A Study of Piezoresistive Sensing Based on Carbon-Nanotube Forests

by

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Abstract

Carbon nanotubes have attracted considerable attention since their discovery due to their exceptional electrical, mechanical, and optical properties. Piezoresistance of carbon nanotubes is promising, and can be utilized to enable various types of devices. This work investigates devices functionalized with vertically aligned multi-walled carbon-nanotube forests, with a focus on pressure and strain sensors. A fabrication process based on Si-micromachining techniques that overcomes the challenges associated with using carbon-nanotube forests was developed for the devices construction.

A pressure sensor is fabricated to have a multi-walled carbon-nanotube forest supported by a deflectable 8-µm-thick Parylene-C membrane suspended by a silicon frame. The responses of the fabricated sensors are experimentally characterized. The sensitivities to positive and negative gauge pressures are found to be comparable in magnitude with the average values of -986 ppm/KPa and +816 ppm/KPa, respectively. The measurement also reveals that the temperature coefficient of the resistance for a forest suspended with a Parylene membrane is -515 ppm/°C, ~3× smaller than that for a forest fixed onto a silicon substrate.

A strain gauge is also fabricated to have a multi-walled carbon-nanotube forest supported by an 8-µm-thick Parylene-C membrane that is supported by two silicon substrates at both ends. The response of the fabricated strain gauge is experimentally characterized. The experiments show that the fabricated device has two sensitivity regions: a sensitive region with a gauge factor of 4.52, about 3.76× more than that for a
previously reported carbon-nanotube forest/PDMS based strain gauge, and a less sensitive region with a gauge factor of 0.87. Moreover, the response to gradual strain decreases is very similar to that for gradual strain increases, and the measured gauge factors are 4.4 and 0.77 for both sensitivity regions. The results are analyzed and the source of piezoresistance is explained.

Finite element analysis is performed for the strain gauge. The results show that the change in lateral separations between the carbons nanotubes, which are transversal to the direction of the applied force, are not equal in the center region, whereas the change in longitudinal separations between the carbon nanotubes, which are parallel to the applied force, are more equal.
Preface

A part of this thesis has been published in Bsoul, A., Mohamed Ali, M.S., Takahata, K. (2011) Piezoresistive Pressure Sensor Using Vertically Aligned Carbon-Nanotube Forests. IET Electronics Letters, 47(14), pp. 807-808. 255:139-144. In that publication, the work of the co-authors, Dr. Kenichi Takahata and Mohamed Sultan Mohamed Ali, is acknowledged; however, I conducted most of the fabrication and testing, and I wrote most of the paper’s manuscript.
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Dedication

To My Family
Chapter 1

Introduction

Carbon nanotubes (CNTs) are hollow cylindrical structures with a few nanometers in diameter made of carbon atoms and belong to the fullerene family [1-5]. A CNT can be thought of as a graphene sheet, with a hexagonal lattice, that is rolled up to make a nanotube [6]. Iijima was the first to discover carbon nanotubes in 1991 [7]. Since that time, CNTs have attracted considerable attention due to their exceptional electrical, mechanical, and optical properties [8-13].

Carbon nanotubes may be classified electronically depending on their geometrical structure, diameter and chirality, into metallic and semiconducting nanotubes [14]. Chirality is defined as the vector along which the graphene sheet is rolled up to make a carbon nanotube (Figure 1.1) [6].

Nanotubes can be classified into single-walled carbon nanotubes (SWCNTs) and multi-walled carbon nanotubes (MWCNTs) [15]. If the number of the rolled graphene sheets is more than one and the produced structure is a concentric tubes with various diameters, then the structure is called MWCNT [15]. On the other hand, the structure known as SWCNTs if it is only a single rolled up graphene sheet [15]. SWCNTs can be either metallic or semiconducting depending on the geometry, whereas MWCNTs are usually metallic [15].
Figure 1.1: The chirality vector describing an unrolled CNT

CNTs can be synthesized in different forms such as individual CNTs, mesh or unaligned CNTs, or vertically aligned CNTs (so called CNT forest, more details will be discussed in section 1.3).

There are a few applications for CNTs as additives, which add value to the product, have already been commercialized, such as the use of MWCNTs in lithium-ion batteries to achieve better mechanical stability [16]. However, there are still a large number of potential applications. Electronics; energy; sensors; field emission; and structural uses are some areas in which CNTs could potentially be used [16]. Carbon nanotube field effect transistors (CNT-FETs) using semiconducting SWCNTs and
interconnects made of bunch of metallic SWCNTs or large MWCNT, are potential applications of CNTs. Long mean free path and high current densities in CNTs were two reasons to propose the aforementioned applications [16, 17]. It was shown that some types of CNTs are more conductive than copper [18]. Large surface area and electrochemical stability of CNTs made them useful for energy applications research such as supercapacitors and batteries [16]. The outstanding mechanical properties of CNTs made them a potential material to be used in fibers for wide range of applications such as lightweight bullet-proof vests and aircraft body parts [16, 19].

Table 1.1 shows some of the mechanical properties of CNTs and other materials that are widely used in structural applications [16]. Low threshold voltage and high current densities for MWCNTs made them attractive field emitters [16]. Field emission displays using CNTs were demonstrated by well known companies such as Samsung and Motorola [16]. Sensors, which are used in our daily life, are other potential applications for CNTs. The CNTs-based sensors utilize the electrical, optical, and electromechanical properties of CNTs [16]. Gas sensors, biosensors, and physical sensors are examples on proposed CNTs based sensor [16].

The interesting electromechanical properties of CNTs made them considered as piezoresistors in various types of physical sensors, such as pressure sensors and strain gauges [20-24]. In CNTs-based piezoresistive sensing, which is the main topic of this thesis, CNTs were used in different forms, such as dispersed CNTs, individual CNTs, and CNT forests (more details are discussed in section 1.3).
### Table 1.1: Mechanical properties of CNTs and other structural materials [16]

<table>
<thead>
<tr>
<th>Material</th>
<th>Young’s modulus (GPa)</th>
<th>Tensile strength (GPa)</th>
<th>Density (g.cm(^{-3}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>SWNT/MWNT</td>
<td>~1000</td>
<td>~100-200</td>
<td>~0.7-1.7</td>
</tr>
<tr>
<td>High tensile steel</td>
<td>210</td>
<td>1.3</td>
<td>7.8</td>
</tr>
<tr>
<td>Carbon fibers</td>
<td>230</td>
<td>3.5</td>
<td>1.75</td>
</tr>
</tbody>
</table>

The context in the first section of this chapter points to the different techniques for nanotube synthesis. The second section presents the properties and deposition process of Parylene-C which is an important material in this work. Then, a background on the piezoresistive effect is presented. The fourth section brings out a detailed discussion on previous related work. The final section provides the summary and the outline of this thesis.

### 1.1 Synthesis of Carbon Nanotubes

The lack of understanding CNTs growth process made their production with a cost-effective synthesis technique and well-defined properties in large quantities is not possible until now [18]. However, several techniques are available to produce CNTs. Arc discharge, laser ablation, and chemical vapor deposition (CVD) are the three major available techniques [18, 25-28].
CNTs were produced and recognized for the first time by arc discharge method [28]. In this method, parts of the graphite anode are evaporated by arc discharge, which is generated between two closely spaced graphite rods with a DC voltage applied between them, in a reactor under inert atmosphere, usually helium [25, 28]. These evaporated parts of the graphite anode are deposited on the reactor walls as well as the cathode [28]. MWCNTs can be found in the deposit on the cathode [28]. SWCNTs can be produced by including metal catalyst in the anode, such as Co and Ni [25, 28]. One drawback with this method is the need to harvest the CNTs and separate them from other carbon deposits. Additionally, it is difficult to produce aligned CNTs [28]. The yield and the quality of the produced CNTs using this method are high [28].

Similar to arc discharge method, the synthesis of CNTs using laser ablation (known also as laser-furnace method) is based on evaporating a graphite target. The major components of the system are: a furnace which is used to heat a quartz tube containing a graphite target that might be doped with catalyst, and a cooled collector or trap to collect the evaporated carbon deposits [28]. A high power laser beam is used to ablate the graphite target while argon gas flowing through the tube [28]. Similar to arc discharge method, harvesting the CNTs is required, and the yield as well as quality of the produced CNTs are high.

The most popular method to produce CNTs is thermal chemical vapor deposition (T-CVD) [18]. A hydrocarbon vapor such as C$_2$H$_4$ or CH$_4$ is flowed through a tubular reactor heated by a furnace to thermally decompose the vapor and getting the carbon deposited on the substrate, which sits inside the reactor and has the catalyst material, forming the CNTs [18, 28]. The quality of the grown CNTs is lower than that for CNTs
produced using arc discharge and laser ablation methods [28]. T-CVD has many advantages over arc discharge and laser ablation due to its low cost, simplicity, ease of parameters control, high production yield, ability to produce CNT forests, and operation under low temperature as well as ambient pressure [18, 28]. Two-heating zones T-CVD system is used to synthesize CNTs in this work, more details are in section 2.4.

1.2 Parylene-C

Parylene-C is a biocompatible polymer with interesting properties that make it widely used in research and industry [29]. It is used as moisture protective layer for some electronics due to its low permittivity [30]. It is also used as coating for medical devices, such as stents, due to its biocompatibility [29]. Some mechanical properties of Parylene-C are listed in Table 1.2.

Parylene-C is a perfect match for CNT forests. This is because Parylene-C is deposited in vapor deposition process at room temperature making it unlikely to disturb the alignment of the CNTs within the forest. In this work, Parylene-C is used to bind the tops of the CNTs in the forest together. Parylene-C deposition system from Specialty Coating Systems (SCS) Inc. is used in this work [39]. Embedding a CNT forest in Parylene-C will change its mechanical properties. For example, coating a CNT forest with polydimethylsiloxane (PDMS) reduces the Poisson’s ratio of PDMS from 0.45 to 0.2 [37]. This reduction in Poisson’s ratio has been related to the limited contraction of the composite during strain due to embedding the CNT forest in the PDMS [37]. Same reduction rate is used for simulation in this work. Previous experimental work found that
coating a CNT forest with polyurethane (PU) increases the Young’s modulus by about 240% [38]. The Young’s modulus change rate found in [38] is used in our study when the CNT forest is embedded in Parylene-C.

**Table 1.2: Mechanical properties of Parylene-C [31-36]**

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Young’s modulus (GPa)</td>
<td>2.7</td>
</tr>
<tr>
<td>Tensile strength (MPa)</td>
<td>40-110</td>
</tr>
<tr>
<td>Poisson’s ratio</td>
<td>0.4</td>
</tr>
</tbody>
</table>

Parylene-C comes in granular form of dimer [30, 39]. As shown in Figure 1.2, the deposition process starts with the vaporizer that evaporates the dimer into gas by heating it at 150° C under vacuum of 0.1 Torr [29, 30, 39]. The dimer is decomposed to monomer by a thermo-chemical process called pyrolysis at 690° C under 0.5 Torr [29, 30, 39]. The deposition of the polymer form of Parylene-C takes place in the deposition chamber where the sample to be coated should be placed. This chamber is at room temperature and under a pressure of 0.05 Torr. Unlike many other CVD processes, the sample does not need to be placed in a hot chamber which allows variety of materials to be coated with parylene-C. Usually the sample is placed on a rotary stage to achieve a good conformal coating. The monomer gas that did not deposit on any surface in the deposition chamber will condense on the cold trap, which is kept at -90° C [30, 39].
Thicknesses from submicron to several microns can be achieved by controlling the amount of the loaded dimer [30, 39].

Figure 1.2: Block diagram represents the main components in Parylene-C deposition system with the temperature and pressure in each component [30, 39]

1.3 Piezoresistive Effect

The description of electrical resistance change due to applied mechanical stress is called piezoresistive effect [40, 41]. The electrical resistance \( R \) of a physical material is given by [42]:

\[
R \propto \frac{1}{\varepsilon}
\]
where $\rho$ is the resistivity of the material, $L$ is the length of the material, and $A$ is the cross-section area of the material. The relative change in the electrical resistance of physical material is approximated by [42]:

$$\frac{\Delta R}{R} = \frac{\Delta \rho}{\rho} + \frac{\Delta L}{L} - \frac{\Delta A}{A}$$  \hspace{1cm} (1.2)

The piezoresistive effect in metals is dominated by the change in metal geometry and the term $\Delta \rho/\rho$ can be neglected [42]. The effect in semiconductors is much larger than that in metals, about 1-2 orders of magnitude, and it is dominated by the change in electrical resistivity $\rho$ [42, 43]. This change in electrical resistivity has been related to the change in bandgap energy caused by mechanical stress [43].

Gauge factor is a measure of sensitivity of a piezoresistive material and it is defined as the ratio of relative change in electrical resistance to the relative change in length, strain, and it is given by [40, 42, 43]:

$$GF = \frac{\Delta R/R}{\Delta L/L} = \frac{\Delta R}{R \times \varepsilon}$$  \hspace{1cm} (1.3)

where $GF$ is the gauge factor, $R$ is the electrical resistance, and $\varepsilon$ is the mechanical strain. Piezoresistive effect has been used in many sensors, including pressure sensors, accelerometers, gyroscopes, and tactile sensors [44-48].

The observed piezoresistive effect of CNTs under applied mechanical stress could be related to intrinsic piezoresistive effect in them and/or to the interaction between the
CNTs themselves. Some types of CNTs have intrinsic piezoresistive effect whereas others do not. In metallic SWCNTs, an applied mechanical stress may open gaps in the band structure leading to change in the electrical resistance [49, 50]. Mechanical stress may change semiconducting SWCNTs electrical resistance by changing their band gap energy [49, 50]. The piezoresistive effect of different single-walled CNTs was studied [24]. The gauge factors for the studied CNTs ranges from -300 to 856 which are relatively high compared to that for Si [24].

However, MWCNTs, which are metallic most of the time due to the large diameters and multiple shells, do not have any intrinsic piezoresistive effect [15, 51]. Multi-walled CNTs were strained to their breaking point and did not show intrinsic piezoresistive behavior [51].

1.4 Related Work

Pressure sensors are one of the most commonly used sensors worldwide, and have an extensive range of applications in a variety of industrial fields (automotive, transportation, manufacturing, aerospace, environmental etc.), consumer products, as well as in biomedical areas. Wide use of pressure sensors is the fruit of research efforts over the last years [48, 52-56].

Piezoelectric, capacitive, and piezoresistive transduction techniques are widely used in pressure sensors and strain gauges. The input to be sensed is converted to its corresponding electrical signal [57]. Pressure on piezoelectric pressure sensor
generates stress on the piezoelectric element which generates electrical voltage that is used to measure the applied pressure [57].

The capacitive pressure sensors are usually designed as parallel plate capacitor and are comprised of an elastic electrode, which is sensitive to pressure, and a ridge electrode [57, 58]. The incident pressure deforms the elastic electrode that works as a diaphragm, changing the capacitance. This change in capacitance is translated to the value of the applied pressure [57].

The piezoresistive sensors exploit the piezoresistive effect in different materials. When the diaphragm of the pressure sensor is deformed due to an applied pressure, or a strain gauge is stretched due to an applied force, a mechanical stress is exerted on the piezoresistive element, which is integrated into the diaphragm, changing its electrical resistance. Usually, four piezoresistive elements are used to form a Wheatstone bridge circuit to achieve higher sensitivity in piezoresistive sensors [57]. Doped-silicon is one of the most popular used materials in the production of piezoresistive sensors due to its high gauge factor and well-developed fabrication techniques [59]. The gauge factor of state-of-the-art doped-silicon piezoresistors is up to 200 [23, 60]. Although the capacitive sensors have higher pressure sensitivity and less temperature sensitivity compared to piezoresistive sensors, the latter has simple circuit requirements which simplified the development of miniaturized piezoresistive sensors; moreover, piezoresistive sensors have highly linear outputs [58, 61].

Piezoresistivity of CNT-dispersed films as well as individual CNTs have been exploited to construct pressure sensors and strain gauges [22, 23, 60-63]. The CNTs
are horizontally placed in random or self-assembled manners, and are subjected to axial strains with applied pressures or forces.

CNTs, cellulose fibers, and polymer thin film is fabricated and tested under strain in [22]. The thin film fabrication steps included the removing of the CNTs from the substrate, and sonicating them in acetone as well as molding the film. In later steps, the film was peeled off and epoxied. The maximum achieved gauge factor was about 65 under a 500 micro-strains. In [23], commercially available single-walled CNTs were used to fabricate thin films and testing them under strain. First, the single-walled CNTs were purified, washed, dried, and then they were sonicated with surfactants. The thin films were made by filtering the suspension through polycarbonate membrane which was then attached to Si substrate. A strain was applied to compare the sensitivities of the single-walled CNTs membrane and the Si substrate. The achieved gauge factor was about 2.5x larger than that for the Si substrate that was used in the experiment. A pressure sensor utilizes carbon nanotubes film embedded in PDMS was demonstrated in [62]. The fabrication process included sonication of single-walled CNTs, photolithography, and peeling off the film. The achieved gauge factors were 9.6-20.5.

A bulk micromachined pressure sensor functionalized by an individual metallic single-walled CNT was fabricated and characterized in [60]. In addition to the conventional micromachining steps, arc discharge synthesized single-walled CNTs were dispersed in a liquid, and then deposited randomly on the top surface of an alumina membrane. Metal electrodes contacting both ends of the individual nanotube were defined by two steps: electron-beam lithography, which is time consuming, and lift off process. Both steps are based on the positions of the randomly adsorbed CNTs on
the alumina membrane. These positions were allocated by scanning the surface with atomic force microscopy (AFM). This fabrication process implies that the position of the randomly deposited CNTs might be different for each device, and hence the position of the metal electrodes might be different as well. The achieved gauge factor using this process is about 210 [60].

A prototype of a pressure sensor functionalized with a bundle of single-walled CNTs embedded in polymer was presented in [63]. The body of the sensor was made of Polymethylmethacrylate (PMMA). The CNTs were aligned between gold electrodes using a dielectrophoretic (DEP) nanoassembly technique. The achieved gauge factor was about 20. Since semiconducting nanotubes may show positive or negative gauge factors, having metallic and semiconducting nanotubes in the CNTs bundle was identified as a reason for having gauge factor lower than that for a single metallic single-walled CNT.

All the aforementioned approaches require additional coating/assembling steps to place CNT structures on the substrate. Vertically aligned CNTs (so called CNT forests) are attracting significant attention as they offer unique properties for many applications [64-66]. The CNT forest is a collection of vertically aligned CNTs; it can be viewed as a new class of functional bulk material with anisotropic electromechanical properties that can be utilized for many potential applications in micro-electro-mechanical systems (MEMS) and other emerging products. In carbon-nanotube forests, CNTs are self-aligned due to crowding at the beginning of their growth in the CVD process [64]. High aspect ratio geometries of carbon nanotube forests, which are desired in MEMS, are also achievable [65]. Large surface area and high conductivity are two of the unique
properties offered by CNT forests [66]. Furthermore, the total electrical resistance across the CNT forest can be highly dependent on the electrical resistances between the carbon nanotubes [66]. In the CNT forest, the current flow direction is from side contacts of individual CNTs, whereas the current flow direction is along the length direction of the nanotube in other cases [66]. The variation of individual carbon nanotube structures compared to carbon nanotube forests synthesized by CVD limits the production of devices based on individual carbon nanotubes [66].

CNT forests can be grown directly on a substrate using CVD process which eliminates the need for additional coating/assembling steps. The forests also exhibit piezoresistivity when a lateral stress is applied; a pressure sensor and a strain gauge based on this principle were recently reported [67, 37]. The source of piezoresistive effect in the CNT forest was related to the change in the gap between the vertically aligned CNTs [37, 67].

1.5 Summary and Outline

This thesis* presents the design, fabrication, and experimental characterization of a pressure sensor and a strain gauge. Both of them use multi-walled CNT forest supported by a flexible polymer (Parylene-C) membrane as the sensing element. The presented fabrication process overcomes the difficulties associated with using CNT forests.

* Part of the work which is related to the pressure sensor appeared in [68].
The results of the fabricated pressure sensor show different responses with higher performance compared to the results in [67] with keeping simple unpatterned forest structure. Unlike [67], nearly symmetric responses for both positive and negative gauge pressures are achieved in the developed pressure sensor. In addition, the work discusses a possible mechanism that explains the polarity of the pressure sensor response.

This thesis also presents characterization of the temperature coefficient of resistance (TCR) for CNT forest on a suspended Parylene membrane and for CNT forest on a silicon substrate. Based on the TCR characterization for the CNT forests, an approach that may contribute to suppressing the temperature dependence of similar devices is suggested.

We also show that the proposed strain gauge has a higher sensitivity compared to the one presented in [37]. A finite element analysis (FEA) is performed to analyze the deformation of the membrane in the strain gauge.

The entire thesis is organized in the following order: Chapter 2 presents the design of the proposed pressure sensor and strain gauge. Additionally, it brings out the details of the developed fabrication processes for the two devices. Chapter 3 explains the experimental set-ups which were used to characterize the devices, the results for the experimental work, and for the performed finite element analysis. Moreover, it discusses possible mechanisms to explain the observed results. Chapter 4 concludes this thesis and provides suggestions for future work directions.
Chapter 2*

Design and Fabrication of the Devices

Surface and bulk micromachining are two main fabrication techniques in MEMS. The fabrication processes for the pressure sensor and strain gauge in this work were developed through a Si-micromachining approach. The main processes used here are photolithography, thin film deposition, and etching. The rest of this chapter discusses the design and fabrication steps for the pressure sensor, and then describes the differences between the design and fabrication process for the pressure sensor and the strain gauge.

2.1 Pressure Sensor Design and Fabrication

The pressure sensor developed in this study is designed to have a suspended square membrane (5×5 mm²) of Parylene-C that supports an unpatterned multi-walled CNT forest (Figure 2.1). The two opposing ends of the forest are contacted with the metal pads on the substrate that are used to measure the forest electrical resistance. The metal pads are designed to have their edges overlapped with the two opposing ends of the forest to have a good electrical connection. These metal pads have electrical connection with the forest from the bottom (Figure 2.2).

* Part of this chapter which is related to the pressure sensor appeared in [68].
When a pressure is applied on the membrane, the membrane is deflected and a strain is generated in it as well as in the forest, leading to a change in the forest electrical resistance due to its piezoresistive effect. This design is different from previously reported CNT forest-based pressure sensor [67] in the shape of the membrane, which was circular, the CNT forest which was patterned, and the fabrication process as well. Additionally, the CNTs type and the Parylene-C thickness in [67] are not mentioned. These differences led to different responses.
The fabrication process for the sensor was developed through a Si-micromachining approach. The process steps are illustrated in Figure 2.3. The process starts from the formation of an 800-nm of Si nitride mask on both sides of a Si substrate using plasma enhanced chemical vapor deposition (PECVD). The purpose of forming Si nitride on the Si front side is to electrically isolate the metal pads, which will be deposited and patterned in later steps, from the Si substrate. This step is followed by patterning of a square window in the mask on the backside of the substrate to be used for etching the Si substrate using Xenon Di-Fluoride (XeF$_2$) in later step (Figure 2.3 step-1). A 130-nm-thick molybdenum film is deposited with an electron-beam evaporation technique and patterned with a lift-off step to form the metal pads on the front side of the substrate (Figure 2.3 step-2). A catalyst layer (2-nm Fe on 10-nm Al$_2$O$_3$) is then deposited and patterned in a similar manner to define the region where the CNT forest will be grown in the next step. The catalyst layer was deposited between the two molybdenum pads and overlapped with their edges to achieve better electrical connection between them and the CNT forest. A T-CVD system is used to grow a forest to the height of 400-600 µm using C$_2$H$_4$ as the carbon source (more details in section 2.4) (Figure 2.3 step-3). Next, an 8-µm-thick Parylene-C film is deposited enveloping the forest of CNTs and binding the tops together with the deposited Parylene film (Figure 2.3 step-4). Coating the CNT forest with Parylene-C is less likely to disturb the alignment of the CNTs compared to using drop coating to deposit other polymers like PDMS in [37]. The Si substrate is dry etched with XeF$_2$ through the backside window of the Si nitride mask, releasing the Parylene membrane with the CNT forest (Figure 2.3
Finally, the top layer of the nitride mask is stripped using CF$_4$ plasma to complete the fabrication (Figure 2.3 step-6).

Potassium hydroxide (KOH) was not used to etch the Si substrate because the etching process using KOH needs to be done at temperature close to the glass transition temperature of the Parylene-C. On the other hand, HNA (hydrofluoric, nitric, and acetic acids) was not used because it etches both of Si oxide and Si nitride which means a timed etching is required when any of these materials are used as etch-stopper; however, timed etching is usually difficult to control. Moreover, HNA is considered an extremely aggressive and dangerous acidic mixture. Additionally, using wet etching might disturb the alignment and drastically modify the CNTs structure because of the capillarity effect [69]. Due to the aforementioned reasons, using XeF$_2$ to dry etch the Si substrate and using CF$_4$ plasma to dry etch the Si nitride layer was more suitable alternative. Figure 2.4 shows a typical sensor fabricated by the above process.

1) Deposit Si nitride and patterning of a square window
2) Deposit molybdenum with electron-beam evaporation and pattern it with lift-off

3) Deposit the catalyst layer and grow the CNT forest

4) Deposit the Parylene-C
5) Etch the Si using XeF$_2$ (Dry etch)

6) Etch the top Si nitride using CF$_4$ Plasma

**Figure 2.3:** Top and cross-sectional views of the fabrication process for the pressure sensor
Figure 2.4: Optical picture of fabricated sample device (top left) with close up of CNT forest (top right) and SEM picture of fabricated sample device (bottom left) with close up of CNT forest (bottom right)

2.2 Strain Gauge Design and Fabrication

The strain gauge developed in this study is designed to have a suspended rectangular membrane (15×2.5 mm²) of Parylene-C that has an unpatterned square multi-walled CNT forest (5×2.5 mm²) in its center with both ends attached to Si
substrates (Figure 2.5). Similar to the pressure sensor in section 2.1, the two opposing ends of the forest are contacted with the metal pads on the substrate that are used to measure the forest electrical resistance. When a force is applied to stretch one of membrane ends, the membrane is deformed and a strain is generated in it as well as in the forest, leading to a change in the forest electrical resistance due to its piezoresistive effect.

**Figure 2.5:** The design of piezoresistive strain gauge with CNT forest

The main difference between this design and the design in section 2.1 is that the two silicon ends are completely separated after the backside etching using the XeF$_2$. This is achieved by changing the shape of the patterned window in the Si nitride mask on the backside from a square window to a rectangle one extended along the width of
the Si substrate. Figure 2.6a shows the backside of a pressure sensor and Figure 2.6b shows the backside of a strain gauge. The metal pads are realized using electron-beam evaporation technique and followed by lift-off process; however, 100-nm of copper (Cu) on top of an adhesive layer of 20-nm of chromium (Cr) is used. Cr is used because it has good adhesive properties to Cu, Si, Si nitride, and Si oxide. The height of the vertically aligned CNT-forest is different as well.

Before the backside etching of the substrate using XeF$_2$, the strain gauge is attached to a supporting plastic frame that has a window in its center to ensure that the two Si substrates will not move after releasing the membrane, maintaining the original shape of the membrane. The edges of the frame are separated before testing. Figure 2.7 shows a typical strain gauge fabricated by the process.
Figure 2.6: (a) The backside of piezoresistive pressure sensor CNT forest (b) The backside of a piezoresistive strain gauge with CNT forest
2.3 Carbon Nanotubes Chemical Vapor Deposition

The CNT forests used in this project are synthesized using an atmospheric thermal CVD reactor tube* built in-house. The reactor tube consists of two heating zones: the first zone is used to preheat the gases that flow within the quartz tube up to 850 °C by a tube oven, and the second zone is a subsequent larger diameter portion of the quartz tube. The sample is placed on top of a resistive heater (highly doped silicon) in the second heating zone. When the preheated gas reaches the heated sample (up to 800 °C), vertically aligned multi-walled CNTs start its growth. Figure 2.8 shows a schematic of the synthesis system whereas Figure 2.9 shows the real synthesis system.

* This reactor tube is similar to a system designed by Dr. Alireza Nojeh’s Group and was built by A. Bsoul and M. Dahmardeh.
Figure 2.8: Schematic of the CVD system
After loading the sample, the oven is turned ON while argon (Ar) is flowing at 338 ccm until the temperature reaches 850 °C. An electrical current is then passed through the resistive heater (highly doped silicon) to heat it up to 800 °C for 2 minutes. The flow rate of Ar is then reduced to 137 ccm and hydrogen (H₂) is flowed at 260 ccm for 5 minutes. Then, Ar flow rate is reduced again to 100 ccm, H₂ is kept at 260 ccm, and Ethylene (C₂H₄) is flowed at 110 ccm for the growth period. General basis, in an hour time, a few 100’s of μm of vertically aligned multi-walled CNTs could be grown. To end the growth process, the flow of C₂H₄ and H₂ is turned OFF. The electrical current and the oven are also turned off, whereas argon is flowing until the system cools down. The reaction tube used in this work has an advantage over other one-heating zone systems; it gives the ability to separately control the temperatures of the gasses and the sample.

It is important to note that the CNT forests growth was not always repeatable. Sometimes, the growth was homogeneous across the whole area of the CNT forest. During other times, empty areas or non-uniform growth were observed. Having no growth at all was observed as well. The reasons for these variations in the CNT forests growth were not clear. These variations also led to have insensitive sensors in some cases. Repeated heating of semiconductors might lead to change in its properties including the resistivity [70]. This means a change in the properties of the Si resistive heater in the presented reaction tube. Accordingly, a change in the resistive heater generated temperature is expected. It was observed that temperature is an important parameter in the growth process. This possible temperature change in the resistive heater might be a possible reason for the limited repeatability in growing CNT forests.
Figure 2.9: Carbon nanotube growth system
Chapter 3*

Experimental Set-ups, Results, and Analysis

Characterization of any device is essential to judge its success. Devices characterization is done by using experimental set-ups that allow varying the input parameters, pressure and strain, and noting the output in response to these variations. The results are analyzed and the possible mechanisms led to the results are discussed. Moreover, comparing results with other similar studies is pursued. Finite element analysis is performed to analyze the deformation of the membrane in the strain gauge due to the applied mechanical strain. The rest of this chapter presents the experimental set-ups for the CNT-forest based pressure sensor and strain gauge, and then it brings out the results, analysis, and detailed discussions.

3.1 Pressure Sensor Experimental Set-up

The fabricated sensor was attached to a glass plate and hermetically sealed using silicone glue so that the cavity contains air at atmospheric pressure. The sensor was placed inside a pressure chamber with a commercial pressure sensor (PX26, Omega Engineering Inc.) that measures the chamber's inner pressure. The chamber's inner
pressure was varied, using a pump, to have the chamber’s inner pressure ranging from negative gauge pressures, lower than atmospheric pressure to deflect the sensor’s membrane upward, to positive gauge pressures, higher than atmospheric pressure to deflect the sensor’s membrane downward. The change in electrical resistance between the two molybdenum pads was recorded in response to the chamber’s inner pressure variations. Figure 3.1 illustrates the experimental set-up.

![Figure 3.1](image)

**Figure 3.1**: Schematic of pressure sensor experimental set-up

The dependence of CNT forest electrical resistance on ambient temperature was tested due to its importance for the sensor calibration purposes. This was carried out by placing the chamber containing the device on a hotplate. A thermocouple was used to
read the chamber’s temperature (Figure 3.2). The changes in electrical resistance in response to the chamber’s temperature increments were recorded for both of a pressure sensor, which has a CNT forest on the Parylene membrane suspended in air, and for a CNT forest fixed on the Si substrate (before Parylene coating).

![Figure 3.2: Schematic of temperature experimental set-up](image)

**3.2 Strain Gauge Experimental Set-up**

The strain gauge was attached to a plastic supporting frame, before releasing the membrane and separating the two Si substrates by dry etching, to prevent any deformation and possibly damaging the membrane before testing. The plastic frame has a window with dimensions larger than that for the window in the Si nitride film on the backside of the device and overlapping with it, so the frame will not be an obstacle for
etching the Si substrate through the window in the Si nitride film. At the end of the dry etching process, the frame was fixed to the experimental set-up which consists of two stages fixed on the same base; the first is a stationary stage, and the second is a movable one that can move in the x-direction with resolution of 10 μm / step. Afterward, the two edges of the plastic frame were separated by melting them instead of cutting them to avoid any application of physical force that might affect the positioning and alignment of the sample. Finally, one of the ends was stretched using the moveable stage. The changes in the device electrical resistance due to the generated strains in the CNT forest were recorded. The experimental set-up is shown in Figure 3.3

![Figure 3.3: Strain gauge experimental set-up](image)
3.3 Pressure Sensor: Experimental Results

Figure 3.4 and Figure 3.5 show the responses of a fabricated pressure sensor with initial resistance of 200 Ω. The measurements done at room temperature for positive and negative gauge pressures, over five and three measurement cycles, respectively. As can be seen in the graphs, positive gauge pressures, which deflect the membrane downward by increasing the chamber's inner pressure to be higher than the cavity's pressure, decrease the sensor's electrical resistance, whereas negative gauge pressures, which deflect the membrane upward by decreasing the chamber's inner pressure to be lower than the cavity's pressure, increase the electrical resistance.

![Graph showing the response of a pressure sensor](image)

**Figure 3.4:** Measured CNT-forest resistance vs. positive gauge pressure
The average sensitivities in the positive and negative pressures are calculated to be -986 ppm/KPa and +816 ppm/KPa, respectively. It is notable that the polarities of these responses are opposite to the results reported in [67], which has average sensitivities in the positive and negative pressures of +1400 ppm/KPa and -170 ppm/KPa, respectively. Moreover, the presented sensor is 4.8× more sensitive than the sensor reported in [67] for negative gauge pressures, providing comparable sensitivities in both pressure polarities, which is an advantage over the device in [67]. These differences may be related to various factors, including the shape and area of the diaphragm, the type and height of the CNTs, the pattern of the CNTs, or any...
combination thereof. The diaphragm in [67] has a circular shape whereas it is a square shape in this study. Additionally, the type and height of the CNTs in [67] are not mentioned. The CNT forest was also patterned in meander shape in [67].

![Diagram of CNTs under positive gauge pressure]

**Figure 3.6**: Cross-sectional view of CNTs under positive gauge pressure

The source of piezoresistive effects of CNT forests has been related to the change in distances between individual CNTs due to lateral strains applied to the forest [37, 67]. This change in distances between the individual CNTs might increase or decrease the number of junctions between the entangled CNTs and hence the forest electrical resistance might change.

A positive pressure that deflects the membrane downward can widen the CNT separation around the center region of the membrane and increase the electrical resistance in the region; however, it can also narrow the separation around the two
border regions between the membrane and the substrate thereby decreasing the electrical resistance in these regions (Figure 3.6). On the other hand, a negative pressure that deflects the membrane upward can narrow the CNT separation around the center region of the membrane and decrease the electrical resistance in the region; however, it can also widen the separation around the two border regions between the membrane and the substrate thereby increasing the electrical resistance in these regions. The polarity may be determined by collective piezoresistive effects across the forest including the two border regions. In the present sensor, these border regions are relatively large and could have played the dominant role in the overall piezoresistive effect. It was noted also that there is no generated current due to applied stress. This means that the CNT forest embedded in Parylene-C does not have a piezoelectric effect.

The dependence of CNT forest electrical resistance on ambient temperature is an important characteristic of the sensor for calibration purposes. Figure 3.7 plots measured electrical resistance changes against temperature from room temperature up to 60 °C for a forest fixed on the Si substrate (before Parylene coating) as well as for a forest on Parylene membrane suspended in air. An approximate temperature coefficient of resistance (TCR) for the forest suspended with the Parylene membrane was measured to be -515 ppm/°C, about 3× smaller than the TCR for the forest-on-Si case as shown in Figure 3.7. The TCR for patterned CNT forest coated with Parylene-C was -1900 ppm/°C in [67] which is comparable to the observed TCR, -1412 ppm/°C, for unpatterned CNT forest before coating with Parylene-C. Although the exact mechanism of the difference in TCR for a forest fixed on the Si substrate (before Parylene coating)
and for a forest on a Parylene membrane suspended in air is unclear, it may be related to the difference in the coefficients of thermal expansion of Parylene-C and Si. The coefficient of thermal expansion of Parylene-C is $3.5 \times 10^{-5} \text{K}^{-1}$ [71], about 10× larger than that for Si, which is $2.6 \times 10^{-6} \text{K}^{-1}$ [31]. A lateral expansion of the Parylene membrane due to a temperature increase leads to a widening of CNT separation or an increase in forest electrical resistance, counteracting the negative TCR and reducing its magnitude. This is suggesting that minimizing the forest area on Si may contribute to suppressing the temperature dependence of the sensor.

Figure 3.7: Measured temperature dependence of CNT-forest resistance
3.4 Strain Gauge: Experimental Results

Figure 3.8 shows the response of a fabricated strain gauge, with initial resistance of 70 Ω, for the applied strain, measured at room temperature, over five measurement cycles. The applied strain is generated by stretching one of the device ends. As can be seen in the graph, applying a strain increases the device electrical resistance. The curve in Figure 3.8 may be divided into high and low sensitivity regions, ranging from 0% strain to ~1.5% strain and from ~1.5% strain to ~3% strain, respectively. The high sensitivity region has a gauge factor of 4.52, about 3.76× more than that for a previously reported carbon nanotube-forest/PDMS based strain gauge in [37], whereas the low sensitivity region has a gauge factor of 0.87. It is notable that the polarity of this response is similar to the results reported in [37]; however, the sensitivities are different. This differences in sensitivities may be related to various factors, including the shape and area of the diaphragm, the height of the CNTs (9.3 μm in [37] compared to 300-400 μm in this study), the polymer material (PDMS in [37] compared to Parylene-C in this study), the quality of the CNTs alignment which might be disturbed in case of [37] due to drop coating of PDMS on the forest and peeling off the forest/PDMS composite from the Si substrate, or any combination thereof.
Figure 3.8: Measured CNT-forest resistance vs. strain

Studying the behavior of the device while restoring its original values, by gradually reducing the input from its maximum value to its minimum value, is an important indicator of the device ability to respond for vibrating input values. After reaching the maximum applied strain during the experiments, the applied strain was reduced gradually and the output readings were taken (Figure 3.9). The results in Figure 3.9 are slightly different than the results in Fig 3.8. The high sensitivity region has a gauge factor of 4.4, about 2.65% less than that for the same region in Figure 3.8, whereas the low sensitivity region has a gauge factor of 0.77, about 11.49% less than that for the
same region in Figure 3.8. The mechanism of these deviations is not clear but might be related to stress relaxation in the parylene membrane.

Figure 3.9: Measured CNT-forest resistance vs. strain while gradually reducing the applied strain

Unlike the pressure sensor in section 3.3, the deformation nature of the CNT forests here is different due to the difference between the applied inputs; a pressure normal to the membrane compared to a force parallel to the membrane.

FEA using COMSOL Multiphysics 4.0 indicates that stretching one of the device ends increases the length of the membrane (the dimension of the CNT forest parallel to the applied force) and decreases its width (Figure 3.10). The increment in length is
expected to widen the CNT separation and increase the electrical resistance of the CNT forest. However, the decrement in width is expected to narrow the CNT separation and decrease the electrical resistance of the CNT forest. The overall change in electrical resistance is determined by collective piezoresistive effects across the forest.

![Figure 3.10](image.png)

**Figure 3.10:** Deformed shape of the membrane that has been stretched 100 µm, the deformation is scaled 10×

The simulation results show that the increment in length is larger than the decrement in width in most of the membrane regions. This means an increment in the forest electrical resistance is expected due to the applied strain, which agrees with the experimental results. The results in Figure 3.8 were compared to results calculated based on the equation of a piezoresistive effect in metals; however, they didn't match.
This means the piezoresistive effect a CNT forest embedded in Parylene-C can not be modeled with this equation [42]:

\[
\frac{\Delta R}{R} = \frac{\Delta L}{L} - \frac{\Delta A}{A}
\]  \hspace{1cm} (3.1)

Figure 3.11 shows the change in separation in lateral direction between equally spaced points (about 20 µm between each two points) along the line A-A’, which is shown in Figure 3.10. The X-axis shows the position on the line A-A’, whereas the Y-axis shows the changes in separation between each point and the one next to it. The negative values indicate reduction in the separation between the points. Since the curve does not show a constant value, then the changes in separation are not equal. According to the curve, the maximum reduction in separation between the points is located at the edges, whereas the minimum is located at the center. Figure 3.12 represents the change in separation in longitudinal direction between equally spaced points (about 10 µm between each two points) along the line B-B’, which is shown in Figure 3.10. Similar to Figure 3.11, the X-axis shows the position on the line B-B’, whereas the Y-axis shows the changes in separation between each point the one next to it. The positive values indicate increase in the separation between the points. Since the curve does not show a constant value, then the changes in separation are also not equal; however, the non-uniformity is low. According to the curve, the maximum increase in separation between the points is located at the center, whereas the minimum is located at the edges.
Figure 3.11: The change is distance in lateral direction between equally spaced points on line A-A' due to strain.

Figure 3.12: The change is distance in longitudinal direction between equally spaced points on line B-B' due to strain.
A comparison between the results in Figure 3.11 and Figure 3.12 shows that the non-uniformity of the change in separation between equally spaced points along the line A-A’ is higher than that for the line B-B’. The absolute maximum value in Figure 3.11 is 0.192 µm, whereas the absolute minimum value is 0.059 µm. The percentage difference between them, as an indicator on the non-uniformity, is about 225%. The absolute maximum value in Figure 3.12 is 0.410 µm, whereas the absolute minimum value is 0.393 µm. The percentage difference between them, as an indicator on the non-uniformity, is about 4%. This means that the change in separation between CNTs in longitudinal direction (parallel to the line B-B’) is almost equal and uniform, whereas the change in separation between the CNTs in lateral direction (parallel to the line A-A’) is not uniform in the region around the line A-A’. The level of non-uniformity of the change in separation between the CNTs in lateral direction is expected to drop as the position gets closer to the Si substrates because the membranes is fixed there and does not move. The results in Figures 3.11 and 3.12 were extracted from the simulation using COMSOL Multiphysics 4.0. One step of FEA is to mesh the structure by taking representative points to find the solution for them in later steps. Figure 3.13 shows the meshed structure. The points are not selected to form a uniform grid. They density of points changes from one region to another within the structure based on the necessity to get the solution. Accordingly, it is very unlikely to find equally spaced points aligned on a single line like A-A’ or B-B’ in Figure 3.10. Based on that, the extraction of the data in Figures 3.11 and 3.12 was not direct, extra steps after the FEA were required to overcome this problem. Firstly, the original positions of the points aligned on lines A-A’ and B-B’ are extracted from the simulation although these positions are not equally
spaced. Then, the new positions after the deformation of the membrane for the same points are extracted. Curve fitting technique is used to generate functions that can generate the new positions of points on the membrane after applying strain from uniformly separated points. From these functions, the change in separation between the points, which represent the change in separation between CNTs, is extracted. Figure 3.14 illustrates the process.

**Figure 3.13:** The structure after meshing in COMSOL Multiphysics 4.0 (Free Tetrahedral extreme coarse meshing is used)

This FEA is an initial step toward better understanding for the electrometrical properties of CNT forests embedded in polymer. Moreover, it can be the first step toward producing software packages that can predict the piezo resistive effect of CNT forests embedded in polymer under mechanical forces by relating the membrane
deformation to the change in the forest electrical resistance. Understanding the mechanical interaction between the individual CNTs within the forest combined with the observed electrical resistance behavior might lead for better understanding the properties of the individual CNTs as well.

Figure 3.14: The process flow of generating the data representing the change in separation between the CNTs due to the applied stress
Chapter 4

Conclusions

The presented research work consists of design, fabrication, and characterization of two CNT-forest-based devices; a pressure sensor and a strain gauge. A fabrication process based on Si-micromachining techniques that overcomes the challenges associated with use of CNT forests was developed for the devices construction. These challenges were imposed because the CNT forests are fragile due to low spatial density of CNTs; making it difficult to integrate them with standard microfabrication processes. For example, the fabrication process is required to avoid wet processing as it causes densification of CNTs when dried and that changes the geometries and properties of the forest. Another challenge associated with the use of CNT forests is the need for relatively high temperatures (several 100s °C) to synthesize them in CVD process. This limits the materials that could be used in the fabrication process.

The pressure sensor was fabricated to have a multi-walled carbon-nanotube forest supported by a deflectable 8-µm-thick Parylene-C membrane that is suspended by the silicon frame. The responses of the fabricated sensor were experimentally characterized. The fabricated pressure sensor exhibited average sensitivities of 816-986 ppm/KPa comparable in both positive and negative gauge pressures, an advantageous feature as a gauge pressure sensor while using simple forest structures. New findings and possible mechanisms in the polarity of the piezoresistive response of
the forest, as well as temperature dependence of the forest electrical resistance were also reported. The CNT forest suspended with the Parylene membrane was observed to provide a 3× smaller TCR in comparison with the forest fixed on the Si substrate. This suggests that minimizing the forest area on Si may contribute to suppressing the temperature dependence of the sensor.

The strain gauge was fabricated to have a multi-walled carbon-nanotube forest supported by an 8-µm-thick Parylene-C membrane that is supported by two silicon substrates at both ends. The response of the fabricated strain gauge was experimentally characterized. The results showed two sensitivity regions based on the sensitivity; sensitive and less sensitive regions. The exhibited gauge factor in the first region was 4.52, about 3.76× more than that for previously reported CNT-forest/PDMS based strain gauge. The gauge factor in the second region was 0.87. The observed hysteresis was relatively small. The exhibited gauge factors to gradual strain decreases, in the same two regions, were 4.4 and 0.77. The results were analyzed and the source of piezoresistance was explained. Finite element analysis was performed to explain the deformation of the membrane in response for the applied strain.

The presented devices have potential applications in such broad areas that involve temperatures below glass-transition temperature of Parylene (typically 80-100 °C). They include medical applications (with appropriate device optimization), because the devices are coated with Parylene-C, a biocompatible polymer, and usually operated only around the body temperature.
This effort has advanced the knowledge and technology in CNT-forest-based pressure and strain sensors as well as in piezoresistive properties of CNT forests. The results will facilitate further developments for not only pressure and strain sensors, but also broader MEMS devices based on CNT forests.

The development of software packages that can predict the piezoresistance of CNT forests under applied mechanical stress is an important future work direction for better understanding of the electromechanical properties of CNT forests. This will be an important step toward the commercialization of CNT forests-based sensors. Another direction can be the improvement of the sensitivities of the presented sensors by modifying the geometrical structure of the CNT forest embedded in Parylene-C, such as having a corrugated structure. Moreover, fabricating a CNT forest-based accelerometer using the presented fabrication process is another direction.
References


