge buildup.

Another possibility would be to use the cantilever as a means of transmitting charge. A conducting cantilever attached to a DC power supply would be mounted a short distance from an uncharged plate enclosed in a gaseous environment to allow migration of ionizations from radiation interacting with the gas. The attractive force between the tip and the plate would cause the cantilever to deflect and come into contact with the plate. Upon contact, the plate would be charged up. After the plate became charged and the tip-plate electrostatic attraction gone, the cantilever would break away from the plate due to the strength of its restoring force and return to an undeflected state. (Of course, the microcantilever would have to be mounted at a suitable distance from the plate to start with for all this to occur correctly.) Once the plate lost sufficient charge the microcantilever would again jump into contact with the plate due to the strength of the electrostatic tip-plate force and recharge the plate. The rate at which these cycles proceeded would be indicative of the strength of the radiation flux incident on the detector.

While access was not available to all of the equipment required for the construction of such a detection system, it was possible to perform a simple experiment to verify the general principle behind such detectors.[16] A plate charged by a DC power supply was placed very close to an unmodified silicon nitride microcantilever. Electrostatic force caused the cantilever to jump to contact with plate. A water droplet present at the tip of the cantilever picked up charge from the charged plate, vastly reducing the electrostatic attraction between the tip and the sample (if sufficient charge were transferred to the water droplet, the force would become repulsive). The strength of the restoring force caused the cantilever to break free of the surface whereupon the water droplet began to dissipate charge to the air. Once the droplet had lost sufficient charge, the tip-plate attractive electrostatic force caused the cantilever to again jump to contact with the plate. This process went on ad infinitum. Each cycle was fairly slow (taking a minute or two), largely because of the time taken to dissipate the charge on the cantilever tip. Variations in the cycle time also resulted from changes in the size of the water droplet on the tip.

In the two cases just discussed (charging of the plate leading to cantilever motion discharging it and charging of the plate via the cantilever), response time would not be a problem if the equipment were designed properly. It is to be noted that we did try setting up the case in which the cantilever would act as a conduit for charge transfer to the plate, but the charge flow through the tip to the plate was really far too slow to be practical. (Charging of the plate would literally take many minutes.) Ways of alleviating this situation would be to have a larger contact area for the current to flow through (our contact area was literally a few nanometers across) and to use a smaller plate (ours was a 1/16" steel ball bearing – a ridiculously large plate for the capacity of the connection supplying charge to it). Despite the limitations of our experiments, we did verify that the idea of the charging or uncharging of the plate leading to tapping of microcantilever against the plate at a rate proportional to the strength of the incident radiation flux is in fact valid.

6.8. Dosimetry Experiments and Possible Future Directions

Investigations have shown that microcantilevers ought to work well as radiation dosimeters. The simplest method of dosimetry involves coating a cantilever with a radiation-sensitive material. When a layer of coating material is put down on a cantilever, the material becomes part of the system. Changes in the mass, elasticity, and other properties of the coating material as a result of exposure to ionizing radiation will cause changes in characteristics of the system such as resonance frequency and deflection. The shape of the response curve for the cantilever (change in parameter versus exposure level) will depend on the relationship between material response and exposure. In general, though, we would expect response per unit dose to be highest at low levels of exposure and to fall steadily and predictably with increasing total dose.

Some experiments were conducted with polychloroprene-coated cantilevers. (Polychloroprene is a rubber that cross-links under exposure to radiation, increasing its strength.) At the time, it had not been possible to locate any data on how much exposure was required to significantly affect the materials characteristics of polychloroprene. Technical problems involving the formation of mobile solvent "bubbles" between the polychloroprene coated layer and the cantilever surface were encountered and were not immediately recognized. Nevertheless, data taken using high levels of exposures (well above lethal for a human) suggested that polychloroprene would work in a dosimeter but would not be sufficiently sensitive to be of any real use for non-lethal doses. This was subsequently confirmed when some data was located on materials response as a function of dose.

More work has been on dosimetry and materials with significantly greater sensitivity than polychloroprene have been identified. Based on theoretical calculations, it is believed that some materials used in x-ray lithography would work well as cantilever coatings for dosimetry. Limitations do exist in that some materials would have to be chemically "developed," complicating processing, and limiting an individual unit to a single use. Other materials exist. Some photographic materials, in particular some of the silver halides (silver bromide and silver chloride) are particularly susceptible to ionizing radiation. These materials ought to be easily reusable (since they lose mass with exposure, although at some point a silver halide based dosimeter would have to be discarded when it ran low on mass to loose) and produce very predictable response curves. The primary drawbacks to the silver halides are their sensitivity to certain frequencies of light (blue light on up), necessitating processing in special facilities where such light would be excluded and keeping the dosimeters in light-proof encasings, and their lower sensitivity than some other materials.

Damage to DNA in cells is the most important type of radiation damage to the body. Thus it is highly appropriate that damage to DNA by radiation ought to be able to be measured, either for dosimetry or for experimentally investigating DNA damage by particular types of radiation, using microcantilevers. It is relatively straightforward to bond DNA chains to gold-coated microcantilevers. The change in the mass of system produced by adding DNA could be measured via changes in the resonance frequency of the cantilever. Similarly if radiation was used to induce double-stranded breaks in DNA, the loss of mass caused by the dissociation of DNA from the cantilever system could be measured. It is anticipated that DNA-coated cantilevers would be immersed in water and that quantities of enzymes for repairing single-stranded breaks would be added in order to filter out noise and to simulate conditions in a cell. Natural processes would not cause much in the way of the kind of catastrophic DNA damage that causes complete breaks in DNA strands in such a situation (the DNA repair enzymes would largely preclude this), and dissociation of DNA under these circumstances would be primarily due to ionizing radiation.

A couple of important limiting factors for DNA-coated microcantilever dosimeters would exist, however. The greater the DNA mass as a fraction of the whole system, the greater the sensitivity of a DNA-coated cantilever to radiation. The most logical approach, then, would be to maximize the surface area per volume of the microcantilever by making it as thin as possible, using the longest DNA strands practicable, and packing the DNA strands as tightly on the surface as possible. Limitations exist on all three modes of attack, the first being that there is a minimum thickness for microcantilevers related to current manufacturing capabilities and issues of cantilever durability. (In general, the thinner the cantilever, the easier it breaks; at some point decreasing thickness will drastically affect the strength of the lever.) The second and third factors are both subject to limitations in that excessive length and packing density of DNA strands would cause them to become entangled with each other and unable to dissociate when damaged by radiation. Calculations made taking into account expected technical limits as described herein suggest that damage to DNA could be measured by microcantilevers without any improvement in equipment or technical methods if high levels of exposure (several hundred rads or more) were to be used. Methods for achieving greater packing density and longer strand lengths without causing irrevocable entangulation and improvements in surface to volume ratios would allow detection and measurement of lower exposure doses.

Chapter 7

Modifying Factors

7.1. Introduction

Many factors affect the behavior of a cantilever during and after breakaway from a surface. These include such things as tip-surface geometry and environmental conditions such as humidity. Items to be discussed include the effect of operation on a sloped surface and the effect of the level of relative humidity on cantilever behavior. In considering humidity effects, tip-surface attractive forces and the behavior of the water bridge that exists between the tip and the surface at very small separation distances will be examined. All these phenomena significantly affect, or have the potential to significantly affect, the behavior of a microcantilever radiation detector. Many other factors that can modify cantilever behavior were observed and studied but will be omitted for reasons of time and space. It is considered that factors considered in this chapter are the most significant and relevant to the detector design used for this thesis.

7.2. General Comments on Humidity Effects

In general, water vapor is incapable of penetrating to any great depth in a cantilever; the surface oxide layer restricts water to within 10 nm of the surface.[4] This means that practically all humidity and water effects will have their origin at the surface. This might initially make one suspect that the relative humidity of the environment in which a cantilever is operating would have little or no effect on the cantilever. In fact, the opposite is the case; cantilevers are profoundly affected by humidity.

Humidity affects cantilevers in several different ways, mostly by controlling the depth of the water layer on the surface of the cantilever and any adjacent object with which it might be interacting (for example, a surface being imaged by an AFM). A thin layer of water is present on practically all surfaces, even at extremely low levels of humidity. In many cases only ultrahigh vacuum will completely remove the water layer. It is well known that changes in the amount of water on the surface of a cantilever cause shifts in its resonance frequency by changing both the mass and the spring constant of the cantilever. It is also well known that humidity levels affect the force between the tip of a cantilever and an adjacent surface. In general, the higher the level of humidity, the greater the force between the tip and the surface. Force is present not only during tip-surface contact but also when the tip is near the surface. If a cantilever tip is slowly lowered down towards a surface, it will at some point jump-to-contact; that is, it will experience a force attracting it to the surface, deflect, and come into contact with the surface before it is actually lowered the full distance down. (See Figure VII-1.) Jumping in is important because it places limitations on the range of possible distances from a surface that a cantilever may operate over without making contact with the surface. Other effects do occur, although we are not as interested in them as we are in these three.

7.3. Humidity and Cantilever Resonance Frequency

The variation of frequency with relative humidity has been extensively studied; in fact it was the basis of the first demonstration of a microcantilever sensor. Water on the surface of a cantilever increases the mass of the cantilever system, tending to cause a drop in the resonance frequency of the cantilever. However, the water on the surface is generally held tightly against it and will tend to increase the surface tension and thus the spring constant, increasing the frequency of the cantilever. In the majority of cases, the net effect of an increase in humidity is a decrease in frequency (the effect due to the change in mass is larger than the effect due to the change in surface tension). However, the frequency can sometimes be more or less constant over the full range of humidities one is likely to encounter (the two effects cancel each other out) or even rise with increasing humidity (the surface tension effect is bigger than the mass effect). An individual cantilever will show the same pattern of behavior consistently, but the pattern will

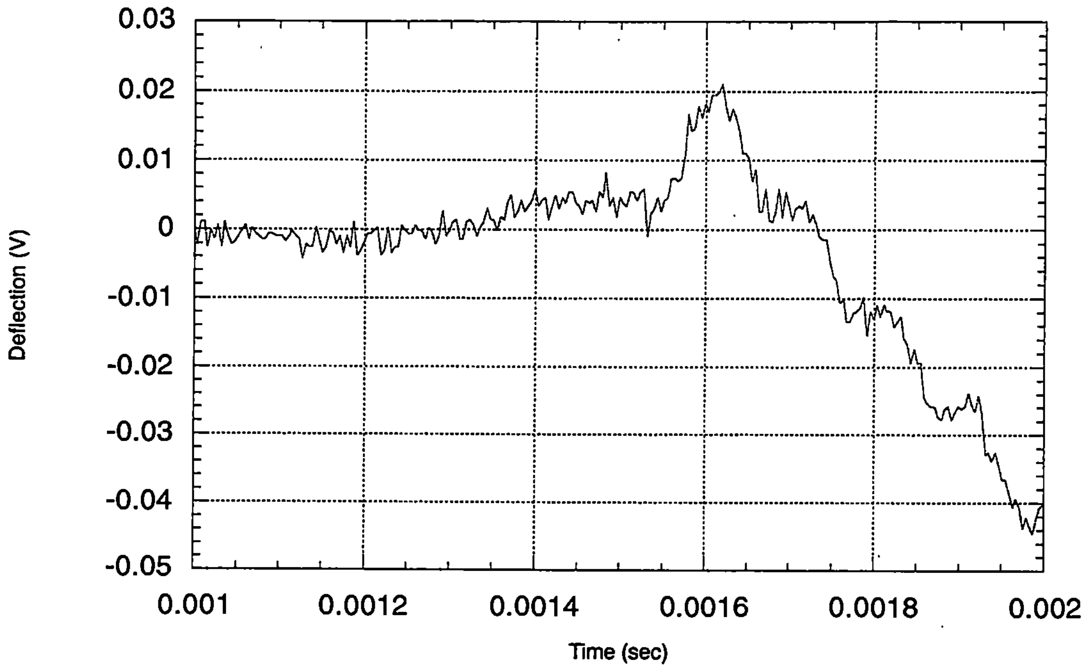

Figure VII-1. Cantilever Jumping into Contact with a Surface

At first the cantilever is free of any adjacent surface and slowly drifts with thermal and other changes. As a surface is moved towards the cantilever, some effect is seen as tip-surface force begins to become significant. At some point when the tip is just a few nanometers from the surface, a water bridge forms and the tip-surface attractive force exceeds the restoring force (due to the spring constant) of the cantilever, causing it to jump into contact with the surface. The surface continues to be moved up towards and against the cantilever, causing it to deflect as pressure is applied to it. The unevenness of the apparent cantilever motion as the surface pushes and deflects it is due to the cantilever tip undergoing some slippage relative to the sample surface while being pushed.

vary from cantilever to cantilever.[22] In general, differences in frequency between cases of very low and very high levels of relative humidity do not exceed several hundred hertz. This effect was not considered at any great length as it is essentially irrelevant if environmental conditions are fairly constant (as they were in these experiments, except when it was desirable for them to be otherwise). In an actual device that might be subject to wide variations in environmental conditions, resonance frequency shifts resulting from changes in ambient conditions can be compensated for by reference cantilevers, or various mitigation strategies can be employed.

7.4. Humidity and Tip-Sample Attractive Forces

7.4.1. General Comments

It was deemed important to study the effect of relative humidity on tip-sample attractive forces, especially with regard to breakaway and jump-to-contact distances. Operation of a near or at-surface microcantilever radiation detector would likely be strongly affected by tip-surface forces as was the detector described in this Thesis. One way in which the detector behavior depends on the tip-surface force is through the distance from the surface at which the excited cantilever vibrates after breakaway (which by definition is the breakaway distance) which is a function of the tip-surface force. This distance determines how much effect a given electric field will have on the resonance frequency of the cantilever. There are other reasons why the magnitude of this force and its dependence on ambient relative humidity are of general interest in the area of microcantilevers and AFM imaging. The lower the magnitude of the force acting between an AFM tip and a surface the better the resolution of acquired images[23,24] and acquisition of the best images possible is highly desirable. Characterizing the forces provides insight into the behavior of the water meniscus or bridge present between the tip and sample at small separation distances[17,18,25-27]. Force distance curves are an area of considerable interest[18,25-30] and they are strongly affected by capillary forces. So plenty of good reasons exist to study tip-surface forces. As it turns out, the detection experiments were conducted at around the same relative humidity level and with similar tip-surface forces so the effect of humidity in this way on the charged-particle detector was probably not all that great.

Both tip-sample attractive forces via breakaway distance measurements and jump-to-contact distance were studied as a function of relative humidity. With the exception of the work by Xu et al[31], tip-sample forces had not previously been studied above 70% relative humidity. Until now jump-to-contact distance had not been characterized as a function of relative humidity.

When the tip of a cantilever is in contact with a surface, an attractive force is present between the tip and the surface. As discussed previously, many forces can come into play, but the two types of forces predominate. These are the van der Waals force and the capillary force resulting from the presence of a doughnut-shaped water meniscus between and around the cantilever tip and the adjacent area of surface under normal atmospheric conditions. The magnitude of the capillary force is strongly dependent on the amount of water present in the meniscus. This is in turn a function of the relative humidity of the ambient atmosphere.[32]

It will be recalled that when a cantilever in contact with a surface is slowly retracted, the tip will not immediately lose contact with the surface but will remain in contact until the restoring force of the cantilever exceeds the tip-surface attractive force. When that occurs, the cantilever suddenly snaps away and a breakaway curve is produced. Measuring the distance a cantilever must be retracted before breakaway occurs allows one to calculate the strength of the attractive force between the tip and the surface by simply multiplying this distance by the spring constant of the cantilever.

Another effect of interest is the magnitude of the jump-to-contact distance and its dependence on relative humidity levels. If a cantilever is brought towards a surface, the attractive force between the tip and the surface will at some point exceed the restoring force of the cantilever, which will deflect and come into contact with the surface[26,28,33-37]. Initially the van der Walls force was thought to be the primary force involved in this[38], but it is now well known that the capillary force is a major player in this process[26,28,36]. This affects the variation of the cantilever's resonance frequency as it approaches the surface as well as the distance at which jump-to-contact occurs.

Both these phenomena will be considered with particular reference to their dependence on ambient relative humidity levels.

7.4.2. Experimental Results and Observations

For each humidity level investigated, a substantial number of data points were taken and used to generate an average value and standard deviation. Specifically, twenty data points were taken in each set of jump-to-contact measurements, while a rather lower number, generally six, were taken for each set of breakaway measurements. The reason for this was that jump-to-contact distances were rather smaller (and thus more subject to errors in measurement, relatively speaking) than breakaway distances, besides which they tended to show rather larger relative variations in distance due to natural causes than breakaway distances.

The jump-to-contact distance was measured at points over a range from 0-83% relative humidity. (See Figure VII-2.) This distance showed a large increase between 0 and 10% relative humidity, generally constant values beyond this up to about 50% RH, and a rapid, approximately exponential rise beyond this. Between 10 and 50% RH, small rises in jump-to-contact distance did occur as humidity rose. Jump-to-contact distances ranged from around 4.3 nm at 0% RH up to 5.5 nm at 83% RH. The standard deviation of the jump-to-contact distance as a fraction of the actual distance was very large, in some cases reaching approximately 0.3. (See Figure VII-3.) Despite this, variation in the actual average jump-to-contact distance was very small, as evinced by the smooth curve produced by the data points.

The distance required for the cantilever to be retracted before it broke free of the surface was quite repeatable and exhibited a definite dependence on relative humidity. The breakaway distance was fairly constant over 0-10% RH, rose fairly over 10-20%, crept up slowly over 20-70%, and exhibited a rapid rise beyond this. (See Figure VII-4.) In terms of the range of distances this represents, the breakaway distance varied from about 97 nm at very low humidities to around 154 nm at approximately 90% relative humidity. For a cantilever with a spring constant of approximately 0.06 N/m, this corresponds to a range of forces between 5.8 and 9.2 nN. (See Figure VII-5.) The breakaway values showed rather the opposite of

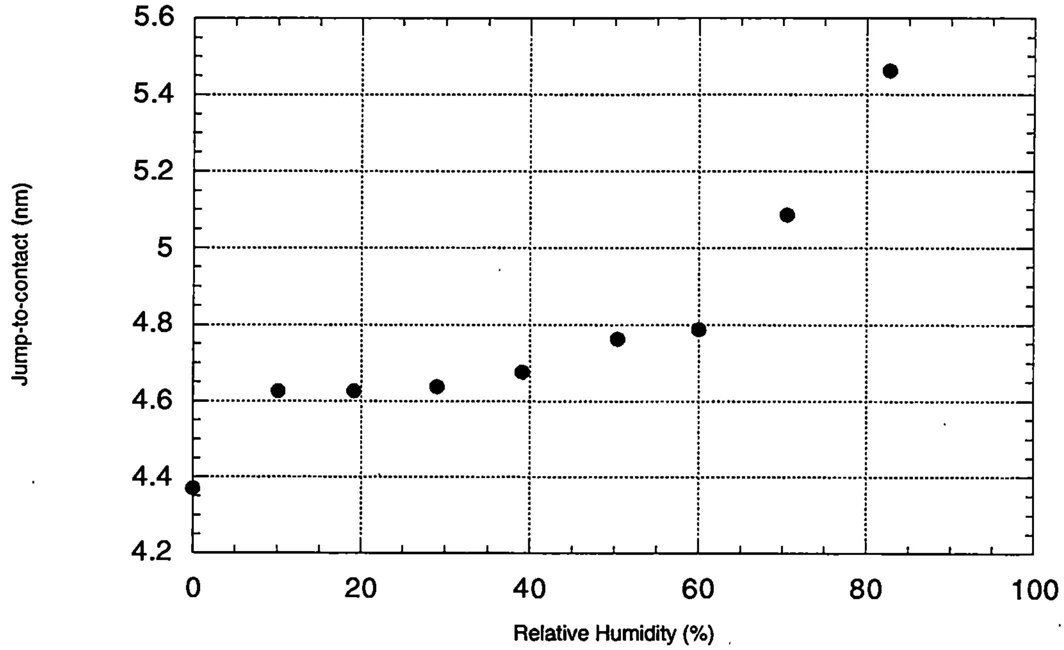

Figure VII-2. Jump-to-Contact Distance as a Function of Relative Humidity

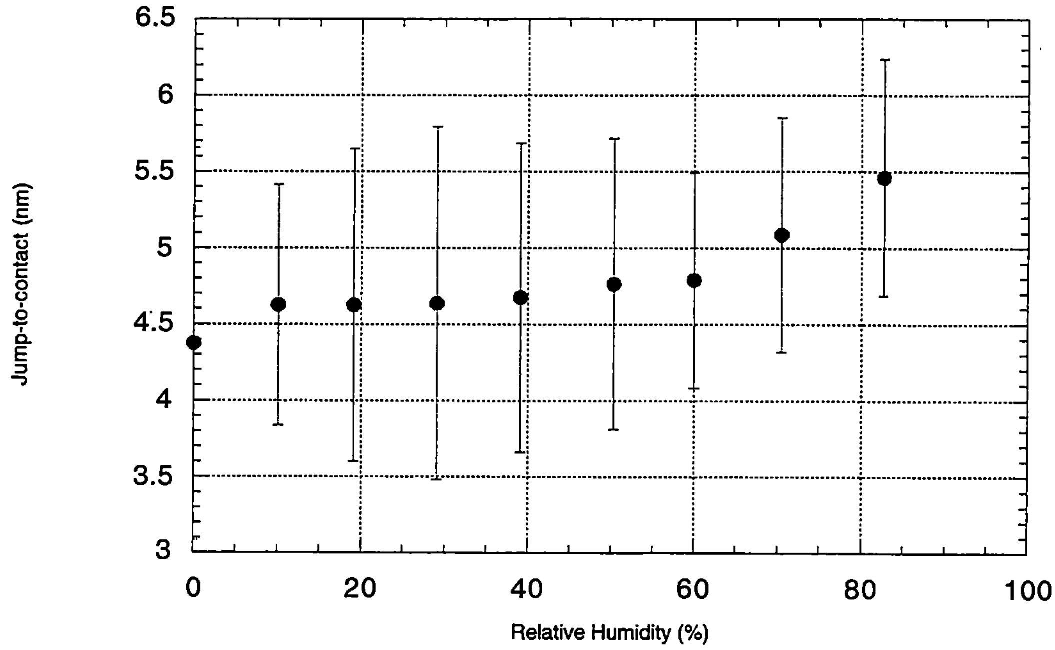

Figure VII-3. Jump-to-Contact Distance with Error Bars

Jump-to-contact distance (nm) as a function of relative humidity with error bars (one standard deviation) added. Note how smooth the curve is despite having such large error bars. This indicates that j-t-c distance varied considerably between individual measurements, but that the average distance remained very constant.

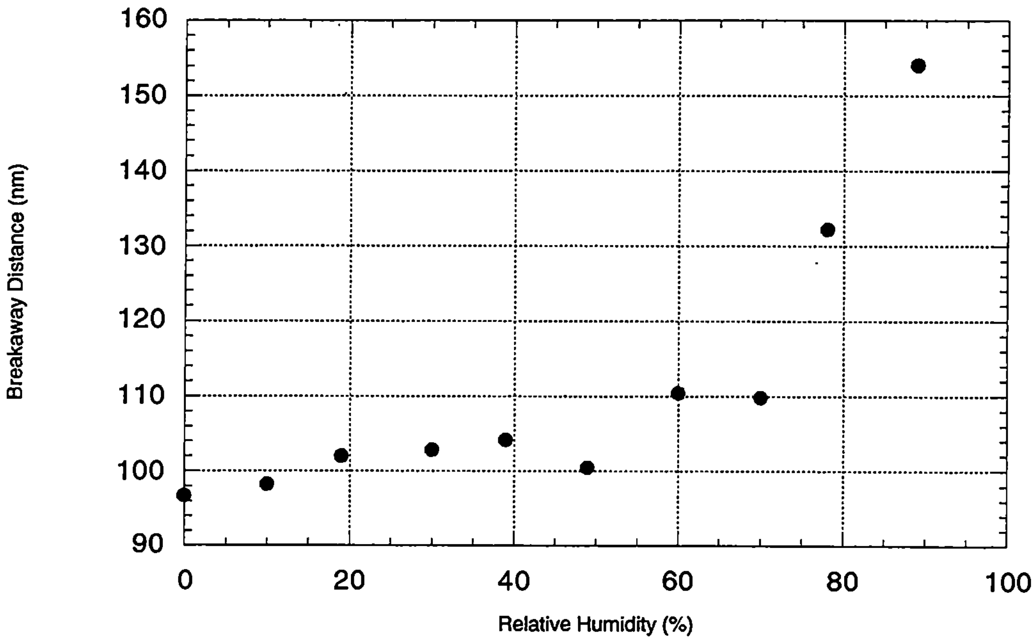

Figure VII-4. Breakaway Distance as a Function of Relative Humidity

Breakaway distance (nm) as a function of relative humidity. Although significant noise is present in the data, the regions described in the text can be clearly seen.

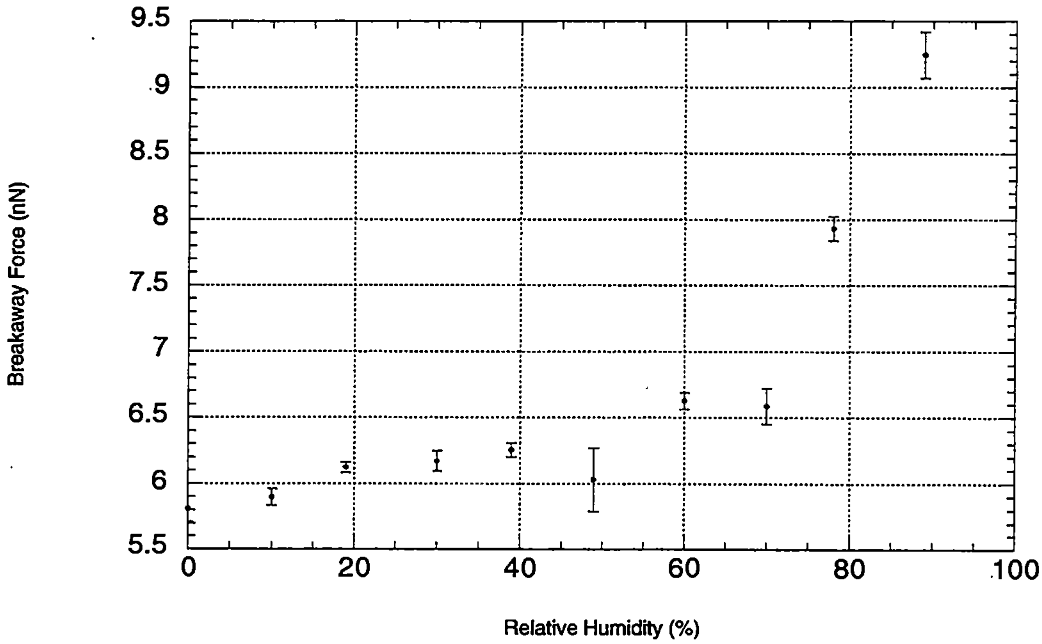

Figure VII-5. Breakaway Force with Error Bars

Breakaway distance converted to breakaway force as a function of relative humidity and with error bars (one standard deviation) added. Note the small error bars as compared with jump-to-contact distance but yet the noisier curve.

the jump-to-contact distance, namely the relative standard deviations were much smaller, yet there was much more noise apparent in the data points.

The exact point of transition to a rapid rise in the breakaway experiment is a bit fuzzy due to the presence of noise in the experiment. This experiment was repeated twice and the results obtained were all essentially the same. In particular, the transition to a rapid rise occurred between 60% and 70% RH in each case. One point of variation was the magnitude of the increase in breakaway distance between 20% RH and the point where the exponential rise began; this varied between four and ten percent.

7.4.3. Discussion and Analysis of Results

Both the breakaway and jump-to-contact distances showed increases that were approximately exponential in character before exhibiting something of a slow-down in the rate of distance change per unit relative humidity. Other breakaway distance data taken (not shown) supported this observation. The deviation from the earlier exponential rise was not dramatic, although it was there. This is attributed to a difference between the relative humidity at the point of measurement and at the actual tip-surface interface. The area immediately around the tip-surface interface was subject to heating by laser action as well as to other effects that had no impact on the area around the hygrometer. It is also common for very confined spaces (such as the area immediately around the point of contact between the cantilever tip and the adjacent surface) to have very different levels of relative humidity than adjacent, open areas and for this state of affairs to persist indefinitely (i.e. a state of equilibrium exists). Experiments by other researchers[39] show that at very high levels of relative humidity, substantial differences between measured relative humidity and actual relative humidity at the tip-surface interface exist. In particular, the relative humidity at the tip-surface interface tends to be much lower than was measured by the hygrometer. These differences did not show up at normal levels of relative humidity. An increasing difference between measured relative humidity and true relative humidity at very high levels of humidity would explain why the rise of tip-surface attractive force with relative humidity began to slow after showing an initial approximately exponential rise. This explanation is supported by the data garnered by Beaglehole and Christenson[32] in which the rate of increase of water layer thickness on a mica surface did not show a decrease at extremely high levels of relative humidity. As long as water layer thickness growth continued unabated, one would expect tip-sample attractive force to do the same. A slightly lower relative humidity level at the tip-sample interface than indicated by the hygrometer at high levels of relative humidity would easily account for the odd behavior of attractive force at high humidity, particularly given the rapid change in adsorbed water layer thickness with humidity in this region.

That cantilevers exhibit jump-to-contact behavior when very close to surfaces is well known[26,28,33-37]. A number of researchers have measured jump-to-contact distances for laboratory conditions, with distances generally being around 3 nm[17,37,38]. Theoretical jump-to-contact distances that were calculated on the basis of van der Waals forces have generally been less than 1 nm, suggesting that capillary forces are also very important[38]. While the exact jump-to-contact distances vary with tip and surface characteristics, measurements have been very consistent. The results reported in this thesis are in very good agreement with previous work in this area.

The most striking thing about the jump-to-contact is the strong similarity between its dependence on relative humidity and the dependence of breakaway distance on relative humidity (see Figures VII-2 and VII-4). Two differences are noticeable, however. The first is that the onset of the rapid rise in distance (and therefore force) occurred at a lower level of relative humidity in the jump-to-contact experiment than in the adhesion force experiment. The second is that the initial jump-to-contact rise was complete by the time 10% RH was reached, while the adhesion force did not complete this until around 20% RH.

Several things could explain the first difference. Noise in the breakaway force experiment could have prevented the rapid rise of the breakaway distance with humidity from being visible in its early stages. The breakaway force was subject to a lot more noise than the jump-to-contact experiment, so at first glance this seems very possible. However, the results of Thundat et al[37] tend to undermine this conclusion as no rapid rise in force/distance was observed although relative humidity levels used were as high as 70%. In contrast, the rise in the jump-to-contact distance certainly began by the time 50% relative humidity was reached. Another possibility is simply that water adsorption as a function of relative humidity was different in the two experiments. Adsorption of liquids on surfaces is strongly dependent on temperature and other variables; changes of a few degrees can substantially impact the adsorption curve[40]. This is a possible explanation for the second difference. Lastly, the water adsorption on the surface and on the tip could have been the same in both cases, but some other mechanism was at play. One possibility is that during tip-sample contact and subsequent separation triboelectrification or other contact charge transfer occurred[6]. The resulting potential difference between the tip and the sample may have allowed the liquid neck or meniscus that mediates the capillary force between the tip and surface to exhibit a transition to a rapid rise in force at lower levels of humidity than is ordinarily the case.

As mentioned before, there is a rapid rise in attractive force between the tip and sample at high levels of relative humidity that has not been reported in the literature until now. Until this point is reached, the results presented herein are essentially the same as those of Thundat et al[37]; what matters when comparing results of this kind is the shape of the force vs. humidity curve, as opposed to the magnitude of the force per se. Xu et al[31] found a very different force vs. humidity relationship, with the force falling at higher levels of relative humidity. The reason for the difference is unknown at this time. Beaglehole and Christenson[32] measured the thickness of the adsorbed water film on mica as a function of relative humidity. Their results showed an approximately exponential rise in film thickness at very high levels of relative humidity that was very similar to the rapid rise in tip-sample forces shown here. It is well known that the force exerted by the meniscus or liquid neck between the tip and the sample is proportional to the radius of the meniscus. Whether the meniscus is formed primarily from water on the sample surface as some suggest[26,36] or is formed by spontaneous condensation as others believe[17,18], the rapid rise in force observed at high humidity levels would in either case be expected. The increasing thickness of water on the mica surface, and most likely the tip also, would lead to a wider meniscus and thus a greater magnitude of force.

The seemingly odd relationship between standard deviation and variability between points for both jump-to-contact and adhesion force (see Figures VII-3 and VII-5) is easily explained. Jump-to-contact distances change a great deal from one jump to the next, but their average value is very steady; evidently it is not very inclined to drift due to environmental or other changes. Breakaway distances, on the other hand, generally demonstrate only very small variations from one pull-away to the next, except when a sudden change in surface or tip conditions can cause them to change substantially and rapidly. It is well known that water thickness on mica surfaces can vary substantially with microscopic puddles or islands being present in some areas while other areas remain relatively dry. These puddles can migrate across the surface[31]. Breakaway forces (and thus distances) can change greatly if such a puddle moves into the immediate area of tip-surface interaction or moves away from the area. We have observed sudden increases and decreases in breakaway force; in some cases forces would easily change by a factor of three or four. Other phenomena, not all of which are well understood at this time, are capable of causing significant changes in breakaway force. The point of all this is not that the specific reason for the substantial variability of breakaway force at different relative humidities can be nailed down in this case, rather that breakaway forces seem particularly susceptible to changes in environmental, surface, and tip conditions. Measurements of jump-to-contact distance showed substantially less variability between points than breakaway force. In general we have found this to be true, implying that average jump-to-contact distances are steadier than breakaway distances, despite showing greater variability from one individual measurement to the next.

7.4.4. Summary

Adhesion force and jump-to-contact distance demonstrate very similar responses to changes in relative humidity. When starting with dry air and increasing the humidity levels, typical behavior is a quick early rise, then a region of little change, and finally large rises at high levels of relative humidity. This behavior was found to be very consistent with the adhesion force/breakaway distance experiment being repeated on another two occasions. Some differences were found between the jump-to-contact and adhesion experiments regarding the points of transition between the initial rise and the region of approximately constant distance/force and again between the approximately constant region and the rapid rise found at high levels of relative humidity. The general shape of the response curve, however, was essentially the same.

Based on the voltage experiments detailed in Chapter 8, we would not expect changes in adhesion force due to variations in relative humidity to have a great deal of effect on the cantilever alpha detector. Changes in adhesion force resulted in variations in the breakaway distance of just over +/- 20% around the average value. It will remembered that variations in cantilever response parameters were theoretically predicted to go as one over the natural log of the distance to the surface, meaning that the parameters would change slowly with distance. This was confirmed via experimentation. Therefore, changes in adhesion force and breakaway distance due to varying levels of relative humidity ought to change cantilever response only fractionally.

7.5. Humidity and Water Bridge Characteristics and Behavior

7.5.1. Introduction

The phenomena just considered do not by any means constitute the only way in which humidity can affect the behavior of a microcantilever. One effect that was extremely important in the detection experiments was the behavior of the water bridge immediately subsequent to the occurrence of breakaway. (The behavior of the water bridge was in large measure a function of the amount of water on the surface and on the tip, which in turn was strongly influenced by relative humidity, as has just been discussed.) The water bridge was capable of affecting cantilever behavior by absorbing the potential energy released by the cantilever, by causing the cantilever to vibrate asymmetrically for a brief period after breakaway, and even by eliminating oscillatory behavior on the part of the cantilever entirely.

7.5.2. Water Bridge Dynamics

It will be recalled that the liquid drop present between the cantilever tip and the surface when they are in contact can act as a kind of sticky and flexible glue holding the two surfaces together as a result of the high surface tension of the water droplet. When a microcantilever tip breaks free of the surface, the water droplet, in the form of a meniscus around the tip-surface interface, does not immediately divide into two separate parts or all get carried away by one of the two surfaces. Instead the initial reaction of the droplet is to stretch out and form a kind of bridge or liquid neck stretching between the two surfaces. Of course, this structure has a limit as to how far it can be stretched before it becomes unstable and breaks. This point of instability is dependent on the surface tension of the water, the relative hydrophobicity or hydrophillicity of the two surfaces, and the thickness of the bridge. The water will seek to minimize its energy and the bridge will snap when continuing to maintain a minimized energy becomes incompatible with the persistence of the bridge.

Nevertheless, the water bridge is capable of persisting over a considerable distance of stretching, even to a distance of several hundred nanometers or more[39]. The bridge displays several interesting behavioral characteristics when stretched. The nature of these behaviors and their effect on the detector were investigated. We will examine these in their order of occurrence in a force calibration curve from the point of breakaway to the point of reattachment to the surface.

Breakaway curves can show substantial variation from curve to curve. See Figure VII-6 for an idea of what a typical breakaway curve looks like. Looking at the curve, it can be seen that an exponential decay mode and a damping oscillatory mode are present. As discussed elsewhere, the exponential decay mode can often be fit better using two separate components rather than a single exponential decay equation. This does not mean that two separate effects are at work here, but rather that the single one that is present is not a perfect exponential decay, but instead approximates one. This mode is in fact due to the effect of the water bridge on the cantilever. What is happening is that the water bridge is being steadily stretched out as the cantilever moves away from the surface and the effect of the bridge on the cantilever is to initially skew the motion of the cantilever towards the surface. The bridge can and does exhibit both an elastic and inelastic component when stretched. It exhibits an inelastic component in two ways. One way in a limited sense is that the stretching of the bridge does not reverse itself (at least not unless the restoring force of the cantilever is removed). A more general way is that if it is stretched quickly it will demonstrate significant internal friction and other processes that dissipate energy. It also exhibits an elastic component in that the cantilever can and

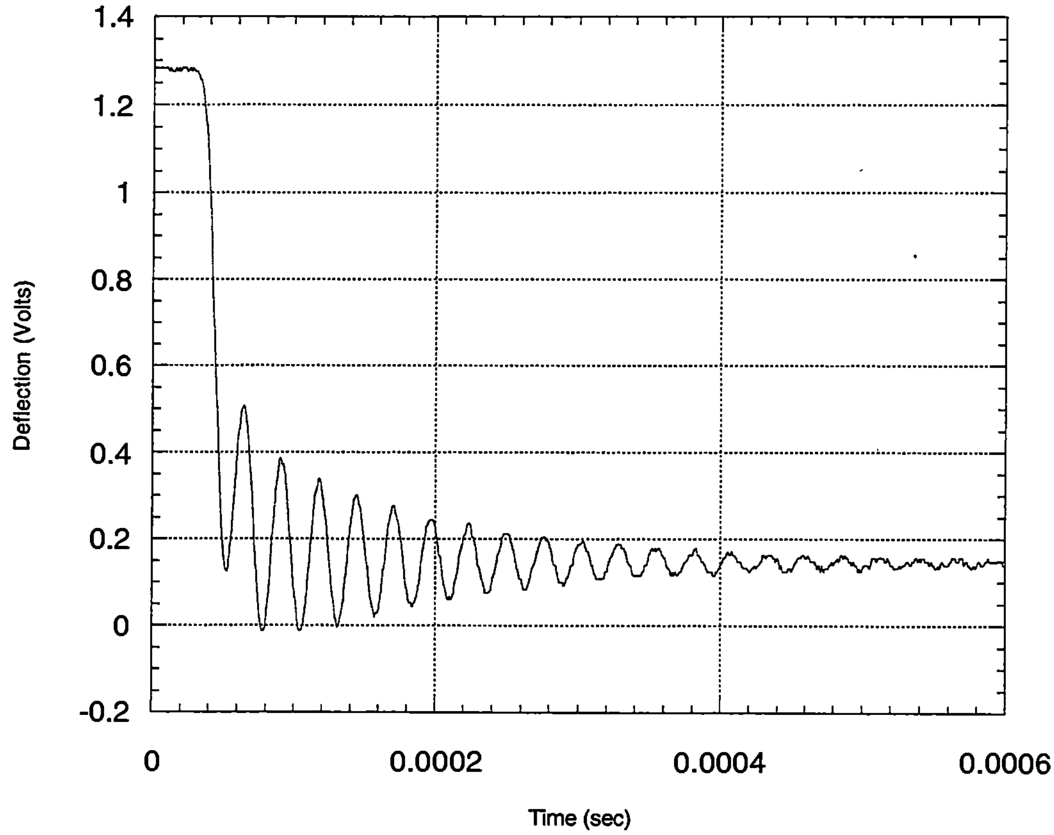

Figure VII-6. Another Typical Breakaway Curve

After breaking loose from the surface, the cantilever goes into harmonic oscillation as it becomes free to convert its potential energy (due to its restoring force and the fact that it was stretched) into kinetic energy. As time goes on, it damps out due to various internal and external friction processes. Note that although deflection is in volts, as in other cases this really represents deflection in nanometers (a conversion ratio can be found and applied).

usually does oscillate while the bridge is still in place with the bridge acting as a kind of rubber band. The upshot of both of these effects is that when a water bridge is stretched out from a surface a long distance it acts in many ways like a rubber band that is somehow steadily getting longer (at least as long as sufficient force is applied to it).

7.5.3. Effect on Breakaway Curves

The skewing of the motion of the cantilever occurs in this way. As the cantilever moves closer and closer to an undeflected position, its restoring force becomes less and less. The water bridge, however, is still exerting a force trying to pull the cantilever back towards the surface. Besides the simple restoring force of the bridge, other forces come into play, especially if the bridge is stretched very quickly. Excessively rapid expansion of the bridge greatly increases the drag it exerts on the cantilever. If the distances are right, the force exerted by the bridge will approach that which is exerted by the cantilever as the cantilever begins to approach its zero deflection point. In this case, the cantilever will continue to move towards its zero deflection point, but the rate at which it does so will decrease. This is because the restoring force is no longer sufficient to stretch the bridge at a rate beyond what could be termed its elastic stretch rate where inelastic effects come into play (because this requires so much energy). The end result is that the rate of motion of the cantilever towards its zero deflection point is substantially reduced, giving the appearance of a flattening off on an oscilloscope trace. This becomes steadily flatter as the cantilever approaches its zero deflection position (or at least the point at which the water bridge and restoring forces cancel each other out). The degree to which skewness occurs is dependent on such factors as the breakaway distance and the amount of water in the bridge. (The breakaway distance is not solely a function of the capillary force, of course, or even always predictable from it; forces such as the van der Waals can vary from case to case.) For an example of a breakaway curve showing greater amounts of skewness, see Figure VII-7.

Several comments on the behavior of the water bridge need to be made at this point. The water bridge is capable of persisting a short period of time after it becomes unstable due to excessive stretching. Its restoring force drops off roughly as the inverse of the square root of the separation distance. The equations for the force between the tip and the surface are quite complex, but an approximation can be made in this way. The restoring force of the water bridge or neck is proportional to its radius[41]. If we consider the water bridge as being a cylinder extending between the apex of the tip and the surface (this works well for distances of tens of nanometers, although it breaks down at small distances), we find the following

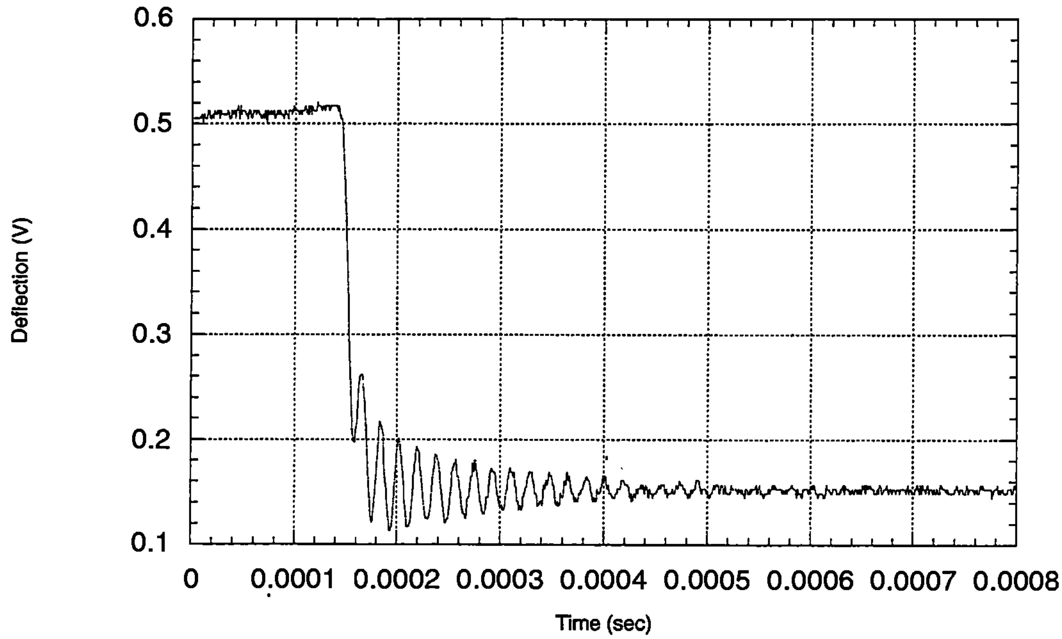

Figure VII-7. Breakaway Curve with Much Skewness

A breakaway curve showing a greater degree of skewness than the last example. Note that although deflection is in volts, this represents some deflection in nanometers.

equation for the volume, V, of the water bridge.

$$V=\Pi r^2 x.$$

If we assume that the volume of the water in bridge is constant (a reasonable assumption given the speed of breakaway[42]), we find the relationship between the radius and the length to which the bridge is stretched.

$$r=\sqrt{\frac{V}{\Pi x}}$$

Since the volume is constant and the force exerted by the bridge is proportional to the radius of the bridge, we find get the following relationship between the force and the length to which the bridge is stretched.

$$F(x)\approx x^{-1/2}$$

Thus when the cantilever initially breaks away from the surface, the restoring force of the water bridge falls fastest at first, slowing down as the length of bridge extension increases.

Returning to the effect being described, the water bridge has been stretched a sufficient distance that the force it exerts is small and does not have much of an effect on the cantilever. Normally the bridge has become unstable by this distance and will simply break after a short period of time. When this break occurs, the change in force on the tip is so small that no significant and measurable changes in the cantilever (such as deflection) are seen.

One could imagine that variations on this type of water bridge behavior might be seen. For example, a combination of circumstances might occur in which the bridge might stretch out but not break due to the breakaway distance being less than the critical distance at which the bridge becomes unstable. It is also possible that a water bridge simply might not form either due to the lack of mobile water on the surface. Both these cases can and do occur and we can learn much from them. They produce breakaway curves that are distinctly different from the regular case in which the water bridge breaks shortly after breakaway. In the case in which the water bridge remains in place, a difference is also seen in the characteristic shape of the jump-to-contact curve.

7.5.4. Different Bridge Behavior Scenarios

It has been seen that a typical breakaway curve shows some skewness (an exponential decay function) superimposed on the vibration of the cantilever (a damped oscillatory mode). (See Figures VII-6 and VII-7.) In cases where the water bridge remains in place, the characteristics change markedly. (See Figure VII-8.) The first major difference is the lack of any substantial oscillatory motion on the part of the cantilever. Instead, the cantilever shows only an exponential decay mode after an initial acceleration in velocity immediately after breaking loose

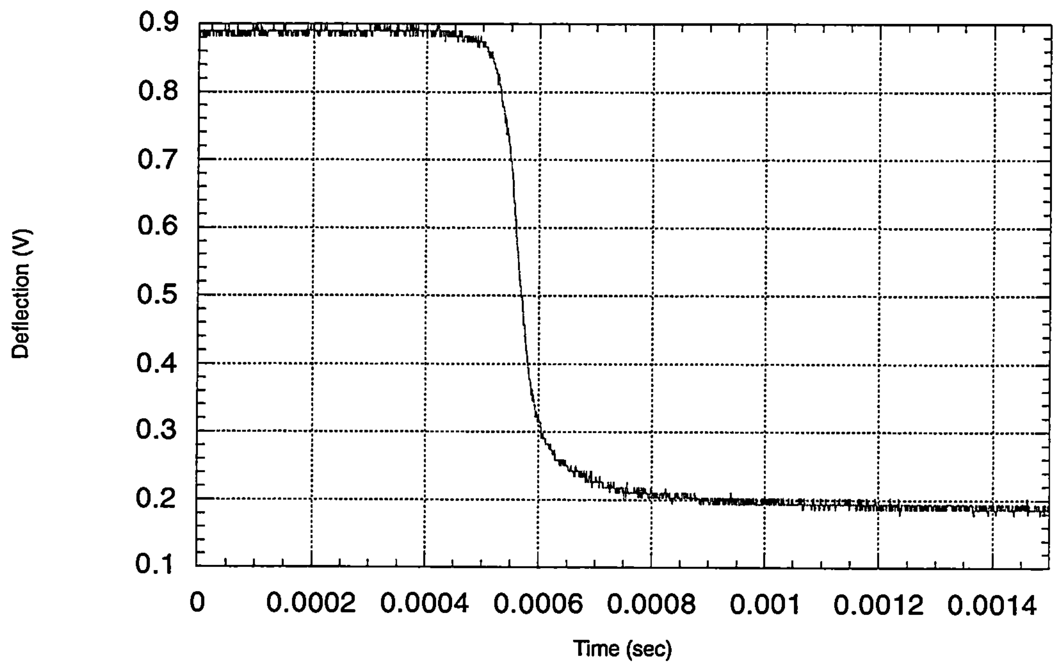

Figure VII-8. Breakaway Curve without Severing of Water Bridge

A breakaway curve in which the water bridge is never severed. Instead the bridge stays in place between the tip and the surface and absorbs all of the potential energy the cantilever gives up after it breaks free of the adjacent surface.

from the surface. The duration of time over which this mode is plainly visible to the eye is rather greater than in the common kind of breakaway curves. (Compare Figure VII-8 with Figures VII-6 and VII-7.) This is indicative of the fact that the water bridge is persisting and is continuing to affect the cantilever. This is because the bridge has not been stretched beyond its point of instability and thus has not suffered any loss of structural integrity. With the restoring force of the cantilever diminishing quickly as its deflection approaches zero, the velocity of the cantilever slows down considerably as the cantilever has little besides its momentum to keep it moving and its kinetic energy is being converted in part to potential energy in the water bridge. (The bridge has something analogous to a spring constant, except that it is not constant and varies greatly with distance of stretching.) At some point the cantilever settles down. Cantilever to surface distance, however, is still changing as the surface is moved relative to the cantilever, first away from it and then towards it as the force calibration curve proceeds.

The effect on the breakaway curve is not the end of the story, however. The continuing presence of the water bridge between the tip and the surface affects the characteristics of the jump-to-contact as the cantilever again approaches the surface during the force calibration curve. (See Figure VII-9.) As the cantilever tip is moved near to the surface, the bridge becomes much less compact and less

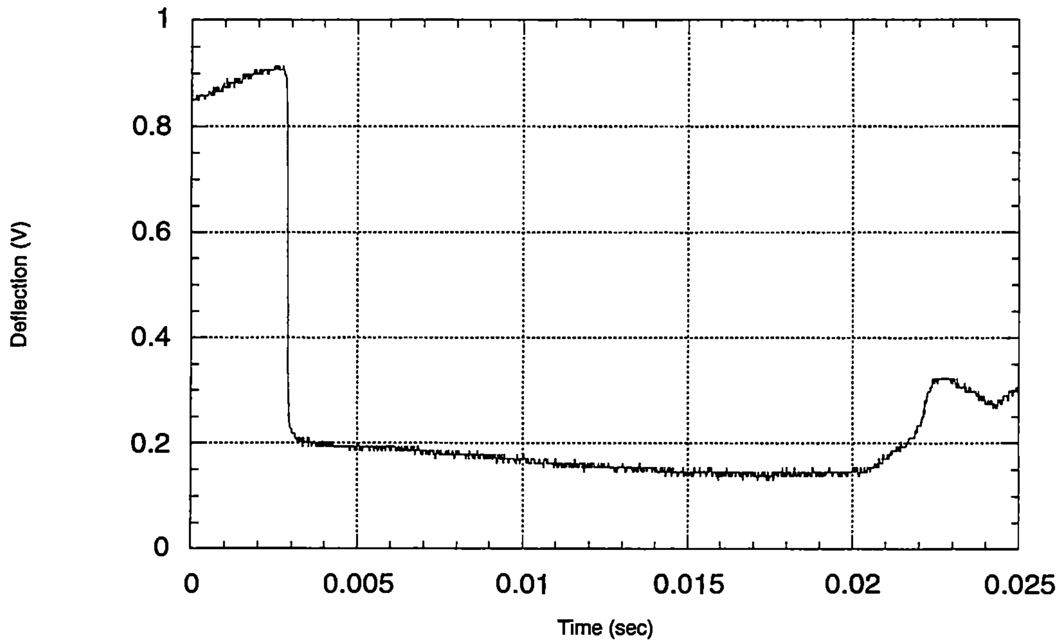

Figure VII-9. Force Calibration Curve with Intact Water Bridge

Another picture of a case in which the water bridge is not severed. On the left, breakaway occurs and the cantilever moves towards equilibrium – quickly at first and then slowly. On the right, the cantilever moves up to meet the surface as it returns, making contact with it and then following it as it is moved as part of the force calibration curve. Note how the presence of the bridge affects the curve by causing cantilever motion long after breakaway and the change in the jump-to-contact behavior especially.

stretched. As the cross-sectional area of the bridge becomes larger, the restoring force trying to pull the two ends of the bridge together becomes greater. The result is that the force becomes strong enough to start producing measurable cantilever deflection in the direction of the surface. This further increases the tip-surface force produced by the bridge, causing the cantilever to deflect more. This motion is far more drawn out than a regular jump-to-contact, taking a period of time that is literally orders of magnitude greater, but it usually culminates in a final rapid jump similar to the type examined previously.

Other kinds of variations on water bridge behavior are sometimes seen. One is one in which a kind of equilibrium is temporarily achieved between stretching of the water bridge and deflection of the cantilever. (See Figures VII-10 and VII-11.) This occurs when the breakaway distance is very nearly the critical distance for bridge instability. As the cantilever approaches its zero deflection point, it comes into a short-lived equilibrium where it stops a few nanometers short of its zero deflection point. For a brief period, the bridge force and the cantilever restoring force almost exactly offset each other. This kind of equilibrium has only been observed around the critical distance, suggesting that perhaps some additional energy loss to the water bridge is required at the point of instability beyond what would be expected simply on the basis of stretching the bridge in order to finally

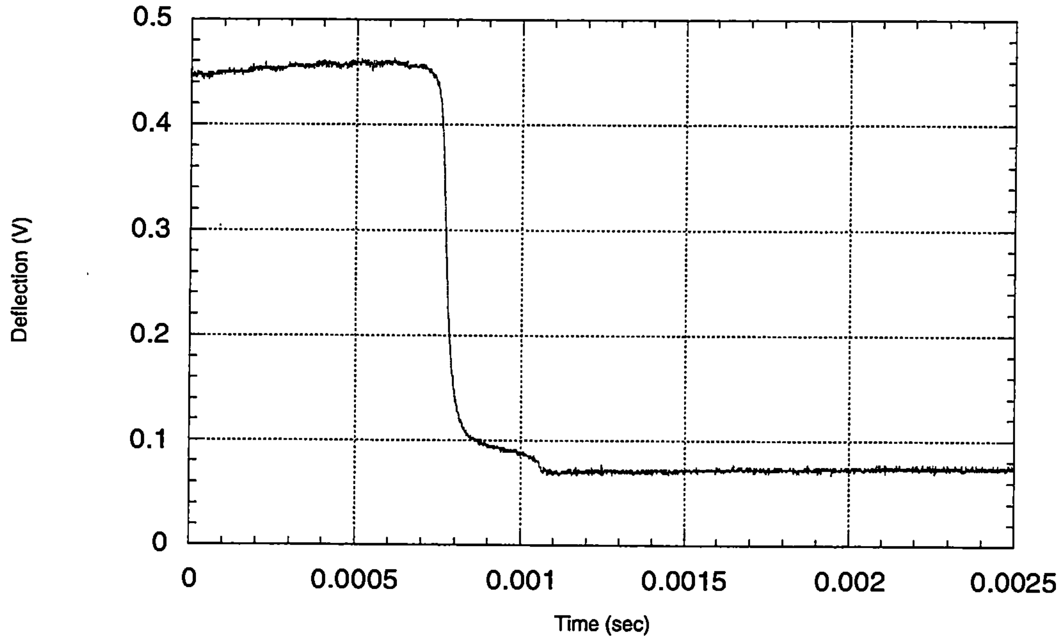

Figure VII-10. Short Temporary Equilibrium

A breakaway curve in which a temporary equilibrium is briefly achieved between the cantilever and the water bridge.

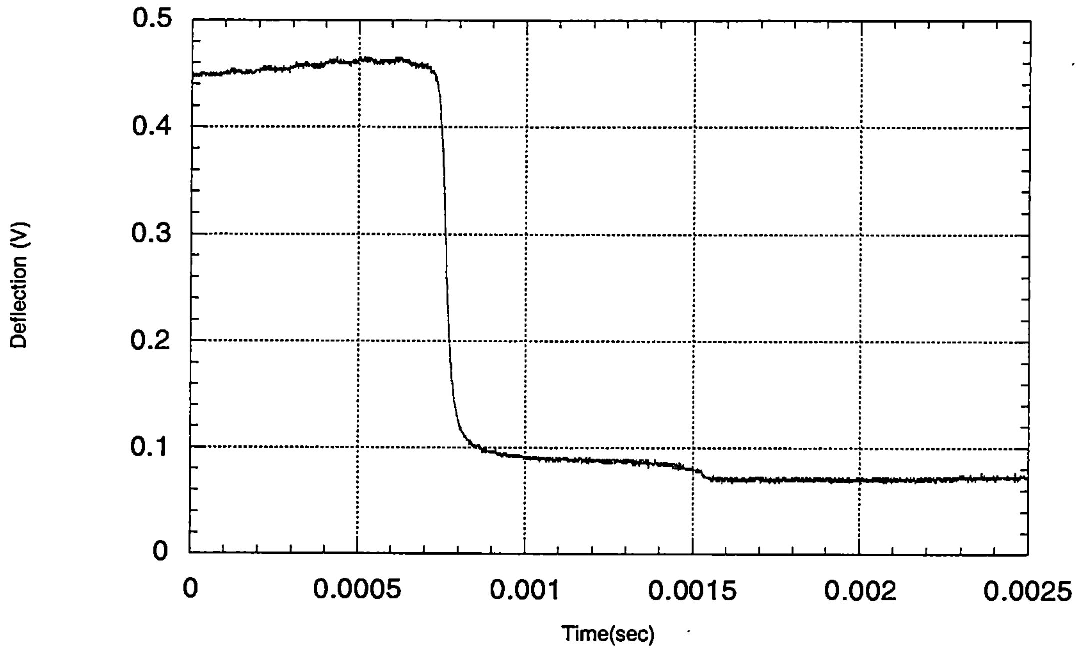

Figure VII-11. Longer Temporary Equilibrium

Another case of equilibrium, this one lasting longer.

sever it. These temporary equilibriums are of variable duration. (Compare Figures VII-10 and VII-11.) During this time, the surface has continued to be retracted as part of the force calibration curve and the bridge at some point is stretched beyond its critical distance and starts to break, producing what looks like a second breakaway. This second breakaway demonstrates many of the same kinds of characteristics as a regular breakaway including oscillation and even a limited degree of skewness. This further reinforces the idea that after the bridge has passed its point of instability, it can continue to affect the cantilever for a short period of time as it breaks. Breakaway curves that demonstrate a temporary equilibrium that is lost show the same jump-to-contact behavior as is seen in the most common type of force calibration curve (one in which skewness is present in the breakaway curve, but nothing else), that is, a sudden jump with no drawn out motion. This suggests that the water bridge does in fact become completely severed in these cases.

It is normally the case that the breakaway distance is fixed for a given situation, being a function of such parameters as the quantity and mobility of water on the sample surface (which is the collector plate in an actual detection experiment) and the cantilever tip. It is possible, however, to manipulate this distance slightly by altering the parameters of the force calibration curve such as rate, start distance, and distance covered. Doing so produced some interesting results. The start case was one in which a temporary equilibrium was achieved, as detailed above. It was assumed that the breakaway distance was right around the critical distance and the lack of cantilever oscillation was the result of all the potential energy released by the cantilever being lost to the bridge. The breakaway distance was increased slightly, resulting in it being a bit greater than the critical distance. The result was that cantilever oscillations appeared (see Figure VII-12) which is what one would expect if the breakaway distance were greater than the critical distance. The amplitude of the oscillations relative to the magnitude of the breakaway distance was very small; indicating that very little cantilever potential energy was converted into kinetic energy in the form of oscillatory behavior.

The different forms of breakaway and jump-to-contact curves were examined, and, to the limited extent that it was possible, were manipulated in order to find out how they related to the state and behavior of the water bridge. Several things were concluded. The first was that during a typical breakaway curve, the breakaway distance most often exceeded the maximum distance to which the water bridge could be stretched without it becoming unstable and disintegrating. This accounts for the fact that most breakaway curves observed looked rather like Figure VII-6, with moderate oscillation size and pronounced skewness early on. If the

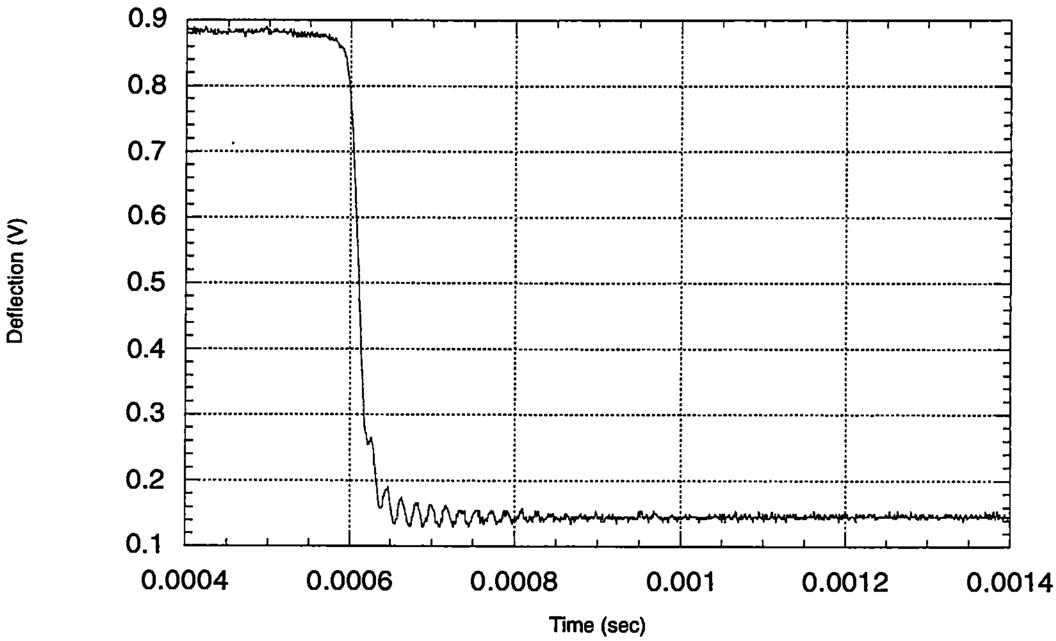

Figure VII-12. Breakaway Curve with Small Oscillations

A breakaway curve produced when the breakaway distance was just slightly larger than the critical distance to produce water bridge instability and breakup. Note the very small oscillation amplitudes resulting from the loss of nearly all the cantilever's potential energy to the water bridge.

breakaway distance was less than what was required to sever the bridge, only a very long-lived exponential decay component was observed as part of the cantilever's behavior with no real oscillatory behavior. If the breakaway distance was almost the same as the critical distance, a temporary equilibrium could be achieved, possibly followed by very small amplitude oscillatory behavior. As the breakaway distance became increasingly greater than the critical distance for bridge breakup to occur, cantilever behavior showed two general components, a exponential decay component (skewness – in some cases two exponential decay components produced a slightly better fit) and an increasingly large damped harmonic oscillatory component. The damped exponential component did not signify that the water bridge necessarily persisted indefinitely but rather that after exceeding its critical distance and becoming unstable it took some finite period of time to disintegrate during which time it could exert a limited amount of force on the cantilever. This last case where the breakaway distance exceeded the critical distance for instability was what was usually observed in a breakaway curve. Confirmation of the presumed state of the water bridge (intact throughout a force calibration curve or severed) in these different cases was provided by the behavior of the cantilever during reattachment to the surface (jump-to-contact). Distinctly different patterns of cantilever behavior occurred during reattachment depending on whether or not the water bridge was intact and thus whether the force it was capable of exerting would come into play or not.

The change in the amplitude of oscillations relative to the breakaway distance can be explained in this way. Stretching out the water bridge to its maximum length before it began to disintegrate required a substantial infusion of energy into the water bridge system (although tiny, substantial relative to the energy of the cantilever). This energy came from the release of cantilever potential energy (resulting from its spring-like behavior). Energy left over for the cantilever after the water bridge had been effectively dealt with went into producing harmonic oscillations. Thus, for cases where the breakaway distance was only barely more than the critical distance, the harmonic oscillations were very small and for cases where the breakaway distance was much larger, the oscillations became much larger relative to the breakaway distance. Some very small oscillation amplitudes as a fraction of the breakaway distance were only around 0.02. Typical breakaway curves (breakaway distance much larger than the critical distance for instability) generally ran between 0.15 and 0.3, depending on the conditions in that particular experiment.

7.6. The Effect of Sloped Surfaces on Breakaway Curves

7.6.1. Slope Effects

The cantilever-surface geometry was discovered to be very important as it could drastically affect the behavior of a cantilever immediately after breakaway from a surface. Under normal conditions, such as in AFM imaging, cantilevers are used on flat or almost flat surfaces. The cantilever itself is held in the holder at an angle of approximately 10.5°, meaning, of course, that the tip deviates from being at right angles to the surface by that amount. As a ball bearing was used as the collector plate, most of the available surface area on the ball bearing was not level or nearly level relative to the tip of the cantilever, although any individual small area was flat in the sense of being in the same plane as far as the tip was concerned. (A cantilever's tip is so small that the surface appears to be flat to it, although the plane of the surface might of course be at an angle relative to the tip.) This being the case, the cantilever may behave differently depending on its position on the surface of the ball bearing if the tip to surface angle has an effect on its behavior during and immediately subsequent to breakaway.

As it turns out, the tip to surface angle has a major effect on cantilever behavior. When a cantilever breaks away from a surface, it goes into harmonic motion as potential energy is exchanged for the energy of the oscillatory motions.

(A cantilever being rather like a spring, it will vibrate if displaced and then released to move freely.) Studies of a large number of cantilevers where the surface used was level (and thus at a 10.5° angle to the vertical direction of the cantilever's tip) always produced vibrations at a single frequency (the main resonance frequency of the cantilever). However, if the tip-surface angle was different from this, it was possible for multiple resonance frequencies to become excited. There did not seem to be any hard and fast rules for determining whether or not multiple resonances would become excited (indeed, the amount of water present at a particular site could make the difference between single frequency and multiple frequency excitation), but some general principles can be sketched out. If the surface was at a substantial slope going crossways across the cantilever (perhaps twenty degrees or more) or in rare cases if the tip-surface angle was very large going lengthwise along the cantilever, multiple frequency excitation became possible. Generally, the greater the angle, the greater the likelihood of the excitation of multiple frequencies, particularly for an angle across the cantilever, rather than down its length.

The following graphs (Figures VII-13, VII-14, VII-15, and VII-16) show breakaway curves produced on slopes with one graph produced on a flat surface (the first one) for comparison. As can be seen on the first graph, Figure VII-13, breakaway curves produced on flat surfaces show a single resonance frequency. It

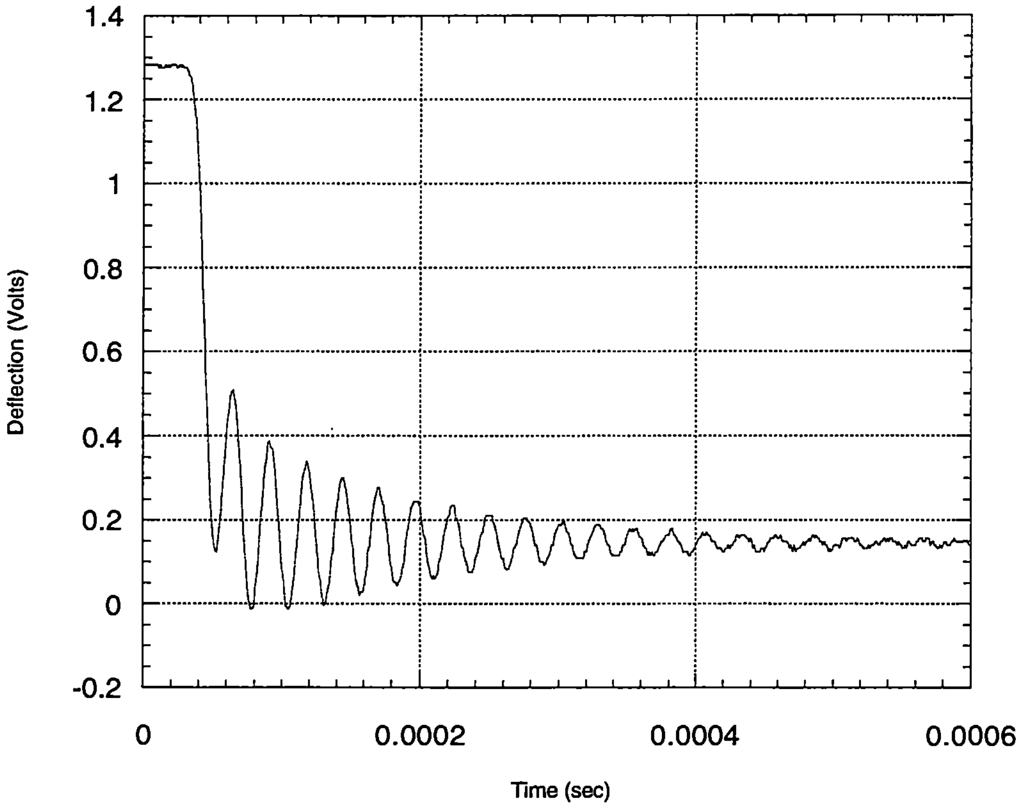

Figure VII-13. Breakaway on a Flat Surface

A breakaway curve produced on a flat surface, resulting in the excitation of a single resonance frequency.

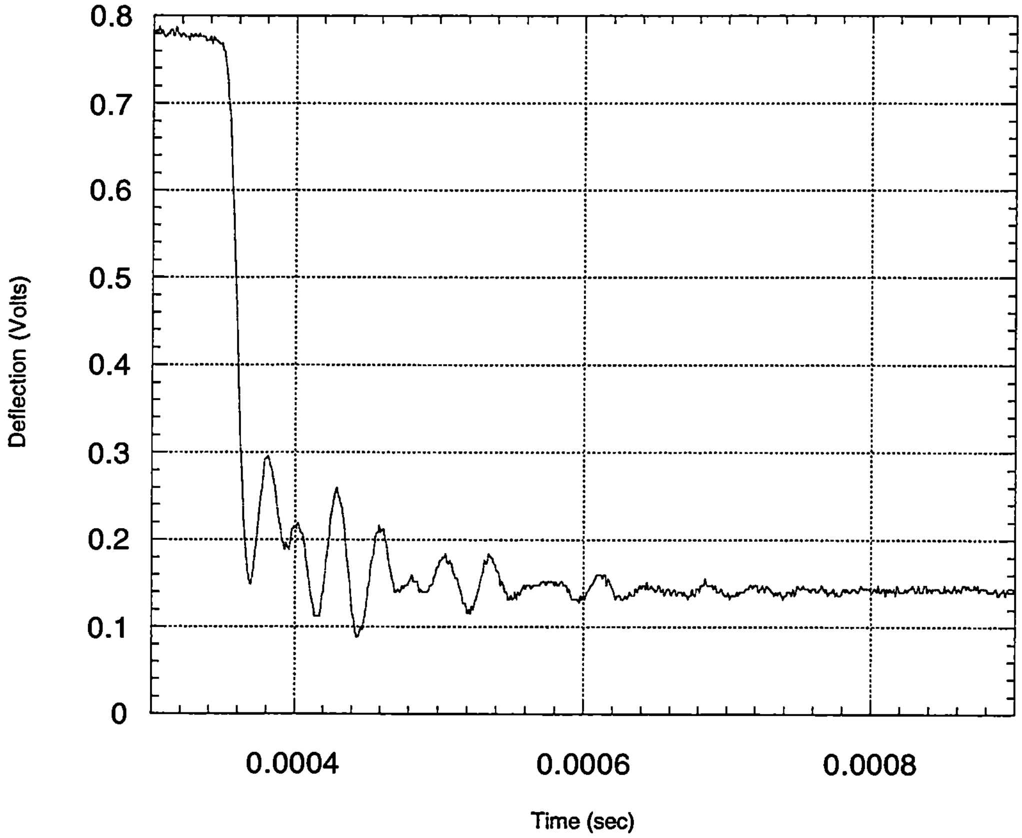

Figure VII-14. First Example of Breakaway on a Sloped Surface

A breakaway curve produced on a sloped surface, causing excitation of multiple resonance frequencies.

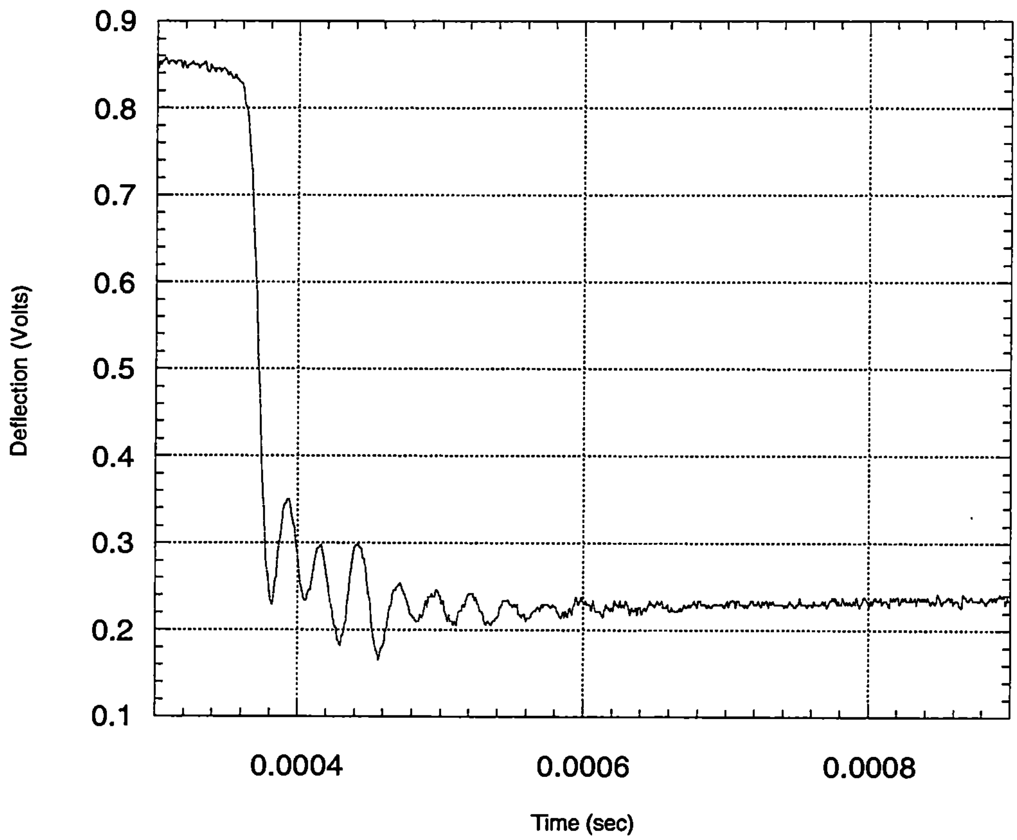

Figure VII-15. Second Example of Breakaway on a Sloped Surface

Another breakaway curve produced on a substantially sloped surface, showing the excitation of multiple resonance frequencies.

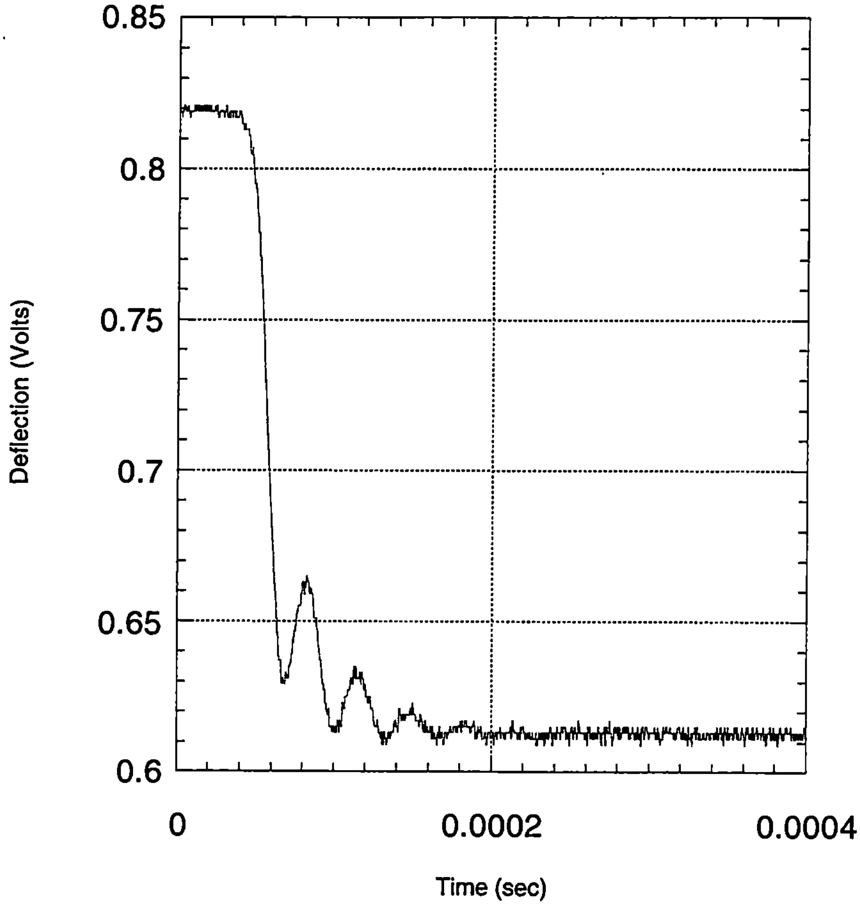

Figure VII-16. Third Example of Breakaway on a Sloped Surface

Another breakaway curve produced on a highly sloped surface - this time sloped from front to back, not side to side. Note the very rapid damping resulting from surface-tip friction mediated by the water bridge even after the point of breakaway.

is worth noting that in general the graphs with multiple frequencies excited (Figures VII-14 and VII-15) tend to look very similar with the same frequencies having seemingly become excited in approximately the same proportion to each other. The damping rates also visibly change, particularly in Figure VII-16. More data that shows the similarities in frequency distribution was taken but is not shown here.

Both the important effects (multiple harmonic resonance excitation and rapid damping) occur only in this particular situation (when breaking free from a highly sloped surface). Why should the angle of the cantilever relative to the surface affect the cantilever's oscillatory motion in this way? The fact that both occur on angled surfaces would suggest that the mechanisms of occurrence might be related.

7.6.2. Mechanisms of Slope Effects

The first phenomenon, the excitation of multiple resonance modes, is explainable in this way. Typical cantilever tips have a radius of 15 to 20 nm at the apex of the tip. When in contact with a flat surface, a circular contact zone occurs approximately centered around the apex. (Figure VII-17.) The effective contact zone is rather larger than would be expected purely on the basis of the contact between the two surfaces. The reason for this is that, except in exceptional circumstances such as ultrahigh vacuum, a tiny drop of water in the shape of a

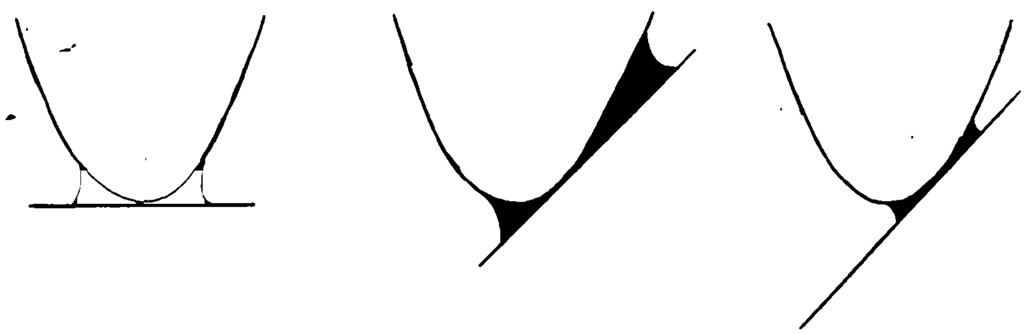

Figure VII-17. Effect of Slope on Tip-Surface Effective Contact Zone

On the left, a flat surface results in a symmetric contact zone centered on the apex of the tip. In the middle, a steep surface relative to the tip results in an effective contact zone that is greatly skewed to one side of the tip. The skewness is greater than this picture suggests as the width of the contact zone coming in and out of the page is higher on the upper side than on the lower. If the cantilever is slowly pulled away from the surface, the cantilever will deflect with some rotation about the center of the contact zone, causing side-to-side twisting and thus torsional vibration when breakaway occurs. Low humidity will result in a small effective contact zone as shown on the right. The center of this zone is closer to the apex of the tip than in the previous case, although this is not necessarily obvious in a side-view.

doughnut sits around the tip of the cantilever and acts like glue holding it to the surface. (The water bridge/meniscus is discussed in much greater detail elsewhere.) When the tip is angled crossways to the surface, the effective contact zone will be skewed to one side (the side which is closest to the surface by virtue of being angled towards it). When the cantilever is begun to be pulled away from the surface, the cantilever will twist as the restoring (spring) force is not quite opposite in direction to the attractive force between the tip and the surface. By the time the tip breaks free of the surface, the cantilever may well have been substantially twisted, causing it to be set into oscillatory lateral motion. (See Figure VII-18.) Lateral resonance modes will occur at different frequencies from the main harmonic and will show up as oscillatory motions of the tip; they are not distinguishable from regular resonance modes on the basis of waveform, or at least not when the two types of signals are mixed together.

That the skewing of the contact zone is responsible for these other resonance modes and that said modes are the result of lateral excitation of the cantilever is supported by a couple of observations. One is that there was a zone of angles over which multiple resonances might or might not occur. Specifically, they might consistently occur one day and completely fail to occur some days later. Changes in relative humidity were implicated in this. Evidently the amount of water present

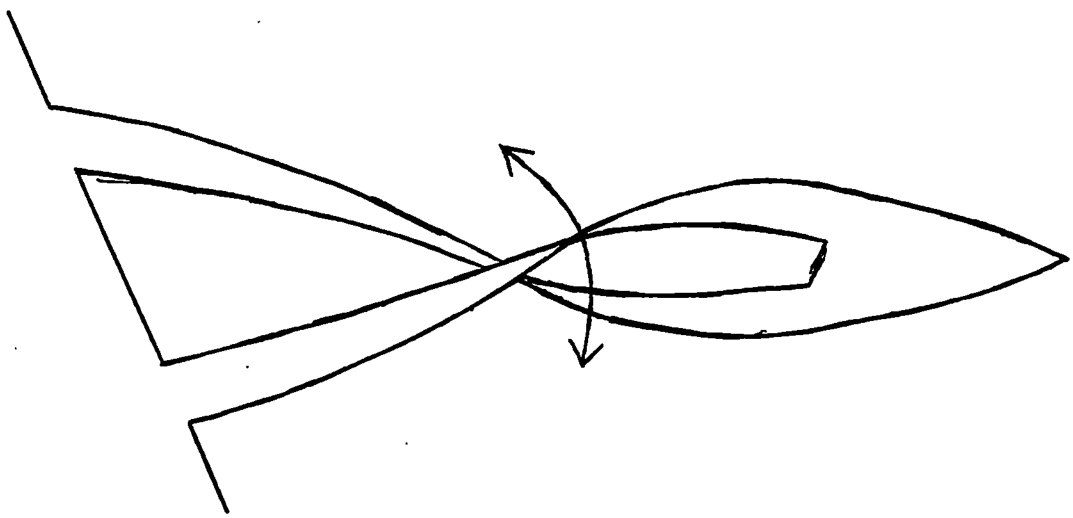

Figure VII-18. Torsional Vibration of a Microcantilever

Torsional (side-to-side) vibration of a triangular microcantilever. Such modes can become excited when operating against a surface with a significant sideways slant.

around the tip-surface contact was changing, making the effective contact zone larger or smaller. One would expect that the greater the amount of water present, the greater the skewing of the effective contact zone, and conversely the lesser the amount of water present, the closer to being centered about the tip the zone would be. This would in turn help to determine (the other main factor being the angle of contact) whether or not lateral resonance modes were excited or not. Another clue is that bar cantilevers have not been observed to demonstrate this multiple resonance excitation when breaking away from a sloped surface. (It is possible that higher order resonance modes that were back and forth rather than side to side could become excited during a sloped breakaway, although this is not specifically known to have occurred. If this did occur, it would be possible for bar cantilevers to demonstrate multiple resonance excitation.) Triangular cantilevers can easily support lateral resonance modes, whereas bar cantilevers cannot[39].

The final graph of the four breakaway curve graphs just shown (Figure VII-16) demonstrates a different kind of behavior – very rapid damping. This particular trace was acquired when the cantilever was on a very steep part of the surface of the ball bearing. The ball bearing rose up ahead of the tip of the cantilever at an angle of around forty-five degrees. With the cantilever at an angle of approximately ten degrees already, the total angle was about fifty-five degrees. It is evident that a lot more energy was given up immediately subsequent to breakaway than would normally be the case, thus the rapid damping. The effect leading to this behavior is similar to effect causing lateral resonance excitation for triangular cantilevers when breaking away from a sloped surface. In this case, the effective contact zone between the cantilever tip and the surface is skewed towards the front of the cantilever. Immediately subsequent to breakaway as the cantilever moves away from the surface, the water bridge present between the tip and the surface is stretched. The water bridge exerts a restoring force when it is stretched, somewhat like a spring, but different in that the force decreases as the water bridge is stretched out more and more. For tip motion so far from being perpendicular to the surface, the horizontal component of the tip motion relative to the surface is greater than the vertical component. Were the surface end of the water bridge to move along the surface following the motion of the tip, the length of the water bridge at any point in time after breakaway (but prior to the final severing of the bridge) could be substantially reduced. This is in fact what happens. The dragging of the water bridge along the surface involves substantial amounts of friction. The energy required to overcome this friction is supplied by the cantilever; thus the very rapid damping of the cantilever's oscillatory motion. This effect was only observed at a very steep angle. Experiments with side-to-side angles never reached the extreme angle used in this case and such big increases in damping rates were not observed. Presumably if the side-to-side angle were large enough, this phenomenon of extremely rapid damping would be observed.

The effects that can occur when the cantilever is used against a sloped surface are important for a couple of reasons. If the method employed for detection of alpha particles is not modified, care will have to be taken to ensure that the cantilever is positioned in such a way that only a single mode becomes excited during breakaway. It is quite difficult to analyze multiple modes and pick out individual frequencies and their amplitudes, especially when significant damping is occurring. Multiple mode excitation and rapid damping have the potential to affect AFM imaging. Triangular cantilevers are most often used for this application and surfaces are sometimes quite rough, making them susceptible to this kind of interference. Tapping mode AFM usually keeps the cantilever tip in contact with the surface (via the water bridge) as it only vibrates an AFM one or two nanometers and thus would not normally be very susceptible to oddities in cantilever amplitude, but it could be detrimentally affected in particular circumstances. Force calibration curves are obviously prone to being affected as we have shown, although the actual breakaway curves, the only part directly affected, are generally not used that often. Situations that may result in multiple mode excitation, namely when a surface is substantially sloped relative to the tip of a cantilever, are also prone to other effects such as noisy data caused by roughness of the surface and the slipping of the tip.

Chapter 8

Suggestions for Future Work

As was seen in Chapter 6, the investigations conducted during the course of completing this thesis should in no way be regarded as having exhausted all avenues of investigation in the area of microcantilever-based radiation detectors. Rather, new and interesting possibilities have opened up, having been seen to likely be fruitful areas of potential research. Initial work on radiation dosimetry shows it to appear promising, despite the technical problems encountered in the dosimetry experiments. Besides experimentation with radiation, several areas of cantilever physics and chemistry also merit further investigation.

Several specific methods for radiation detection are worth mentioning here. Detectors based around the charged plate principle show promise for sensitivity over a wide range of fluxes, particularly if the cantilever electric field detector were of variable voltage. Investigations should focus on two cases. The first is the case in which the plate is already charged to a certain level and then caused to decline in voltage as a result of cancellation by ion showers induced by incoming radiation.

The second is when the collector plate is contained in a vacuum system designed to prevent migration of ions to the plate, the actual charged particles being the only factor affecting the charge on the plate. (Other than when the voltage on the plate is reset to either ground or some particular level of charging, of course.) Investigating both of these designs would be worthwhile from the standpoint of moving towards a practical, field detector, rather than one operating only on a laboratory bench top. In the course of this work, it would be desirable to experiment with different types of cantilever setups such as using cantilevers to ground the collector plate or to charge it, as well as other variations that could be used.

Dosimetry experiments should have a high priority placed upon them, in large measure because they are the most likely means by which to produce strong results as quickly as possible. Two issues are problematic in dosimetry; identifying radiosensitive materials (most work has revolved around making materials less susceptible to radiation damage, not more so) and coating those materials on a cantilever (surface coating is a whole branch of its own in chemistry). Once these two problems are overcome, the rest of the work is fairly straightforward. Experimentation with DNA as a coating for cantilever dosimeters is highly recommended by this author as DNA can be easily coated onto gold-coated cantilevers and it is highly sensitive to radiation. Catastrophic entanglement is a potential problem, and care will have to be taken in order that sufficient mobility of severed DNA strands is maintained without compromising sensitivity (by making the DNA fractional mass of the system too low). Attachment of severed portions of DNA to the cantilever surface is also a potential problem. It should be possible to avoid this by choosing DNA for coating that is strongly adhesive to gold only at one end of the strand and not at the other end or anywhere in between when the strand is cut into two. If necessary, the solution (DNA-coated cantilevers must be kept in liquid except for brief periods during transfer) can be chemically treated in order to reduce DNA-gold bonding strength[43]. Other coatings worth investigating include silver halides and polymers used in x-ray lithography.

Areas of cantilever physics and chemistry of interest for research are many. Specific ways in which this work could be continued relate primarily to the behavior of the water bridge that can form between the cantilever tip and an adjacent surface. As an example, measurement of cantilever resonance frequency shifts when the water bridge is in place for different distances of bridge extension would provide a measure of the restoring force exerted by the bridge and how it varies with distance. The application of work in atmospheric chemistry to the formation of a water droplet at the apex of the cantilever tip would result in an improved understanding of said droplet and the dependence of its characteristics on atmospheric conditions, primarily relative humidity. Estimates of the size of the droplet could perhaps be acquired experimentally by charging the droplet through contact with a charged surface and then measuring the response of the cantilever to an electric field. The idea here is that the greater the size of the droplet, the greater the charge that would be deposited at the cantilever tip, and thus the greater the cantilever response. Success would be most likely to be achieved if an insulating cantilever were to be used. It would also be very interesting to find the critical angle of the surface slope that is required to produce multiple resonance excitation of a cantilever during breakaway and how this varies with relative humidity level.

Chapter 9

Conclusions

- The feasibility of microcantilever-based charged particle detectors was demonstrated.
- Detector response was characterized as a function of various environmental parameters. Specific studies and conclusions include:
    - Breakaway distance was studied as a function of relative humidity.
    - Jump-to-contact distance was studied as a function of relative humidity.
    - Both breakaway and jump-to-contact distances show a characteristic variation with relative humidity levels that corresponds well to the variation of the thickness of the water layer present on the surface.
    - It was concluded that changes in relative humidity change the magnitude of detector response only fractionally.
- Conclusions about measurements include the following:
    - The use of breakaway curves is a good method for finding parameter changes in charged particle (electric field) experiments.

- Conclusions about detector design included the following:
  - A spherical collector plate works very well and does not have problems with charge distribution over its surface being uneven and unpredictable.
  - The collector plate ought to be kept well grounded and have a minimum amount of contacts with adjacent materials in order to prevent electron sprays filling traps in said materials and altering charge distribution on the plate.
  - Cantilever detectors should be coated on the underside with a layer of conducting material in order to increase cantilever response.
  - Putting a voltage on the cantilever can greatly increase cantilever response.
  - Cantilevers with small spring constants will show the greatest response to the electric field emanating from the collector plate.
  - Collector plates in open atmosphere will show a maximum level of charging, beyond which migration of ions in the air to the plate will prevent any further increases in charge on the plate.
  - For an uncharged cantilever and a plate in the open atmosphere, contact between the cantilever tip and the plate prior to measurement of parameter change can be very desirable as then both the tip and the plate will be charged, maximizing detector response.

- Conclusions about cantilever response and data analysis include the following:
  - Damping rate shifts can be highly sensitive although not very reliable.
  - Both frequency shifts and deflection work well for detection of electric fields (and thus charged particles). Care must be taken however, because if the tip and plate do not contact each other, the force acting between them may be very weak and detector response insufficient to be useful.

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