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Mechanical Properties and MEMS Applications of Carbon-Infiltrated Carbon Nanotube Forests

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Mechanical Properties and MEMS Applications

of Carbon-Infiltrated Carbon

Nanotube Forests

Walter C. Fazio

A thesis submitted to the faculty of
Brigham Young University
in partial fulfillment of the requirements for the degree of

Master of Science

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ABSTRACT

Mechanical Properties and MEMS Applications of Carbon-Infiltrated Carbon Nanotube Forests

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Master of Science

This work explores the use of carbon-infiltrated carbon nanotube (CI-CNT) forests as a material for fabricating compliant MEMS devices. The impacts of iron catalyst layer thickness and carbon infiltration time are examined. An iron layer of 7nm or 10nm with an infiltration time of 30 minutes produces CI-CNT best suited for compliant applications. Average maximum strains of 2% and 2.48% were observed for these parameters. The corresponding elastic moduli were 5.4 GPa and 4.1 GPa, respectively. A direct comparison of similar geometry suggested CI-CNT is 80% more flexible than single-crystal silicon. A torsional testing procedure provided an initial shear modulus of about 5 GPa for the 7-nm, 30-min CI-CNT. The strain and elastic modulus values were used to design numerous functional devices which were then fabricated in CI-CNT. A series of compliant cell restraint mechanisms were developed, assessed, and revised. A passive restraint with no moving parts was found to be both the most effective design and the easiest design to produce economically. A refined version of the passive restraint has been released commercially. Another series of designed devices successfully demonstrates the implementation of CI-CNT LEM designs.

Keywords: Carbon nanotubes, CNTs, compliant mechanisms, microelectromechanical systems, MEMS, material properties, modulus, tensile strength, microbiology, cell restraints, torsion, LEMs, lamina emergent mechanisms, design, Walter C. Fazio.
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CHAPTER 1. INTRODUCTION

1.1 Background

1.1.1 Microelectromechanical Systems

Microelectromechanical systems (MEMS) are microscopic devices that undergo some form of mechanical or electromechanical motion. Sharing their principal fabrication methods with computer microchips, the first micromachined devices (pressure sensors) appeared in the late 1960’s, with MEMS development beginning in earnest in the 1980’s, leading to commercial applications for MEMS by the end of the 20th century [1]. In the 21st century, MEMS technology has seen widespread commercial use in such forms as micromirrors for digital projection systems [2]; crash sensors to more reliably deploy vehicle airbags; inkjet printer heads; and smartphone sensors such as microphones, accelerometers, magnetometers, and gyroscopes. Silicon is by far the most common MEMS material. Its favorable mechanical properties have been well understood for years, and it also benefits from being readily available (and thus relatively inexpensive) and permitting easy interfacing with existing microchip technologies [3].

Two general process categories dominate MEMS fabrication: surface micromachining and bulk micromachining. Surface micromachining involves the patterned deposition of one or more layers of material onto a substrate. The process includes sacrificial layers that are etched away to create a device that can move. With layer thicknesses rarely exceeding a dozen microns, devices produced through surface micromachining typically have very low aspect ratios.

Bulk micromachining consists of the patterned shaping of the substrate material itself. This permits the creation of MEMS with much thicker layers and higher aspect ratios, since the substrate can be very deep. A common method for bulk micromachining single-crystal silicon is chemical
etching, which allows a designer to take advantage of the crystalline structure to achieve a desired shape. A more versatile method that can produce vertical sidewalls regardless of crystalline structure is deep reactive-ion etching (DRIE). Complex patterns hundreds of microns thick and with very high aspect ratios are possible with this method. Another high-aspect-ratio technique is LIGA (a German acronym meaning “lithography, electroplating, and molding”), which uses high-energy x-rays to produce highly precise, vertical-sidewalled patterns from very thick photoresists. This process is very expensive, however, due to the need for a source of high-energy x-rays [4, 5].

1.1.2 Compliant Mechanisms in MEMS

Compliant mechanisms, which depend on flexible members to achieve motion rather than rigid members with joints, have seen significant use in MEMS for many years [4]. Employing compliant mechanisms on the micro-scale is beneficial for a number of reasons, including their ability to simplify manufacturing and achieve motion with little or no friction [6, 7]. This has led to the development of both compliant MEMS actuators [8–10] and sensors [11, 12]. A survey of texts on MEMS (for example, [13]) reveals that nearly all MEMS systems achieve motion using one or more compliant elements.

An important design limitation arises where compliance is used, however. Since such devices operate using flexible members, the degree to which their constituent material can deflect is a critical design parameter. When using compliant elements, material selection is crucial, since the devices must be designed around their material’s maximum stress and strain. Unfortunately, conventional MEMS fabrication methods are limited to a handful of materials, most of which are ill-suited for compliant mechanism design when compared to common macro-scale compliant materials (especially polymers). For example, the maximum strain of polypropylene—frequently used in commercial products for living hinges and other compliant mechanisms—approaches 2.5% before yielding [14] and, if allowed to deform plastically, can exceed 10-20% in some applications [15]. In contrast, an acceptable value for polysilicon—one of the most common MEMS materials—is approximately 0.7% [13]. This places a notable burden on MEMS designers as they attempt to achieve a specific motion with the limited deflections permitted. The result is often a very large mechanism relative to the motions it is designed to experience.
1.1.3 Carbon Nanotube-Templated Microfabrication

Recently, research at Brigham Young University has led to a new method for fabricating microsystems, including compliant MEMS, using carbon nanotubes (CNTs) [16, 17]. Carbon nanotube-templated microfabrication (CNT-M) is achieved by applying a desired pattern to a silicon wafer using photolithography, depositing a growth catalyst using thermal evaporation, and growing a forest of vertically-aligned CNTs atop that pattern. The empty space between nanotubes is then filled in, or infiltrated, with a desired material using chemical vapor deposition (CVD) to form a solid structure. Figure 1.1 illustrates the fabrication process. Because the nanotubes occupy so little volume compared to the space around them, the properties of the resulting structure are determined largely by the infiltration material.

The CNT-M process theoretically permits a very wide range of deposition materials to be used; for example, [16] focused on depositing both silicon and silicon nitride, and [17] focused on creating silicon oxide structures. The process also allows the creation of structures with very high aspect ratios, where features as narrow as ten microns can be grown to hundreds of microns in height during fabrication.

Preliminary research at BYU suggested that a CNT structure infiltrated with carbon could undergo a much greater deflection before failure than other MEMS materials, including silicon. In addition, the material appeared to be purely elastic, with no plastic deformation or stress relaxation when loaded. Thus, CNTs infiltrated with carbon appeared to have very favorable characteristics for compliant MEMS. The material will be referred to in this thesis as carbon-infiltrated carbon nanotubes (CI-CNT).

1.2 Thesis Objectives

The overall purpose of the work described in this thesis was to examine the viability of CI-CNT as a compliant MEMS material and verify whether it did indeed possess superior properties compared to existing options. The specific objectives of the work were threefold. First, the key material properties for compliance—that is, elastic modulus and maximum strain—were to be determined. Additionally, since the quality of both uninfiltrated and infiltrated CNT structures
Figure 1.1: The CNT-M process, in this case done with carbon. Photoresist is spun on and developed to remain where CNT growth is not desired (a). Alumina is deposited (b), followed by iron (c), which serves as the CNT growth catalyst. The final pattern for CNT growth is formed by removing the remaining photoresist via lift-off (d). A forest of vertically-aligned CNTs is then grown (e) and infiltrated with carbon (f) in a furnace, after which the sample is cooled and removed.
were found to be influenced by several fabrication parameters [18], the effects of varying two key parameters—growth catalyst layer thickness and duration of infiltration—were also investigated.

The second objective was to demonstrate the use of CI-CNT in the fabrication of compliant MEMS devices using the measured material properties to guide the design process. The third objective, closely related to the second, was to explore new MEMS designs made possible by using CI-CNT as opposed to another MEMS material.

1.3 Thesis Outline

This chapter has provided a brief overview of MEMS and the use of compliance in their design, has introduced the CNT-M process developed at BYU, and has explained the objectives of this research regarding CI-CNT produced through CNT-M. Chapter 2, based on a conference paper presented by the author at the IMECE 2011 conference, discusses the investigation of the material properties of CI-CNT described in the first objective above. Chapter 3 compares the in-plane compliance of CI-CNT with that of single-crystal silicon using the deflection of similar cantilever beams as the basis for comparison. An initial exploration of CI-CNT torsional properties and behavior is given in Chapter 4.

Chapter 5 presents the development of several compliant CI-CNT cell restraints designed using the material properties from Chapter 2. Chapter 6 explores a portion of the MEMS design space potentially opened by the use of CI-CNT, especially as applied to lamina emergent mechanisms (LEMs). Chapter 7 reviews the work of the previous chapters and discusses potential future work for better understanding and implementing CI-CNT.
2.1 Introduction

Preliminary experiments with depositing carbon in carbon nanotube (CNT) forests have shown that carbon-infiltrated CNT (CI-CNT) structures exhibit a remarkable degree of compliance compared to existing MEMS materials. Devices designed using CI-CNT could achieve a much greater degree of deflection, improving existing compliant MEMS designs and opening up additional possibilities for new ones. The high structural aspect ratio inherently possible with this process further expands its potential.

Ours is not the first exploration of applying carbon to MEMS fabrication. The use of amorphous carbon coatings in tribological applications has been explored [19, 20], and thin-film amorphous and “diamond-like” carbon MEMS structures and devices have also been created [21, 22]. Other approaches have examined the utility of carbonizing precursor structures through various techniques. Micromolding of polymeric precursors followed by carbonization through thermal treatment has been successfully demonstrated [23], as has pyrolysis of thick SU-8 patterns formed using photolithography [24]. However, the fabrication of MEMS via CVD infiltration of CNT forests with carbon has not previously been explored.

In order to better determine the potential of this material, we conducted an experiment to both reliably quantify the material properties of CI-CNT and identify the influence of certain fabrication parameters on these properties. We then used the resulting data to design and build a demonstrative compliant gripper device for restraining mammalian egg cells. We also used a finite-element analysis model of the device to compare the performance of CI-CNT with that of polysilicon for the same geometry.

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1This chapter was presented as a paper of the same title by the author at the ASME IMECE 2011 conference in Denver, CO. Paper number IMECE2011-64168.
2.2 Procedure

To determine mechanical properties of macro-scale materials, tensile testing is an established standard. At the micro-scale, however, difficulties handling and manipulating the samples make this method unfeasible. Beam bending is therefore a common and simple way to test microscale material samples [25]. In this method, cantilever beams are vertically loaded at the tip and deflected downward until failure, which typically occurs at the fixed end. The measured force and deflection at failure are then used to calculate the properties of the beam’s material.

For cantilever beams loaded by an end force $F$, linear beam theory gives

$$\sigma = \frac{FL(h/2)}{I} \quad (2.1)$$

$$E = \frac{FL^3}{3EI} \quad (2.2)$$

$$\epsilon = \frac{E}{\sigma} \quad (2.3)$$

to calculate the fixed-end stress, the Young’s modulus, and the maximum strain, respectively. Here, $L$ is the distance from the fixed end of the beam to the point of loading, $h$ is the height of the beam in the direction of deflection, $I$ is the beam’s second moment of area, and $\delta$ is the deflection of the loading point. The ultimate strength was assumed to be the stress calculated at the fixed end, where failure occurred. However, during the testing process we noted that the deflections in our samples exceeded the valid range for linear beam theory. This led us to develop a nonlinear finite element model with which to calculate the desired properties. The above equations were used to provide
initial property estimates for the model, which was then used in an iterative process to arrive at more accurate values. This process is detailed below in the “Data Analysis” section.

2.2.1 Test Pattern Design

To employ the beam bending method, we created a cantilever beam design measuring 0.267 mm wide by 2.063 mm from the fixed end to the point of loading, as shown in Figure 2.1. Beam height was planned to be approximately 200 µm, with the exact dimension determined by the CNT growth time during fabrication. Exact thicknesses for all samples were measured after breaking using scanning electron microscopy. Past beam bending experience showed that consistent load placement is difficult on a plain cantilever beam, so we included small notches on both sides of the loading point and on the end of the beam to aid with visual alignment. A test pattern was designed that consisted of an outer frame surrounding a space containing 13 cantilevers. (Figure 2.3 on page 11 shows a fabricated specimen in this configuration.)

The test pattern design was used to create a full-size, dark-field photolithography mask for use with a 4-inch wafer and positive photoresist. Multiple instances of the pattern were arranged to make effective use of the entire mask and wafer area.

2.2.2 Fabrication

Fabrication followed the same general CNT-M process as used in [16], with the necessary modifications made to the procedure to infiltrate the sample with carbon instead of silicon. The process is described below and illustrated in Figure 2.2.

Standard photolithography procedures were used to pattern AZ-3312 positive photoresist on a silicon substrate. Standard 4-inch silicon wafers were used with a single full-size photolithography mask. After photolithography, a 30-nm layer of alumina (Al₂O₃) was deposited using an e-beam evaporator. The alumina serves as a buffer layer to prevent diffusion of the iron layer into the silicon at the elevated CNT growth and infiltration temperatures. Following this, an iron layer was deposited using a thermal evaporator to act as the catalyst for CNT growth. The thickness of the iron layer was one of the parameters of our experiment; thus, thicknesses of 4 nm, 7 nm, and 10 nm were deposited. The final catalyst pattern was obtained using liftoff (15 minutes of
Figure 2.2: CNT-M process with carbon. Photoresist is spun on and developed to remain where CNT growth is not desired (a). Alumina is deposited (b), followed by iron (c), which serves as the CNT growth catalyst. The final pattern for CNT growth is formed by removing the remaining photoresist via lift-off (d). A forest of vertically-aligned CNTs (VACNTs) is then grown (e) and infiltrated with carbon (f) in a furnace, after which the sample is cooled and removed.

sonication in a Microposit 1165 bath) to remove the remaining photoresist. The wafers were then diced into individual samples.

The CNTs were grown and infiltrated with carbon using a CVD process. Samples were placed in a 1-inch quartz tube furnace and heated from room temperature to 750 °C in about 12 minutes while flowing H₂ at 214 sccm. Once the temperature stabilized, CNT growth was carried out with the addition of 146 sccm of C₂H₄. After 6 minutes the H₂ and C₂H₄ were switched off, and 230 sccm of Ar was flowed while the temperature ramped up to the infiltration temperature of 900 °C. Once temperature re-stabilized, 214 sccm of C₂H₄ was added, and infiltration using CVD commenced. As infiltration was another experimental parameter, infiltration time was either 30 min or 120 min. Once infiltration was complete, the furnace was opened and the sample allowed to cool inside the tube while Ar continued to flow. Once the equipment was sufficiently cool for handling, the sample was removed. Intrinsic stresses within the sample caused the infiltrated CNT
structure to curve slightly and self-separate from the substrate, likely during cooling. A typical sample is shown in Figure 2.3.

2.2.3 Testing

Each sample was tested in an Instron tabletop tensile testing machine, using a needle probe attached to a force transducer to gradually deflect each beam and record the resulting force. The large frame on each sample was used to secure the sample in a custom-built clamp, which was designed to hold the sample flat and prevent the fixed end from deflecting. The clamp was mounted on a stage beneath the Instron’s probe tip, allowing us to precisely move the sample and position each beam tip under the probe. To ensure consistent loading of all beams, we aligned the probe at a target point using the alignment notches of each beam. We used a digital optical microscope to assist with alignment and to observe and visually record each test.

Each beam was tested individually by positioning the force probe slightly above the beam, as shown in Figure 2.4, and lowering it at a constant rate of 0.5 mm/min, allowing it to contact the beam and continue lowering until the beam failed. Both the force and deflection values were
continuously recorded during each test. Following each beam test, the data were copied from the testing program into a spreadsheet.

2.2.4 Data Analysis

As noted previously, we found during testing that many of the beams experienced deflections greater than 10% of their length. As linear beam theory loses accuracy for such large deflections, we constructed a nonlinear finite element model of the cantilever beam design using ANSYS. The model accounted for non-linearity in the beam’s motion as well as the presence of fillets (conservatively measured to be approximately 90 μm) that existed at the fixed end of the beam. Element type “SOLID95” was employed. We used symmetry along the vertical axial plane of the beam to reduce computational demands while preserving model fidelity.

The linear beam theory results were used as the initial properties in the ANSYS model. We then ran the ANSYS simulation with the measured maximum deflection as the loading condition and observed the force at the loading point. This force was compared with the actual measured
force acquired during testing. Since linear beam theory states that there is a direct relationship between Young’s modulus and the force being applied to the end of a beam, we used this ratio to scale the Young’s modulus as follows:

\[
E_{corrected} = \frac{F_{measured}}{F_{ANSYS}} E_{linear}
\]  

(2.4)

We then re-ran the ANSYS model using this corrected modulus. When the forces were compared again, they were found to match very closely. A failure stress correction factor could then be calculated in the form

\[
C = \frac{\sigma_{corrected}}{\sigma_{linear}}
\]  

(2.5)

where \(\sigma_{linear}\) is the failure stress found using linear beam theory and \(\sigma_{corrected}\) is the failure stress found using the second-run ANSYS-generated values.

We discovered that, in a given sample, all of the beams tended to have the same correction factor. We therefore used a subset of the beams—the center and the two end beams—to determine each sample’s correction factor. We then applied that correction factor to the calculated values of all the beams in that sample.

When developing the ANSYS model, we also discovered that changing the Poisson ratio made little difference in the result when kept within reasonable bounds. We therefore used a Poisson ratio of 0.3 for all simulations.

2.2.5 Design of Experiment

The objective of our experiment was to determine the influence of two sample preparation factors—iron layer thickness and infiltration time—on the material properties of CI-CNT structures. To do this, we conducted a full-factorial design of experiments with three levels of iron layer thickness and two levels of infiltration time, resulting in the experiment design in Figure 2.5. Two replicates of each treatment combination were prepared, with the exception of the 4-nm, 30-min treatment, for which three replicates were performed. In some cases, errors during fabrication or testing invalidated the results from a sample, requiring the fabrication of an additional sample to take its place.
Figure 2.5: Experiment treatments. Two replicates of each treatment were tested, with the exception of the 4-nm, 30-min treatment, where three replicates were performed.

Figure 2.6: Force-deflection curve for a beam in the 7-nm, 120-min sample.

2.3 Results and Discussion

Figure 2.6 shows a typical force-deflection curve for a CI-CNT beam. As mentioned previously, the plot reveals that the curve is nearly linear. Using Equations 2.1–2.3, this translates to
Table 2.1: Ultimate Strength (MPa)

<table>
<thead>
<tr>
<th>Infiltration time</th>
<th>30 min</th>
<th>120 min</th>
</tr>
</thead>
<tbody>
<tr>
<td>4 nm iron</td>
<td>140.37 ±21.01</td>
<td>123.38 ±24.61</td>
</tr>
<tr>
<td>7 nm iron</td>
<td>108.31 ±23.28</td>
<td>152.06 ±41.03</td>
</tr>
<tr>
<td>10 nm iron</td>
<td>101.77 ±51.60</td>
<td>231.80 ±29.03</td>
</tr>
</tbody>
</table>

Table 2.2: Elastic Modulus (GPa)

<table>
<thead>
<tr>
<th>Infiltration time</th>
<th>30 min</th>
<th>120 min</th>
</tr>
</thead>
<tbody>
<tr>
<td>4 nm iron</td>
<td>8.66 ±1.12</td>
<td>7.43 ±1.43</td>
</tr>
<tr>
<td>7 nm iron</td>
<td>5.37 ±1.28</td>
<td>9.40 ±0.98</td>
</tr>
<tr>
<td>10 nm iron</td>
<td>4.10 ±1.56</td>
<td>15.7 ±2.26</td>
</tr>
</tbody>
</table>

a linear stress-strain relation for the material up to failure. The sudden drop of force back to zero indicates that failure is characteristic of an instantaneous, brittle failure.

Tables 2.1 and 2.2 summarize for each treatment the values and standard deviations of ultimate strength and elastic modulus. Table 2.3 gives the strain values calculated from these summaries, as well as their standard deviations. The number of individual cantilevers per treatment determined the $n$ values used to calculate the standard deviations. The $n$ is therefore 26 for all but the 4-nm/30-min and 7-nm/120-min treatments, which were both missing a cantilever and thus have an $n$ of 25. For compliant devices, a high maximum strain is desirable, as this results in a higher achievable deflection before failure. The highest maximum strain for CI-CNT was 2.48%, associated with the 10-nm, 30-min growth parameters, followed by the 7-nm, 30-min sample with 2.02%. The lowest maximum strain of 1.48% was experienced by the 10-nm, 120-min sample. Thus, of the levels tested, an iron layer thickness of 7-10 nm with a 30-min infiltration time appears to be the most favorable for fabricating a compliant CNT-MEMS device with this process.

2.3.1 Infiltration Verification

In conducting our analyses, we assumed that the beams had uniform properties through the entire cross-section. To ensure that this assumption was correct, we examined some of the fracture
Table 2.3: Maximum Strain (%)

<table>
<thead>
<tr>
<th>Infiltration time</th>
<th>30 min</th>
<th>120 min</th>
</tr>
</thead>
<tbody>
<tr>
<td>4 nm iron</td>
<td>1.62 ±0.30</td>
<td>1.66 ±0.54</td>
</tr>
<tr>
<td>7 nm iron</td>
<td>2.02 ±0.99</td>
<td>1.62 ±0.50</td>
</tr>
<tr>
<td>10 nm iron</td>
<td>2.48 ±0.51</td>
<td>1.48 ±0.28</td>
</tr>
</tbody>
</table>

Figure 2.7: SEM image of a typical fracture surface due to beam failure during testing.

surfaces for each sample using a scanning electron microscope (Figures 2.7 and 2.8). These images showed that, while the structures were not completely filled, the distribution of voids within the beams was fairly uniform. As a result, our assumption of uniform properties within each sample was validated. We also observed that the fracture surface was rough and jagged, rather than flat. This suggests a failure mode more complex than simple brittle fracture and merits further study.
2.3.2 Statistical Analysis

To better understand correlation of the growth parameters with the material properties, we performed an analysis of variance on the full set of individual beam data gathered. This was especially useful in managing the high variability in the data, which is evidenced by the large standard deviations given in the tables. The corrected Young’s modulus and failure stress values for each beam were grouped by sample for the replicates of each experiment treatment. Correlations of iron thickness, infiltration time, and the interaction to failure stress, modulus, and maximum strain were then computed. The $P$ values for each correlation are given in Table 2.4.

Based on the analysis, we can conclude with a 95% confidence level that infiltration time has a statistically significant effect on both failure stress and Young’s modulus. Additionally, the interaction of infiltration time and iron thickness appears to affect Young’s modulus as well, with a 95% confidence level. Iron thickness alone does not correlate significantly with any of the material properties, and none of the effects seem to have a significant influence directly on maximum strain. This latter result may be because the variation between samples is on the same
Table 2.4: Main Effect and Interaction $P$ Values. (Values of $P < 0.05$ in Bold)

<table>
<thead>
<tr>
<th>Effect</th>
<th>Failure Stress</th>
<th>Modulus</th>
<th>Max Strain</th>
</tr>
</thead>
<tbody>
<tr>
<td>Infiltration Time</td>
<td>0.0375</td>
<td>0.0064</td>
<td>0.1930</td>
</tr>
<tr>
<td>Iron Thickness</td>
<td>0.2862</td>
<td>0.2731</td>
<td>0.9355</td>
</tr>
<tr>
<td>Interaction</td>
<td>0.1898</td>
<td>0.0459</td>
<td>0.7037</td>
</tr>
</tbody>
</table>

order as the differences between the average values in Table 2.3. This suggests that relatively high strain can be achieved over a wide range of fabrication parameters.

An inspection of the summary tables reveals a curious trend in the maximum strain values. For 30-min infiltrations, maximum strain increases with increasing iron layer thickness, but for 120-min infiltrations, the reverse occurs. We suspect this peculiar difference is due to the fact that, according to our observations, thicker iron layers tend to produce less dense CNT forests. A less dense forest may not have enough surface area to permit complete infiltration via CVD during 30 min, leaving numerous voids throughout the framework that result in a more flexible structure but a lower effective failure strength. In contrast, a 120-min infiltration time (used with the sample in Figure 2.8) likely ensures a more complete infiltration, resulting in a stronger but less flexible structure. Because a denser forest provides more surface area for infiltration to act on, this extra time makes less of a difference for thinner iron layers than for thick ones. Hence the difference in maximum strain between the two infiltration times for the 4-nm iron layer samples is small compared to the difference for the 10-nm iron layer samples.

The impact of this behavior on design depends on the application of the mechanism being designed. If a high deflection is the sole need, then the lower strength of the high-strain CI-CNT is of less importance. If the device must bear or apply a load, however, then a slightly lower strain may be used in order to employ CI-CNT of sufficient strength—though, as the statistical analysis shows, this tradeoff is not significant.

### 2.3.3 Comparison with Other Materials

Figure 2.9 compares the maximum strain of the 10-nm, 30-min CI-CNT with three macro-scale engineering materials (1040 steel, aluminum, and polypropylene) and two common MEMS
Figure 2.9: Maximum strain of CI-CNT (10-nm, 30-min) compared with common MEMS and macro-scale engineering materials.

Figure 2.10: Failure strength of CI-CNT (10-nm, 30-min) compared with common MEMS and macro-scale engineering materials.
structural materials (polysilicon and SU-8 photoresist). Figure 2.10 compares failure strength for the same materials. Macro-scale and MEMS material values are from [14] and [26], respectively. With regards to maximum strain, CI-CNT is notably higher than SU-8 in the MEMS category and compares favorably with polypropylene. It is several times more flexible than the engineering metals listed. Its failure strength is roughly a third that of steel and 1/10 that of polysilicon.

2.4 Implementation Example

To demonstrate the application of these properties to a carbon-infiltrated CNT-M design process, we used the results of this experiment to guide the development of a prototype CI-CNT device. We chose to work in concert with another project in our lab to design and fabricate a compliant cell gripper device. The device needed to actuate and grasp a 100-µm diameter egg cell, holding it firmly in place so a microscopic injection needle could inject it with DNA without the cell slipping or being pulled out of place. The ultra-high aspect ratio of CI-CNT was ideal for this task, as it provides the vertical stiffness and high surface area needed to restrain a cell. Furthermore, the high compliance of the material could allow the device to remain reasonably small and still provide the range of motion necessary to allow unimpeded access to the cell before actuation and a firm hold on the cell once actuated. Finally, the short cycle time and potentially low cost of fabrication promised a more convenient route to prototyping and testing the design than would be possible with conventional high aspect ratio MEMS fabrication techniques. The full cell restraint design project, which included this and other restraint designs, is discussed in great detail in Chapter 5.

An initial concept was developed on paper that employed a stiff lever bar and several long, slender beams in tension, compression, and bending to close a pair of graspers and hold the cell. Because of the anticipated complex motion of the mechanism, the geometry for this concept was then refined using a finite-element analysis beam model in ANSYS. 1-D beams were drawn according to the initial geometry and then assigned geometric and material property values. All flexible beams were given a width of 15 µm—the minimum feasible feature resolution for our selected mask fabrication process—and all beams were given a height of 100 µm. A worst-case modulus of 15 GPa was assumed. The “BEAM3” element type was used with all beams to ensure proper, nonlinear analysis. A desired deflection of the actuation bar of 50 µm was assigned as the sole
loading condition. Results were output as deflections and principal stresses. Once the model was complete, the appropriate commands were saved as an ANSYS script file to facilitate repeated runs with varying parameters. The simulation was run multiple times using a trial-and-error method to arrive at a configuration that delivered the desired tip deflections while remaining below the expected failure stress at all points. Once sufficient geometry had been identified, a CAD model was developed using the measurements, and the design was included in a mask for CNT-MEMS fabrication.

The device was grown using the same procedure and similar parameters as the beam samples. A 7.5-nm iron layer was deposited, and CNTs were grown for 5 min and infiltrated for 30 min. The finished device was mounted on a standard microscope slide using an adhesive transfer tape. A glass cell holding pipette was used to actuate the mechanism, and a digital optical microscope was used to observe and record the procedure. Figure 2.11a shows the device mounted and ready for actuation, while Figure 2.11b shows the device fully actuated. Figure 2.12 shows a tilted SEM view of a second sample of the device.

When actuated, the device behaved as predicted by the ANSYS model. The graspers could be fully closed without any sign of failure. After testing, a small crack was noticed on the end of the fulcrum beam, which experiences compression during operation, but a review of imagery from the test revealed that the crack was present before any actuation had taken place and likely formed during fabrication or transport. In any case, the point experiencing the maximum stress occurs in the longest beam and held successfully.

To compare this material’s performance with that of polysilicon, the ANSYS model was run with properties for both materials. The CI-CNT material properties used were those of the 7-nm, 30-min configuration: that is, a Young’s modulus of 5.4 GPa and an ultimate strength of 108.3 MPa. A Poisson ratio of 0.33 was assumed. For polysilicon, a Young’s modulus of 165 GPa, an ultimate strength of 1 GPa, and a Poisson ratio of 0.28 were used. With an actuation point deflection of 50 µm, the maximum stress predicted by the model was 23.7 MPa for CI-CNT, resulting in a safety factor of about 4.7 for the CNT device. In comparison, the maximum stress predicted by the polysilicon to attain the same actuation was 729 MPa, providing a safety factor of about 1.4. This indicates that, in this application, CI-CNT could likely undergo significantly more deflection, while polysilicon is already approaching its limit. Alternatively, the CI-CNT device
Figure 2.11: (a) Optical microscope image of the CI-CNT cell gripper device prior to actuation by a cell holding pipette (lower-right). (b) Cell gripper device deflected by the holding pipette to a fully closed position.

Figure 2.12: Perspective-view scanning electron micrograph of the CI-CNT cell gripper device at 70x magnification. Beam width is 15 µm, and structure height is approximately 100 µm.
could be further reduced in size and still achieve the same motion with the same beam widths, while the polysilicon device would require narrower beams simply to avoid failure.

2.5 Conclusion and Recommendations

An experiment to determine the elastic modulus and ultimate strength of carbon-infiltrated carbon nanotube microstructures was conducted in order to obtain maximum strain values for compliant MEMS design. Two CI-CNT fabrication parameters were varied in order to determine their effects on the material’s mechanical properties. We found that, in our experiment, the most favorable parameters for obtaining highly compliant CI-CNT were a 7-10 nm iron catalyst layer and a 30 min infiltration time, resulting in maximum strains of around 2.3%. The material’s compliance exceeds that of current MEMS materials and compares favorably with conventional compliant macro-scale engineering materials. Additionally, we demonstrated the use of the modulus, ultimate strength, and maximum strain data to guide the design of a compliant cell gripper device made using the carbon-infiltrated CNT-M process. We also showed that, for the given geometry, CI-CNT was able to provide the necessary deflection with a much higher safety factor than would be possible using polysilicon.

CI-CNT holds great potential for future use in high aspect ratio, compliant MEMS applications. Its ease and potential low cost of fabrication could make it useful not only for final products but for rapid prototyping of devices that will ultimately be made using a more expensive or time-consuming process. However, further exploration of the material properties and behavior of the material is necessary before it can be considered a reliable MEMS material. A more detailed factorial experiment could be used to characterize growth parameters and pinpoint the levels that maximize compliance. The impact of additional fabrication parameters can also be explored. Furthermore, as previously mentioned, the failure mode showed indications of a more complex behavior than simple brittle failure. A closer investigation of these fracture surfaces and their significance, as well as the role infiltration voids may play in causing this, is warranted. Useful approaches would include focused ion beam cuts to better reveal the internal structure, especially in the vicinity of the fracture.

Furthermore, this study only explored the material properties in vertical bending. Due to the volumetric dominance of infiltrated carbon in these structures, we expect lateral and axial
loading of the beams to behave similarly; however, it is possible that the presence of CNTs in the material may cause unexpected deviations from the properties given above. Additionally, torsional behavior, which would be important for out-of-plane motion in many MEMS applications, remains to be explored. Nevertheless, the properties quantified in this experiment provide a promising indication of the capabilities of this material and its potential to expand the range of possibilities for MEMS devices.
CHAPTER 3. A COMPARISON OF CI-CNT AND SINGLE-CRYSTAL SILICON IN-PLANE BENDING

3.1 Introduction

One of the primary reasons for the interest in using CI-CNT for compliant-MEMS fabrication is its greater flexibility compared to other MEMS materials, which allows devices to accomplish the same motion with a greater safety factor or a smaller device, or to achieve greater ranges of motion than could previously be feasibly attained. But how significant of an advantage does CI-CNT really provide? Since silicon is one of the most common MEMS materials, we decided that a comparison of similar devices made from both materials would be informative. It would provide a convenient and straightforward way to both quantify and visualize the superior performance of CI-CNT. We therefore measured the deflection of in-plane cantilever beams made from both CI-CNT and silicon and compared their performance in terms of maximum strain. This chapter describes this process and presents the results thereof.

3.2 Procedure

The beams of a compliant cell restraint mechanism (described in detail in Section 5.3.4 on page 50) were deemed well suited for a direct comparison. The same restraint device had been fabricated using both the CI-CNT procedure and a conventional silicon on insulator (SOI) process that produces single-crystal silicon devices. Measurement with a digital microscope confirmed that the devices from both processes had nearly identical dimensions. The beams were approximately 600 µm long. Widths varied by approximately 20% between the two materials, with the SOI beams measured at 15-16 µm thick and the CI-CNT beams slightly wider at 18-20 µm thick. For the analysis we specified widths of 15.42 µm for the SOI beams and 19 µm for the CI-CNT beams. Beam height was not measured, as an approximation was sufficient for the analysis procedure.
We tested the beams by deflecting them with a tungsten microprobe attached to a manually operated micromanipulator. To deflect a beam, the microprobe pushed the grasper head at the end of the beam forward (towards closed position), as shown in Figure 3.1. Deflection was increased until the beam failed. The operation was observed and recorded using a digital microscope to allow measurement after the fact.

Following testing, the videos were reviewed frame by frame, and still images were extracted of each beam in its undeflected and maximum deflected positions. Each pair of images was then overlaid and aligned to create a single coordinate system for each beam. X-Y coordinates of the fixed end, the undeflected tip location, and the deflected tip location were found with Photoshop CS5 using the program’s coordinate readout tool. X-Y coordinates were also found for the ends of line segments that paralleled the top of the grasper head in its default and deflected positions. These values would be used to identify the change in angle of the head and thus determine the angle at which the force is being applied when fully deflected. Figure 3.2 shows the locations of these points and line segments.

A MATLAB script was developed to process the collected coordinate points and align them with the standard-form compliant beam for deflection analysis (a horizontal beam deflecting upward with the fixed end on the left). The script reads in the three sets of points for each beam, flips and rotates them as needed, and scales them using a predefined scaling factor. The scaling factor is necessary in order to convert the pixel-based coordinate values into the correct dimensional values in microns. It was determined by examining pixel values for distances measured on the digital microscope and superimposed on a screenshot taken prior to testing. That same pixel
measurement was made in a video frame grab to find the ratio between the pixel sizes of the two images, producing the microns-to-pixels scaling factor needed for the conversion.

The MATLAB script also reads in the coordinates for the other four points, converts them to vectors, and uses a dot product to compute the angle of the deflected beam tip relative to the undeflected beam. The rotated and scaled coordinate sets and calculated deflection angles for all processed beams are then saved to a file.

Our ultimate objective in this analysis was to arrive at maximum strain values that could be used to compare the silicon SOI beams to the CI-CNT beams of identical geometry. To find the strain values, we modified a fixed-free cantilever beam solver developed in Excel to determine maximum strain in a beam having given $a$ and $b$ values, which are the $x$ and $y$ coordinates of the beam’s tip after deflection. The solver software included in Excel was used to accomplish this. Two cells were created to calculate the positive difference between the measured and the calculated $a$ and $b$. The spreadsheet’s original optimization objective was to minimize the difference between two $\alpha$ values, one found using linear beam theory and the other calculated using elliptical integrals. We modified the objective by adding the $|\Delta a|$ and $|\Delta b|$ cells to it, causing the solver to minimize all three values. The solver did so by adjusting the applied force and the beam’s $k$ value. Once optimized to the point where $|\Delta a|$ and $|\Delta b|$ were nearly zero, a cell added to the spreadsheet calculated the beam’s maximum stress, which occurs at its fixed end. Using Hooke’s law,
the maximum stress was divided by the $E$ specified in the spreadsheet’s parameters section. The resulting strain value was displayed in a final cell. It was not necessary to know the correct $E$ for the material, since whatever value is used is divided back out of the result when Hooke’s law is applied.

### 3.3 Results and Discussion

Seven silicon SOI beams and two CI-CNT beams were tested to failure and analyzed. Calculated maximum strains for each beam can be seen in Table 3.1. As expected, the CI-CNT beams reached a higher maximum strain before failure than the silicon beams. The average strain in the SOI silicon beams was 1.25%, while the average strain for the two CI-CNT beams was 2.25%. Though the sample size for CI-CNT is small, this result is consistent with what had been previously measured for CI-CNT. The tests in Chapter 2 placed the maximum strain of CI-CNT made with a 7-nm iron layer (as the cell restraint devices were) at $2.02 \pm 0.99\%$. The strain values obtained in this experiment fall well within that range. Additionally, maximum strain of the CI-

<table>
<thead>
<tr>
<th>Beam Tested</th>
<th>Max Strain</th>
</tr>
</thead>
<tbody>
<tr>
<td>SOI-A-L</td>
<td>1.23%</td>
</tr>
<tr>
<td>SOI-A-R</td>
<td>1.30%</td>
</tr>
<tr>
<td>SOI-B-L</td>
<td>0.87%</td>
</tr>
<tr>
<td>SOI-C-L</td>
<td>1.41%</td>
</tr>
<tr>
<td>SOI-C-R</td>
<td>1.27%</td>
</tr>
<tr>
<td>SOI-D-L</td>
<td>1.51%</td>
</tr>
<tr>
<td>SOI-D-R</td>
<td>1.19%</td>
</tr>
<tr>
<td>CNT-CellR-L</td>
<td>2.45%</td>
</tr>
<tr>
<td>CNT-CellR-R</td>
<td>2.05%</td>
</tr>
<tr>
<td><strong>Average SOI</strong></td>
<td><strong>1.25%</strong></td>
</tr>
<tr>
<td><strong>Average CI-CNT</strong></td>
<td><strong>2.25%</strong></td>
</tr>
</tbody>
</table>

CNT beams is, on average, 44% higher than the average maximum strain of the single-crystal silicon beams. Even the worst CI-CNT value bests the top SOI value by over 26%. Clearly, this difference is sufficient to provide a significant advantage when designing compliant MEMS devices using CI-CNT. Figure 3.3 shows an overlay of the best cantilever from each material (SOI-D-L and
Figure 3.3: Deflection comparison overlay of the beam from each material with highest deflection. The darker material is CI-CNT and the lighter material silicon. A broken CI-CNT beam is seen beneath the deflected beam.

Note that the CI-CNT beam achieves a greater deflection despite being 15%–20% thicker than the silicon beam.

3.4 Conclusion

A simple comparison was made of the maximum strains in beams with near-identical geometry fabricated from both silicon and CI-CNT. The values found for the two sets of beams reinforced previous observations that CI-CNT is notably more flexible than silicon. This comparison of the two materials using identical geometry provides a good, simple visualization of this difference.

It should be noted as part of this discussion on in-plane deflection that recent CI-CNT device tests produced drastically different elastic modulus values than previously seen, casting some doubt on the applicability of the established modulus values to in-plane deflection. Since that is the primary mode of actuation for compliant MEMS devices, this discrepancy merits further investigation. An in-plane beam test pattern has been developed to do just that, but data have yet to be obtained. Notwithstanding that difference, this experiment quantitatively demonstrated an
in-plane strain comparable to the out-of-plane strain previously established. Future work in this area will add valuable understanding to the in-plane behavior of this material.
CHAPTER 4. AN EXPLORATION OF CI-CNT TORSIONAL PROPERTIES

4.1 Introduction

Our initial work provided a good understanding of the basic mechanical properties of CI-CNT. The elastic modulus and maximum strain values we had obtained provided a foundation from which to design and build successful compliant MEMS devices featuring in-plane motion.

An increasing body of work by BYU and others focuses on a field of compliant mechanisms termed “lamina emergent mechanisms,” or LEMs [27]. Like any compliant mechanism, LEMs derive their motion from the flexibility of their constituent material. LEMs are unique, however, in that they consist of one or more planar layers of material with features designed to cause motion in the out-of-plane direction. This behavior is readily observed in such things as pop-up books but is only now beginning to be systematically explored and implemented in a mechanistic fashion. The advantages of LEMs over other types of mechanisms, compliant or otherwise, include:

- Ease of fabrication
- Low cost of fabrication
- Compact storage and shipping
- Complex motion achievable with simple topology

To achieve out-of-plane motion from a single material layer, several forms of compliant joints exist. For linear motion normal to the material layer, such as that provided by orthoplanar springs, fixed-guided beams in series and parallel function well. To enable more complex motions, such as those experienced by four-bar linkages and the like, a new joint termed the lamina-emergent torsional (LET) joint was developed [28]. LET joints, which exist in several forms, derive their motion from the twisting of a long, relatively narrow segment of material. The rotation is distributed uniformly throughout the torsional member, preventing failure by ensuring that no single
portion of the joint ever exceeds the material’s maximum shear stress. When designed properly, LET joints can achieve substantial rotational deflection. In combination, they enable the design of many different and intricate mechanisms from simple sheets of material.

Their manner of fabrication makes LEMs particularly attractive for MEMS applications, where thin-film processes are routinely patterned into very intricate layer-based designs. The CI-CNT process, while currently strictly a single-layer process, is no exception. With the material’s excellent compliant properties, it was expected that it would lend itself to LEM fabrication as well. However, to understand its behavior in torsion—an important piece of information for optimal LET-based CI-CNT LEM design—elastic modulus and strain alone are insufficient. Additional properties relevant to out-of-plane motion and torsion needed to be investigated and quantified if effective design beyond a trial-and-error approach was to be accomplished. Two related material properties are relevant when considering torsion: shear modulus $G$ and poisson ratio $\nu$. In an isotropic material, the two are related to the elastic modulus $E$ by

$$ E = 2G(1 + \nu). $$

The purpose of this experiment was to lay a groundwork for obtaining these important properties for CI-CNT. For this initial examination, we assumed isotropy in the material. While this assumption is almost certainly inaccurate, we expected the results to provide insight into CI-CNT torsional behavior and a “ballpark” value for shear modulus that could be refined through later
work. A secondary objective was to investigate the potential impact of the floor layer—a thin carbon film deposited during infiltration which often spans small gaps in CI-CNT patterns—on the testing process.

4.2 Procedure

4.2.1 Design

To examine torsion, a design was needed that could apply a rotational deflection to a slender piece of CI-CNT. Given the small size and brittle nature of CI-CNT samples, applying rotation by twisting a slender beam directly was highly unfeasible. No equipment existed on the necessary scale to make it otherwise. Instead, a CI-CNT test design would have to convert a linear deflection applied by available testing equipment into rotation applied to such a beam incorporated into the design’s geometry.

A simple and reasonably accurate method of accomplishing this can be found in [13]. In that design, two thin torsional bars support a stiff beam between them (Figure 4.2). The beam acts as a lever which can be pressed down at the tip to twist the torsional bars. If a specified deflection is applied at the tip and the resulting reaction force is measured (or vice versa), the approximate
shear properties of the material can be determined. Ideally, the torsional bars would have a circular cross-section, but a rectangular cross-section can be analyzed in the same manner by using an effective torsional moment of inertia.

To implement this design in CI-CNT, we chose dimensions that we expected would produce a range of motion similar to that experienced by the simple cantilever beams discussed in Chapter 2. The length of each torsional bar was set at 1000 µm. A loading arm 500 µm wide and 1250 µm long was used. As with the bending-test cantilevers, notches were incorporated into the arm tip to facilitate force probe alignment (Figure 4.3a). The notches were placed such that the load would always be applied 1100 µm from the horizontal center of the torsion bars for every test sample, regardless of bar width. Since the cross-section expected to provide the best results was unknown, three bar widths—25, 50, and 100 µm—were chosen. Bar height would be determined by CNT growth time, but the bar widths were selected with a CNT forest height of 100 µm in mind.

The sample set layout used for testing can be seen in Figure 4.3b. It consists of a linear array of five torsional test devices. The test devices were arranged with a 1000-µm buffer between each sample to prevent the failure of one from damaging any others. A wide area behind the devices was included to give the test fixture an ample surface to grasp securely. The resulting sample set was mirrored so that two sets would face each other on the wafer. The resulting 1 x 2 cm pattern was a convenient size for fabrication and also maximized yield. A mask was prepared
Figure 4.4: (a) Diced wafers containing torsion samples. (b) A torsion sample in the custom-built chuck being tested on the Instron testing machine. The needle visible near the center is attached to the machine’s head and force transducer assembly, which moves vertically to apply a deflection.

which included several sample sets for each torsional bar width. Some unpaired sample sets were also included in available space on another mask being prepared at the time.

4.2.2 Fabrication

We prepared two wafers using the procedure described in Chapter 2. Due to changes in the photolithography machines, exposure time had to be increased to 10-11 seconds in order to prevent translucent specks in the mask’s transparency material from being patterned in the photoresist. We deposited iron on both wafers using a DENTON thermal evaporator system. The primary torsion pattern wafer received approximately 7 nm of iron, while the other wafer, which included only a few torsion sample sets, received approximately 10 nm. Both wafers were diced into the individual pattern blocks using the cleanroom dicing saw. We performed liftoff on the individual pieces following dicing. Since a previous wafer had developed odd specks of contamination using the standard liftoff equipment in the ESC underground lab, the liftoff procedure for these two wafers was done in the cleanroom using fresh 1165. Both wafers turned out clean and with no apparent flaws from preparation. Figure 4.4a shows the numerous sample pieces of both wafers. CNT growth and carbon infiltration followed liftoff. Specific values for the relevant fabrication parameters of each sample set can be reviewed in Table 4.1.
4.2.3 Testing

The torsion test devices were tested with the same Instron machine and force transducer described in Section 2.2.3 (page 11). We loaded the sample sets into a chuck that was custom-built by the CNT research group for such tests. (An earlier version of the same chuck was used for the bending tests.) We then tested the devices one by one as shown in Figure 4.4b, using the chuck’s x-y stage to align the probe tip with the notches on each beam. During testing, we discovered that alignment consistency could be improved by placing two lights in an orthogonal arrangement and moving the stage until the shadows cast by the force probe onto the beam lined up with the notches.

To apply a controlled deflection, we re-used the bending test profile developed for the simple cantilevers, which specifies a steady, 0.5 mm/min downward probe deflection and records the time, deflection, and reaction force on the probe tip. Most of the devices were tested to failure. Some of these were subjected to a sub-failure load cycle prior to loading them to failure in order to check for any changes or variations due to cycled loading. On most of the sample sets, one test device was preserved for future examination by never loading it entirely to failure.

4.2.4 Data Analysis

Data from the tests were processed with a MATLAB script developed to calculate a shear modulus. The script first loads the raw force and deflection data output by the Instron machine. It then trims the data, removing data points occurring after breakage or before probe contact with the loading arm, leaving only the data acquired during loading. The script then calculates a slope for the deflection-force data of each test device using a linear least-squares fit curve, providing a force-to-deflection ratio $\psi$ for that sample. To avoid any pre-failure irregularities and to decrease the likelihood that probe tip slippage might skew results, only the first half of the data from each test device were utilized. The first 20 data points (1 second) of probe contact were also omitted to remove any initial data noise. The resulting $\psi$ value was reasonably insensitive to any remaining data anomalies.

With the slopes determined, the shear modulus for each sample was calculated. This was done using the equations for analyzing a symmetric torsional test pattern described on pp. 85-86 of [13]. Given a torsional bar with a rectangular cross-section of width $2w$ and height $2t$, a torsional
moment of inertia is first calculated as

\[ J = wt^3 \left[ \frac{16}{3} \left( 1 - \frac{t^4}{12w^4} \right) \right] \text{ for } w \geq t \]  

(4.2)

SEM measurements of a torsional bar’s width and height for each sample set were used by the script for this calculation. The shear modulus appears in the torsional deflection equation

\[ \delta_{\text{torsional}} = \phi L = \frac{TIL}{2JG} = \frac{FL^2l}{2JG} \]  

(4.3)

where \( l \) is the length of an individual torsion bar (in this case 1000 \( \mu \)m), \( L \) is the distance from the horizontal center of the bar to the point of loading on the loading arm (1100 \( \mu \)m), \( \delta_{\text{torsional}} \) is the vertical deflection of the loading arm at the loading point, and \( F \) is the force applied to achieve that deflection. This can be rearranged as

\[ G = \frac{FL^2l}{2J\delta_{\text{torsional}}} \]  

(4.4)

to use the measured force and deflection to calculate the shear modulus. Having found the slope \( \psi \) for each test run, which is essentially a ratio of force to deflection (and is constant for a linear force-deflection data set), the equation further simplifies to

\[ G = \psi \left( \frac{L^2l}{2J} \right) \]  

(4.5)

If \( \psi \) is in standard MEMS units of \( \mu N/\mu m \), the calculated shear modulus is obtained in MPa.

For post-test identification, each test device was given a label in the form of TX-Y, where X is the sample set number and Y is the device number (ranging from 1 to 5, with 5 being the closest to the CMR logo on the end of the frame). Five sample sets, labeled T1 through T5, were tested. Device T2-1 was destroyed prior to testing and could not be tested. Prior to testing, the floor layer was removed from sets T3 through T5. It was left intact on sets T1 and T2 to examine its impact on the testing process. To do this, T1 was tested right side up, while T2 was tested with the floor layer facing up (Figure 4.5).
4.3 Results and Discussion

Table 4.1 presents the fabrication parameters and calculated shear modulus for each sample, and the shear modulus values are plotted in Figure 4.6. The results show reasonable agreement on a shear modulus across sample sets T4 and T5. The average modulus for these two together was 4.8 GPa. Sample set T1 was lower and more variable but in the same range. Set T2 agreed more perfectly with T4 and T5 with the exception of T2-2, which at 13.4 GPa was well over twice the modulus of the other samples in that set. The cause of this anomaly is unclear, but the raw data
do show a much higher reaction force for that loading arm than the remainder of the set. It is possible that the floor layer or some defect in the sample’s geometry led to this discrepancy. When the value of T2-2 is omitted along with the values of T3, the average shear modulus remains 4.8 GPa. Sample set T1’s values were lower than the T4-T5 average and varied notably, while the three similar T2 modulus values were higher than that average. Together, however, the T1-T2 average with T2-2 omitted was also approximately 4.8 GPa. Together, these results could indicate that the floor layer does indeed have an effect on torsion tests. Averaging the two tests together may have cancelled out this effect. To be sure, more data across a range of torsion bar configurations is needed.

T3 clearly differs from the other sample sets, with an average $G$ of 10.38 GPa. Two factors—its higher torsion bar aspect ratio (250 µm by 50 µm) and its thicker iron layer (10 nm)—may have caused this difference.

The shear modulus was not the only item of interest in these tests. We were curious also to examine the failure surfaces of the samples to see the mode of shear failure and consider its implications regarding the nature of CI-CNT. We examined all failure surfaces on the remaining base of each sample set. Figure 4.7 shows representative pairs of failure surfaces (left and right torsional bar) from T1-2 and T3-1.

The failure surfaces confirm the brittle nature of the CI-CNT material. The curvature of the surface also adds to the evidence that the material is not isotropic. Curvature was only observed in the plane parallel to the axes of the CNTs; in the transverse direction (cutting through the CNTs), the failure profile is linear. This pattern was observed on all torsion samples, regardless of torsion bar aspect ratio or fabrication iron layer thickness. (Indeed, in T3 with its higher aspect ratio, the curvature is even more pronounced.)

As mentioned previously, sample sets T1 and T2 (which were fabricated together) were used to investigate whether the presence of a floor layer has any substantial impact on the torsion testing process. To test this, sample set T2 was tested bottom side-up. As is evident in Figure 4.5b, the floor layer spans the gap between the torsion bars and the frame of the sample set. As the loading arm deflects and the bars twist, the layer tends to ripple and warp until failure of the torsion bars causes it to rip. The results are inconclusive but do hint at the possibility that the floor layer influences the torsion test process. Since the upward-facing sides of the torsional bars
Figure 4.7: (a) and (b): Scanning electron micrographs of the left and right torsional failure surfaces of sample T1-2. (c) and (d): Torsional failure surfaces of sample T3-1.

twist away from the base, the tension in the floor layer may have worked against torsion slightly, allowing simple bending in both the bars and the beam to contribute more substantially to the measured deflection. Much as aluminum foil can handle significant tensile forces despite being a relatively thin film, the floor layer may have been able to provide a measurable counteracting force to the torsion. In samples that are right-side up, the floor layer would be in compression, and its contributions would be negligible.
Table 4.1: Shear Modulus and Fabrication Parameters for Torsional Test Devices

<table>
<thead>
<tr>
<th>Sample</th>
<th>Shear modulus G (GPa)</th>
<th>Fe thickness</th>
<th>Grow time</th>
<th>Infiltration time</th>
<th>Torsion bar height</th>
<th>Torsion bar width</th>
</tr>
</thead>
<tbody>
<tr>
<td>T1-1</td>
<td>4.684</td>
<td>7 nm</td>
<td>5 min</td>
<td>27 min</td>
<td>146 um</td>
<td>104 um</td>
</tr>
<tr>
<td>T1-2</td>
<td>5.429</td>
<td>7 nm</td>
<td>5 min</td>
<td>27 min</td>
<td>146 um</td>
<td>104 um</td>
</tr>
<tr>
<td>T1-3</td>
<td>4.803</td>
<td>7 nm</td>
<td>5 min</td>
<td>27 min</td>
<td>146 um</td>
<td>104 um</td>
</tr>
<tr>
<td>T1-4</td>
<td>4.069</td>
<td>7 nm</td>
<td>5 min</td>
<td>27 min</td>
<td>146 um</td>
<td>104 um</td>
</tr>
<tr>
<td>T1-5</td>
<td>3.505</td>
<td>7 nm</td>
<td>5 min</td>
<td>27 min</td>
<td>146 um</td>
<td>104 um</td>
</tr>
<tr>
<td>T2-2</td>
<td>13.405</td>
<td>7 nm</td>
<td>5 min</td>
<td>27 min</td>
<td>136 um</td>
<td>100 um</td>
</tr>
<tr>
<td>T2-3</td>
<td>5.352</td>
<td>7 nm</td>
<td>5 min</td>
<td>27 min</td>
<td>136 um</td>
<td>100 um</td>
</tr>
<tr>
<td>T2-4</td>
<td>5.449</td>
<td>7 nm</td>
<td>5 min</td>
<td>27 min</td>
<td>136 um</td>
<td>100 um</td>
</tr>
<tr>
<td>T2-5</td>
<td>5.191</td>
<td>7 nm</td>
<td>5 min</td>
<td>27 min</td>
<td>136 um</td>
<td>100 um</td>
</tr>
<tr>
<td>T3-1</td>
<td>9.042</td>
<td>10 nm</td>
<td>7.5 min</td>
<td>27 min</td>
<td>233 um</td>
<td>51 um</td>
</tr>
<tr>
<td>T3-2</td>
<td>8.922</td>
<td>10 nm</td>
<td>7.5 min</td>
<td>27 min</td>
<td>233 um</td>
<td>51 um</td>
</tr>
<tr>
<td>T3-3</td>
<td>10.343</td>
<td>10 nm</td>
<td>7.5 min</td>
<td>27 min</td>
<td>233 um</td>
<td>51 um</td>
</tr>
<tr>
<td>T3-4a</td>
<td>11.108</td>
<td>10 nm</td>
<td>7.5 min</td>
<td>27 min</td>
<td>233 um</td>
<td>51 um</td>
</tr>
<tr>
<td>T3-5</td>
<td>12.479</td>
<td>10 nm</td>
<td>7.5 min</td>
<td>27 min</td>
<td>233 um</td>
<td>51 um</td>
</tr>
<tr>
<td>T4-1</td>
<td>5.143</td>
<td>7 nm</td>
<td>5 min</td>
<td>30 min</td>
<td>179 um</td>
<td>104 um</td>
</tr>
<tr>
<td>T4-2</td>
<td>4.849</td>
<td>7 nm</td>
<td>5 min</td>
<td>30 min</td>
<td>179 um</td>
<td>104 um</td>
</tr>
<tr>
<td>T4-3</td>
<td>4.865</td>
<td>7 nm</td>
<td>5 min</td>
<td>30 min</td>
<td>179 um</td>
<td>104 um</td>
</tr>
<tr>
<td>T4-4a</td>
<td>4.776</td>
<td>7 nm</td>
<td>5 min</td>
<td>30 min</td>
<td>179 um</td>
<td>104 um</td>
</tr>
<tr>
<td>T4-5a</td>
<td>5.002</td>
<td>7 nm</td>
<td>5 min</td>
<td>30 min</td>
<td>179 um</td>
<td>104 um</td>
</tr>
<tr>
<td>T5-1</td>
<td>4.858</td>
<td>7 nm</td>
<td>5 min</td>
<td>30 min</td>
<td>185 um</td>
<td>105 um</td>
</tr>
<tr>
<td>T5-2</td>
<td>4.823</td>
<td>7 nm</td>
<td>5 min</td>
<td>30 min</td>
<td>185 um</td>
<td>105 um</td>
</tr>
<tr>
<td>T5-3</td>
<td>4.606</td>
<td>7 nm</td>
<td>5 min</td>
<td>30 min</td>
<td>185 um</td>
<td>105 um</td>
</tr>
<tr>
<td>T5-4a</td>
<td>4.275</td>
<td>7 nm</td>
<td>5 min</td>
<td>30 min</td>
<td>185 um</td>
<td>105 um</td>
</tr>
<tr>
<td>T5-5a</td>
<td>4.828</td>
<td>7 nm</td>
<td>5 min</td>
<td>30 min</td>
<td>185 um</td>
<td>105 um</td>
</tr>
</tbody>
</table>
4.4 Conclusion

We performed an exploratory experiment with torsional supported CI-CNT test devices to assess the efficacy of a method for finding the material’s shear modulus and to obtain some initial values. We also visually examined the mode of shear failure. Having found a semi-consistent and reasonable value for $G$, this approach appears effective. We recommend that a more systematic test of samples with various fabrication parameters now be carried out using this method in order to determine more definitive shear modulus values and to better understand how those parameters impact $G$. Future torsion patterns may also include fillets on the inside corners of the torsion bars in order to control the impact of stress concentrations.

Given the difference in values between the sample sets with the near-square (1.4:1) aspect ratio and sample set T3 with its 5:1 aspect ratio, it is possible that shear modulus measurements over a variety of aspect ratios could also lead to a deeper understanding of CI-CNT anisotropy. Since the location of maximum stress is dependent on the cross-sectional dimensions of the beam, both the numerical values and the visual inspection of the failure surfaces could be informative. We recommend the further exploration of this possibility.
CHAPTER 5. CNT CELL RESTRAINT DEVICES—DESIGN AND IMPLEMENTATION

5.1 Introduction and Background

The introduction of DNA and other genetic material into cells is a common part of transgenic microbiological research, facilitating studies of a variety of challenges, including cancer and other genetic diseases [29]. A popular method for transferring this genetic material is microinjection. Microinjection entails fluid flow through a microns-wide hollow micropipette which is inserted into the cell. During this process, the cell is typically held in place using a larger pipette through which the operator manually applies suction. This procedure, while effective, has several shortcomings. A great deal of concentration and skill are often necessary to process large numbers of cells in this manner, since the alignment of the injection needle, which is achieved manually, must be precise to avoid damaging the cell. Injection can only take place if the axes of the injection needle and holding pipette are aligned; otherwise, the off-axis force applies a moment to the cell, causing it to rotate during injection and become damaged. The cell must therefore be oriented such that the targeted section of the cell is both facing the needle and located on the axis. Finally, the operator must maintain an adequate suction force throughout the entire injection process lest the cell move or escape.

To simplify the process, shorten cycle time, and increase the likelihood of a successful injection, an external cell restraint potentially fabricated out of CI-CNT was desired. An effective cell restraint design would satisfy the needs of both a company producing the device and a microinjection operator using it. Through consultation and interaction with industry representatives and microbiologists who regularly performed microinjection, a set of customer needs was developed. These needs are listed in Table 5.1.
Table 5.1: Initial list of cell restraint customer needs.

**The restraint must . . .**

. . . be compatible with existing microinjection equipment
. . . maximize visibility of the cell to the operator
. . . be reusable
. . . operate in a fluid environment
. . . restrain cells without damaging them
. . . be simple to operate
. . . actuate reliably/consistently
. . . restrain the cell on both insertion and removal of the injection needle
. . . properly align the cell with the injection needle
. . . permit off-axis injections

5.2 Concept Generation and Selection

A brainstorming session was held with relevant attendees from the research group to generate a wide variety of concepts for consideration. Pieces of paper were made available at the meeting table. Each participant was instructed to put forth any ideas that came to them through drawing or writing. Ideas could include a complete mechanism design or a partial concept that could be integrated into a design. Discussion and interaction were encouraged in order to promote/foster the inspiration of additional concepts. All concept sheets were collected at the end of the session and were later sorted to combine identical or similar ideas. The original concept drawings can be reviewed in Appendix A.

The brainstorming session produced 38 unique cell restraint concepts. Each concept was assigned a unique identifier for ease of reference throughout the design process. A basic concept screening process was then employed to identify those concepts most worth considering in more detail. Five criteria were used to judge the concepts. A sixth criterion was used to flag those concepts we considered worth examining later but were not deemed feasible for the current design project. The criteria are presented in Table 5.2. The screening matrix is presented in Table 5.3.

5.3 Preliminary Design Refinement

From the screening results, personal judgment of the design team was used to select a handful of concepts to develop more fully. Variations on two of those concepts, C25 and H1, were
Table 5.2: Concept screening criteria.

Criteria
A Manufacturability using CNT method
B Gripping ability
C Cell accessibility
D Backwards compatibility with microinjection
E Ease of use
F Examine-later flag

Table 5.3: Screening Matrix and Results for Cell Restraint Concepts.

| Tag | C1 | C2 | C3 | C4 | C5 | C6 | C7 | C8 | C9 | C10 | C11 | C12 | C13 | C14 | C15 | C16 | C17 | C18 | C19 |
|-----|----|----|----|----|----|----|----|----|----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| A   | +  | +  | +  | +  | -  | -  | +  | +  | 0  | +   | -   | +   | +   | +   | +   | +   | +   | -   | -   |
| B   | +  | 0  | +  | +  | +  | +  | 0  | +  | +  | +   | +   | +   | +   | +   | +   | +   | +   | -   |
| C   | +  | +  | +  | +  | -  | 0  | +  | +  | +  | +   | +   | +   | +   | +   | +   | +   | +   | -   |
| D   | 0  | +  | +  | +  | -  | +  | 0  | 0  | 0  | 0   | +   | 0   | +   | +   | +   | +   | 0   | +   |
| E   | 0  | 0  | 0  | 0  | 0  | 0  | +  | -  | 0  | 0   | +   | -   | 0   | +   | +   | 0   | +   | +   |
| F   | Y  | Y  | N  | N  | Y  | Y  | Y  | Y  | Y  | Y   | Y   | Y   | Y   | Y   | Y   | Y   | Y   | Y   |

Tot 3 3 5 4 -2 1 4 4 0 3 1 5 0 5 4 4 0 3 |1 3

also developed and included. Some concepts that scored highly in the screening were rejected after further consideration. For example, concepts C12 and C23 were rejected because their grounding points would be difficult to implement with CI-CNT. The list of selected designs can be found in Table 5.4.

Table 5.4: Cell Restraint Concepts Selected for Further Development.

<table>
<thead>
<tr>
<th>Tag</th>
<th>Name</th>
</tr>
</thead>
<tbody>
<tr>
<td>C4</td>
<td>Push hammer</td>
</tr>
<tr>
<td>C14</td>
<td>XBob Push</td>
</tr>
<tr>
<td>C21</td>
<td>Pincer</td>
</tr>
<tr>
<td>C25</td>
<td>Static hole</td>
</tr>
<tr>
<td>C25-MOD1</td>
<td>Hole with keystone</td>
</tr>
<tr>
<td>C25-MOD2</td>
<td>Lateral push hammer</td>
</tr>
<tr>
<td>H1</td>
<td>Fixed-guided pull tweezers</td>
</tr>
<tr>
<td>H1-MOD1</td>
<td>XBob pull tweezers</td>
</tr>
</tbody>
</table>

45
5.3.1 General Design Parameters

To maximize surface contact with the cell, all restraint areas were designed to be circular when engaged. The mammalian egg cells to be restrained have a typical diameter of 90 µm. Each restraint design had to successfully prevent the motion or escape of the cell in any direction when engaged. In order to accomplish this without impeding injection access to the cell, each restraint area was designed as a space that circumscribed slightly greater than 180°. The amount of wrap-around past 180° required to sufficiently restrain the cell was unknown. Therefore, multiple configurations of each design were prepared. Three configurations were created for all designs but C14: 200°, 220°, and 240°. These three configurations provided openings for the injection needle 88-µm, 84-µm, and 77-µm wide, respectively, as found by

\[ C = 2r \sin \left( \frac{\theta}{2} \right) \]  

(5.1)

where \( \theta \) is the vertex angle of the isosceles triangle formed by a chord \( C \) and the two radii of length \( r \) that intersect its endpoints.
During the project, the configurations were referred to as the 10°, 20°, and 30° configurations, respectively, referring to the angle past 180° that the device enclosed on each side of the restraint area. In this document, however, they will be referred to by their full enclosure angles. The C14 design, developed by Greg Teichert, included two configurations: a 210° enclosure and a 230° enclosure.

A description of the development process for each design is given below. A visual overview of all the designs is provided by Figure 5.1.

5.3.2 Design C4: Back-Push Hammer device

Device Description and Operation

This design, shown in Figure 5.2, consists of a structural frame and a movable shuttle, with the front end of the shuttle sized and shaped to conform to the profile of a cell. A gap in the frame, aligned with the shuttle, tapers from the shuttle’s width on the inner side down to less than the width of a cell on its outer side. A long, slender beam connects the shuttle to the frame and provides the controlled motion of the shuttle into place when actuated.
Device Design

Design C4 was developed using a pseudo-rigid body model of a fixed-free cantilever beam. A specified amount of deflection (approximately 125 µm) was chosen as the design requirement, and the beam parameters were modified using the CNT material properties determined in Chapter 2 as a guide. The geometry was developed in SolidWorks, using the beam parameters (length, thickness, tip motion path, etc.) as construction guides. Appropriate fillets were added to mitigate stress concentrations. Due to the CAD features used to create the gap geometry, only 220° and 240° configurations of this design were created.

5.3.3 Design C14: XBob Push

Device Description and Operation

The C14 design, developed by Greg Teichert, operates in a manner similar to the C4 design, but the shuttle actuation is provided by an XBob mechanism (Figure 5.3). The operator places the cell in the restraint area and then closes the restraint by pushing on the back surface of the XBob shuttle.
Device Design

The principles and guidelines presented in [30] were used to design the XBob mechanism, which is essentially a grid of compliant Robert’s Mechanisms that achieve linear movement with negligible transverse motion. The XBob was designed to permit a shuttle translation of 50 $\mu$m with a safety factor of 2.

5.3.4 Design C21: Pincer

Device Description and Operation

The pincer design—a combination of concepts C17 and C21—consists of a structural frame, two rigid, separated grasper halves, several long, flexible members, and a rigid actuation bar (Figure 5.4). A cell is placed between the grasper halves, and the tip of the actuation bar is pushed forward. The force is transferred through the flexible members to pull the grasper halves towards each other, securely restraining the cell. Following injection, the cell is released by releasing the actuation bar. The elasticity of the flexible members automatically returns the device to its default open position.
Device Design

Due to the complexity of the geometry, the PRBM method could not be used for design of the full mechanism. The results of the PRBM analyses for the C4 and C25-MOD2 designs were used to estimate rough minimum lengths of the flexible members in the C21 design. A parametric CAD sketch in SolidWorks was then used to create reasonable skeleton geometry for the mechanism. X-Y coordinates for key points in the geometry—beam ends and intersections—were then obtained from the sketch and used as keypoints in a beam element ANSYS model. The model assumed a worst-case CNT material elastic modulus of 15 MPa and employed a BEAM3 element type to accommodate the nonlinear large deflections expected during actuation. A poisson ratio of 0.33 was assumed. A 50-µm deflection was applied to the tip of the actuation bar as the sole loading condition. Using the results of the simulation, the geometry was refined by manually adjusting keypoint locations until the desired grasper motion was achieved with a maximum stress well under the expected failure stress of the CNT material. These keypoint locations then served as the skeletal framework for the final device design, which included the support frame, curved grasper surfaces, appropriate beam thicknesses (15 µm for the flexible members, 150 µm for the stiff actuator bar), and other, aesthetic finishing features.

See Section 2.4 for additional details regarding the design and analysis procedures.

5.3.5 Designs C25 and C25-MOD1: Static “Hole” Restraints

Device Description and Operation

Unlike the other designs, C25 is a static cell restraint. It consists of a simple, circular hole in a support frame that is intended to accommodate a cell (Figure 5.5a). The structure provides the support needed to prevent cell motion during injection. C25-MOD1 includes additional geometry to facilitate cell removal (Figure 5.5b).

Device Design

With the absence of moving components, the design of C25 was quite simple. The circular hole is simply the exact embodiment of the circular restraint area measurements discussed at the
beginning of this section. C25-MOD1 includes a keystone-shaped slot behind but connected to the restraint area. This addition was developed due to the expected difficulty of removing a cell from a simple hole. The keystone slot was intended to allow the operator to “blow” fluid towards the cell, producing turbulence around the cell that would theoretically flush the cell out of the containment area.

5.3.6 Design C25-MOD2: Side-Push Hammer

Device Description and Operation

This design evolved from the idea of having a C25-type pattern with an adjustable restraint area. A movable shuttle comprises half of the restraint area, while the other half remains fixed (Figure 5.6). Once the cell is placed, the shuttle is pushed laterally to secure the cell.

Device Design

The geometry of the C25-MOD2 design is functionally identical to that of the C4 design. The same equations, techniques, and tools were therefore used for the development of this design, which undergoes a deflection of approximately 25 µm.
5.3.7 Design H1: Fixed-Guided Pull Tweezers

Device Description and Operation

This design consists of a pair of cupped graspers at the end of two long, slender beams, forming a set of tweezers placed far enough apart to permit the rough placement of a cell between them without interference (Figure 5.7). The beams are sufficiently long so as to accommodate the full horizontal length of the lab’s standard cell holding pipette (approximately 1 mm). This design is actuated by pulling the holding pipette straight back following cell placement. The slender beams are attached to a movable stage, which is in turn connected to the fixed frame by several symmetric, fixed-guided beam segments that permit a controlled, spring-loaded translation. As the stage translates, cam surfaces at the front end of the fixed frame force the graspers laterally towards the cell until it is securely restrained for injection. Like the other designs, the graspers possess curved surfaces for cell containment. Upon completion of injection and release of the stage, the energy stored in the fixed-guided spring mechanism returns the device to its open position.

Device Design

The long, slender beams of the H1 design are nearly twice the length of the beam in the C4 design and required to deflect only 30 µm; therefore, the same beam thickness was utilized without further analysis. Similarly, the fixed-guided beam segments comprising the stage’s spring are approximately double the length of the fixed-free beam of the C25-MOD2 design. Fixed-guided beams are equivalent in the PRBM method to two fixed-free beams with their tips pinned together.
This design required that each fixed-guided segment deflect 25 \( \mu \text{m} \) and includes structural features to prevent deflection beyond that amount. Since the lone C25-MOD2 beam was designed for this deflection range, the fixed-guided segments, with a beam thickness of 15 \( \mu \text{m} \), were assumed to be sufficient.

In order to allow the use of the same geometry on both this and the H1-MOD1 design (which utilizes the XBob design discussed previously), a maximum actuation deflection range of 50 \( \mu \text{m} \) was selected. The cam surfaces on the frame and the graspers therefore required careful design in order to deflect the graspers the full 30 \( \mu \text{m} \). The main limitation was the minimum guaranteed resolution of 10 \( \mu \text{m} \) of the photolithography mask printing process. A gap of at least that distance was needed between cam surfaces in their initial position to ensure correct device fabrication. Because of the angled cam surface required on the grasper, the gap distance along the travel direction of the stage is 31 \( \mu \text{m} \), nearly twice that of the 18.5-\( \mu \text{m} \) gap normal to the cam surface. The actual deflection must thus take place in the remaining 19 \( \mu \text{m} \) of stage travel. The device design accomplishes this.
5.3.8 Design H1-MOD1: XBob Pull Tweezers

Device Description and Operation

This design is identical to the H1 design, except that the deflection mechanism is the XBob from the C14 design rather than the fixed-guided beam set (Figure 5.8). The grasper and beam geometry was designed to accommodate the limited range of motion of the XBob mechanism.

Device Design

With the exception of the geometry needed to frame the functional components of the mechanism, the design of this device was completed in connection with the H1 design and is detailed above.

5.4 Prototype Fabrication and Testing

5.4.1 Pattern Arrangement

To make optimal use of the space available on the wafer but still accommodate device testing, we considered several potential pattern arrangements (Figure 5.9a). The devices needed to
be accessible to both the cell holding pipette and the injection needle while fitting in as compact a space as possible. Since the devices would need to be attached to a substrate before use, the ability to separate each device relatively easily was also sought. The final arrangement consisted of two opposing rows, with three devices on each row (Figure 5.9b). Each device was fastened to the main row strut by a stem which included perforations to facilitate detachment if desired. The wrap-around configuration number was marked on the stem of each device. Each row included a larger pad at one end for handling purposes, and the two pads were connected by a thinner strut which was to be broken to separate the rows.

Previous research and experience had shown that narrow CNT features turned out better when in close proximity to other features, possibly due to the greater presence of iron [18]. For this reason, block features were included around and between many thin, isolated beams. These growth support features were not connected to anything so as to be easily discarded after fabrication.

### 5.4.2 Mask Layout

The devices were to be fabricated using the same procedure described in Section 2.2, and so a photolithography mask was required. The layout consisted of a 4-column series of repeating device patterns. The first three columns contained the 200°, 220°, and 240° design configurations,
respectively. The fourth column was a repetition of the first. The 210° C14 design was included with the 220° set and the 230° C14 design with the 240° set. At the bottom of the mask pattern, several of the earlier pattern arrangements were also included for potential future assessment. Most of these did not include growth support blocks.

5.4.3 Wafer Preparation

A 4-inch silicon wafer was prepared using the completed mask. AZ-3312 positive photoresist was used with a 4-second exposure time and 45-second develop time following standard cleanroom procedures. A second wafer was processed parallel to the first but had to be discarded due to a lithography machine malfunction.

When the mask, fabricated by CAD Art Services, Inc. in Oregon, was received, inspection revealed scattered spots and specks across the transparency, including its transparent regions. The developed wafer was examined under a microscope to determine whether these spots had affected the quality of the pattern, and it was determined that the exposure levels had been sufficient to overcome nearly all aberrations produced by the defects. Those that remained did not appear to affect pattern integrity. A shorter exposure can be expected to make their effects more pronounced.

Following photolithography, the patterned wafer was split in half in preparation for iron deposition. The wafer segment with the top half of the pattern was deposited with 7.5 nm of iron using thermal evaporation, and liftoff was performed to remove the remaining photoresist. The wafer segment was then manually split into individual samples.

5.4.4 CNT Growth and Infiltration

Two samples, a 220° set and a 240° set, were prepared following the standard furnace procedures described in Chapter 2. Growth time was 5 minutes to achieve a device height of approximately 100 µm—the rough diameter of the mouse egg cells to be used in testing. Infiltration time was 30 minutes. Figure 5.10 shows one half of the finished 220° set.
5.4.5 Device Mounting

Testing the devices required that they be fastened to an appropriate substrate. For initial tests, standard microscope slides were used. Several types of glues were tested for fastening the devices to the slides. Elmer’s glue was tried first, due to its viscosity, availability, and more likely biocompatibility with the egg cells. However, its water solubility rendered it ineffective after a couple of minutes of exposure to the biofluid in which microinjection takes place. Standard superglue was tested next but too readily wicked along the device-slide interface and ultimately immobilized operational features of the restraints. A superglue gel proved most effective, as it had sufficient viscosity to minimize wicking but would not dissolve in the biofluid. Once refined, the glue method was used as follows:

1. A line of glue is placed on the slide and allowed to dry/set, forming a ridged bump.
2. A small dab of fresh glue is placed atop a section of the ridge.
3. The back end of the device to be mounted is lowered onto the liquid glue just applied.
4. The positioning of the device is promptly adjusted until its curvature brings the front of the device into parallel contact with the substrate.
This approach allowed multiple devices to be placed in close proximity on a single slide, all positioned and oriented for testing. As noted in the procedure, the intrinsic curvature of the CNT material actually facilitated effective mounting of the devices, since placement of the back of the device on the glue ridge resulted in the functional end of the device being parallel to the substrate.

The majority of the preliminary devices were mounted using this technique. However, during the testing period, a thin, removable-backing adhesive from 3M was found that was faster and more effective for mounting the devices. Mounting involved applying the adhesive to a substrate, placing the device onto it, and then tapping the device down gently in several places until secure. The backing could be shaped prior to application to precisely control which portions of the devices would be attached—necessary to both control curvature and to keep kinetic portions of the device free and functional. This simpler approach was used to mount the C21 device and all second-round designs.

5.4.6 Evaluation of Preliminary Designs

Tests were conducted at two stations. One consisted of an optical microscope equipped with a vacuum stage to hold the slide in place. The other employed a digital microscope with double-sided tape for slide positioning. Each kinetic device was actuated using a cell holding pipette or a tungsten microprobe, both mounted on manually operated micromanipulators. Basic range of motion, device functionality, and ease of use were evaluated. Test observations for the individual designs are described in the following sections.

Design C4: Back-push Hammer device

Operation of this design proved to be one of the simplest and most straightforward. It was actuated using the cell pipette, which by its configuration is best able to apply a force in the forward direction. The flexible member deflected as expected and provided the proper alignment for the shuttle when fully actuated. Because of an imperfect mounting job, the restraint area of the device was suspended nearly 100 \( \mu \text{m} \) above the slide surface. Multiple tungsten microprobes were used to push the device down onto the substrate so that cells would not slip down below the restraint while being placed. Figure 5.11 shows the testing of the C4 device.
One downside to this design that proved to be rather common among all the designs was the difficulty in placing and retrieving the cell. To place the cell, the pipette would be used to position it above the restraint area, at which point suction through the pipette would be deactivated, allowing the cell to fall into place. Careful aim had to be employed in order for the cell to fall correctly and not drift, taking valuable time and concentration. Retrieval also proved taxing, since the presence of the mechanism prevented the pipette from coming into direct contact with the injected cell. Instead, the pipette was brought as close to the cell as possible above and behind it, and a combination of puffing and sucking was used to stir up the cell until it was caught by suction. Both stages of the injection procedure required significant user involvement and were largely dependent on skill. Additionally, a mock DNA injection run, using a tungsten microprobe tip as the injection needle
stand-in, resulted in the cell being pulled out with the needle, indicating a possibly insufficient wrap-around to restrain the cell. This design was considered less effective for those reasons.

**Design C14: XBob Push**

No C14 device was fabricated successfully until after the testing period. The design was therefore not tested directly. However, the functionality of the XBob mechanism (Figure 5.12) was validated by the H1-MOD1 design, and its restraining capability and limitations would likely have been equivalent to the C4 design, given their similar restraint area geometry.

**Design C21: Pincer**

The C21 “Pincer” design was one of the last to be tested because of the difficulty of obtaining an intact device during fabrication. This was largely due to the tenuous nature of its structure, which was easily damaged or destroyed by accident. However, once an intact device was obtained, it was tested for functionality to compare with the predicted ANSYS simulation. Due to the fabrica-
Figure 5.13: C21 pincer cell restraint in its (a) open position and (b) actuated position.

tion difficulties, this design was quickly quickly discounted as a commercially viable cell restraint device. Additionally, the significant distance between the cell injection location and the device actuation point would require unacceptable downtime to move the holding pipette and likely the microscope view. C21 was largely considered a material capability demonstration device.

Figure 5.13 shows the Pincer device in its default and actuated positions. Like C4, actuation was easy to accomplish using the cell holding pipette. Actuation of the device worked as expected, with no beam failures. A crack in the fixed end of the actuation bar fulcrum beam was noticed while reviewing videos of the procedure, but inspection of pre-actuation images revealed that the crack was present prior to testing. This location was under compressive stress but was not the point of maximum compressive stress.

**Designs C25 and C25-MOD1: Static “Hole” Restraints**

As shown in Figure 5.14b, the restraint area of the static C25 designs proved too small to accommodate a test cell. Additionally, based on the C4 design tests, placement and extraction of the cell would have proven very difficult, even with the keystone geometry.

**Design C25-MOD2: Side-push Hammer**

The C25-MOD2 “Hammer” design was the first device to be tested. Beam deflection behaved as expected, as did the rotational and translational positioning of the shuttle (Figures 5.14c
Figure 5.14: (a) Restraint area profile of the C25 device. (b) The C25-MOD1 device, which proved too small to admit a cell. (c) The C25-MOD2 “Hammer” device open to admit a cell. (c) The Hammer device being actuated using a tungsten microprobe.

and 5.14d). The device was tested with both a cell-sized latex bead and real cells. However, the design of the device required a lateral force for deflection, and it was quickly discovered that the holding pipette configuration could not apply sufficient force in that direction to actuate the mechanism. For this reason, the design was deemed less effective.

**Design H1: Fixed-guided Pull Tweezers**

The H1 device functioned as expected (Figure 5.15). The fixed-guided beams remained intact through their full range of motion and properly returned the mechanism to their default position when the actuation force was removed. The grasper beams and cam surfaces also moved properly, providing sufficient closure to fully grasp a cell during testing (Figures 5.15c and 5.15d).
A grasper strength test was conducted by pulling on the restrained cell using the holding pipette, and this indicated a reasonably good grip. A later mock injection test with a tungsten microprobe, however, resulted in cell pull-out.

An additional problem was encountered that affected both H1 designs. The devices were designed to be actuated using the cell holding pipette, which does so by pulling straight back after placing the cell. Because of the curvature in the bent pipette, however, the pipette tended to deflect upwards onto the top of the device rather than apply force laterally to the mechanism. This essentially rendered the device nonfunctional in the intended working conditions. Thus, while the cam-based grasper design worked very well and would likely have restrained the cells with greater wrap-around, the actuation mode proved ineffective.
Figure 5.16: Actuation of an H1-MOD1 cell restraint using a tungsten microprobe.

**Design H1-MOD1: XBob Pull Tweezers**

Cam geometry and actuation method being identical, the observations and conclusions made while testing the H1 design apply also to the H1-MOD1 design. The only difference is the actuation mechanism, which for the H1-MOD1 was the XBob mechanism. The XBob proved much stiffer than the fixed-guided beam stack but moved as predicted (Figure 5.16). Linear motion was achieved through the full actuation range of the device. Furthermore, one of the beams on the mechanism was broken before testing, yet it still worked properly because of the redundancy in the design.
5.4.7 Evaluation Outcomes

Analysis of testing results produced several useful conclusions with which to refine our approach. First, the limitations of the injection equipment were more fully realized. The cell holding pipette, which serves as the actuation tool, could only effectively apply force in the forward direction. Any successful design’s actuation mechanism would need this to be its sole input. Second, it was recognized that the actuation point must be as near as possible to the restraint area to make operation of the device easy and convenient for the user. Third, direct pipette access to the restraint area made cell placement and retrieval must faster and easier, while having device features block such access greatly increased the time and skill needed to successfully interact with the cell. Fourth, more intricate designs, such as the C21 and XBob-based devices, had poorer yields, while the less intricate and more robust devices consistently turned out well, highlighting one of the benefits of design simplicity. Fifth, the double-sided adhesive was found to be a superior mounting technique to the glue-based approach, enabling more precise fastening and also providing a way to counteract the stress curvature of the CNT material. Finally, the 240° wrap-around was the best of the options at restraining the cells but was still not sufficient.

5.5 Second-Round Device Design

Using the results from device testing, a new set of designs was developed. Following testing, three new restraint designs were developed based on the information gathered. These designs are described below.

5.5.1 Grasper Mechanism Design

The mechanism termed the “Grasper” (Figure 5.17a) was a redesign of the H1-MOD1 mechanism. The tweezer beams were moved to the stationary outer frame, and the cam surfaces were attached to the front of the mobile stage, effectively inverting the operation of the device. The stage was reconfigured as a push-actuation mechanism, with its length increased and a center space provided to accommodate the cell holding pipette. One side of the space included a flat surface for the pipette to push on, while the other side provided a slanted surface along which to roll a cell to reorient it if desired. The successful XBob pattern from the preliminary designs was reused,
with the spacing increased to better stabilize the larger stage. The cupped grasper surfaces were modified to provide greater wrap-around than those of the preliminary designs. The grasper beams were angled outward to provide ample injection working space for the operator. The graspers’ cam surfaces were redesigned to achieve full closure with less stage travel and maintain a fixed closure distance beyond the required input.

An additional experimental feature for organizing pre- and post-injection cells was incorporated into the Grasper design. A series of grooves or channels, informally termed “bowling racks” by the design team, were placed on either side of the injection area. The intent was to provide a feature where cells could be placed and expected to remain in an organized fashion until retrieved. With a width approximately half that of a typical egg cell, the grooves were intended to accomplish this by utilizing gravity to cause contained cells to sit slightly lower in the grooves than the surrounding surface, preventing them from readily drifting or rolling away with incidental fluid motion.

5.5.2 Viper Design

The “Viper” mechanism design (Figure 5.17b), so named for its snake-like appearance, was a unique cell restraint design intended to restrain the cell by pinching it between two flat
surfaces: the device’s mobile head and its stationary gate. The design would also allow an operator to reorient the cell by rolling it between the flat surfaces until the desired positioning was reached. The folds in the flexible beam were designed to be sufficiently flexible to allow the head relatively free movement and large range of motion. The head was designed with a flat surface for actuation by the holding pipette. An angled surface on the opposite surface of the head allowed the head to slide along one side of the gate, producing controlled, nonrotating movement.

5.5.3 Passive Restraint Design

A third design was conceived which arose while examining some broken C14 devices. The XBob mechanism and stage were missing, leaving only the outer frame. The suggestion arose of a restraint device consisting solely of such a frame with a narrow, tapered gap in it that would restrain the cell in three directions. The holding pipette itself would provide restraint in the fourth direction. Once the cell was placed in the restraint area, the pipette would be left in place, and no additional actuation would be required. The frame would also provide ample working space for the pipette. The device would be a passive restraint after the manner of preliminary designs C25 and C25-MOD1. Implementations of this design can be seen in Figure 5.18.
Because the restraint device would not grasp the cell with a cylindrical surface as the other designs did, it was determined that the amount of enclosure would be measured in linear “aperture width” rather than degrees of wrap-around. The aperture width was defined as the width of the narrow end of the taper. The narrowest aperture width of the preliminary designs, approximately 78 µm in the 240° configuration, was unable to reliably restrain a cell, so the aperture width on the new restraints needed to be narrower. However, the aperture had to also remain wide enough to provide the injection needle easy access to the cell. To help determine the best aperture width, three widths—50 µm, 60 µm, and 70 µm—were decided upon for testing.

The tapered sides of the gap forming the restraint area would prevent lateral movement of the cell. The taper would also make it possible for the restraint to work with cells of various sizes. Out of concern that too steep a taper would make it more likely that a cell could become jammed in the restraint, three levels of taper were selected for testing. These levels, measured as the angle subtended by two sides, were 30°, 40°, and 50°. Combined with the aperture width, this produced nine unique restraint area configurations to test.

In connection with the passive restraint design, the cell organization concepts were explored further. One design (Figure 5.18b) was prepared that included the “bowling rack” patterns of the Grasper device. A second configuration (Figure 5.18a) explored the use of two cell “corrals”—partitioned open spaces where cells could be divided into pre- and post-injection clusters. This configuration was nicknamed the “Castle” restraint device due to its appearance.

5.6 Second-Round Device Fabrication and Testing

The new restraint devices were arranged into pattern blocks to facilitate testing. One block consisted of several identical copies of the Grasper design facing one direction and several identical copies of the Viper design facing the other direction. A second block consisted of the nine Castle passive restraint configurations facing outward in three directions from a central supporting area. A third block consisted of the nine “bowling rack” passive restraints in the same configuration. These blocks are visible post-fabrication in Figure 5.19. Because the blocks were included with other devices on a mask designed to test a sacrificial-release method for CNT structures, etch holes were incorporated into the large solid areas of the patterns.
Device fabrication followed the same procedure used with the preliminary designs: photolithography on a wafer, followed by iron deposition and liftoff, followed by CNT growth and infiltration via CVD in a furnace. Issues with the furnace temperature sensor caused issues in the first several samples. Once the issue was resolved, good samples were obtained. Figure 5.20 provides optical microscope images of the four designs.

Upon fabrication, the pattern blocks were fastened to a substrate for device testing. The pattern blocks were designed to be used with the 3M adhesive, though limited space on the wafer prevented ideal layouts. Where necessary, the adhesive profile was shaped to avoid kinetic features. Applying and keeping fluid atop the preliminary designs mounted to flat microscope slides proved difficult and often resulted in significant viewing distortion due to refraction through the puddle’s curved surface. The second-round devices were therefore mounted inside 1.5-inch diameter standard petri dish lids (Figure 5.21), providing ample fluid containment and a flat fluid surface.

5.6.1 Evaluation

The second-round cell restraint designs were examined and evaluated as described below.
Grasper Design

The grasper stage and beams all functioned successfully and as designed. Stage motion was initially hampered during testing due to the petri dish not being adequately secured prior to device actuation. The forces applied by the pipette and the stiffness of the XBob device were sufficient to move the entire dish before the device would actuate. Additionally, the thickness of the adhesive layer, combined with the remaining curvature of the CNT material, suspended the restraint area of the device at least 100 µm above the bottom of the petri dish. This made it difficult to apply the needed force with the cell pipette without its tip sliding off of the actuation surface and under or over the device. Once the petri dish was secured, actuation was achieved using a tungsten...
Figure 5.21: Castle restraints mounted to the inside bottom of a petri dish using the 3M adhesive.

Figure 5.22: Functional testing of the Grasper cell restraint.

microprobe (Figure 5.22). Mock injections were not performed using the Grasper device, since the Castle restraint had already proven more effective by the time the Grasper functionality was tested.

**Viper Design**

The Viper restraint device moved as predicted. Since the device was suspended above the substrate like the Grasper device, pushing it down closer to the substrate with additional micromanipulators helped keep cells from slipping beneath the device. However, low out-of-plane stiffness
made the device head slide off the pipette tip easily during actuation, sometimes causing it to get caught on the device frame. This proved to be very troublesome during testing, so a microprobe was used to successfully actuate the device.

Once a cell was placed and the device actuated fully, the head properly contacted the cell and rolled it along the gate surface (Figure 5.23). This was accomplished with multiple cells. Mock injection was attempted in an effort to verify the effectiveness of the restraint while experiencing injection forces, but alignment problems with the mock injection needle prevented it from being able to reach the cell.

**Passive Restraint Designs**

Because significant interest was placed in this design by the sponsoring company, these devices were tested first. Mounting of the passive restraints was very straightforward. Without any moving parts to accommodate, the entire restraints could be placed straight onto the adhesive, removing any possibility of cells being able to slip underneath the structure and counteracting all intrinsic curvature. No special preparation of the adhesive profile was necessary.

Measurement of the restraint area geometry of the nine castle patterns confirmed that fabrication produced the expected aperture sizes and taper angles. Mock injection testing was conducted with standard egg cells on the 70-µm/50° Castle restraint. Placement of the cell was very quick and straightforward. The restraint successfully prevented cell movement throughout the entire in-
jection process, including needle retraction (Figure 5.24). Furthermore, when off-axis injection was simulated, no rotation of the cell was observed. Removal of the cell following injection was just as quick and straightforward as placement.

Several Castle restraints were tested to examine the effects of the varying taper angles and aperture widths. With the 50° taper angle, no tendency of the cell to jam within the restraint area was observed. Some difficulty was experienced with the 30° angle on the 70-µm device. The 70-µm aperture width was found to be sufficiently narrow to prevent cells from being pulled out during needle retraction. The 60-µm aperture width provided the same capability but felt substantially more restrictive to use and precluded easy off-axis positioning. The 50-µm aperture designs were not tested, since it was expected that these issues would be even more pronounced on those configurations.
5.6.2 Testing Outcomes

While all three restraint designs generally functioned as expected, the results quickly showed that the passive restraint design was the most effective and could best meet customer needs. Without moving parts, functionality was robust and easily achieved. Placement and retrieval of the cell were very straightforward, relying on skills already possessed by the operator. Restraint of the cell required no further action on the part of the operator following placement. Injection was also straightforward, with off-axis injection accomplished just as easily by simply repositioning the needle. Total injection cycle time was very short due to the use of the pipette in the restraint process. Even if the other designs had functioned exactly as expected with no complications, they would still have required more time and user actions per injection than performing the same injections using the passive restraints. The passive restraint design was therefore selected as the final, most effective cell restraint device.

5.7 Conclusion

A prototype-centric product development process was used to develop an effective cell restraint device to facilitate microinjection. CI-CNT was used for fabrication due to its ability to form high aspect-ratio devices, provide sufficient flexibility for the ranges of motion desired, and allow rapid fabrication of designs. Design requirements were identified through interaction with experienced microinjection operators and were refined through the evaluation of preliminary designs. They were then satisfied in a final restraint design which was delivered to the customer.

After further consideration, the company decided to utilize a more conventional MEMS fabrication process to produce the restraints, largely due to concerns regarding production ramp-up. This is understandable, considering the wider availability of such processes and the as-yet incomplete characterization of CI-CNT and its fabrication procedures. Nevertheless, this cell restraint development project demonstrated the utility of using CI-CNT for rapid and iterative prototyping of high aspect-ratio compliant MEMS devices. Both the swift concept-to-prototype turnaround time and low fabrication cost relative to conventional MEMS technologies encouraged frequent design iteration, liberal testing, and increased willingness to innovate and explore high-risk possibilities.
CHAPTER 6. QUALITATIVE EXPLORATION OF CI-CNT IN-PLANE AND LEM DESIGN

6.1 Introduction

As discussed at the beginning of Chapter 4, lamina emergent mechanisms (LEMs) are mechanisms that are fabricated from a planar material that produce out-of-plane motion. While exploring the torsional properties of CI-CNT is important for enabling the systematic design of LEMs from the material, we decided to attempt some preliminary designs using the known material properties (elastic modulus and maximum strain) by employing some assumptions. The first assumption was that CI-CNT is isotropic. The second was that its Poisson ratio \( \nu \) was 0.33. These assumptions, though known to be erroneous to at least some degree, provided a starting point from which to design. To compensate for the use of these assumptions and the variability in the known properties, a large safety factor was used.

Using these design guidelines, we first modeled the basic geometry of a LET joint in ANSYS and tuned the parameters to permit the desired motion. We then used that and other building blocks to create a variety of meso-scale LEM devices that could be fabricated using CI-CNT for qualitative testing and observation. Our objectives were twofold: (1) to assess the feasibility of making LEMs using CI-CNT, and (2) upon successful fabrication, to provide visual demonstration of CI-CNT being used in that capacity.

This chapter discusses the design work that led to the development of these CNT-LEMs and provides results of testing some of the devices.

6.2 Design Summaries

We created several different designs to showcase CI-CNT out-of-plane LEM behavior as well as device design in general. Two basic building blocks were designed first that were utilized
in the various devices. Those are discussed first, after which a brief description of each device is given.

6.2.1 Bistable Beam Array

We used a fixed-guided beam bending MATLAB script based on elliptic integral calculations to design an array of bistable fixed-guided beams. The beams were given a 25-µm thickness in the direction of motion, a 1100-µm length, and a 100-µm width. An elastic modulus of 5 GPa was used. The pre-deflection beam angle $\gamma$ was set at 8 degrees. This configuration produces a bistable beam design that switches to its second position under a 2.6 mN force and deflects approximately 200 µm. The maximum stress experienced by the beam is 70 MPa, or about 65% the expected ultimate strength for a 7-nm Fe CI-CNT device. Figure 6.1a shows the force-deflection curve, and Figure 6.1b shows the corresponding stress-displacement plot. This design was used to create an eight-beam bistable device—four beams on either side of a translating shuttle block—that would require a total force of about 21 mN to actuate.

Figure 6.1: Force and stress vs. displacement for the CI-CNT bistable beam in standard SI units.
6.2.2 LET Joint

The key element of our demonstration CI-CNT LEMs was a LET joint that would enable out-of-plane motion. Using the assumptions stated in Section 6.1, we created an ANSYS model for use in designing a LET joint appropriate for CI-CNT. Once again, an $E$ of 5 GPa was used, as was a Poisson ratio of 0.33. Taking advantage of symmetry to reduce the processing load, the model consisted of only half of the joint, as shown in Figure 6.2. By manually adjusting the geometry, a design was developed that could ideally rotate 45° out of plane with a maximum stress of 59.2 MPa—approximately 65% of the expected ultimate strength. ANSYS calculated that a LET joint with beams 650 µm long, 100 µm tall, and 25 µm thick would accomplish this.

6.2.3 Bistable Threshold Accelerometer

Perhaps the simplest design developed was a CI-CNT threshold accelerometer—a device that would, in theory, switch bistable positions upon experiencing a specified level of acceleration. While not a LEM, we still wanted to test the bistable beam array in this potential application. CAD models of an accelerometer design using the beam array are shown in Figure 6.3. A larger pad is
connected to the shuttle to facilitate actuation. Holes were included to allow mounting on a test platform using pins if desired.

### 6.2.4 LEM “Prop-Up” Device

One basic LEM design we created was a simple 4-bar crank-slider with an extra protrusion on the coupler link (Figure 6.4). When the device is actuated, the protrusion would be lifted up above the rest of the device. It could be considered the first stage of a Nuremberg scissor mechanism. The intent in this case was to lift the bistable mechanism attached to the protrusion high enough that it could actuate above the entire device. When actuated, a portion of the shuttle would overlap with the crank-slider links. The overlap would, it was hoped, prop the device up in an actuated position when the actuation force was removed. A variation of the device was developed with a second bistable mechanism built into the surrounding frame that could be switched once the rest of the device was actuated, the intent being to prevent it from returning to its default change-point position.
6.2.5 Bricard 6R Linkage

Previous research has demonstrated the successful fabrication and operation of a LEM Bricard 6R linkage [31]. The successful fabrication of a similar design from CI-CNT would be a compelling visual demonstration of the material’s capability and flexibility. Figure 6.5a shows the mechanism we developed. Since the range of motion of each joint in the mechanism far exceeded the designed capabilities of the CI-CNT LET joint, three “inside” LET joints were used in series for the joints in the middle of the sides. A snaking, thin, flexible beam was placed on the corners to handle the required motion there. One link of the linkage was connected to a larger frame to make handling and actuation easier.

6.2.6 Pivoting Ball-Socket Joint

The mechanism in Figure 6.5b is a simple CI-CNT ball-and-socket joint designed to test whether such a joint would be feasible. The beams connected to either side of the joint have the same dimensions as the beams in the bistable mechanisms to potentially make the joint bistable.

6.2.7 Bi-Layer Clamp

Additional design concepts arise when the notion of combining multiple CI-CNT layers is considered. On the macro-scale, such a proposition would be relatively straightforward, since
bonding materials and techniques are readily available. On the meso- and micro-scale, fastening layers can be both labor intensive and challenging. Nevertheless, we were interested in the added possibilities that eliminating this constraint could offer.

The first two-layer device we devised of was a clamp. Two simple crank-sliders, similar to the one in the prop-up device but with an extra link in the middle, would be stacked on top of each other. The two slider links on the linkages’ free ends would be stacked and fastened together in some manner. The mechanisms’ frames would be joined as well, possibly with an intermediate
spacer between them. The two halves of the resulting bi-layer device would move symmetrically when actuated, moving the two intermediate links in each crank-slider away from each other. With all links being of equal length, the intermediate links would not rotate but remain parallel to each other at all times. This configuration could be used as a tiny clamp in which other devices or components could be held.

A spacer frame with the same dimensions as the mechanisms’ frames was included with some of the devices on the photolithography mask. Inserting this frame between the two device layers was intended to move them away from their change-point positions and make initial actuation easier and less likely to damage the LET joints due to compression along the links.

6.2.8 Bistable Switching Sign

Bi-layer devices that do not move out-of-plane could be created as well. One such device is a bistable switching sign. In this design, a shuttle is placed between two bistable mechanisms. The shuttle possesses a series of slots at regular intervals running perpendicular to its direction of motion. The static layer beneath the mechanism has rows of small holes distributed across its main area. The rows of holes are spaced such that some rows are visible through the shuttle slots when the device is in default position while others are visible when the device has been actuated. The sets of holes could be arranged to spell two different words (“OFF” and “ON” in this case) or show two different geometric symbols (such as a square and a triangle).

6.2.9 Circle-Back LEM

Figure 6.8 shows the design of a simple but ambitious CI-CNT LEM. It is essentially a long chain of links connected to each other by inside LET joints. The last link is terminated by a pair of long, flexible beams designed to act as clips, allowing the chain to curl back towards its anchor and clip into a slot in the frame, leaving it standing above the substrate under its own support. Its purpose was to show the possibility of creating and assembling self-supporting structures based on CI-CNT LEMs.
Figure 6.7: (a) Trimetric CAD view of the two layers comprising the bistable switching sign. (b) Simulated view of a message pattern viewed through the top grating. The holes visible in the bottom layer spell the word “OFF.”

Figure 6.8: Top and trimetric CAD views of the circle-back LEM pattern.

6.2.10 Foldable Boxes

Another pair of designs intended to demonstrate assemblable, self-supporting LEMs can be seen in Figure 6.9. These are essentially foldable boxes, consisting of stiff segments joined by three series LET joints and framed by interlocking clips and slots. The device in Figure 6.9a most closely resembles the layout of a conventional foldable box, while Figure 6.9b shows a simpler folding structure that forms a triangular prism.
6.3 Testing

6.3.1 Bistable Threshold Accelerometer

Several CI-CNT accelerometers were successfully fabricated. Figure 6.10a shows a scanning electron micrograph of one of the accelerometers. Figures 6.10b and 6.10c show an accelerometer in its default and actuated positions. Bistability was achieved, though imperfectly and with very little room for error. It is evident in Figure 6.10c that at least one beam broke prior to reaching the bistable position, and one clearly failed to switch. Other accelerometers, as well as bistable arrays on other LEM designs, either broke or failed to exhibit bistable behavior. With such a small holding force for the secondary bistable position, deviation of the beam geometry from the design during fabrication is most likely the cause. The presence of a substantial floor layer may also have interfered with the device’s operation. Optimizing the configuration of the fixed-guided beams for a more equal actuation force and improving fabrication quality would produce a more robust design.

6.3.2 LEM “Prop-Up” Device

Two prop-up LEMs were actuated using tungsten microprobes on manually operated micromanipulators. One microprobe was used to hold the slider in sliding contact with the substrate,
Figure 6.10: (a) Scanning electron micrograph of a CI-CNT bistable threshold accelerometer. (b) and (c) Accelerometer in its default and stable switched positions, respectively.
Figure 6.11: (a) Prop-up device being actuated by three microprobes (right). The substrate is being held in place by an alligator clamp (left). (b) Optical microscope view of the LET joints allowing the prop-up device to actuate. The protrusion, with its bistable device, is visible on the right.

while another was used to actuate the device. A third microprobe was used to assist the device out of its change-point position to prevent compression from destroying the LET joints.

As Figure 6.11 shows, the prop-up LEM actuated properly, though one LET joint failed on the first LEM before the protrusion had risen high enough to try engaging the holding bistable device. (Even so, the bistable device proved not to be bistable.) On the second LEM, the stiffness of the bistable device was too great to actuate it without applying a destructive lateral force on the nearby LET joint. Clearly, any bistable device on the emergent portion of a LEM would need to have a very low stiffness to avoid damaging the mechanism during actuation. Alternatively, an external counter-force would be required to brace the device while the bistable device is actuated.

As noted above, a LET joint on the first device failed before actuation was completed. This happened because device was too thick, causing stress to build faster than intended in the torsional beams of the LET joints. This highlights the importance of using thin CI-CNT layers in LEM applications. Unfortunately, thin layers are more fragile, and the thinner bistable mechanisms were more difficult to actuate without the shuttle slipping out of plane away from the probe tip. Clearly, tradeoffs must be made when both elements are combined, and an appropriate balance or an alternative design must be found.
6.3.3 Bricard 6R Linkage

No Bricard 6R linkage emerged completely intact from the fabrication procedure. The thin features attached directly to the large frame broke when the frame curved under intrinsic stress. Placing the linkage-frame connection near the center of a side of the frame rather than on a corner would have had a more favorable outcome.

While the linkage as a whole was inoperable, a portion of it remained intact, so the functionality of the three series LET joints was tested. Figure 6.12 shows the joint in both flat and highly deflected positions. The test showed that the series LET joints very effectively enabled high out-of-plane rotations. The joint was deflected to approximately 135° without failing. The joint was not tested to failure in order to preserve it for further examination and testing.

6.3.4 Pivoting Ball-Socket Joint

We fabricated two devices that included the ball-and-socket joint in their frames, but in both instances the ball and the socket were fused together. Testing one in hopes of breaking it free merely resulted in the attached beam breaking. The 20-µm gap between the two halves was minimized to avoid unnecessary play in the joint, but it is evident that a greater space is required unless fabrication quality can be improved. Its usefulness as a CNT-MEMS building block remains undetermined.
6.3.5 Bi-Layer Clamp

The bi-layer clamp has yet to be tested in its assembled state, though the actuation of the individual layers would behave in a similar manner to the prop-up LEM.

6.3.6 Bistable Switching Device

The switching device has yet to be tested. The bistable beams in this design being identical to those in the other designs, the device may or may not behave in a truly bistable manner.
6.3.7 Circle-Back LEM

Three circle-back LEMs were tested. The first was from a poor growth that was overly fragile. It was used for practicing the tricky manipulations that would be needed for complete assembly. Its tip achieved a $90^\circ$ rotation before failure (Figure 6.13a). The second device was one that had turned out well. However, part-way through the assembly process, the operator bumped one of the micromanipulator arms, resulting in the premature destruction of the device. The third device, also a good sample, reached a full $180^\circ$ rotation before an intermediate LET joint failed (Figure 6.13b), likely due to a fabrication defect, seen on the device before testing, that fused part of a LET joint together. The tip of the remaining four links was deflected an additional $30^\circ$ before the LET joint at the frame failed (Figure 6.13c).
6.3.8 Foldable Boxes

The triangular-prism foldable box was successfully assembled. The folding process is shown in Figures 6.14a–6.14d. Figures 6.14e and 6.14f show the completed structure. Two microprobes were used to fold the two side flaps and then keep them upright while a third microprobe folded the main flap into position. With only a little difficulty the slots slid over the clips on the side flaps. The microprobes bracing the sides were then removed, followed by the one supporting the main flap. The clips successfully prevented the structure from unfolding. The clips are visible on the left edge of the side flap tips in Figure 6.14f.

One folding box specimen was prepared with a 500-µm thickness during the fabrication of a separate device for another project. Part of the sample was broken, but at least one series LET joint remained. The joint was deflected to see how well a 0.5-mm thick set of torsional beams would perform. Visual inspection suggests the attached section had rotated at least 60° before failing (Figure 6.15). By that point, the compressive side of the LET joints were all in physical contact, most likely causing the stress on the tensile and bending regions of the joint to rise much faster than before. Still, achieving a 60° rotation with such a thick sample is quite remarkable and showcases CI-CNT’s simultaneous flexibility and high aspect ratio well. This serves as further demonstration of the impressive range of motion of the three inside LET joints in series.
6.4 Conclusion

Several CI-CNT in-plane and LEM designs were designed from basic building blocks, fabricated, and tested. Tests showed varying degrees of success but proved very informative as to the uses and limitations of CI-CNT in such applications.

A number of insights were gained through this experimentation. First, it appears that some mechanism components, such as the bistable beams and the ball-socket joint, need further design refinement before they could be integrated reliably into CI-CNT mechanism designs. On the other hand, LET joints were successfully implemented in CI-CNT and achieved a good range of motion. The multi-LET joints, which consisted of three LET joints in series, were the most successful joints utilized. They provided significant ranges of motion in a surprisingly compact space. Indeed, they were compact and flexible enough to allow the creation of folding structures. Just as they do on the macro-scale, LET joints could enable the creation of a wide variety of CI-CNT mechanisms. Those tested and described in this chapter are only the beginning.

Notwithstanding our success in mechanism actuation and structure assembly, we also observed how difficult it is to operate and manipulate devices on this scale. Out-of-plane motion was achieved, but only through extensive micromanipulator involvement. Successfully coordinating multiple micromanipulators in such close proximity and around such delicate structures is very challenging and requires significant time, focus, and skill from the operator. Several samples were destroyed during testing due to inadvertent contact with a micromanipulator rod or the platform supporting the device. The manipulation abilities of the microprobe tips are also a significant limitation. Using them to actuate devices could be compared to washing dishes with one’s feet while wearing shoes: the basic ability to move and hold objects exists, but no fine control is possible, and the process must be planned out in advance and well coordinated during execution in order to be successful. Some devices included features intended to make operation easier (such as the notch in the prop-up actuation point visible in Figure 6.11b), and they did make a difference. Including more such features (like tabs and holes) as specific actuation points, or planning the layout of a device with actuation modes in mind, would produce much more successful designs.

Expanding the tools available for device manipulation would also be useful in future work. While elaborate articulating manipulators would be nice, a simple set of microprobes with varying surface geometry would make a significant difference. The existing tungsten microprobes used in
testing these devices all have very smooth shafts and sharp tips. A probe tipped with a wide bar or flat face would make it much easier to push on a feature without the probe slipping off of it. Additionally, a spade-like flat probe would facilitate getting beneath a feature to push it upward. Probes coated with a material with a high friction coefficient (such as rubber) would also be very useful, since the features of existing probes could be much more useful if their surfaces were able to apply a lateral force on the CI-CNT features using friction. Tool enhancements such as these could be made in-house, and the time invested in their creation would almost certainly pay off in the increased dexterity they provide.

Overall, this experiment revealed that there are many possibilities for unique and capable CI-CNT LEMs. The ones described here could all be considered meso-scale devices, but with a higher-resolution mask, the same mechanism components could be further miniaturized to create true MEMS-scale devices. The handful of prototypes discussed in this chapter show that the CI-CNT mechanism design space is rich, with many possibilities waiting to be explored.
CHAPTER 7. CONCLUSION

This thesis has presented several material properties of CI-CNT relevant to compliance, demonstrated its viability as a MEMS material through the development of numerous devices, and showcased CI-CNT LEMs to illustrate the capabilities this material adds to the MEMS design space. A list of specific contributions is given below:

• Values for CI-CNT elastic modulus and maximum strain

• An understanding of how the iron layer thickness and infiltration time used to prepare CI-CNT devices influence elastic modulus and strain

• Several viable cell restraint devices intended to aid and simplify cellular microinjection, demonstrating an iterative design process that can be carried out with CI-CNT

• A passive cell restraint structure which has been further refined and marketed as a product by NanoInjection Technologies, Inc.

• Initial values for a CI-CNT shear modulus

• The development of a viable process for determining the shear modulus of CI-CNT and CNT-M materials in general

• A quantitative and visual comparison of CI-CNT and single-crystal silicon flexibility

• The development of a CI-CNT LET joint design that enables the creation of CI-CNT LEMs

• A demonstration of CI-CNT’s capabilities through the design, fabrication, and testing of several devices, including LEMs.
7.1 Future Work

While CI-CNT has been examined and successfully used as a MEMS material, this work has only begun to explore the possibilities it presents. Numerous opportunities remain for further understanding and utilizing CI-CNT. Some potential future work is described in the following sections.

7.1.1 In-Plane Elastic Modulus

We mentioned in Section 3.4 that recent tests had produced very different elastic modulus values in the in-plane direction than were determined from our out-of-plane measurements. To investigate this further, beams of known dimensions should be tested by deflecting them in both in-plane and out-of-plane directions. Analyzing the force-deflection data as was done in Chapter 2 would reveal whether there is indeed a difference. We developed a test pattern to do this, but data has not yet been collected and analyzed. The pattern includes beams of various thicknesses to help determine whether and to what extent completeness of infiltration affects modulus and strain. The results of this experiment would help refine the known material properties of CI-CNT when actuated in directions most relevant to MEMS applications.

7.1.2 Shear Modulus

Several areas worth exploring exist related to the shear modulus work described in Chapter 4. First, an ANSYS model of a torsional cantilever was partially developed that could be completed to more accurately model the tests conducted and calculate the shear modulus. Second, a more systematic experiment using torsional cantilevers could be conducted to better pin down values for a CI-CNT shear modulus. Using a design of experiments approach similar to that used in Chapter 2, any relation between fabrication parameters and the shear modulus could be identified. Knowing this relation would better inform anyone intending to design CI-CNT LEMs, as they could select the best fabrication parameters accordingly.

Third, as mentioned in Section 4.4, the possibility exists that insight into the amount of anisotropy in CI-CNT could be gleaned by conducting torsion tests using torsional bars of various aspect ratios. The measured modulus as well as the appearance of the fracture surface could
be analyzed to achieve this. The relationship between the aspect ratio and the various stresses experienced by a given stress element in the torsional bar would have to be explored in greater detail.

Finally, the torsion test method may have inadvertently provided a way to investigate the strength and influence of the thin carbon floor layer film typically attached to fabricated devices. The following procedure was conceived after testing of these samples had concluded. A sample with floor layer present could be loaded bottom-side up in the testing apparatus. The beams could first be loaded to a pre-failure level and then released. Then, using a tungsten microprobe on a micromanipulator, the floor layer in the gap between the torsion bars and the base could be torn so that it would no longer transmit a load between the two. The beams could then be deflected again and any differences in the force-deflection curves analyzed. If successful, this procedure could be used not only to determine whether the floor layer has an impact but to also provide a way of quantifying differences in the floor layer developed under different fabrication conditions. Better understanding the floor layer could improve understanding of the underlying mechanics of carbon infiltration.

7.1.3 Refined Material Properties Design of Experiments

Now that the process for preparing and fabricating CNT devices has been refined and streamlined, the uniformity of the properties in the resulting structures is likely greater. With this reduced variability, the opportunity arises for a more in-depth exploration of the fabrication parameters on all mechanical properties of CI-CNT. Specifically, center points could be added to the $2^k$ factorial design used in Chapter 2 to uncover nonlinearities in the responses. If nonlinearities are discovered, more elaborate statistical methods (such as a central composite method) could then be implemented to obtain a full response surface for the parameters of interest. Having a response surface would make it possible to interpolate the precise parameter settings that would produce the most compliant material. The necessary additional test points for both of these approaches could be added to the existing data, or new tests at all points could be conducted to ensure data integrity.
7.1.4 Material Properties Universal Test Pattern

In connection with refining the understanding of CI-CNT material properties, a device pattern that integrates all of the features used to test the various properties into one universal set would be very useful. Not only would this place all test features conveniently in one piece of CI-CNT, but it would also improve data consistency, since properties would vary less within a single piece of CI-CNT than they would across multiple pieces prepared separately.

7.1.5 Additional Areas of Exploration

Some additional areas exist that may be worth exploring. Listed in no particular order, these areas include:

- Implementing the LEM designs presented in this thesis on a smaller scale using the new photolithography mask fabrickator being installed in the cleanroom
- Developing improved micromanipulator tips to facilitate future mechanism actuation, as discussed in Section 6.4
- Investigating other material properties of CI-CNT, including thermal and electrical properties
- Exploring the uniformity of CI-CNT produced along the entire length of the furnaces used for growth and infiltration, using modulus to gauge the quality of material at different points
- Assessing tensile and torsional failure surfaces in greater detail to ascertain the exact mode of failure and its implications regarding the nature of CI-CNT
- Using mechanical or focused-ion beam cutting to closely examine the cross-section of a CI-CNT sample and better examine degree of infiltration and void geometry.

In the end, there is much that could be explored as far as carbon-infiltrated carbon nanotubes are concerned. Since it would require a great deal of manpower to accomplish all of the ideas outlined above, not all of these ideas may be implemented. Any lines of investigation that are pursued will only add to the knowledge and tools already developed that make CI-CNT a promising new material for microfabrication of compliant devices.
REFERENCES


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APPENDIX A. CELL RESTRAINT DEVICE BRAINSTORMED CONCEPTS

A.1 Concepts

This appendix contains scans of all the concepts generated during the initial brainstorming session for the cell restraint project. Included on each sheet is the date, the initials of the person who created the concept, and a drawing (sometimes annotated) depicting the concept. A unique identifier label was added to each sheet after the fact to enable direct reference to each concept in an organized manner. It appears in the form of either the letter C or the letter H, followed by a number. In this appendix, two scanned sheets are presented on each page. The initials of those who participated are listed in the following table.

Table A.1: Cell Restraint Brainstorming Session Participants (Names and Initials)

<table>
<thead>
<tr>
<th>Initials</th>
<th>Name</th>
</tr>
</thead>
<tbody>
<tr>
<td>QA</td>
<td>Quentin Aten</td>
</tr>
<tr>
<td>MAE</td>
<td>Melanie Easter</td>
</tr>
<tr>
<td>WCF</td>
<td>Walter Fazio</td>
</tr>
<tr>
<td>HG</td>
<td>Holly Greenberg</td>
</tr>
<tr>
<td>GH</td>
<td>Greg Holst</td>
</tr>
<tr>
<td>LLH</td>
<td>Larry Howell (Professor)</td>
</tr>
<tr>
<td>BDJ</td>
<td>Brian Jensen (Professor)</td>
</tr>
<tr>
<td>GHT</td>
<td>Greg Teichert</td>
</tr>
</tbody>
</table>
Figure A.1: Concept C1 drawing.

Figure A.2: Concept C2 drawing.
Figure A.3: Concept C3 drawing.

Figure A.4: Concept C4 drawing.
Figure A.5: Concept C5 drawing.

Figure A.6: Concept C6 drawing.
Figure A.7: Concept C7 drawing.

Figure A.8: Concept C8 drawing.
Figure A.9: Concept C9 drawing.

Figure A.10: Concept C10 drawing.
Figure A.11: Concept C11 drawing.

Figure A.12: Concept C12 drawing.
Figure A.13: Concept C13 drawing.

Figure A.14: Concept C14 drawing.
Figure A.15: Concept C15 drawing.

Figure A.16: Concept C16 drawing.
Figure A.17: Concept C17 drawing.

Figure A.18: Concept C18 drawing.
Figure A.19: Concept C19 drawing.

Figure A.20: Concept C20 drawing.
Figure A.21: Concept C21 drawing.

Figure A.22: Concept C22 drawing.
Figure A.23: Concept C23 drawing.

Figure A.24: Concept C24 drawing.
Figure A.25: Concept C25 drawing.

Figure A.26: Concept H1 drawing.
Figure A.27: Concept H2 drawing.

Figure A.28: Concept H3 drawing.
Figure A.29: Concept H4 drawing.

Figure A.30: Concept H5 drawing.
Figure A.31: Concept H6 drawing.

Figure A.32: Concept H7 drawing.
Figure A.33: Concept H8 drawing.

Figure A.34: Concept H9 drawing.
Figure A.35: Concept H10 drawing.

Figure A.36: Concept H11 drawing.
Figure A.37: Concept H12 drawing.

Figure A.38: Concept H13 drawing.
Figure A.39: Concepts list generated by Holly Greenberg.

- Can opener
- Hand
- "Hand" gripping arcade game to grab toys aka "the claw"
- Claws—birds
- Pincher—scorpion, cat mandibles
- Pin holder with edges
- Tongs
- Forceps
- Hemostat
- Clamps
- Vice
- Tweezers
- Eyelash curler
- Vice-grips
- Alligator clips
- Jaws
- Suction
- Vacuum
- Cable tie
- Catching a ball
- Unit "gripping" to clothes
- "Gripping" through capillary action
- Adhesion vs. gripping?
- Key grip to a lock
- Magnetic gripping
- Snaps on an clothing
- "Simple"
  - paperclip
  - binder clip
  - paper crimper
APPENDIX B. ANSYS CODE FOR CANTILEVER BEAM ANALYSIS

An earlier version of the ANSYS code below was written by Jason Lund to model and analyze the cantilever beams in Chapter 2 in order to obtain accurate elastic modulus and maximum strain data using the testing results. The code included below is an updated version which increases automation of the process. The beam dimensions given here are not the ones used for the analysis, but they are easily adjusted for those or any other size beams.

!ANSYS model for MEMS cantilever tests
!Used to account for geometry that beam theory ignores
!! Description:
!! The following code accepts a deflection and measured force for a given geometry
!! and iterates the non-linear solution until the corresponding modulus is found

!! Code accepts orthotropic material properties but currently uses a transversely
!! orthotropic material

!! What to change for use:
!! one should input the deflection and reaction force and change the geometry
!! as required.
!! Changing the filename would be advised

!Author: Jason M Lund
!Date: 04/23/2012

finish
/clear

!/CWD,'C:\Jason FEA\Bending Tests'
/Filename,CFRPBEAM,0 !Changes the filename from the default

finish
/begin

!!!!!! INPUT PARAMETERS HERE !!!!!!
!Parameters
!/INPUT,CFRPbeam1_1.txt !FIND string variable commands
! The script can be
!further automated by reading in the important
!variables from an external file
!Adding an additional loop could make the program
!capable of calculating the modulus for multiple
!tests with minimal supervision

deflection = -788.44
testforce = 6.9165e5 !microNewtons

filletRadius = 200
length = 1800
width = 250
tipLength = 150
tipRadius = 100
holeOffset = 50
elthruthick = 4
thick = 130

!!Parameters
!!Define Material Properties for the orthotropic carbon fiber
E1 = 48.0535e3 !MPa -Young’s Modulus in x-direction
E2 = 10e3 !MPa -Young’s Modulus in y-direction
E3 = E2 !MPa -Young’s Modulus in z-direction
nu12 = 0.3 !Poisson’s Ratio
nu23 = 0.3 !Poisson’s Ratio
nu31 = (E3/E1)*nu12 !Poisson’s Ratio
G12 = (E1)/(2*(1+nu12))
G23 = (E2)/(2*(1+nu23))
G31 = (E3)/(2*(1+nu31))

!set material properties
!material 1 = carbon fiber
mp,ex,1,E3
mp,ey,1,E1
mp,ez,1,E2
mp,nuxy,1,nu31
mp,nuyz,1,nu12
mp,nuxz,1,nu23
mp,gxy,1,G31
mp,gxz,1,G12
mp,gxz,1,G23

k,,(width/2)+filletRadius,0,0
k,,(width/2)+filletRadius,filletRadius,0
k,,(width/2),filletRadius+length+tipLength,0
k,,0,filletRadius+length+tipLength,0
k,,0,0,0
k,,0,-2*filletRadius,0
k,,(width/2)+2*filletRadius,-2*filletRadius,0
k,,(width/2)+2*filletRadius,0,0

larc,1,3,2,filletRadius
l,3,4
l,4,5
l,5,6
l,6,7
l,7,8
l,8,9
l,9,1
al,all
cyl4,(width/2)+holeOffset,filletRadius+length,tipRadius

asba,1,2,,delete,delete
firstoffset = -40
wpro,,-90
wpro,,firstoffset
asbw,all
numcmp,all
steps = 15
amount = (((filletRadius+80)/steps)-1

wpro,,filletRadius+80
asbw,all
numcmp,all
WPCSYS,-1,0
wpro,,-90
wpro,,filletRadius+length
asbw,all

allsel,all
numcmp,all

!*get,lasta,area,,num,max
!*get,firsta,area,,num,min

et,1,shell281
sectype,1,shell,,dummy
secdata,thick,1,,5,carbfib
et,2,solid185

lrgelements = width/4
smallelements = steps*.75
secnum,1
mshape,1,2d
mshkey,0
esize,smallelements
!asel,s,area,,all
!asel,u,area,,firsta
!asel,u,area,,lasta
amesh,2
esize,lrgelements
allsel,all
asel,u,area,,2
amesh,all
allsel,all
mshape,1,3d

! extrude the volume
TYPE, 2 ! selects the element type #2 (the solid elements)
! as the ones to be extruded
EXTOPT,ESIZE,elthruthick,0, ! sets the number of element
! divisions in the extruded direction
EXTOPT,ACLEAR,1 ! clears the 2d elements once the solids are made
allsel,all
vext,all,,,0,0,-thick

allsel,all

nsel,s,loc,x,0
d,all,ux,0
d,all,roty,0
allsel,all
nsel,s,loc,z,-thick
nsel,r,loc,y,-2*filletRadius,0,1
d,all,ux,0
d,all,uy,0
d,all,uz,0

allsel,all
nsel,s,loc,y,filletRadius+length
nsel,r,loc,z,0
d,all,uz,deflection

allsel,all
finish
/solu

ANTYPE,0 ! sets the analysis type as a static analysis
NLGEOM,1 ! sets the analysis mode to non-linear analysis
NSUBST,20,30,10 ! sets the target,maximum,minium number
    ! of substeps (15,30,5) (5,20,3)
OUTRES,ALL,1 ! output results ever substep
TIME,1 ! time at the end of the load step
solve
FINISH
/POST1
numloop = numloop+1
*ENDDO

nsel,s,loc,y,filletRadius+length
nsel,r,loc,z,0
FSUM,,
*GET,ANSYSforce,FSUM,0,ITEM,FZ
check = testforce-ANSYSforce
Eratio = testforce/ANSYSforce
E1 = E1*Eratio
!E2 = E2*Eratio
*IF,Eratio,eq,1,EXIT
FINISH
/PREP7
numloop = numloop+1
*ENDDO
APPENDIX C. MATLAB CODE FOR TORSION TEST DEVICE ANALYSIS

The MATLAB code below was used to process the test data from the torsion test devices discussed in Chapter 4 and calculate shear modulus values for each device.

```matlab
% Torsion testing data analysis script
% Masters thesis research
% Walter C. Fazio
% faziow@et.byu.edu
% Measurements conducted 23 Feb 2012
% Data chopping code based on a similar MATLAB script by Jason Lund from 09 Aug 2011
clc
clear all

%% USER-DEFINED VALUES
% Design details
% Torsion bar width: variable
% Torsion bar height: variable
beamDimensions = [146 104; 136 100; 233 51; 179 104; 185 105]; % height width

% Torsion bar length:
barLength = 1000;

% Cantilever distance from central axis of torsion beam:
cantileverLength = 1100;

% Fillet radius
% N/A

% number of data points to use when finding zero-val’s
MeanSampleSize = 25;
```
%% FILENAME READER
% Read in sample ID names from file

% Data files are named using their test ID
% File 'filenames.dat' contains a manually generated
% list of test IDs
fid = fopen('filenames.dat');
A = textscan(fid, '%s');
fclose(fid);
filenames = A{1};
extension = '.csv';

%% SAMPLE WIDTH AND HEIGHT READER
load sampleheightwidth.dat;
sampleHeights = sampleheightwidth(:,1);
sampleWidths = sampleheightwidth(:,2);

%% DATA-FROM-FILES READER
% Reads in data from data files and stores them in a cell array

% Create storage variables
datafull = cell(0,0); % Variable for initial data storage
data1 = cell(0,0); % Temporary storage of test ID strings
data2 = cell(0,0); % Temporary storage of test data
data = cell(0,0); % Variable for final data storage

% Pull data from data files
for i=1:length(filenames)
    file = filenames{i};
    fullname = strcat(file,extension);
datatemp = csvread(fullname);
data1{i} = file;
data2{i} = datatemp;
%readout = i;
end

% Store test data in cell array 'data'
for i=1:length(filenames)
    datafull{i,1}=data1{i}; % Test ID strings in column 1
    datafull{i,2}=data2{i}; % Test data matrix in column 2
end

%plot((-datafull{5,2}(:,2)),1*(datafull{5,2}(:,3)))

%% DATA CHOPPER

130
% Removes irrelevant data points from before loading and 
% after breakage

for i = 1:length(filenames)

    SampleToChop = datafull{i,2}(:,2:3);

    [value,minCell] = min(SampleToChop(:,2));
    SampleToChop = SampleToChop(1:minCell,:);

    % Average (mean) of force values of first 25 data points
    SampleSubset = SampleToChop(1:MeanSampleSize,2);
    zeroForce = mean(SampleSubset);
    i
    test = 1;
    for h = (1:length(SampleToChop))
        if (SampleToChop(h,2) < zeroForce)
            if (SampleToChop(h+4,2) < SampleToChop(h,2))
                if (SampleToChop(h+8,2) < SampleToChop(h+4,2))
                    if (SampleToChop(h+12,2) < SampleToChop(h+8,2))
                        if (SampleToChop(h+16,2) < SampleToChop(h+12,2))
                            if (SampleToChop(h+20,2) < SampleToChop(h+16,2))
                                test2 = h;
                                break
                            end
                        end
                    end
                end
            end
        end
    end
    test = test+1;
end

% counter verifiers
test
test2

% chop to remove leading zero-force data
SampleToChop = SampleToChop(test:length(SampleToChop),:);

% flip data values to positive values
SampleToChop = -1*SampleToChop;
zeroForce = -zeroForce;
% zero out deflection values with respect to initial position
SampleToChop(:,1) = SampleToChop(:,1) - SampleToChop(1,1);

% zero out force values with respect to mean zero-load force calculated
% previously
SampleToChop(:,2) = SampleToChop(:,2) - zeroForce;

% convert force and deflection values to MEMS units
SampleToChop(:,1) = SampleToChop(:,1)*1e3; % mm to microns (um)
SampleToChop(:,2) = SampleToChop(:,2)*1e6; % N to microN (uN)

% store cleaned-up data in new cell array 'data'
data{i,1} = SampleToChop; % Test data matrix in column 2
data{i,2} = datafull{i,1};
data{i,3} = i; % Test number
end

%% INTERESTING DATA EXTRACTOR

for i = 1:length(filenames)

datasetlengths(i) = length(data{i,1});

% Determination of which two datapoints to use when calculating slope
% if length(data{i,1})>600 % used for data sets with >600 datapoints
%     datapoint1 = 50;
%     datapoint2 = datapoint1 + 150;
%     %outputword = ’>600’
%     %
% else
%     datapoint1 = 50; % used for data sets with <600 datapoints
%     datapoint2 = length(data{i,1})-100;
%     %outputword = ’<600’
%     %
%     if datapoint2 < datapoint1 % use last datapoint if dp2 is before dp1
%     datapoint2 = length(data{i,1});
%     %status = [i, length(data{i,1}), datapoint2,]

end
% Slope finder
% 

datapoint2 = round(length(data{i,1})/2);
datapoint1 = 20;
datapoints1(i) = datapoint1;
datapoints2(i) = datapoint2;
deltadatapoints(i) = datapoint2 - datapoint1;

deflpoint2 = data{i,1}(datapoint2,1);
deflpoint1 = data{i,1}(datapoint1,1);
forcepoint2 = data{i,1}(datapoint2,2);
forcepoint1 = data{i,1}(datapoint1,2);

polyslope = polyfit(data{i,1}(datapoint1:datapoint2,1),data{i,1} ...  
 (datapoint1:datapoint2,2),1);

slope = polyslope(1); %(forcepoint2-forcepoint1)/(deflpoint2-deflpoint1)
slopes(i) = slope;
%
% Actual max point finder
%
maxDeflection = data{i,1}(length(data{i,1}),1);
maxForce = data{i,1}(length(data{i,1}),2)

% Use intermediate points as "max points"
maxDeflection = data{i,1}(datapoint2,1);
maxForce = data{i,1}(datapoint2,2);
%FoverD = maxForce/maxDeflection;

% Store slope and max points in cell array 'data'
data{i,4} = slope;
data{i,5} = maxDeflection;
data{i,6} = maxForce;

% Store torsion bar dimensions in cell array 'data'
data{i,7} = sampleHeights(i);
data{i,8} = sampleWidths(i);

end

%% ATTACH HEADERS TO END OF DATA ARRAY

data{end+1,1} = 'datapoints';
data{end,2} = 'sample name';
data{end,3} = 'sample #';
data{end,4} = 'def-force slope';
data{end,5} = 'max def';
data{end,6} = 'max F';
data{end,7} = 'TBar height';
data{end,8} = 'TBar width';

numberOfColumns = 8;

%data

%% MANUAL SAMPLE FILTERING

% Wanted: only main data samples (those testing torsion)

wanted = [1,2,3,4,5,7,8,9,10,11,12,13,14,15,16,22,23,24,25,26,29,30, ... 
31,34,35,36,37,38,39,40, 45];
wantedStrict = [1,2,3,4,5,7,8,9,10,11,12,13,14,16,22,23,24,25,30,34, ... 
35,36,37,39, 45];

dataTorsion = cell(0,0);
dataTorsionStrict = cell(0,0);

% fill dataTorsion cell array with all datapoints that are torsion tests
for i = 1:length(wanted)
    for j = 1:numberOfColumns
        dataTorsion{i,j} = data{wanted(i),j};
    end
end

% fill dataTorsionStrict cell array with one data sample for each beam
for i = 1:length(wantedStrict)
    for j = 1:numberOfColumns
        dataTorsionStrict{i,j} = data{wantedStrict(i),j};
    end
end

% list trimmed-version datapoints whose values are used for shear calculation
for i = 1:length(wantedStrict)-1
    datapoints2Strict(i) = datapoints2(wantedStrict(i));
deltadatapointsStrict(i) = deltadatapoints(wantedStrict(i));
datasetlengthsStrict(i) = datasetlengths(wantedStrict(i));
end

%% SHEAR MODULUS CALCULATION
for i = 1:length(dataTorsionStrict)-1
    thickness = dataTorsionStrict{i,7};
    width = dataTorsionStrict{i,8};
    w = thickness/2;
    t = width/2;

    J = w*t^3*(16/3 - 3.36*(t/w)*(1 - (t^4/(12*w^4))));
    Jarray(i,1) = i*1e10;
    Jarray(i,2) = J;

    slope = dataTorsionStrict{i,4};
    D = dataTorsionStrict{i,5};
    F = dataTorsionStrict{i,6};
    l = barLength;
    L = cantileverLength;
    shearModulus = (F*L^2*l)/(2*J*D);
    shearModulusFromSlope = slope*(L^2*l)/(2*J);
    %deltashear(i) = shearModulus - shearModulusFromSlope;

    dataTorsionStrict{i,9} = shearModulusFromSlope;
end

dataTorsionStrict{end,9} = 'G';

% OUTPUT PROCESSED DATA
readout = cell(0,0);
for i=1:length(dataTorsionStrict)
    readout{i,1} = dataTorsionStrict{i,2};
    readout{i,2} = dataTorsionStrict{i,3};
    readout{i,3} = dataTorsionStrict{i,4};
    readout{i,4} = dataTorsionStrict{i,5};
    readout{i,5} = dataTorsionStrict{i,6};
    readout{i,6} = dataTorsionStrict{i,7};
    readout{i,7} = dataTorsionStrict{i,8};
    readout{i,8} = dataTorsionStrict{i,9};
end

[nrows,ncols]= size(readout);

outfilename = 'torsionresults.csv';
fid = fopen(outfilename, 'w');
for row=1:nrows-1
    fprintf(fid, '%s, %d, %d, %d, %d, %d, %d, %d\n', readout{row,:});
end

fprintf(fid, '%s, %s, %s, %s, %s, %s, %s, %s\n', readout{nrows,:});
fclose(fid);

%%%% DATA PLOTTING TOOLS

% Load up relevant sample labels
for i=1:length(wantedStrict)-1
    filenamevector(i) = filenames{wantedStrict(i)};
    filenamevectorarray(i) = filenames{wantedStrict(i)};
end

for i=1:length(dataTorsionStrict)-1
    xaxis(i) = dataTorsionStrict{i,3};
    xaxis2(i) = i;
    yaxis(i) = dataTorsionStrict{i,9};
end

figure(1)
bar(xaxis,yaxis)
figure(2)
bar(xaxis2,yaxis)
figure(3)
bar(yaxis)
set(gca, 'XTick', 1:1:24)
set(gca,'XTickLabel',filenamevectorarray)
xlabel('Torsion Sample')
ylabel('Shear Modulus (MPa)')
APPENDIX D.  NANOWOCKY

The poem that follows is a personal adaptation of Lewis Caroll’s brilliant and whimsical poem “Jabberwocky,” applied to my research and read aloud at the end of my graduate seminar.

_Nanowocky_

’Twas brillig, and the seminar
Did gyre and gimble in the Clyde;
All mimsy was the CMR
And the nanotubes inside.

“Beware the cell restraint, RA!
The jaws that clamp, the beams that bend!
Go test it! Find out if we may
On that design depend!”

He took his lab notebook in hand:
Long time the manxome proof he sought–
He worked with hope at the microscope,
Then stood awhile in thought.

And as, in uffish thought he stood,
The cell pipette, of recent fame,
Came whiffling through the field of view,
And burbled as it came!

One, two! One, two! And through and through
The cell restraint went snicker-snack!
He watched it so; with video
He went galumphing back.

“And, does it work, the cell restraint?
Go publish now, my beamish boy!
O frabjous day! Callooh! Callay!”
He chortled in his joy.

’Twas brillig, and the seminar
Did gyre and gimble in the Clyde;
All mimsy was the CMR
And the nanotubes inside.
APPENDIX E. GALLERY

E.1 SEM Gallery

When frequently examining MEMS under the microscope, one inevitably encounters some intriguing views. CNT-MEMS are no exception. Mix in a little creativity and some proper photographic principles, and the scenes in the viewfinder or on the screen become compelling explorations of the microscopic world. The final pages of this thesis contain a sampling of interesting scanning electron micrographs obtained in the course of this research. A brief description accompanies each.
Figure E.1: Detail scanning electron microscope view of the letter “Y” in an uninfiltrated test pattern grown on 2 nm of iron. (10 kV beam, spot size 3, 900x magnification. Field of view approximately 150 µm.)
Figure E.2: Slanted view of uninfilitrated test pattern beams grown on 3 nm of iron. (10 kV beam, spot size 3, 350x magnification. Field of view approximately 250 μm.)
Figure E.3: Uninfiltrated hub-and-spoke test pattern. Splits reveal a few of the countless CNTs that make up the overall forest. This micrograph won third place at the 2010 ASME DETC Micro- and Nanosystems Photo Contest. (2.5 kV beam, spot size 3, 2000x magnification. Field of view approximately 60 µm.)
Figure E.4: Collapsed hub-and-spoke test pattern infiltrated with silicon. (10 kV beam, spot size 3, 200x magnification. Field of view approximately 600 µm.)
Figure E.5: Top view of fingerlike features partially infiltrated with silicon. (10 kV beam, spot size 3, 3300x magnification. Field of view approximately 40 µm.)
Figure E.6: Rubble-like arrangement formed by jumbled test pattern beams partially infiltrated with silicon. (10 kV, spot size 3, 285x magnification. Field of view approximately 430 µm.)
Figure E.7: Silicon-coated CNT strands in a thinner patch of the forest. (2.5 kV beam, spot size 3, 1060x magnification. Field of view approximately 100 µm.)
Figure E.8: Detail of an uninfiltrated set of beams grown on 4 nm of iron. (10 kV beam, spot size 3, 1100x magnification. Field of view approximately 100 µm.)
Figure E.9: A very “tall” letter B, partially infiltrated with silicon. (10 kV beam, spot size 3, 200x magnification. Field of view approximately 600 µm.)
Figure E.10: Detail of the base of the “B” feature. This micrograph won first place at the 2010 ASME DETC Micro- and Nanosystems Photo Contest. (10 kV beam, spot size 3, 5000x magnification. Field of view approximately 25 μm.)
Figure E.11: A peculiar pattern of fissures and protrusions based around etch holes in an unfilled test structure. (20 kV beam, spot size 3, 2375x magnification. Field of view approximately 50 µm.)
Figure E.12: Detail view of an XBob in a carbon-infiltrated cell restraint device. Triangular growth support blocks are visible between the beams. (5 kV, spot size 4, 175x magnification. Field of view approximately 700 µm.)
Figure E.13: View of the C21 “pincer” cell restraint device (see Chapter 5). This micrograph won second place at the 2011 ASME DETC Micro- and Nanosystems Photo Contest. (5 kV beam, spot size 5, 70x magnification. Field of view approximately 1775 µm.)
Figure E.14: Angled view of the grasper arms of an H1 cell restraint device (see Chapter 5). (5 kV beam, spot size 5, 90x magnification. Field of view approximately 1375 µm, or 1.375 mm.)
Figure E.15: Closeup of a well-infiltrated CI-CNT structure, as viewed from above. (5 kV beam, spot size 3, 3200x magnification. Field of view approximately 40 µm.)
Figure E.16: Fully assembled triangular folding box (see Section 6.2.10). (5 kV beam, spot size 3, 33x magnification. Field of view approximately 3750 $\mu$m, or 3.75 mm.)
Figure E.17: A feature seen occasionally on the surfaces of CI-CNT samples, affectionately termed “snow worms” due to the bright appearance of the background CI-CNT surface. (10 kV beam, spot size 4, 676x magnification. Field of view approximately 180 μm.)
Figure E.18: A CI-CNT rendition of the Compliant Mechanisms Research (CMR) Lab logo. (5 kV beam, spot size 3, 125x magnification. Field of view approximately 1000 µm, or 1 mm.)
Figure E.19: Brigham Young University’s classic “Y” logo, in all its microscopic glory. It was included in some of the preliminary design test patterns and thus fabricated alongside them. (5 kV beam, spot size 5, 62x magnification. Field of view approximately 2000 µm, or 2 mm.)