The effects of air flow on the resonance properties of microcantilevers

Stephanie Elaine Gregor
To the Graduate Council:

I am submitting herewith a thesis written by Stephanie Elaine Gregor entitled "The effects of air flow on the resonance properties of microcantilevers." 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 Physics.

Panos Datskos, Major Professor

We have read this thesis and recommend its acceptance:

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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And recommend its acceptance:

Stephen C. Jacobson

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Accepted for the Council:

Vice Provost and Dean of Graduate Studies
ABSTRACT

The development of the atomic force microscope introduced the idea of using a microlength cantilever to detect a microsized phenomenon. It was found that a microlength cantilever could provide a useful optical or piezoresistive signal when it was bent or when its natural vibrations were altered. As research developed, the fluid medium surrounding the microcantilever became an influential parameter of experiments. As the fluid medium of microcantilevers was altered to create flow, the behavior of the microcantilevers was questioned and examined. In this experiment, measurements were made on the effect of air flow of various rates over microcantilevers in order to characterize some of the effects of flow. The complexity of flow was reduced by creating laminar parallel and perpendicular air flow over assorted microcantilevers (standard Si₃N₄ triangular and rectangular microcantilevers and Si rectangular microcantilevers) in a range of common flow rates (approximately 0-80 ml/min). The effects of the flow were measured by obtaining the resonance frequency spectrum and deflection of the microcantilevers from an optical signal. As the flow rate increased, it was found that the resonance frequency increased, the quality factor decreased, the resonance amplitude increased, and static deflection occurred proportional to perpendicular velocity. The increases in resonance frequency and resonance amplitude were not expected, yet they do not contradict theory or existing research. They indicate more measurement possibilities in the realm of microcantilever research. The results obtained open the door to further work with different types of fluids where the properties of viscosity, density and fluid inertia could be further characterized and measured.

High velocity airflow was examined in the hope that further signal amplification and velocity sensitivity could be obtained in the airfoil effect. No airfoil effect was measured, but the experimental conditions limited the flow velocity to below the speed of sound (which marks a change in functional dependence of the drag force to velocity-squared instead of velocity) and there were indications that turbulence may have occurred at the higher velocities. An attempt was made to utilize curved microcantilevers in the expectation that they would be sensitive enough to respond to the airfoil effect at lower velocities, but this did not work out. Further research with an improved cell is recommended to determine the potential for the airfoil effect to occur at a microscopic level, in order to obtain information for both microcantilever amplification and the understanding of fluids.
THE EFFECTS OF AIR FLOW ON THE RESONANCE PROPERTIES OF
MICROCANTILEVERS

A Thesis
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1. INTRODUCTION

The development of the atomic force microscope (AFM) opened up a powerful new form of measurement for microscopic phenomena. A microsize cantilever was used in the AFM to detect the surface of a material.\(^1\) As the cantilever was brought near the surface, the force exerted on the cantilever could be detected by the bending of the cantilever. The force was commonly detected either by linear displacement of a laser focused on the back of the cantilever or by the change in electric signal due to the bending of a piezoresistive cantilever.\(^1\) This ingenious piece of equipment has since been applied extensively to creative measurement experiments.\(^2,3,4\)

One very useful application of the AFM system has been to consider the dynamic properties of the microcantilever. As a lever, the microcantilever vibrates with ambient and applied energy, creating a spectrum in the frequency domain.\(^5\) The signal obtained from the cantilever, optically or piezoresistively, can be input into a spectrum analyzer. A spectrum analyzer decomposes the position signal into its Fourier frequency components and outputs the frequency spectrum. The resulting frequency spectrum is well understood theoretically in terms of harmonic oscillation.\(^6\) By matching the shape of the cantilever to the boundary conditions of a resonant wave\(^5\), the frequency spectrum can be predicted in terms of resonant frequency harmonics, the quality factor (Q) of the resonant peaks, and the phase shift of the outgoing signal, as displayed in Figures 1 and 2.

![Harmonic Resonance](Figure 1. Theoretical plot of a resonance frequency spectrum for various quality factor (Q) values.)
Figure 2. Theoretical plot of the phase shift for various quality factor (Q) values for a resonance curve in the frequency spectrum.

There is a wealth of physical information in the dynamics of a cantilever. The dynamics take into account both the forces applied to the cantilever externally and the intrinsic properties of the cantilever material which affect the flexibility and motion.\(^5\) Microcantilevers have thus been utilized in a variety of ways to measure physical properties at a microscopic scale.

The application of microcantilevers to commercial systems was initially marked by the introduction of the resonant gate transistor by H. C. Nathanson et al. at Westinghouse in 1965.\(^8\) The ability to utilize silicon to integrate mechanical microsensors with electronics was a key development in this field. In 1982, Kurt Petersen\(^2\) formally addressed the mechanical properties of silicon in light of its inexpensive, high-precision, high-reliability, miniturizable, batch-fabrication and thin-film properties in a paper designed to integrate the mechanical and electrical fields of microdevice research. At that time research at places such as Stanford, Texas Instruments, Hewlett-Packard, and IBM had already developed such miniaturized technologies as ink jet nozzles, circuit boards, optical benches/wave guides, gas chromatographs, refrigeration systems, pressure transducers, strain transducers, torsional scanners, image projection displays, arrays of voltage-addressable optical modulators, signal switching arrays, temperature sensors, and magnetic field sensors. In the next decade, research proliferated. By the early 1990’s, another major field integration occurred with microsystems and fluidics. In 1993, Peter Graveson

\(Q = \infty\)

\(Q = 13\)

\(Q = 8\)

\(Q = 3\)
et al. recognized the fluid microsensing capabilities of thermal, shear force, charge pulse, and pressure drop detection and the integrated microsystem capabilities of nozzle arrays, chemical analysis systems, and biologically implanted sensing and precision dosing. Shuchi Shoji and Masayoshi Esashi at that time similarly summed up the developments of microflow actuators, valves, pumps, sensors and systems.

With the advent of silicon technology and fluidics, the initial AFM system developed significantly from simply probing the surface of materials. Though information was initially sought for molecular configuration and surface forces, surface forces were revealed to be quite complex, and microcantilever research was used to further probe the various forms of energy in material surfaces. Thermal energy was typically probed with the amplitude and frequency of the microcantilevers themselves. Acoustic energy was measured through mechanical wave propagation over surfaces which can now be viewed through video imaging techniques. Magnetic forces could be probed by attaching a magnetic material to the end of the cantilever. Measurement techniques have also been developed to increase the sensitivity of measurements. Thermal and acoustic stimulation, phase measurements and interferometric equipment have greatly increased the sensitivity of microcantilever measurements, advancing the frontiers to nanomechanics.

In the field of microfluidics, AFM microcantilever research has followed two significant directions. First, a large amount research has been directed at the interaction of the fluid with the microcantilever. Absorption of molecules from the fluid medium was shown to result in a shift in the microcantilever’s effective mass which could be detected by the ensuing shift in the resonant frequency of the microcantilever. Microlabs were created by layering a chemically sensitive material onto systems of microcantilevers and allowed measurements to be taken from the ensuing bending of the microcantilevers. Research was finally focused on creating self-assembling monolayers on the layered cantilevers and trapping thiol-sensitive DNA. A second major development of microcantilever measurement in a fluid medium dealt with the properties of fluids. The complexity of the fluid environment was broken down into its effects on the microcantilever through terms of inertial and frictional resistance. Inertial resistance is due to the microcantilever pushing the fluid mass out of its way as it vibrates. Frictional resistance, identified through viscosity, is due to the intermolecular forces at work on the surface of the microcantilever. Such resistance to motion damps the vibrations of the microcantilever, creating
a signature in the frequency spectrum which can be measured. The theoretical cornerstone of fluid damping is Landau's ideal analysis of a sphere on a spring in a fluid.\textsuperscript{16,7} Ambitious endeavors have pushed the theoretical frontier, for example Xu et al.\textsuperscript{17} addressed the statistical nature of microgeometry and viscosity (through intermolecular forces and microgeometry), the degrees of freedom in a liquid as a compressed gas, and the balancing of microreversibility with macro entropy in gas theory; but no universally applicable or analytic solutions have been developed. The field is so complex that the general practice has followed the traditional fluidics method of taking a lot of specific, applied data in the hope that the bigger picture will eventually form. Typical research experiments have probed viscosity\textsuperscript{18,19}, increased the quality factor (Q)\textsuperscript{20}, simultaneously measured viscosity and density\textsuperscript{20}, examined the functional form of skin friction damping\textsuperscript{21}, and examined the effects of viscous drag on geometry, resonant frequency, quality factor, and amplitude\textsuperscript{22}.

The applications of microfluid research are valuable and powerful to both medicine and scientific research, but the science of fluidics has long defied analytic solutions. At the crux of the microfluidic arena are the inherent complexity of fluids and how the properties of fluids scale down to the micro realm. The Navier-Stokes equations, connecting pressure with acceleration and friction with viscosity, are nonlinear and quite complicated. Much of fluid science history has been developed either by mathematical development of frictionless hydrodynamic theory or experimental hydraulics.\textsuperscript{23} Prandtl, Reynolds and Raleigh worked at the turn of the century to develop analytic theory by analyzing the effects of geometry on fluids in boundary layers (exploring the limits of laminar vs. turbulent flow and friction) and dimensional analysis. These results provide a basis from which qualitative behavior can be extrapolated by holding some of the many parameters constant and measuring the effects of varying one or two parameters, as has been done with viscosity and density. Even so, the nonlinear analysis is often accomplished through chaos theory or computer simulation. The complexity is addressed here by compiling the research available and seeking the properties that would be useful to measure. Research has characterized many general properties of fluids in microsystems. Graveson\textsuperscript{3} stated that for laminar gas flows in microgeometry, it is possible to have low Reynolds numbers close to the velocity of sound (for flow diameters < 100 µm) and interface slip tends to disappear causing velocities to exceed predictions (diameters ~ 1 µm). For liquids, Graveson noted that viscosity is dependent on both polarity and the dimension of the flow channel and that gas bubbles interfere with flow at dimensions ~ 1 µm. Gass et al.\textsuperscript{19} examined shear force and viscosity in liquids. The
equations used assume that the drag force $F_D$ is parallel to the cantilever, the velocity profile is based on pressure difference, etc. Viscosity was found to dominate shear forces in liquids. Flow was found to have a functional relationship such that $F_D = f(v)$ for laminar flows and $F_D = f(v^2)$ for turbulent flow, given fixed geometric conditions. Chen et al.\textsuperscript{7} showed that gas damping could be generally accounted for with a change in the quality factor $Q$ and resonant frequency ($f_R$), while liquid damping is better accounted for with a change in the effective mass ($m_{\text{eff}}$). These results helped shape the development of the research performed here.

Considering the union of microcantilever measurements and macrofluidics, it is intriguing to look at the behavior of airfoils. Airfoils are tremendously sensitive to geometry and fluid characteristics. For a thin airfoil with a high-velocity air flow (approaching the speed of sound) and a large Reynolds number ($\sim 7 \times 10^6$), the angle of the airfoil can be adjusted to obtain a lift force. The flow at very low angle creates a smooth boundary layer and laminar flow around the airfoil. For moderate angles greater than zero and less than 10 degrees, a lift force is generated due to a pressure difference from boundary layer separation in general and air traversing different length paths on the top and bottom of the airfoil in specific. At larger angles, bubbles form around the sharp trailing edge when the boundary layer separates and turbulence develops.\textsuperscript{23} The situation when the angle is between 0 and 10 degrees is utilized in aircraft to harness the lift force. A force so sensitive to physical conditions would provide a very useful detection tool. A lot of research has been conducted at low velocities for biological systems, but very little has been conducted at high velocities, where microgeometry can reduce the slip resistance and where we now turn.
2. FOUNDATIONS

2.1 Cantilevers

The microcantilever is the critical sensing tool of the AFM. It is required to repeatedly and elastically flex under force. As such, a microcantilever has to be strong, flexible, and long-lived. Though many tips, such as STM (scanning tunneling microscope) tips, are chosen for their conductivity properties, AFM tips are not so restricted. Rather, metals are generally not chosen because they do not have the hardness necessary to stand up to AFM conditions. Research has turned instead to silicon, a well-understood semiconductor with the mechanical strength of crystalline structure. Traditional silicon wafers, with a diameter of 5-13 cm and a thickness of 0.5 mm, had a reputation for being extremely fragile and brittle. Research has revealed the fragility of single-crystal silicon wafers to be due to defects and pressure along their crystallographic planes. As the behavior of silicon was analyzed, processing was developed to minimize defects and points of stress concentration. With the minimization of silicon’s weaknesses, the strengths of the crystalline structure could be brought out. It was found that etching rather than cutting silicon not only prevented fracture, but allowed for a geometrical control of the final silicon structure.

Etching is a complicated chemical process that removes silicon in either isotropic or anisotropic directions. It is believed that the process is accomplished in four steps: 1) injection of holes into silicon (to increase the oxidation state), 2) attachment of hydroxyls to Si⁺, 3) reaction of hydrated silicon with a complexing agent, and 4) dissolution of reacted products. The most common etchant systems, notably EDP (ethylene diamine, pyrocatechol, and water), KOH and water, and HNA (HF, HNO₃ and acetic acid CH₃OOH), have various isotropy, masking and dopant properties. All of the etchants, however, attack silicon most slowly along the dense (111) surface. By coating the (100) surface of silicon with an etch-resistant mask, such as SiO₂ or Si₃N₄, the etchant will dissolve the silicon along the (111) plane wherever the mask is not present, forming pyramidal pits with an inclination of 55 degrees. This creates a mold for a hard material, such as Si₃N₄, to form a pyramidal tip on one side of a microcantilever for an AFM probe, while the opposite side is left flat to reflect a laser beam. (See picture in Appendix A) Often, the flat side is coated with gold to increase the quality of the reflected laser.
A common microcantilever set is sold by Thermomicroscopes. (See Appendix A) A Si$_3$N$_4$ base comes with one rectangular cantilever and four triangular cantilevers, all of differing sizes. Each has a pyramidal tip on one side and a gold coating on the other. The cantilevers have varying width, length and resonant frequency. The resonant frequencies are chosen by the geometry so that they fall in the range of 7-120 kHz, a range easily measured by common spectrum analyzers. Additionally, the spring constants are ranged between 0.01 and 0.5 N/m. Figure 3 shows a camera view of a Thermomicroscopes microcantilever chip mounted in a cell with a laser reflecting off the tip of the rectangular microcantilever.

2.2 Dynamics

As stated previously, the dynamics of a microcantilever contain a wealth of information. It is important to understand the basic information that will be used to analyze the results of microcantilever experiments.

2.2.1 Oscillation

The dynamic behavior of a microcantilever is derived theoretically by a number of different methods. One such method starts with a lever that is supported on one end but free on the other end. The position and motion of the free end are then analyzed in almost any Newtonian formulation. Once the position and velocity for a free lever are analyzed, external effects can be fit added to the mathematical framework. An analysis of the basic motion of an elastic lever can be found in most undergraduate mechanics courses through idealization as a one-
dimensional simple harmonic oscillator. Solving the simple harmonic equation with a velocity­
proportional damping factor and a forcing function yields common solutions:

\[
\frac{d^2y}{dt^2} + \gamma \frac{dy}{dt} + \omega_0^2 y = \frac{F_0}{m_{\text{eff}}} e^{-i\omega t}
\]

where \( y \) is the amplitude, \( t \) is the time, \( \gamma \) is the damping factor, \( \omega_0 = \sqrt{k/m_{\text{eff}}} \) = the resonant
frequency, \( F_0 e^{i\omega t} \) is the forcing function, and \( m_{\text{eff}} \) is the effective mass. A plot of the amplitude \( y \)
verses the frequency \( \omega \) from Eqn (2) yields a Gaussian curve with a peak at \( \omega_0 \). A plot of the
phase \( \delta \) versus the frequency \( \omega \) from Eqn (3) yields a curve in which the phase changes through
180 degrees (or \( \pi \) radians) as it passes through the Gaussian region. (Refer to Figures 1-2) This
behavior can be directly measured in a microcantilever experiment with a lock-in amplifier. A
lock-in amplifier stimulates the cantilever with a signal at a set phase and records both the
frequency spectrum and the relative phase of the outgoing signal. Data collected from a
microcantilever would be expected to look like Figure 4.

Figure 4. Theoretical plot of amplitude and phase verses frequency for a resonance system.
2.2.2 Resonant Frequency

The resonant frequency \( (\omega_0 = \sqrt{k/m_{\text{eff}}}) \) is defined from the spring constant of a harmonic oscillator. This spring constant is due to the elasticity of the cantilever. This elasticity can be defined in terms of the material properties and the geometry of the cantilever and then compared in a detailed fashion to the equations of motion for the cantilever. A common approach given by Sarid\(^5\) is to start with a lever’s degrees of freedom. Let the shear force be equal to \( ma \) and the partial derivative of the moment:

\[
F = ma = \rho A \frac{\partial^2 Y}{\partial t^2} = \frac{\partial M}{\partial x}.
\]  

(\(F = \text{force}, \ a = \text{acceleration}, \ \rho = \text{mass density}, \ A = \text{area}, \ M = \text{moment}, \ x = \text{length}\)) The moment can be expressed as \( E (\text{Young’s modulus}) \) multiplied by \( I (\text{the moment of inertia}) \) multiplied by the curvature of the lever \( (1/R = 1/\sqrt{x^2 + y^2}) \) so that:

\[
M = EI \frac{1}{R} = EI \frac{\partial^3 Y}{\partial x^2}.
\]  

(5)

By letting the lever be in equilibrium, \( \partial F/\partial x = 0 \), the following equation is obtained:

\[
\frac{\partial^2}{\partial x^2} \left( EI \frac{\partial^2 Y}{\partial x^2} \right) + \rho A \frac{\partial^2 Y}{\partial t^2} = 0,
\]  

(6)

leading to the result,

\[
Y(x, t) = y(x)e^{i\omega t}.
\]  

(7)

Applying the boundary conditions with one end free yields the solution:

\[
Y(x, t) = y_0 \left[ (\cos \kappa x - \cosh \kappa x) + \frac{D}{B} (\sin \kappa x - \sinh \kappa x) \right] e^{i\omega t},
\]  

(8)

where

\[
\omega_n = \kappa_n^2 \sqrt{\frac{EI}{\rho A}}.
\]  

(9)

Applying these results to a solid rectangular cantilever with a moment of inertia \( I = wt^3/12 \) and a spring constant \( k = F/x = 3EI/l^3 \) (for the cantilever: \( w = \text{width}, \ t = \text{thickness}, \ l = \text{length} \)) allows the resonant frequency to therefore be expressed as

\[
\omega_n = \frac{t}{l^2} \sqrt{\frac{E}{\rho}}.
\]  

(10)

Commonly used references\(^{29}\) express the resonant frequency \( F_R \) with the following equation:
2.2.3 Resonant Amplitude

The derivation of a harmonic oscillator resulted in a superposition of sinusoidal functions describing the amplitude of the cantilever at resonance. The question arises of what is the laser measuring? It is expected that the microcantilever oscillates at the resonant frequency in the shape of a quarter sine wave, increasing in amplitude non-linearly from zero at the fixed end to a maximum at the free end. The laser is expected to measure the quarter sine wave envelope of the oscillating microcantilever at the fundamental resonant frequency.

2.2.4 Harmonics

The microcantilever is expected to behave at harmonics of the fundamental in a fashion similar to that of the fundamental resonance frequency. The microcantilever is expected to form envelopes of fractions of the sine wave in which a maximum occurs at the free end of the microcantilever (i.e., $1 = \frac{3}{4}$ sine wave, $1 = 1 \frac{1}{4}$ sine wave, etc.). The microcantilever amplitude at the free end is expected to decrease with each increase in harmonic mode, which makes the harmonics difficult to measure.

2.2.5 Quality Factor

Finally, it is important to turn our consideration to the shape of the resonance frequency curve. If there were no damping, the peak of the curve would be infinite and the system would lose no energy. (Ref. Figure 1) The energy loss to the system of the experiment is dependent on the frequency and defines the height and width of the curve in the frequency spectrum. As the peak gets higher and the bandwidth gets narrower, the resonant frequency becomes more defined and easier to measure. To quantify this behavior of damping across experiments, a quality factor $Q$ is defined. From the denominator of the function for the amplitude of the oscillator in Eq (2), the damping can be related to the frequency. This definition can then be manipulated into an expression for quality:

$$F_r = \frac{1}{2\pi} \sqrt{\frac{k}{m}} \frac{1.03t}{2\pi^2} \sqrt{\frac{E}{\rho}}$$

(See alternate methods of derivation in Appendix B.)
\[
\omega_{\text{max}}^2 = \omega_0^2 - \frac{1}{2} \gamma^2 \quad \text{(12)}
\]

\[
\gamma_{\text{max}} = \frac{F_0 Q}{\kappa \sqrt{1 - \left(1/4Q^2\right)}} \quad \text{(13)}
\]

\[
Q = \frac{\omega_R}{\gamma} = \frac{\omega_R}{C_d A \rho v + c} \approx \frac{\omega_0}{\Delta \omega_{\text{Peak}}^{0.707}} \quad \text{(14)}
\]

The last equation expresses the quality factor in the familiar terms of total energy divided by the energy loss (energy is proportional to \( \omega \)).

The quality of a resonance peak is vital to analysis of resonance behavior and its prolific use has led to shorthand approximations. For casual analysis within an experiment, a qualitative approximation of the quality factor \( Q \) is given by the full width at half maximum (FWHM) of the frequency peak, as indicated in the last term of Eq (14). This value is not as universal as the \( Q \) factor, but it is independent of the amplitude of the signal in an experiment, fairly accurate and quick to use.

As stated above, the quality factor is defined from the effects of damping, which are the signature of the experimental conditions. Crucial to any experimental analysis of the frequency spectrum is the inclusion of damping forces, which give theoretical form to the interaction of the microcantilever with the experiment. This is the topic of the next section, where the issue of fluidics is addressed.
3. RESEARCH

3.1 Theory

The fundamental harmonic equations for the microcantilever carry information relating frequency and phase, quality and damping, geometry, material properties, and fluid variables. In this experiment, the measurable variables are the resonant frequency, the resonant amplitude, the quality factor, the phase shift in the output signal, and the static deflection of the microcantilever. Without fluid surroundings, the resonant frequency is dependent only on the cantilever material and its dimensions. Selection and manipulation of the cantilever provides the basis for the frequency information. To this basis, fluid damping can then be added.

3.1.1 Fluid Damping

To consider the detailed effects of fluids on cantilever resonance, the basic resonance equations need to take into account more detailed damping factors that include different damping functions and macroscopic factors such as viscosity. The starting place is generally Landau’s\(^\text{16}\) analytic solution to a sphere on a high-frequency spring in a static viscous fluid, where the drag force can be represented as

\[
F_D = \left(\frac{2}{3}\pi \rho R^3\right) \frac{dy}{dt} + (3\pi R^2 \sqrt{2\eta \rho \omega}) \frac{dy}{dt}.
\]  

Shih et al.\(^\text{22}\) utilize Landau’s model for fluid damping. The assumptions, that the cantilever is much longer than wide, that the axial displacement is significantly larger at and near the tip of the cantilever, and that the amplitude is smaller than the length, are requirements for linear behavior of cantilevers in fluid and are applicable to this experiment. (Hosaka and Itao\(^\text{30}\) provide cantilever parameters of length < 1.6 mm, width ~ length/10, thickness ~ length/100, and first resonant amplitude ~ length/1000 for a Reynolds number less than 1.) The harmonic differential equation can be written out by incorporating the damping terms proportional to the acceleration and velocity as induced mass and damping due to the viscous liquid. When the equations of motion are obtained, the amplitude and frequency can be expressed with

\[
\omega_b = \sqrt{\frac{\kappa}{M_{\text{eff}} + M_I}} \quad \text{and} \quad \gamma_L = \frac{b_m + b}{M_{\text{eff}} + M_I},
\]
where $M_1$ is the induced mass (acceleration coefficient of Eq (15)) and $b$ is the damping (velocity coefficient of Eq (15)) of the viscous fluid.

Chen et al.\textsuperscript{7} additionally employed Landau's analysis, with attention to the differences between gases and liquids. For liquids with a Reynolds number that is not small, an induced mass is similarly defined to be $M_1 = 2\pi \rho R^3/3$ and the dissipation constant is given as

$$\frac{b}{M_{\text{eff}} + M_1} = \frac{3\pi R^2 \sqrt{2\rho \omega}}{M_{\text{eff}} + M_1}. \tag{17}$$

This results in a resonant frequency of

$$\omega_{\text{max}} = \frac{1}{8} \left( \sqrt{9B^4 + 64\omega_0^2 - 3B^2} \right) \tag{18}$$

with $B = (\text{geometrical constant})^{*}(3\pi R^2 \rho)/\left(M_{\text{eff}} + M_1\right)$. The derivation for viscous damping of a gas is a little different. By taking the Chapman-Enskog viscosity equation, the nonpolar Leonard-Jones potential and assuming small Reynolds numbers (making the drag force proportional to the velocity and viscosity), the damping constant is determined and related to $Q^7$:

$$\gamma_{\text{gas}} = \frac{\alpha \eta}{M_{\text{eff}}} = \frac{\omega_{\text{max}}}{Q}, \tag{19}$$

where $\alpha$ is the drag proportional coefficient which can be expressed here as either $6\pi R$ or

$$\alpha = \frac{\kappa}{\eta Q \omega} \left(1 - \frac{1}{2Q^2}\right). \tag{20}$$

The complexity of a fluidic cantilever environment can be summed up from these analyses in the statement that gas damping is dominated by a velocity-dependent factor, the viscosity, while the liquid damping is dominated by an acceleration-dependent factor, the induced mass.\textsuperscript{7,11} This makes sense intuitively, as one would expect the increase in density of a liquid to provide significant resistance to the motion of the cantilever. It is thus expected that static gas damping can be taken into account primarily with a decrease in quality and secondarily with a decrease in resonant frequency. Certainly in static liquids, there is a decrease in resonant frequency and quality.\textsuperscript{11,21} SFM (scanning force microscopy) experiments, which explore damping because it increases the scan speed limit of SFM surface detection, show that gas systems shift the resonance frequencies of microcantilevers due to absorbed mass, change in surface stress and encountering the mass of the gas, in addition to damping.\textsuperscript{7}
However, before applying these results, a couple of issues must be addressed. The first is the issue of turbulence. The equations of motion are derived from linear differential equations. Turbulence is generally associated with nonlinearity and is dependent on a number of parameters, including velocity and geometry. Generally, when a fluid is allowed to travel in a straight line, laminar flow is expected and a linear flow profile is expected to exist between a wall, where there is no velocity, out towards the center of the passage where there is maximum velocity. In microfluid systems, it is generally stated that the drag force is a linear function of a fluid’s velocity in laminar flows and a quadratic function of velocity in turbulent flow. It is also stated that linear equations can be applied when the velocity is less than the speed of sound. Both high speeds and nonlinear geometries are possible causes of turbulence. The Reynolds transition number is used as a measure of the turbulence in a system. It is given as a range around 2000-2300 and calculated in microflow systems as \( \text{Re} = \frac{R \rho u}{\eta} \), where \( R \) is the radius, \( \rho \) is the density, \( u \) is the transverse velocity and \( \eta \) is the viscosity. It is difficult to measure or calculate for certain the Reynolds number for a system, but some of the conclusions of previous research provide guidance. Chen et al. calculated the Reynolds number for a similar system to this experiment and found it to be on an order of 0.01, but that was for a non-flow gas situation. It is generally concluded that turbulence is not an issue for systems with Reynolds numbers that are not small if the peak amplitude of oscillation is significantly smaller than the length. In contrast, Graveson et al. warned that turbulence can occur on dimensions below 100 \( \mu \)m and boundary slip can occur at a \( \mu \)m level, leading to a higher flow than expected. Given these insights, it is concluded that turbulence is possible in this system, even with a linear flow path, and probably likely as the velocity increases.

The second issue of flow is the source of interest for this experiment. Neither static microcantilever systems nor typical low-velocity microflow systems have a lot of information about the effects of flow on the dynamics of microcantilevers, especially for velocities in the m/s range. Parallel flow at low speed is expected to have minimal effect on the cantilever at low angle, merely forming streamlines (see references [23] and [30] for streamline pictures and discussion) around it and probably causing a decrease in the quality factor and resonant frequency. Parallel flow at high speed is theorized to affect an asymmetric and angled cantilever with a lift force,

\[
F_L = \frac{1}{2} C_L \rho v^2 L \tag{21}
\]
where $C_L$ is the lift coefficient, $\rho$ is density, $v$ is velocity, and $L$ is chord length, bending it to an equilibrium position proportional to the square of the velocity. The effects of parallel flow at high speeds on resonant oscillation is unknown. The bending of the cantilevers and the dynamic characteristics will be measured.

The force due to perpendicular gas flow is expected to bend the cantilever, according to Newton’s equations, to an equilibrium position that corresponds to the magnitude of the velocity and hence the force applied. It is expected that the effects of flow will reveal themselves in the quality factor and static bending of the microcantilever. Effects of high velocity will also await measurement.

Of direct relevance to this experiment, Neuzil et al. ran parallel flow over both SiN$_x$ ($Q \approx 20$) and SiO$_x$ ($Q = 187$) cantilevers to amplify the resonant signal. The quality factor was additionally analyzed to decrease with flow rate. However, it is stated that nonlinear effects are associated with a significant decrease in quality factor and resonant frequency.

3.1.2 Noise, humidity and temperature

As cantilevers take on microscopic dimensions and are utilized for microscopic measurements, the issue of noise becomes important. The environment stimulates the cantilever to move, providing the resonance data, but it also provides noise and damps the resonant vibrations. Many common factors which influence the frequency spectrum and the damping of the resonance curve are temperature, humidity, stray photons (light), and stray vibrations (mechanical and acoustic). Mechanical, acoustic, and light effects can be minimized through choice of equipment. Statistical mechanics can be utilized to calculate the noise inherent in a microsystem. Temperature and humidity effects are more unique to the experiment and must be addressed on an individual basis.

Butt and Jaschke performed a thermal analysis of microcantilevers by applying the equipartition theorem to the harmonic degrees of freedom. By expressing the thermal variations in position in terms of $\frac{1}{2}k_0T$, one would expect that you could simply set the thermal vibration energy $\frac{1}{2}k_x^2$ equal to $\frac{1}{2}k_0T$ and solve for the amplitude of the thermal vibrations as the square root of $k_0T/k$. Butt and Jaschke noted that the optical detection of cantilever position measures
the inclination rather than the position of the lever and the position must be expressed in terms of inclination: \( z = (2/3)L(dz/dx) \). Taking this into account, setting the kinetic and potential energies of the cantilever equal to \( \frac{1}{2}k_\beta T \), and summing over all the modes of vibration, the beam deflection of the rectangular cantilever due to noise was found to be:

\[
\sqrt{\langle z'^2 \rangle} = \sqrt{\frac{4k_\beta T}{3k'}} = \sqrt{\frac{4}{3} \langle z'^2 \rangle} .
\]

Similarly, Mehta et al.\(^{26}\) calculated the Brownian noise, a mechanical-thermal effect resulting from molecular agitation. The noise, defined to be proportional to the mass and quality factor of a resonator, was estimated to be in the range of 0.1 nm in amplitude for a typical microcantilever with a spring constant of 0.1 N/m. This is a negligible amount for the equipment being considered in this experiment.

Finally, John Vig performed a comprehensive theoretical analysis of noise in MEMS (microelectromechanical systems) and NEMS (nanoelectromechanical systems) in order to determine the scale at which the noise substantially affected the systems.\(^{27}\) For temperature, absorption/desorption, outgassing, Brownian motion, Johnson noise, drive power, self-heating, and random vibration, noise was found to be negligible except in the case of temperature and Johnson noise (to a lesser extent) in nanometer-dimensioned equipment in the MHz range.

For this experiment, the noise issues to be addressed are then large fluctuations of temperature or humidity and damping effects due to the level of moisture in air. The controlled environment of the laboratory removes concern about large temperature or humidity fluctuations. Temperature data was nonetheless taken throughout a couple of experiments without any noticeable effect. Humidity is predicted to decrease the quality of the resonance curve and act as an induced mass on the motion of the cantilever, potentially shifting the resonance curve down. However, the effects of humidity were neglected in this experiment for the following reasons: 1. ambient air is commonly utilized in MEMS research and some average humidity is expected in air data, 2. data was not taken in this experiment for absolute values of viscosity or quality, 3. data was compared over time without noticeable variations, and 4. the effects of flow were measured over timescales of minutes, in which the humidity would have no cause to drastically change.
Thus, it is expected that noise will occur from a variety of sources and in a generally random manner, but resonance data will eclipse the noise and fluctuations will be small enough to not affect the measurements. The randomness of such noise will mean that a baseline cannot be subtracted, but it also means that it will not mimic relevant data. Errant signals will be discussed in the experimental section.

3.2 Experimental work

To measure the effects of flow, a flow cell needed to be designed to allow parallel and perpendicular flow over the optically detected cantilever. Air was chosen to probe the potential for lift due to the data on macroscopic airfoils which is available for comparison. The cantilever is supposed to be at a small angle (less than 10 degrees) to the airflow in laminar flow in order to experience the lift force. In this experiment, the imprecision of securing the cantilever parallel to the flow will result in some small angle between the cantilever plane and the flow. Detailed measurement of the angle would be required to quantify airfoil behavior, but the small angle inherent to the equipment is a positive happenstance that is expected to allow qualitative response to the airfoil effect. Curved cantilevers are also used to insure that there is some asymmetry, but turbulence and nonlinear effects are expected to accompany drastic curvature. For either situation, it is expected that there will be damping and a static bending that will increase as the flow rate is increased. A range of velocities is chosen for the air. Although large velocities would be expected to have the greatest effect, the cell will have limits to the velocity it can contain. The flow rate will begin with small velocities that are comparable to that of experiments done in other fields and increase to the limitations of the cell.

The velocity of a fluid can be controlled and measured in a number of ways. It can be controlled through the flow rate and geometry. It has been measured in a variety of forms: mass, momentum, viscosity, drag, etc, by a variety of methods: mass flow, ion pulse generation, differential pressure, thermal anemometry, cantilever damping, etc. The equipment is designed here so that the velocity can be controlled through flow rate and geometry and measured with a digital meter on the outflow. In this way, the cantilever frequency and damping can be compared to the velocity readings for analysis. Much of the research available for comparison has been performed at low flow rates for biological applications or for submerged AFM tips.
3.2.1 Instrumentation

In this experiment, a cantilever was secured to a wall in a sealed cell with input and output tubes. A laser was focused through a glass slide window of the cell onto the end of the cantilever and reflected into a quad cell photodiode detector, which is a common AFM or SFM optical detection system. The final experimental setup consisted of the following equipment: 1) a source of flow leading into the cell chamber, 2) a cell chamber defined by an o-ring clamped between a groove on a backing plate and a glass slide, 3) a cantilever on a base clamped to the backing plate within the cell, 4) an outflow leading through a digital flow meter, 5) a 5 mW laser collimated and focused on the cantilever through the glass slide, 6) a quad cell photodiode detector with three output channels: total, horizontal, and vertical relative signals in volts, 7) a voltmeter receiving the total photodiode signal, 8) an oscilloscope receiving horizontal and vertical photodiode signals, and 9) a spectrum analyzer receiving a split of the horizontal photodiode signal.

1) The inflow system for low flow rates (1-10 ml/min) consisted of a model YA-12 Yale syringe pump with a Gastight 1050 syringe (dia = 32.5 mm). (See Figure 5.) The syringe pump motor operated with an input diameter programmed to 32 mm. For a larger range of flow rates, flow was also obtained in a less precise method by connecting the cell inflow to a valve from

![Figure 5. Syringe pump.](image-url)
the vibration-resistant table’s pneumatic source. All cell tubing had an inner diameter of approximately 1/32”. A 100 µm capillary was inserted into the tubing for perpendicular flow.  

2) The glass slide was standard, 1 mm thick.  

3) A clamp for the cantilever was made by placing a thin piece of metal over the base and tightening it down with a screw into the solid aluminum backing.  

4) The flow meter was a Cole Parmer model 32915-04 (calibrated by MM 1/18/00; serial number 3521) with a range of 0-10 ml/min.  

5) The laser was class 3a, with a power of 5 mW and a coherent wavelength of 635 nm (HeNe is 632.8 nm). To obtain a quality signal, the laser was focused on a pinhole, collimated and focused on the cantilever. (See Figure 6.) The intensity was controlled by sending the beam through a circular opening that could be contracted or expanded to obtain the desired intensity. The laser had enough strength to match the range of the photodiode detector. It was decided that a 13 V signal saturated the photodiode and a signal below 2 V tended to suffer from diffraction at the circular opening. Signals were generally set to give a 5 V reading on the photodiode.  

6) The photodiode quad cell was a unique construction of handy materials. The output voltage signals were weighted (see instrument noise section 3.2.2.b for details) and absolute magnitude was not an issue. The total output signal had a range of 0-13 V; however, to avoid saturation, signals were kept to the middle of range.  

7) The multimeter was a model DM501 module in a Tektronix TM504 case.  

8) The oscilloscope was a model SC502, 15MHz module in a Tektronix TM504 case.  

9) The spectrum analyzer was a Stanford Research SR770 FFT model, with a range of 0-100 kHz.  

3.2.1.a Flow control system  

A calibrated 50 ml syringe was filled with ambient air and secured. A motor with thread windings was placed at the handle of the syringe so that the motor would push the syringe closed at a consistent rate. The motor was controlled with software that allowed parameters to be set. The syringe pump was fed through a meter, both with and without the cell installed in between, to determine the flow rate and thus the velocity. The meter consistently registered approximately 80% of the syringe rate. (Refer to Section 3.2.3.) The cantilever signal took time (typically minutes) to come to equilibrium when the syringe pump was turned on. Therefore, it was
a. Picture of laser equipment.

b. Schematic of laser setup.

Figure 6. Laser setup.
assumed that flow velocity depended on the cell geometry more than the syringe pump and the
digital meter was used on the outflow to record flow rate. Velocity in cm/s could then be found
by dividing the flow rate (ml/min or ccm) by $60 \pi r^2$. For perpendicular flow in the second cell,
a capillary of fixed radius ($r = 50 \mu m$) was inserted into the input channel of the cell, resulting in
a calculable velocity from the flow rate of $\approx 210$ cm/s per ml/min.

3.2.1.b Flow cell

The design of cantilever cells was focused on flow requirements. The geometry of the
cantilever cell was developed to allow for parallel and perpendicular laminar flow. It was
designed to minimize dead volume for the purposes of reducing settling time and reducing
turbulence. In general, as the geometry is reduced, the size of the equipment must be compared
to the size of the molecules in the fluid to evaluate potential effects. In this experiment, the cells
and tubing were on scales of 0.2 cc and 0.1 mm diameter, which is much larger than the molecule
size.

The initial test cantilever cell consisted of a cylinder with circular glass plates screwed
onto each axial end, a cantilever holder inserted horizontally into the middle, and flow ports
inserted vertically above and horizontally at the end of the cantilever in the sides of the cylinder.
(See Figure 7.) This arrangement was relatively large, with an inner diameter of 0.8” and a
thickness of 0.5” (volume $\approx 0.25$ in$^3$ or 4.1 cc), minus the cantilever support. It was found that
data taken with this cell proved susceptible to large settling time and drift when the flow was
turned on, as shown in Figure 8. It was decided that a cell with linear flow geometry would
provide the most laminar properties.

A second and final cell was machined to allow parallel and perpendicular flow, to reduce
dead volume and to improve the alignment of the flow with respect to the cantilever. (See Figure
9.) The cell was made of aluminum, as corrosion was not an issue for air flow. The cantilever
was secured to the end of a cylindrical holder and inserted into the back of the cell. Two ports on
either side of the cylinder/cantilever allowed parallel flow. A hole was drilled into the length of
the cylinder near to the cantilever so that the cantilever could be placed to sit directly over the
capillary opening. This configuration was utilized for perpendicular flow. This cell was also
positioned so that the cantilever was horizontal for data collection.
Figure 7. Picture of the first flow cell. Note the cylindrical hole in the bottom for the insertion of the cantilever holder. One hole in the side was to act as an inlet port and the other was sealed. A hole was drilled in the top for an outlet port. O rings were fit to the grooves on each opening of the cylinder and circular glass slides were tightened and sealed over each using screws in the four corner holes. The cell was secured sideways (90 degree rotation of the picture) and horizontal signals were measured.

Figure 8. Flow response in the first flow cell. The rectangular cantilever B was placed under successively larger flow rates and allowed time to come to equilibrium. Drift and slow response led to the conclusion that the flow was not laminar and a new cell would be required.
a. Picture of front view of final flow cell.

b. Picture of back view of final flow cell.

Figure 9. Final flow cell.
c. Drawing of front view of final flow cell.

d. Drawing of side view of final flow cell.

Figure 9. (Continued)
3.2.1.c Cantilever selection

Cantilevers were selected for performance that correlated well with the range of the equipment. The gold-plated Si$_3$N$_4$ cantilevers designed for AFM heads were used initially. (Specifications are in Appendix A.) When the largest cantilevers (B, C, D) were placed in perpendicular flow (0-10 ml/min), they bent the signal past the range of the photodiode horizontal stage (10 mm). This could be corrected by placing a lens in front of the photodiode, but that was unnecessary for this experiment, as cantilevers E and A were well-suited to the experiment. The smallest Si$_3$N$_4$ cantilever F resonated above the 100 kHz range of the spectrum analyzer. Medium size cantilevers E ($f_R \sim 37.5$ kHz) and A ($f_R \sim 22.9$ kHz) were utilized primarily in the experiment. Figure 10 shows the resonance spectra of cantilevers A-E.

Additional cantilevers were selected to probe for the airfoil effect. The first chip contained four straight rectangular silicon cantilevers of similar length, resonant frequency, and quality factor and one shortened cantilever, which was too small for this experiment. The cantilevers were developed by milling in a focused ion beam (FIB) and layering aluminum thinly onto the surface for reflectivity. The resonant frequencies of the four cantilevers were approximately 48 kHz (see Figure 11) and the lengths approximately 200 µm, making the
thickness approximately 1.5 µm. The second chip was a standard Si$_3$N$_4$ chip layered with a thicker substrate, so that the stress difference of the material structures forced curvature. In this way, previous data on Si$_3$N$_4$ cantilevers could be compared with this data for curvature. Finally, a chip with different length rectangular silicon cantilevers was FIB milled and thickly sputtered to force slightly greater curvature. (See Figure 12.) Unfortunately, these cantilevers were too small and curved to obtain a clear signal.

3.2.2 Basic equipment measurements

3.2.2.a Accuracy and sensitivity

The limits of the spectrum analyzer in bandwidth was in the 10’s of Hertz. The measured bandwidth and amplitude could be calculated from dividing the span by a set number of intervals. Data was generally taken as an average of 300 spectra. When greater accuracy was desired in the resonant frequency and shifts, a statistically significant number of spectra were recorded and then analyzed according to a normal distribution. (See section 4.1) The photodiode operated in an analog mode and was centered horizontally and vertically to the nearest tenth of a volt at the beginning of each data set. The flow meter registered flow to three significant digits through a range of 0-10 ml/min.
3.2.2.b Control of noise, humidity and temperature

The spectrum analyzer contained background noise of $\leq 100 \, \mu V$. It is assumed that this signal was due to thermal noise, the ventilation system, and other sources of unavoidable noise. First, and sometimes second, cantilever resonances could typically be viewed over this noise. The experiment was isolated on a hydraulic table, yet the signal was extremely sensitive to ambient activity, such as nearby voices and the door closing. The monitor used to view the cantilever during setup contributed noticeably and was turned off during data collection whenever possible. The overhead fluorescent lights and the syringe pump were found to not contribute noticeably, at least above 4 kHz. It was found that the signal could not be boosted with the input amplification on the spectrum analyzer due to amplification of the noise and so it was set to 0 dB. However, peaks in the resonance spectrum due to equipment noise were identified by boosting the input amplification and examining phase data for a lack of phase shift at these peaks. Three clear peaks of noise were identified. Two peaks at 31.5 and 52.3 kHz (see Figure 11) were found to correspond to the spectrum analyzer. One peak at 55.8 kHz (see Figures 10, 11) proved universal and of large amplitude. On examination, it was found to lack a phase shift and to contain a repeating structure that was not a property of cantilever resonance, as shown in Figure 13. This peak was found to occur when a laser was reflected off the bases of the cantilevers. It was concluded that it was due to the base itself or some invariant of the equipment. In an attempt to remove the noise, a background signal was measured and subtracted from the data; however, this did not improve the results as the majority of the noise was random. However, with the noise thus identified, the cantilever data could be identified and measured.

The photodiode was a quad cell arrangement (cells A,B,C,D; refer to Figure 14) with three voltage outputs of a horizontal signal $[(B+D)-(A+C)]/(A+B+C+D)$, a vertical signal
Figure 13. Zoom of artifact frequency spectrum peak caused by the equipment. When measured on a large span width, the noise appears to have a Gaussian shape (upper curve). However, the lack of a phase shift and the shape when zoomed indicated that it is not a resonant curve of the cantilever. See Figures 10, 11 and 15 for comparison.

Figure 14. Photodiode cell divided into quadrants.
([(A+B)-(C+D)] / (A+B+C+D)) and a total signal (A+B+C+D). Photodiode saturation occurred at 13 V. Besides receiving the laser reflection, the photodiode measured a few sources of noise. The overhead fluorescent lighting added ~0.3 V on the total photodiode cell voltage reading. This was not considered an important factor, but was allowed to be absorbed into the calibration of the measurements. One source of noise was found to be due to the photodiode. When the cantilever was deflected during flow experimentation, a signal at 55.5 kHz was amplified beyond that of the cantilever resonance. To isolate the cause for this signal, the photodiode was shifted horizontally away from the center of the laser reflection of an ambient cantilever signal. Spectrum data was taken at 1 V increments, from 0-9 V of the horizontal photodiode signal. The result is displayed in Figure 15. As the laser signal approached the horizontal fringe of the photodiode, the cantilever E resonant peak at 37 kHz lost amplitude while noise was amplified. When the horizontal signal reached 4 V, the resonant peak began to lose significant amplitude; by 9 V, the resonant signal was not distinguishable. As a consequence, the resonance signals of statically deflected cantilevers were allowed to come to equilibrium and then recentered before measurement.

![Effects of shifting the photodiode on cantilever E](image)

Figure 15. Noise due to uncentered photodiode signal. When the laser signal became uncentered horizontally, as when the cantilever experienced deflection, resonance data was lost and noise was amplified. At 4 V, significant signal loss occurred. Signals with cantilever deflection were thus brought to equilibrium and recentered for data collection.
Humidity and temperature are a concern to AFM measurements. The laboratory of this experiment had a temperature meter on the wall. The variations were not large from day to day in this controlled environment. The temperature was generally within a few degrees of 72°F and the humidity was not very large. The temperature was recorded at the beginning of the spectrum characterization of the cantilevers used in this experiment, but no noticeable change could be perceived. It was concluded that the effects of temperature and humidity were quite small compared to the cantilever behavior.

3.2.2.c Fundamental cantilever properties

The fundamental resonance properties of the cantilevers were measured for characterization and familiarity purposes. First, resonance behavior was measured by with a lock-in amplifier. The resonant frequency spectrum and phase shift occurred just as predicted by theory, as shown in Figure 16.

Second, the resonance frequency of Si$_3$N$_4$ Thermomicroscopes cantilever C was calculated according to the theory of the mechanical properties (Eqn (11)) with the manufacturer’s data ($t = 0.6$ µm, $l = 320$ µm, $E = 130$ GPa, and $\rho = 0.33$ g/cm$^3$) to have a

![Acoustically stimulated cantilever resonance](image)

Figure 16. Experimental plot of a resonance frequency spectrum and phase shift. Measured with a spectrum analyzer and lock-in amplifier, the microcantilever displayed the theoretically predicted behavior of simple harmonic oscillation.
Acoustically stimulated cantilever C resonance

Figure 17. Resonance frequency spectrum for cantilever C. The resonant frequency was predicted by theory, based on the geometrical and material properties of the microcantilever.

resonant frequency of 7 kHz. Data taken with cantilever C yielded the plot in Figure 17. The experimental data is found to agree with the calculations, though the calculations are based on data that is only given to one significant digit. The precision of the calculations can be increased by measuring the physical properties of the cantilevers in greater detail, if desired. For this experiment, the calculations are used only for approximation and no greater detail is required.

Third, the fundamental harmonic amplitude of a cantilever was plotted at various points along the length by measuring the displacement of the laser at these points. This was done for triangular cantilever C. The position of the laser along the length was measured with a camera picture. (See Figure 18) The fraction of the length was multiplied by the length of 320 µm to determine the distance. The amplitude was determined relatively from the magnitude of the photodiode signal of the deflected laser beam. The amplitude is shown in Figure 19. These results match the expectations.

Fourth, the first harmonic of the resonant frequency was measured. One method of measuring a small microcantilever signal was to acoustically stimulate motion with a speaker. The excitation frequency was controlled with a signal generator set to 5 V output. Since resonance is an amplification property, the signal magnitude did not matter; however, it was
Figure 18. Method of measuring the vibration amplitude versus length. The laser was focused at various positions along the length of the cantilever. The amplitude of vibration was determined from the photodiode and an oscilloscope. The position was determined from comparing the length of the cantilever (320 µm) with the position of the laser on the camera picture.

Figure 19. Amplitude versus length for $\text{Si}_3\text{N}_4$ cantilever C at the first resonance. The bottom leg is indicated by diamond markers, the top by triangles and the reference sine wave with a dotted line.
frequency range with this speaker (Panasonic model WM-R30B card speaker) and signal
generator. In order to avoid the complexity of the harmonics of triangular geometry, rectangular
microcantilever B was used. Calculating that three fourths of a wavelength would resonate along
the length of the rectangular cantilever for the second harmonic, the frequency was calculated
from Eq (11) to be approximately 55 kHz. Data points were taken at small increments of
frequency around 55 kHz. The data were then fit to a Gaussian curve:

\[ y = y_0 + a e^{-\left(\frac{x-x_0}{\sigma}\right)^2}. \]  

(23)

A rough fit yielded values of \( y_0 = 53.6, a = 142, x_0 = 52.6, \) and \( \sigma = 0.178. \) (See Figure 20) The
measured resonant frequency 53 kHz is within 4% of the predicted value 55 kHz, which is
actually only good to one significant digit, as explained previously. This method of determining
the second resonance verifies theory of harmonics, yet the method is somewhat unwieldy for
precise measurements.

A second measurement method was tested for potential use. The microcantilever was
used as the reflector for an interferometer signal. This provided great sensitivity to amplification
at resonance, allowing four resonant harmonics of silicon cantilevers to be identified. However,

![Gaussian fit to second resonance of cantilever B](image)

Figure 20. The second harmonic frequency of the Si_3N_4 rectangle cantilever B. The signal was increased
with acoustic stimulation and the amplification was measured. At various points in the frequency spectrum
the resonance was measured to obtain a peak distribution that could be fit with a Gaussian function.
these signals were quite sensitive to background noise and the quality of the signal. While interferometry might conceivably be accomplished through a fluid medium, the likeliness seemed problematic. An additional problem was that the resonant frequencies quickly exceeded the range of readily available spectrum analyzer equipment (0-100 kHz).

Finally, the equipment was calibrated to measure the amplitude of oscillation so that order-of-magnitude information could be obtained. A change in laser path of 10 µm was measured with the horizontal micrometer on the cantilever cell. The resulting 1 V signal on the horizontal photodiode channel allowed the cantilever tip displacement to be easily correlated to 10 µm per 1 V or 1 nm per 100 µV in this instance. However, the relationship is probably not linear and should be repeated carefully for significant data. Si₃N₄ microcantilever D was shown to have an amplitude of approximately 24 nm for the first harmonic near 17 kHz and 1.2 nm for the second harmonic near 65 kHz.

3.2.3 Measured effects of flow

Once the cell and cantilevers were set up and the static behavior characterized, the measurements were repeated with airflow. The flow was begun in the range of 0-10 ml/min. The seals were tested and tightened sufficiently so that there were no leaks. A spectrum of the base was taken during flow for comparison. Various flow levels were then applied to Si₃N₄ cantilever E. When the flow behavior was characterized, the experiment was repeated with cantilever A.

An important characteristic of the cell was the time it took to settle into equilibrium. Once the syringe pump was turned on, the outflow rate would steadily increase until it came to equilibrium, as would the changes in the cantilever’s frequency spectrum. Figure 21 displays the outflow rate as a function of time given various syringe pump rates on cantilever E. The length of the trial in time was defined by the volume of the syringe (50 cc) and the flow rate. As such, the size of the syringe limited the time in which stable data could be taken. A second flow source was obtained by connecting the cell to a pneumatic source, but it was difficult to obtain precise velocities and so was not used for small flow rates. For the smaller flow rates that were able to come to equilibrium, the programmed syringe pump rate was consistently 125% larger than the
Figure 21. Outflow rate versus time for various input syringe pump rates in the final cell. (Cantilever E) The largest rate is seen to overwhelm the system while the rates below 8 ml/min fall short of the inflow rate, consistently reaching approximately 80% of the inflow rate. The limitation of the syringe volume is seen in the time allowed each rate. Experiments optimized the available equilibrium time.

flow meter registered. This was viewed as a parameter entry error in the syringe pump and the flow meter was used for flow values with both the syringe pump and the pneumatic source.

3.2.3.a Static deflections

Perpendicular air flow immediately and significantly bent the triangular Si$_3$N$_4$ cantilever signals on the horizontal photodiode channel over the range of flow rates. Figure 22 displays the horizontal photodiode signal versus outflow rate for three representative samples of 2-7 ml/min. This plot displays the reading of the photodiode and outflow rate over time as the syringe pump was turned on and brought the cell up to an equilibrium state. The deflection was found to increase with the rate of flow, as expected. Additionally, the deflection on the photodiode read a consistent value for a given outflow rate regardless of what the final outflow rate would be. The deflection was too large to measure flow rates of 8-9 ml/min. Due to the errors discussed in Section 3.2.2.b, the resonance behavior in perpendicular airflows was measured after allowing the cantilever to come to equilibrium and then recentering the photodiode to the signal.
3.2.3.b Shifts in resonance frequency

When low perpendicular flow (2-7 ml/min) was applied, an increase in resonant frequency was graphically obvious for the triangular Si$_3$N$_4$ cantilevers. This trend can be displayed in two ways. The first is in a comparison of resonant spectra peaks for cantilever A under different flow rates. As displayed in Figure 23, the resonant frequency increased consistently with flow rate. There was concern that the flow might not be consistent throughout the cell, as was the case in the initial cell. Therefore, the experiment was repeated by altering the distance from the capillary inflow to the cantilever to determine whether or not this was a factor. However, for distances of approximately 100, 200, and 300 µm from the cantilever, there was no significance to the proximity of the inflow capillary. (The distance from the capillary to the cantilever was approximated by focusing the camera on the cell and comparing the known size of the cantilever and capillary with the distance between the capillary and the cantilever.) It appears from this data that the flow velocity was consistent within the cell over the ranges of flows used.

The second method to display the shift in frequency was to measure the frequency spectrum of Si$_3$N$_4$ cantilever E as a relatively large flow of 9 ml/min was applied. As seen in
Figure 23. Resonant frequency shift of Si$_3$N$_4$ cantilever A under perpendicular flow rates. The data was taken for various positions of the inflow capillary. The capillary was moved to positions of 100 (diamond symbol), 200 (square symbol), and 300 (triangle symbol) µm away from the cantilever to test the consistency of the flow in the cell, but no effect was measured. The dotted line represents the resonant frequency for zero flow. A decrease is evident for low flows, as expected, but an unexpected increase occurs for rates above 5ml/min. The deflected signal was brought to equilibrium and recentered on the photodiode before resonance data was taken.

Figure 24, the frequency of the cantilever spectrum was shifted and, additionally, the overall amplitude had increased as the flow in the cell increased.

3.2.3.c Change in quality factor

The quality factor, calculated from Eqn (14), was found to decrease with flow rate, as expected and displayed in Figure 25. Again, the experiment was repeated as the capillary inflow distance to the cantilever was varied and no influence was measured.

3.2.3.d Change in resonance amplitude

When the perpendicular flow rate was increased over the Si$_3$N$_4$ cantilevers, the amplitude of resonance was found to increase dramatically. Figure 26 displays the change that occurred in the middle of the velocity range. To test the repeatability of this unexpected phenomenon, parallel flow was passed over the cantilevers over a large range of flow rates. The valve to the
Figure 24. Perpendicular flow over cantilever E: resonant frequency shift. Spectra were taken at two different times after the flow was turned on. The effect of the flow rate increasing can be seen in the change in resonant frequency and amplitude. The signal was recentered before measurement.

Figure 25. Perpendicular flow over cantilever A: quality factor change. The quality factor, determined from Eq (14), decreases consistently as the flow rate increases. The data was taken for various positions of the inflow capillary. The capillary was moved to positions of 100 (diamond symbol), 200 (square symbol), and 300 (triangle symbol) µm away from the cantilever to test the consistency of the flow in the cell, but no effect was measured. The dotted line represents the quality factor for zero flow. The deflected signal was brought to equilibrium and recentered on the photodiode before resonance data was taken.
Figure 26. Perpendicular flow over cantilever E: resonant amplitude change. The resonant amplitude increases significantly with the perpendicular flow rate for flow > 5 ml/min. The deflected signal was brought to equilibrium and recentered on the photodiode before resonance data was taken.

A pneumatic source was opened up to four revolutions of the valve past 10 ml/min flow (which occurred at less than one revolution). For all flow rates, the amplitude at resonance increased with the flow rate. (See Figure 27.) At four revolutions, the flow rate was high enough to begin making the cell leak, so the flow rate was not increased any more. It is conclusive that for each of the cantilevers tested (A, E, R and C) and for both parallel and perpendicular flow, the amplitude was shown to dramatically increase.
Figure 27. Parallel flow over microcantilevers C, B, A and E. The large flow rates exceeded the meter. The flow was gauged by recording the number of revolutions the valve to the pneumatic source had been opened. Four revolutions was the maximum the cell could withstand without leaking. A dramatic increase in amplitude is shown to accompany an increase in flow rate. The same results were obtained with perpendicular flow.
4. ANALYSIS

4.1 Statistical analysis procedure

The unexpectedness of the increase in resonant frequency makes it important to quantify the results. To verify that the shift in resonant frequency is distinct, a statistical analysis can be performed which calculates the variation inherent to the resonance curve data and the confidence that can be assumed for the resonant frequency value. Fundamental statistics determine that random samples of a piece of data will fall around the average (mean) value in a normal (Gaussian) curve. A normal curve is characterized by a mean, which is the true value of the data being sampled, and a standard deviation (σ), which is a measure of how far the data tend to range from the mean. The standard deviation is the distance from the mean to the inflection point in the normal curve and represents the range in which a random sample of data is likely to fall. In reference to the mean, approximately 68% of data will fall within ±σ, 95% of data will fall within ±2σ, and 99.7% will fall within ±3σ. As the number of data points increases, the standard deviation and the mean can be calculated to a greater precision. Once the mean and the standard deviation are known for a sample of data, the accuracy of the mean (how close the sampled mean is to the true value of the experiment) can be calculated. Whereas the standard deviation provides the probability that a measurement will be close in range to the mean, the standard deviation of the mean provides the probability that the mean is close in range to the true value being measured.

A large sample of resonance data was taken for cantilever A and the resonant frequencies were analyzed as a normal distribution. The spectrum analyzer was set to average 300 spectra at a span of 6.25 kHz centered on the resonant frequency. The peak was recorded 100 times at 0 ml/min and 70 times at 4.3 ml/min and then each was fit to a normal distribution. The mean and the standard deviation were conservatively calculated (without inclusion of the 300 spectra average in the sample size) to be 22.906 kHz ±54 Hz for 4 ml/min and 22.853 kHz ±42 Hz for zero flow. (See Table 1.) The standard deviation of the mean was then calculated, taking into account the 300 sample average of the spectrum analyzer, to be ±3 Hz for 4 ml/min and ±2 Hz for zero flow. Applying the probabilities associated with standard deviations of normal distributions, there is a 99.7% probability that the resonant frequency of zero flow is within 0.007
Table 1. Statistical analysis of resonant frequency shift for cantilever A. Units are in kHz.

<table>
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<th>Statistical analysis of normal fit:</th>
<th>Flow = 0</th>
<th>Flow=4ml/min</th>
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<td>Variance:</td>
<td>0.002</td>
<td>0.003</td>
</tr>
<tr>
<td>Standard Deviation:</td>
<td>0.042</td>
<td>0.054</td>
</tr>
<tr>
<td>Mean:</td>
<td>22.853</td>
<td>22.906</td>
</tr>
<tr>
<td>Standard deviation of mean:</td>
<td>0.002</td>
<td>0.003</td>
</tr>
<tr>
<td>Difference:</td>
<td></td>
<td>0.053</td>
</tr>
</tbody>
</table>

kHz of 22.853 kHz (22.846 kHz ≤ ω_R ≤ 22.860 kHz) and the resonant frequency of 4 ml/min flow is within 0.009 kHz of 22.906 kHz (22.897 kHz ≤ ω_R ≤ 22.915 kHz). This analysis confirms the conclusion that there is a significant shift in the resonance frequency when air flow is applied to the cantilever.

4.2 Signatures of air flow

There were four signatures of the air flow. The first is the correlation of cantilever deflection to outflow rate for perpendicular flow. (Figure 21.) This signature will allow the identification of flow rate with a microcantilever, at least for low flow rates (0-10 ml/min). The second signature is the decrease in quality factor with increased flow rate. A decrease in quality is expected and can be quantified and compared with other research. The third signature is the increase in resonant frequency with flow rate. It is unexpected and requires analysis. The fourth signature is amplitude increase at resonance under flow. It is consistent, occurring for low flow rates and high flow rates. It is also dramatic, increasing the peak by an order of magnitude for high flow rates. Finally, it is versatile, occurring for both parallel and perpendicular flow. Considering the field of microelectromechanical devices (MEMS), it is also quite useful. The ability to amplify microscope signals is highly sought after in research.

4.3 Comparison with theory

The deflection of the cantilever with perpendicular flow is expected by theory (first paragraph, p. 15) and can be compared with the drag force of air for the given velocities. Because it is expected and has been measured before\(^2\), there is no further analysis here. The decrease in quality is also expected and can be quantified in light of the damping expression for a
cantilever oscillating in a static gas (Eqns (1) and (19)). The effect of velocity could be addressed, but it would be difficult to isolate the velocity effect with the data taken. As stated previously, the increase in resonant frequency is unexpected. The decrease of resonant frequency for rates below 5 ml/min (Figure 23) is indicative of other research findings7,25 and theory for static gas (Eq (17) and (18)), where the presence of molecules around the cantilever are believed to impede the oscillation of the cantilever and act as a load, or an increased effective mass. (Eqn (16)) The increase seems to indicate the opposite of damping, an energy increase. The fact that the amplitude increases dramatically supports an increase in energy in the cantilever. The increase in amplitude of oscillation is correlated directly in theory to an increase in energy (E = ½ Ky²).6 However, the mechanism is unknown. It is possible that undetected static bending increased the stress and effective spring constant of the cantilevers, thereby increasing the resonant frequency (Eq (16)), but then the amplitude would be expected to decrease, not increase.

In all the results of air flow, there was no detected static deflection due to parallel flow. (Eqn (21)) It is likely that the velocity was not near enough to the speed of sound to have a laminar yet velocity-squared dependence. However, the microcantilevers are more flexible than airfoils so they would be expected to respond readily to the lift force if one were present. Most likely, the geometry of the cell combined with the large velocities created turbulence in the flow. One of the requirements for a laminar system, as stated in the turbulence discussion of Section 3.1.1, was that the peak amplitude of oscillation be significantly smaller than the length. The amplification of the amplitude may be an indication that turbulence was occurring.

4.4 Other research findings

Neuzil et al.25 utilized parallel air flow to actuate both SiNₓ (Q = 20) and SiOₓ (Q = 187) cantilever resonances. The bulk of the results are based on SiOₓ cantilevers custom shaped into paddles. The resonance amplitudes were magnified by over three orders of magnitude over a range of 0-20 ml/min; but the whole signal, baseline included, was raised, unlike the results conducted here. The resonant frequency was found to increase and the quality factor to decrease as flow rate increased. A dramatic decrease in quality at 12 ml/min was attributed to nonlinear air flow. The qualitative results of increased amplitude, increased resonant frequency and decreased quality agree with the results of this experiment.
5. CONCLUSION

5.1 Discussion

Four useful signatures of air flow over a resonating microcantilever were measured: static deflection, decreased quality, increased resonant frequency and increased amplitude. Static deflection and decreased quality are expected and supported by previous research and theory. Increased resonant frequency and amplitude were unexpected and likely due to turbulence. They can be harnessed though and better understood with further research. It is intriguing to consider flow as a source of energy and amplification in the field of microcantilevers.

The airfoil effect was not detected, though that would be expected if turbulence existed at the higher velocities. It is also possible that the macroscopic airfoil effect does not work on the microscale, but that does not seem to be the most likely explanation.

5.2 Future work

There is plenty of room for development of the results measured in this experiment. The signatures measured here can all be used as indicators of flow rate. Deflection appears especially reliable and straightforward as a flow meter. Amplitude and frequency increases are indications that flow should be, and evidently already is, considered as an actuation mechanism for microcantilevers. Additionally, they can be further examined to explore the theory of flow and viscosity. It is recommended to perform this experiment with various gases and liquids. The effects of different viscosities in velocity and acceleration damping would be intriguing, especially since the flow seems to act more as a driving signal that dumps energy into the oscillation rather than a damping factor. The difference between pressure effects and drag effects could be explored. At low Reynold’s numbers inertia effectively disappears and the viscosity dominates damping. This behavior could be explored in liquids where the inertia of such a dense phase usually dominates.

The airfoil effect could be further explored. It is recommended that a sturdier cell with larger tubing be developed so that higher velocities and alternate geometries could be used to test
for lift forces. Cantilevers could also be shaped more like airfoils. The spring constants of cantilevers are quite varied and could provide great sensitivity.


Hemmert, W., et al., “Nanometer resolution of 3D motions using video interference microscopy”, 1998, Research Laboratory of Electronics, MIT, Cambridge, MA.


APPENDICES
APPENDIX A. Thermomicroscopes cantilevers

Microlevers™

GENERAL PURPOSE CANTILEVERS

FEATURES:
- Compatible with all major AFM brands.
- Typical radius of curvature: sharpened tips: < 20 nm, unsharpened tips: < 50 nm.
- Available with gold coating for high reflectivity.
- Processed corners for easy sample approach.
- The widest range of spring constants commercially available on a single chip.

ThermoMicroscopes Microlevers are ideal for all contact imaging modes, force modulation microscopy, and liquid operation. The range in force constants enable users to image soft samples in contact as well as high load force vs. distance spectroscopy.

Typical Mechanical Characteristics

<table>
<thead>
<tr>
<th>Cantilever type</th>
<th>A - triangular</th>
<th>B - rectangular</th>
<th>C - triangular</th>
<th>D - triangular</th>
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<th>F - triangular</th>
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<tbody>
<tr>
<td>Standard mode of operation</td>
<td>Contact</td>
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<tr>
<td>Cantilever length</td>
<td>180 µm</td>
<td>200 µm</td>
<td>320 µm</td>
<td>220 µm</td>
<td>140 µm</td>
<td>85 µm</td>
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<tr>
<td>Cantilever width</td>
<td>18 µm</td>
<td>20 µm</td>
<td>22 µm</td>
<td>22 µm</td>
<td>18 µm</td>
<td>15 µm</td>
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<tr>
<td>Cantilever thickness</td>
<td>0.6 µm</td>
<td>0.6 µm</td>
<td>0.6 µm</td>
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<tr>
<td>Force Constant</td>
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<td>0.02 N/m</td>
<td>0.01 N/m</td>
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<td>0.10 N/m</td>
<td>0.50 N/m</td>
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<tr>
<td>Resonant Frequency</td>
<td>22 kHz</td>
<td>15 kHz</td>
<td>7 kHz</td>
<td>15 kHz</td>
<td>38 kHz</td>
<td>120 kHz</td>
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Ordering Information

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</table>

* Not for use with AutoProbe MS systems

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APPENDIX B. Two alternate methods of calculating the resonant frequency.

A second method of vibration analysis, credited to Raleigh, finds the fundamental resonant mode by setting up the conservation of kinetic and potential energy (setting the potential = strain energy) and solving for the parameters. Sarid\cite{5} expresses the strain and kinetic energies as follows:

\[ W_s = \frac{EI}{2} \int_a^b \left( \frac{\partial^2 Y}{\partial x^2} \right)^2 dx = \frac{8EIz_0^2}{l^3} \]  

(24)

\[ W_k = \int_0^l \frac{A\rho}{2} \left( \frac{\partial Y}{\partial x} \right)^2 dx = \frac{A\rho \omega^2}{2} \int_0^l Y^2(x)dx = \frac{52}{405} A\rho \omega^2 l z_0^2 \]  

(25)

Setting them equal yields the same \( \omega_n \) as previously, and \( \kappa = 1.8788/1 \). Chen et al.\cite{7} similarly utilize the variational method in order to identify error due to variations in the cross sectional area and moment of inertia. Their corrections to the resonant frequency are not significant for this experiment.

A curious compilation method takes the deflection of the tip of the cantilever and applies the variational method to \( \delta(U-KE) \) to describe the motion. Walters et al.\cite{11} present equations for determining the spring constant through the equipartition theorem and some geometry, but have difficulty with mass-produced cantilever thickness.
Vita

Stephanie Gregor was born near Chicago in 1975. She graduated from Christian Liberty Academy in 1995. During her five years of undergraduate study, she participated in a variety of research projects and coursework investigating the fields of physics, engineering and chemistry. In 2000, she graduated Georgia Southern University with a B.S. in Physics.

Stephanie is currently working in Oak Ridge, Tennessee.