A Technique for the Optimization of Actuation Characteristics of Ionic Polymer-Metal Composites

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A Technique for the Optimization of Actuation Characteristics of Ionic Polymer-Metal Composites

by

Vaughn Varma

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A Technique for the Optimization of Actuation Characteristics of Ionic Polymer-Metal Composites

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Abstract

A Technique for the Optimization of Actuation Characteristics of Ionic Polymer-Metal Composites

Vaughn Varma, M.S.
Rochester Institute of Technology, 2018

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Ionic-Polymer-Metal Composites (IPMCs) are a subset of Electroactive Polymers (EAPs), which are an actively-researched class of electromechanical actuator. IPMCs are similar in function to piezoelectric actuators, however require substantially lower input voltage, requiring as little as 1V to actuate, and with a very high maximum no-load strain of over 300%. IPMCs can be built from biocompatible materials, and do not require the use of any permanent magnets, making them suitable for medical applications, or for use in environments subject to strong or fluctuating magnetic fields. IPMCs are additionally soft and flexible, and are suitable for wet environments, further improving their biocompatibility. This, along with their other properties, makes IPMCs ideal for small robotic applications and for biomimetics. However, IPMCs typically exert, maximally, a small fraction of the total force
which can be provided by piezoelectric actuators or other types of EAPs. This study expands on previous efforts to improve IPMC performance by proposing a method for optimizing desired aspects of IPMC performance with respect to any number of input parameters by applying the method of Gradient Descent utilizing the Backtracking Line Search. This method is outlined generally and demonstrated here, showing the process used through most of one iteration to optimize for IPMC blocking force with respect to changes in the amount of platinum used during the primary and secondary plating procedures. The incomplete backtracking line search led to performance comparable with the initial results (within 0.7% of this initial guess, 2.38 [mN] vs 2.37 [mN]), with an IPMC made during the initial gradient estimation exhibiting 30% improvement over this initial guess, 3.13 [mN], indicating that subsequent iterations of the backtracking line search could lead to further improvements in IPMC blocking force. It was additionally found that the sanding process in particular, as a process excluded wholesale from the gradient descent search used, had a relatively large impact on the IPMC blocking force, and thus should be controlled more carefully when continuing or extending the method carried out here.
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Chapter 1

Introduction and Literature Review

1.1 Introduction of Problem

IPMCs offer advantages over many other material actuators due to their low actuation voltage and high strains under actuation (in some cases, beyond 90° deflection in a cantilever configuration)[9]. However, the range of applications for IPMCs is still relatively limited, since the actuator force is low (typically the order of 1-10 mN [22]). Optimization methods have been applied

Figure 1.1: IPMC operating principle (from [17])
to improve the maximal actuator force, and forces up to approx. 0.3 N [16] have been observed, however the forces are still too small to find widespread use. Furthermore, the ratio of output force to input voltage is much less than for more widely-used actuators, such as piezoelectric actuators. Most research into the optimization of IPMC performance to date is centered around varying a few design variables at a time, such as actuator thickness [9] or variations in the properties of a few chemical processes involved in IPMC manufacturing, such as the concentration of a reducing agent [22], with only one or a few different outputs to optimize for a given study, such as blocking force [9] or response time [13]. Several aspects of the performance of IPMCs, such as saturation voltage and steady-state power draw have received relatively little attention, and could be used as metrics in classifying the utility of IPMCs as actuators. This study seeks to build on previous research to expand knowledge related to optimization of IPMC performance.

In order to optimize any performance metric of IPMCs, without a valid analytical model relating that performance metric to a select set of input parameters, a technique must be used which operates totally independently of such a model. Several of these exist and are used in practice for different applications, such as Gradient Descent, Coordinate Descent, Genetic Algorithm, and Simulated Annealing. These methods are most frequently used in simulation, rather than in conjunction with experimentation, and are typically designed under the assumption that it takes a relatively small amount of time to calculate the value of a desired metric from the set of varied input
parameters. IPMCs can, however, take several days each to make, and thus a technique is desired which requires the preparation of a minimal number of samples. This study seeks to utilize such an optimization technique to make it suitable for use in optimizing IPMC performance. To that end, the following was performed:

- Aggregated existing research on the optimization of IPMC performance
  - Selected 2 process parameters to target: the concentrations of platinum salt in the initial reduction and secondary developing processes in IPMC preparation.
  - Selected Gradient Descent as a suitable optimization technique to use
  - Selected blocking force as an appropriate performance metric to optimize

- Demonstrated the chosen optimization technique as applied to improving the selected performance metric over the parameters targeted
  - Fabricated 2 IPMC samples for an initial set of values for the targeted parameters
  - iterated on these values according to the gradient descent technique, using multiple samples for each set of parameter values required for gradient descent
1.2 Literature Review

EAPs have potential as actuators due to their large deformation under small input voltages and have generated significant interest within the last ten years [19]. During this time, new materials were developed which drastically improved the potential for EAPs, specifically IPMCs, for practical use. There is, however, still a substantial amount of improvement necessary to overcome current limitations of EAPs (such as low actuation force and mechanical energy density [19]) before they can see more widespread use. Several previous authors have explored the notion of altering various parameters related to IPMC fabrication for better performance. Labrador [9] found, for example, that doping the actuator in an LiCl solution drastically increased the amount of strain exhibited under actuation. There exists a consensus among authors who have included membrane thickness as a design variable in an optimization study that there is inverse relationship between membrane thickness and maximum deflection [13, 16, 19] as well as between membrane thickness and response time [11, 16]. In addition, it is generally seen that, along with a decrease in maximum deflection, a thicker membrane leads to a higher maximum actuator force [9, 11, 16]. Ruiz [19] found, however, that, at least for the case of a rod-shaped IPMC actuator, the blocking force does not increase in an unbounded fashion with respect to thickness; i.e. there is a finite thickness for which the actuator force is maximum. The result was, however, derived indirectly from a computational model of an IPMC and was not directly verified with a physical IPMC specimen. In addition, it is unclear how the nature of
the relationship between space-charge density (which Ruiz argues is analogous to blocking force) and specimen radius changes, if at all, with respect to other parameters. For example, the thickness yielding maximum blocking force for an IPMC actuator may depend on the length of the specimen. Other re-

![IPMC blocking force vs membrane thickness](image)

**Figure 1.2: IPMC blocking force vs membrane thickness [9]**

searchers have also looked at altering different chemical properties of IPMCs to improve actuator performance. Labrador [9] found, for instance, that doping the Nafion membrane in LiCl before manufacturing the actuator resulted in an increase in maximum deformation of two orders of magnitude vs. non-doped Nafion. Another approach to altering IPMC chemistry is changing the electrode material. Researchers have used everything from Buckypaper [9] to pure platinum [22] to palladium-platinum (Pd-Pt) [16] to gold-sputtered platinum [11] to reduce the resistivity of the IPMCs electrodes (Figure 1.2).
spite of the fact that so many different approaches exist for fabricating IPMC electrodes, it is difficult to directly compare the performance of different materials against one another, as there exists no controlled set of parameters under which a wide variety of materials have been tested. Each study uses IPMCs of a varying sizes, with varying input voltages and with inconsistent manufacturing processes. Along with the chemical composition of the electrodes, different methods have been explored to improve the electrical properties of the electrodes. In general, increasing the amount of electrode material seems to improve actuator force without significantly affecting maximum deflection [22]. Yu et al. [22] found that increasing the concentration of platinum during the manufacturing stage, where an electrode is chemically grown on the Nafion membrane, can allow the electrode to be more thoroughly embedded into the membrane, significantly reducing the overall resistance at the interface between the electrode and the nafion membrane. The decrease in resistance was found to lead to a significantly higher blocking force.

In general, IPMCs seem to perform better when the resistance in each electrode is reduced, and the penetration depth of the electrode is increased. However, researchers have yet to quantify the effects of penetration depth in any meaningful way. One further property of the interface between the membrane and electrode, which seems to be of importance along with electrode penetration depth, is surface roughness of the ionic membrane onto which the electrode is deposited. Some researchers have included surface roughing (normally via sandpaper) as a method to improve actuator performance [16, 22],
but have not included any experimental justification for doing so. Wang et al. [21] specifically observed the effectiveness of surface roughing on actuator performance. Wang et al. highlighted several methods for roughing the nafion membrane (Figure 1.2, and of these selected sandblasting, which introduced an added step in IPMC manufacturing, where residual granules must be chemically rinsed from the membrane before plating. Results from the study demonstrated that using rougher sandblasting powder, over a longer period of time, yields both higher maximum deflection and higher blocking force. Plasma etching, while more costly than sandblasting, could avoid the aforementioned complexity in manufacturing introduced during sandblasting while potentially improving IPMC performance further. Kim et al. [7] found that plasma etching can somewhat improve IPMC performance, however over-treatment can reduce the actuator performance when compared against that of an untreated specimen. Zhang et al. [23] directly compared the effectiveness of plasma etching against sand blasting as a method for improving IPMC performance. Zhang et al. [23] found that, while plasma etching can out-perform sandblasting in both maximum deflection and blocking force of the IPMC, plasma etching also greatly increases the dependence of each of those characteristics on voltage. It is unclear, however, by the work done by Zhang et al., how each of these treatments compares to an untreated specimen or how the altered dependence on voltage behaves over the entire range of useful input values (from minimum voltage for actuation to saturation voltage), as only two input voltages were tested. The authors recommend sandblasting,
due to the increased sensitivity of the output characteristics to changes in the input voltage for plasma treatment. However, if this sensitivity drops off as the voltage approaches the saturation voltage, and plasma etching can provide consistently superior actuator performance for a range of input voltages, then it can still be considered a viable option for improving IPMC performance, for cases in which the input voltage can be held near or within that range during operation.

There has been relatively little research performed to date into the efficacy of using existing optimization methods to optimize various metrics of IPMC performance. Lacking an accurate analytical representation of these metrics as a function of process parameters used when manufacturing the IPMCs, optimization techniques designed for black-box testing are an attractive option, as they explicitly operate without this prior knowledge. Yu et al. [22] sought to improve IPMC performance using Orthogonal Array Testing, a technique traditionally used for fault testing in software [18]. This technique, for a set of discrete inputs, seeks to find the set of inputs which leads to some particular output (i.e. the presence of some fault) without needing to test every single combination of inputs. The technique requires selection of orthogonal combinations of inputs, that is inputs that are both balanced and that are dissimilar from one another. To improve IPMC performance, Yu et al. [22] used a set of three possible values for each of three process parameters. Low, center and high values used were used for the concentration of reducing agent for the growth of platinum onto Nafion, the concentration of platinum salt dur-
ing this process, and the concentration of Tetraethyl Orthosilicate (TEOS), which was mixed into Nafion solution and then cast as such into the shape of a sample. The technique provided large improvements in blocking force (Figure 1.5), and provided some insight into the sensitivity of the blocking force and displacement of IPMCs subject to changes in these parameters, with the platinum salt concentration (Figure 1.2) and TEOS concentration having a substantially larger impact than reducing agent concentration on blocking force and maximum deflection. However, the approach taken by Yu et al. [22] was limited in resolution, as the input variables must be discretized roughly to avoid excessive testing. The technique is thus most suitable for situations where the desired variables to test are already discrete, as techniques designed for continuous variables can often not be applied for discrete variables. Some examples of such variables are type of metal to plate onto IPMC, cation element, and the addition of some extra manufacturing step. However, such a technique designed for continuous variables is desirable where the variables are, in fact, continuous, as useful information can be gained in between adjacent discrete steps.

Lee et al. [10] used a simple form of such a method, and attempted to improve the blocking force of a simulated actuator assembly constructed from multiple IPMCs, although the technique used can also be applied to physical IPMCs as opposed to simulated ones, and can be applied to optimize the blocking force of a single cantilevered IPMC rather than an actuator assembly. Lee et al. [10] simply used an iterative search to find the best value for a
single parameter (IPMC length), given that the other parameters were all fixed. However, this does not translate well to higher-dimensional parameter space, as the desired output parameter may not depend on each input variable independently of one another. Thus, if this technique were extended even to just two parameters to vary, varying one at a time, each time a best value is found for one parameter, another search would need to be performed with the other parameter (Figure 1.2), until some convergence criterion is met for both parameters. The approach could lead to a high number of iterations as the algorithm zig-zags or spirals in on the combination of values which, when combined, produce the best overall result (in this case, the highest blocking force). The generalization of this technique is referred to as Coordinate Ascent, or Descent if it is desired to minimize the output metric.

The technique of Gradient Descent described by Boyd & Vandenberghe [1] serves as an iterative method for optimizing any quantifiable, continuous performance metric resulting from any finite number of continuous input parameters, and avoids some of the issues arising from a more naïve approach when used for higher dimensions. The technique involves finding the gradient of the desired output metric (cost), then iteratively performing a line search in the direction of greatest descent and recalculating the gradient until a convergence criterion is met. The approach is similar to coordinate descent/ascent, but each line search is performed in a direction believed to give the greatest improvement in cost.

Figure 1.8 shows some examples of iterations of the gradient descent
algorithm, and highlights some of the challenges associated with the method. If the cost is much more sensitive to changes in some directions than others, the algorithm will have difficulty converging, and may do so very slowly. In addition, the gradient descent method is a convex optimization technique. Thus, it is only guaranteed to find the true minimum of the cost if the cost is a convex manifold over the set of input parameters, ensuring that any local minimum is also a global minimum. Otherwise, the technique may converge to a local minimum which is not the true global minimum, providing improvement over the initial cost, but not as much improvement as possible.
Figure 1.3: Diagram showing the effects of the addition of a secondary electrode layer (via gold sputtering) on IPMC performance by reducing surface resistivity [11].
Figure 1.4: SEM images of the nafion surface after various roughing [21]

Figure 1.5: Improvements in IPMC blocking force from Orthogonal Array Optimization [22]
Figure 1.6: Scanning Electron Microscope (SEM) images of IPMC electrode penetration resulting from different platinum concentrations during manufacturing [22]
Figure 1.7: Example of the coordinate descent algorithm applied to an elliptical paraboloid [14]

Figure 1.8: Examples of Gradient Descent algorithm for poorly-scaled (left) and well-scaled (right) coordinate frames. Figures 9.14 and 9.15 from [1]
Chapter 2

Background

In order to improve some quantified attribute (e.g. financial cost, efficiency, impact toughness) of a process, device or system lacking a closed-form solution by varying some controllable parameter or set thereof, numerical techniques are often utilized. The most common approaches are variants of the Gradient Descent Algorithm, which is used to iteratively drive some arbitrary cost toward a local minimum with respect to any number of continuous input variables. Because of the generality of this method, it is investigated here in further depth. In addition, because any numerical technique as applied here depends on precision in recorded measurements, a test setup is required which is sensitive to incremental differences in loads of the scale of those supplied by IPMCs (e.g. sufficient to differentiate between samples with 1 [mN] and 1.2 [mN] blocking force); such a test rig is described here based on [2].

2.1 The Gradient Descent Algorithm

The general outline for the gradient descent algorithm is as follows:

1. Decide on a numerical calculation to minimize (for applications where a higher value is desired, e.g. improving the isentropic efficiency of a
2. Begin with some "best guess", i.e. estimate of the optimal input parameters (i.e. the set of input parameters estimated to produce an optimal cost)

3. Estimate the gradient of the cost with respect to the input parameters, evaluated at the current best guess
4. Move the best guess some amount in the direction of the steepest descent of the gradient.

5. Repeat steps 3-4 until some convergence criterion is met, or some maximum number of iterations is reached.

### 2.1.1 Gradient Estimation

Step 3 of the above process is performed in several ways, depending on the application and specific implementation of the gradient descent algorithm. Here, the gradient is approximated by predictably sampling points near the best guess and summing the vectors representing the shifts in input parameters between the best guess and each sample. The vector is then normalized and weighted by the improvement in the cost as follows:

\[ \vec{\nabla} C |_{\vec{x}_i} \approx \frac{1}{n} \sum_{j=1}^{n} \left[ \frac{(C(\vec{x}^*_j) - C(\vec{x}_i)) \ast (\vec{x}^*_j - \vec{x}_i)}{||\vec{x}^*_j - \vec{x}_i||} \right] \] (2.1)

where $C$ is the cost function, $\vec{x}_i$ is a vector of parameter values for the $i_{th}$ best guess, and $\vec{x}^*_j$ is the vector of parameters for the $j_{th}$ sampled point near $\vec{x}_i$. To calculate the euclidian norm $||\vec{x}^*_j - \vec{x}_i||$, there must be equivalence of units across every parameter in $\vec{x}_i$. For applications where the units do not naturally equate, some equivalence factor must be used. For example, if one parameter is temperature, and a second is concentration of a chemical, it could be decided that a change of 1 mmol is ‘equivalent’ to a change of 0.3 °C. Ideally, these should be selected such that a change of one “unit” in any direction from the initial best guess produces a change in the cost of a roughly similar magnitude,
irrespective of which variable or variables are changed.

2.1.2 Selection of Sample Points for Gradient Estimation

Figure 2.2: Possible positions of points to sample for gradient estimation in a 2D input space using n points (left) and n+1 points (right), with the best guess (⋆) already at the minimum cost.

For cases where sampling a single point is time-consuming, such as in the production of IPMC samples, which takes multiple days, it is desirable to sample as few points as possible while still ensuring that there exists sufficient information at each iteration for the gradient descent algorithm. Naïve thinking would indicate that n points in n-dimensional space (i.e. for varying n different parameters to improve cost) should suffice, where each point lies along an axis aligned with the input parameters, but in the numerical case, this may be insufficient, and may prevent the gradient descent algorithm from converging properly. To illustrate this for a simple case, if a current best guess truly lies on a local minimum of a smooth cost function in 2 input dimensions (arbitrarily, x and y) as in Figure 2.1.2, then selecting only 2 samples as de-
scribed would indicate that the cost decreases most steeply in the direction 
(-x, -y), when, in fact, the gradient should be approximately 0 (indicating 
convergence). Thus, it is necessary that the points selected be roughly evenly 
spaced about the best guess. However, if only 2 points are selected for 2 input 
variables, and they are spaced evenly around the best guess, they will be di-
rectly opposite one another, and will provide no information in the transverse 
direction. This can be avoided by adding a single point to sample. As a result, 
for n input variables, at least n+1 points must be sampled to have sufficient 
information to allow the gradient descent algorithm to function properly. The 
problem of placing a minimum number of points about the current best-guess 
thus becomes the problem of placing n+1 points evenly on an n-dimensional 
hypersphere. A convenient method for doing this follows without proof:

1. Select one point a distance of one unit along the first input parameter 
   $(\vec{x}_1^* = [1 \ 0 \ \cdots \ 0]^T)$

2. Assign a value of $-\frac{1}{n-1}$ for the first index of every remaining point (this 
   ensures that the arithmetic mean of the set of n vectors is the zero vector)

3. For the second point, assign the second coordinate a value such that the 
   magnitude of the vector is 1, and all values after the second coordinate 
   are 0 (for the 4D case, where $\vec{x}_1^* = [1 \ 0 \ 0 \ 0]^T$, $\vec{x}_2^* = [-\frac{1}{4} \ \frac{\sqrt{15}}{4} \ 0 \ 0]^T$.

4. For the remaining n-1 points, select the second coordinate as a uniform 
   value such that the arithmetic mean across the second coordinate is 0 
   for the entire set of n+1 vectors
5. Continue this process for all remaining coordinates and vectors. For the last two vectors, there should be two possible values for the final coordinate such that it has a magnitude of 1 (a positive and a negative); these are the values for the last coordinate of each of the last two vectors. For the 4D case, the vectors are thus:

\[
\begin{bmatrix}
1 \\
0 \\
0 \\
0
\end{bmatrix}, \begin{bmatrix}
\frac{1}{4} \\
\sqrt{\frac{5}{48}} \\
\frac{1}{4} \\
\sqrt{\frac{5}{8}}
\end{bmatrix}, \begin{bmatrix}
\frac{1}{4} \\
\sqrt{\frac{5}{48}} \\
\frac{5}{6} \\
0
\end{bmatrix}, \begin{bmatrix}
\frac{1}{4} \\
\sqrt{\frac{5}{48}} \\
\sqrt{\frac{5}{24}} \\
\sqrt{\frac{5}{8}}
\end{bmatrix}
\] (2.2)

6. Using the previously set equivalence factors, convert each value of each vector into a corresponding input parameter with units

7. This set of vectors represents perturbations from the best guess. The entire set may be scaled by a constant factor to ensure the following:

- That the changes in input values are large enough they produce a measurable change (i.e. large enough with respect to all noise resulting from process and measurement uncertainty) in the overall cost, and
- That the perturbations are not so large as to skip over important features of the cost over the input space (e.g. local extrema). Generally speaking, the smaller the perturbations, the closer the approximation is expected to be to the true gradient, but also the more sensitive it becomes to uncertainty and noise.
8. Add to the set of vectors the current best guess, and these are the points to sample at each iteration.

The perturbation vectors do not depend on the current best guess, so these can be precomputed once and used for every iteration, meaning only the last step is necessary to perform at every iteration.

2.1.3 Step Size

Figure 2.3: Diagram showing iterations of the Gradient Descent algorithm with too large (left) and too small (right) of a fixed step [4]

Once the estimate of the gradient has been calculated, the best guess must be moved in the direction of steepest descend (opposite the gradient vector) by some amount. There are several methods for determining the step size to use, such as simply using a fixed scalar multiplied onto the magnitude of the gradient (Figure 2.1.3), but using a fixed step size requires precise tuning to ensure convergence without requiring an excessive number of iterations.
There exist, however, methods which choose a step size adaptively, and one such method is used here, namely the Backtracking Line Search as described by Boyd & Vandenberghe [1]. This method is more robust than a fixed step size to deviations in the scale of the gradient, is relatively simple, and tends to select an appropriate step size without too many iterations (Figure 2.1.3). The Backtracking Line Search is carried out as follows:

- Select parameters $0 < \alpha < 0.5$ and $0 < \beta < 1$

  - $\alpha$ is related to the exit condition for the backtracking line search, and is recommended by Boyd & Vandenberghe [1] to be selected between 0.01 and 0.3 (0.1 used here)

  - $\beta$ relates to how quickly the step size decreases (values closer to 1 decrease more slowly). Boyd & Vandenberghe [1] recommend a value of $\beta$ between 0.1 and 0.8 for crude and more fine searches,
respectively. A slightly more crude search (0.4) is to be used here for the initial line search, to balance the number of iterations required for the line search with the desire to take a suitably large step, which can reduce the number of times the gradient must be estimated, and thus reduce the number of samples required overall.

• begin with a parameter $t = t_0$, and do the following:

1. If the exit condition
   \[
   C(\vec{x}_i - t\vec{V}C(\vec{x}_i)) \leq C(\vec{x}_i) - \alpha t * ||\vec{V}C(\vec{x}_i)||^2
   \]
   (2.3)
   is satisfied (Figure 2.1.3), exit the search with the new best guess as $\vec{x}_i - t\vec{V}C(\vec{x}_i)$

2. Otherwise, update $t := \beta t$ (this requires the production of an additional sample) and repeat.

Figure 2.5: Diagram illustrating the exit condition for the Backtracking Line Search [4].
Figure 2.1.3 provides a visualization of the Backtracking exit condition. The slope of the lower dashed line is the gradient of the cost at t=0, f(x) is the cost along the direction of the gradient, and the upper dashed line is the threshold for the line search. t is updated until the sampled point \( f(x + t\Delta x) \) lies below the upper dashed line.

The entire gradient search is carried out until some exit criterion is reached. There are a few commonly used ways to formulate this exit condition, such as a threshold on the proportion of improvement of cost between one iteration and the next, i.e. \( \frac{\Delta C}{C} \leq \epsilon \), where \( \epsilon \) is a cutoff threshold, such as \( \epsilon = 10^{-4} \). Another common condition is that the gradient have a sufficiently small magnitude, e.g. \( \|\nabla C\| < \epsilon \). These two exit criteria are similar for the gradient descent method, as the step size at any iteration is proportional to the magnitude of the gradient. The exit criterion \( \frac{\Delta C}{C} \leq \epsilon \) may cause the algorithm used here to exit earlier than desired for a given \( \epsilon \), in particular if the \( \beta \) selected for the backtracking search is small and \( \alpha \) is large, as this can cause the line search to take a step which is especially small proportional to the gradient when compared with other steps taken, causing a smooth cost function to have very similar values between iterations of the gradient descent search, leading to undesired termination. The gradient descent search is not carried out for multiple iterations in this study, and thus the selection of an exit criterion was omitted here.
2.2 Testing Equipment

2.2.1 Test Rig

Figure 2.6: Fixture used for testing IPMC blocking force.

For testing the blocking force of the IPMC samples produced, a test rig, shown in Figure 2.2.1 and slightly modified from that described by Chiu [2], was used. The rig utilizes a force sensor with a Nitinol whisker and a pair of
opposing strain gauges affixed to the wired with JB Weld (Figures 2.2.1, 2.2.1) as a voltage divider to measure bend in the wire, and thereby applied force at the tip of the wire. The IPMC specimen was screwed between two pads made of 1/8” graphite gasket, each of which is connected to a wire lead with Nickel-based conductive glue (Figure 2.2.1). The connection was sealed with Amazing Goop all-purpose adhesive to prevent oxidation of exposed copper in the attached wire. The entire assembly was submerged in deionized water.
Figure 2.8: Diagram of force sensor used to measure IPMC blocking force [2] during testing, as seen in 2.2.1, along with the tip of the nitinol wire, which was set at a distance of 1.065 [in] from the top of the graphite pads for all tests performed. The nitinol was angled such that an IPMC sample would not easily lose contact with the wire during testing. In order to allow the bottom of the assembly to be submerged while minimizing the amount of exposed metal kept underwater to reduce rust formation, and to fit the bottom part of the test rig into the available glassware, a new bracket (Appendix A) was designed and 3D printed to replace the lower aluminum bracket in [2] holding the IPMC sample.

2.2.2 Data Acquisition

An NI MyDAQ with a 10-bit Analog-Digital Converter (ADC) was used to capture data. Both the signal pin as well as the voltage source for the voltage divider were measured, and the sensor signal was taken to be the signal pin voltage divided by the source voltage at every time step. The process was done to keep the signal constant with respect to variations in the input voltage. The MyDAQ was also used to control the voltage applied to the IPMC,
although due to the MyDAQs relatively low current limit on the analog output pins, an op-amp circuit with unit gain was used as a buffer (Appendix B), so that the current could be supplied by a secondary power supply rather than the MyDAQ. Fairly severe current-limiting was still observed for higher input voltages and for certain IPMC samples, and so the second channel of the op amp integrated circuit (IC), as well as both channels of six additional identical LM2094N op amps, also with a unit gain, and the outputs were connected to the graphite pad leads in parallel.
Figure 2.10: Layout of sensor showing strain gauge configuration and attachment to the nitinol wire.

Figure 2.11: Layout of sensor showing strain gauge positioning on the nitinol wire.
Chapter 3

Methods

3.1 Sample Production

In order to execute the gradient descent search, several IPMC samples must be prepared and measured. For this study, IPMC samples of size 6cm x 1cm x - were made using a tweaked version of the IPMC cookbook provided by NASA JPL [16], outlined Below. All values are presented per \( cm^2 \) of sample area and all values were multiplied by 6 \( cm^2 \) for the samples used here. The approach used was to:

1. Sand and cut IPMC sample to size from Nafion sheet. Each sample was sanded lightly with 150 grit sandpaper, then vigorously with 800 grit sandpaper until the IPMC took on a matte, minimally translucent appearance. In future studies, this should be replaced, ideally, with a process which can be more precisely controlled, such as sandblasting. Oguro [15] recommends fine glass beads in a dry sandblasting process for 1 \( [s] \) per \( [cm^2] \) of area of the IPMC sample.

2. The sample was cleaned in DI water in an ultrasonic cleaner for 1-3 minutes to remove particles on the sample surface.
3. The sample is set in boiling 2M HCl for 30 minutes

4. The sample is rinsed in DI water, then set in boiling DI water for 30 minutes.

5. The sample is immersed in Pt solution comprised of \( \geq 3 \text{[mg/cm}^2\text{]} \) Platinum salt in \( 2 \text{[mL/cm}^2\text{]} \) DI water

6. After immersing in Pt solution, \( \frac{1}{60} \text{[mL/cm}^2\text{]} \) 10\% Ammonium Hydroxide solution is added to balance the solution’s pH. This solution is left to sit overnight.

7. Sample is placed in 6 \( \text{[mL/cm}^2\text{]} \) stirring water at 40°C

8. Every 30 minutes, 7 times, \( \frac{1}{15} \text{[mL/cm}^2\text{]} \) 5\% Sodium Borohydride is added to the stirring water, gradually increasing the temperature to 60°C at the end of the 7th 30-minute period.

9. \( \frac{2}{3} \text{[mL/cm}^2\text{]} \) Sodium Borohydride is added to the stirring water 30 minutes after the 7th addition in step 8, and the solution is left to stir at 60°C for 90 minutes.

10. The sample is rinsed in DI water and immersed in 0.1M HCl for 1 hour. After this, the specimen was rinsed and allowed to sit in DI water overnight. This step may be skipped, but was included here to allow the samples to be manufactured at more convenient times.
11. The sample is immersed in a stirring solution comprising $4\ \text{mg cm}^{-2}$ Pt salt, $8\ \text{mL cm}^{-2}$ DI water, and $\frac{1}{12} \text{mL cm}^{-2}$ 10% Ammonium Hydroxide, at $40^\circ C$.

12. Every 30 minutes, 8 times, $\frac{1}{5} \text{mL cm}^{-2}$ 5% Hydroxylamine Hydrochloride and $\frac{1}{10} \text{mL cm}^{-2}$ 20% Hydrazine Hydrate were each added to the stirring solution, while gradually increasing the temperature to $60^\circ C$. At the end of the 8th 30-minute period, some of the stirring solution was sampled along with some sodium borohydride. The solution was brought to a boil in a water bath. If black flecks formed, the temperature of the stirring solution was maintained at $60^\circ C$, and more hydroxylamine hydrochloride and hydrazine hydrate were added. Periodic sampling was done to test for the formation of black flecks. Additional hydroxylamine hydrochloride and hydrazine hydrate were added until said flecks no longer formed.

13. The sample was rinsed in DI water and boiled in 0.1M HCl for 30 minutes

14. The sample was allowed to soak in 1.5M LiCl solution at room temperature for 3 days.

15. The edges of the sample were trimmed by hand with a sharp knife to remove platinum from the edges of the sample. Trimming prevented current from routing around the Nafion due to the voltage difference between the faces of the IPMC. Instead, it is sent through the Nafion, inducing mechanical action. The amount of deflection was visibly much larger for a constant input voltage in the same IPMC specimen after
performing this so it was been performed for each subsequent sample. The exact effects thereof are not quantified here. The amount trimmed off was as little as possible while ensuring that no more platinum on the edges of the IPMC was visible (Appendix D). Ideally, this should be performed in some more precise way than by hand with a knife, but trimmings of width 0.008 [in] were achieved in this manner. For the purposes of this study, to reduce the number of separate sample preparation procedures, each sample was additionally cut in half (to make two 1 [cm] x 3 [cm] samples), and each half was tested separately.

The cost function was selected as the negative of the IPMC blocking force, thus maximizing the blocking force. Steps 5 and 11 were selected to vary for the Gradient Descent algorithm, varying the mass of platinum salt during each step while keeping the volume of water fixed. It was desired that sanding also be included as a varying parameter, however due to the unavailability of a suitable sandblaster, this became a difficult parameter to appropriately quantify and vary precisely, and was thus excluded from the gradient descent search. Because the two parameters to be varied have identical units, the conversion factors comparing the units of each parameter to one another is unnecessary. One could be used if it was predicted that the cost would be much more sensitive to changes in one step as compared with the other, but lacking that insight here, this was omitted. Points were sampled at a radius of 2 [mg] from the current best guess, with an "equivalency factor" of the same 2 [mg]. Although this does not serve to equate the units with one another, it
allows that \( ||\vec{x}_j^* - \vec{x}_i|| = 1 \) for all \( j \). The coordinates were ordered with the steps chronologically. Thus, using the method outlined in 2.1.1, the perturbation vectors are as follows:

\[
\delta\vec{x}_1^* = \begin{bmatrix} 2 \\ 0 \end{bmatrix} [mg], \delta\vec{x}_2^* = \begin{bmatrix} -1 \\ \sqrt{3} \end{bmatrix} [mg], \delta\vec{x}_3^* = \begin{bmatrix} -1 \\ -\sqrt{3} \end{bmatrix} [mg] \quad (3.1)
\]

Selecting the initial best guess as 25 mg Pt for both parameters (for an IPMC with area 6 \([cm^2]\)), the points to sample for the gradient approximation are thus:

\[
\vec{x}_1^* = \begin{bmatrix} 27 \\ 25 \end{bmatrix} [mg], \vec{x}_2^* = \begin{bmatrix} 24 \\ 26.73 \end{bmatrix} [mg], \vec{x}_3^* = \begin{bmatrix} 24 \\ 23.27 \end{bmatrix} [mg] \quad (3.2)
\]

IPMC samples were prepared for each of the above, as well as the best guess itself.

### 3.2 Materials and Equipment

The materials used to create the IPMC samples as per Section 3.1 are as follows:

- 2N Hydrochloric Acid (HCl)
- 0.1N HCl
- Nafion 117 from DuPont
- Platinum Salt (Tetraammineplatinum(II) chloride hydrate)
- Hydroxylamine Hydrochloride
• Hydrazine Hydrate 20%
• Sodium Borohydride 5%
• Deionized (DI) water
• Ammonium hydroxide 10%

The equipment used during the manufacturing of IPMC samples is as follows:

• Digital Scale

• Fisher Scientific IsoTemp hot plate/stirrer with closed-loop control (temperature input with resolution 5°C)

• Thermo Scientific Chemical fume hood

• Small pocket knife

• Branson 2510 ultrasonic cleaner

• 800 grit sandpaper

• 150 grit sandpaper

• assorted beakers and test tubes

• Eppendorf 1000 μL pipette and tips

• Aluminum foil

• small magnetic stirring bar
The materials and equipment used in testing the manufactured samples is as follows:

- 7x LM2904N dual-channel digital op-amp
- 4x 10kΩ resistors, 5% tolerance
- Omega TrueRMS SuperMeter multimeter
- MPJA 14601PS DC power supply
- Shenzhen Mastech HY3003-3 dual-channel DC power supply
- Weller General-Duty Soldering Iron
- Assorted header pins and wires
- National Instruments MyDAQ module with LabView 2014

### 3.3 Sensor Calibration

To calibrate the sensor, it was removed from the test rig and clamped to a table as shown in Figure 3.1. In Figure 3.1, a loop string was affixed to the end of the nitinol wire with a small dab of glue. Weights of various masses (0.05 [g] to 1.2 [g]) were cut from scrap wire to hang on this loop, and were massed via a digital scale. While the sensor data was being collected, the setup was covered with a plastic box jutting over the edge of the table, as shown in Figure 3.2, to prevent drafts in the room from impacting sensor readings. A LabView script was used which sampled the voltages of the sensor signal and
supply voltage pins at 1 [kHz] for 10 [s]. The script displayed a graph of the set of measurements (as $\frac{V_s}{V_i}$, i.e. $\frac{V_{signal}}{V_{supply}}$) for visual verification, and reported the arithmetic mean of the sensor signals. All loads were measured as the ratio of these signals, during calibration as well as data acquisition. This helps prevent noise in the input voltage as well as changes from touching the power supply’s voltage knobs from impacting the sensor reading.

The sensor was measured with various combinations of weights hanging from it, beginning and ending with none, and the results were collected in an Excel spreadsheet. It was found that the sensor did not initially produce meaningful results. As shown in Figure 3.3, there is a cluster of relatively unordered data points with no clear trend. This occurred during initial loading and unloading of the sensor and tended to drive the sensor readings toward a more steady value. It is believed this is due to some minor slippage in the
Figure 3.2: Calibration sensor setup showing transparent box used to protect setup from drafts

sensor, and the data taken before the readings settled to a consistent no-load value were discarded. Because the order in which the data were collected was preserved, and the slippage occurred only near the beginning of the calibration procedure, the rejected data were selected as those data having been recorded on or before the last clear outlier, as determined visually. A linear regression was performed on the remaining data (Figure 3.4), and the slope was used to interpret IMPC signal data as a force. Signal values were converted to [N]
Figure 3.3: Sensor Calibration Data (incl. discarded data). Applied loads given in grams-force ([gf])

from [gf] and the slope of the curve was found to be 54.4 [N]. The sensor was only calibrated in one direction, as all samples can be set up to press into the nitinol wire from the same direction. Testing in a single direction is also performed to prevent the sensor from continuing to slip after settling initially.

It was noted that, if the nitinol whisker was deflected too much (≥ 2 [cm] at the tip) during setup or during manual adjustment of the IPMC setup, the zero-weight reading may change significantly and unpredictably. Upon further investigation, this appears to have nonnegligible dynamic behavior (Figure 3.5). It is believed that this is due to the nitinol wire slipping against the two metal set screws keeping it fixed, changing the angle of and thus
Figure 3.4: Sensor Calibration Data with Linear Fit

bend in the nitinol wire. Ideally, the nitinol wire should be clamped in such a way that it cannot slip (e.g. by epoxy). In this study, to allow the nitinol wire to be removed or adjusted if necessary, the sensor was left with just set screws fixing the nitinol wire in place. To prevent the slippage from altering data, the weights were added and removed carefully, and the sensor was tested with no weights both before and after all other sensor readings to ensure this value had not changed during testing. During testing of the IPMCs, this dynamic sensor behavior was avoided in a similar way, by not disturbing the sensor during testing, and by testing loading conditions in the increasing and decreasing directions, to and from 0 [V]. Initially, during testing, to prevent the
slippage from altering test readings, the test procedure was performed without recording data, until the LabView display showed no signs of transient effects in the sensor readings outside of those expected, i.e. when the data look like Figure 3.7 as opposed to Figure 3.6. Later, post processing of the data was performed to correct for this slipping (Section 3.4.1) thereby reducing the number of tests necessary to gather the required data. The apparent square wave in the data is an expected feature, and is explained in detail in the following section.
3.4 Sample Testing

A LabView script was developed to automate the test process (Appendix C). The script applies voltage in 0.1 [V] increments to the IPMC, up to 3.3 [V], then back down to 0 [V]. Above some voltage (2.5 [V] for most samples tested), oxidation began at the interface between the graphite pads and the IPMC sample, and, as some current is diverted to the oxidation process, and lacking a way to accurately measure the voltage drop through the graphite pads and through the interface with the IPMCs resulting from this, test data was truncated such that data from input voltages exceeding this threshold were not considered.
3.4.1 Data Collection and Processing

Each data point was taken as the arithmetic mean of the data beginning at some offset from the time when the voltage was first applied, to allow the dynamics of the IPMC/sensor combination to settle to approximately steady-state condition, and the time when the input voltage shifts again. A good value for the offset was determined empirically, for the IPMC samples tested here, to be 15 [s]. A smaller value may be sufficient, but this left 45 [s] of data at a sampling rate of 1000 [Hz] for each data point, which is a substantial amount of data, and which leaves acceptably small error bars as discussed in Section 4.3.

It was noticed during testing that, after loading the specimen from 0
[V] to 3 [V] and back, the force did not return to its original value. However, unlike during the sensor calibration, the deflections of the tip of the nitinol remained well under 1 [cm] during testing, and this amount was consistent between tests. It was discovered that creating a short circuit across the IPMC leads, only after removing any applied voltage, caused dynamic behavior in the IPMC (the opposite direction as when applying the voltage initially, indicating relaxation in the specimen), even if the IPMC had been provided with a 0 [V] difference between its faces prior to shorting the actuator. A similar phenomenon was observed by Kim & Kim [8], that IPMCs retain some displacement, and thus, due to the electromechanical action involved in IPMC displacement, some voltage, even after the IPMC is supplied with a 0V difference across its two faces (Figure 3.4.1). The hysteresis was circumvented
Figure 3.9: Hysteresis observed in IPMC displacement as a function of input voltage [8]

during the automated testing by applying a -3 [V] signal to the IPMC in between each 0.1 [V] increment/decrement. This was not predicted to have precisely the same effect as short circuiting, however it ensured that every test was performed in the “voltage increasing” direction, and could thus be compared against one another. With these changes, the overall testing procedure for a single prepared IPMC specimen was as follows:

1. Mount the IPMC specimen in the designated fixture (Figure 2.2.1) by screwing the graphite pads together with the IPMC in contact with the last 1 [mm] of the nitinol wire

2. Ensure all electrical connections are wired properly, with no visible short circuits and with all connections attached

3. Turn on power supplies ($\geq 5$ [V] for the Op Amp array input power, 5 [V] for sensor input voltage)
4. Run test procedure in LabView

- Step from an input voltage $V_{ipmc}$ of 0 [V] up to 3.3 [V] in 0.1 [V] increments, holding at each value for 60 [s] (to allow setup to approach steady-state) and with -3 [V] applied for 30 [s] before each 60 [s] interval (to ensure all measurements are in increasing direction).

- Decrement the applied voltage by 0.1 [V] from 3.3 [V] down to 0 [V], still with the 30 [s] sections of -3 [V] before each 60 [s] section.

After applying the -3 [V] signal, however, large abnormalities in the data persisted. It was found that the sensor data was still occasionally slipping (Figure 3.4.1) by enough to clearly disrupt the blocking force data. To correct for this, the difference between each recorded data point and the sensor value associated with the previous -3 [V] section was taken, and the values were subsequently shifted by a constant value such that the arithmetic mean of the first and last data points (each at an input voltage of 0 [V]) lies at 0 [N] applied force. The processing made the final data robust to shifts in the sensor data occurring outside of regions of data used to produce each data point. The correction does not ensure that all sources of inaccuracy of recorded data are removed, but regularized the final data substantially, and allowed for a more reasonable comparison between IPMC specimens. All of the processing on the data was performed using a script written in Matlab R2017a (Appendix E).
Figure 3.10: Transient shift in data baseline leads to what appears to be a large amount of hysteresis, which can be corrected by post-processing data
Chapter 4
Results and Discussion

In order to reduce the effect of random uncertainty in the preparation process on the final results, it is desired to have multiple samples to compare against one another. However, due to the time taken to manufacture a sample, it can be difficult to produce these samples in a timely fashion. As a compromise, a single sample was prepared and split in two, leaving two samples to measure for each set of variables while still only requiring a single manufacturing process for each pair. Overall, an initial guess was prepared, as well as 3 pairs of nearby samples to calculate the gradient, and a single pair of samples for the first iteration of the backtracking line search. Some differences in appearance during the sample preparation process were observed from the expectation outlined in [15], such as the earlier-than-expected formation of a dull metallic layer in every sample produced; additionally, the samples cut from the same IPMC sometimes varied significantly in blocking force, in spite of the uniformity of the processes affecting each half of the original IPMC. The execution of the gradient descent algorithm was carried out mostly as expected, with the caveat that it was not anticipated that an increase in platinum salt concentration should lead to much of a decrease in blocking force from any initial amount of salt, which is one of the results implied from the
data collected.

4.1 Sample Production

Figure 4.1: Appearance of IPMC during step 5 of preparation (initial reduction process); this differs from the description provided in [15]

When preparing IPMC samples, it was expected that the initial reduction process would leave a black layer of platinum particles as described in Step 3 of [15], and a metallic sheen was not expected until the secondary plating process (step 4 of [15], step 11 in Section 3.1), however it was frequently observed that the IPMC took on a metallic sheen before finishing with the initial reduction process. Figure 4.1 shows this, with the gray portion on the right overtaking the black portion on the left over the course of the reduction process. More platinum is used in each step than the minimum recommended
in [15], but additionally, during the preparation of the first few samples, it was found that inadequate sanding caused this process to occur more quickly, indicating that some more effective way of sanding to increase the effective surface area of the IPMC (e.g. sandblasting) could cause a closer correlation with the results of [15].

Figure 4.2: Appearance of aluminum foil lid after step 11 of IPMC preparation (secondary plating process)

For the longer processes during sample preparation, aluminum foil was used to fashion coverings for the glassware used to limit evaporation of the solution, which otherwise sits uncovered, on heat (up to 60°C), for several hours at a time. It was noted that after the secondary platinum developing process, the aluminum foil changed in appearance, developing a thin black layer on the surface exposed to the evaporated vapors of the stirring solution (Figure 4.1), which condensed on the aluminum foil. It is possible that this black layer is platinum deposited from salt carried with the evaporated water, along with
the weak reducing agent, which condensed onto the aluminum foil. This might indicate that a nonmetallic cover should be used, to ensure that the IPMC is the only surface onto which the platinum in solution will develop, however since an aluminum cover was used during initial sample preparation, one was used for every sample made, to ensure comparability between all prepared samples.

4.2 Results and Interpretation

\[
\begin{array}{cccc}
\bar{x}_1 & \bar{x}_2^* & \bar{x}_3^* & \bar{x}_4^* \\
25 & 27 & 24 & 24 \\
25 & 25 & 26.7 & 23.3 \\
\end{array}
\]

Table 4.1: A list of IPMC samples prepared for gradient estimation (two 1 [cm] x 3 [cm] samples for each pair of values), from Section 3.1. The first coordinate is mass of platinum salt in step 5 (initial electrode reduction step) of sample preparation, and the second coordinate is the mass of platinum salt in step 11 (secondary electrode developing process), for the full 1 [cm] x 6 [cm] sample manufactured.

\[
\begin{array}{cccc}
\bar{F}_{block} & \bar{x}_1^* & \bar{x}_2^* & \bar{x}_3^* \\
2.26 & 3.41 & 1.35 & 1.18 \\
2.48 & 2.84 & 0.94 & 0.53 \\
\end{array}
\]

Table 4.2: Blocking forces (in [mN]) of each half of each prepared specimen

After the data was processed as outlined in Section 3.4.1, the values at each voltage increment in the increasing and decreasing directions were averaged together, and the highest of these was recorded in Table 4.2 for each
3 cm x 1 cm specimen prepared. The average of these values was used to calculate the gradient as follows:

$$\vec{\nabla} F|_{\vec{x} = \vec{x}_1} \approx \frac{1}{3} \sum_{j=1}^{3} \left[ (F(\vec{x}_j^\ast) - F(\vec{x}_1)) \ast \delta \vec{x}_j^\ast \right] = \begin{bmatrix} 0.708 \times 10^{-3} \\ 0.0829 \end{bmatrix} [mN] = \begin{bmatrix} 1.42 \\ 0.166 \end{bmatrix} \left[ \frac{N}{g} \right]$$

(4.1)

where $\delta \vec{x}_j^\ast$ is unit-normalized using the conversion factors described in Section 2.1.1, giving it a magnitude of 1 [-] for all j. This result indicates that, for the IPMC preparation process described in Section 3.1 for 1 [cm] x 6 [cm] samples, using 25 [mg] of Pt each for steps 5 and 11, the blocking force is much more sensitive to the addition of platinum in the initial reduction process (step 5) than in step 11. This could be related to the formation of a metallic sheen in the initial reduction phase, and, if it is the case that the sensitivity of blocking force to changes in platinum concentration in the secondary plating process is itself dependent on the amount of sanding the IPMC receives, this only serves to further demonstrate the need for an optimization technique which can account for multiple interdependent factors, such as the gradient descent method used here.

Since the Cost $C(x)$ is taken to be the negative of the blocking force for the gradient descent algorithm, the behavior of the algorithm is that of the analogous gradient ascent with the blocking force as the objective function.

A value of $t_0 = 5[N^{-1}]$ was selected to ensure the first sample in the line search is sufficiently far from the initial best guess to reduce the number of times the gradient must be estimated. This means the first condition to
Table 4.3: Blocking forces (in [mN]) of each half of the initial best guess \( \vec{x}_1 \) and the first iteration of the backtracking search \( \vec{x}_2 \)

<table>
<thead>
<tr>
<th>( F_{\text{block}} )</th>
<th>( \vec{x}_1 )</th>
<th>( \vec{x}_2 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.26</td>
<td>2.44</td>
<td></td>
</tr>
<tr>
<td>2.48</td>
<td>2.33</td>
<td></td>
</tr>
</tbody>
</table>

check in the line search (from Equation 2.3) is

\[
C(\vec{x}_1 - 5\vec{\nabla}C(\vec{x}_1)) \leq C(\vec{x}_1) - 0.5 \times ||\vec{\nabla}C(\vec{x}_1)||^2
\]  

This leaves the first point to be sampled for the line search at

\[
x = \vec{x}_1 - 5\vec{\nabla}C(\vec{x}_1)
\]

\[
= \begin{bmatrix} \frac{25}{25} \ [mg] + 5 \times \begin{bmatrix} 1.42 \ [mg] \\ 0.166 \ [mg] \end{bmatrix} \end{bmatrix}
= \begin{bmatrix} 32.1 \ [mg] \\ 25.8 \ [mg] \end{bmatrix}
\]  

One pair of 1 [cm] x 3 [cm] (one 1 [cm] x 6 [cm] Nafion strip) IPMCs were manufactured with these amounts of platinum, and the maximum blocking forces of each are collected in Table 4.2. The average of these (2.38 [mN]) does not satisfy the line search termination criterion, as

\[
C(\vec{x}_2) > C(\vec{x}_1) - 0.5 \times ||\vec{\nabla}C(\vec{x}_1)||^2
\]

\[
-2.38[mN] > -3.39[mN]
\]

Although it appears that this method has led the search in a direction with little benefit (as the blocking forces of this and for \( \vec{x}_1 \) are similar to one another), however circumstances like this are accounted for with the backtracking line search; at the least, a single point was sampled in a very similar direction (\( \vec{x}_1^* \))

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but closer to $\tilde{x}_1$ with substantially better blocking force, and the backtracking search will walk back toward $\tilde{x}_1$ to find a suitable point. Thus, the next step of the gradient descent algorithm is to continue with another iteration of the backtracking line search, with $t = \beta t_0 = 2$, now checking the criterion

$$C(\tilde{x}_1 - 2\tilde{\nabla}C(\tilde{x}_1)) \leq C(\tilde{x}_1) - 0.2 \times ||\tilde{\nabla}C(\tilde{x}_1)||^2 \quad (4.5)$$

One interesting result of this, however, is the implication of the existance of a local minimum between the initial guess and first iteration of the line search. Although nothing precludes this from occurring, conventional reasoning indicates that an increase in platinum salt concentration consequently increases conductivity on the surface of the IPMC, while also affecting the IPMC thickness and overall stiffness minimally, thus increasing the maximum deflection and blocking force at the tip of the IPMC. However, barring some unexpected perturbation to the data forcing both samples of $\tilde{x}_1^*$ to be recorded with an abnormally high blocking force, or $\tilde{x}_2$ with an abnormally low blocking force (both relative to $\tilde{x}_1$), that is what this data indicates: that by increasing the platinum salt concentration primarily in step 5, the IPMC samples exhibit the expected increase in blocking force, then, somewhere, the minimum is reached and the blocking force declines thereafter, such that by $\tilde{x}_2^*$ with $t = [5N^{-1}]$, the blocking force is approximately identical to that at $\tilde{x}_1$. As can be seen in Table 4.2, some values (esp. $\tilde{x}_3^*$) have a fairly large disparity in blocking force between the two samples. These samples were produced from the same strip of Nafion, as a single unit, so it is somewhat surprising to see blocking forces this disparate. The sanding process of the Nafion sheet, however, has

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relatively poor control over uniformity, as it is performed here by hand and verified visually. It is noted in [21] that the blocking force is fairly sensitive to changes to the sanding process, and thus it would likely be advantageous to implement this process in a more predictable, uniform fashion. This would additionally provide a way to include sanding in the gradient descent search, for instance by varying grain size and time in a sandblaster; given the sensitivity of the desired metric to improve, blocking force, to sanding, this could allow for much greater improvement overall in IPMC blocking force than is possible by only varying the concentrations of platinum as performed here.

4.3 Measurement Uncertainty

One interesting finding from the data gathered is that some of the apparent uncertainty or noise in the blocking force values is not captured in the sensor noise. The error bars in Figure 4.3 are small enough to indicate that, for the amount of data averaged and the noise in that data for each input voltage, the blocking force values indicated should be more precise than they apparently are. None of the data points sharing an input voltage, for instance, have overlap in their error bars, indicating that the sensor noise represented by the shown error bars is possibly not indicative of all measurement uncertainty. This is possibly due to a change in the contact location or angle between the IPMC tip and the sensor whisker, as the specimen is not affixed to the sensor, but merely rests against it. Given the sensitivity of the sensor, and the scale of forces being measured, these changes could be imperceptible to
the eye and still produce this behavior. That said, the data still do form a relatively clear underlying curve from which information can be drawn, but further characterization of the utilized sensor device as it interacts with IPMCs is required to generate a better model for uncertainty in force readings.

Figure 4.3: Sample Test Data, with error bars representing the 95% Confidence Interval for the sensor readings
Chapter 5

Conclusion and Future Work

5.1 Conclusion

Based on prior research, platinum salt mass in each plating process was targeted as the set of parameters to vary, and the gradient descent technique with a backtracking line search was selected as an iterative optimization method for improving IPMC blocking force. This process has been generally outlined to optimize arbitrary performance metrics of IPMCs with respect to any number of relevant, controllable input parameters, and was partially demonstrated for the inputs of platinum salt mass and output metric of blocking force. This method, if carried out through several iterations, could serve to establish precise process parameters to prepare IPMCs with optimal (locally, at least) performance, thus improving their overall viability as actuators. This leaves some work for future researchers to carry out in order to see the optimization of IPMC actuation characteristics fully come to fruition.

5.2 Future Work

The process outlined here for optimizing IPMC performance was carried out for most of one iteration, for two input variables, and could just as easily
be performed with some other performance metric in mind (e.g. maximum deflection, peak power transfer, manufacturing cost, etc., or some arbitrary combination thereof), with some larger set of process parameters (e.g. including concentration of a secondary plating metal such as used in [16, 20]), and could be carried through several iterations to actually find the sets of process parameters which lead to (locally) optimal performance with respect to the desired metric. For several reasons mentioned in Chapter 4.2, it is particularly desirable to better control the sanding process, and to include this in the gradient descent method.

In addition to future work regarding the IPMCs, a better characterization of the force sensor used here is desired. There were several artifacts in the data with unknown origin, and these needed to be circumvented here, but they should be ideally studied more closely to better understand how to avoid them, as well as to understand their effects on the resulting sensor signal.
Appendices
Bracket designed for modifying test rig for water immersion. All shown measurements in [in]
Parallel Op Amp array for driving additional current into IPMC samples
Appendix C

Figure C.1: Script used to sample sensor and drive IPMC specimen, 1/2

The shown portions of the LabView code connect to one another left to right, with distinct nodes labelled separate numbers 1-5.
Figure C.2: Script used to sample sensor and drive IPMC specimen, 2/2

Figure C.3: Test Script Front Panel
Figure D.1: Early completed IPMC sample (left) and later sample (right). Boiling longer in 2N hydrochloric acid and additional sanding reduced the amount of surface area of the IPMC covered by the visible dark splotches.
Figure D.2: Untrimmed specimen edge (left) compared against trimmed edge (right)
Appendix E

close all
calfac=54.4; % [N]; converts measurements to force, determined separately during calibration

n=0;
files={'5_2nd_half'};%File or Files to be read
out=[];%measurements at 3V for gradient approximation
for file=files
  n=n+1;
  A=importdata(['automated test data/'] file{:},'\t'); %relative path for test data
  %Data structured as: [ time, input voltage, unfiltered sensor data]
  t=A(:,1);
  unfiltered=A(:,3);
  in=A(:,2);
  I=find(in(1:end-1)-in(2:end))+1;%indices where input voltage changes
  I=[1;I];
  x=[];%input voltage
  y=[];%IPMC force
  s=[];%error bars for y
  neg_offs=[];%the IPMC values at -3V, for shifting y
  offset=15/(t(2)-t(1));%amount of data to ignore as IPMC settles
  for i=1:numel(I)
    i1=I(i);
    if i1 == I(end)
      i2=numel(t);
    else
      i2=I(i+1)-1;
    end

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if in(i1)>=0 %Does not add data to y for sections where −3V are applied to IPMC
   if (i1~numel(t))
      x=[x in(i1)];
      y=[y mean(unfiltered(i1+offset:i2))];% arithmetic mean of region of interest
      s=[s std (unfiltered(i1+offset:i2))*1.96/
         sqrt(i2−i1−offset)];%sample standard deviation, 95% confidence
   end
   elseif in(i1)<0
      if (i1~numel(t))
         neg_offs=[neg_offs mean(unfiltered(i1+
            offset:i2))];
      end
   end
end
y=y;%keep copy of unshifted data for comparison
for i=2:numel(y)
    y(i)=y(i)+neg_offs(1)−neg_offs(i);%shift y values
end
y=y−(y(end−1)+y(1))/2;%shift y values such that 0 [V ] → 0 [N]
y=y*calfac;%convert to [N] from [−]
y=y−(y_(end−1)+y_(1))/2;
y=y−(y(1));
end
plot(x,y);
hold on;%plot curve from each file on same graph
out=[out mean(y(x==3))];%Value for gradient estimation
Matlab code written to post-process and interpret sensor readings from automated tests

```matlab
end
legend(files{:});
xlabel('Input Voltage V_{ipmc} [V]');
ylabel('IPMC Force [N]');

Matlab code to display filtered signal time history.

```
Bibliography


