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INVESTIGATING MEMS DEVICES IN FLOW CONDITIONS RELEVANT TO FLOW-THROUGH SYSTEMS

By

Mohammad Shafquatul Islam B.Sc., American Intl. Univ. – Bangladesh, 2009 M.Sc., University of Leeds, 2012

A Dissertation Submitted to the Faculty of the J.B. Speed School of Engineering of the University of Louisville In Partial Fulfillment of the Requirements for the Degree of

Doctor of Philosophy in Electrical Engineering

Department of Electrical and Computer Engineering University of Louisville Louisville, Kentucky

December 2023

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A Dissertation Approved on

November 20, 2023

by the following Dissertation Committee:

Cindy K. Harnett, PhD Dissertation Chair

Mark Running, PhD

John F. Naber, PhD

Shamus McNamara, PhD

DEDICATION

To my family, friends, and future.

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I would like to express my deepest gratitude to Professor Cindy Harnett, my advisor, for her invaluable guidance, unwavering support, and remarkable patience throughout the years. My journey to this point would not have been possible without her. I would also like to thank the rest of my dissertation committee members, Prof. Mark Running, Prof. John Naber, and Prof. Shamus McNamara, for their helpful discussions and invaluable feedback in shaping and finalizing this thesis.

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ABSTRACT

INVESTIGATING MEMS DEVICES IN FLOW CONDITIONS RELEVANT TO FLOW-THROUGH SYSTEMS

Mohammad Shafquatul Islam

November 20, 2023

Advancements in microscale actuating technologies has substantially expanded the possibilities of interacting with the surrounding environment. Microstructures that deflect in response to mechanical forces are one of the largest application areas of microelectromechanical systems (MEMS). MEMS devices, functioning as sensors, actuators, and support structures, find applications in inertial sensors, pressure sensors, chemical sensors, and robotics, among others. Driven by the critical role of catalytic membrane reactors, this dissertation aims to evaluate enzyme activity on polymeric membranes and explore how fabrication methods from the field of Electrical and Computer Engineering (ECE) can incorporate sensing and actuation into these porous surfaces. Toward better understanding of conditions in flowing systems, this dissertation investigates how MEMS devices perform in flows, demonstrating a set of thin-film out of plane cantilevers that deflect in the flow velocity range of 0.5 to 5.7 mm/s in high viscosity solution (glycerol). We show with the same processing methods, MEMS devices can be

developed for actuation over angles 0 to 90 degrees at speeds in the millisecond range and for resistive temperature sensing (temperature range 20 to 500 °C). Finally, this dissertation presents an innovative packaging approach that employs mechanical tangling, allowing the integration of MEMS microgrippers with fibrous materials commonly used in wearables, soft robotics, and applications requiring large deformation.

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CHAPTER I

INTRODUCTION

The scope of this work presented the development and optimization of a flowthrough spectroscopy device employing a laccase enzyme-coated membrane and a colorimetric laccase activity indicator. This cm-scale microreactor enables the measurement of reaction rate and percent conversion as functions of flow rate and membrane microstructure. The integration of *Trametes versicolor* laccase enzyme onto screen-printed electrode (SPE) surfaces, as confirmed by voltammetric analysis, demonstrated a more than twofold increase in current compared to enzyme-free electrodes, establishing a signature of active enzyme presence. The microreactor, subjected to fluid flow, demonstrated a tradeoff between achieving a high flow rate and maintaining a short dwell time in the active region. This unique enzyme-coated membrane system holds potential applications in sorting biological products for their productivity in polymeric membrane bioreactors.

Toward better understanding of conditions in flowing systems, this dissertation investigates how MEMS devices perform in flows. These inorganic devices could potentially sense conditions inside membrane reactors and other flow-through systems. In this thesis, we investigate their compatibility and develop integration strategies. By combining insights from fluid dynamics, materials science, and MEMS technology, we aim to gain a clear understanding of their behavior in dynamic environments. Conventional sensing methods used by MEMS flow sensors are thermal, drag force, differential pressure, piezoresistivity, hot-wire anemometry, and optical detection. However, most of these structures are fabricated in the plane of the flow. We introduce a flow sensitive mechanism based on thin-film metal-oxide out-of-plane microcantilevers with optical deflection read-out. Mechanical characterization within a flow-through system revealed their robustness at varying flow rates, exhibiting deflection without damage. The advantage of this mechanism comes from ease of setup in fluidic channels, and many points of measurement due to ease of fabrication in arrays.

The developed flow-based mechanical testing platform caters to arrays of out-ofplane deflecting structures. Unlike conventional probe-based force testing, which is burdened by challenges such as high equipment costs, stress concentration issues stemming from point contact, and the necessity for skilled operators to ensure precise cantilever landing, the flow-based mechanical testing method emerges as a superior alternative. This approach enables the concurrent assessment of multiple devices, streamlining contact initiation without requiring time-consuming alignment. Moreover, it facilitates the evaluation of devices in orientations closely resembling their real-world applications. This approach provides a more practical and efficient means for characterizing lateral deflection devices compared to conventional probe-based testing methods.

In the realm of Micro Electromechanical Systems (MEMS), miniscule devices work as sensors, actuators, and passive 3D support structures. Their functions include inertial sensors like accelerometers and gyroscopes, pressure sensors, displacement and strain sensors, radio frequency switches and antennas, resonators, ink-jet nozzles, chemical sensors, mass sensors, robotics, and an array of other applications [1,2]. In the next section of the thesis, we went beyond flow-based deflection to investigate their performance as resistive temperature sensors and actuators, and developed methods to move the devices off the silicon substrate and onto fibrous materials commonly used in wearables, soft robotics, and applications requiring high deformation. Despite the tremendous potential of MEMS technology, a significant drawback arises from their conventional fabrication on rigid silicon substrates, limiting flexibility and adaptability when affixed to soft, stretchable, and porous materials. Microgrippers are created using the strain architecture technique and more importantly, their integration with soft surfaces does not compromise the MEMS fabrication process or the thermal functionality of these devices. The demonstrated reversible clasping mechanism could serve as a programmable method for selectively transferring MEMS devices to fibrous substrates, providing versatility in applications with high deformation requirements.

CHAPTER II

MINIATURIZED SYSTEM FOR EVALUATING ENZYME ACTIVITY

Motivated by the importance of catalytic membrane reactors, in this chapter we propose to characterize enzyme activity on membranes and investigate how fabrication methods from ECE can bring sensing and actuation to these porous surfaces. This chapter describes a demonstration cm-scale reactor. Its purpose in the larger thesis is to provide a motivating example for the sensing parameters of interest, and specify the environmental conditions of temperature, flow rate, and viscosity that an embedded MEMS device must withstand for process control in a model microreactor system. Polymeric membranes coated with enzymes prove to be versatile catalysts, facilitating biofuel production and other chemical processes utilizing feedstock like plant biomass. Such bioreactors are more energy efficient than high temperature methods because enzymes catalyze chemical reactions near room temperature.

A significant challenge in processing plant biomass is the presence of lignin, a complex aromatic polymer resistant to chemical breakdown. Therefore, membranes coated with enzymes such as laccase that can degrade lignin are sought for energy extraction systems. The experimental study presented here is dedicated to optimizing an enzyme-based membrane bioreactor, investigating the tradeoff between high flow rates and short dwell times in the active region.

2.1 Example Application: Breaking Down Plant Biomass at a Catalytic Membrane

The major challenge associated with processing plant biomass is the presence of lignin, a hydrophobic and heterogeneous biopolymer that resists chemical and biological degradation. It is the second most abundant naturally synthesized compound after cellulose comprising 15-40% of dry weight in most plants [3]. It is a complex aromatic polymer rich in phenolic compounds; enzyme-based reactors that can oxidize these phenols and depolymerize lignin could provide a new route to sustainable biofuels and aromatic fine chemicals beyond conventional refineries [4]. Research groups are looking at laccase enzymes for breaking down organic materials [5]; these enzymes can adhere to charged membrane surfaces and can also be engineered with binding sites such as his-tag sequences to attach to metal-ion coated synthetic membranes. Membrane-immobilized enzymes that can break down lignin from woody and non-woody plants are sought after for compact and room-temperature energy extraction systems. In this work, a flow-through device was developed with a goal of evaluating genetically engineered enzymes' activity in a microreactor environment using a membrane-based lab-on-chip sensor system. The smallscale screening system is designed to test sub-100 microgram quantities of enzymes in a membrane reactor format so the most active variants can be identified for scale-up.

Laccase, an environmentally friendly and functionally diverse enzyme, is well known for its lignolytic activity. It is produced by a variety of fungi, bacteria, and plants [6,7] and serves as an efficient catalyst for bioremediation, due to its ability to catalyze the oxidation of a variety of substrates, such as phenolic compounds, metal ions, and aromatic amines [8]. On account of its relatively low redox potential (450-800 mV) laccase cannot directly catalyze the oxidation of most nonphenolic substrates (e.g., 80% of lignin). But its substrate scope can be widened to nonphenolic compounds once combined with low molecular weight mediators, which simultaneously act as substrates for the enzyme [9–11]. As laccase's range of substrates have broadened in the last couple of decades, so has its influence in bioremediation applications, wood pulping, paper, and textile industry, municipal sewage, electrochemical analysis, and organic synthesis applications [12–19].



Figure 1. Active laccase enzyme from *Trametes versicolor* complexed with 2,5-xylidine [20].

The range of compounds identified over the decades as mediators for the laccasemediator system has increased dramatically. Currently, 2,2'-azino-bis(3ethylbenzthiazoline-6-sulfonic acid) (ABTS) and m-cresol are regarded as the best mediators for laccase [21–23]. ABTS has gained significant popularity as a laccase mediator in various biochemical and environmental applications. This synthetic compound plays a crucial role in enhancing the enzymatic activity of laccases, which are coppercontaining enzymes found in many fungi and plants. It serves as an efficient electron mediator, facilitating the transfer of electrons between the enzyme and the substrate. This electron transfer process is indispensable for laccase-driven reactions, where these enzymes catalyze the oxidation of a diverse array of substrates. In the realm of environmental science, ABTS-assisted laccase reactions play a vital role in the degradation of pollutants. Laccases have gained recognition for their remarkable ability to break down stubborn organic compounds, and ABTS enhances their efficiency when applied to tasks like wastewater treatment and environmental remediation. Within these critical applications, the attributes of ABTS, notably its stability and efficiency, take center stage. These qualities are of paramount significance, ensuring the rapid and effective breakdown of contaminants, thereby addressing the pressing need for environmental remediation and preservation.

For this research, *Trametes versicolor* laccase was employed due to its promising activity in bioremediation presented in [24,25]. *Trametes versicolor* is also known as *Coriolus versicolor* which is a white rot fungi. The developed enzyme screening system seeks to work with sub-milligram laccase quantities produced in early-stage genetic engineering research. Voltammetric analysis performed in the presence of m-cresol isomer determined the enzyme activity without flow. A flow-through spectroscopy device with enzyme-coated membranes and ABTS, which is a well-characterized colorimetric indicator for spectroscopy, will be designed and employed to measure the percentage of conversion as a function of flow rate and membrane microstructure.

2.2 Evaluating Commercially Available Membranes as Supports for Laccase

Several polymer membranes were evaluated for absorbance measurement experiments. Supercharged Nylon filters (Whatman Nytran SPC, 0.45 µm pore size) were obtained from Tisch Scientific; this treated nylon has a high positive charge per area for picking up protein in blotting assays. Thicker than other membranes in Table 1, the Nytran membrane can also lie flat without curling, for easier coating and assembly into flow-through systems.

Table 1

Survey of commercially available polymer membranes for flow-through enzyme reactor system

Membrane	Pore Size (µm)	Manufacturer	Nominal Thickness (μm)	Binding Capacity (µg/cm²)
Polyethersulfone	1.2	Sterlitech Co.	110 - 150	20
Polycarbonate	0.2	Sterlitech Co.	10	5
Polyester	0.2	Sterlitech Co.	10	< 5
Polyvinylidene difluoride (PVDF)	0.2	Sterlitech Co.	125	4
Nylon	0.2	Sterlitech Co.	65 - 130	120
Nytran SPC Nylon	0.45	Tisch Scientific	140 - 170	600

2.3 Electrochemical Detection of Immobilized Enzyme Activity

Voltammetric measurements will be conducted using three-electrode configured screen-printed electrodes (SPE) from Dropsens Inc. These disposable electrodes are constructed on a flexible plastic substrate consisting of a PEDOT working electrode, a carbon counter electrode, and a silver reference electrode.



Figure 2. (a) Typical experimental setup showing the reference, counter, and working electrodes; (b) setup employed for in-lab experiments with SPEs; (c) Metrohm Autolab N series potentiostat; (d) design of a flow-through environment using a membrane-based lab-on-chip sensor system.

Cyclic voltammetry provides direct information about the redox properties of laccase by monitoring currents into and out of electrode-bound enzyme coatings. Analysis

of the resulting electrochemically active products is another source of data about enzyme activity. Such studies could potentially evaluate the quality and lifespan of laccase-based enzyme biosensors and bioreactors. To demonstrate the feasibility of the proposed approach in a small-volume format, cyclic voltammetry analysis of m-cresol will be performed at bare and laccase-modified screen-printed electrodes (SPE) as illustrated in Figure 2. Although m-cresol is a small molecule, its structure resembles lignin's phenolic sub-units, making it a convenient substrate for modeling lignin breakdown.

Voltammetric measurements will be carried out using Metrohm Autolab N series potentiostat/galvanostat models 128N and 302N, running the latest version of NOVA software. The SPEs need to be soaked in laccase solution (*Trametes versicolor* laccase, Sigma-Aldrich, 3 mg/mL in pH 7 0.5 M potassium phosphate buffer with 100 mM KCl) overnight and dried for 4-5 hours afterwards to develop the coating. The analysis will be carried out with a m-cresol concentration of 5 x 10⁻⁴ mol/L in a 0.01 mol/L non-deaerated acetate buffer (pH 5.0) as the supporting electrolyte. The recipe for acetate buffer (0.01 M 500 ml) consists of sodium acetate solution (0.01 M 352.5 ml), acetic acid solution (0.01 M 147.5 ml), and 27 mg of m-cresol. The potential range for the cyclic voltammetry was set from -0.4 to +1.0 V, while scan rates of 5 to 20 mV/s will be used.

2.4 Results from Electrochemical Measurement of Laccase Activity

The electrochemical properties of cresol at bare SPEs and modified SPEs with *Trametes versicolor* laccase enzyme (Laccase/SPE) were investigated using cyclic voltammetry over a range of scan rates. The m-cresol isomer provided two oxidation peaks

at bare SPE in the acetate buffer solution at pH 5.0. The highest oxidation peaks observed were 13.91 μ A and 15.75 μ A, while the peak potentials of the oxidation peaks were recorded at 0.606 V and 0.95 V, respectively. On the other hand, highest oxidation peak height detected for the laccase-modified SPEs were 31.45 μ A and 25 μ A at applied potentials of 0.67 V and 0.74 V, respectively. It is evident from Figure 3(a) that the oxidation peaks are higher when the SPEs are exposed to the active laccase enzyme, which supports the development of membrane-based lab-on-chip biosensors using enzymes such as *Trametes versicolor* laccase. The magnitude of peak currents increased with increasing scan rates as shown in Figure 3(b).



Figure 3. (a) Cyclic voltammetry of 5 x 10⁻⁴ mol/L m-cresol at bare SPE and Laccase/SPE performed at 10 mV/s scan rate; (b) bare SPE voltammetry performed at different scan rates in 0.01 mol/L non-deaerated acetate buffer.

For slow voltage scan rates, the diffusion layer will grow farther from electrode compared to faster scan rates, which produce a relatively thinner diffusion layer [26]. Because of the shorter diffusion length, reactant flux to the electrode is higher at fast scan rates compared to slow scan rates. Since current is proportional to flux towards the electrode, faster scan rates increase the current produced for the voltage applied. For quasi-reversible or irreversible cases like this instance, the peak potentials increase with the applied scan rate. Faster scan rates will encourage greater electrochemical irreversibility [26].

Cyclic voltammograms presented in Figure 3 indicate the presence of active laccase enzyme on SPE electrodes with an oxidation peak that more than doubles in current compared to enzyme-free electrodes. This signature of an active enzyme could be used to sort biological products for their potential productivity in synthetic membrane bioreactors.

2.5 Immobilizing Enzymes on Membranes for Colorimetric Detection of Laccase Activity

Nytran SPC membrane sheets were laser-cut into 1 cm diameter discs for laccase coating. The laccase stock solution recipe consisted of the following: potassium chloride (200 mM 50 ml), potassium phosphate monobasic (1 M 19.25 ml), potassium phosphate dibasic (1 M 30.75 ml), and 300 mg laccase (*Trametes versicolor*) powder; for 100 ml of an 0.5 mM pH 7 solution. The laccase buffer solution was divided into 1 ml centrifuge tubes and flash-frozen within a day to keep the protein fresh; tubes were thawed one at a time for experiments. Nytran membranes were then functionalized with laccase enzyme

via a drop casting method; each membrane disc was exposed to $20 \ \mu$ l drops of the laccase solution from a pipette. The membranes were left to dry for 5-6 hours.



Figure 4. SEM images of the Nytran SPC membrane – (a) plain membrane; (b) with immobilized laccase.

The enzyme immobilization was further confirmed by colorimetric analysis of ABTS solution exposed to laccase-coated membrane discs by means of dipping followed by scanning electron microscopic (SEM) inspection. Comparison of the as-received Nytran SPC membrane and the same membrane after coating with laccase is shown in Figure 4. Scanning electron micrographs revealed surface films that were not present on as-received membranes, which are later washed away when the membrane placed inside the microreactor is rinsed with DIUF (de-ionized ultra-filtered) water.

2.6 Design and Fabrication of a Microstructured Membrane Bioreactor for Flowthrough Experiments

To demonstrate colorimetric detection of the immobilized enzyme in a flowthrough format, a plastic housing was designed to drive convective flow through membranes. The schematic of the flow-through device consists of a functionalized membrane sandwiched between several acrylic laser-cut pieces (3 mm thick) which served as scaffolding to hold the membrane in place and as reservoirs for the flow medium. Two stainless steel hypodermic tubes (Ziggy's Tubes and Wires part no. 18R316-0.787) connected silicone tubing (McMaster Carr 51845K52) to a syringe pump at the inlet, and a spectrometer cuvette at the outlet. The individual pieces were glued together with epoxy potting compound (3M Scotch-Weld DP270). A breakdown of the flow-through device can be seen in Figures 5 and 6.

After the enzyme immobilization step, the membrane was placed between laser cut acrylic pieces and glued together to form the flow-through reactor. The membranes were then rinsed thoroughly by flowing DIUF water through the reactor with a syringe pump to wash off loosely bound enzymes. This process was carried out with several (2-4) 20 ml aliquots of DIUF water. Removing loosely bound enzyme promotes a steady-state situation in the membrane reactor by keeping the enzyme surface density constant during testing, and by preventing enzymes from falling into suspension and catalyzing reactions downstream.



Figure 5. Schematic of the laser cut acrylic flow-through device. Membrane (yellow disc) is sandwiched between the pieces and glued.



Figure 6. Assembled flow-through laccase-coated membrane device.

2.7 Optical Absorbance Measurement of Immobilized Enzyme Activity with Flowing Reactant

ABTS is a chemical compound used to observe enzyme reaction kinetics. When ABTS is oxidized by laccase, it changes from nearly colorless to deep greenish blue with an absorbance peak at 405 nm. ABTS may be used at different concentrations (0.02-9.1 mM) in phosphate buffer (25 mM to 100 mM) and pH 5 to pH 7 [27,28]. A syringe pump was connected to the flow-through device to feed it with ABTS solution at varying flow rates. With higher flow rates, the solution will have a shorter residence time near the membrane and vice versa for lower flow rates. The average dwell time in the microreactor was calculated by dividing the microreactor volume (76 μ l) by the volumetric flow rate from the pump. It is expected that increased dwell time will result in more reactions between the ABTS and immobilized enzymes on the membrane. Consequently, slower flow rates should yield higher absorption at 405 nm. Flow rates between approximately 2 and 50 ml/h yielded about 5 to 120 seconds of average dwell time. The reacted solution will be directed into a cuvette and analyzed in an optical spectrophotometer for absorbance measurements. For this purpose, a USB2000 spectrophotometer running on the Oceanview spectroscopy software (both from Ocean Optics, Inc.) will be used with an ultraviolet LED as the light source. The overall setup is presented in Figure 7.



Figure 7. (a) Flow-through reactor connected to a syringe pump; (b) cuvette containing reacted ABTS solution analyzed by a spectrophotometer.

Concentration of the reacted ABTS solution was calculated by rearranging the Beer-Lambert law – as presented in Equations (1) and (2). Here, c_0 is molarity of the ABTS used, c is the concentration of the reacted ABTS solution, ε is the molar absorption coefficient (36,000 M⁻¹cm⁻¹ for ABTS), and L is the width of the cuvette, giving the absorption A.

$$A = \varepsilon L c \tag{1}$$

$$c = \frac{A}{\varepsilon L} \tag{2}$$

Percentage of reacted molecules =
$$100 \frac{c}{c_0}$$
 (3)



Figure 8. Concentration of oxidized ABTS product in the permeate solution after reacting with laccase on flushed and unflushed membranes. The same flow rate series and ABTS concentration (1 mM) was used for both reactors.

2.8 Results from Optical Absorbance Measurements with Flowing Reactant

Optical absorbance measurements depended on flow rate, ABTS concentration, density of immobilized enzyme, and the protocol for handling the protein-coated membrane after drop-casting. In order to achieve steady-state, the flow-through membrane reactor should be washed meticulously so that the membrane is free of loosely bound enzymes. Aliquots from the permeate were tested for the presence of free laccase enzyme by exposing it to ABTS solution. It was observed that the amount of leached enzyme was
negligible after the third wash as the ABTS did not change color. Figure 8 shows that without flushing the reactor with DIUF water before introducing ABTS, the recorded product concentration was higher than when it was flushed. This is because the loosely bound enzymes ended up in the permeate solution and continued to oxidize ABTS. Not only did these suspended enzymes interfere with the measurement, but their departure decreased the enzyme density on the membrane, leading to a decrease in reactor efficiency over time.



Figure 9. Concentration of oxidized ABTS product in the permeate solution after reacting with laccase on (a) flushed and (b) unflushed membrane reactors. The same ABTS concentration (0.5 mM) and flow rate series was used for both reactors.

Figure 9 compares the effect of enzyme loss in a flushed vs. un-flushed reactor over time, with green arrows indicating the testing sequence. At the top right of the un-flushed plot in Figure 9(b), the product concentration is high (0.055 mM) for a dwell time of 120 seconds. However, after moving to higher flow rates (and shorter dwell times), then returning to the same 120 second dwell time, the reactor produces a lower product concentration (0.04 mM). Meanwhile, the round-trip product concentration in the flushed reactor Figure 9(a) starts out at a lower value, but is nearly reversible and is comparable to the final product concentration in the un-flushed reactor. In either case, flushed or unflushed, longer dwell times caused reactants to spend more time in contact with enzymes, enabling more reactions to take place and increasing the concentration of oxidized product. Such round-trip tests can not only measure enzyme activity but also help optimize the choice of flushing protocol and membrane material to promote steady-state conditions in the reactor. These cyclic tests can also validate that old enzyme has been eluted in applications that require periodic cleaning and refreshing of the membrane reactor surface.

Besides enzyme variant, dwell time, membrane material, and flushing protocol, the reactant concentration must be optimized for maximum percent conversion in a given time. Figure 10 compares two concentrations of ABTS over the same flow rate series. Both 0.5 mM and 1 mM ABTS concentrations lead to increased product concentration with greater dwell time as shown in Figure 10(a). The percentage conversion for the 0.5 mM reactant is higher than that for the 1 mM reactant at short dwell times, but as dwell time lengthens, the two start to converge as presented in Figure 10(b), suggesting that access to enzyme is limiting the conversion rate. Although higher concentrations of reactant do produce more product per time, they also lead to greater amounts of wasted reactants in the effluent

stream. If reactants are expensive relative to enzyme, this reactor could be redesigned to fit more membrane surface area in the same volume, putting more enzyme within a diffusion length of the reactants. Folded membranes and hollow-fiber membrane arrays are two common area-expanding strategies to increase the throughput of both membrane reactors and membrane filters.



Figure 10. (a) Concentration of oxidized ABTS in the permeate solution after reacting with membrane-immobilized laccase at two different ABTS concentrations ("25%" = 0.5 mM, "50%" = 1 mM). (b) Percentage of oxidized ABTS molecules over a range of dwell times at two ABTS concentrations.

Enzyme-catalyzed membrane reactors offer environmentally friendly, compact, and energy-efficient replacements for chemical production and energy generation. However, the best combination of enzyme variant, membrane structure, membrane coating, reactant concentration, and flow rate must be identified through modeling and experiment. One of the key variables is the residence time of reactants near the membrane-bound enzymes. A long dwell time is ideal for high conversion percentage, but not for fast production rates. Because the colorimetric and voltammetric assays in this work only require milliliter volumes of outflow, it is practical to explore a wider range of residence times. Another important variable is the enzyme itself. An enzyme such as laccase has hundreds of variants that evolved for optimal activity in different organisms with different environmental conditions. The optimal working temperature for laccase enzyme can vary depending on the source of the enzyme and its specific application. Generally, laccase enzymes exhibit activity over a broad temperature range, but their optimal working temperature is often in the range of 40 °C to 70 °C [29–33]. However, some laccases may have optimal temperatures outside this range, and their activity can be influenced by factors such as the enzyme's origin and any modifications it may have undergone.

The process yield of a membrane reactor is a strong function of temperature and flow rate, and it may even vary across a membrane thanks to positive thermal feedback. Temperature and flow sensing using microdevices in or near membranes could therefore potentially improve the process yield. In applications requiring MEMS to interact with the external environment, the incorporation of out-of-plane micro- and nanostructures is highly sought after because it creates three-dimensional features with increased environmental contact area. Our objective in the next chapter is to develop a method for the seamless integration of MEMS actuators with soft surfaces. Leveraging a sophisticated strain engineering technique, these structures are released and delicately transferred from silicon wafers to soft, porous, and stretchable fiber-based materials. Activation mechanisms, including heating, exposure to magnetic fields, or interaction with fluid flow, enable diverse functionalities.



Figure 11. Schematic of a microreactor incorporated with a fabric swatch/mesh near the membrane. The fabric swatch contains the MEMS actuators which will potentially provide sensing capabilities and local stirring in this flow-through format.

The strategic placement of MEMS actuators on a channel-spanning mesh near a membrane introduces local stirring, illustrated in Figure 11, which holds the potential to elevate reaction rates by bringing unreacted molecules closer to the membrane. Alongside actuators, the integration of membrane-based sensors provide real-time insights for optimizing flowthrough enzyme microreactors. Possible sensor types achievable through MEMS technology encompass local temperature monitoring through thin-film thermocouples or resistive traces, optical measurements for colorimetric indicators, and flow rate sensing via resistive bend sensing or thermal anemometry. Expanding the scope, these MEMS-based devices extend their utility to breathable, high-conduction flow-through structures such as tissue engineering scaffolds, bandages, and air filters.

Obtaining a thorough understanding of the optimal conditions for enzyme activity involves measuring these variables at a local level, ideally as close to the membrane surface as possible. The utilization of temperature sensors becomes crucial in this context, allowing for the measurement of local enzyme activity, particularly in exothermic or endothermic reactions where traditional voltammetric or colorimetric indicators are not readily available. Such a localized measurement approach provides valuable insights, facilitating the effective scaling up of enzymatic processes.

CHAPTER III

FLOW INDUCED MECHANICS OF STRAIN-ENGINEERED MICROCANTILEVERS INTEGRATED IN A FLOW-THROUGH SYSTEM

In this chapter, we report the fabrication process of stress-driven out-of-plane microcantilevers based on metal-oxide bilayer design and their mechanical characterization in a flow-through system. Microcantilever arrays are realized by using conventional micromachining techniques involving optical lithography and etching processes. Due to the geometry of the curled-up cantilever, the point of maximum load applied by the fluid flow is distributed along its body. Arrays of microcantilevers underwent testing within a flow medium, utilizing water and glycerol as the selected fluids. To corroborate our experimental findings and optimize parameters, we conducted fluid-structure interaction finite element modeling simulations for both fluid scenarios. By adopting this fluid-driven approach, we can simultaneously evaluate multiple structures and collect statistical data regarding their mechanical performance, durability, and suitability in different devices.

<u>3.1 Background: MEMS Fabrication Techniques</u>

MEMS microfabrication typically involves two main techniques: bulk micromachining and surface micromachining. Bulk micromachining involves shaping the MEMS device by patterning and etching the substrate. On the other hand, surface micromachining is characterized by depositing and patterning thin films on top of the substrate. These films can serve as structural support, conductive layers, or passivation coatings. When dealing with surface micromachined MEMS, it becomes crucial to carefully manage the residual stress within the thin-film structural layer. This stress has significant implications for the mechanical behavior of the devices. It can even lead to substrate bending and affect how well the film sticks to the substrate. Depending on how the thin films are deposited, they can either cause structures to bend or buckle [34] caused by compressive stress on clamped–clamped devices, or result in cracks and peeling of the film from the substrate due to tensile stress. Furthermore, the stress in the films also has a direct impact on the dynamic performance of MEMS, influencing factors like quality factor and resonance frequency [35,36].

Biological structures exhibit inherent hierarchical and intricate three-dimensional (3D) designs that span length scales from the nano to the macro scale [37]. The replication and utilization of these unique 3D structures in biomaterials offer a promising avenue for the design and fabrication of devices with advanced capabilities, emulating functionalities found in natural biomaterials [38]. This burgeoning interest in recreating diverse 3D structures, varying in shapes and scales, extends to numerous applications such as electronics [39,40], biomedicine [41,42], mechanical and metamaterials [43,44], energy harvesters [45,46], microelectromechanical systems (MEMS) [47,48], etc.

Strain engineering, a technique facilitating self-bending or self-rolling, stands out for its compatibility with standard microfabrication techniques. This quality positions it as a highly promising method for seamlessly integrating complex 3D sensors with microelectronic devices. The applications of this technology are extensive, covering a diverse range of fields. From biological applications involving the capture and interaction with cells to optical devices encompassing non-planar or suspended mirrors, waveguides, and photosensors, strain engineering proves its versatility. Moreover, it extends its utility to electrical and magnetic probes, including neural probes, as well as to the development of flow sensors for gases and liquids and temperature sensors. In essence, the exploration of 3D structures in biomaterials, particularly through strain engineering, not only opens new frontiers in technology but also lays the groundwork for innovations that mimic and integrate seamlessly with the intricate designs found in the natural world.

Various existing fabrication methods, including 3D printing, twophoton/multiphoton lithography, templated growth, and self-assembly, have been utilized for creating 3D structures. Despite their contributions, each of these approaches carries inherent limitations that hinder both production efficiency and device performance. Notably, their applicability is confined to a limited range of materials, and they prove incompatible with lithographic techniques [37]. To overcome these drawbacks, alternative techniques based on strain-induced bending/folding have been proposed [49,50]. Drawing inspiration from the ancient Japanese art of Kirigami (the art of cutting and folding), recent research has pioneered an innovative fabrication method for 3D structures. This method involves the compressive buckling of 2D precursors already bonded to predefined locations on a prestretched elastomer substrate, showcasing a promising leap forward in overcoming the existing limitations of conventional approaches.

Microactuators enabling vertical displacement have found widespread applications in various fields, including micromirror arrays [51], microgrippers [52,53], switches [54], programmable microstructures [55], and soft microrobotics [56]. The development of these microstructures involves the strategic use of various materials, including ceramics [57,58], metals [59-61], polymers [62], and hybrid combinations [63]. Numerous approaches and materials are available for constructing out-of-plane microactuators [64], yet thermal actuators consistently demonstrate distinct advantages, particularly in terms of large displacement, cost-effective mass production, and a diverse selection of candidate materials like ceramics, metals, and polymers [65,66]. Additionally, electrical thermal actuators comprising bilayer beams inherently offer the benefits of being lightweight with large areal densities [67,68]. The application of surface microfabrication provides a mature and efficient methodology for developing bilayer thermal actuators, facilitating testing and characterization on silicon wafers [55]. Although these lightweight microactuators have gained attention for their significant displacement, especially in applications like microgrippers and switches, other contexts such as micro clot retrievers [69,70], micro robots [71–73], and micro sails for chip-scale spacecraft [74,75], demand enhanced stiffness and bending strength as fundamental prerequisites.

The distinct benefit of incorporating MEMS components lies in their capacity to offer a transduction mechanism, converting analog physical signals into digital electrical signals and vice versa. A comprehensive overview of the common transduction mechanisms used by MEMS sensors and actuators have been outlined in [76]. Notably, there is a current emphasis on the adoption of out-of-plane micro- and nanostructures, especially in applications requiring MEMS/NEMS to interface effectively with the external environment. In contemporary applications, there is a growing preference for out-of-plane micro- and nanostructures, particularly when MEMS/NEMS need to establish effective interfaces with the external environment.

Despite the successful production of complex MEMS using conventional lithographic patterning and etch methods, there exists a demand for simpler fabrication techniques that can efficiently produce complex 3D structures from planar precursors in a reproducible and parallel manner. Methods inspired by origami (paper folding), kirigami (paper folding and cutting), and self-assembly have been proposed to actualize the transformation of 2D designs into intricate 3D structures [48].

The innovative application of the kirigami/origami technique introduces an additional level of flexibility in generating previously inconceivable 3D geometries, surpassing the limitations of conventional subtractive and additive fabrication designs. As a result, kirigami/origami and associated techniques have begun to find diverse applications, including MEMS/NEMS [48], energy storage systems [77], biomedical devices [78], devices for aerospace industry [79], and materials for mechanical and photonic applications [80–82].

Traditional fabrication methods encounter challenges when intricate nonplanar shapes, like cylinders, helices, and other complex geometries vital for capacitors, inductors, RF antennas, and magnetic devices, are required. Drawing inspiration from the principles of origami and kirigami [49,83–85], researchers have introduced a method involving strainengineered planar functional nanomembranes on sacrificial layers. This approach enables the construction of elaborate rolled-up structures, such as rings [86–88], tubes [89–93], and helices [94–97].

In the realm of micro-scale industries, a significant obstacle revolves around the fabrication of three-dimensional structures, particularly when aiming for parallel and costeffective production. This challenge arises due to the prevalent design of semiconductor processing equipment, primarily geared towards generating thin-film planar electrical connections. The self-folding technique, a form of self-assembly, emerges as a solution for achieving three-dimensional out-of-plane structures. This method capitalizes on the strain mismatch between two thin films, paving the way for the creation of structures essential in various applications, especially in instances where microelectromechanical systems need to seamlessly interface with elements in the external environment.

Proposed as an alternative to robot-based pick-and-place methods, self-assembly emerges as a solution to challenges like stiction and fragility. This approach facilitates the simultaneous assembly of numerous components across a broad scale, ranging from millimeters to nanometers [98–100]. The dynamics of self-assembly and re-orientation are often driven by surface tension and capillary forces, typically induced by the local melting of materials such as solder, polymers, and glass [101–105]. Another innovative strategy involves magnetic lifting with localized welding, contributing to the repertoire of self-assembly techniques [106]. Beyond traditional applications, self-assembly concepts find relevance in the realm of microfluidics, showcasing their versatility and potential in diverse fields [107–109].

Analogous to self-folding is the concept of self-rolling, a process typically involving the deposition of a single or multi-layer film with a stress gradient across its thickness. This layer is then released by removing a sacrificial layer. The structural layer bends or rolls as the stress is allowed to relax, ultimately adopting a conformation that minimizes potential energy. The literature abounds with examples of self-rolling and selfbending structures, showcasing the versatility of this approach. Some instances include the fabrication of self-positioned mirrors utilizing the strain in lattice-mismatched epitaxial layers [110]. Nano-belts made of SiGe/Si and SiGe/Si/Cr exhibit a helical curling effect [111]. Micro- and nanotubes, crafted from single-material thin films (tensile Si films on Ge sacrificial layers), demonstrate self-rolling capabilities [112]. Rolled-up tubes, formed from pre-stressed inorganic nanomembranes deposited on photoresist (polymer) sacrificial layers, illustrate another variation of this technique [91]. Additionally, 3D polymer structures, created using lithographically defined areas of stress in SU-8 photoresist [113], and self-assembled microstructures employing Cr/Cu bilayers [114], contribute to the diverse landscape of self-rolling applications.

The scope extends to various domains, encompassing the production of multilayer C/Si/C microtube anodes for lithium-ion batteries [77], microtube optical cavities fashioned from SiO_x/SiO_2 [115], cantilevers bending out-of-plane with different radii of curvature using a composite structural layer of silicon nitride and silicon oxide [116], and the self-assembly of inductors, transformers, and resonators using shapeable ultrathin films [117]. This assortment of applications underscores the adaptability and potential of self-rolling methodologies in creating intricate and functional structures across multiple disciplines.

Table 2

Comparison of different actuation principles and characteristics of microstructures

[118,119]

Actuation	Working Principle	Typical geometries Pros		Cons	
Electrostatic	Electrostatic force	Electrostatic Comb drive and force parallel plate Fast response and low power consumption		Large dimension and pull-in issue	
Electromagnetic	Magnetization effect	Switches for mirrors, pumps, etc.	Large displacement, quick response, and high precision	Large dimension and difficult to fabricate	
Electrothermal	Thermal expansion	Bimaterial bending cantilever	Large displacement and low voltage	High working temperature	
Piezoelectric	Piezoelectric effect	Bimorph bending cantilever	Large force and good operating bandwidth	Simple planar structures and high operating voltage	
Shape memory effect	Materials' deformation	Bimaterial bending cantilever	High energy density and flexibility	Hysteresis and large power consumption	

As highlighted earlier, microactuators play a crucial role in enabling microelectromechanical systems to execute various physical functions, such as the conversion of electrical signals into mechanical actions. The operational principles of MEMS actuators are contingent upon factors like structural dimensions, technology, response time, maximum power consumption, and the force and torque generated in relation to displacement. Table 2 provides a comprehensive classification of MEMS actuators and sensors based on their actuation principles [120]. This classification serves as a valuable reference for assessing the diverse functionalities and characteristics of MEMS devices, aiding in their optimal design and application across different domains.

MEMS devices employ various actuation mechanisms, including electrostatic, electromagnetic, piezoelectric, and thermal actuation [121–123]. Among these, electrostatic actuation stands out as the most prevalent due to its simplicity, compatibility with CMOS processes and materials, and relatively low power consumption. However, it comes with certain drawbacks such as the potential for high driving voltage and a non-linear voltage response.

While piezoelectric actuators boast significant actuation forces and rapid switching capabilities, they face limitations such as the inability to produce large strokes, a requirement for high operating voltage, and susceptibility to temperature fluctuations. In the context of piezoelectric actuation, the induced strain aligns closely with the applied electric field. Furthermore, it's essential to acknowledge the intricate fabrication processes that these actuators necessitate.

Electromagnetic actuators exhibit a considerable range of motion and force, contingent upon a solenoid with a magnetic core for generating a practical electromagnetic field. However, this effectiveness comes at the cost of a considerable power input, with a notable portion being converted into heat. The primary challenge associated with this actuation principle arises from the lack of a well-established fabrication technology, primarily due to the intricate nature of components involved, such as coils and magnetic materials.

Thermal actuation emerges as a compelling choice, offering substantial force and deflection based on the strategic selection of expansion materials. One of its distinctive strengths lies in the low driving voltage and the establishment of a nearly linear relationship between deflection and power. The fabrication processes for thermally actuated devices are generally uncomplicated, facilitating seamless integration with circuitry through standard CMOS processes. However, the trade-offs include high power consumption and limitations imposed by the speed of heating and cooling microactuators for operational frequency. Despite these challenges, thermal actuation can generate significantly higher forces compared to electrostatic actuation. Moreover, it enables versatile movements both away from and towards the substrate, thereby mitigating the risk of the pull-in voltage collapse inherent in electrostatic actuation structures.

Alternative actuation mechanisms, including shape memory effect actuation, chemical reaction actuation, or a combination of previously discussed mechanisms, offer specialized applications in MEMS. The choice of actuation mechanism is influenced by factors such as design specifications, application constraints, cost, and process compatibility. While no single mechanism dominates, some prove more effective in specific areas, while others exhibit maturity or promise untapped potential.

3.2 Background on Thin Film Stress-Based Assembly

Microstructures that deflect in response to mechanical forces are one of the largest application areas of microelectromechanical systems (MEMS). In the realm of microfluidics, flow rate measurements have become essential in many fields of engineering [124–126]. MEMS based flow sensors, generally classified as thermal or non-thermal [127], can detect a wide range of flow rates ranging from minute to significant. They are often put into fluid flow monitoring operations because of their small-scale size, while other attributes such as rapid response, low power requirement, increased precision, and lower manufacturing cost make them a good fit for microfluidics [128]. MEMS flow sensors incorporate various miniaturized moving structures such as springs, cantilevers, diaphragms, etc. developed using silicon and polymer materials [129–131]. Conventional sensing methods used by MEMS flow sensors are thermal [127], drag force [132], differential pressure [133], piezoresistivity [132,134], hot-wire anemometry [135], and optical detection [130,131,136]. However, most of these structures are fabricated in the plane of the flow. In this paper, we introduce a flow sensitive mechanism based on out-ofplane Au/SiO₂ microcantilevers with optical deflection read-out. The advantage of this mechanism comes from ease of setup in fluidic channels, and many points of measurement due to ease of fabrication in arrays.

MEMS microcantilevers are well known for their versatility and high sensitivity acting as sensing elements for scanning probe microscopy [137], air and liquid flow sensing [128], detection of numerous biological and chemical target analytes [138–140], humidity [141] and pH [142] measurements, novel applications in microbiology and genomics [143]. Typically, microcantilevers are placed in a microfluidic channel so that the fluid flows perpendicularly to them and the fluid momentum in turn deflects or bends the microcantilevers. The flow is acquired by quantifying the amount of deflection or strain caused by the drag force using optical or electrical approaches. Flow sensors reported in [144,145] used optical images of the deflected cantilever to detect the flow sensitivity. [146] reported in-line monitoring of flow fluctuations with SU-8 and SiN cantilevers integrated in a microfluidic channel. They used a laser beam focused on the surface of the cantilever to measure the displacement of the reflected laser beam on a position sensitive detector. [144] developed a multilayer soft lithography process to fabricate a polydimethylsiloxane (PDMS) microcantilever-based flow sensor which reported a minimum water flow rate of 35 µl/min. The deflection of the cantilever was measured for various flow rates between 0.2–1.3 ml/min by monitoring the cantilever deflection under an optical microscope and off-line image processing techniques. Stress-driven out-of-plane piezoresistive microcantilevers have been developed to effectively detect and measure flow velocity in water and gas [132,134,147]. The change in cantilever deflection due to flow velocity in the microfluidic system subsequently alters the resistance of the piezoresistor. Such cantilever deflection measurements are performed with optical or piezoresistive readout.

Significant research has been performed in the last decade to integrate thin filmbased flexible large area devices onto substrates that are porous, stretchable, breathable, and mechanically robust in nature [148–150]. Flowrate based measurements find a great deal of applicability in measuring the mechanical properties of 3D functional MEMS devices realized through micro-origami using forces derived from material properties such as strain mismatch, capillary forces, tensile forces, [48] etc. Especially in stretchable and flexible substrates [151–153], these flow systems can be employed to study the mechanical properties under various deformation modes such as biaxial, radial, etc. Compatible MEMS devices, which come in many shapes and manufactured with various materials, require careful measurement of their mechanical properties to be employed in systems where high-performance electronics and deformable mechanics perform collectively. Measuring device responses to flow stimuli can help explore mechanical properties such as bending stiffness, tensile stiffness, safe strain levels to avoid mechanical breakdown of the device [48].

In the following sections, we describe the residual stress-based fabrication of our microcantilever arrays, and the setup and analysis of optical deflection measurements on the cantilevers under controlled flow conditions in order to determine their mechanical properties and behavior as sensors. Fluid flow along the cantilever surface impacts and bends the microcantilever. Optical microscope video is analyzed with an edge detection algorithm developed using the MATLAB image processing toolbox to detect cantilever deflection under a steady flow of glycerol at increasing flow rates. Finite element modeling (FEM) simulation was carried out using COMSOL to analyze the deformation behaviors of microcantilevers due to applied force. The model was then validated using mechanical lateral bending from a calibrated direct contact probe.

We also developed an algorithm to track displacements of an array of cantilevers at >90% accuracy from microscope image data where two deflection regimes were observed over an array of 6 cantilevers. The cantilever edges were detected with 2-pixel tolerance. The accuracy of cantilever displacement detection was evaluated from the ratio of number of true positives for edge detection to number of points detected on the image. Results show mechanical robustness at flow velocity and drag force ranges of 0.48-5.7 mm/s and $0.35-4.23 \mu$ N, respectively, for operation with glycerol.

<u>3.3 Experimental Methods</u>

3.3.1 Theory of Thermal Bending

Out-of-plane structures can be created by depositing two layers with different thermal expansion coefficients onto each other. As thin films are deposited on substrate surfaces, by sputtering or chemical vapor deposition techniques, intrinsic stresses build up inside the film.



Figure 12. Schematic of metal-insulator bimorph bending because of stress-mismatch induced curvature upon release from the substrate.

The stress mismatch is created by patterning a metal-on-SiO₂ layer on a silicon substrate. Thermally grown SiO₂ generally has compressive stress, while the metal film used in this work has less compressive stress or tensile stress. Once the patterned bilayer structure is released from the sacrificial layer or substrate, the difference in stress between the two layers causes the cantilever structure to curl upwards as illustrated in Figure 12. During testing, fluid along the surface of the out-of-plane curled geometry impacts and bends the microcantilever. Optical microscope incorporated with an edge detection algorithm is employed to measure cantilever deflection.

Table 3

Material	Coefficient of Thermal Expansion	Young's Modulus <i>E</i> (GPa)

Physical properties of conventional materials employed in bilayer MEMS devices [154]

Material	Expansion α (×10 ⁻⁶ K ⁻¹)	Young's Modulus <i>E</i> (GPa)
Si ₃ N ₄	2.8	260
SiC	4.51	460
SiO ₂	0.5	70
Si	2.49	165
Al	23.1	70
Au	14.4	78
Ni	13.1	207
Cu	16.4	110

Among the conventional materials that are used for bilayer MEMS devices (Table 3), gold (Au) and silicon dioxide (SiO₂) are chosen for their large difference in coefficient of thermal expansion (CTE). Gold is an inert material hence, it does not form an oxide layer in water. The dimension of the layers are designed in a way so that the microcantilever works in the elastic region. This implies that the generated stress in each layer should be less than the yield stress of each layer. It is the region where the material can be deformed and once released will return back to its initial position. Based on Euler-Bernoulli beam theory, the ratio between length, width and thickness of the cantilever beam should be enough to consider the structure as a beam. The ratio of length to width and length to bilayer thickness are 30 and 300, respectively. The design parameters employed in the development of bimorph microcantilevers are presented in Table 4.

Table 4

Desig	<u>yn</u>	parameters	of	MEMS	cantil	levers	in	this	work

Material	Length (µm)	Width (µm)	Thickness (µm)	Young's Modulus (GPa)	Poisson's Ratio	Residual Stress (MPa)
SiO ₂	300	10	~ 0.5	70 [155]	0.2 [156]	-300 [157]
Au	300	10	~ 0.5	78 [158,159]	0.44 [155,160,161]	57 [162]

The bilayer develops a radius of curvature (ρ) when released from the surface because of differential thermal expansion between layers during fabrication. This is the radius that a cantilever attains when it is released, and no fluid is flowing. The inverse radius ($1/\rho$), or curvature, is expressed by Equation 4 [110]:

$$\frac{1}{\rho} = \frac{6\varepsilon(1+m)^2}{d[3(1+m)^2 + (1+mn)\{m^2 + (mn)^{-1}\}]}$$
(4)

where ε is the strain mismatch or fractional difference in the unconstrained relaxed lengths of the two layers $(l_b - l_a)/l_o$, *n* is the ratio of the elastic modulus *E* of the layers, $(n=E_a/E_b)$, *d* is the total thickness of the cantilever and *m* is the ratio of their thicknesses, $(m=d_a/d_b)$. Subscripts '*a*' and '*b*' refer to the upper metal and lower oxide layers, respectively. When the elastic moduli of the two layers are close to each other, i.e., m=1, and the ratio of the layer thicknesses are equal, n=1, then Equation 4 reduces to Equation 5.

$$\frac{1}{\rho} = \frac{6\varepsilon d_a d_b}{d^3} \tag{5}$$

$$\varepsilon = \frac{\sigma_{metal}(1 - v_{metal})}{E_{metal}} - \frac{\sigma_{oxide}(1 - v_{oxide})}{E_{oxide}}$$
(6)

The design parameters such as choice of materials and thickness of the bilayer determine the radius of curvature of the bimorph cantilever. The strain mismatch (ε) required in Equation 4 was calculated from Equation 6, where σ is the biaxial stress of the oxide and metal as measured by a profilometer pre and post deposition. Since we consider an oxidized wafer of 500 nm SiO₂ thickness, the upper metal (Au) layer thickness is what we can adjust to get the desired curled cantilever radius through the fabrication process. Figure 13 shows that the bilayer curls with a large radius when upper metal thickness increases. A theoretical radius of curvature value of ~180 µm is obtained for a curled cantilever by choosing a metal (Au) layer thickness of 470 nm from the graph.



Figure 13. Plot of radius of curvature versus thickness of upper metal layer from

Equation 4.

3.3.2 Device Fabrication

The fabrication process of the integrated microcantilevers within the microfluidic network is detailed in Figure 14. The devices for this project are fabricated with a two-mask cleanroom process. A standard 4-inch silicon wafer acts as the substrate for material deposition. The bilayers required for creating the curled-up geometry are SiO_2 and Au.

The process starts off by thermally growing a 500 nm SiO₂ layer on the substrate in a tube furnace at 1000°C (Figure 14a). Thermally grown oxide on silicon has a very high compressive stress due to the differential thermal expansion of the coating and substrate as the wafers cool. Photoresist (Shipley 1813) is then spun on the wafer and patterned using photolithography (Figure 14b). Next, a 10 nm thick titanium (Ti) adhesion layer and a 470 nm gold (Au) layer is deposited by sputtering. Combined metal layer thickness was measured at 480 nm using a Dektak profilometer (Veeco Instruments Inc.). After the metallization process, excess Au layer is removed by lift-off in the acetone bath followed by water. Thus, the metal layer is patterned after lift-off leaving the cantilever shape behind (Figure 14c). Photolithographic patterning is done once more using a second darkfield photomask to define where the SiO_2 will be etched to create a path to the silicon substrate (Figure 14d). Wafers were processed in a March reactive ion plasma etcher, with 300 mTorr pressure of CF_4 :H₂ at a partial pressure ratio of 50:3 and a RF power of 300 W. After 10 min of anisotropic etching, the 500 nm oxide was completely removed from unprotected areas (Figure 14e).



Figure 14. Microcantilever fabrication process flow: (a) oxidized Si Wafer; (b)photolithographic patterning using mask-1; (c) metal deposition and photoresist lift-off;(d) photolithographic patterning using mask-2; (e) SiO₂ etching and photoresist removal;

(f) metal-oxide bimorph is released by etching the silicon in XeF₂ gas.

The wafer is then diced after a protective layer of photoresist is applied over it. Once diced, the wafer has many individual dies which are later processed. The protective layer of photoresist on the die is cleaned with acetone and deionized water. The wafer is then dried under N₂ gas followed by a dehydration bake of 60 seconds. To release the bilayer cantilever structure, an isotropic xenon difluoride (XeF₂) dry silicon etcher (Xactix Inc.) is used to undercut the device and release it from the die surface (Figure 14f). The etch process fully removes the silicon from the underside of the oxide layer leaving a silicon cusp on the substrate at the centerline of the released structure. The overall process is detailed further in [153]. For 10 μ m lines, the width of the cantilevers, the etch process required about 10 or more 30 sec cycles of exposure to an atmosphere of 3 Torr XeF₂ for complete release from the substrate. The etch rate depends on the area of exposed silicon and will vary from pattern to pattern. However, wider lines always require more etch cycles for complete release, providing a mechanism to control the radius of curvature of microcantilevers based on XeF₂ etch time.



Figure 15. Microcantilever deformation caused by residual stress inside the bilayer structure. Top-view SEM image of (a) released cantilever array; (b) metal/oxide bilayer cantilevers curling up from a silicon substrate.

The stress mismatched bilayers curl up when released as pictured in Figure 15 from a scanning electron microscope (SEM). Cantilevers released successfully had a length of 300 μ m, thickness around 1 μ m and width of 10 μ m. The XeF₂ etch process is highly selective to silicon, enabling a wide range of metals and other materials to be used in the process.

3.3.3 Microcantilever Testing Under Fluid Flow

In order to characterize and observe the deformation of the integrated microcantilevers in a flow-through format, a plastic housing was designed to drive convective flow through it. The microfluidic device consists of a silicon die sandwiched between a plain glass microscope slide and a 3 mm thick acrylic laser-cut piece which serves as scaffolding to hold the die in place and act as the channel for the flow medium. The microfluidic channel has a total length of 4.15 cm and depth of 1000 μ m, while it is 750 μ m wide at its narrowest part. After the die is placed inside the flow-channel rastered on the acrylic piece with a laser cutter, the glass slide is glued to it thus forming the flow-through device. Two stainless steel hypodermic tubes are connected to the microchannel inlet and outlet. Silicone tubing are connected to the tubes for the fluid to enter and exit the device. The schematic of the flow through device is shown in Figure 16. The flow is injected into the device through the inlet by using a syringe pump, faces the microcantilevers and then exits toward the outlet. The individual pieces were glued together using epoxy potting compound.



Figure 16. (a) Schematic of the microfluidic flow channel etched onto a piece of acrylic;

(b) fabricated microdevice.



Figure 17. Schematic of the released cantilever array placed within the microfluidic channel. Liquid flows from left to right deflecting the free end of the curled cantilever beams.

To assess the performance of the microcantilever array suspended within the microfluidic channel, it was subjected to fluid flow (see Figure 17). The microfluidic device consists of a thin silicon die with an integrated microcantilever array encapsulated by a 3 mm piece of acrylic and a glass microscope slide. The microfluidic network is raster etched onto the acrylic piece with a laser cutter. The flow is introduced into the microdevice through the inlet, which induces a loading force on the microcantilever to bend it and then transferred out through the outlet. With water as the fluid, the deflection was too small to be detectable by eye under a microscope $(1-2 \mu m)$. Larger deflections were obtained with higher viscosity glycerol. Glycerol (99%) with a density of $\rho = 1260 \text{ kg/m}^3$ and dynamic viscosity $\eta = 1.412$ Pa s is injected into the microdevice through the inlet using a syringe pump at various flow rates while the cantilever deflection is monitored through an optical microscope. Images are captured before and after inducing flow for future image processing to determine the on-plane displacement of the cantilevers. The free end of the curled cantilever is deflected once the syringe pump is turned on to facilitate the fluid flow. The elasticity of the beam tends to restore it to its initial equilibrium state when the flow is stopped. Once the cantilever deflection reached its steady-state position, images were captured in an optical microscope (Olympus IX-70).



Figure 18. Method for measuring deflection from microscope images: (a) superimposed image of 3 cantilever edges before and after flow; (b) green and yellow points plot the cantilever edge coordinates on the image pixel map for without and during flow respectively; physical representation of the cantilever displacements for flow rates: (c) 20 ml/hr; (d) 30 ml/hr; (e) 40 ml/hr. The uncurling of the device towards a flatter position is evident in this view.

To measure cantilever deflection from microscope images collected under different steady-state flow rates, an edge detection algorithm was developed through MATLAB image processing toolbox and its predefined image processing workflows. The high-resolution images of the cantilever tips projected with high contrast under the scenarios of before and during flow are fused together using the *'imfuse'* function in MATLAB. The composite image is deduced using the *'falsecolor'* method wherein the images are overlaid

in different color bands with same intensity regions shown in gray color while magenta and green colors portray the variability in intensities of the two images. To facilitate edge detection, the composite image is then converted into a grayscale image presented in Figure 18(a), where the color information falls in the range of (0, 255) with the extremes representing black and white pixels respectively. In the merged image the cantilever tip without flow is shown in the grayscale range of 90 to 110 while the cantilever tip during fluid flow is displayed in the gray scale range of 150-170 as shown in Figure 18(b). The cantilever tip edges are identified by the pixel transformation point, their gray scale range and neighborhood pixel distribution and the mean of the edge detection coordinates is computed to get the cantilever tip pixel positions before and during flow conditions. The pixel displacement of the cantilever is thus calculated as difference of the x-coordinates of the edge detection points and is converted into microns using the pixel to micron conversion ratio which varied from 0.92–1.26 over the superimposed images for different flow rates illustrated in Figure 18(c,d,e). The variation in the pixel to micron conversion ratios for different images can be attributed to the image resolution adjustment over different flow rates, number of bits per pixel employed with the 'imread' function and composite image size determination by the 'imfuse' function. Each image consists of 3 cantilever displacements for a given fluid flow. For most of the fluid flow values, the algorithm yielded the displacement information for all 3 cantilevers with an accuracy of 92.3%.

<u>3.4 Finite Element Analysis</u>

3.4.1 Fluid-Structure Interaction: Theory and Equations

In the domain of computational fluid dynamics, data structures and numerical analysis are employed to analyze and solve the problems dealing with fluid flow. COMSOL Multiphysics, a finite element method based commercial software package, is used to produce a model and study the flow of liquid in the microchannel. An arbitrary Lagrangian-Eulerian (ALE) approach is utilized to derive the equations on the deformable domain. The structural deformations are solved using the elastic formulation and nonlinear geometry formulation, which allow large deformations. ALE is used largely in the analysis of Fluid-Structure Interaction (FSI) systems and is really effective when analyzing structural motions in which the structure is severely deformed, such as an impact problem or the analysis of a very flexible structure.

The ALE method handles the dynamics of the deforming geometry and the moving boundaries with a moving grid. COMSOL Multiphysics computes new mesh coordinates on the channel area based on the movement of the structure's boundaries and mesh smoothing. The Navier-Stokes equations solve the flow conditions that are formulated for the moving coordinates. The deformation of this mesh relative to the initial shape of the domain is computed using Hyperelastic smoothing.

The sequential coupled method is used to numerically solve the interactions between the fluid and solid structure. The flow is assumed to be laminar Newtonian, viscous and incompressible. The fluid flow is administered by single-phase, incompressible Navier-Stokes equations and continuity equations given below:

$$\rho \frac{\partial u}{\partial t} - \nabla \left[-pI + \eta (\nabla u + \nabla u^T) \right] + \rho (u \cdot \nabla) \cdot u = F$$
(7)

$$\rho \nabla \cdot u = 0 \tag{8}$$

where *u* denotes the velocity (m/s), η is the viscosity (Pa·s), ρ is the density (kg/m³), *T* is the absolute temperature (K), *I* is the unit diagonal matrix, *p* is the fluid pressure and *F* is the volume force affecting the fluid. It is assumed that no gravitation or other volume forces affect the fluid, hence *F* = 0.

For each flow rate, the fluid velocity at the entrance of the microchannel is provided as the boundary condition for the inlet. The pressure at the outlet is set to atmospheric pressure. No-slip condition is imposed for all the walls of the channel which are nondeforming and defined as u = 0. The fluid flow loading acting on the microcantilever is defined as the force per area:

$$F_T = -n \cdot (-p I + \eta (\nabla u + \nabla u^T)) \tag{9}$$

where u is the velocity field on the cantilever surface pointing out from fluid, n is the normal unit vector to the boundary, and F_T is the fluid loading, which is a sum of pressure and viscous forces. The fluid loading is applied on the cantilever surface as the end point of the microcantilever is affixed to the bottom of the fluidic channel. The structural deformations are solved with an elastic formulation and a nonlinear geometry formulation which enables large deformations.



Figure 19. (a) 2D model geometry in COMSOL; (b) generated mesh for the model.

At the channel inlet, the boundary condition for fluid flow assumes that normal inflow velocity is perpendicular to the flow inlet.

$$u = -u_0 n \tag{10}$$

where u_0 and n are the inlet velocity and normal unit vector to the boundary, respectively. As for the channel outlet, the boundary condition is set to p = 0.

$$p = p_0 \tag{11}$$

3.4.2 Design and Simulation Model

The MEMS based cantilever design includes defining the variables for the required geometry and selection of the parameters. A 2D model was designed in COMSOL where a horizontal microflow channel of 1000 μ m long and 500 μ m high is constructed for experimental analysis. Figure 19(a) presents the 2D model where a microcantilever is placed inside the horizontal channel. The curled-up bilayer microcantilever design has the same thickness of 0.5 μ m for SiO₂ and Au layers. The two materials are joined along their longitudinal axis serving as a single mechanical element and undergoes combined out-of-plane bending upon actuation due to fluid loading. The same material properties presented in Table 4 were used for simulation purposes. Fluid flows from the left side of the geometry, while the outlet is taken from the right side of the flow channel. As the fluid flows into the horizontal channel, it enters with a parabolic velocity profile and comes in contact with the microcantilever placed perpendicular to the flow inside the channel. The Reynolds number of the flow is small (Re << 100) and the flow has fully developed laminar characteristics owing to the channel's small dimensions.




microcantilever at t = 4 sec.

The inlet velocity was calculated using the equation below:

$$v_i = u_{mean} \cdot 6 \cdot (H - Y) \cdot \frac{Y}{H^2}$$
(12)

where *H* is the height of the microchannel, *Y* is length of the channel, u_{mean} is the centerline velocity, *U* is the inlet velocity, and *t* is time. The centerline velocity u_{mean} was calculated using:

$$u_{mean} = \frac{U \cdot t^2}{\sqrt{t^4 - 0.07t^2 + 0.0016}}$$
(13)

In this work, the simulation was performed using the two-dimensional incompressible Navier Stokes equations under the microfluidics model in COMSOL Multiphysics, designed to support the numerical modeling of fluid flow through a channel. The linear elastic model was assigned to the microcantilever, while the rest of the geometry was categorized as a laminar flow region. The two physics were fully coupled using the Fluid-Structure Interaction (FSI) interface in the MEMS module which allows solid and liquid equations to be formulated and solved at the same time.

The 2D numerical analysis is performed to estimate the displacement of the microcantilever under various flow rates. The proposed model consists of a microchannel with one inlet, one outlet while the microcantilever is placed inside the microchannel illustrated in Figure 19. Glycerol with density of $\rho = 1260 \text{ kg/m}^3$ and dynamic viscosity η

= 1.412 Pa·s is used as the medium to test the cantilever performance. The simulation was configured to analyze the cantilever deformation from 0 to 4 seconds, when the cantilever is close to its steady state. Figure 20(a) shows the von Mises stress and displaced microcantilever at its steady-state position (t = 4 sec). The inlet mean velocity for glycerol was set to 35 mm/s. Figure 20(b) shows the geometry deformation and flow at t = 4 sec. The flow stays laminar in most of the area, while the swirls are restricted to a small area behind the cantilever. The amount of displacement, the size and location of the swirls depend on the magnitude of the inflow velocity.



Figure 21. Simulation results of the microcantilever tip displacement under different flow velocities of glycerol (red curve) and water (blue curve).

The numerical results for the microcantilever response to various flow velocities of glycerol are compared with that of water in Figure 21. The simulation results for water as the flow medium confirms the experimental results where it is evident that the cantilever tip displacement is negligible. As for glycerol, there is a linear range of response for flow velocities 0–10 mm/s before the cantilever eventually reaches its steady-state position.

3.5 Results and Discussion

To observe the mechanical strength of the out-of-plane cantilever array, a microneedle probe was used to push it in the downward direction. The probe needle was fixed on an end-effector which was mounted on the 6 degrees of freedom (DOF) manipulator in the microassembly station of the NeXus system, which is a multiscale additive manufacturing platform integrated with 3D printing and robotic assembly [163]. By perpendicularly moving the needle down it gently pushed the microcantilever. The cantilever's behavior under the applied load will give us an idea of its mechanical strength and flexibility. As presented in Figure 22(a), the distance between the tip of the cantilever's free end and the die surface was measured with side view SEM images of the cantilever which was found to be in the range of 220–240 μ m. It can be seen from Figure 22(b), the cantilever tip on the free end almost touches the die surface without breaking, showing tremendous flexibility under load. Once the probe was retracted the cantilever returned back to its previous shape.



Figure 22. (a) Side view SEM image of the curved cantilever showing the distance from tip to the surface; (b) cantilever deformation under load using microneedle probe; (c)
COMSOL simulated Au/SiO₂ bilayer cantilever deformed under 5 μN applied force.

In addition, FEM simulation was carried out in COMSOL by solving a 3D curled cantilever model under applied force. Similar to the probe test, the curled end of the cantilever was chosen as the point of applied force while the other end was kept fixed to monitor tip displacement. In Solid Mechanics, a point load ranging from 0–10 μ N was applied on a node extreme to the fixed constraint set at the bottom end of the cantilever. The cantilever was modeled in accordance with the fabricated cantilever dimensions and using material properties mentioned in Table 4. In Figure 22(c), the displaced tip of the cantilever under a 5 μ N downward directed load can be observed to be touching the surface it rests on.

Optical microscope incorporated with an edge detection algorithm was used to measure cantilever deflection under a steady flow of glycerol at various flow rates. These flow test measurements have been carried out on a 300 μ m long and 10 μ m wide cantilever array by inducing the flow along the cantilever direction. For every flow rate, a set of still photos were captured from the bottom using a charge-coupled device camera mounted on an inverted microscope. The average and standard deviation were calculated from the data collected for each flow rate for the specified range. The displacement of the microcantilever was measured for flow rates ranging from 10–120 ml/hr or flow velocity ranges of 0.48–5.7 mm/s using image processing techniques. Figure 23(a) shows the measured displacement versus flow velocities. Because the drag force depends on the projected area that undergoes fluidic momentum, the displacement increases with initial flow velocities and saturates after flow levels the curled shape of the cantilever. The maximum tip displacement was measured at ~235 μ m which matches the range of the cantilever's typical 220–240 μ m height above the substrate.

Images of deflecting cantilevers were captured with a 10x objective mounted on the optical microscope. Using higher resolution objectives can increase the quality of the image taken by the optical microscope and improve the minimum flow rate induced deflection that can be detected using this setup. However, the trade-off with increased magnification is that fewer cantilevers are in the frame and the depth of field is reduced.



Figure 23. (a) Cantilever tip displacement under glycerol at different flow velocities; (b) Horizontally applied force versus displacement data from COMSOL simulation of a 3D curled cantilever model.

Flow rates in this work resulted in Reynolds numbers of 0.001–0.01, meaning that the flow was well into the laminar regime. In this Stokes drag regime, drag forces (F_D) are proportional to velocity (v) and to fluid viscosity (η) as follows:

$$F_D = k \eta v \tag{14}$$

where k is a geometric factor depending linearly on object width. In the smalldeflection regime, cantilever deflection (δ) is proportional to a distributed load. Here, I is the cantilever's moment of inertia, b is the width, l is the length, and h is the thickness of the microcantilever.

$$\delta = F_D l^4 / 8El \tag{15}$$

$$I = bh^3 / 12$$
 (16)

Table 5

Theoretical calculation parameters

Parameters	Symbols	Values
Fluid viscosity	η	1260 kg/m ³
Cantilever length	l	300 µm
Cantilever width	w	10 µm
Cantilever thickness	b	1 μm
Cross-sectional area of channel	A	5.85 mm ²
Cantilever moment of inertia	Ι	$0.833 \ \mu m^4$
Elastic modulus	E	74 GPa
Max. flow velocity	Vmax	5.7 mm/s
Min. flow velocity	V _{min}	0.48 mm/s
Max. drag force	F _{Dmax}	4.23 μN
Min. drag force	FDmin	0.35 μΝ

The slope of the early part of Figure 23(a) should therefore be proportional to viscosity. To investigate the cantilever displacement in the large-deflection regime (deflection > 0.1 * cantilever length), FEM simulation was performed in COMSOL Multiphysics using a curled-cantilever model to achieve deflections in the same range observed in Figure 23(a). The curled end or tip of the cantilever was chosen as the point of applied force while the other end was fixed as illustrated in the inset of Figure 23(b). A horizontal point load ranging from 0–10 µN was applied to a node located on the tip of the cantilever. The microcantilever was modeled following the fabricated cantilever dimensions and using material properties mentioned in Table 4. It can be observed from Figure 23(b) that a 4–5 μ N horizontal force is required to displace the tip of the cantilever by 250–260 microns. This force range matched well with the theoretical results presented in Table 5, where the maximum drag force calculated was 4.23 µN for a ~235 microns tip displacement presented in Figure 23(a). This analysis suggests that in applications, the curled cantilevers can recover from forces in the few micronewton range consistent with water and low-speed (<5mm/s) glycerol flows. Since the viscosity of water is more than three orders of magnitude smaller than that of glycerol, the deflection caused by water was not visible in our system.

<u>3.6 Potential Application as a Flow Sensor</u>

Table 6 presents a comprehensive comparative analysis of flow sensor design methodologies based on stress-driven cantilever beams. It highlights various aspects, including device geometries, actuation schemes, transduction principles, and the constituent thin-film layers used in these devices.

In [164], researchers utilized an arrangement of four freestanding micro-cantilever beams placed at right angles to each other to detect both air flow rate and direction. The velocity of the air flow is calculated by assessing the associated alterations in resistance within the piezoresistors positioned on the upper surface of each cantilever beam. As air passed through the sensor array, it induced deformations in the beams, resulting in changes in electrical resistance within the piezoresistors positioned on the upper surface of each cantilever beam. The resistance variations of the two cantilevers positioned perpendicular to the air flow direction were negligible. Moreover, the researchers demonstrated that the total resistance variations across all four cantilevers could be used to calculate the flow rate accurately.

In the context of flow sensing, [165] introduced a novel approach featuring a linear array of closely spaced stress-driven artificial hair cells. This array is designed to provide multi-parameter flow measurements, which are intended for use in controlling underwater vehicles. This array comprises multiple flow sensors aligned along the direction of the cantilever beam. To extract information regarding freestream flow direction and velocity from flow fluctuations, a real-time capable cross-correlation procedure was devised.

Table 6

A comparison of the MEMS devices presented in the literature and the device presented in

this work

Reference	Device Geometry	Actuation Scheme	Measuring Principle	Cantilever Layer(s)	Devices Per Experiment	Application
This work	Curled Cantilever	Water Flow	Image Processing	Au/SiO ₂	6	Flow Sensor
[164]	Curled Cantilever	Airflow	Piezoresistor (Pt)	Si ₃ N ₄	4	Flow Sensor
[165]	Curled Cantilever	Air and Water Flow	Piezoresistor	Si ₃ N ₄ /Si	3	Flow Sensor
[132]	Curled Cantilever	Water Flow	Piezoresistor (Si)	SiO ₂ /Si/SiO ₂	1	Flow Sensor
[134]	Curled Cantilever	Airflow	Piezoresistor (Pt)	Si ₃ N ₄	1	Flow Sensor
[166]	Curled Cantilever	Air and Water Flow	Piezoresistor (NiCr)	Si ₃ N ₄ /Si	1	Hair Cell Flow Sensor
[147]	Curled Cantilever	Gas Flow	Piezoresistor (NiCr)	AlN/Mo	1	Flow Sensor
[146]	Perforated Cantilever	Water Flow	Optical Readout	SU8/SiN	1	Flow Sensor
[167]	Free-standing Cantilever	Water Flow	Piezoresistor (N-type)	Au/Si	1	Flow Sensor
[130]	Optical Fiber Cantilever	Water Flow	Optical Readout	Silica	1	Flow Sensor
[144]	Flat Cantilever	Water Flow	Image Processing	PDMS	1	Flow Sensor
[168]	Out-of-plane Bimorph Beams	Temperature Variation	Capacitance	Au/Poly-Si	1	Temperature Sensor
[65]	Curled Cantilever	Electrothermal	Laser Doppler Vibrometry	SU8/NiTi	1	Thermal Sensor
[169]	Slightly bent Cantilever	Airflow	Piezoresistor (Pt)	Si ₃ N ₄	1	Sensor - Temp, humidity, anemometer

[166] introduced a biomimetic Si/SiN multilayered cantilever design, which employs an internal stress gradient to bend the beam out of the plane, facilitating the detection of flow velocity in a water environment. Along the full length of the cantilever beam, four nichrome 80/20 piezoresistors (100 nm thick) were evenly distributed and electrically configured into a Wheatstone-bridge circuit. Subsequently, bonding wires were meticulously attached to the contact pads to establish the necessary electrical connections. To safeguard the cantilever and its components from water exposure, a waterproof parylene coating was applied through chemical vapor deposition. This process uniformly added a 2 µm protective cover layer to envelop all facets of the flow sensor including the cantilever beam. Furthermore, the thin hydrophobic parylene coating does not affect the bending and radius of curvature of the cantilever.

A distinctive feature of our study compared to prior research is the capacity to employ multiple devices simultaneously for in-line flow sensing measurements. Unlike conventional flow sensor systems that typically record data from a single device at a time, our developed system can accommodate up to six cantilevers per flow measurement session. In this setup, the deflection of each cantilever tip is monitored through an optical microscope, with each image capturing all six cantilever beams. This approach allows for the concurrent measurement of several devices and facilitates the collection of statistical data concerning their mechanical performance in response to fluid flow. It's worth noting that the utilization of higher-resolution objectives can enhance image quality and improve the detection of minimal flow-induced deflection. However, it's important to consider that increasing magnification reduces the number of cantilevers visible in the frame and narrows the depth of field.

CHAPTER IV

THERMALLY DRIVEN MEMS FIBER-GRIPPERS

The goal of this chapter is to investigate mechanical tangling for adhesion of microelectromechanical systems (MEMS) to unconventional carrier materials for assembly of highly porous, fiber-based electronics. Adhesion stands as a pivotal factor in the realm of fabrication, presenting formidable challenges, particularly when dealing with continuous thin films of pliable materials like silicone and polyimide. The complexity of achieving adhesion escalates further when faced with discontinuous surfaces, such as fabric meshes. Nevertheless, these substrates hold the potential to expand the scope of MEMS into uncharted material domains. Operations that prove to be difficult on conventional circuit boards, such as facilitating the transfer of electronic contacts and fluids from one side of a mesh to the other, become notably more straightforward when utilizing a mesh as the substrate.

In this work, microgripper arrays are realized by microfabrication and release of strained metal-oxide bilayers. With this project, we aim to develop a process that wraps a MEMS gripper around a conductive fiber and reverses the process using electric current to open the gripper. In order to validate our experimental findings and optimize the parameters, we conducted simulations using finite element modeling.

4.1 Background on MEMS Microgrippers

Functional integration of heterogeneous materials can enable novel MEMS design formats to be implemented on substrates with characteristics such as stretchability, breathability, etc. The reversible geometric transformation of MEMS realized through thermal actuation via pulsed current can allow this integration to be real-time configurable.

MEMS actuation can enable scheduled release of environmental samples from microcontainers for analysis [170], while other MEMS based mechanical devices detect fluid flow events [171], steer and modulate light, produce three dimensional electric fields in microfluidics and other applications, and serve as electromagnetic resonators, antenna elements and probes for observing surface topology in atomic force microscopy [172].

Understanding the electrical and mechanical characterization of MEMS microgripper actuation can enable dynamic formatting of functional gripper contacts with fiber substrates. Decision parameters such as electrical contact area, mechanical clasp strength, and latching-unlatching with the fiber can be controlled by electrical switching between the various stable states of gripper actuation. These actuating MEMS fiber grippers can have a wide scope of applications in microrobot technology as fiber crawlers carrying payloads [173]. Contemplating locomotion, micro assembly and micromanipulations on fiber systems helps envision biological vessel networks for microsurgery [174], textile circuit routing [175], electronic textile fabrication [149] and tissue engineering [151].

Various methods realizing MEMS actuation have been demonstrated in literature such as, actuation triggered by light [176], fluid flow, electrothermal actuation

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[170,171,177], electrostatic actuation [178], electromagnetic actuation [179] and piezoelectric actuation [180]. The electro-thermal actuators can be further classified based on the basis of in-plane and out-of-plane actuation. While U-beam/hot-and-cold-arm actuators [181] and V-beam/chevron actuators [182] are employed for horizontal/lateral actuation, the bimorph actuators in this work and others [183] are used for out-of-plane actuation. MEMS actuators can be bilayer [153], trilayer [184] or multimorph structures implemented with materials with differential expansion. MEMS researchers have demonstrated bilayers of metal/diamond like carbon or metal/oxide, and trimorphs of metal/polymer/oxide for applications in medicine [185]. Bimorphs of Al/SiO₂ [186], and Cu/W [187] have been used to implement micro-mirrors.

Piezoelectric actuators [188,189] introduce hysteresis nonlinearity and demand high actuation voltages for minimal motion range, posing potential mechanical fatigue concerns. The performance of piezoelectric actuators can be influenced by temperature changes, which may require additional thermal management. Moreover, the performance of piezoelectric actuators can be influenced by temperature changes, which may require additional thermal management.

An alternative approach to MEMS-specific device actuation employs electrostatic forces, as demonstrated in studies by [177,190–192]. This method often involves the use of components such as comb drives or capacitors with movable arms. However, electrostatic actuators commonly face limitations due to the relatively small force generation and compact dimensions of capacitors. Furthermore, it's important to note that electrostatic actuation is not suitable for applications in a biological medium, as the applied voltage can lead to electrolysis when operating a microgripper in an aqueous environment.

The thermal actuation method allows for substantial displacement at relatively low voltage levels, relying on the thermal expansion of a specific mechanical component within the gripper when electric currents, driven by the Joule effect, are applied. This approach has been investigated in studies by [193–195]. However, a significant challenge arises from the potential for elevated temperatures in the vicinity of cells. To extend the application of thermal actuation to biological manipulation, it is essential to design the gripper's structure in a way that efficiently dissipates the heat generated by the actuators without causing damage to the surrounding cellular environment. A thermal gripper with significant potential was introduced and analyzed in studies by [196–198]. Subsequent experimental evidence, presented in [199,200], showcased the gripper's effective operation in practical settings, particularly in the micro-manipulation of cells submerged in a biological fluid. [170] introduced microfabricated containers designed for the storage of small environmental samples to facilitate analysis. These microcontainers utilized a strainarchitecture fabrication technique, causing planar microstructures to fold out of the substrate. They remained connected to electrodes, allowing for thermal actuation, electrochemical sample collection, and gas generation through electrolysis. The results demonstrated the successful collection of dissolved metals via electroplating, utilizing low voltages and currents compatible with the power resources of wireless sensor networks. These containers have the potential to expedite in-situ analysis of concentrated samples.

In this work we design, fabricate, and characterize the fiber gripper actuator response in terms of resistance, temperature, and radius of curvature (ROC) of the device with respect to current applied at the contact pads. We envision the gripper clasping and detaching from the fiber, and put forth insights of the gripper actuator carrying payloads.

4.2 Experimental Methods

4.2.1 Theoretical Analysis Methods

The MEMS microgripper is designed to have 12 gripper arms of length 740 μ m built in a continuous trace format (Figure 24) where one of the arms is fixed and connected to contact pads. The gripper's arm trace width is 10 μ m and its contact pads dimensions are 200 μ m * 200 μ m for the purpose of electrical probing for driving current and enabling thermal actuation. A donut-shaped etch window of 1850 μ m outer diameter and inner diameter 330 μ m encompassing the gripper is designed such that the gripper center is fixed to the substrate. The bimorph microgripper curls to a radius of curvature ρ when released from the surface due to differential thermal expansion of the bilayers. This is the radius that a working gripper gets to when no current is applied. The curvature, or inverse radius $1/\rho$, is given by Equation 17:

$$\frac{1}{\rho} = \frac{6\varepsilon(1+m)^2}{d[3(1+m)^2 + (1+mn)\{m^2 + (mn)^{-1}\}]}$$
(17)

where ε is the strain mismatch or fractional difference in the unconstrained relaxed lengths of the two layers $(l_B - l_A)/l_o$, d is total thickness $(d = d_A + d_B)$, n is the ratio of the elastic modulus of the layers, $(n=E_A/E_B)$ and m is the ratio of their thicknesses $(m = d_A/d_B)$. Subscripts 'A' and 'B' refer to the upper metal and lower oxide layers, respectively.



Figure 24. Bimorph MEMS microgripper design.

Equation 17 could also be represented as Equations 18, 19, 20 where t_o is the initial temperature of the gripper, t is the temperature at which the gripper starts to actuate. These equations are useful in obtaining the theoretical radius of curvature of the microgripper actuation for a given thermal condition resulting from applied current.

$$\frac{1}{\rho} = \frac{6(\alpha_B - \alpha_A)(t - t_0)(1 + m)^2}{d[3(1 + m)^2 + (1 + mn)\{m^2 + (mn)^{-1}\}]}$$
(18)

If $d_A = d_B$ then m = 1

$$\frac{1}{\rho} = \frac{24(\alpha_B - \alpha_A)(t - t_o)}{d\left[14 + n + \frac{1}{n}\right]}$$
(19)

If m = 1 and n = 1 that is $E_A = E_B$ and $d_A = d_B$

$$\frac{1}{\rho} = \frac{3(\alpha_B - \alpha_A)(t - t_o)}{2d} \tag{20}$$

The strain mismatch is induced by thermal expansion during fabrication, causing the layers to curl up from the substrate at room temperature. When the structure is heated, it opens and flattens, because the top metal layer has a more than 50 times greater thermal coefficient of expansion (TCE) than the oxide layer. T_f describes the "flat temperature" at which the released pop-up MEMS will become planar again:

$$T_f \approx T_o + \frac{\Delta T_B(\alpha_{si} - \alpha_B) - \Delta T_A(\alpha_{si} - \alpha_A)}{(\alpha_A - \alpha_B)}$$
(21)

where (in standard SI units) T_o is liftoff temperature (room temperature), ΔT_B is oxide deposition temperature - liftoff temperature, ΔT_A is metal deposition temperature - liftoff temperature, α_{Si} is silicon TCE, α_A and α_B are metal and oxide TCE, respectively. With increasing current through the metal layer, gripper temperature increases which is presented in Figure 25(b).

4.2.2 Finite Element Analysis Methods

A finite element modeling (FEM) simulation was carried out using COMSOL Multiphysics 5.5 software to analyze the mechanical deformation behaviors of the Au/SiO₂ microgripper due to applied voltage. The microgripper was modeled in accordance with the design dimensions discussed earlier and using original material properties provided in COMSOL material library. Following the construction of the model geometry, material properties are added to the bilayer structure with the top layer chosen as Au, while the bottom layer is assigned to SiO₂. For each microgripper arm the anchored surfaces around the center of the microgripper for both Au and SiO₂ layers were assigned to have mechanically fixed surface boundary conditions while other surfaces were kept free to move in the Structural Mechanics module. The finite element simulations were performed using physics-controlled meshing elements with a linear solver. Electric potential ranging from 0-1.5 V is applied on the fixed arm of the microgripper which is connected to the contact pads as depicted in Figure 24. This applied voltage induces an electric current and due to the material's resistivity, in this case Au, the current heats up the structure. The thermally induced stress loads the material and deforms the microgripper arms.



Figure 25. FEM simulation of the microgripper: (a) deformation with temperaturedependent electrical conductivity; (b) temperature of the microgripper top layer (Au) for different values of applied current.

By using the Joule Heating and Thermal Expansion predefined multiphysics interface, COMSOL automatically adds the equations for three physics including the necessary multiphysics couplings. COMSOL modules, Heat Transfer and Structural Mechanics, work in conjunction to model the mechanical deformation and performance of the microgripper structure as a function of temperature which is dependent on the voltage applied. Figure 25(a) illustrates maximum gripper tip displacement of 412 μ m resulting from an applied voltage of 1.5 V. The numerical results of the simulated temperature as a function of applied current are presented in Figure 25(b).

4.2.3 Fabrication Methods

The bimorph actuator is fabricated on the Si substrate by depositing strain mismatched layers of different thermal expansion coefficients. A 450 nm thick SiO₂ coating is thermally grown on a silicon wafer by wet oxidation at 1000 °C. The oxidized wafer is coated with Shipley 1813 photoresist, and the wafer is exposed on a contact aligner (Karl Suss) to ultraviolet (UV) light through a bright field mask. Image reversal using a Yes oven is carried out followed by flood exposure at the aligner and a development step in MF319 developer. The image reversal process makes the photoresist sidewalls slanted assisting in small features clearing in the lift off process. A Ti-Au metal layer of 480 nm combined thickness (where Ti and Au are 10 and 470 nm, respectively) is deposited on the wafer using a sputtering machine (Lesker PVD75). A lift-off process is carried out in acetone to obtain the metal patterned oxidized wafer. A second photolithography patterning is carried out using a dark field mask containing the torus shaped etch window design. Plasma assisted selective oxide removal is done in a reactive ion etch chamber (March) for about

10 min with 300 mTorr pressure of CF₄:H₂ at a partial pressure ratio of 50:3 and a RF power of 300 W. The processed wafer is then diced, and each die is wire bonded to a printed circuit board as shown in Figure 27. A single tinned Cu wire (Karl Grimm) is aligned to the gripper with a tolerance of 400 microns. An isotropic XeF₂ assisted Si etch is carried out using Xactix to release the MEMS gripper arms from the substrate, keeping the center of the device and contact pads attached to the wafer. The overall microgripper fabrication process flow is illustrated in Figure 26.



Figure 26. Microgripper Fabrication Process Flow: (a) 450 nm SiO₂ deposited on Si wafer; (b) Photoresist patterning carried out using Yes oven with gripper design mask; (c) 470 nm Au-Ti sputtering and lift-off using acetone; (d) Photoresist patterning for

selective etch windows; (e) SiO_2 plasma etch and fiber-tinned Cu alignment; (f) Dry

silicon etch to release bimorph actuator arms from the substrate.



Figure 27. Fabricated device wire bonded to a printed circuit board.

4.2.4 Device Characterization Methods

Fabricated bimorph cantilever array diced into wafer dies were placed onto a hot plate station to record the change in length (as seen from top view) based on changes in temperature. The experimental setup is illustrated in Figure 28. Optical measurements were taken by visual inspection of the actuators on-screen, with measurement error primarily coming from vibration of the hot plate stage. Optical measurements were recorded at different hotplate temperatures. These are cantilevers not microgrippers, but they have been fabricated using the same process and yields the same radius of curvature as the microgripper arms.



Figure 28. (a) L-Edit design of bilayer cantilever array (300 μm * 15 μm) – representing flat cantilevers before release; (b) Die consisting released cantilever array placed on a hot plate; (c) Top view image of released devices – microscopic image (left), schematic representation (right).

When heated from 30-160 °C, these actuators displayed relatively 'rigid' behavior and there was no visible shaking from the hot plate vibrations. After this threshold (up until the limit of 210 °C), the actuators gradually became more responsive to minor vibrations. This effect is prominent in the error bars of Figure 31. Shaking the heating stage through manual adjustments revealed strong Hookean behavior at high temperatures.

4.3 Results and Discussion

4.3.1 Gripper Actuation Results

The device behavior can be analyzed using the following results on gripper resistance, actuation current threshold, current handling capacity, electrical and mechanical insights on thermal actuation. We tested 34 grippers (1000 μ m diameter probing design gripper) for their average resistance when carrying 10 mA of current, the median value was 96 $\Omega \pm 31\Omega$, within uncertainty of the theoretical value of 108 Ω ; presented in Figure 29. After testing 16 grippers (1000 μ m diameter probing design gripper) for their maximum current density, the median value was 40 mA: vastly superior to the theoretical value of 9.4 mA.



Figure 29. Scatter plot of the measured resistance across different gripper devices at an applied current of 10 mA.

Although theoretical, the best temperature approximation where the metal and oxide flatten after release (Equation 18) is 337 °C (based on a metal deposition temperature of 200 °C). A model extrapolating the flat temperature vs metal deposition temperature (both above and below 200 °C) shows a linear trend.



Figure 30. Current loaded microgripper thermal actuation: (a) gripper at 5 mA applied current; (b) gripper unfolding at 15 mA; (c) gripper unfolding at 25 mA; (d) gripper at 35 mA.

Pop-up MEMS begin to 'unfold' perceptibly at approximately 6.5 mA. Grippers can handle 1 mA of current for 8.5 minutes uninterrupted. Figure 30 shows the thermal actuation of the fiber gripper at various levels of applied current. Working out the gripper actuation at lower currents and small step increments can increase the repeatability and actuation life of the gripper. The present Au-Pt devices can handle up to 50 mA of current without burning out compared to our previous devices made out of Cr/Ni/Cr and Ti/Pt that could handle only 3 mA and 18 mA of currents, respectively [201]. From our previous experiments, the gripper cools down to room temperature in vacuum (change of state) in 10 ms as an upper limit measured by SEM frame rate. The previous Cr/Ni/Cr and Ti/Pt devices could sustain 1000 cycles of state change. With the current gripper design with Au-Pt layers, we observed that higher the initial current induced and the longer amount of time it is induced, the smaller number of iterations the gripper can be re-used. In the future work we wish to employ devices with thin layer of Au over Cr, so that the electrical properties do not dominate the desired mechanical characteristics.

At room pressure and temperature, we cooled the devices by convection, placing an electric fan 30 cm from the devices while running current through the gripper. We observed that the fan reduced the measured resistance from 87.5 Ω to 61 Ω .

4.3.2 Radius of Curvature Versus Temperature

We tested 45 single gripper arms or cantilevers for their radius of curvature as temperature was varied. Despite large variance in individual grippers, an overall trend was modeled using the averaged results. The radius of curvature of our gripper clasps should theoretically agree with Equation 18. Using an initial approximation of α_B (SiO₂) value as 0.65 x 10⁻⁶ °C⁻¹, the calculated values in Table 7 were obtained. Table 7 shows the theoretical values for variables used in Equation 18 and the calculated values within the theoretical range represented by the predicted values in Figure 31. Allowing for tolerances of <5% for our parameters*, we observe that our values lie within the theoretical bound of our model.

Table 7

Variables	Theoretical	Calculated
α_A	13.8 ~ 22.6 x 10 ⁻⁶ °C ⁻¹ [202]	20.66 x 10 ⁻⁶ °C ⁻¹
α_B	$0.55 \sim 1 \ge 10^{-6} \circ C^{-1}$ [202]	0.65 x 10 ⁻⁶ °C ⁻¹
t ₀	~ 337 °C **	329.04 °C
D	~ 1 µm	0.875 μm

Theoretical and calculated values for variables in Equation 18

* Specifically, $\alpha_A \pm 1e-6 \ ^\circ C^{-1}$, $t_0 \pm 10 \ ^\circ C$, $d \pm 0.05 \ \mu m$, $n \pm 2\%$, and $m \pm 3\%$

** This value is obtained by inserting $\alpha_A = 14 \ge 10^{-6} \circ C^{-1}$, $\alpha_B = 0.71 \ge 10^{-6} \circ C^{-1}$, $\alpha_{Si} = 3 \ge 10^{-6} \circ C^{-1}$ in Equation 21

Figure 31 shows Equation 18 fitted to our calculated values as 'Predicted Values', with the tolerance bands represented in the 'Error Bound' range. The experimental values are shown for reference. Though many individual data points fall well outside the error bound (primarily due to manufacturing inconsistencies), our averaged values tended to stay within a 5-8% range.



Figure 31. Actuator curvature versus temperature. Experimental results in red, given error bounds in green. Averaged values generally fall in bound.

The experimental flattening temperature (radius of curvature ~ 0 μ m) was at 329.04 °C, supporting our model from the results presented earlier in section 4.3.1 that predicted 337 °C. Experimental observations saw a large increase in measurement error beginning at ~150 °C, as shown in Figure 31. Further increases in temperature only amplified this effect: small vibrations caused large, persistent gripper oscillations. This suggests that these grippers could be thought of as Hookean springs, with oscillations constrained in the direction of curvature.

Combined with our qualitative observations, the results confirm the theoretical models and suggest that grippers have low rigidity at higher temperatures. We believe this may imply a lower gripper contact strength at temperatures greater than 150 °C, which could be explored by stretching a fabric mesh with grippers latched on. This could also be a product of biaxial tension. Tension on the thin axis of the gripper arm that releases at the critical temperature could explain the sudden appearance of oscillatory behavior.

4.3.3 Radius of Curvature Versus Current

The radius of curvature of the gripper clasp, contact area, and conformability with the fiber are determinants of adhesion. Some of previous gripper to fabric release results illustrate the successful transfer and adhesion of MEMS devices to discontinuous surfaces like fabric meshes as shown in Figure 32. The firmness of the gripper clasp can be fine-tuned towards desired electrical and mechanical contact by understanding the variation of the gripper's radius of curvature with applied current.

The radius of curvature of the top view gripper images at varying currents is obtained through a MATLAB binary and grayscale image analysis algorithm which detects data points on the outer circumference of the gripper. For all the grippers images at different currents (with gripper arms making > 90° and < 90° turns), we applied conditions for detecting outer circumference points at angles where gripper arms were drawn. These data points were averaged to find the mean radius.



Figure 32. Successful transfer and adhesion of grippers to a fiber.

The microgrippers in this work require an applied current of 6 mA or greater to actuate them. Currents ranging from 0-5 mA produced no visible effect on the gripper arms upon inspection under optical microscope. In order to ensure repeated use of these microgrippers one has to ensure that the applied current is active for less than 45 sec during each run and current ranging between 0-25 mA. We noted that sending greater than 40-50 mA would cause the gripper to permanently open circuit, and at this point the gripper arms

curled more than the theoretical value of radius of curvature of a working gripper presented in Figure 33(b). The MEMS micro-hotplate literature has shown that thermal stresses in current-carrying thin metal films can damage SiO₂ underlayers [203]; a similar mechanism is likely at work here. Trigonometry was used to calculate the radius of curvature from the projected length and the known arclength of the gripper arm. Using $r = L/\theta$, where L = 740µm, the radius of curvature was deduced. In Figure 33(a) the gripper has radius of curvature of 146 µm. Here, the microgripper is at its resting state after being released through microfabrication with no applied current. Figure 33(b) on the other hand, was subjected to 50 mA current and has a radius of curvature of 97 µm.



Figure 33. Gripper radius of curvature for 740-micron long gripper arms (a) before carrying current; (b) after carrying 50 mA current.

Figure 34 shows the change in radius of curvature of the microgripper with respect to applied current. The blue plot shows the theoretical plot of applied current versus gripper radius of curvature derived from gripper radius versus temperature calculation from Equation 18 and linear interpolation of the temperature values on the FEM model in Figure 25(b) to obtain the corresponding current values. Also, the red plot shows the experimental data of radius of curvature along with standard deviation error bars at a given current obtained using image processing algorithm discussed above.



Figure 34. Blue curve: Theoretical plot of applied current versus gripper radius of curvature derived from Equation 18 and FEM model in Figure 25(b); Red points: Experimental data of radius of curvature at a given current obtained from image processing on gripper images from Figure 30.

Table 8

Theoretical T (°C)	ρ (x 10 ⁻⁸) (1/ Ω)	Theoretical R (Ω)
20	2.22	69.74
25	2.26	71.03
27	2.27	71.54
127	3.11	97.87
227	3.97	125.06
327	4.87	153.41
427	5.82	183.33
527	6.81	214.52
627	7.86	247.59

Theoretical resistance and temperature data for gold

4.3.4 Resistance Versus Temperature

Since the resistivity of most metals depend on temperature, we investigated the MEMS structures' function as resistive temperature sensors. Because radius of curvature depends on temperature, and temperature depends not only on current but on heat sinking by the surrounding environment, such self-temperature sensing will be useful in controlling the gripper's position. The current conductance happens through the upper Au metal layer of the bimorph. The positive temperature coefficient of resistivity of gold, indicates an increase in resistance with respect to increase in temperature due to applied current, which is shown in the theoretical plot of the graph Figure 35. The resistance is calculated by

multiplying the theoretical resistivity (ρ) by l/a, where l is the total trace length of the gripper actuator, which is 15.8 mm, and a is the cross-sectional area, 5 μ m². The theoretical resistance and temperature values for gold are presented in Table 8.



Figure 35. Blue curve: Theoretical resistance versus temperature for the top metal layer (Au) of the biomorph structure; Red points: Experimental data of measured resistances for 5 to 45 mA applied currents in 5 mA increments, placed on theoretical resistance versus temperature curve.
On the experimental standpoint, currents in the range of 5-45 mA in steps of 5 mA were applied to the device and subsequent resistance values were noted. From the trendline approximation of the theoretical curve, values for the temperature are obtained by linear interpolation into the curve at the measured resistance values (Table 9), and the experimental datapoints are plotted. Figure 35 shows how measured resistances were used to look up temperatures on the theoretical resistance-vs-temperature curve; temperature was not directly measured in this experiment. However, the resistance measurements were subject to instrument uncertainty, illustrated as vertical error bars on the experimental data points.

Table 9

Exp	erimental	resistance	and tem	perature (data 1	tor g	gol	d
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I (mA)	Experimental <i>R</i> (Ω)	ρ (x 10 ⁻⁸) (1/ Ω)	Experimental T (°C)
5	84.55	2.69	76.42
10	87.5	2.78	87.62
15	87.8	2.79	88.76
20	93	2.95	108.51
25	99	3.14	131.16
30	110.5	3.51	173.46
35	127	4.03	233.86
40	175	5.56	399.16
45	193	6.13	458.01



Figure 36. (a) SEM image of gripper actuator clasping fiber; (b) CAD model of similar gripper design carrying a semiconductor device payload: a mini-LED of dimensions 210 μ m * 130 μ m * 110 μ m.

4.3.5 Gripper Interaction with Fibers

Now we focus on how fibers influence the grippers' configuration by exerting forces on the gripper arms. Figure 36 shows the SEM image of a released microgripper latching onto a fiber. Such a structure might connect a sensor or other device payload to a conductive fiber for power, actuation, or communication. The key to connecting payloads is ensuring payload and fiber compatibility with the MEMS fabrication and release process. The tinned copper wire in Figure 36, added during fabrication (see Figure 26(e)), is unaffected by the highly selective XeF_2 release process. If applications require optimal contact the gripper radius can be matched to the fiber diameter (Figure 30) by working out the theoretical parameters (Equations 17 and 18) to get the right radius during fabrication, and fine tuning of the contact area can be done after release by changing the actuation parameters. Other payloads that include silicon, such as logic devices, would need to be passivated with a coating to protect them from the silicon etchant. The CAD model in Figure 36(b) shows how a mini-LED payload (Cree SR1321) would connect to a released gripper of similar design.

Figure 37(a) presents a released out-of-plane MEMS cantilever structure while another identical cantilever can be seen clasping onto a fiber after it had been released in Figure 37(b). The maximum horizontal extent from the attachment point of the 650 µm long cantilever is derived with and without fiber intervention through MATLAB grayscale image analysis algorithm. When the fiber is present, the cantilever is unrolled and is no longer a circle with a single radius of curvature. Since the gripper arms are making greater than 90 degree turns; the maximum horizontal extent from the attachment point is obtained from the top view, measuring the distance between the beginning of the curling part of the cantilever and the contact pad connection of the cantilever. We have narrowed down the detection search using "Range Method" and finding the mean of the data points. Here range method is the visual estimation of the range of values for X and Y coordinates of the point of detection. This image processing algorithm was applied on 5 different SEM images. The deduced mean value of the maximum horizontal extent from the attachment point is 330 μ m for images 'with fiber' which is greater than 137 μ m obtained for images 'without fiber'.



Figure 37. SEM image of (a) released cantilever; (b) out-of-plane cantilever clasping a fiber; 3D FEM simulation of a released gripper geometry – (c) initial position of the cantilever; (d) displaced cantilever due to a 5 μN lateral applied force.

FEM simulation was carried out in COMSOL Multiphysics by solving a 3D curled cantilever model under applied force. The curled body of the cantilever was chosen as the point of applied force while the other end was kept fixed to monitor tip displacement shown in Figure 37(c). In Solid Mechanics, a point load ranging from $0-10 \mu$ N was applied on a node located in the curvature of the microcantilever. The cantilever was modeled in accordance with the fabricated cantilever dimensions and using original material properties provided in COMSOL material library. Figure 37(d) illustrates the displaced cantilever tip recorded at 330 μ m under 5 μ N horizontal load. The distance from the pad to the maximum x-extent of the cantilever (the length of the green line) has approximately doubled in Figure

37(b) in comparison to the one in Figure 37(a). The FEM results show the same thing happening with a 5 μ N force. So, at this displacement, we can use the simulation to estimate that the cantilever is pushing back on the fiber with a 5 μ N force. This information is useful for estimating the force range needed to dislodge the gripper from the fiber.



Figure 38. (a) Optical microscope image of a microgripper arm being flattened using a microneedle probe; (b) COMSOL simulation of a bilayer microgripper arm or cantilever displaced with a 10 μN lateral applied force.

To further investigate the mechanical strength of these microgrippers, a microneedle probe (Dektak) was used to push one of the gripper arms vertically as shown in Figure 38(a). The probe was used to land on the fixed end of a gripper arm and then moved across its length to flatten it; upon release the gripper recovered its original shape.

From our observation, 1 mg or 9.8 μ N force was enough to flatten the gripper and not break it, which compares well with the result obtained through simulation. Figure 38(b) shows the displaced cantilever almost flattened under 10 μ N horizontal load.

4.3.6 Gripper Interaction with Soft and Stretchable Surfaces

In this section we present a method of incorporating MEMS microgrippers with soft, porous, stretchable fiber-based surfaces. As described in earlier sections these gripper structures are created from thin-film metal-oxide bilayers on a silicon substrate using a self-aligned lift-off and etch process to define bilayer beams with lengths in the 0.2-1 mm range [153,204]. These thin-film structures remain planar until released by dry silicon etching, allowing us to cover them with a piece of fabric or fiber mesh before the release step in the fabrication process illustrated in Figure 39. The dry silicon etch process exposes the material to xenon difluoride, a gentle silicon etchant that undercut the grippers through holes in the mesh. The compressively-stressed oxide layer causes the grippers to curl from the surface and interact with the mesh fibers. The grippers' radius of curvature is set by the thickness and elastic modulus of the layers [205,206]. Fabric-based carrier materials can serve multiple functions, including antibacterial properties, electrical conductivity, and responsiveness to heat, as long as they do not interfere with the highly selective silicon etching process. Additionally, these MEMS structures excel at seamlessly establishing electrical connections from one surface to the other, a feat that proves challenging when dealing with alternative soft materials such as silicone or polyimide.



Figure 39. Microgripper Fabrication Process Flow: (a) oxidized Si Wafer; (b)-(c) photolithographic patterning using mask-1; (d) 470 nm Au-Ti deposition using sputtering; (e) photoresist lift-off in acetone; (f)-(g) photolithographic patterning using mask-2; (h) SiO₂ plasma etching; (i) fabric swatch alignment with gripper features on the substrate; (j) fabric swatch attachment using mylar tape; (k) dry silicon etch using XeF₂ to release bimorph gripper arms from the substrate and enable mechanical clasping with the fabric swatch.

The overall fabrication process of the microgrippers and their incorporation with a fabric swatch is detailed in Figure 39. Here, the fabric samples (Matte Tulle Fuschia 100% nylon, Casa Collection Chiffon Chocolate 100% polyester, Glitterbug Micronet Fabric White 100% nylon, Jo Ann Fabrics) were aligned randomly with the gripper features on the substrate. The fabric swatch was firmly attached over the surface using mylar tape. Lastly, the grippers were released through isotropic Si etch using XeF₂ gas.

Figure 40 presents the successful transfer of the microgrippers from silicon substrate to a porous, stretchable, mesh-like carrier. Figure 40(a) shows an idealized vision of grippers transferring semiconductor payloads to a fabric swatch, creating a porous structure with mechanical strength coming from fibers and functionality from thin-film devices. After the grippers are released, they curl out of plane and securely latch onto the fabric, as detailed in Figure 40(d). Then the fabric swatch was carefully detached from the substrate. Subsequently, the fabric swatch is gently detached from the substrate and taken off the substrate. A thorough examination under an optical microscope confirms the successful transfer of the grippers to the mesh, as depicted in Figure 40 (b)-(c). These microgrippers demonstrate resilience when subjected to the self-aligned lift-off process from silicon to fabric, provided that the procedure is executed with care. However, it is essential to handle these fabric samples with caution to prevent any contamination that could potentially disrupt the microgrippers release process using XeF₂.

Porous substrates provide a reduced surface area for adhesion, but they introduce the possibility of mechanical tangling. Porosity allows electronic contacts to cross from one a mesh to the other, which is difficult to do on continuous films. Fluid flowing through a mesh can interact with MEMS for sensing and cooling. Carrier materials relying on

conductive fibers have the potential to advance the field of electronic textiles [149,204]. Radius of curvature for reliable clasping while curling depends on the fabric being clasped. Fabric samples used in this study had fiber diameters ranging from 50-100 μm.



Figure 40. (a) Desired integration of MEMS microgrippers with fabric layout; (b)-(c) optical microscopic image of the released gripper structure clasping the target fabric swatch. Images are taken after the fabric swatch had been detached from the Si-substrate;
(d) SEM image of the released gripper structure clasping the target fabric mesh.

Fabrics provide lightweight structural support and excellent air and fluid conduction, making them particularly advantageous for wearable sensors. Future work will encompass the measurement of forces during pull testing and the deliberate alignment of fibers on supporting structures, aiming to enhance the efficiency of the grippers as illustrated in Figure 40(a). In situations where alignment may not be feasible, the exploration of higher gripper density could offer a viable solution to establish a robust bond between a thin film device array and a fabric.

CHAPTER V

CONCLUSIONS AND FUTURE WORK

A flow-through spectroscopy device with laccase-coated membrane and a colorimetric laccase activity indicator (ABTS) was developed to measure reaction rate and percent conversion as a function of flow rate and membrane microstructure. Voltammetric analysis indicated the presence of *Trametes versicolor* laccase enzyme on SPE electrodes with an oxidation peak that more than doubled in current compared to that of the enzyme-free electrodes. This signature of an active enzyme can be used to sort biological products for their potential productivity in polymeric membrane bioreactors. Membrane porosity before and after laccase coating was verified with scanning electron microscopy. This experimental study focused on optimizing an enzyme-based membrane bioreactor. The primary focus was on modeling the tradeoff between achieving a high flow rate and maintaining a short dwell time in the active region.

To optimize not only the enzyme itself but reactant concentration, reactor geometry, membrane materials and methods, and flow rates, ABTS was used to measure laccase activity in a flow-through format. This scaled-down system had a volume of 76 μ l and used only 60 micrograms of laccase enzyme. The microreactor was subjected to flow rates ranging from 2-50 ml/h which resulted in approximately 5-120 seconds of average dwell

time for ABTS molecules near the laccase immobilized membrane. Key developments toward a miniaturized membrane reactor optimization platform were (1) a round-trip dwell time test to evaluate flushing protocols, and (2) conversion rate comparisons to determine whether output is limited by reactant concentration or enzyme availability. The maximum product yield of 3.9% might be increased by longer dwell times. A prolonged dwell time is favorable for achieving a high conversion percentage but not suitable for rapid production rates. However, the flushing results presented in Figure 9 also suggest better enzyme binding will lead to a more active catalytic surface and higher conversion rates for a given dwell time in this reactor. Successful development of enzyme-based membrane bioreactors demands that enzymes are densely attached, and also attached securely enough to withstand convective flows and washing. They must be oriented to retain their natural catalytic activity and function as much as possible. Thanks to their evolution in natural systems, enzymes work under mild temperatures and in biocompatible chemical environments. Because of these advantages, the enzyme attachment problem has been approached via adsorption, physical entrapment in polymers and sol-gels, and covalent attachment or self-assembly onto various surfaces [207-209]. Histidine-tagged recombinant proteins are an efficient method of controlling the adhesion of a protein on surfaces functionalized with cobalt, nickel, and other metal ions [210,211]. The challenge for genetic engineers is to insert the histidine tag at a location within the protein that does not interfere with regions that are used for catalysis.

In future work, this microscale system could be a platform for evaluating submilligram quantities of genetically engineered enzymes in a membrane reactor environment before investing in production and scale-up. The broader goal is to apply enzyme-coated nanomembranes for efficient processing of lignin-rich biomass into fuels and other valuable chemical products.

Chapter three of the dissertation explores the fabrication process of stress-driven out-of-plane MEMS microcantilevers, employing a metal-oxide bilayer design, and delves into their mechanical characterization within a flow-through system. The performance of bimorph cantilevers has been tested by introducing various flow rates into a microfluidic device and observing their response with an optical microscope. Due to the geometry of these curled cantilevers, the load applied by the fluid flow is distributed along its body. These cantilevers showed mechanical robustness at flow rates ranging from 10-120 ml/hr for operation with glycerol. We found that the thin film cantilevers in this study exhibit deflection without undergoing damage when exposed to flow velocities and drag forces ranging from 0.48–5.7 mm/s and 0.35–4.23 µN, respectively. A fundamental distinction between these cantilevers and the prevailing MEMS deflecting-cantilever designs lies in their three-dimensional structure, enabling lateral deflection parallel to the substrate when subjected to fluid flows. Initially designed as planar structures, they can be enhanced with piezoresistive traces to create an integrated flow sensing device as demonstrated in previous studies [132,134,147]. Parylene conformal coating proves to be a proficient method for water sealing and offers a versatile solution for post-fabrication adjustments to fine-tune the properties of MEMS microcantilevers [166,212]. In this work, the microcantilevers were assessed using a syringe pump. However, employing a more precise flow measurement instrument would facilitate calibration, allowing for operation at considerably lower flow rates. A distinctive feature of our study compared to prior research is the capacity to employ multiple devices simultaneously for in-line flow sensing

measurements. Unlike conventional flow sensor systems that typically record data from a single device at a time, our developed system can accommodate up to six cantilevers per flow measurement session. This approach enables the simultaneous measurement of multiple devices, facilitating the collection of statistical data on their mechanical performance in response to fluid flow. In addition, the sensitivity of these microcantilevers can be furthered by modifying their dimensions and employing higher resolution optical objectives. However, it's important to consider that increasing magnification reduces the number of cantilevers visible in the frame and narrows the depth of field.

In this study, a flow-based mechanical testing platform was also developed for arrays of out-of-plane deflecting structures. While probe-based force testing remains an option for deflection measurement, it comes with significant disadvantages, including high equipment costs, the potential for stress concentration due to point contact, and the need for a skilled operator to precisely land on an individual cantilever. Moreover, conducting lateral probe-based deflection tests presents additional challenges, particularly the risk of inadvertently damaging the substrate with the force probe. In the context of characterizing lateral deflection devices, flow testing emerges as a more favorable option, offering numerous advantages over probe-based testing. These benefits include the ability to simultaneously assess multiple devices, simplified contact initiation without the need for time-consuming alignment, and the capacity to evaluate devices in orientations closely resembling their real-world applications.

The dissertation in chapter four demonstrates a method of integrating MEMS devices with highly porous and stretchable substrates such as fibers, fabric, etc. in a way that does not affect the MEMS fabrication process or the thermal functionality of the

devices. MEMS microgrippers were fabricated using strain architecture technique and once released, the microgripper curl-up due to the stress mismatch between the bilayer structure. This work describes a process that wraps a MEMS gripper around a conductive fiber and reverses the process using electric current to open the gripper. The gripper's electrical resistance serves as a self-temperature sensor over the 20–500 °C range. Beyond their potential for adhering MEMS to fabrics and to flexible, stretchable substrates that are incompatible with or resistant to adhesives, these microgrippers illustrate how MEMS-based microrobots might interact with small-scale (< 200 μ m diameter) soft and biological structures that require sub-millinewton contact forces. The key contribution of this work over our earlier work is demonstrating the grippers' temperature-dependent resistance, which offers a route to improved control of the gripper state.

While MEMS technology offers tremendous potential, especially when combined with other technologies like ICs and/or photonics on a common substrate, a notable limitation is that they are typically fabricated on rigid silicon substrates that lack flexibility and conformity when attached to soft, pliable, and occasionally porous substrates. This study explores a novel packaging method that utilizes mechanical tangling to seamlessly integrate MEMS with fibrous materials commonly found in wearables, soft robotics, and other applications with high deformation requirements. It also addresses bioengineering environments, such as perfusible cell-growth systems, that requires a porous substrate for fluid access. The reversible clasping demonstrated in this research could potentially serve as a "programming" method for transferring devices to fibrous substrates by initially opening all devices on a wafer and subsequently closing only those intended for transfer. This selective fan-out approach already offers economic advantages for producing largearea devices from small wafers, similar to commercial micro-transfer printing used in the manufacture of large-format displays with inorganic LEDs transferred from densely populated donor wafers. Future work on this project will be focused on the investigation of the microgrippers' pull-off strength, electrical contact resistance, and payload integration (as depicted in Figure 36(b)) before the transfer process.

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APPENDIX A

Calculating the Volume of the Microreactor



Figure 1. Cross-section of the micro-reactor reservoir.

Microreactor Volume = π (Reservoir Radius)² (Reservoir Depth)

 $=\pi (0.565/2)^2 (0.3)$

 $= 0.076 \text{ cm}^3$

= 0.076 ml

= 76
$$\mu$$
l [since, 1 ml = 1 cm³]

Recipe for 0.5M 1L laccase buffer solution

- 1M monobasic solution: 192.5 ml
- 1M dibasic solution: 307.5 ml
- 200 mM KCL solution: 500 ml
- *Trametes versicolor* laccase powder: 3g

Recipe for preparing 0.5L 0.01M sodium acetate solution

- 0.01M sodium acetate solution: 352.5 ml
- 0.01M acetic acid solution: 147.5 ml
- m-cresol solution: 0.027 ml at 0.0005 M/L concentration

Chemicals purchased

- KCl > Sigma-Aldrich Product No. P9541
- Potassium phosphate monobasic > Sigma-Aldrich Product No. 60220
- Potassium phosphate dibasic > Sigma-Aldrich Product No. P3786
- Trametes > Sigma-Aldrich Product No. 38429
- Sodium acetate > Sigma-Aldrich Product No. S8750
- Acetic acid > Sigma-Aldrich Product No. A6283
- m-Cresol > Sigma-Aldrich Product No. 65996

APPENDIX B

MATLAB image processing to measure the microcantilever tip displacement under to various fluid flow rates

The cantilever tip displacement due to fluid flow is measured using image processing in MATLAB software. The optical microscopic images of the cantilever tip before and during various flow rates are taken without changing the magnification and position of the sample, with adjusting just the focus to view the tip clearly. This is done so that the overlap of before and after flow images of the tip gives accurate tip displacement positioning as can be observed the Figure 2(c). The pink and green tips in the overlap image in Fig 2(c) show tip position before and during flow respectively. This overlap image is put to grey scale image processing to identify the tip edges and eventually the displacement of the cantilever tip.

The imfuse(A,B), MATLAB function is used to create a composite image from two images, A and B. If A and B are different sizes, *imfuse* pads the smaller dimensions with zeros so that both images are the same size before creating the composite. The output, C, is a numeric matrix containing a fused version of images A and B.



Figure 2. Optical images of cantilever array inside the flow chamber.

The output color channel is specified by "green-magenta" representing the vector [2 1 2], which is a high contrast option.

The Pseudo Code for the MATLAB Program is as follows:

- The fused image which is a *truecolor* image file is initially inputted as an m-by-nby-3 array, I
- The *rgb2gray* function is used to convert RGB fused image to grayscale, I1 by eliminating the hue and saturation information while retaining the luminance
- The grayscale values are evaluated by a weighted sum of the R, G, and B components as follows:

0.2989 * R + 0.5870 * G + 0.1140 * B
- Further, the grayscale information is converted to binary matrix. And image
 properties such as centroid, PixelList, Bounding box, extrema and image matrix
 factorization using SVD (Singular Value Decomposition) are retrieved for
 processing the fused image for cantilever displacement details.
- With the image pixel information (*impixelinfo* MATLAB function), the grayscale value range, and position (x,y) range of the cantilever tips 'before flow' are noted against the background/ surrounding contrast pixels
- The grayscale Matrix I1 is searched for 'before flow' cantilever tip co-ordinates by giving the position and intensity cues against the neighborhood pixels from the above retrieved information by initiating a nested for loop within the row-column size of Matrix I1
- The average value of the bunch of points identified at the cantilever tip which are 23-pixel distance from each other is evaluated as the 'before flow' cantilever tip co-ordinates, (A.B)
- The above three steps are repeated for finding the 'after flow' cantilever tip coordinates, (A1, B1)
- Euclidean distance, d between two points (A, B) and (A1, B2) using the formula
 d = sqrt((A-A1)^2+(B-B1)^2)) is found, which is the required cantilever tip displacement resulting from fluid flow

MATLAB code for Cantilever Edge Detection and Tip Displacement for 30ml/hr

```
I=imread('si1.jpg');
I1=rgb2gray(I);
BW = imbinarize(I1);
doubleB=im2double(BW);
[U,S,V]=svd(doubleB);
V2=transpose(V);
%imshow(BW);
%impixelinfo
L=BW;
stats = regionprops(L,'centroid');
stats1 = regionprops(L,'PixelList');
stats3 = regionprops(L,'BoundingBox');
stats4 = regionprops(L,'Image');
stats5 = regionprops(L,'Extrema');
%[r,c] = find(bwlabel(BW));
b=1;
[h1,11]=size(I1);
  t=1;
  t2=15;
   t3=20:
  t1=1;
   t4=5;
for j=1:1:11
              for i=1:1:h1
                                 C(i,j)=i;
                                    D(i,j)=j;
                                    n=1:
                          if i+t3<h1 && j+t2<l1 && i-t3>0 && j-t2>0
                                                   if I1(i,j) \ge 92 && I1(i,j) \le 110 && I1(i,j+t2) \ge 92 && I1(i,j+t2) \le 110 && I1(i-t) \le 110 && I1(i-t) \le 110
t_{3,i} >= 200 \&\& I1(i-t_{3,i}) <= 255 \&\& I1(i+t_{3,i}) >= 200 \&\& I1(i+t_{3,i}) <= 255 \&\& I1(i-t_{3,i}) <= 255 \&\& I1(i-t_{3,
t3,j+t4 >= 200 && I1(i-t4,j+t2) <= 255 && I1(i,j-t) >= 200 && I1(i,j-t) <= 255 && I
I1(i,j+t4) >= 92 \&\& I1(i,j+t4) < 110
                                            Y(b)=i;
                                            X(b)=j;
                                            b=b+1;
                                                       end
```

```
end
    end
end
imshow(I1);
impixelinfo
k=1;
for i=1:1:b-1
  Sum1=0;
Sum2=0;
h=0;
if i==1
 for j=1:1:b-1
 if abs(X(j)-X(i))<23 && abs(Y(j)-Y(i))<23
   Sum1 = Sum1 + X(j);
   Sum2 = Sum2 + Y(j);
  h=h+1;
 end
 end
 A(k)=Sum1/h;
 B(k)=Sum2/h;
 k=k+1;
end
if i~=1
  r=0;
  for z=1:1:i-1
  if abs(X(i)-X(i-z))>23 || abs(Y(i)-Y(i-z))>23
    r=r+1;
  end
  end
  if r==z
  for j=1:1:b-1
 if abs(X(j)-X(i))<23 && abs(Y(j)-Y(i))<23
   Sum1 = Sum1 + X(j);
   Sum2 = Sum2 + Y(j);
  h=h+1;
 end
  end
 A(k)=Sum1/h;
 B(k)=Sum2/h;
 k=k+1;
  end
end
```

```
end
hold on
plot (A,B,'*g');
hold off
I=imread('si.jpg');
I1=rgb2gray(I);
BW = imbinarize(I1);
doubleB=im2double(BW);
[U,S,V]=svd(doubleB);
V2=transpose(V);
%imshow(BW);
%impixelinfo
L=BW;
stats = regionprops(L,'centroid');
stats1 = regionprops(L,'PixelList');
stats3 = regionprops(L,'BoundingBox');
stats4 = regionprops(L,'Image');
stats5 = regionprops(L,'Extrema');
%[r,c] = find(bwlabel(BW));
b=1;
[h1,11]=size(I1);
t=1;
t2=12;
t3=15;
t1=2;
t4=10;
for j=1:1:11
  for i=1:1:h1
     C(i,j)=i;
     D(i,j)=j;
     n=1;
    if i+t3<h1 && j+t2<l1 && i-t3>0 && j-t2>0
```

```
\begin{split} & \text{if } I1(i,j) >= 140 \ \&\& \ I1(i,j) < 155 \ \&\& \ I1(i,j+t2) >= 140 \ \&\& \ I1(i,j+t2) < 155 \ \&\& \ I1(i+t3,j) >= 200 \ \&\& \ I1(i+t3,j) <= 255 \ \&\& \ I1(i+t3,j) <= 255 \ \&\& \ I1(i+t3,j) <= 255 \ \&\& \ I1(i+t3,j+t4) >= 200 \ \&\& \ I1(i+t3,j+t4) <= 255 \ \&\& \ I1(i,j+t4) >= 200 \ \&\& \ I1(i+t3,j+t4) <= 255 \ \&\& \ I1(i,j+t4) >= 200 \ \&\& \ I1(i,j+t4) <= 255 \ \&\& \ I1(i,j+t4) >= 140 \ \&\& \ I1(i,j+t4) < 155 \ \&\& \ I1(i+t1,j) >= 140 \ \&\& \ I1(i+t1,j) <= 155 \ Y1(b) = i; \end{split}
```

```
X1(b)=j;
       b=b+1;
        end
        end
    end
end
k=1;
for i=1:1:b-1
  Sum1=0;
Sum2=0;
h=0;
if i==1
 for j=1:1:b-1
 if abs(X1(j)-X1(i))<23 && abs(Y1(j)-Y1(i))<23
   Sum1 = Sum1 + X1(j);
   Sum2 = Sum2 + Y1(j);
  h=h+1;
 end
 end
 A1(k)=Sum1/h;
 B1(k)=Sum2/h;
 k=k+1;
end
if i~=1
  r=0;
  for z=1:1:i-1
  if abs(X1(i)-X1(i-z))>23 || abs(Y1(i)-Y1(i-z))>23
    r=r+1;
  end
  end
  if r==z
  for j=1:1:b-1
 if abs(X1(j)-X1(i))<23 && abs(Y1(j)-Y1(i))<23
   Sum1 = Sum1 + X1(j);
   Sum2 = Sum2 + Y1(j);
  h=h+1;
 end
  end
 A1(k)=Sum1/h;
 B1(k)=Sum2/h;
 k=k+1;
  end
```

end

end

```
hold on
plot (A1,B1,'*y');
hold off
```

MATLAB CODE for Cantilever Edge Detection and Tip Displacement for 50ml/hr

```
I=imread('si5.jpg');
I1=rgb2gray(I);
BW = imbinarize(I1);
doubleB=im2double(BW);
[U,S,V]=svd(doubleB);
V2=transpose(V);
%imshow(BW);
%impixelinfo
L=BW;
stats = regionprops(L,'centroid');
stats1 = regionprops(L,'PixelList');
stats3 = regionprops(L,'BoundingBox');
stats4 = regionprops(L,'Image');
stats5 = regionprops(L,'Extrema');
%[r,c] = find(bwlabel(BW));
b=1;
[h1,11]=size(I1);
t=1;
t2=15;
t3=20;
t1=1;
t4=5:
for j=1:1:11
  for i=1:1:h1
     C(i,j)=i;
     D(i,j)=j;
     n=1;
    if i+t3<h1 && j+t2<l1 && i-t3>0 && j-t2>0
```

 $if I1(i,j) \ge 92 \&\& I1(i,j) < 110 \&\& I1(i,j+t2) \ge 92 \&\& I1(i,j+t2) < 110 \&\& I1(i,t3,j) \ge 200 \&\& I1(i-t3,j) \le 255 \&\& I1(i+t3,j) \ge 200 \&\& I1(i+t3,j) \le 255 \&\& I1(i-t3,j) \le 100 \&I1(i-t3,j) \leI1(i-t3,j)$

```
t3,j+t4)>=200 && I1(i-t4,j+t2)<=255 && I1(i,j-t)>=200 && I1(i,j-t)<=255 &&
I1(i,j+t4) >= 92 \&\& I1(i,j+t4) < 110
       Y(b)=i;
       X(b)=j;
       b=b+1;
        end
        end
    end
end
imshow(I1);
impixelinfo
k=1;
for i=1:1:b-1
  Sum1=0;
Sum2=0;
h=0;
if i = 1
 for j=1:1:b-1
 if abs(X(j)-X(i))<23 && abs(Y(j)-Y(i))<23
   Sum1 = Sum1 + X(j);
   Sum2 = Sum2 + Y(j);
  h=h+1;
 end
 end
 A(k)=Sum1/h;
 B(k)=Sum2/h;
 k=k+1;
end
if i~=1
  r=0:
  for z=1:1:i-1
  if abs(X(i)-X(i-z))>23 || abs(Y(i)-Y(i-z))>23
    r=r+1;
  end
  end
  if r==z
  for j=1:1:b-1
 if abs(X(j)-X(i))<23 && abs(Y(j)-Y(i))<23
   Sum1 = Sum1 + X(j);
   Sum2 = Sum2 + Y(j);
```

```
h=h+1;
end
and
A(k)=Sum1/h;
B(k)=Sum2/h;
k=k+1;
end
end
end
end
hold on
plot (A,B,'*g');
hold off
d = sqrt((A-A1)^2+(B-B1)^2))
```

APPENDIX C

Derivation of Flat Temperature Equation and Calculation of Theoretical Flat Temperature Value for Au-SiO₂ Bilayer devices



Figure 3. Graph of arbitrary temperature vs arbitrary, unrestrained feature length of released pop-up MEMS. As more current is induced the greater the pop-up MEMS reverse direction. The flat temperature is the temperature it takes to have the released pop-up MEMS flatten.

Unconstrained fraction length at deposition temperature is its length on the thermally expanded Si

$$\frac{L_m}{L_D}\Big|_{T_{metal}} = \frac{L_D(T_{metal} - T_0)\alpha_{si} + L_D}{L_D}$$
$$\frac{L_{ox}}{L_D}\Big|_{T_{oxide}} = \frac{L_D(T_{oxide} - T_0)\alpha_{si} + L_D}{L_D}$$

Unconstrained Lengths at room temperature when released

$$L_{0_{ox}} = [L_D(1 + (T_{oxide} - T_0)\alpha_{si})](1 + (T_0 - T_{oxide})\alpha_{ox})$$
(1)

$$L_{0_m} = [L_D(1 + (T_{metal} - T_0)\alpha_{si})](1 + (T_0 - T_{metal})\alpha_m)$$
(2)

At flat Temperature both unconstrained lengths are the same

i.e.,

$$L_{0_{ox}} + L_{0_{ox}} (T_f - T_0) \alpha_{ox} = L_{0_m} + L_{0_m} (T_f - T_0) \alpha_m$$

Deriving the flat temperature, we get

$$T_f = T_0 + \frac{L_{0_{ox}} - L_{0_m}}{L_{0_m} \alpha_m - L_{0_{ox}} \alpha_{ox}}$$

Substituting Equations (1) & (2), we get

 $T_f = T_0 +$

 $\frac{[L_D(1+(T_{oxide}-T_0)\alpha_{si})](1+(T_0-T_{oxide})\alpha_{ox})-[L_D(1+(T_{metal}-T_0)\alpha_{si})](1+(T_0-T_{metal})\alpha_m)}{[L_D(1+(T_{metal}-T_0)\alpha_{si})](1+(T_0-T_{metal})\alpha_m)\alpha_m-[L_D(1+(T_{oxide}-T_0)\alpha_{si})](1+(T_0-T_{oxide})\alpha_{ox})\alpha_{ox}}$

Simplifying the above Equation, we get

$$T_f = T_0 + \frac{\Delta T_{oxide} \left(\alpha_{si} - \alpha_{ox}\right) - \Delta T_{metal} \left(\alpha_{si} - \alpha_m\right)}{\alpha_m - \alpha_{ox}} \tag{3}$$

where,

$$T_0 \rightarrow Temperature where lift off was done ~25^{\circ}C$$

 $L_D \rightarrow Designed Length of the Cantilever$
 $\alpha_m \rightarrow Coefficient of Thermal Expansion of Gold ~14 × 10^{-6}/^{\circ}C$
 $\alpha_{ox} \rightarrow Coefficient of Thermal Expansion of SiO_2 ~0.71 × 10^{-6}/^{\circ}C$
 $\alpha_{si} \rightarrow Coefficient of Thermal Expansion of Silicon ~3 × 10^{-6}/^{\circ}C$
 $T_{oxide} \rightarrow Oxide Deposition Temperature~1000^{\circ}C$
 $T_{metal} \rightarrow Metal Deposition Temperature~200^{\circ}C$

 $T_f \rightarrow Flat Temperature$

$$\Delta T_{oxide} = T_{oxide} - T_0$$

$$\Delta T_{oxide} = 1000 - 25 = 975^{\circ}C$$
$$\Delta T_{metal} = T_{metal} - T_0$$
$$\Delta T_{oxide} = 200 - 25 = 175^{\circ}C$$

Substituting these values in Eq. 3, we get

$$T_f = T_0 + \frac{975(3 \times 10^{-6}/^{\circ}\text{C} - 0.71 \times 10^{-6}/^{\circ}\text{C}) - 175(3 \times 10^{-6}/^{\circ}\text{C} - 14 \times 10^{-6}/^{\circ}\text{C})}{14 \times 10^{-6}/^{\circ}\text{C} - 0.71 \times 10^{-6}/^{\circ}\text{C}}$$

$$T_f = 25 + \frac{975(2.29) - 175(-11)}{13.29}$$

$$T_f = 25 + \frac{2230.46 + 1925}{13.29}$$

$$T_f = 25 + 312.67$$

$$T_f = 337.67^{\circ}C$$

CURRICULUM VITAE

Mohammad Shafquatul Islam, Ph.D. Shumaker Research Building 2210 South Brook Street University of Louisville Louisville, Kentucky, 40208 Email: <u>shafquatul.islam@louisville.edu</u> Phone: 502-807-0079

Education

Ph.D. in Electrical Engineering	December 2023
University of Louisville, Speed School of Engineering, Louisville, KY	
M.Sc. in Embedded Systems Engineering University of Leeds, Leeds, United Kingdom	January 2012
B.Sc. in Electrical and Electronics Engineering American Intl. Univ. – Bangladesh, Dhaka, Bangladesh	March 2009

Core Competencies

- Proficient in MEMS/NEMS fabrication techniques in Class 100 Cleanroom 6 years
- **MEMS/IC Layout Design:** MEMS/Photonic IC Design and Mask Layout using Tanner L-Edit, FEM simulation of Si-based MEMS Designs using ANSYS, COMSOL
- Semiconductor Fabrication Processes: Metal Deposition (Lesker PVD-75, Technics 4604, Oxford PECVD Plasmalab 100), DRIE (SPTS System), Dry Etch (March RIE, Xactix XeF₂ Si Etcher), Contact Photolithography (Suss Mask Aligner MA6/MA8, SU8, Shipley), Wet Processes (RCA wafer clean, HF/BOE etching)
- **Metrology:** Veeco Dektak 8M Profilometer, Zygo NewView 7300 Optical Interferometer, Veeco NT1100 Optical Profilometer
- Scanning Electron Microscopy: TESCAN Vega 3 SEM, Apreo C LoVac FESEM, FEI Nova 600 SEM
- **Programming Languages and Design Tools:** Python, C, SQL, JavaScript, PHP, MATLAB, Tanner L-Edit, Klayout, JMP, Cadence Virtuoso, CoventorWare, PSpice, Multisim

Professional Experience

Hardware Engineering Intern – Displays

Apple Inc., Santa Clara Valley, CA

- Worked as an intern in the MEMS process integration team developing novel MEMS devices for consumer products
- Gained experience working in a Class 10 cleanroom (access enabled by Apple) supporting MEMS process development on 200mm silicon wafers and collaborating with multi-disciplinary teams to develop key technology building blocks and deliver proof of concept vehicles
- Duties included optical and SEM inspections, inline metrology supporting process troubleshooting (white light interferometry, optical reflectometry), submission and interpretation of failure analysis reports, and preparation of SOP documents
- Collaborate with process module teams to tighten inline control and recipe optimization for yield improvement

Chip Engineering Intern

Ciena Corporation, Ottawa, ON, Canada

- Generated and maintained photomask layout designs for InP-based photonic ICs
- Quality control of photomask design process
- Automation of photomask layout design process
- Collaborated with internal design groups and external wafer fabrication foundries

Publications

- 1. Islam, M.S., Challa, S., Wei, D., et al. Investigating flow induced mechanics of strainengineered microcantilevers integrated in a flow-through system. *Microsystem Technologies* (2023), under peer review.
- Islam, M.S., Challa, S., Yacin, M.H. et al. Thermally driven MEMS fiber-grippers. J Micro-Bio Robot 18, 89–100 (2022). <u>https://doi.org/10.1007/s12213-023-00161-w</u>
- M. S. Islam, S. Challa, M. H. Yassin, S. S. Vankayala, J. Beharic and C. K. Harnett, "MEMS Bimorph Fiber-Gripping Actuators," 2022 International Conference on Manipulation, Automation and Robotics at Small Scales (MARSS), Toronto, ON, Canada, 2022, pp. 1-6, https://doi.org/10.1109/MARSS55884.2022.9870247
- D. Wei, S. Challa, M. S. Islam, J. Beharic, C. K. Harnett and D. O. Popa, "Multi-Robot Collaboration for Electronic Textile Fabrication," 2022 International Conference on Manipulation, Automation and Robotics at Small Scales (MARSS), Toronto, ON, Canada, 2022, pp. 1-6, <u>https://doi.org/10.1109/MARSS55884.2022.9870470</u>
- Challa, Sushmita, M. Shafquatul Islam, Danming Wei, Jasmin Beharic, Dan O. Popa, and C. K. Harnett. "Functional Fiber Junctions for Circuit Routing in E-Textiles: Deterministic Alignment of MEMS Layout With Fabric Structure." In *International Manufacturing Science and Engineering Conference*, vol. 85079, p. V002T08A009. American Society of Mechanical Engineers, 2021. <u>https://doi.org/10.1115/MSEC2021-63887</u>

May 2022 – February 2023

February 2023 – August 2023

- Islam, Mohammad S., and Harnett, C. K. "Miniaturized systems for evaluating enzyme activity in polymeric membrane bioreactors." *Engineering in Life Sciences* 19, no. 11 (2019): 749-758. https://doi.org/10.1002/elsc.201900059
- S. Challa, M. S. Islam, and C. K. Harnett, "Fiber-Crawling Microrobots," 2019 International Conference on Manipulation, Automation and Robotics at Small Scales (MARSS), Helsinki, Finland, 2019, pp. 1-6, https://doi.org/10.1109/MARSS.2019.8860986
- Challa, S., Ternival, C., Islam, S., Beharic, J., & Harnett, C. (2019). Transferring Microelectromechanical Devices to Breathable Fabric Carriers with Strain-Engineered Grippers. MRS Advances, 4(23), 1327-1334. <u>https://doi.org/10.1557/adv.2019.6</u>

Conference Presentations

1.	Islam, M. S., Challa, S., Yacin, M.H. et al. "MEMS Bimorph Fiber-	
	Gripping Actuators." International Conference on Manipulation,	Presentation
	Automation and Robotics at Small Scales, Toronto, Canada, July 2022.	
2.	Islam, M. S., Harnett, C.K. "Integrating Bioinspired Strain-Engineered	
	MEMS Actuators with Breathable Fabric Carriers." 1st International	Presentation
	Conference on Nature Inspired Surface Engineering, NJ, USA, June 2019.	
3.	Islam, M. S., Harnett, C. K. "Miniaturized Systems for Evaluating Enzyme	
	Activity in Polymeric Membrane Bioreactors." Super Collider Conference	Poster
	organized by KY NSF EPSCoR, Lexington, KY, April 2019.	
4.	Islam, M. S., Harnett, C.K. "Experimental Study in Optimizing Enzyme-	
	Based Polymeric Membrane Bioreactors." 71st Annual Meeting of the	Presentation
	American Physical Society's Division Presentation of Fluid Dynamics,	
	Atlanta, GA, Nov. 2018.	
5.	Islam, M. S., Harnett, C.K. "Electrochemical Study of Trametes Versicolor	
	Laccase Activity Presentation on Screen-Printed Electrodes." 9th	Presentation
	International Conference on Microtechnologies in and Poster Medicine and	and Poster
	Biology, Monterey, CA, March 2018.	

Patents

• Harnett, C.K., Beharic, J., Ternival, C., Challa, S. and Islam, M.S., University of Louisville Research Foundation ULRF, 2021. *Microgrippers for Transferring Devices to Fibrous Surfaces*. U.S. Patent Application 16/983,405

Honors and Awards

- University of Louisville Doctoral Fellowship Award
 Aug 2016 July 20
- Dissertation Completion Award

Aug 2016 – July 2018 August 2021 – December 2021