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Nanoscale Mapping of In-Situ Micro Electro Mechanical Systems with Atomic Force Microscopy

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Nanoscale Mapping of In-Situ Micro Electro Mechanical Systems with Atomic Force Microscopy

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B.S., University of Texas Pan American, 2012

A Thesis
Submitted in Partial Fulfillment of the Requirements for the Degree of Master of Science at the University of Connecticut

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APPROVAL PAGE

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Nanoscale Mapping of In-Situ Micro Electro Mechanical Systems

with Atomic Force Microscopy

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This thesis is dedicated to my abuelita Lety, my siblings, my mother and my late father.

May he rest in peace.
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Abstract

Micro-electro-mechanical-systems (MEMS) are increasingly at our fingertips. To understand and thereby improve their performance, especially given their ever-decreasing sizes, it is crucial to measure their functionality in-situ. Atomic Force Microscopy (AFM) is well suited for such studies, allowing nanoscale lateral and vertical resolution of static displacements, as well as mapping of the dynamic response of these physically actuating microsystems. In this work, the vibration of a tuning fork based viscosity sensor is mapped and compared to model experiments in air, liquid, and a curing collagen gel. The switching response of a MEMS switch with nanosecond time-scale activation is also monitored – including mapping resonances of the driving microcantilever, the displacement of an overhanging contact structure in response to periodic pulsing, and measurements of the synchronization between the switched RF signal and the applied forces. Such nanoscale in-situ AFM investigations of MEMS can be crucial for enhancing modeling, design, and the ultimate performance of these increasingly important and sophisticated devices.
Chapter 1: Introduction/Background

1.1 Overview

Micro electromechanical systems (MEMS) drew attention in the 1980’s as sensors and actuators because they could be produced at low cost and in large volumes using the semiconductor manufacturing techniques and infrastructure already present at the time. [1] While there are several types of MEMS devices, this thesis focuses on a few MEMS that exploit the piezoelectric property of thin film lead zirconate titanate (PZT). “Developments of personal communication devices forced the market to acquire miniaturized efficient devices, which is possible only by the development of radio frequency (RF) MEMS.” [2] Currently, most cellular phone technology uses materials like Aluminum Nitride (AlN) [3] but research shows that PZT exhibits high dielectric constants, lower mechanical and dielectric loss, and has coefficients that are an order of magnitude larger than those of AlN and similar currently used materials [4,5].

A range of characterization methods are used to study the behavior of such structures in order to improve their ultimate functionality. Atomic Force Microscopy is ideal for such purposes, providing nanoscale spatial resolution, the ability to map nanoscale forces, and the possibility for monitoring MEMS devices during in situ actuation. Accordingly, this work focuses on two examples of the implementation of AFM with MEMS. Static measurements are presented for Mechanical Logic cantilever MEMS designs to characterize their mechanical stiffness. Analog to Digital Converter MEMS and Radio Frequency MEMS are then measured during in situ actuation to determine their dynamic performance at the nanoscale. A separate electromechanical system, a millimeter-scale Tuning Fork, is used as a control specimen to prove the measurement concept.
through measurements in air and in tissue engineering collagen gels. This chapter provides background into the relevant experimental capabilities, sample types and configurations, and materials studied in the M.S. thesis research.

1.2 Atomic Force Microscopy

Atomic Force Microscopy (AFM) is a branch of Scanning Probe Microscopy (SPM) that utilizes a cantilever and tip to scan a material and gather different types of data. The information gathered from the interaction between the tip and the sample’s surface can range from topography to magnetic or electrical forces down to the pico-Newton scale. AFM allows us to make measurements in the horizontal X-Y plane as well as in the vertical Z dimension. AFM has several advantages over other forms of characterization; it has high spatial resolution, it does not need a vacuum environment, it can image in air and liquid which allows us to image live biological samples, it can measure vertical dimensions (z direction), samples do not need to be prepared in a special way, and depending on the AFM variation multiple properties can often be acquired simultaneously, allowing them to be superimposed one on another [6]. As seen in Figure 1, AFM detects forces by using a quadrant segmented photodiode to monitor a laser spot that reflects off of the cantilever attached to the tip. As the tip rasteres the sample, the interaction between the tip and the sample causes the cantilever to bend, which in turn causes the laser to change location on the photodiode. The recorded change is what allows us to map loads, and related signals, as a function of position.
Figure 1: Basic schematic view of how AFM works. A laser is reflected from the cantilever to the photodiode. The interaction between the tip and the sample cause the cantilever to deflect, detected by the photodiode to record down to picometer scale changes for property or topography mapping wherever the tip is positioned or scanned.

1.2.1 Contact Mode AFM

Contact mode is one of the two most used operating modes in AFM. Its main distinguishing factor is that it maintains contact with the sample throughout the entire imaging process. This is done by maintaining a constant force to the lever using a feedback signal that is trying to maintain a constant deflection known as the set point. The advantage of contact mode is that it is typically faster than other operating modes, with the major disadvantage especially for soft or fragile specimens that it increases the possibility of damaging the sample.

1.2.2 Tapping Mode AFM

‘Tapping mode’™, also known as Alternating Contact (AC) mode, is the other most common operating mode with AFM. In this mode the probe is tapping or alternating the contact made with the sample. The advantages of tapping mode are that it has better spatial resolution due to it not experiencing lateral force, it exerts a minimal normal force which is ideal for sensitive samples,
and the probing tip is thus able to stay sharper for longer periods of use. Due to the relatively small normal and nearly zero lateral forces, it is more compatible with a wider range of materials, such as those that are very sensitive and could be damaged through the use of the contact operating mode. Another advantage of this mode is that due to the virtue of tapping, one can gather additional information such as phase or obtain data in relation to surface adhesion forces of the sample.

1.3 Cantilever Mechanics

In all of the experiments done for this thesis, we encounter cantilever beams with one fixed end and one free floating end. Furthermore, this type of cantilever beam can be broken down into several more subcategories. For these experiments, three standard types of cantilever beam equations can be used; one with a concentrated load at the free end, another with a concentrated load at any point, and the last with a uniformly distributed load as seen in Figures 2-4. The equations for slope at the free end ($\theta$), maximum deflection ($\delta_{\text{max}}$), and deflection ($y$) at any point in terms of $x$ are shown after their respective figures.

The case of a load applied at the very end of the lever is a reasonable approximation of standard AFM where the tip is located at the end of the cantilever. The point, $l$, is thus where the AFM tip exerts a load ($P$) on the cantilever.
Figure 2: Shows the profile diagram of a cantilever beam with a concentrated load, P, at the free end. $\delta_{\text{max}}$ is the maximum deflection of the cantilever, $\theta$ is the slope at the free end.

To figure out the slope, deflection at any given point, and the maximum deflection, of a cantilever beam with a concentrated load at the free end, beam deflection formulas 1-3 are used.

$$\theta = \frac{P l^2}{2EI} \quad (1)$$

$$y = \frac{Px^2}{6EI}(3l - x) \quad (2)$$

$$\delta_{\text{max}} = \frac{Pl^3}{3EI} \quad (3)$$

$E$ is the modulus of elasticity also known as the Young’s Modulus, which describes the tendency for a material to deform along its axis when an opposing force is applied. The moment of inertia, $I$, is the capacity of the cross section about a neutral axis to oppose bending, typically equal to the lever width times the cube of the thickness divided by 12 and thus very sensitive to the lever thickness. $X$ is any point along the lever, while $l$ is the overall length of the lever and position of applied load (P).

A profile diagram for a cantilever beam with a concentrated load at any point along the x axis, which is relevant to the condition of the AFM tip scanning a static MEMS lever seen in the non-dynamic imaging section of this thesis is shown in Figure 3 and described by equations 4-7. The same parameters are applicable as for Equations 1-3, with additional terms defining the distance
from the base of the lever to the loading location (a) and the remaining distance (b) to the lever end (at l).

Figure 3: Profile diagram of a cantilever beam with a concentrated load at any point along the x axis.

\[ \theta = \frac{P a^2}{2EI} \]  
(4)

\[ y = \frac{P x^2}{6EI} (3a - x) \text{ for } 0 < x < a \]  
(5)

\[ y = \frac{P x^2}{6EI} (3x - a) \text{ for } a < x < l \]  
(6)

\[ \delta_{max} = \frac{P a^3}{6E l} (3l - a) \]  
(7)

The most common experimental condition for in situ actuation experiments presented in this thesis combines the above with actuating the MEMS levers with a function generator. Thus we can assume the cantilever beam experienced a uniformly distributed load as seen in Figure 4. Omega (ω) is the distributed load per unit length, assumed for simplicity to be constant across the entire lever length as shown. Equations 8-10 are relevant to Figure 4. Technically, an AFM interacting with a MEMS lever actuated with such a distributed load also requires superposition of the AFM set point force (P) at some position along the lever (a=0 to L). In practice, however, the AFM load
is negligible compared to the distributed load and is ignored in this work for in situ measurements of piezoactuating MEMS devices.

![Profile diagram of a cantilever beam with a uniformly distributed load, $\omega$.](image)

**Figure 4: Profile diagram of a cantilever beam with a uniformly distributed load, $\omega$.**

\[
\theta = \frac{\alpha x^3}{6EI} \quad (8)
\]

\[
y = \frac{\alpha x^2}{24EI} (x^2 + 6l^2 - 4lx) \quad (9)
\]

\[
\delta_{max} = \frac{\alpha l^4}{8EI} \quad (10)
\]

These equations for cantilever mechanics are used to describe the behavior of the AFM cantilever during our measurements, and to interpret the results from the actuating levers in the MEMS devices studied in this work. Figure 5 is an illustration of a single actuating lever from the Analog Digital Converter MEMS device explained later in this thesis. The long rectangular beam is the PZT, the slightly narrower thin structure on top is an electrode (always grounded for AFM experiments since the tip contacts it), an underlying electrode (biased) cannot be seen due to the perspective, and the integrated perpendicular bar making up the ‘T’ shape is actuated by this lever to make contact with independent overhanging electrodes (not shown). The AFM probe will be positioned either along the static or actuated MEMS beam, or on any overhanging electrodes, to determine the local mechanical properties and or to monitor the actuation directly.
1.4 MEMS

The inexorable march for faster, smaller, cheaper, lower power consumption, more mass producible, more functional, and more integrated devices led to the development of the related field of Micro-Electro-Mechanical-Systems (MEMS) and their still smaller nanoscale equivalent Nano-Electro-Mechanical-Systems (NEMS). Strongly leveraging semiconductor fabrication technologies, these devices with sub-micron scale mechanical features are approaching a $10 billion per year industry. This is largely driven by the incorporation of MEMS accelerometers and
gyroscopes [7-9] in automobiles, smartphones, and other consumer products. Micro-electro-
mecanically actuated structures are also implemented in adaptive optics for improved imaging
[10-12], and are widely employed commercially in projection displays [13]. Because of their
diminutive size, mass, and inertia, MEMS and NEMS can operate from DC to GHz frequencies or
beyond, enabling low power RF switches [14], high Q electro-mechanical filters [15], and
mechanical digital logic functions [16]. They are central to miniature timing and frequency control
applications [17] as well, including a highly portable Atomic clock [18].

A rapidly expanding offshoot of such electromechanical devices is the development of
microfluidics [19] and micro-pumping [20]. Active and passive MEMS and related technologies
have enabled advances in DNA separation [21], biology [22], medicine [23], and drug delivery
[24]. MEMS implants [25] are also being developed and implemented to assist healing and/or
enhance biological functions, with many microrobotics systems leveraging MEMS [26]. As tools,
dedicated MEMS chips exist allowing materials characterization [27], with recent MEMS chips
being explored for power generation, primarily as resonant [28] or non-resonant [29] energy
harvesters.

Naturally, this range of applications presents substantial challenges in materials, processing,
design, and characterization [30]. Electro-mechanical performance depends on the actuation
principle, typically achieved with capacitive forces, shape memory effects, or piezoelectric effect
[31]. These devices can be easily tuned through dimensional control and electrode shaping [32],
to manipulate spatial and temporal electrical and strain fields. Self-assembled-monolayer coatings
[33] can address the very common issue of adhesion and wear [34] and device stability. Uniformly,
though, practical applications of MEMS necessitate a thorough understanding of the behavior of
the electromechanical system in the appropriate end-use environment. Measuring the response to static or dynamic natural loads, as well as the environment, is therefore crucial [35].

Along these lines, optical microscopy of operating MEMS structures is an obvious and common solution that includes implementing high speed cameras or stroboscopy to observe motion of MEMS components. Such homodyne or heterodyne [36] interferometry can accurately measure positions and/or displacements during actuation; therefore, local mechanical properties can be calculated when loading and boundary conditions are known [37]. Digital image correlation of feature positions or markers during actuation can be used to map active strain fields as well [38].

Many examples of in-situ electron microscopy of MEMS materials have also been reported. TEM in particular has been implemented during indentation to locally explore elastic moduli, pressure-induced phase transitions, and especially size effects [39], even at cryogenic temperatures [40]. In-situ nanoindentation and electron microscopy of MEMS structures themselves have primarily focused on 1D structures, for example wires or ribbons [41].

In order to determine the mechanical and tribological properties of MEMS/NEMS components, atomic force microscopy is widely implemented. This includes numerous studies of wear effects for MEMS materials [42], lubrication layers [43], and biological molecular coatings and biomimetic nanostructured surfaces [44]. The focus of this work, on the other hand, is to leverage the excellent spatial resolution of AFM, along with its capability to map periodic motion with amplitudes down to pm, for mapping the static and dynamic functionality of fully assembled MEMS systems operating in-situ.
1.4.1 PZT MEMS

Lead zirconate titanate (PZT) possesses piezoelectric, pyroelectric, ferroelectric, and electrostrictive properties. This versatility is employed in a wide range of applications. It is used in bulk actuator applications and in low cost ceramic filters due to its piezoelectric properties [45,46]. Its pyroelectric properties are utilized in infrared radiation (IR) detectors and imagers [47]. The ferroelectric properties are used in non-volatile memory (memory that doesn’t need to be constantly refreshed and is saved regardless if the device is on or off) like ferroelectric random access memory (FRAM) technology [48-51], which is faster and uses low power compared to other storage technologies. Of course for piezo MEMS devices, the piezoelectric property of thin film PZT is the primary property exploited.

There were 3 types of PZT MEMS designs used in this thesis: the Basic Block, Analog Digital Converter (ADC), and RF Block. The Tuning Fork is a larger scale example of such cantilever MEMS designs used for control experiments, and though it technically uses LiNbO3 levers instead of PZT, it operates equivalently at least for the frequencies of interest.

1.4.2 Mechanical Relay Basic Block MEMS

More energy efficient technology is needed for improving battery lifetime and reducing the scale of portable consumer electronics. One route is to implement low power integrated circuits, achieved through low voltage operation but often at the cost of degraded performance. Furthermore, the miniaturization of IC devices has increased static power losses to levels
comparable to the dynamic power consumed (i.e. during operation) [52]. Utilizing mechanical relays as in MEMS switches enables zero static power consumption. This is because when in the open condition, there is an air gap of approximately 2 µm, completely preventing the flow of leakage currents resulting in a relay with over three orders of magnitude lower static leakage than the more common CMOS technology [3]. Furthermore, low resistance contacts coupled with the large displacement PZT actuator enable a dynamic power I-V slope that is significantly sharper than those achieved by other methods such as MOSFET, while the activation voltage is about a third of a theoretically optimal MOSFET device [53]. MEMS relays have been demonstrated throughout the military specification operating temperatures as well, ranging from -55 °C to 125 °C. The mechanical relay basic block MEMS studied in this thesis comprises seven sets of 1 bit basic structures, as seen in Figure 6a.

Figure 6: Optical images of: (a) typical specimen with 7 distinct Basic Block MEMS devices (blue colored regions); (b) one basic block unit with 2 sets of actuators with 6 distinct contact lines and 3 electrode pads shown, and (c) contacts for directly biasing (from top left) and grounding (at center approaching from top) a single side of the Basic Block, displaying the dual-driven-cantilever design to force an attached gold pad (at right) into contact with two independent overhanging electrodes (far right).
The purpose of these MEMS devices is to provide 6 types of logic functionality based on binary biasing configurations (ground or V) for six independent IO connections per basic block, as seen in table 1. A and B are two independent input electrodes, the operation is read at output O, while D, S, and G indicate drain, source, and gate electrodes labelled according to CMOS notation. This device therefore mimics CMOS transistor logic, except with enhanced off-state performance.

<table>
<thead>
<tr>
<th>Operation</th>
<th>IO</th>
<th>GS</th>
<th>VS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Buffer</td>
<td>A</td>
<td>GND</td>
<td>VDD</td>
</tr>
<tr>
<td>Inverter</td>
<td>A</td>
<td>VDD</td>
<td>GND</td>
</tr>
<tr>
<td>AND</td>
<td>A</td>
<td>GND</td>
<td>B</td>
</tr>
<tr>
<td>OR</td>
<td>A</td>
<td>B</td>
<td>VDD</td>
</tr>
<tr>
<td>Not A or B</td>
<td>A</td>
<td>VDD</td>
<td>B</td>
</tr>
<tr>
<td>Not A and B</td>
<td>A</td>
<td>B</td>
<td>GND</td>
</tr>
</tbody>
</table>

Table 1: Details of the six basic operating sets for the basic block MEMS device.

1.4.3 Analog Digital Converter MEMS

In our current digital age, data is still characterized by analog signals, even though almost everything we use in today’s technology has a microprocessor that internally works with digital signals. Analog to Digital Converters (ADC) are therefore necessary, as they read analog signals from external inputs and output digital signals with which a microprocessor perform its various functions. Common applications for ADCs include converting the analog signals being received
through a telecommunications line, (analog) TV tuner cards, microcontrollers, digital storage oscilloscopes, computer music reproduction, microphones, and mobile devices to name a few. Mobile device technology has especially advanced the development of ultra-low power digital communication systems. In terms of actuation voltage and leakage current specifications sought by the mobile industry, PZT based piezoelectric MEMS such as those studied herein have an advantage over the more common AlN and electrostatic MEMS [54]. For this thesis, a PZT piezoelectric Analog Digital Converter MEMS design, as seen in Figure 7a, was studied. It comprises a series of eight functional PZT actuating levers of varying lengths that provide eight unique digital output levels. Each of these actuator elements includes an actuating lever of a distinct length, and a pair of normally open contacts. The bank of 16 gold (Au) contacts are shown at left, paired up to overhang Au contact pad affixed to the end of one of the 8 possibly actuating levers. As labelled, there is a single analog input bias (the analog signal to be converted, which is equally applied to the tops of all 8 of the MEMS cantilever actuators), a single input ground connection (back side of the actuating levers), a digital input (constant positive voltage applied to 1 of each of the 8 pairs of overhanging electrodes), and eight digital outputs that only yield a high bias (positive voltage from the digital input) if the underlying lever(s) are actuated and bridge any given pair(s) of contacts. One PZT lever without an output is also designed at each end for control purposes, visible only at the bottom of Figure 7b.
Figure 7: Optical images of a single ADC MEMS layout (a), and a bank of 8 distinct PZT piezoMEMs actuators providing 8-bit digital output of the analog input bias (b).

1.4.4 RF MEMS

As discussed herein, RF MEMS refer to the design and fabrication of MEMS devices used in RF integrated circuits. They may also refer to MEMS that operate from 0.1 to 100 GHz, which encompasses millimeter to microwave frequency ranges [2, 55]. RF MEMS can be classified into 3 groups: 1) RF Extrinsic, where the MEMS is located outside the RF circuit and can control other devices in the RF circuit, 2) RF Intrinsic, where the MEMS is located inside the RF circuit and can serve as an actuator as well as perform RF circuit functions, and 3) RF Reactive, where the MEMS is inside, and has an RF function that is coupled with attenuation. The RF MEMS in this thesis fall under the RF Intrinsic category [2].
The RF switch is likely the most studied RF MEMS component. There are two types of RF MEMS switch contacts which separates them into two corresponding groups: capacitive and ohmic. Capacitive contact switches use a thin dielectric thin film between two metals, while the ohmic contact switch uses two metals to create the switch contact. Capacitive contact switches are optimal for higher frequencies and rely on capacitive coupling of the RF energy and the dielectric to the switch contact material. Ohmic switches allow broadband operation operating from DC and upward [56]. Either RF MEMS switches offer advantages over other switching devices like PIN diodes and FET switches [57] such as: near zero power consumption, high isolation, low insertion loss, linearity, and low cost [55, 58]. Two critical criteria to the design of RF MEMS are isolation and insertion loss [56]. Isolation refers to the measure of power leaking when there is no signal transmission in the off state or open switch. Generally, isolation is measured as the ratio of the output power to the input power in terms of decibels (see Equation 11) [59]. Power can be leaked by capacitive coupling, conductive losses at the surfaces, and mismatch losses [59]. Insertion loss is the measure of the signal transmission efficiency in the on state or closed switch. It is the measure of the incident power minus the reflected and transmitted power. Insertion loss can be attributed to contact resistances, conductive losses from skin effects, dielectric absorption, and reflective losses from impedance mismatches [59]. Insertion loss is defined as the ratio of input signal to output signal, given in decibels (see Equation 12) [59]. Since the output power is always less than the input power, insertion loss is reported as a value greater than 0 dB [56]. Linearity refers to the mathematical relationship that two variables are linearly proportional to one another, in the case of RF MEMS it refers to the linearity of the input power and output power of the device. A key issue in RF MEMS switch technology is the compact design of MEMS cantilever beams [55]. The RF Block MEMS design used for this thesis, as seen in Figure 8a, shows a block of RF
MEMS sets with different designs. Each design has one or two bias electrodes, one ground electrode, an RF input electrode and an RF output electrode as seen in Figure 8b.

\[
Isolation = 10\log \left( \frac{P_{\text{output}}}{P_{\text{source}}} \right)
\]  

(11)

\[
Insertion\ Loss = -10\log \left( \frac{P_{\text{output}}}{P_{\text{input}}} \right)
\]  

(12)

Figure 8: RF MEMS block design (a), zoomed in RF MEMS with relevant electrodes labeled RF Input, RF Output, Ground, and bias (b).
1.5 Tuning Fork

Electro-mechanically actuating devices are important for a wide range of applications, ranging from scientific tools to commercially available products. Tuning forks, for example, are used as mass sensors for molecular [60] and biological adhesion [61], and can detect gas density [62] including humidity [63]. They have similarly been employed in magnetometry [64] or as spectroscopic gas sensors [65]. They can even be designed for compatibility in harsh environments, acting as strain sensors [66], to measure viscosity [67, 68], or serve as accelerometers in automotive applications [69]. Scientifically, they are central to many advanced variations of the family of characterization techniques known as scanning probe microscopy, including Atomic Force Microscopy (AFM) [70], near field scanning optical microscopy [71, 72], scanning electrochemical microscopy [73], atomic resolution imaging and atomic manipulation [74], and high-speed AFM [75]. They have been crucial to many recent advances including superfluidity phenomena in quantum fluids such as He II [76, 77], and even identifying phase transitions in solid He [78]. In the simplest case, they operate by detecting a shift in the frequency, amplitude, phase, or Q-factor of a mechanical resonance that is usually piezoelectrically driven, providing sensitivity to the surrounding environment. Depending on their design, this typically leverages a primary resonance in the Hz to kHz range.

The piezoelectrically activated tuning forks used in this study were produced by micromachining LiNbO₃ wafers (IDB technologies, UK) with Cr/Au electrodes sandwiched between two wafers that were subsequently bonded together producing a liquid tight seal, similar to ones described elsewhere [78]. The outer surface of the tuning forks is coated with thermally evaporated Cr/Au conductive layer eliminating stray electric field in the surrounding media under test. The tuning
fork electrodes were contacted to isolated holder wires via standard conductive epoxy and sealed using high performance epoxy (3M, USA). Tuning fork tine dimensions include a length of 4 mm and width of 2 mm, resulting in a resonance frequency in the range of 50 to 60 KHz.

1.6 Collagen Gel

Collagen is the main structural protein of the various connective tissues in animals, anywhere between 25-35% of the body’s protein is collagen [79]. Collagen is also found in muscle tissue, approximately between one to two percent of muscle tissue is collagen. Collagen facilitates successful adaptation in vitro culture and enhances the expression of cell-specific morphology and function. High Concentration (HC) collagen is used in applications where a sturdy gel provides maximal support to maintain a three dimensional structure. This gel has been used as a culture medium for cells [80], and has great utility as a scaffold for engineering several tissue types. Twenty eight types of collagen have been identified and described, the most abundant in the human body (~90%) being Type I collagen. Type I collagen forms elongated fine fibers which are present in scar tissue, tendons, ligaments, vascular ligature, organ capsules, corneas, and organic components of bone and teeth. The gel used for this thesis was 3 mg/mL gel Type 1 High Concentration (HC) collagen from Rat tail.
Chapter 2: Procedures

2.1 Sample Preparation

The MEMS samples were created in the PiezoMEMS Technology Lab at the Army Research Lab. Wirebonding, as seen in Figure 9, was performed in the Institute of Material Science (IMS) clean room at the University of Connecticut. The collagen gel samples were made in the Nanomeasurements Lab in the IMS after the constituents were obtained from collaborators in the University of Connecticut Health department in Farmington.

![Optical Image of RF Block MEMS with nanowire bonding](image)

Figure 9: Optical Image of RF Block MEMS with nanowire bonding. AFM tip can be seen out of focus on the lower left side.

2.1.1 MEMS Sample Preparation

At the Army Research Lab in Adelphi, MD, 500 nm of PZT (52/48) is deposited onto a matching layer of TiO2/Pt (33/100 nm), a base of 300 nm of SiO2, 50 nm of Si3N4, and 150 nm of additional SiO2 60 as seen in Figure 10.
Using <nanobonding machine name>, electrical contacts on the ADC and the RF Block MEMS were wire bonded such that the top electrode on the piezoMEMS actuators is grounded (to protect a contacting AFM probe) while the bottom electrode can be biased. Care was taken to route the wiring to allow AFM imaging of the MEMS structures during their actuation (essentially all wire traces can only cover a ~225° arc, allowing the AFM tip, lever, and holder to approach the specimen without interference). The RF Block MEMS had two additional electrodes wire bonded, allowing the RF Input Signal and the RF Output Signal to be monitored. The Basic Block MEMS was not wire bonded since its mechanical properties were studied but its performance was not mapped in situ.

2.1.2 Gel Sample Preparation

Two types of gel samples were prepared, a 3mg/mL collagen gel sample and a control sample that had no collagen added. To obtain the 3 mg/mL sample, the collagen gel is formed according to the manufacturer’s protocol (BD Biosciences) based on the following constituents: 300 μl sterile 10X Phosphate Buffered Saline, 22.310 μl sterile 1N NaOH, 1.7 ml cell culture media, and for all but
control experiments 970 μl of Rat Tail Collagen HC (0.3% type 1, BD Biosciences). Collagen gel is maintained on ice to prevent gelation up to 2 hours.

2.2 General Set Up

One Lock-In-Amplifier (LIA) (Stanford Research Systems Model SR830 DSP, USA) was used to obtain the results for the Basic Block MEMS. One lock-in-amplifier (Stanford Research Systems Model SR8440 DSP, USA) and a Function Generator (Agilent 33220A, USA) were used to measure the results in the ADC MEMS. Two lock-in-amplifiers (SR830 and SR844) and a Function Generator (Agilent 33220A) were used to perform the experiments of the RF Block MEMS. A different lock-in-amplifier (Stanford Research Systems Model SR850 DSP, USA) was used for the Tuning Fork experiment with the collagen gel. In each case, the purpose of the LIA is to monitor the amplitude and/or phase of surface oscillations, detected by the AFM probe resting on the specimen surface, with respect to an AC driving voltage applied to the MEMS actuators. For the RF-block MEMS device, the 2nd LIA also serves to measure the RF transmission across the RF switch of a MHz scale signal, and how this transmission varies with MEMS actuation conditions.

In most cases, the AC voltage amplitude, frequency, pulse width, and/or other parameters are automatically controlled using Agilent Vee equipment control software. Signals are detected either using an ADC (Measurement Computing DAS4020), or by the AFM during scanning.
2.2.1 MEMS Set Up

The Basic Block MEMS was positioned on the AFM and topography images were obtained using Tapping mode. Different forces were applied by the AFM tip to measure the deflection of the Basic Block actuating levers and to obtain their local spring constants.

The ADC MEMS were set up on the AFM and the input bias and ground electrodes of the actuating cantilevers were connected to the function generator’s output channel. The deflection channel from the AFM and the Sync Channel from the function generator were then connected to the lock-in-amplifier input and reference signal pathways.

The RF Block MEMS was connected as described for the ADC MEMS case, with the addition of a second lock-in-amplifier. The reference output channel of the second lock-in-amplifier was connected to the RF Input electrode, providing a signal of 1 V at a frequency of 20 MHz. The RF Output electrode was connected to the signal-in channel of this second lock-in-amplifier, thereby continuously monitoring the RF transmission and any changes resulting from RF-MEMS switching.

2.2.2 Tuning Fork Set Up

The tuning forks were studied in two ways. In the first setup, their resonant frequency was monitored in ambient air for control purposes, and separately in liquid during curing of a nanofibrous collagen gel to monitor the curing process by detecting changes in the gel viscosity. In either case the resonant frequency was determined by sweeping a drive frequency and monitoring the amplitude and phase of the impedance with a lock-in-amplifier (Model SR850
DSP, USA). These measurements were performed in a thermally isolated chamber since the resonant frequencies can be sensitive to temperature changes.

In the second setup, a tuning fork was mounted lengthwise in the AFM such that the tines oscillated along the tip axis (perpendicular to the lever). The exposed tine was contacted directly by the AFM probe to finely detect vibration amplitudes down to the picometer scale.

2.3 AFM imaging parameters

AFM measurements were performed with an Asylum Research MFP-3d microscope, operated in contact mode using Silicon probes (Olympus AC160 or AC240) with free resonant frequencies of approximately 300 or 70 kHz and stiffness values of approximately 40 or 4 N/m, respectively. In order to measure the displacements of the MEMS structures or the tuning forks, the deflection of these levers due to sample actuation is calibrated before every experiment using Asylum Research’s built in GetReal™ Calibration function to units of nm from Volts of displacement on the AFM’s quadrant photodetector. This calibration is based on the widely employed static indentation method [81], with typical values on the order of $50 \text{ nm V}^{-1}$. Contact set points on the order of 0.1-1 Volts are employed throughout, although in one measurement values as high as 8 V were purposefully employed as described in this thesis. It is of the upmost importance to calibrate these levers daily due to their sensitivity to several factors including room temperature and humidity. The deflection and spring constant of the same lever can vary more than 10 nm $\text{V}^{-1}$ and 5 nN/nm respectively from one day to the next. Calibrating these levers before every experiment is essential to obtain the most accurate data for proper analysis.
2.3.1 Static Imaging of MEMS

The first MEMS sample experimented on was the Basic Block design. Using the AFM tip, topography images of the actuating lever were obtained. Using contact mode, five different forces ranging from 1000 nN to 6000 nN were applied to the actuating cantilever to determine their deflection and the spring constant of the actuating levers, which plays a key role in determining the actuating voltage of a MEMS [55]. After obtaining sufficient data, a Scanning Electron Microscope (SEM) (JEOL JSM 6330F, Japan) was used to obtain images of the various MEMS structures in order to understand the often complicated device and electrode layout as well gather more detail of the Basic Block MEMS for topographic comparisons as exemplified by Figure 11.

Figure 11: SEM image of the base of the actuating cantilevers of the Basic Block MEMS (a). An AFM topography image of the area roughly enclosed in the rectangle seen in part a (b).
Topography images of the ADC MEMS structures were also obtained as seen in Figure 12.

2.4.1 In-situ Imaging of MEMS Dynamics

Analog to Digital Converter MEMS switches and RF MEMS switches were experimented on for in-situ imaging. Typical AFM imaging conditions for in-situ actuating piezo MEMS devices include set point forces of 0.3 V and a standard gain setting of a range between 10 and 18. Some measurements were performed with the AFM probe fixed at a single location, while most were conducted during scanning anywhere from 10 nm to 35 µm. Given the long aspect ratio of the MEMS levers, image dimensions of 4:1 through 32:1 were implemented in order to both save time as well as not to ‘fall off’ of the suspended MEMS cantilevers. Using two lock-in-amplifiers, data was recorded for the AFM tip using the Height, Deflection and Tip Amplitude channels as well as the amplitude of the RF Output signal. Varying actuating frequencies and actuating voltages were performed and analyzed.

2.4.2 In situ Measurements of Tuning Fork

Two types of in-situ Tuning Fork measurements were performed. As a macroscopic control, Figure 3 (a) presents common frequency responses of a macroscopic tuning fork, measured by analyzing the AC signal from a voltage divider across the tuning fork with a lock-in-amplifier (arbitrary units are shown) during frequency sweeps across 3 kHz in 25 Hz steps (i.e. 120 points per line) in air. The peak amplitude and frequency, i.e. resonance, are analyzed, which depend on a range of parameters related to the surrounding media as noted in the introduction. Since this does not involve the AFM, such measurements are compatible with any environment, in this case employed
in a curing solvent and collagen gel, in a liquid of the gel solvents but without the collagen gel agent as a control, and in air as a second control.

A similar process as the In situ measurements for the MEMS devices described in the previous section was also performed with the AFM for nanoscale investigations of the tuning fork operation. In particular, the vibration amplitude of the Tuning Fork was measured as a function of frequency by a contacting atomic force microscope probe positioned near the end of one of the counter-vibrating tines. This required mounting the tuning fork as sketched in Figure 2(a), the amplitude is directly measured by monitoring deflection of the high frequency AFM probe and again analyzing the signal with the lock-in-amplifier. For a fixed driving amplitude of the tuning fork, 15 consecutive frequency sweeps were made, Figure 2(b), again each offset for visual clarity (by 200 a.u.).

2.5 Collagen Gel

After setting up the Tuning Fork inside the thermally isolated chamber, and adding the collagen to the rest of the thermally equilibrated components as described earlier, the collagen gel (or control solvents without the gelling agent) is held at 37 °C according to the manufacturer’s protocol. The Agilent-Vee program then automatically sweeps frequency and measures the impedance via a voltage divider every two minutes. In this manner, any change in resonant frequency of the vibrating tuning fork can be identified in order to draw conclusions about the stiffness and density of the collagen gel during the roughly 30 minute curing process.
Chapter 3 Analysis and Results

3.1 Non Dynamic Imaging of MEMS

Figure 12a shows a backscattered electron SEM image of the T-shaped actuators described in Figure 13, with AFM topographic images, a 2x2 micron image to try to see the surface resolution of the overhang in Figure 12b, while a 16:1 ratio of 20 micron image of the overhang itself to further understand its shape and its response in Figure 12c. A profile cross section is displayed in Figure 12d, to give us an easier understanding of the shape of these crucial components.

Figure 12: Backscattered electron SEM image of a MEMS structure, overhanging contacts on the left with the T-shaped actuator on the right as described in Figure 13, the red rectangle depicts the area scanned in part c (a), a zoomed in AFM topography image of the blue square in part c of the overhang (b), a topographic image of the overhang contact (c), with the corresponding profile (d).
For a given AFM set point force (F), the active MEMS lever should respond similar to an AFM cantilever, and thus deflect normally (in the z direction) proportional to the load according to Hooke’s law \( z = \frac{F}{kc} \). But since the load is applied by the AFM tip at every imaged location \( l \) while scanning along the lever, it is important to note that the measured change in height due to distortion of the MEMS structure at that location \( z_l \) can reveal a local spring constant \( (kc, \text{local}) \) according to Equation 11. Only at the end of the lever \( (l=L) \) would the conventional spring constant \( (kc) \) be reached.

\[
k_{c,\text{local}} = k_c \left( \frac{L}{l} \right)^3 = \left( \frac{Ewt^3}{4l^2} \right) \left( \frac{L}{l} \right)^3 = \left( \frac{Ewt^3}{4l^3} \right) = \frac{F}{z_l} \quad (11)
\]

After obtaining data on the deflection of the actuating cantilever beams, the data was normalized for better visual representation with the initial force of the tip being 0 and all successive deflections due to the respective increase in force applied by the tip is shown in Figure 13. By successfully tuning and obtaining the AFM cantilever tip spring constant and deflection invOLS prior to every experiment, the local spring constant of the actuating cantilever in relation to the length of the cantilever beam can be calculated. Figure 13a), depicts a secondary electron SEM image of a T-shaped cantilever, where the suspended T-structure is outlined by light dashed lines. The AFM is first scanned along this 35μm long microcantilever within the indicated dark dotted region, such that the left edge identifies the end of the underlying supporting structure (the ‘suspended base’). Two primary measurements are conducted in this manner, one to determine the spring constant of the MEMS structure, and the other to monitor its electro-mechanical actuation in-situ. Overhanging the T-shaped structure are two gold cantilevers approaching from the right of Figure 13(a). These independent electrical leads are suspended above the T-section, allowing them to be connected when the T-shaped cantilever contacts them both (upon actuation by sufficiently biasing
the PZT thin film). Additional AFM measurements on one of these overhangs, within the slightly rotated rectangle, are also described below.

Figure 13: Secondary electron SEM image of a MEMS structure suspended where noted by the dashed lines (a), AFM measured height profiles of the MEMS structure for a sequence of distinct AFM applied loads up to 6 μN, all imaged from the base of the suspended region within the area sketched by the dotted lines (b), and the corresponding local spring constant calculated based on Hooke’s law every 63 nm (each original image pixel in the x-direction) along the lever length (c).

Accordingly, Figure 13(b) presents the measured height along the MEMS lever from a multiparametric sequence of consecutive images similar to Figure (5Mp), except height is acquired with a different set point for each image frame. For brevity only the average height across the lever (in the y-direction) is displayed for position (l) at each load (F), but of course it could be fully mapped. To accommodate tilting in AFM images that is omnipresent, though constant for a given
imaging session, each height profile is adjusted so that the linearly extrapolated MEMS distortion for zero applied force is exactly zero (i.e. a constant background slope is subtracted from all data, as is common in SPM analysis). Visually, the deflected height of the MEMS lever does indeed appear to be linearly proportional to the applied force for any given position \(l\) along the length of the lever.

Calculating the slope at every single location, resolved down to a spacing of 63 nm, yields the measured local spring constants, Figure 13(c). The fitting quality \(R^2\) coefficient indicates a good fit except near to the base of the structure (>0.98 beyond 1 \(\mu \text{m}\)), which is where deflection is minimal and hence the instrumental signal to noise ratio is expected to introduce error. In fact error bars are overlaid displaying the standard deviation of the slope for each position, but are only prevalent nearest the lever base. This validates the linear (Hooke’s law) fit, as well as the linear approximation to determine the suitable baseline height and slope when calculating the \(F=0\) condition. For a fixed lever width, thickness, and materials parameters, according to the local spring constant is expected to change with position, inversely proportional to the 3\(^{rd}\) power. These measured results do not, however. This is likely due to the fact that the MEMS structures are more complex compared to a simple cantilever beam, with multilayers of Silicon, PZT, electrodes, and matching layers, as well as a width and thickness that is not designed to be constant along the length. Furthermore, strain within the beam components, and/or at the interfaces, may also play a role that could spatially vary. Such seemingly subtle effects may be difficult to anticipate and/or model, confirming the value of direct measurements of such MEMS devices.
3.2 In Situ Imaging of MEMS

3.2.1 In Situ Imaging of Analog to Digital Converter MEMS

In the Analog to Digital Converter MEMS, data was obtained from imaging the actuating cantilever as seen in Figure 14a. Offset deflection, offset amplitude, and offset phase were all collected with the set point as the variable. Another set of experiments were done with the variable being the drive amplitude. Drive frequency was the next variable that was experimented on. After sufficient data was obtained from the actuating cantilevers imaging of the ‘overhang’ with the AFM was performed. The overhang, as seen in Figure 14b, is the area made of two overhanging contacts that the cantilever beam impacts when actuated allowing for the circuit to close.

![Figure 14: Optical image of the AFM tip scanning on an actuating cantilever in a ADC MEMS (a). Optical Image of same size of AFM tip scanning on the overhang contacts (b).](image)

To monitor the dynamic response of the MEMS structure during in-situ actuation, the same experimental configuration is employed as the non dynamic imaging except an AC sinusoidal bias
is applied to the electrodes that sandwich the piezoelectric film embedded in the suspended active cantilever. The tip, riding on the piezoactuating surface akin to Figure 30 (a) with the tuning fork tine, is then scanned in a 20 by 1.25 μm swath (16:1 aspect ratio) of the MEMS lever as for Figure 13. This is then repeated 11 times in the same area, each with a distinct ac frequency of actuation of the MEMS structure similar to Figure 31, in this case from 800 to 900 kHz in steps of 10 kHz with 3 Vac. The ac-deflection of the AFM lever is analyzed with a lock in amplifier, just as with Figure 30 (b), and recorded during imaging.

Figure 15 presents the resulting montage of AC amplitude (left) and phase (right) images for the MEMS structure in this nearly MHz frequency range.

Figure 15: Resonant nodes revealed in eleven maps of amplitude (left) and phase (right) of an actuating piezoelectric MEMS lever in the same 20 μm by 1.25 μm area, as a function of drive frequency between 800 kHz (base) and 900 kHz (top) with steps of 10 kHz between frames as labelled.

A node in the AC response is clearly apparent in the amplitude and phase data, located at approximately 16 μm during 900 kHz excitations but evidently shifting left with lower frequencies (down in the figure). To the left of the node the amplitude is weaker than to the right. The node
itself exhibits a paired antipeak and peak. Near $x=0$ μm, a second node becomes apparent at 830 kHz. Both nodes converge at approximately 810 kHz, with hardly any evidence of these nodes at 800 kHz where the lever response is almost uniform over the measured length.

The actual mode and harmonic excited in this frequency range is unknown for the particular MEMS structure imaged (but could be with laser Doppler vibrometry). Sophisticated modelling has been performed for similar devices, though, to consider first and higher order modes and even leverage these modes for enhanced MEMS performance [82]. It is certainly the case that the nearly-MHz frequencies measured are beyond the first harmonic of the first mode for the lever. Qualitatively similar results were separately observed in other frequency ranges. Furthermore, this experiment was repeated on identically designed, but shorter or longer, MEMS structures, with similar results as seen in Figure 16. As expected, the nodes were found at a different frequency, since they strongly depend on the geometry of the resonating device.

![Figure 16: Resonant nodes in a five amplitude image collage of 20 x 1.25 μm area of the piezo actuating levers at lower frequencies due to the geometry.](image)
Similar experiment is repeated five times on a different actuating lever, each with a distinct set point increasing from 0.1 to 0.5 V in 0.1 V steps with a drive frequency of 395 kHz and a drive amplitude of 3 V where the collage of height images can be seen in Figure 17. An oscillating pattern is visible while the increasing set point is creating a larger magnitude in the lever. This could possibly be due to a stronger contact between the conducting tip and the sample or the image is not real due to the lack of synchronization paired with the rastering pattern of the AFM.

![Figure 17: Height topography of Analog to Digital Converter actuating lever with varying 0.1 V steps of the set point as labeled.](image)

The overhanging electrode area as seen in Figure 13(a) was imaged obtaining the height profile at different actuating frequencies. The data shows that different frequencies have a negligible effect on the distance traveled by the overhanging electrodes as seen in Figure 18 below. The green vertical dashed line represents the approximate location where the overhanging electrode ends. The maximum (800 kHz) and minimum (200 kHz) frequencies are shown to prevent clutter.
Figure 18: A cross sectional diagram of the height of the overhanging electrodes (base on the left, free end on the right) at different actuating frequencies with the green vertical dashed line representing the end of the overhanging electrode.

Amplitude data was also obtained and a cross section of the varying actuating frequencies was plotted as shown in Figure 19. Two different frequencies (200 and 600 kHz) display a large increase of amplitude on the overhanging electrode structure, but the 600 kHz frequency displayed a large change in the free end where the actuating lever strikes the overhanging electrode.
Figure 19: Amplitude cross section profile of the overhanging electrode structure at seven different actuating frequencies as labelled with a constant actuating voltage of 8 V.

The same experiment was done but with varying actuating voltages with a constant actuating frequency as seen in Figure 20. Amplitude shows a linear relationship with the lower voltages but increases significantly once we reach the higher end of the voltages allowed by the MEMS device.
Figure 20: Amplitude cross section profile of the overhanging electrode structure at four different actuating voltages as labelled with a constant actuating frequency of 200 kHz.

Finally, a T-shaped suspended lever as shown in Figure 13(a) was actuated with voltage pulses instead of an AC sinusoidal signal. In addition to this actuating lever, though, there is also a pair of MEMS structures approaching from the opposite side, and in fact overhanging each half of the top of the ‘T’ shape (Figure 20). Each is an independent electrode, such that the actuating MEMS device is designed to mechanically make or break electrical connections between the two contacts, by short circuiting them together from below if driven with sufficiently voltage [16]. The crossbar of the T-shape acts as the short circuiting element. AFM data was then acquired with the tip contacting one of these overhangs, allowing mapping of surface displacements in the slightly rotated area sketched at the right of Figure 13(a). The oblique view secondary electron SEM image of Figure 21(a), again with a rotated overlaid sketch of the AFM imaging region, clarifies the three-dimensionality of the lever and overhanging contacts. The tip scans from the end of the overhang (at left) to more than 10 μm away (at right).
Figure 21: SEM of T-shaped MEMS Structure designed to electrically contact the two parallel electrodes at right by electro-mechanically actuating from below (a); AFM topography of one of the overhanging electrodes in the region sketched by a rotated rectangle (b) with equal lateral scaling as the SEM image; and AC vibration amplitude detected at the surface of the overhang due to periodic electro-mechanical contact from below with a range of pulse widths (c).

By design, the height of the overhanging structure is not uniform. This is evident in the SEM image, as well as the topography determined by the AFM, Figure 21(b). Note that the lateral scales of these images (a-b) are matched for direct visual comparison. The simultaneously acquired amplitude on the overhang is shown in Figure 21(c), for pulse excitations ranging from 50 ns to
20 μs. These pulses repeated at a frequency of 25 kHz, with a magnitude (8 V) sufficient to cause the underlying T-shaped lever to strike the overhanging electrodes. The plot is presented with a log scale for the measured rms amplitude to more clearly reveal the various responses.

Directly on top of the electrode overhang, the rms amplitude of surface vibrations reaches as high as 15.7 nm, due to the repeated closing of the contact. Even for pulses of just 50 ns, though, measureable displacements are detected as far as 15 μm from the end of the overhanging structure, confirming coupling of the periodic mechanical contact many microns away from the contact point itself. Focusing on a single position of a similar overhanging electrode, Figure 22 plots the rms amplitude of surface displacements upon repeated contacts activated by voltage pulses with durations ranging over 3 orders of magnitude, from 20 ns to 20 μs. Taken alone, this result suggests a ~5 μs pulse threshold, below which the MEMS structure does not substantially strike the overhang and hence the electrical contact may not be reliably made. This is compatible with electrical measurements of the relay signals from equivalent devices. Interestingly, though, the mapped AC response of the overhang structure in Figure 21(c) shows that even 50 ns pulses generate a still-detectible surface displacement. In fact, reliable contact can be made in as fast as a few ns with more sophisticated MEMS designs [16]. Future measurements will couple the relay signal with direct maps of the overhang displacement to elucidate the threshold pulse amplitudes and durations for reliable electrical contacts, and their correlations to displacements of the overhanging contacts. The ability to image the in -situ response of such MEMS structures is therefore valuable for accurately investigating MEMS performance and reliability. It is reasonable to expect that the overall dynamic response of the MEMS structure will be sensitive to a combination of the strike rate as well as the impulse (applied force times the contact duration). The
repeat rate here is fixed at 25 kHz (every 40 μs) regardless of the pulse dimensions, such that the results of Figure 21(c) and Figure 22 are for a fixed number of strikes in a given time, independent of the pulse shape. Therefore, the cumulative pulse time (i.e. the contacting time, reasonably assuming a 1:1 correlation) is smaller for the narrower pulses, perhaps contributing to the apparent turn-on behavior in Figure 11. To normalize for this effect, similar measurements can of course be made that implement higher repeat rates for the shorter pulse durations. However, difficulties may arise since these different repeat rates can excite distinct modes or harmonics for the independent or coupled MEMS structures, including the AFM cantilever itself. As a result, the dynamics of the AFM lever itself could convolute the results, and therefore in this work a single frequency far from the AFM cantilever resonance is employed.

![Graph](image)

Figure 22: RMS amplitude of surface displacement for an overhanging contact struck by an underlying piezo-MEMS structure at a rate of 25 kHz, as a function of pulse duration.

### 3.2.2 Temporal Response of RF MEMS

For this section (alone), discussions or figures of the ‘RF Output’ refer to the signal read by the output electrode using one of the lock in amplifiers described in Chapter 2 of this thesis. This RF
output signal is not obtained using the AFM tip itself, though it is recorded during AFM ‘imaging’ to essentially map the conductivity of the RF signal pathway as a function of a range of experimental parameters. This and related AFM signals could equivalently be recorded with a data acquisition card or an oscilloscope since the surface is not physically scanned. But, when data is acquired through ‘imaging’ as performed in this section, the concepts are easily extended for future work to investigate any spatial variations in the RF or corresponding AFM signal.

Figure 23 shows an example of this, displaying the amplitude of the output signal of the RF MEMS device with a constant actuating voltage of the RF switch, but varying frequencies as a function of the imaged line. Lower actuating frequencies (near the base of the figure) display wider stripes, while higher actuating frequencies display narrow stripe patterns as a function of the cycles per second of the actuating frequency and the AFM scan rate. Near 0.7 um in the y direction and up to ~-.95 um, the actuating frequency is higher than the sampling rate, such that even though the RF signal is still being switched, a meaningful image of this switching is not possible. The magnitude of the RF signal for the on and off states can be seen to shift with frequency as well (bright band near 0.95 um), related to the RF circuit impedance which has a frequency dependence as well. But again, the image size and positions referred to above are artificial (with a dummy size of 1 um on a side), as the AFM is simply being used to record signals as a function of time, not a spatially varying response.
Figure 23: Amplitude of the RF output signal at varying actuating frequencies with a constant actuating voltage.

Figure 24 is a montage of six images of the amplitude of the RF output signal with varying actuating voltages and a constant frequency with a 25% pulse width, meaning the actuating switch lever is only making contact with the contact electrodes to close the rf circuit for one quarter of the time in any given actuation cycle. Subtle periodic noise is also superimposed, particularly apparent in the image with the lowest piezomems actuation voltage of 1 V. Focusing instead on the 1:4 periodicity of the real switched RF signal (dark and light bands), it is clear that the higher the actuating voltage, the stronger the modulation of the RF signal.
Figure 24: Amplitude of RF Output signal with varying actuating voltages as labelled and a constant frequency with a 25% pulse width.

Figure 25 shows height and deflection images, which are acquired with the AFM tip contacting the surface, in the top two rows. The amplitude of the RF output signal is presented in the bottom row of the figure. At 0 V (left column), when the RF MEMS switch is ‘off,’ an essentially uniform background noise signal is observed in all three of the channels (rows) as expected for no actuation of the MEMS RF switch. With an intermediate actuating voltage of 5 V (center column), the change in amplitude of the RF output signal is pronounced, while features are only barely discernible in the height (nanometer range) and deflection channels. This suggests that the MEMS RF switch requires very little actual force between the actuating microlevers and the overhanging electrodes to latch the RF signal into the output electrode.
Figure 25: Montage of 9 images of Height (Ht), Deflection (Df), and RF Output signal amplitude (Amp) with varying actuating voltages of the underlying microlever as labelled (note the contrast ranges are necessarily increased from left to right to identify the effects of the stronger actuation conditions).

For the maximum actuating bias used in this experiment (10 V), the height and deflection channels reveal clear contrast for each strike from the lever below into the physical overhang on which the AFM is resting and recording the response, with a corresponding stronger change in the RF response (note the much wider signal range in the contrast scales for the 3rd column). The AFM tip is recording a height change (bounce from below) in the picometer range for an actuating
voltage of 5 V, but detects jumps in the range of nanometers for 10 V of actuation. This is more clearly observed in the cross sectional analysis of the height image, Figure 26.

Figure 26: AFM image of height (left) with a horizontal profile cross section (right) along the sketched line for two 25% duty cycle pulses of the RF MEMS switch.

The equivalent cross sectional data (same image line), but for the deflection channel (essentially the instantaneous force experienced by the AFM probe), is shown in Figure 27. A magnified portion of the signal as sketched by the box. The asymmetry of the features as compared to the height signal actually indicates that this image was acquired in the ‘retrace’ pass of the AFM tip (while moving from right to left). This is because the deflection image reveals an increase at the right side of each stripe (bright band), i.e. the instant the lever is actuated but before the AFM feedback has equilibrated to the new surface position. Next, scanning to the left, the deflection (force) gradually returns to the initial set point position, as the tip height is adjusted by the AFM z-piezo according to the constant-force feedback-circuit. The opposite occurs when the microlever bias returns to zero, unloading the overhanging structure and thus lowering it, causing an instantaneous decrease in the deflection image on the left side of each actuating strip (dark bands).
This proves a physical rise and fall of the overhang when the actuating lever strikes, contacts, and then releases from the contacts, as strong as several nanometers for 10 V actuating biases.

Figure 27: Deflection image (left) corresponding to Figure 26 of RF MEMS device with a profile cross section (right) of the same horizontal section as in the previous figure, confirming an actual change in height during contact between the actuating lever and the overhanging electrodes on which the AFM probe is resting.

Finally, the previous data was obtained with low switching frequencies to be able to correlate the forces on actuating levers and overhanging contacts with the RF signal itself. In reality, though, RF MEMS will be switched at higher frequencies. Accordingly, Figure 28 displays the RF response with an actuating frequency of 5 kHz. Again the AFM scan size is essentially artificial as the AFM is simply being used to record the RF response as a function of time. Unlike the
previous images where the sampling rate was approximately 512 points per second, though, in this case ~40,960 points were acquired per second to try to obtain as much accurate data as possible. This was achieved by setting the AFM scan rate close to 10 Hz (10 traces and retraces per second), with 2048 points per line in each direction.

**Figure 28:** Amplitude image of the RF output signal switched at 5 kHz (upper left) but recorded at ~41 kHz, with a complete cross sectional profile (below) of an arbitrary section of the amplitude data as well as a magnified portion from this line (upper right).

The Moire pattern apparent in the 2d image of this data is due to the lack of synchronization between the amount and rate of pixels acquired in the raw data, the periodic signals they represent, and the pixel resolution of the image. The cross sectional profile graph at the base of the figure...
makes this more clear, where there is a slight sin-like wave pattern superimposed on the rapidly switched RF output signal. The cross section at above right is magnified from the full profile. Counting the number of imaged peaks and valleys (RF on/off cycles) and relating this to the acquisition time, per line, confirms that the AFM is gathering real and accurate data about the in situ operation of the RF MEMS device. Unfortunately simultaneous AFM height or deflection images, as with prior figures in this section, are not possible at such high data acquisition rates due to data-throughput limitations in the instrument on available due to the necessity of the AFM scan rate being high and such scan speed would do irreparable damage to the sample.

To conclude this section, AFM is demonstrated to be able to accurately record the RF signal response as a function of time with up to ~40 kHz acquisition rates, and to be able to relate the RF signal with forces or changes in height caused by the RF MEMS switches themselves as they physically latch overhanging electrodes with an underlying microactuating lever. This is valuable and unique insight available only through such in situ measurements, with one particularly important contribution to be made in terms of establishing threshold actuating voltages necessary to reliably switch the devices. Specifically, for the devices tested, moderate voltages on the order of 5 V are sufficient for achieving RF switching functionality with hardly discernible strains imposed on the overhang structure (sub pm). 10 V signals, on the other hand, cause nanometers of strain for the overhang, a likely source of eventual mechanical failure and thus a possible reliability concern that can now be avoided since it has been directly observed.
3.3 In Situ Imaging of Tuning Fork

The final MEMS structure considered in this work with in situ AFM measurements is a tuning fork, with the description from our recently published paper on the topic [83]. As introduced in Chapter 1, this is a practical MEMS device available commercially and manufactured on the scale of millions per year for pennies apiece. This serves as an excellent control for relating the nanoscale and milli- to micro- second resolved data described above, with real world applications.

Figure 29(a) presents common frequency responses of such a macroscopic tuning fork, measured by analyzing the AC bias on a voltage divider across the tuning fork with a lock-in-amplifier (arbitrary units are shown) during frequency sweeps across 3 kHz in 25 Hz steps (i.e. 120 points per line) in air. For each frequency sweep past the primary resonance at approximately 55.6 kHz, 7 distinct driving amplitudes are employed from 10 to 730 mV (peak to peak). The results for each condition are offset by 40 a.u. for visual clarity in the figure, with the strongest driving voltage offset by 240 a.u. and the weakest barely rising above 0. The mean peak amplitude is displayed for each of these conditions in Figure 31 (b), with error bars representing the peak-to-peak standard deviation based on 5 measurements for each driving amplitude to establish repeatability. As expected, these results indicate a nearly perfectly linear correlation between the driving amplitude and the maximum response of the tuning fork ($R^2=0.9985$).
Figure 29: Tuning fork response (arbitrary units) as measured by a voltage divider while sweeping the driving frequency past the 55.56 kHz primary resonant frequency multiple times for 7 distinct AC amplitudes (a), revealing a linear maximum response with driving amplitude (b) and ± standard deviation error bars of 1-5%. Note that each set of frequency sweeps at distinct driving amplitudes is offset by 40 a.u in (a) for clarity.

Mounting the tuning fork from Figure 29 in an AFM and contacting one of the tines as sketched in Figure 30(a), the amplitude is directly measured by monitoring deflection of the high frequency AFM probe and again analyzing the signal with the lock-in-amplifier. For a fixed driving amplitude of the tuning fork, 15 consecutive frequency sweeps were made, Figure 30(b), again each offset for visual clarity (by 200 a.u.).

The sweeps are visually indistinguishable except for subtle shifts in the peak frequency, which amount to a maximum of 5 frequency steps of 10 Hz, attributed to thermal effects for these measurements made in ambient conditions. Importantly, these 15 measurements are actually 5 groups of 3, each acquired with a distinct AFM contact force applied (i.e. set point) ranging from 39 to 1540 nN. Figure 2(c) displays the average maximum amplitude for the peaks as a function
of this set point, along with the essentially negligible standard deviation. These results indicate a nearly constant electromechanical response of the TF except for the highest AFM load (1.5 μN). The units here have been converted to actual root mean square (rms) nanometer amplitudes via a conversion factor of 48.1 nm/V as described in the methods section. The results yield an effectively flat response for the resonant frequency as well.

![Figure 30: Sketch of in-situ AFM and tuning fork structure (a), AFM-detected amplitude (shown in raw arbitrary units) during 15 frequency sweeps driving the resonance of the same tuning fork as in Figure 1, and the resulting peak amplitude (calibrated to nm of rms amplitude, with standard deviation error bars) as a function of the set point force.](image)

Of course at some high load the AFM tip may cause the tuning fork to behave like a damped harmonic oscillator. While this threshold could be near 1 μN, a barely apparent maximum in Figure 2(c), we have not explored this possibility since higher loads are not reliable with the AFM probes.
and system used in these experiments. In fact, the 1 μN condition required 5 V of set point deflection for this lever and alignment with the AFM, while the slightly diminished response at a 1.5 μN load necessitated an 8 V set point, where nonlinearities of the photodetector may somewhat mask the true behavior. Therefore, loads much less than 1 μN are otherwise used throughout the remainder of this work (typically ~200 nN), where the contacting AFM can reasonably be assumed not to perturb the simple harmonic oscillator behavior of the tuning fork. In fact, the experiments could be performed faster by sampling fewer frequencies and fitting the results to a simple harmonic oscillator response, but instead the pure data, maximum signals, and frequencies for these maxima are considered throughout.

Figure 31 presents a montage of 18 corresponding AFM images of the tuning fork, each 5x80 μm (1:16 aspect ratio), all acquired in the same location where the left edge is aligned with the free end of the imaged tine (4 mm from its base and therefore where the normal amplitude should be a maximum). The displayed contrast does not present height as is common with AFM, since it is essentially identical for these multiple images of a single area. Instead, the distinct images map the amplitude of the tine for a range of specific driving frequencies. Specifically, the images sequentially step through the primary resonant peak of the tuning fork, from 55.56 to 55.73 kHz in steps of 10 Hz. The overall image contrast clearly is brighter (stronger amplitude) near 55.63 kHz, revealing the vibrational resonant peak. While the amplitude should also be strongest at the ends of the tines and hence the left edge of each of the images, this is difficult to discern from the amplitude contrast alone.
Figure 31: 18 images of the tuning fork amplitude as a function of frequency for a 5x80 μm area near the end of a 4 mm tine (just beyond the left edge).
To better visualize the overall amplitude and frequency response of the tuning fork tine, a two-dimensional histogram of the entire dataset in Figure 31 is shown in Figure 32. In this representation, the histogram count is displayed as color contrast. The histogram bins are established for the 18 discrete driving frequencies (i.e. images) with steps of 10 Hz as indicated in the x-axis of the figure, and for a range of possible vibration amplitudes as identified by the y-axis based on steps of 2 pm rms. At each frequency, the detected amplitudes are tightly grouped, indicating a relatively uniform response in the imaged region. Optical interferometry measurements of the same structure, with its common μm scale or worse lateral resolution, could resolve similar average signals. However, interferometry cannot differentiate the nanoscale responses evident in Figure 31.

Figure 32: Two-dimensional histogram of the number of image pixels (i.e. relative area of the imaged region of the tuning fork, color contrast) for defined amplitudes (y-axis) and frequencies which in this case means the 18 discrete images (x-axis).
Using such high spatial resolution, multiparameteric data, maps of a range of properties such as the local maximum amplitude ideally can be determined down to the resolved pixel size in the initial AFM images. This is achieved by: i) simply “drilling” through the stack of distinct images (i.e. Figure 31) for any given image pixel as described elsewhere 64; ii) calculating and recording the image frame at which the contrast is strongest (e.g. the frequency corresponding to the peak amplitude), or reaches some other indicative magnitude (e.g. where a phase signal reaches 90°, current reaches 0 nA, etc.); and iii) repeating this process for every pixel in the imaged region. In this case, the resulting images of the maximum amplitude, frequency for the maximum amplitude, etc. can then be easily compiled. The spatial resolution of such maps depends on two parameters, any uncorrectable spatial drift during the experiment (linear or measured drift can easily be accommodated), and of course the initial information density (pixel size). The pixel density for the images of Figure 33 are relatively modest (~312 nm on a side simply due to a low acquired pixel density), but practically can extend down to the limit of the AFM tip contact area (~5 nm for similar condition contact-mode AFM images and tips). This general concept has previously been employed for efficient, high resolution mapping of conductivity 64, photoconduction 65, stiffness 66, and friction coefficients 67.

Of course both the resonant frequency and amplitude for the millimeter scale tuning fork are expected to be relatively uniform when imaged on the μm scale with AFM. Indeed, calculated images of the maximum amplitude and frequency for this maxima (i.e. resonance) simply correspond to the contrast in the single image of Figure 31 with the strongest signal (i.e. at 55.62 kHz). Therefore, for this millimeter sized feature, an image of the average vibrational response of the tine as a function of the distance from its end is both more revealing, and more appropriate given the spatial scales of the measurement. Specifically: i) the 18 images of a single area from
Figure 31 are each normalized to the maximum amplitude at that frequency (which in fact occurs at the left edge of each image, i.e. nearest the end of the tine); ii) drilling through this data pixel by pixel as described before, the average normalized amplitude with respect to the end of the tine is calculated; iii) this is repeated for every image pixel to generate the average amplitude response for the last 80 μm of the tine, Figure 33(a). A cross section of this entire result, Figure 33(b), now reveals the general decrease in amplitude from the tine end toward the base that was expected but visually unapparent in the raw data of Figure 31. Specifically, the measured change in amplitude over the 80 μm imaged length is ~1.5%, which compares favorably with the fraction of the overall 4mm tine length that is directly investigated, i.e. the last 2% of the tine.

It is important to note that topography induced artifacts can superimpose on such results. In this case, poorly bound particles on the surface yield apparently vertical stripes of diminished amplitude in Figure 33(b), due to poor tip-particle-tine coupling. Here, inconveniently this is coincidentally convoluted with several μm of thermal drift in the y-direction. This can be accommodated in future measurements with feature tracking, or employing high speed AFM to minimize thermal drift. Regardless, though, these model results validate the applicability of AFM for in-situ measurements of electromechanical systems.

The maximum measured amplitude of approximately 11 nm (rms) for the tines further confirms the applicability of such tuning forks for investigating curing effects of nano-fibrous gels, since the vibration magnitude is on the same order as the gel porosity. Future investigations will focus on the tuning fork response to gel curing time for a much larger range of vibrational amplitudes, which should provide further insight into the mechanical properties of the mesoscopic interconnected network of fibers.
Figure 33: Image of average normalized amplitude (a) and cross section of this result (b) from Figure 31 revealing a 1.5% decrease in the normalized vibrational amplitude over the last 2% of the tine length.

Returning the measurement configuration to a voltage divider circuit instead of AFM detection of the tine amplitude, the tuning fork response was next measured in liquid, Figure 34. Specifically, the tines were submerged into a mixture of cell culture media and buffer solution, with and without the addition of a nanofibrous collagen that typically gels in approximately 30 minutes. The peaks between 52.8 and 53 kHz, acquired every 10 minutes as noted, display a subset of the more than 20 regularly repeated frequency sweeps during curing of the nanofibrous collagen gel. The amplitude, and the frequency, clearly rise smoothly with curing time. For comparison, an identical control experiment was also performed with the cell culture media and buffer solution alone, i.e. without the collagen and hence a gel will not form. The peaks for this no-collagen case at ~52.5 kHz (labelled as ‘NC’ in the plot) are stable over the same duration, supporting use of such tuning forks to monitor curing of gels.
Tuning fork spectra similar to those in Figure 34 were in fact acquired every 2 minutes. Analyzing the maximum amplitudes and frequencies leads to Figure 35. Beyond 10 minutes, an increase in the tuning fork resonant amplitude (a) and similarly the resonance frequency (b) is observed, implying an increase in viscosity as expected during gelling. In the first 10 minutes, on the other hand, the response shows certain frequency drift, possibly suggesting the detection of distinct reaction stages, but most likely related to the temperature evolution during the early stages of the measurement. While endothermic or exothermic reactions can be detected via the temperature sensitivity of the tuning forks 62, it is more likely that the first 4 data points are indicating nonlinearities during thermal equilibration of the system overall because even though the measurements are performed in a thermally isolated chamber, the lid is open at time zero in order to add the Rat Tail Collagen HC and initiate the reaction. This possibility is supported by the identically acquired tuning fork response for the control conditions, i.e. without the collagen gel,
which is also plotted for an equivalent overall duration. The resonant frequency remains essentially constant as anticipated, with a less substantial but still apparent drift in the first few measurement points (~5 minutes), therefore attributed to thermal equilibration effects.

Figure 35: Resonant amplitude (a) and frequency (b) for a tuning fork as a function of time when submerged in a liquid of cell culture media, PBS, NaOH, and collagen during curing (‘with collagen’) as compared to a stable control measurement without the curing collagen gel (‘no collagen’).

The gel is expected to have fibers and pores on the ~10 nm scale, such that it is important to know the amplitude of the tines in order to relate the magnitude of the mechanical perturbations they cause to the surrounding effective microstructure. Accordingly, the same tuning fork was mounted in an AFM in air for direct measurements of the resonant amplitude normal to the tuning fork axis,
i.e. the direction of maximum amplitude. Rotating the horizontally mounted tuning fork by 90 degrees would allow shear of the tuning fork tines to be detected, or even leveraged as a fast scanner as in some high speed AFM systems [84].

Chapter 4 Conclusions and Recommendations for Future Work

As the wide use of MEMS and NEMS structures continues to increase in real-world devices, the development and application of in-situ techniques capable of measuring and mapping their static and dynamic functionality are similarly important. Atomic Force Microscopy is particularly well suited for such investigations, providing the topography of MEMS as well as the local stiffness, actuation, resonance, or other parameters with nanoscale spatial resolution. This is directly demonstrated with model measurements of tuning fork resonances, as well as maps of the performance of piezoelectric MEMS cantilevers and RF signal switches. Challenges include decoupling any results from the dynamics of the AFM cantilever itself, as well as the persistent issue of acquisition speed with nearly all AFM systems. Even so, AFM can obtain valuable and novel insight into the performance of MEMS devices at the nanoscale, enhancing future efforts in MEMS modelling, design, functionality, and reliability.

The project inspires several ideas for future work. Future work involves the author traveling to the U.S. Army Research Laboratory in Adelphi, MD. The location where the MEMS structures used in this thesis are fabricated. There, he intends to include the use of an AFM system to perform the measurements shown in this thesis to advance with the characterization and modeling of these and future devices. During this time, different top electrode material will be investigated to understand its effects on the mechanical and electrical properties of the devices.
References


