Nanomanufacturing Methods for the Reduction of Noise in Carbon Nanotube-Based Piezoresistive Sensor Systems

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Carbon nanotube (CNT)-based piezoresistive strain sensors have the potential to outperform traditional silicon-based piezoresistors in MEMS devices due to their high strain sensitivity. However, the resolution of CNT-based piezoresistive sensors is currently limited by excessive 1/f or flicker noise. In this paper, we will demonstrate several nanomanufacturing methods that can be used to decrease noise in the CNT-based sensor system without reducing the sensor's strain sensitivity. First, the CNTs were placed in a parallel resistor network to increase the total number of charge carriers in the sensor system. By carefully selecting the types of CNTs used in the sensor system and by correctly designing the system, it is possible to reduce the noise in the sensor system without reducing sensitivity. The CNTs were also coated with aluminum oxide to help protect the CNTs from environmental effects. Finally, the CNTs were annealed to improve contact resistance and to remove adsorbates from the CNT sidewall. The optimal annealing conditions were determined using a design-of-experiments (DOE). Overall, using these noise mitigation techniques it is possible to reduce the total noise in the sensor system by almost 3 orders of magnitude and increase the dynamic range of the sensors by 48 dB.
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Keywords: carbon nanotube, MEMS, piezoresistor, flicker noise

1 Introduction

Currently, many biological, materials science, and nanomanufacturing applications could benefit from multi-axis micro- and nanoscale sensors with fine displacement resolution (nanometers) and/or force resolution (piconewtons) [1,2]. Unfortunately, because of the size, sensitivity and fabrication limitations of traditional MEMS sensing techniques such sensing systems do not yet exist. CNT-based piezoresistive transducers offer the potential to overcome these limitations due to their high strain sensitivity and inherent nanoscale size. However, CNT-based piezoresistive sensors tend to have large amounts of noise due to their interactions with the substrate, the metal electrodes, and the environment. In this paper, we will present several techniques that can be used to improve the CNT-electrode contact interface, isolate the CNT-based piezoresistive sensor from the outside environment and reduce the total noise in the piezoresistive sensor system. Using these techniques, it is possible to reduce the environmental sensitivity and increase the resolution of CNT-based piezoresistive sensor systems.

2 Sources of Performance Limitations

2.1 Flicker Noise. Carbon nanotube-based piezoresistive strain sensors have the potential to outperform many traditional MEMS force and displacement sensors due to their high strain sensitivity [3]. However, CNT-based sensors typically suffer from large amounts of flicker noise which limits the resolution of these sensors [4,5]. Flicker noise is believed to be caused by the capture and release of charge carriers in localized trap states in the CNT [6]. The flicker noise in a CNT is given by Eq. (1), where α is the Hooge constant, V_s is the source voltage, f is the frequency, and N is the number of charge carriers in the resistor.

$$\sigma_{1/f} = \sqrt{\frac{\alpha V_S^2}{N} \ln\left(\frac{f_{\max}}{f_{\min}}\right)} \tag{1}$$

Overall, the Hooge constant for CNT-based sensors has been measured to be similar to other semiconducting materials such as polysilicon [7]. However, as sensor systems are scaled down to the nanoscale, the number of charge carriers is reduced and flicker noise increases. Therefore, due to the small number of charge carriers in the CNT-based devices, the noise in these devices tends to be dominated by flicker noise.

In general, the amount of noise in the CNT sensor scales with the resistance of the sensor [4]. However, the total amount of noise in the in the CNT-based sensor system is highly dependent on a number of sources related to the design of the sensor system and the conditions under which the sensor is manufactured and tested. For example, the amount of flicker noise in the sensor is higher when the sensor is exposed to the environment compared to when it is tested in a vacuum [8]. This indicates that molecules that are absorbed onto the surface of the CNT can act as extra scattering sites and significantly increase the noise in the sensor [9]. Similarly, the gate voltage used during testing can have a significant effect on the sensor noise by altering the number of charge carriers in device [10,11]. Even the surface on which the CNTs are deposited can have a significant effect on the noise in the sensor since the motion of charged defects in the dielectric surface or at the dielectric/CNT interface can result in increased noise in the CNT [12]. For example, it has been shown that CNTs on a SiO_2 substrate have 10–20 times greater flicker noise than suspended CNTs [13,14].

In addition, the type of CNTs in the sensor can have a significant effect on the sensor noise. For example, metallic CNTs have

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been shown to have up to 2 orders of magnitude less flicker noise than semiconducting CNTs [15]. Also, if the CNTs are longer than the ballistic conduction length, the length of the CNT can also affect the amount of noise in the sensor [10,11,16]. Finally, the temperature at which the CNT-based sensors are tested can have a significant effect on the flicker noise. For example, samples tested at room temperature can have up to 3 orders of magnitude greater noise power than samples tested at 8 K [17]. It is thought that this decrease in noise power can be attributed to reduced noise in the contact between the electrode and the CNT [18].

In general, sensors based on CNT network films tend to have higher 1/f noise than single CNTs, due to the noise from tube–tube junctions [19]. Therefore, the percolation process is the primary physical mechanism influencing the noise level in a CNT film and percolation theory can be used to describe the noise in these films [20]. This means that the noise in CNT films is sensitive to the disorder of the film and the quality of the film [21,22]. For example, it has been shown that the signal-to-noise ratio of CNT network films can be increased by up to two orders of magnitude by improving the alignment of the CNTs within the film [23]. In addition, it has been shown that flicker noise in CNT films scales inversely with the device size for a given resistance [24].

In order to reduce the amount of flicker noise in CNT-based sensors and transistors, several noise mitigation techniques have been investigated. One of the major sources of flicker noise in CNT sensors is the adsorption and desorption of gas molecules on the surface of the CNT [25]. This adsorption and desorption of gas molecules changes the number of mobile carriers in the carbon nanotube, which leads to a change in electron mobility, resulting in increased flicker noise. This source of flicker noise can be reduced in CNT-based sensors and transistors by encapsulating the CNTs in a ceramic coating in order to protect the CNTs from the outside environment. For example, it has been shown that encapsulating CNTs in 100 nm of atomic-layer-deposited aluminum oxide can increase device stability and decrease noise by almost an order of magnitude [26]. Similar results have been demonstrated for top-gated transistors where the ceramic encapsulation layer is used as the gate dielectric and passivation layer [27,28].

In addition to adding an encapsulation layer, annealing has also been used to reduce noise in the sensors. Annealing improves both the intertube coupling and the CNT-electrode coupling. These improvements can lead to an order of magnitude decrease in flicker noise in CNT-based sensors [29,30]. Similarly, increasing the number of CNTs in the sensor has been shown to reduce the noise power, since the noise power scales with the number of carriers, which is proportional to the number of CNTs in the sensor [31]. Finally, the amount of flicker noise in a CNT-based sensor is also a function of the gate bias voltage [32]. As the gate bias is increased, the flicker noise tends to decrease, since more charge carriers are injected into the device. However, the gauge factor of the CNT-based piezoresistor decreases as the gate bias voltage increases due to the decrease in the piezoresistive effect. Therefore, the signal-to-noise ratio of such sensors tends to be maximized when the device is in the off-state [33].

2.2 Contact Resistance. The contact resistance of the carbon nanotubes to the electrodes can have a large effect on the resolution of the sensor system. This is because the contact resistance tends to remain approximately constant as the CNT is strained. Therefore, a high contact resistance can significantly reduce the sensitivity of the sensor system. Contact between the electrodes and the CNTs can either be Schottky or ohmic, depending on the contact metal and the processing conditions [34]. For example, thermal annealing can be used to create ohmic contacts and to decrease the contact resistance between the CNT and the electrode by up to 3 orders of magnitude [35]. In general, ohmic contact is preferred to Schottky contact in CNT-based piezoresistive strain sensors because the current in ohmic contacts is linear with voltage and ohmic contacts tend to have lower contact resistances due to the lack of a Schottky barrier. The Schottky barrier is a potential barrier between the CNT and the electrode, which prevents free flow of electrons between the CNT and the electrode due to potential energy differences between them. The height of the Schottky barrier is set by the difference between the work functions of the CNT and the electrode as well as the diameter of the CNT [36].

Overall, the contact resistance is a function of the difference in work functions between the metal electrodes and the CNT as well as the wettability of the electrode [37]. The quantum coupling between the CNT and the metal electrode depends on the contact area, CNT diameter, CNT chirality, and whether the CNT is side or end contacted [38,39]. In addition, coupling between the d-orbitals in the metal and the π-orbital in the CNT plays an important role in determining both the cohesive and the electronic interactions at the contacts [40]. Therefore, metals with good quantum coupling to CNTs, good wettability, and work functions similar to CNTs such as titanium tend to produce the best contacts.

3 Experimental Setup and Noise Measurement

A precision bridge circuit was set up in order to measure the noise in the CNT-based piezoresistors. The bridge circuit consists of (1) a precision voltage reference, (2) a Wheatstone bridge, and (3) an instrumentation amplifier. The voltage reference in the bridge circuit is used to convert the “noisy” supply voltage from the power supply into a low noise input reference for the Wheatstone bridge. The Wheatstone bridge is set up in a quarter bridge configuration with the CNT-based piezoresistor (R_1 in Fig. 1(*a*)) acting as the active element and three metal film resistors ($R2$ through $R4$) used to complete the bridge. The instrumentation amplifier is used to scale the output signal of the Wheatstone bridge by a gain, G, so that the signal can be read accurately by an

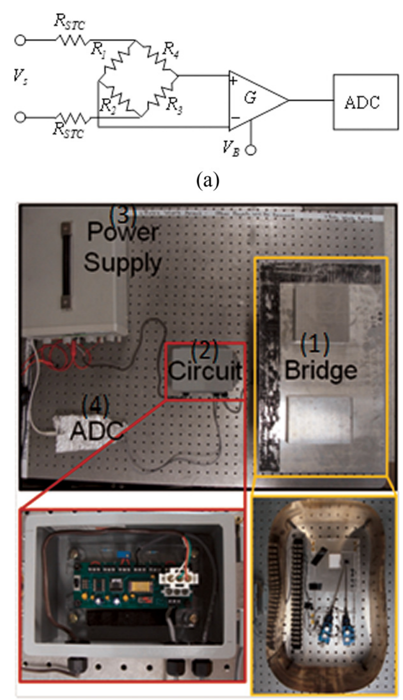

Fig. 1 (*a*) Schematic of experimental setup where V_s is the voltage source, V_b is the voltage bias and R_{stc} are the span temperature compensation resistors. (*b*) Experimental setup consisting of (1) a Wheatstone bridge, (2) a precision bridge circuit with a precision voltage reference and instrumentation amplifier, (3) a dc power supply, and (4) an analog-to-digital converter [41,42].

analog-to-digital converter (ADC). Both the electronics and the Wheatstone bridge are placed in metal boxes in order to shield them from electromagnetic interference, as shown in Fig. 1. Overall, this bridge circuit design results in less than 2 μV of noise in the electronics. This is much lower than the noise in the CNT-based piezoresistors. Therefore, it is possible to get an accurate measurement of the noise in the CNT-based piezoresistive sensor using this experimental setup.

A MEMS test structure was designed and microfabricated in order measure the noise and gauge factor of the CNT-based piezoresistors (Fig. 2). The test structure consists of a fixed–fixed flexure beam and electrodes connected to the base of the flexure. The outer four sets of electrodes are connected to polysilicon piezoresistors while the inner two electrodes are left empty so that CNTs may be connected across them. These central electrodes are spaced 1 μm apart. This architecture enables independent measurement of strain with both the polysilicon and CNT-based piezoresistors. The center of the flexure has a locating hole where small, known weights may be placed, thereby loading the structure and straining the CNTs. A detailed description of the device structure and the fabrication process is given in Ref. [43].

SWCNTs are deposited onto the test flexures via dielectrophoresis. The SWCNTs are grown using thermal chemical vapor deposition and are purified to over 99% pure SWCNTs using ultracentrifugation. In the typical deposition process used for these tests, a droplet of a 3 μg/ml solution of SWCNTs in deionized water was placed on the middle electrodes of the test structure and a 5 MHz, 5 V peak-to-peak AC voltage was used to direct the deposition of the SWCNTs [43]. After 5 min, the test structure was rinsed with DI water and dried.

4 Methods to Reduce Contact Resistance

4.1 Effect of Electrode Material. The selection of the electrode material can have a significant impact on both the magnitude and the type of contact resistance. For example, electrode materials with low work functions can sometimes produce Schottky-type contact resistances. This type of contact resistance is undesirable because it is nonlinear and can reduce the sensitivity of the sensor system. In addition, oxidation of the electrode material can increase the contact resistance and result in Schottky-type contact barriers.

For this study, aluminum, titanium, and platinum contacts were tested. Both the titanium and platinum electrodes generally resulted in low resistance, ohmic contacts. However, the aluminum contacts sometimes resulted in Schottky-type resistors. This is because aluminum has a low work function and oxidizes easily. In order to overcome these limitations, the aluminum contacts were typically coated in platinum and annealed to produce low resistance ohmic contacts.

4.2 Platinum Coating. The platinum coating of the aluminum electrodes was performed using the gas injection system in the focused ion beam (FIB). In this process, the platinum organometallic gas is injected into the FIB vacuum chamber where it is chemisorbed onto the surface of the device. When the desired deposition area is scanned with the ion beam, the precursor gas is decomposed into volatile and nonvolatile components. The nonvolatile component of the gas, such as the platinum, remains on the surface while the volatile reactants are released back into the vacuum chamber. Through this process it is possible to coat both the CNTs and the aluminum electrode with platinum. This helps to improve the contact resistance by increasing the work function of the electrode, increasing the contact area between the CNT and the electrode, and by preventing oxidation of the contact area.

4.3 Annealing. After the platinum deposition, the aluminum contacts with the CNTs attached to them are typically annealed in order to reduce the contact resistance and improve the linearity of the contact. Annealing can be used to remove surface contaminants such as water molecules that can adhere to the electrode surface. In addition, annealing can create a composite structure between the electrode and the CNT which can significantly decrease the contact resistance and create an ohmic contact between the electrode and the CNT. A typical current–voltage curve for a CNT with the aluminum electrode after platinum deposition and annealing is shown in Fig. 3. From this curve, it is

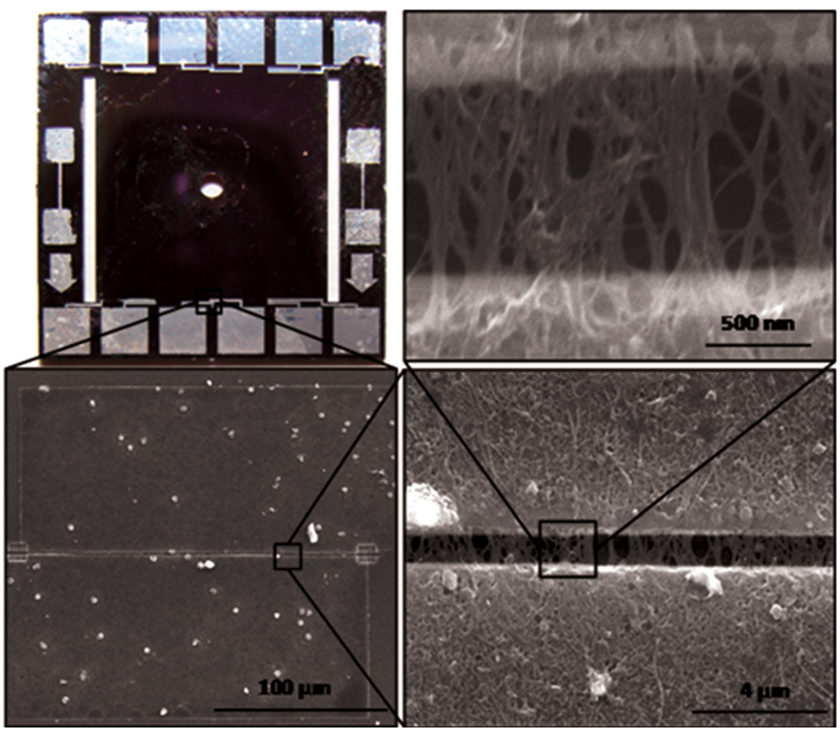

Fig. 2 Test Structure with CNTs connected between the two central electrodes. Reprinted from Ref. [43] with permission from the American Physical Society.

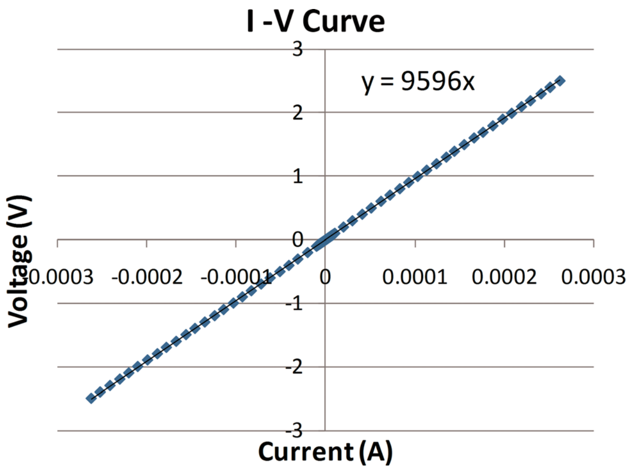

Fig. 3 ***I-V* curve for CNT-based sensor showing ohmic contact**

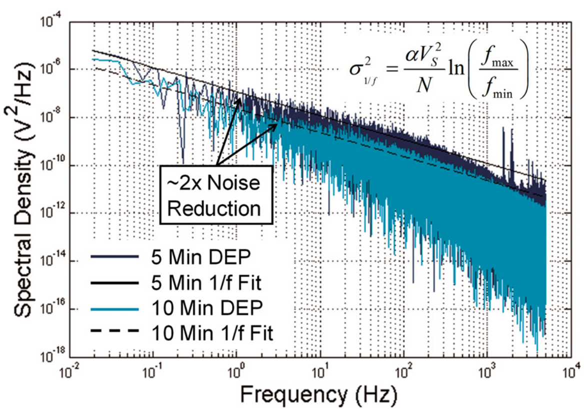

Fig. 4 Power spectral densities of CNT-based piezoresistive sensors with dielectrophoresis deposition times of 5 and 10 min

clear that good ohmic contact has been achieved between the CNT and the aluminum electrode due to the linearity of the curve.

5 Noise Mitigation Techniques and Results

5.1 Increasing Number of CNTs. Several different design and fabrication methods were investigated in order to determine their effects on flicker noise in the CNT-based piezoresistive sensor. First, the effect of the number of CNTs in the sensor system was investigated. In order to increase the number of CNTs in the sensor, the deposition time in the dielectrophoresis process was increased from 5 min to 10 min. This should approximately double the number of CNTs in the sensor. As can be seen in Fig. 4, this change in the manufacturing process resulted in a two-fold decrease in the flicker noise in the sensor. This result is consistent with Eq. (1), where the noise power is inversely proportional to the number of charge carriers. Increasing the number of CNTs in the sensor system should increase the number of charge carriers in the sensor since the number of CNTs is directly related number of carriers. Therefore, doubling the number of CNTs in the sensor should reduce the power spectral density by a factor of two and result in lower flicker noise.

5.2 Coating CNTs. In addition to increasing the number of CNTs in the sensor system, several other manufacturing methods were investigated to reduce the flicker noise in the CNT-based piezoresistive sensor system. First, electron beam evaporation was used to coat the devices in 500 nm of aluminum oxide in order to isolate the CNTs from the outside environment. This aluminum oxide coating helps to prevent molecules in the air from absorbing onto the surface of the CNT where they can become additional scattering sources and increase the noise in the CNT-based sensor. A schematic of fabrication steps used to reduce noise in the CNT-based piezoresistive sensors after the deposition of the carbon nanotubes by dielectrophoresis is given in Fig. 5.

After the CNTs are coated in aluminum oxide, the samples are annealed at 500 °C for 30 min. This annealing process helps both to reduce the contact resistance between the CNTs and aluminum electrodes and to remove some of the adsorbates from the surface of the CNTs. Overall, the aluminum oxide coating and annealing steps reduce the flicker noise in the sensor by about 1 order-of-magnitude. The effect of coating the CNTs in aluminum oxide and annealing the devices on the noise in the sensor is shown in Fig. 6.

5.3 Annealing CNTs. In order to determine the optimal annealing conditions for the CNT-based piezoresistive sensors, a simple DOE was set up to test the effects of annealing time and temperature on the noise in the sensor. A full factorial DOE was used with four temperature levels between 475 °C and 550 °C and time intervals of 30 min and 1 h. Initially, as the time and temperature were increased the flicker noise in the sensors decreased, as seen in Table 1. This decrease in noise is likely due to improved CNT-electrode contacts and removal of adsorbed molecules. However, at high temperatures and long annealing times the flicker noise started to increase due to the degradation of the CNTs in the sensors. Overall, the optimal annealing conditions for the CNT-based piezoresistor in this test were determined to be an annealing temperature of 525 °C and an annealing time of 30 min. Using these processing conditions it is possible to reduce the flicker noise in the sensors by a factor of 229 over as deposited samples.

By using the optimal annealing conditions it is possible to start to hit the Johnson noise limit for these sensors, as shown in Fig. 7. The crossover frequency between the measured flicker noise and the Johnson noise limit predicted from theory is approximately 7 Hz. This indicates that for measurements that take less that 1/10th of a second, flicker noise is negligible and the Johnson noise becomes the dominant noise source. However, for longer experiments, flicker noise is still the dominant noise source.

5.4 Overall Results. Overall, by coating the CNT-based piezoresistors in a layer of aluminum oxide and annealing them it is

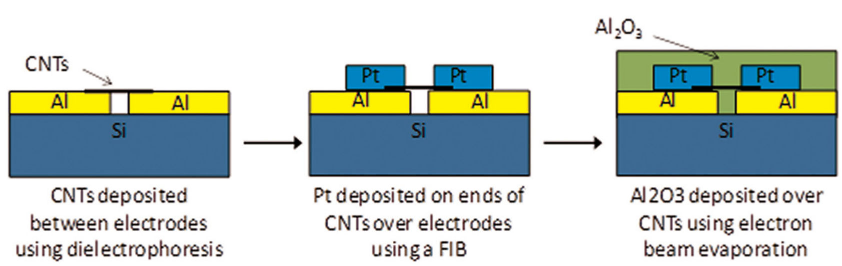

Fig. 5 Schematic of noise reduction fabrication process

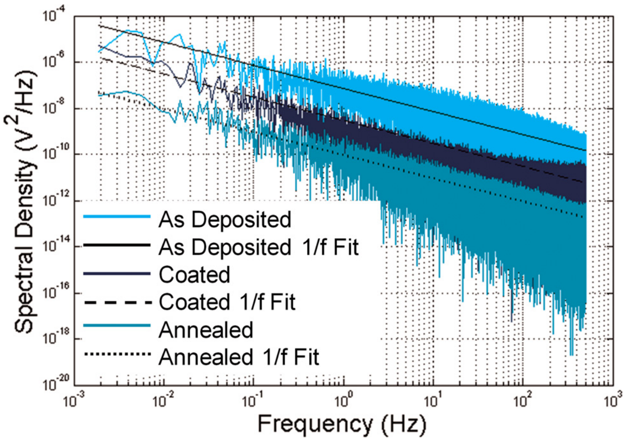

Fig. 6 Power spectral densities of as deposited, Al_2O_3 and annealed CNT-based piezoresistive sensors

Table 1 Measured noise as a function of annealing time and temperature

Time \ Temp	475 °C	500 °C	525 °C	550 °C
30 min	370 μV	190 μV	4.37 μV	49.7 μV
60 min	150 μV	34.3 μV	44.9 μV	99.5 μV

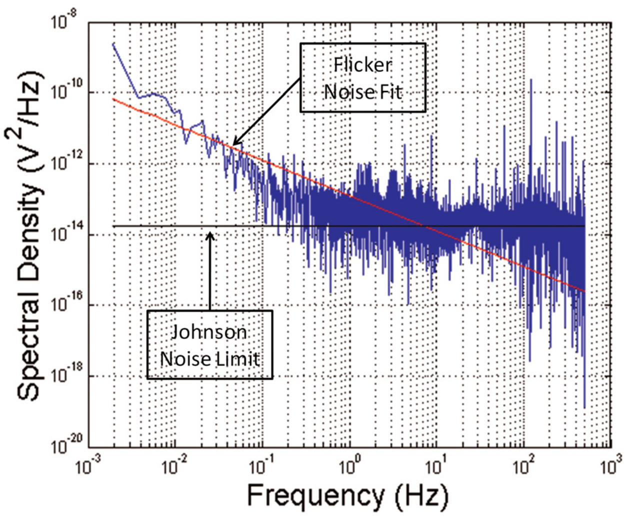

Fig. 7 Power spectral densities of CNR-based sensor annealed at 525 °C

Table 2 Measured noise and dynamic range for CNT-based sensors produced under various processing conditions

	Total noise (μV)	α/N	Dynamic range (dB)
As deposited	1000	8×10^{-8}	50
Coated	200	3×10^{-10}	64
Annealed	4	2×10^{-13}	98

possible to reduce the total noise in the sensors by almost 3 orders of magnitude, as shown in Table 2. This reduction in noise also results in a significant improvement in the dynamic range of the CNT-based piezoresistive sensors. For example, coating the CNTs in aluminum oxide increased the dynamic range by 14 dB, while annealing the samples increased the dynamic range by another 34 dB. Overall, this demonstrates that through the use of proper design and manufacturing procedures, the resolution of CNT-based piezoresistive sensors can be significantly improved.

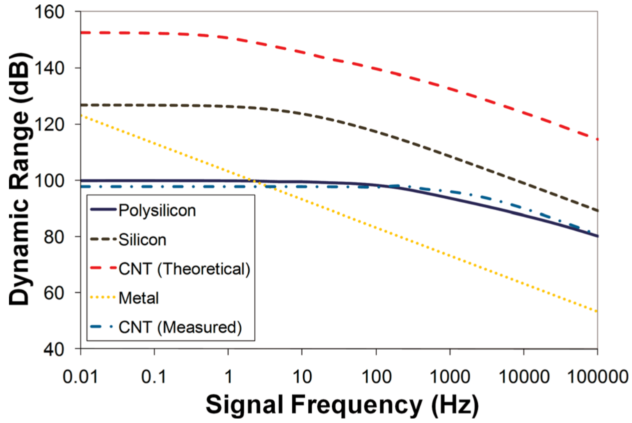

Fig. 8 Performance of various piezoresistive materials on microscale flexure beams

6 Comparison to Conventional MEMS Piezoresistors

Overall, through the use of theory and experimentation this we have been able to increase the performance of CNT based piezoresistive sensors by more than 3 orders of magnitude over previous results [3]. These improvements make CNT-based piezoresistive sensors very competitive with more conventional MEMS piezoresistors such as metal, silicon and polysilicon, as shown in Fig. 8. However, more work still needs to be done on the sorting of CNTs in order to reach the theoretical maximum performance characteristics of CNT-based piezoresistive sensors.

As can be seen in Fig. 8, for flexures on the scale of tens of microns, current CNT-based piezoresistors outperform metal gauges for signal frequencies greater than 10 Hz. In addition, CNT-based piezoresistors perform about as well as optimized designs for polysilicon piezoresistors. Silicon-based piezoresistors would outperform CNTs in a single axis device but are not capable of high performance on multi-axis devices due to the crystallographic orientation-dependent gauge factor of single crystal silicon. For smaller devices, the performance of CNT-based piezoresistors will continue to improve in comparison with silicon and polysilicon since the noise in the CNT-based sensors scales with the number of CNTs in the sensor while the noise in the silicon-based sensors scales with the sensor volume. On the other hand, for larger devices polysilicon and silicon-based piezoresistors are probably the best material selections. For slow measurements, metal gauges become a feasible option. Based on this analysis, CNT-based piezoresistors are currently the best material for submicron sensor systems. In addition, they have the potential to significantly increase in performance as chirality sorting technology improves [43].

7 Conclusions About Best Practices for CNT-Based Piezoresistors

7.1 Materials Selection. In general, in order to ensure low contact resistance and ohmic contact between the CNTs and the electrodes, electrode materials should be selected that have high work functions and that do not oxidize. However, these considerations must be weighed against fabrication considerations. For example, gold and palladium both have high work functions and do not oxidize easily. However, these materials tend to delaminate from the device structure and can contaminate microfabrication equipment. Alternatively, aluminum is easy to pattern and etch and does not create contamination issues. However, aluminum suffers from a low work function and it rapidly forms a surface oxide. Therefore, in this work aluminum was selected as the electrodes material but platinum was added at the electrode-CNT interface in order to ensure good ohmic contact.

7.2 Design and Manufacturing. This study found that in order to minimize the amount of noise in the CNT-based piezoresistive sensors, five basic guidelines should be followed:

(1) Always maximize the size of the CNT-based piezoresistor.
(2) Maximize the number of CNTs in the piezoresistive sensor.
(3) Coat the sample CNT-electrode interface in platinum to minimize contact resistance.
(4) Use ceramic coating to protect the CNT-based piezoresistor.
(5) Anneal the CNT-based piezoresistive sensor at 525 °C for 30 min to reduce noise and improve contact resistance.

Through the use of these design and manufacturing guidelines it is possible to reduce the noise in the CNT-based piezoresistive sensors by almost 3 orders of magnitude. This makes CNT-based piezoresistive sensor competitive with other types of piezoresistive sensors at the microscale for a large range of frequencies. In addition, as sensors are scaled down to the nanoscale, these noise mitigation techniques will help to make CNT-based piezoresistive sensors the dominant sensor system for measuring nanoscale forces and displacements.

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