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Carbon Nanotube Based Sensors on Polymer Substrates for Pressure and Strain Measurements

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Outline:

Introduction

Silicon pressure sensors based on micro-electro-mechanical systems (MEMS) technologies are gaining popularity for applications in bio-medical devices over the past few decades. These MEMS-based sensors are inexpensive to manufacture and give many advantages in performance. The major drawbacks of using a silicon-based pressure/strain sensor in implant and in-vivo medical diagnosis tools are that it is not biocompatible, is rigid, and provides no flexibility compared to polymeric materials [1].

Owing to these reasons, a search for alternative materials to build a pressure/strain sensor is carried out. After an intensive search and a study of current research outputs, as well as interviews with other researchers, we have come across two suitable materials, which are, carbon nanotubes and SU-8 photoresist.

SU-8 photoresist is a special kind of photoresist that can be used as a standalone building material. It provides great flexibility, and most importantly, it is biocompatible. The SU-8 will eventually eliminate and replace the use of conventional silicon as a core sensor construction material.

Carbon nanotubes are getting widely known for their unique characteristics and sensing capability. This has led us to investigate and research its use as the sensing medium on polymeric-based sensing device.

SU-8 Photoresist

SU-8 is an epoxy-type, negative tone, near-UV (365nm) photoresist. It has been widely used in microelectronics packaging in the past decade, and will be continue to get popular for use in photolithography in MEMS applications due to it thermal stability, good chemical resistance, and compatibility to conventional MEMS fabrication processes.

The thickness of a cross-linked SU-8 photoresist can extend from 0.1nm to 100µm. Thus, SU-8 provides high aspect ratio and tall structure features which yields advantages for a low cost LIGA process (lithography, electroplating and moulding). SU-8 can withstand high temperature and chemical environments when it is fully cured and cross-linked. Therefore it is often used as a permanent structure material in MEMS applications.

Carbon Nanotubes

Carbon Nanotubes (CNTs) is a material that has an outstanding potential to become a new type of sensing medium in novel MEMS based sensors, due to its properties, such as nano size, high strength, high specific surface area, high electrical and thermal conductivities and possesses semiconducting characteristic [2].

The CNTs can be visualised as a cylindrically shaped graphite hollow fibers, whose walls are layers made of hexagonal structured networks of carbon atoms. Depending on the graphite cylinder arrangement, two types of CNTs can be produced, the single-walled carbon nanotubes (SWNTs) and the multi-walled carbon nanotubes (MWNTs). The diameter of the carbon nanotubes is approximately 1.5 to 2nm and around 1 to 15µm in length.

SWNTs and MWNTs have their own distinctive properties. In general, there are four important properties found in CNTs, the chemical reactivity, electrical conductivity, mechanical strength, and optical interaction. This chapter focuses only on the electrical conductivity of the CNTs. The conducting properties are influenced by the molecular structure within the nanotube. This results in different band structure and leads to differences in the conducting band gap [3]. Therefore, the CNTs can be either metallic or semiconducting.

Using CNTs as a novel sensing medium for pressure/strain sensors has been investigated and documented. This investigation shows that the mechanical deformation and electrical properties of CNTs give promising evidence for a measureable piezoresistive effect in the CNTs to be used as a sensing medium. The benefit of using CNTs compared to conventional doped polysilicon pressure/strain sensors is that CNTs are very elastic and can be bent to a small radius without damaging the nanotube [4]. Furthermore, and more importantly, they provide a much higher gauge factor, which is up to 100 times as reported initially [5].

Using the CNTs as a sensing medium is promising, but they are also prone to certain limitations and handling difficulties. For example, production of pure and uncontaminated nanotubes is time consuming and costly. As well, the handling of CNTs is extremely difficult.

Approach and Methodology

The standard fabrication procedure adopted in MEMS-based sensor fabrication provides a high degree of intergrability into a microelectronic sensor subsystem [6]. In view of drawbacks of using the silicon wafer as the substrate material mentioned previously, an investigation on the feasibility of realising pressure/strain sensors using polymeric materials and CNTs was undertaken, and has been reported by the authors recently [7, 8].

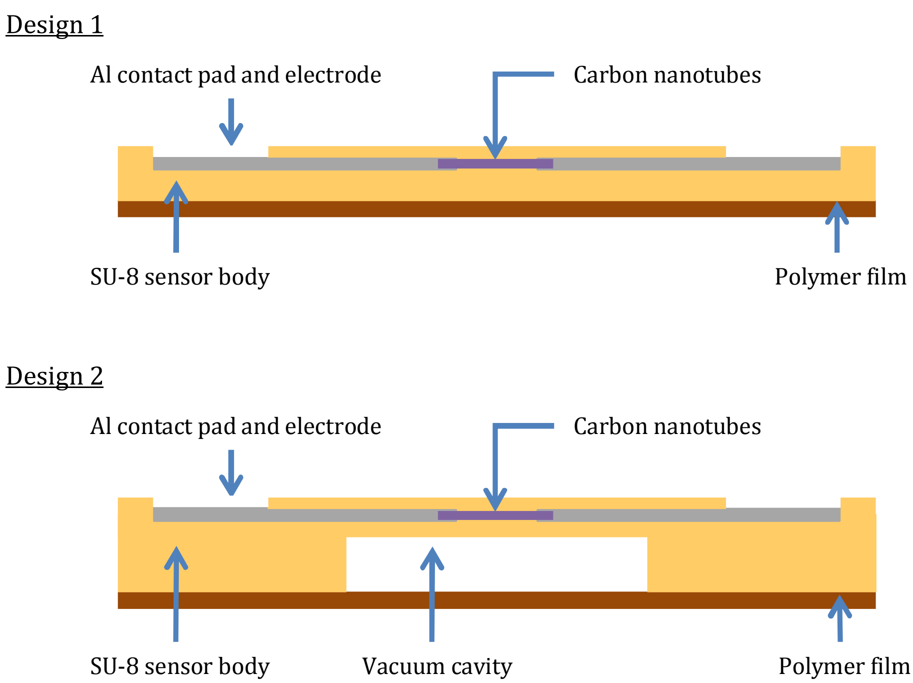

FIGURE 3.1
New sensor design concepts; design 1: Flat, and design 2: Cavity

The concept of a flexible polymer- and CNT-based sensors is shown in figure 3.1. There are two different designs; namely, “flat design” and “cavity design” sensors. The “flat design” sensor is easy to fabricate and is best used in bending force or strain measurements, whereas the “cavity design” concept involves advanced procedures to create a vacuum cavity.
These proposed sensors use a flexible substrate made of polyimide film as the sensor platform. The overall body structure of the sensor was made using SU-8. The “flat design” sensor prototype is used in our current research stage to examine the functionality of the CNTs under applied pressure and bending forces.

Prototype Fabrication Process

A prototype sensor was manufactured and both the “flat design” and “cavity design” sensors are fabricated and tested. During the early phase of the fabrication experiments, a silicon wafer was used as a carrier. This allowed us to easily investigate and obtain an optimum recipe in creating the SU-8 structure, and also to examine and understand the CNTs deposition process.
Once all major parameters are investigated, polyimide film is substituted for the silicon substrate, and henceforth, forms the actual base structure of the sensor as originally proposed.
The procedures for fabricating the prototype sensor using either a silicon wafer or polyimide film are similar in many ways, and the process is illustrated in figure 3.2. The illustration shows the fabrication process of the “flat design” sensor only, with polyimide film as the base material.

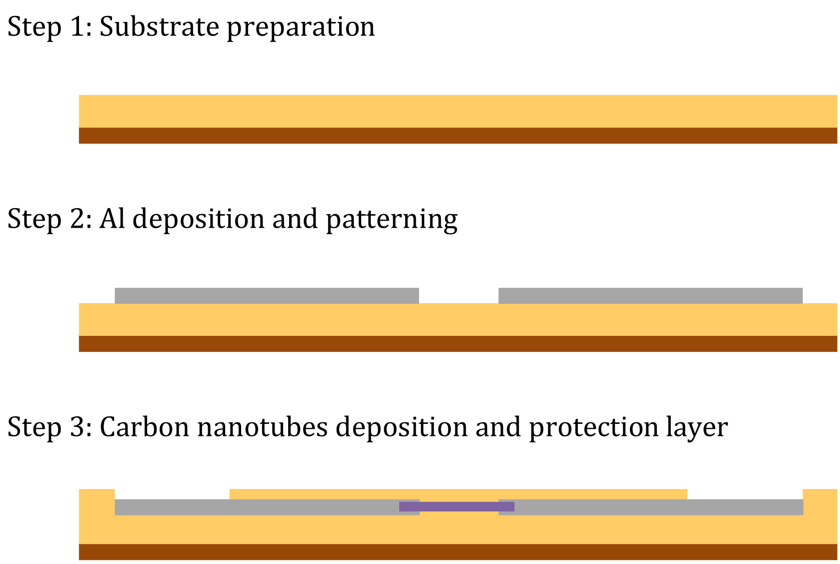

FIGURE 3.2
Fabrication process of the “flat design” sensor

The fabrication process starts with the preparation of the base substrate, followed by the definition of the microelectrodes, which are shown in figure 3.3. To deposit and manipulate the CNTs across the microelectrode gap, dielectrophoresis (DEP) method is used. The DEP method requires an AC current to align the CNTs between electrode pairs.

Following the DEP process, the sensor is subjected to a voltage test. This test is to ascertain that the CNTs have been properly deposited across the gap of the microelectrodes, and that a continuous bridge is thus formed.

FIGURE 3.3
The aluminium miroelectrodes

Finally, a protection layer of SU-8 is layered to protect the sensor and to allow it to withstand the subsequent test with applied pressure or bending force. The completed prototype “flat design” sensor array is shown in figure 3.4.

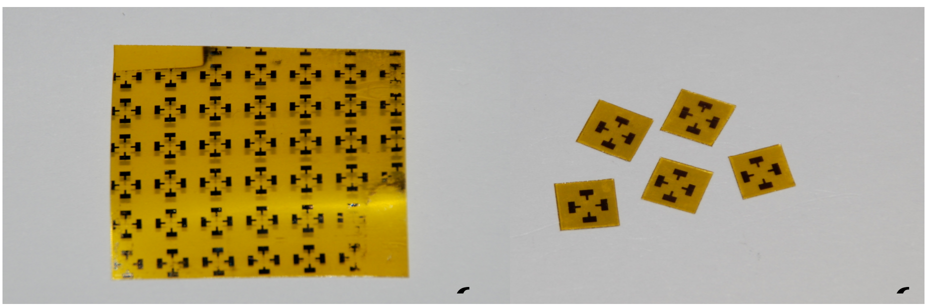

FIGURE 3.4
(a) Flexible “flat design” sensor array, (b) cut out of individual sensor

The fabrication process for the “cavity design” sensor is similar compared to the “flat design” sensor, where additional procedures are required to create the cavity structure directly under the microelectrodes tips. Figure 3.5 shows the prototype sensor array “cavity design” on silicon wafer, as well as individual sensors after the lift-off process.

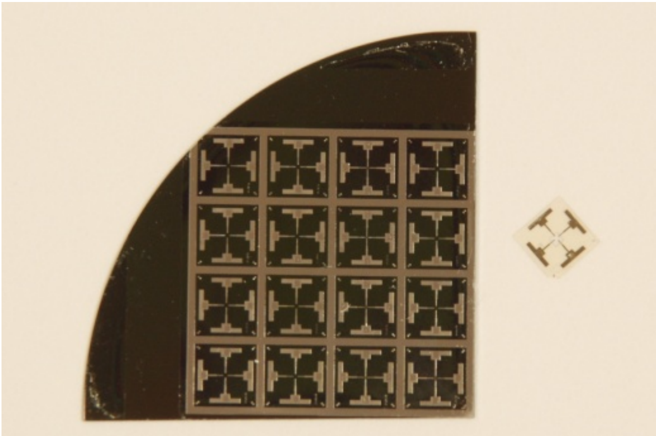

FIGURE 3.5
(a) Flexible "flat design" sensor array, (b) cut out of an individual sensor

Preliminary Test Results

The outcomes of the DEP process and the prototype sensor testing results are presented in this section. Before the DEP process takes place, the carbon nanotube solution needs to be prepared. In this particular experiment, 2mg of single-walled CNTs is diluted in 1ml of deionised (DI) water. The applied electric field is set to from 5 to 10Vpp and the frequency from 1 to 5 MHz of a sinusoidal signal.

DEP Experimental Results

A drop of 2µl of the solution containing CNTs is placed in between the tips of the microelectrodes and the AC signal is applied for 4 minutes. Figure 3.6 shows an SEM image which illustrates the before-and-after effect of the CNTs deposition process.
As shown in the SEM image, the DEP process is successfully carried out to deposit the CNTs and in a manner that bridges the microelectrodes. The measurement of resistance values across the entire sensor sample is consistent with respect to specific deposition times. Resistance values ranging between 250kΩ and 500kΩ are recorded. Further analysis shows, the longer the DEP deposition time the lower the resistance value. This is caused by the gradual CNT alignment with and across the electrode gap, hence reducing the resistance to the passing current.

Sensor Pressure Test Results

Two types of tests are carried out after the completion of the DEP deposition of the CNTs between the microelectrode's tip. The first test is the pressure test, which applies to the "cavity design" sensor. Second, the bending test which is applied to the "flat design" sensor. Both tests are intended to determine the resistance change in CNT-formed conduit when pressure and bending forces are applied.

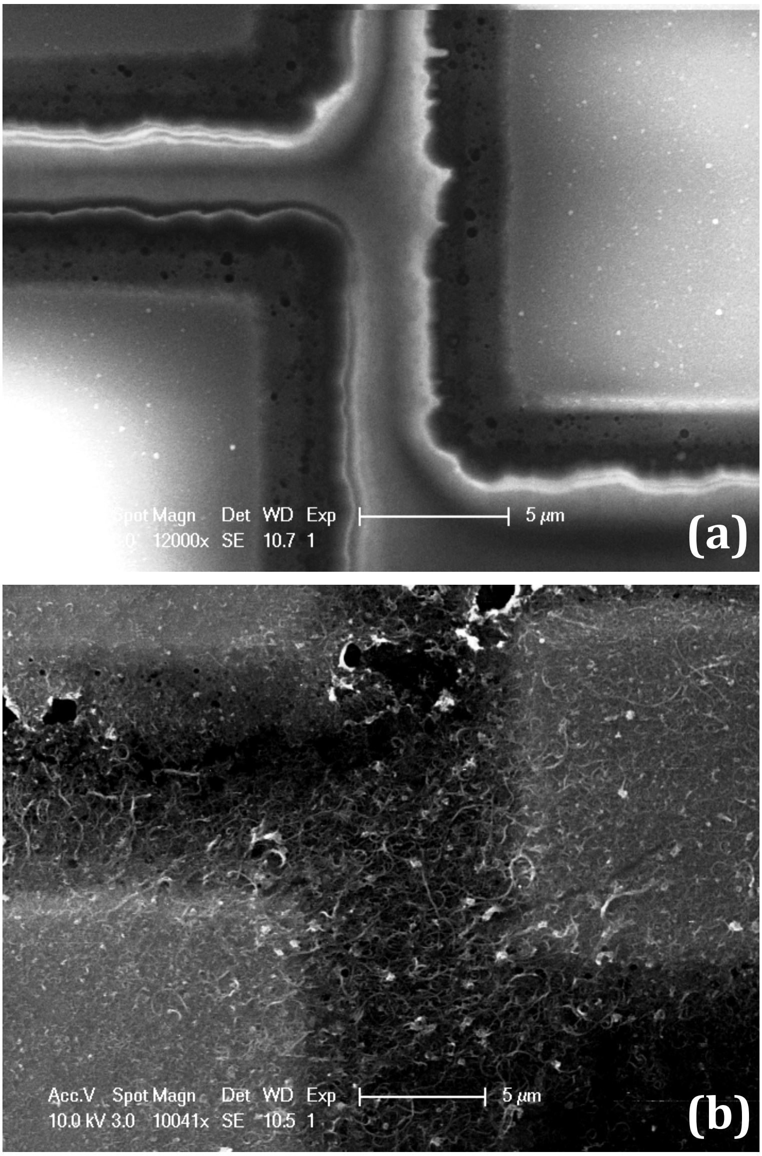

FIGURE 3.6
(a) Before DEP process, (b) after DEP process lasting 4mins

The pressure testing setup is illustrated in figure 3.7. In order to obtain the resistance change in the "cavity design" sensor, a predetermined weight is placed on top of the sensor's diaphragm. This weight induces stress on the diaphragm and causes the CNTs to deform [9]. This pressure test is also run on a commercial sensor as a control device.
The weight used in the experiment is 20g. The resistance reading is recorded before and after the weight is placed on the sensor. This procedure is repeated ten times.

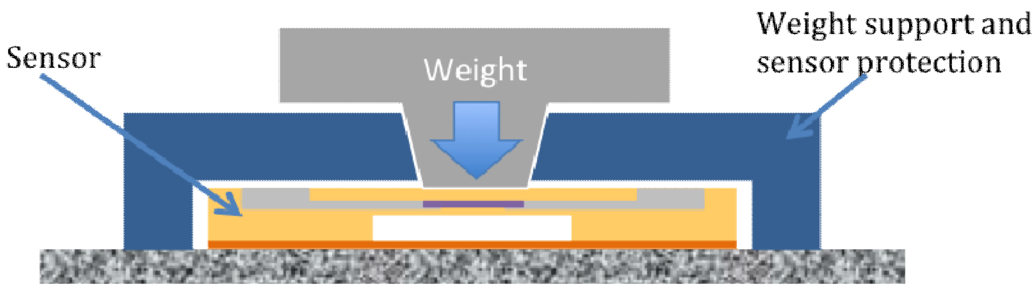

FIGURE 3.7
Pressure testing setup for “cavity design”

The same procedure was carried out on the commercial pressure sensor as well. The results of this pressure testing are presented in figure 3.8.

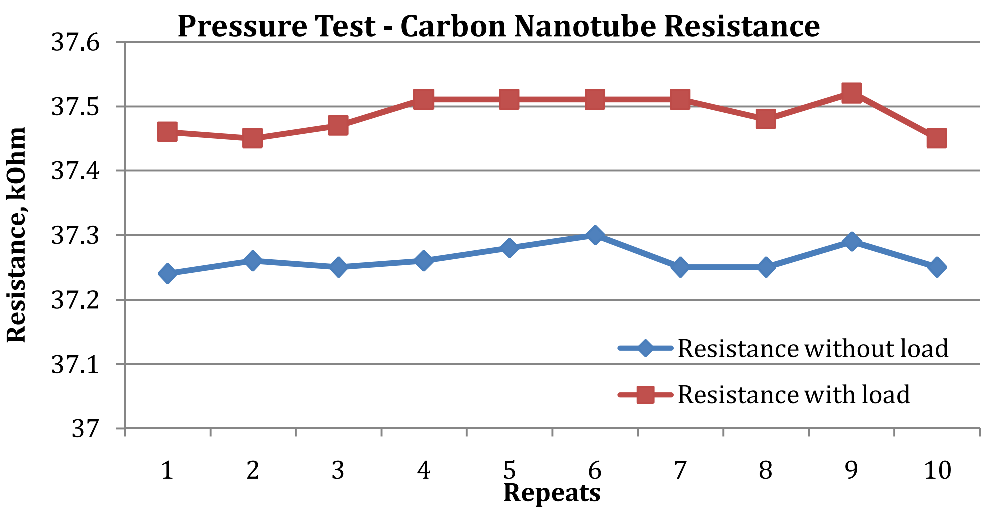

FIGURE 3.8
Pressure test results, CNTs resistance value

On average, the CNTs’ resistance value of 37.26kΩ is recorded without the 20g of pressure load. With load, the average resistance value of 37.49kΩ is measured.

Figure 3.9 illustrates the bending test experiment setup for the sensor. The prototype sensor is partially placed on a firm platform as shown. The CNTs’ resistance value is recorded. A weight of 20g is then placed on the overhang of the sensor. This weight induces strain on the sensor and causes the structure to bend and cause the CNTs to deform. The resistance value is recorded again with this weight on. The experiment is repeated ten times to obtain the average resistance value of the CNTs with and without the load.

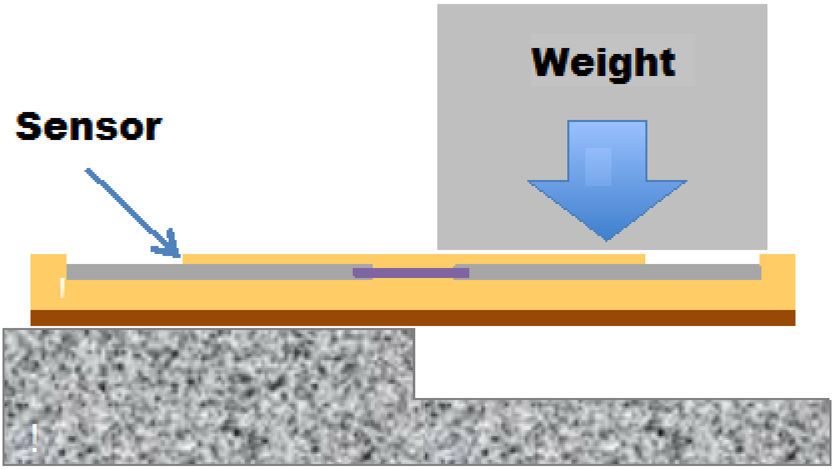

FIGURE 3.9
Bending test experiment setup schematic

On average, without the 20g load, the CNTs resistance is 37.85kΩ, with the weight load, the resistance value increased to 38.10kΩ. Furthermore, the tests showed no hysteresis or memory effects from repeated strain, thus indicating once more the resilience and elasticity of nanotubes in continual usage.

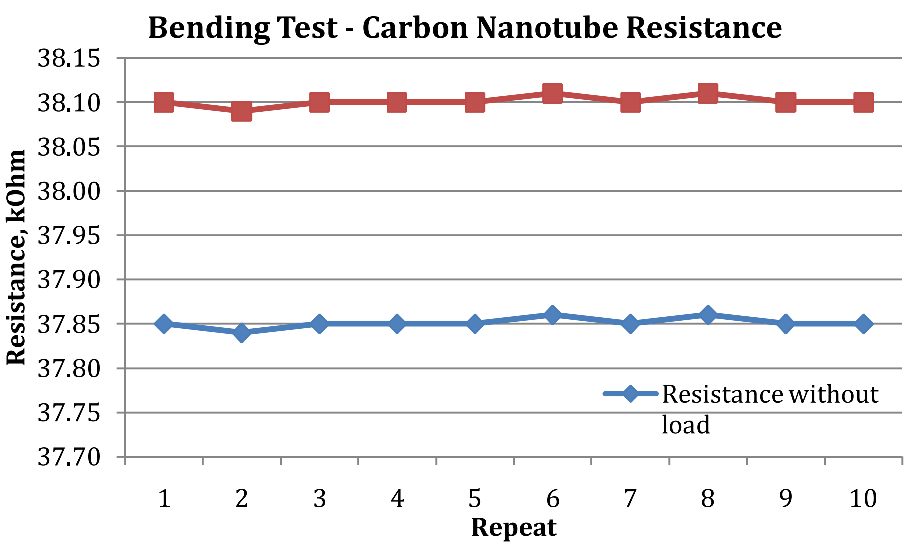

FIGURE 3.10
Bending test results, CNTs' resistance value with and without load

The experimental results confirm the behavior expected from any conductive element of length L in the direction of current flow, Young modulus E, and piezoresistive constant p_L, which emulates a nanotube. The relative change in electrical resistance $\Delta R/R$ is given by:

$$\frac{\Delta R}{R} = Ep_L \frac{\Delta L}{L}$$

Conclusions

The knowledge and understanding gained from our previously published works as well as the experimental work carried out in this research have put us today in the cusp of developing a novel flexible micro sensor based on polymer and carbon nanotubes. The DEP process used to manipulate the CNTs between the microelectrodes has proven successful and has yielded useful results in pressure/strain measurements.

Since the conclusion of this pioneering work, other researchers have utilised nanotubes in the realisation of pressure devices. Wang [10] has used the principle of nanotube sensitivity to pressure to design and realise a prototype of fringe-electrodes element. The fringe-electrodes-element encompasses the pressure sensitive part, the connection part, and the interface part. Different from the traditional sandwich element, the pressure sensitive part of the fringe-electrodes-element has no electrodes, contributing to increasing the flexibility of the element.

Chauhan et al. [11] reported on the growth of multiwall carbon nanotubes (CNTs) at the centre of a bow tie micro-cavity and demonstrated the change in resistance of these CNTs under gas pressure loading ($\Delta R/R \cong 16\%/\text{atm}$). By adapting the Euler-Bernoulli theory of beams to CNTs that bridge opposite walls of the cavity, they were able to fit the piezoresistance curves and extract the Young's modulus, the piezoresistive constant, and the nanotube radius, for a range of CNT growth conditions. By detecting pressures as low as 0.1 atm, they have demonstrated a membrane-less technology capable of sensing pressures with micron scale resolution.

Future works will have to investigate drift with temperature and packaging [12], and carry out reliability tests to further establish commercial viability as a robust sensing system for precision measurements.

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