Modeling and Effects of Non-Homogeneous Infiltration on Material Properties of Carbon-Infiltrated Carbon Nanotube Forests

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Modeling and Effects of Non-Homogeneous Infiltration on Material Properties of Carbon

Infiltrated Carbon Nanotube Forests

Daniel O. Snow

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

Modeling and Effects of Non-Homogeneous Infiltration on Material Properties of Carbon Infiltrated Carbon Nanotube Forests

Daniel O. Snow
Department of Mechanical Engineering, BYU
Master of Science

This work investigates the material properties and production parameters of carbon infiltrated carbon nanotube structures (CI-CNT’s). The impact of non homogeneous infiltration and the porosity of cross section regions, coupled with changes in designed geometry, in this case beam width, on the density and modulus of elasticity are compared. Three potential geometric models of beam cross section are proposed and evaluated. 3-point bending, SEM images, and numerical optimization are used to assess the validity of each model and the implications they have for future CI-CNT material applications. Carbon capping near exterior beam surfaces is observed and determined to be a contributing factor to variations in material properties correlated with changes in designed geometry and infiltration parameters (temperature, time, and hydrogen flow rate). Unexpected relationships between beam width and elastic modulus are partially explained by modeling the carbon-capped beams as C-shaped structural members consisting of a graphitic carbon shell of varying porosity and thickness and uninfiltrated carbon nanotube internal regions with a near negligible stiffness. Findings of previous works on the effects of infiltration parameters and carbon capping on materials properties are confirmed and expanded. Flange and web thickness and porosity of the graphitic carbon shell are identified as potential design parameters for pursuing tunable material properties in high precision geometry MEMS and compliant mechanism applications.

Keywords: CI-CNT, Carbon Nanotube, material properties, 3-Point Bending
ACKNOWLEDGMENTS

I have been working on CI-CNT research on and off for almost 8 years. During that time I have had the privilege to work with amazing dedicated and talented individuals who have enriched the learning process and enhanced the reach of my own talents and efforts.

Foremost among those people is Dr. Brian Jensen. I would like to thank him for giving me the opportunity to get involved in research early in my undergraduate years, and also for his patience and optimism as my advisor for this thesis. It is a pleasure to work with and learn from him.

I would also like to thank Dr. David Fullwood and Dr. Eric Homer, whose classes ignited my passion and curiosity for materials science and materials selection. I took more credits from Dr. Fullwood in my undergrad and graduate degrees than any other professor, most of which were elective courses. Not coincidentally, they are the other members of my thesis committee, as their expertise and passion for materials has and will continue to be, invaluable.

I would like to thank Shane Sypherd for joining me on this investigation and for his countless hours of work in sample preparation and data gathering. This thesis would not be possible without his efforts. Fred Fagergren and Andrew Cunningham also made valuable contributions to the data gathering and processing efforts.

Big thanks goes out to the Mechanical Engineering, Electrical engineering, Physics, and Chemistry departments of Brigham Young University for the use of all the amazing equipment and for facilitating the necessary training to use it all. Brigham Young University and Tag-Heuer also deserve special thanks for their funding of the research.

I would like to thank my amazing wife, Jazmin, for her patience, encouragement, and faith throughout this entire project and my entire education.
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CHAPTER 1. INTRODUCTION

1.1 Background

In the fields of microelectromechanical systems (MEMS) and compliant mechanisms, carbon infiltrated-carbon nanotube (CI-CNT) structures have been shown to have many advantages as a material [1] [2]. Principal among these are the ability to produce these structures with precise geometry on the micro scale, reasonable strength, and significant control over material properties by varying parameters during the production/manufacturing process. CI-CNT structures are created by patterning 2 dimensional geometry onto a prepared substrate, then growing a vertically aligned CNT forest on that patterned substrate, and finally infiltrating the CNT forest with amorphous carbon before releasing the structure from the substrate. This research seeks to further determine the effects of process variations during the infiltration stage and of designed geometric parameters of the structures on effective mechanical material properties. This will allow for improved utility of the CI-CNT material in current applications, and an expansion of its use to additional fields and applications.

1.2 Previous Works

Previous work with carbon nanotubes and carbon infiltrated carbon nanotubes as a nanomaterial covers a broad range of intended applications and includes various findings based on highly constrained testing of the material. Hutchison et al. [3] demonstrated the ability to produce CI-CNT structures with precise dimensional tolerances and accuracy. Fazio et al. explored modulus and strength properties of CI-CNT beams in large deflection scenarios and out of plane bending [4]. Hanna et al. developed best practices and methodology for measuring mechanical properties of CI-CNT structures intended for use in compliant micromechanism applications [5]. Robison and Jones et al. explored applications in compliant mechanisms and medical devices such as coronary
stents [2] [6]. Wang et al. explored the addition of CNTs to carbon fiber composites and the resulting influences on microstructure and properties [7]. While developing CI-CNT structures for the restraint of individual cells, Toone et al. [8] showed several material property advantages of CI-CNTs including bio-compatibility, good thermal properties, and sufficient strength for use in compliant mechanism applications.

Mechanical properties have been explored using forces applied in different scenarios, directions, and structure orientations. Kwon et al. studied torsional behavior in polymer-CNT structures using atomic force microscopy [9].

Several studies have explored infiltration materials aside from amorphous carbon. Chen et al. created a wax infiltrated CNT sponge composite to explore electro- and photodriven phase changes in a material [10]. Zhou et al. used aluminum alloy CNT composites to explore tribological properties and fabrication techniques [11]. Polymer infiltration of CNT forests or CNT carpets to improve mechanical properties of micro-structures was explored by Jung et al. and Dassios [12] [13]. Gu et al. infiltrated CNT forests with silicon carbide [14].

Infiltration behavior, parameters and constraints have been evaluated to understand how CNT forests can be infiltrated more predictably and completely with a variety of materials. Boncel et al. studied capillary infiltration of aqueous solutions into CNT films [15]. Liu et al. explored using pressure to and applied charges to infiltrate CNTs with water [16].

Other research focused on evaluating material properties that were not directly mechanical in nature. Gong et al. explored thermal properties in CI-CNT nanocomposites, and Borca et al. characterized anisotropic thermal diffusivity in CNT-polymer composites [17] [18].

1.3 Motivation

This work will utilize and build on prior work by expanding the exploration of material properties by exploring in plane bending with the nanotubes aligned horizontally, and in a 3-point bending environment which prevents the need of large deflection analysis. Prior work also commonly makes the assumption that CI-CNT structures behave as a homogeneous material where the effective modulus of elasticity is essentially equivalent to a Young’s modulus for pyrolytic deposited carbon. Though several prior investigations have noted incomplete infiltration and carbon capping as potential sources of variation or error, none have explored the possibility of quantifying
the effects of the partial infiltration. This work will focus on the assumption that CI-CNT structures have non-homogeneous cross sections, as well as varied porosity in infiltrated areas, due to incomplete infiltration and carbon capping, and the effects or process parameter variation. Explanations of differences between the effective material properties of the beams will be posited. 3 cross section models will also be developed to quantify and understand the effects of porosity and capping and its geometric design implications.

1.4 Thesis Objective

For this thesis, the objective was to determine to what extent the effective material properties and stiffness of CI-CNT beams are affected by non-homogeneous infiltration, infiltration parameters (temperature, time, and hydrogen flow rate) and designed variations in beam width, and to further facilitate design and modeling decisions as well as manufacturing process/parameter development.

To explore these questions, a full factorial experiment was developed to test the effects of various manipulable parameters on the effective mechanical material properties of CI-CNT beams. In conjunction with a parallel thesis effort, CI-CNT beams were produced with 4 infiltration duration times (10 min, 25 min, 45 min, 70 min), 4 infiltration temperatures (800 C, 850 C, 900 C, 950 C), 3 hydrogen gas flow rates (311 sccm, 396.5 sccm, 492.4 sccm), and 3 patterned beam widths (200 microns, 250 microns, 300 microns). Each combination of growth parameters was replicated 3 times with a sample containing all 3 beam widths, resulting in 144 sample sets. Each sample set consisted of 25 beams for each width or 10,800 total beams. 10 beams per width and growth parameter combination were desired, but a significant number of beams were expected to be damaged during the growth, infiltration, removal, and testing sequence, so 25 beams were produced to ensure sufficient sample quantity. Beams of all widths were patterned with 6mm length. The desired growth height was between 300 and 425 microns. Growth height is controlled by the same parameters as infiltration (time, temp, hydrogen flow rate), but is very difficult to control, which is why the experiment allowed for a large range of acceptable heights. Testing of the beams in the sample sets included:

- Removing 12 beams from growth and infiltration substrate using a razor blade.
• Weighing 12 beams together as a batch using a high precision scale, in order to determine average density of the batch.

• Optical measurement of height and width of each beam individually at 300x magnification. These measurements were then used to calculate density and several other values during analysis.

• 3-point bending to failure of each beam using a custom fixture with a 3.2mm gap. This was done with a razor blade attached to a 1 pound load cell on a mini Instron force deflection measurement instrument.

Data output from the load cell was collected and processed with a Python code script and then analyzed graphically and statistically to further understand the effects of infiltration parameters and beam geometry on CI-CNT structure mechanical properties. The beams infiltrated at 800 C lacked sufficient stiffness and strength to yield useful results during 3-point bending. In many cases, the beams never broke, and simply deflected more and more until the end slipped into the gap which the beam was placed across for the test. In other cases, the beams simply sheared in half without deflecting at all and registered no readable force on the load cell.
CHAPTER 2. EXPERIMENT

2.1 Sample Creation

The sample CI-CNT beams were fabricated using a process similar to that outlined by Fazio [4] with modifications to the growth and infiltration parameters intended to serve as the basis of the experiment. The process is described in this section and is shown in Figure 2.1 [19].

2.1.1 Substrate Preparation

The substrates used for the beams were 4 inch (100) silicon wafers. A chromium plated mask was created specifically for this project and had sets of 25 beams all 6 mm in length, with 3 groupings in close proximity, one each of 200, 250, and 300 micron width beams. This was repeated in rows and columns to fill the entire usable wafer surface. AZ-3312 positive photoresist was used with the mask in a photolithography aligner to create the 2-dimensional footprint of the beam sets (step (a) of Figure 2.1). The photoresist coated wafer was then developed using MIF300, and a 40 nm layer of alumina (AL2O3) was deposited using an electron beam evaporator (step (b) of Figure 2.1). The following layer was 4nm of iron deposited in a thermal evaporator (step (c) of Figure 2.1). The iron was the most critical layer as it serves as a catalyst to induce the growth of vertically aligned CNT’s. Alumina acts as a buffer or seal between the iron and silicon and prevents the iron layer from diffusing into the wafer during periods of elevated temperature that occur later in the fabrication process. The patterned wafer was placed in a sonicated bath of NMP (N-Methyl-2-pyrrolidone) to remove the photoresist that was undeveloped in the aligner (step (d) of Figure 2.1). A new layer of photoresist was then applied to the entire wafer to protect the patterned surface while the wafer was cut into individual sample sets using a diamond powder blade in a wafer dicing saw. The wafer was then cleaned again to remove the protective photoresist, and the samples were ready for the growth and infiltration phases.
2.1.2 Growth

The process by which the nanotubes form is known as chemical vapor deposition (CVD). CNT forests were grown in an induction furnace that uses a 24mm inner diameter quartz tube as the heating chamber. The tube was heated to 750 C with argon flowing through it. Then hydrogen gas began flowing (311 sccm or 0.0115 m/s) and the argon was shut off. Ethylene (C2H4) then began to flow (338 sccm or 0.0125 m/s). This state was held for 5 minutes and 30 seconds for all samples. This period allowed CNT’s to form and the time control along with the specific temperature and gas flow rates were chosen to constrain the growth height of the CNT’s within the desired range. After the growth time, ethylene flow was shut off, and argon flow was restored, then hydrogen flow was shut off and the temperature was ramped up to the infiltration temperature, which was varied as part of the experiment. The sample at this stage consisted of the original patterned wafer chip with a vertically aligned CNT forest bonded to the surface wherever exposed iron was present (step (e) of Figure 2.1).

2.1.3 Infiltration

This stage was where the sample preparation parameters began to vary according to the experiment outlay. Table 2.1 shows the entire range of parameters for temperature, infiltration time, hydrogen flow rate and beam width. The order of growth and infiltration of the 144 sample sets was randomized to minimize any potential bias. All 3 beam widths for a given sample were
grown and infiltrated on a single wafer chip at the same time to ensure identical conditions for all beams in given set.

Once the furnace temperature finished ramping to the appropriate infiltration temperature, hydrogen at the corresponding flow rate was switched on. Argon was switched off and ethylene flow was restored at the same flow rate as the growth phase (338 sccm). This state was maintained for the duration determined by the experiment parameters. Ethylene was then shut off, argon flow was restored, and then hydrogen was shut off. The furnace was then opened and shut off to allow the quartz tube, which remained sealed with argon flowing, to cool to a temperature low enough to safely remove the sample.

### Table 2.1: Complete Experiment Design Parameters

<table>
<thead>
<tr>
<th>Infiltration Temp (C)</th>
<th>Infiltration Time (min)</th>
<th>H2 Flow (sccm)</th>
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<tbody>
<tr>
<td></td>
<td>10</td>
<td>25</td>
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<tr>
<td>850</td>
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<tr>
<td>950</td>
<td>200 μ 250 μ 300 μ</td>
<td></td>
</tr>
</tbody>
</table>

μ indicates microns of patterned beam width

### 2.1.4 Beam Release

Removal of the samples from the wafer substrate is a delicate process. The method used for this research consisted of using a razor blade to separate the base of the beams from the wafer gently by pressing the blade edge against the entire base along the beam’s length. 12 beams of each width were gathered, to ensure at least 10 beams survived the rest of the measurement process. Figure 2.2 shows a sample with several beams already removed.
2.2 Measurements

2.2.1 Weight, Dimension, Density

Beams were weighed using a high precision enclosed scale in batches of 12 where all beams had the same infiltration parameters and width. The scale used was accurate to 0.0001 grams.

Each beam was then measured for height and width individually using a Keyence digital microscope at 300x magnification.

With measurements for grown height, patterned length and width, the average density of each batch was calculated.

2.2.2 3-Point Bending

A 3-point bending test was performed for every beam on a Mini Instron. A 1 pound load cell and a razor blade were used to apply a point load in the middle of the beam, which was placed across a 3.2mm gap (Figure 2.3).
Figure 2.3: Mini Instron and stage used for 3-point bending tests and data acquisition.

The beams were simply supported and free to move during the test. The program to run the test was written such that the razor blade would continue to descend until the beam shattered. Force and deflection data were gathered continuously. As the primary field of applications for this project was MEMS, the beams were placed such that the CNTs were aligned parallel to the length of the razor blade (Figure 2.4), as this is the orientation that would most likely be available to MEMS devices, and is the orientation with least prior investigation.
2.2.3 Data Extraction and Cleanup

Collected data was reduced to only the data gathered from the moment the blade made contact with the beam until the beam broke. This was done with a Python program which then calculated the slope of the force deflection curve which is the beam’s stiffness, and is a critical value for later analysis. This data set was utilized partially or entirely for this thesis as well as thesis research by Shane Sypherd and Fred Fagergren [19], [20]. Figure 2.5 is an example of the force-deflection curves produced by the data gathering program on the Mini-Instron.

2.3 Analysis

In order to determine what effect, if any, incomplete infiltration due to the beam’s patterned width and porosity had on the material properties of the CI-CNT material, 3 different models were used to calculate values for 2nd moment of area, effective elastic modulus, and theoretical stiffness given the measured density. An important note is that for all models, the beam’s “width” as patterned on the substrate is actually considered the “height” for the 3-point bending test results, since it was the width that was aligned vertically during the test. From this point forward, patterned
Figure 2.5: Example of force-deflection curves produced by 3-point bending test.

width will be denoted by \(w\), and height grown away from the substrate will be denoted by \(h\). For the equations and relationships of these models to be valid, the following assumptions were made:

- Deposited pyrolytic carbon has a Young’s Modulus of 15 GPa and density of 1800 kg per cubic meter. These values are the midpoints of the normal range for graphite [21]. Different values for either property can be used without damaging validity of our models, but this was our starting point since we do not have exact values for either.

- Pure bending - this assumes a shear force of zero, and no torsional or axial loads.

- Hooke’s Law applies for this material.

- The material is isotropic.

- The beam has an axis of symmetry in the bending plane.

- Failure mode is bending, not crushing, wrinkling, or sideways buckling.

- Plane cross sections of the beam remain in plane during bending.

The effective Young’s modulus for the deposited carbon can be calculated using Equation (2.1) where \(E_g\) is the Young’s modulus of graphite and \(\phi\) is the porosity of the graphite [22] [23].
\[ E_{\text{eff}} = E_0 (1 - \phi)^2, \]  
\[ (2.1) \]

\[ k_t = \frac{48EI}{L^3} \]  
\[ (2.2) \]

### 2.3.1 Porous Rectangular Cross Section

The first model assumes that the beams have homogeneous infiltration and a simple rectangular cross section, and that the density of the beam is primarily driven by porosity. The effective modulus was determined by first calculating a value for the porosity of the beams using Equation (2.3) and then using Equation (2.1). 2nd moment of area for a rectangular cross section or \( I_{\text{Rect}} \) is calculated using Equation (2.4). Finally the theoretical modeled stiffness \( k_t \) is calculated using Equation (2.2) where \( L \) is the length of the gap spanned by the beam (3.2 mm).

\[ \phi_{\text{Rect}} = 1 - \left( \frac{d_{\text{avg}}}{d_g} \right), \]  
\[ (2.3) \]

\[ I_{\text{Rect}} = \frac{hw^3}{12}, \]  
\[ (2.4) \]

This is similar to the model used by Sypherd in his investigation of the 200 micron beams from this same data set [19], with the principal difference being the use of porosity to modify the effective Young’s modulus. This research expanded Sypherd’s investigation to include the 250 and 300 micron beam widths to determine if incomplete or non-homogeneous infiltration and/or patterned geometry had any influence on effective modulus.

### 2.3.2 Solid C-Shaped Cross Section

In previous research efforts, it was hypothesized that during infiltration, amorphous carbon forms more quickly near the exterior surfaces of the CI-CNT structure, and under certain conditions, that carbon can effectively seal off the beam and prevent carbon infiltration from creating a homogeneous infiltration. This “capping” could change the mechanical nature of the beam if most of the load bearing material is found at the edges. The hypothesis developed during the planning
of this thesis research was that the changes in effective modulus observed in beams of differing geometries could be due to carbon capping, and that the beams might be more accurately modeled using a C-shaped structural beam model (Figure 2.6) as opposed to a simple rectangular model.

![C-shaped cross section diagram oriented as beams were during 3-point bending.](image)

Figure 2.6: C-shaped cross section diagram oriented as beams were during 3-point bending.

2nd Moment of Area

If capping is indeed causing the beam to behave similar to a C-shaped beam, one source of the difference from the simple rectangular model is the 2nd moment of area. Using the parallel axis theorem, we can derive an equation for the 2nd moment of area of a C-shaped beam or $I_C$ (Equation 2.5), where $T1$ and $T2$ are the thicknesses of the flanges and web as shown in Figure 2.6, and $h$ and $w$ represent the height and width as they did in the simple rectangular beam model [24].

$$I_C = \frac{hw^3 - (h - T_2)(w - 2T_1)^3}{12}, \quad (2.5)$$

Model Optimization

This model attributes a portion of variation in flexibility to the differences in overall geometry, $h$ and $w$, and internal geometry formed during infiltration and capping, $T1$, $T2$, and $g$, where $g$ is the internal distance between the two flanges. There are several important assumptions in addition to those made for the simple rectangular cross section to make this model valid:
• The amorphous carbon infiltration has Young’s modulus \( Y \) and density properties similar to graphite (15GPa, 1800 kg/m\(^3\)).

• All beams in a single production batch have similar values for \( T1, T2, \) and \( Y \), and the cross section has a plane of symmetry parallel to the bending plane.

• CNT forests aligned parallel to the bending axis have a negligible density and Young’s modulus compared to amorphous carbon.

To determine the values for the geometric variables in Figure 2.6, this model uses the measured beam stiffness and density and known values for Young’s modulus and density of graphite \( d_g \). First, the cross sectional area \( A_{cs} \) is calculated using Equation (2.6), where \( d_{avg} \) is the average density of the beams in the given batch.

\[
A_{cs} = \left( \frac{d_{avg}}{d_g} \right)wh,
\]  
(2.6)

SEM images [19] and measurements indicate that \( T2 \) is thicker than \( T1 \) in beams where infiltration capping occurs [25]. In this analysis a fixed ratio of \( T2 \) being twice as thick as \( T1 \) is used (Equation 2.7). Without a fixed ratio, there are nearly infinite values of web and flange thickness that result in the calculated cross sectional area for the measured height and width of the beams. Additionally, change to this ratio produces little difference in the model outcome, since \( T1 \) affects the value of 2nd moment of area much more than \( T2 \), given Equation (2.5). With limited information about infiltration across the CNTs vs. along them, the approximation is good enough for our current objective. With that ratio, \( T1 \) is then derived as a function of \( A_{cs} \), height and width (Equation 2.8).

\[
T_2 = 2T_1, 
\]  
(2.7)

\[
T_1 = -\frac{\sqrt{(-4(A_{cs})) + h^2 + 2wh + w^2 - w - h}}{4},
\]  
(2.8)

With the geometries of each C-shaped cross section approximated, \( I_C \) can then be calculated for each beam using Equation (2.5). Theoretical modeled stiffness can then be calculated for the solid C-shaped cross section using Equation (2.2).
2.3.3 Porous C-Shaped Cross Section

The third model we used is essentially a hybrid approach which incorporates the calculations for porosity from the first model and the C-shaped geometry of the second model. Using Equations (2.3) and (2.6), we can derive a new equation for the area of the C-shaped cross section, Equation (2.9). We then calculate $I_C$, flange and web thickness, and finally theoretical modeled stiffness, just as in the solid C-shaped cross section model.

$$A_{cs} = \left( \frac{d_{avg}}{d_g(1 - \phi)} \right) wh,$$

(2.9)

Model Optimization

By allowing porosity to vary, we allow the web and flange thickness and effective Young’s modulus to vary relative to each other. Using the sum of squared percent error and a VBA script with Excel Solver, we selected the value of $\phi$ for each batch that minimized the difference between the measured stiffness of the beams $k_m$ and the theoretical modeled stiffness $k_t$. 


CHAPTER 3. RESULTS AND DISCUSSION

Table 3.1: Summarized Experimental Results - 200 micron

<table>
<thead>
<tr>
<th>Temp (C)</th>
<th>Infil. Time (min)</th>
<th>H2 Flow (sccm)</th>
<th>Density (kg/m3)</th>
<th>Stiffness (N/m)</th>
<th>Porosity</th>
<th>Flange Width (T1, microns)</th>
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</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Measured Porous Solid C Porous C Porous Rect Porous C Solid C Porous C</td>
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<td>0.83 0.83</td>
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<td>91.22</td>
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<td>3.36 96.44</td>
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<td>2.95 103.75</td>
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<td>6.60 68.83</td>
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Results of the full experiment are summarized in Tables 3.1, 3.2, and 3.3, and explained in the following sections. As explained above, the beams infiltrated at 800 C were not sufficiently stiff or strong to yield useful results in the 3-point bending test, and are also omitted from the
experimental results. Of the original 10,800 beams, more than half of the beams created were intended only as assurance that enough beams would survive release from the substrate for testing. After removing all the 800 C beams from the sample, and all measurement and testing procedures, 2,640 or approximately 81%, of the tested beams yielded data that was usable.

### 3.1 Initial Findings

The expectation at the onset on this experiment was that stiffness would increase as density increases and as beam width increases. This is confirmed with the measured stiffness data as shown in Figure 3.2. The purpose of the comparison of the three geometric models is to identify...
Table 3.3: Summarized Experimental Results - 300 Micron

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a potential explanation as to why beams of differing thicknesses exhibit different effective material properties, and to arrive at a useful model to inform future design decisions and tradeoffs. Figure 3.1 shows the percent error of theoretical stiffness relative to measured stiffness vs. the patterned beams width, for the porous rectangular cross section model. This model, like the models used in past works, assumes that the entire cross section is infiltrated homogeneously, though porosity may vary. Like past models it becomes less accurate for wider beams unless a very low temperature is used. This is consistent with past findings that were the source of our motivation to explore the effects of beam geometry on material properties.
The data in Tables 3.1, 3.2, and 3.3 show that by using a C-shaped beam model and accounting for differences in the porosity of infiltrated pyrolytic carbon, inconsistency of material properties can be attributed to non-homogeneous infiltration, differences in beam width, and variation in the porosity of the infiltrated carbon caused by varying infiltration process parameters. Our belief is that the temperature of deposition causes differences in the microstructure of the infiltrated carbon and, coupled with infiltration time and hydrogen flow rate, the carbon density/porosity as well. Variation in pyrolytic carbon modulus and other material properties as a result of deposition temperature has been demonstrated in several prior studies [26], [27], [28].

3.2 Stiffness Model Comparison

Figure 3.2 also gives us a first look at the accuracy of each of the three models we use to approximate the theoretical stiffness based on density. We see that for beams of lower densities,
the rectangular cross section is a much better approximation than a non porous C-shaped cross section, but as density increases, the opposite becomes true. It is therefore not surprising to see that the porous C-shaped cross section model is a very good approximation of measured stiffness regardless of density, since it takes into account the physical phenomena from both of the other models.

Figure 3.3 shows us the resulting theoretical stiffness values of each model plotted against the measured stiffness for the corresponding beams. Again we see that the Porous C-shaped model provides values that are very close to reality.

### 3.3 Infiltration Parameters

Porosity is affected by all three of the parameters that we varied during the infiltration process. Figure 3.4 shows that as infiltration temperature increases, porosity generally decreases,
Figure 3.3: Modeled vs Measured Stiffness

Figure 3.4: Porosity vs. Infiltration Temperature
Figure 3.5: Porosity vs. Infiltration Time

Figure 3.6: Porosity vs. Infiltration Hydrogen flow rate
which tracks with our understanding that carbon is deposited more quickly as temperature increases. Similarly, as infiltration time increases, porosity generally decreases, as shown in Figure 3.5, since longer exposure of the beams to deposition increases the amount of carbon that finds its way into the beam. Increased Hydrogen flow rate increases porosity, see Figure 3.6. Individually each of these findings makes sense based on prior works.

![Figure 3.7: % Error in Stiffness vs. Infiltration Time, 850 C](image)

Figures 3.7, 3.8, and 3.9 show the % error between the theoretical stiffness values produced for each model and the measured values of stiffness from the actual beams. Each graph shows all the beams for a single infiltration temperature to allow for better comparison. For lower temperatures and infiltration times, not how poorly the solid C-shaped cross section model approximates stiffness relative to the other models that account for porosity. As temperature and infiltration time increase, this model becomes much more accurate, since decreasing porosity makes the assumption of webs and flanges that resemble solid graphite more and more plausible. For beams infiltrated at 950 C and more than 40 minutes, it becomes a better approximation than the rectangular porous model.
Figure 3.8: % Error in Stiffness vs. Infiltration Time, 900 C

Figure 3.9: % Error in Stiffness vs. Infiltration Time, 950 C
These results become more interesting when we look at porosity relative to T1. Beams infiltrated at a lower temperature tended to have thicker web and flanges when porosity is low, but as porosity increases, the higher temperature beams begin to have thicker webs and flanges. This supports our hypothesis of carbon capping at the surface, since we expect higher temperatures to produce thinner, more dense edges along the beams and lower temperatures to produce wider less dense regions, while the stiffness of the beams is similar. Capping had been theorized and then observed (Figure 3.11) [19], which is what pointed us towards a C-shaped model early in our research. The rectangular cross section with homogeneous infiltration seemed unlikely to capture the complexity of the structural and material properties involved in 3 point bending. With
the bottom edge of the beam completely sealed by the silicon substrate, the capping could only occur at the remaining 3 edges of the beam. The C-shaped model was intended to more closely resemble to actual structural cross section and therefore render results that made sense based on prior research and known phenomena in the field of mechanics of materials.

Figure 3.11: SEM image of capping in a CI-CNT structure; note the resemblance to a C-shaped structural member cross section.
CHAPTER 4. CONCLUSION

4.1 Implications

Comparing all 3 cross sectional geometry models has improved understanding of porosity, elastic modulus, stiffness, density, and manufacturing methods in CI-CNT structures. Sypherd commented on the tunability of material properties of CI-CNT structures made possible through varying the conditions of infiltration [19], and Stevens et al. similarly explored tunability of geometry [29].

Through this investigation of beams of several patterned widths, it is now more clear how these parameters can affect stiffness, density, and effective modulus. Our results definitively confirm that the patterned geometry affects the resulting material properties in CI-CNT material, which has been a source of confusion and doubt in several past research and application efforts. Porosity and carbon capping are the most likely driving factors in material and mechanical property variations across beams or members of varying geometries. Future research and design projects can take this into account to improve their processes and results.

Designers of CI-CNT structures can now understand the potential impacts of designed geometry in terms of flange and web thickness and porosity. Design and production decisions are not limited to simply minimizing carbon capping and porosity, though this is one valid approach. Using the porous C-shaped cross section model, and to some extent the other two models as well, it is entirely feasible to tune the stiffness and beam geometry by selecting appropriate values for temperature, time and hydrogen flow during infiltration. This will ultimately reduce the variability and uncertainty of resulting properties when utilizing this material.
4.2 Future Work

Further investigation into the actual cross sectional geometry of the beams would be valuable. It is unlikely that the beams consistently resemble a c-shaped beam or a homogeneous rectangular beam. Depending on infiltration parameters, beams likely fall on a spectrum between the two models. One potential tool to further understand the internal geometry would be extensive SEM imaging of broken beams with measurements of visible capped areas. A better understanding how the internal geometry develops over time would further improve the tunability of material properties that make CI-CNT structures so unique.

Another area for future work could involve producing much wider and much shorter beams in an attempt to get a homogeneous infiltration. 3 point bend tests of these beams could be compared to beams of the same dimensions made from graphite to further understand the composition of the infiltrated amorphous carbon and potential mechanical effects of the CNT forests contained in the carbon matrix.

Research into how the infiltration parameters of gas flow rates and temperatures affect the type of carbon that infiltrate the CNT forest would also be of great value [30]. The ability to infiltrate with carbon of a much higher Young’s modulus and strength could greatly expand the list of potential applications for the CI-CNT nano material. Confirming the density of the shell or cap material would also help to improve the accuracy of all three of the beam models used here.

Understanding how the microstructure and density of the infiltrated pyrolytic carbon is affected by infiltration parameters such as temperature and time could also further improve the validity of the C-shape model, or possibly point to a different geometric model that resembles the resulting geometry and effective material properties even better. This could be done through experiments similar to those perfomed by Zhang et al [28] using the same furnace and parameters for infiltration we used, but removing the CNT structures and simply creating a pyrolytic carbon film and testing it using nanoindentation or other common methods.
REFERENCES


