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Printable low-cost and flexible carbon nanotube buckypaper motion sensors

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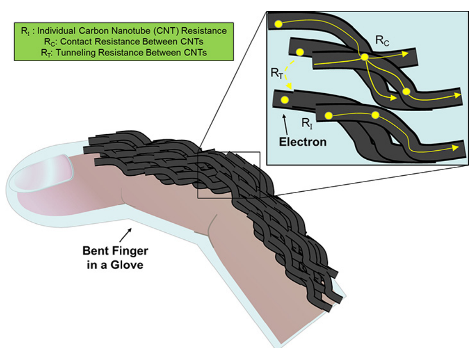
HIGHLIGHTS

- Low-profile tactile sensors can be manufactured using low-cost printing technology and ultra-thin carbon nanotube buckypaper.
- The printed buckypaper sensor can sense tensile and compressive strains as low as 0.005% and as high as 1%.
- The printed buckypaper sensors are flexible and can be designed to conform to surfaces on the body.
- Finger movements were easily detected using a glove that was integrated with sensors.

GRAPHICAL ABSTRACT



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ABSTRACT

Wearable technology, which features affordable and flexible sensors integrated into fabrics and garments to detect both deliberate and subtle body movements, will reshape the way we approach self-rehabilitation, physical training, and many high-dexterity tasks by harvesting data about the wearer's activity. Metallic and semi-conductor sensors are currently the most commercially viable sensors. Metallic sensors designs are low profile and flexible; however, they are limited by low sensitivity and complex manufacturing. Semi-conductor sensor designs are highly sensitive but limited by their rigidity and brittle nature. Wearable sensors that are low profile, flexible, and sensitive to micro-strains are highly desired. We have developed a printable and low profile strain sensor using multi-wall carbon nanotube thin films called buckypaper (MWCNT-BP). Our tests indicate that the buckypaper sensors are 77% more sensitive than similar sensor designs. This paper explains the low-cost printing technology and displays the sensors' performance after integration into a fabric glove.

1. Introduction

Wearable technology will reform the methods of collecting biomechanical data [1–6]. Smart clothing and garments with tactile sensing capabilities could provide concurrent feedback for self-rehabilitation, intelligent prosthetics, and physical training [4–6]. New human-machine interfaces have the potential to significantly improve the quality of life. Tactile sensors must be affordable, low profile and flexible enough to conform to the arbitrary curves and crevices of the human body. They must also provide quick and stable responses to micro-strains with high sensitivity and low power requirements. Given these design specifications, micro-strain (<100 μm/m) has remained a

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challenge to the scientific community. Tactile sensors that are based on piezoresistivity mechanisms, in which electrical resistance changes under applied strain, are the most common due to their simplicity and affordability. Metal-foil sensors have been a standard due to their flexibility and low profile; however, they are limited by low sensitivity with a gauge factor (GF) $\approx$ 2. The resistance variation derives from changes in the effective length of the sensing element. As a result, they behave as fixed directional sensors that are limited to sensing strain in a specific direction. Alternatively, highly sensitive (GF $\approx$ 200) semi-conducting sensors made of crystalline silicone (Si) are limited by their rigidity and a restricted working range. Achieving wearable tactile sensing requires novel approaches in design and structural engineering.

Recent research has focused on manufacturing carbon nanotube (CNT)-based sensors. CNTs demonstrate high tensile strength (~100 GPa), Young's modulus (~1 TPa), and electrical conductivity (10^6–10^7 S/m), and feature a large aspect ratio (>10^6). The unique combination of high elastic modulus and impressive electrical properties promotes excellent sensor characteristics compared to traditional sensors [1–3,5,7–16]. The structure of carbon nanotubes has a significant influence on their electrical properties. Thus, mechanical deformation alters the structure, which then alters the conductivity of the CNT. This is the essence of the carbon nanotube piezo-resistivity [17].

Researchers have developed CNT-based sensors following a manufacturing process that involves dispersing CNTs into a viable polymer matrix. Hu et al. performed a detailed investigation on various process parameters including different polymers, CNT types, curing processes, mixing processes, and types of additives. Hu discovered that the process complexity affects how well the CNTs form the conductive pathways within the matrix to achieve high sensing performance [18]. CNT/polymer films follow a percolation-like power law, in which a CNT concentration threshold (wt%) is required to begin cultivating the percolation network in the host matrix [19–24]. As concentration surpasses the percolation threshold, electrical conductivity in the matrix significantly increases. However, once the network forms, the improvement slows as CNT concentration increases beyond this threshold value [21,25–27]. Darabi et al. recently showcased the potential of carbon nanotubes by dispersing a small amount of CNTs (<10 wt%) into chewing gum to make flexible tactile sensors that can detect sneezes and breathing patterns [1].

The use of individual carbon nanotubes is showing promise, but the sensor designs are limited to the laboratory scale. In addition, sensitivity is considerably low, and manufacturing methods are difficult to repeat [10]. Carbon nanotube buckypaper (CNT-BP) films have helped eliminate process complexity [13,14,23,28,29]. Paper materials, such as CNT-BP, have recently attracted increased research and commercial interest for next-generation wearable technology due to their lightweight, scalable manufacturing processes, and conformability [30].

Buckypaper is a dense, carbon nanotube network that exhibits improved sensitivity to local distributions of stress compared to individual CNTs. Multi-walled CNT-BP (MWCNT-BP) has a well-distributed, yet random structure that provides several degrees of freedom, which is a key advantage over metallic foil strain gauges [3]. MWCNT-BP film sensors have displayed a greater working range than single-walled carbon nanotube buckypaper [10].

Recently, Suzuki et al. designed highly sensitive (GF > 10) buckypaper strain sensors by spin-coating aligned MWCNT buckypaper films with low-modulus polyurethane (PU) resin and an elasticity-assist layer made of polytetrahydrofuran (PTHF) [14]. The sensors were successfully integrated into fabric gloves to sense a pianist's finger motions. Compatibility with a printing process is highly desirable for next-generation tactile sensors. With the advances of printing technology, printing is simple, cost-efficient, and capable of large-area and high-throughput production. In this report, we present a highly sensitive, low-cost strain sensor that takes advantage of the advances in printed electronics in conjunction with the low-profile (7 μm), lightweight (5 g/m^2) and multi-functionality of MWCNT buckypaper. This type of affordable, flexible strain sensor can be deployed in various applications ranging from tactile sensing in wearable technology to structural health monitoring.

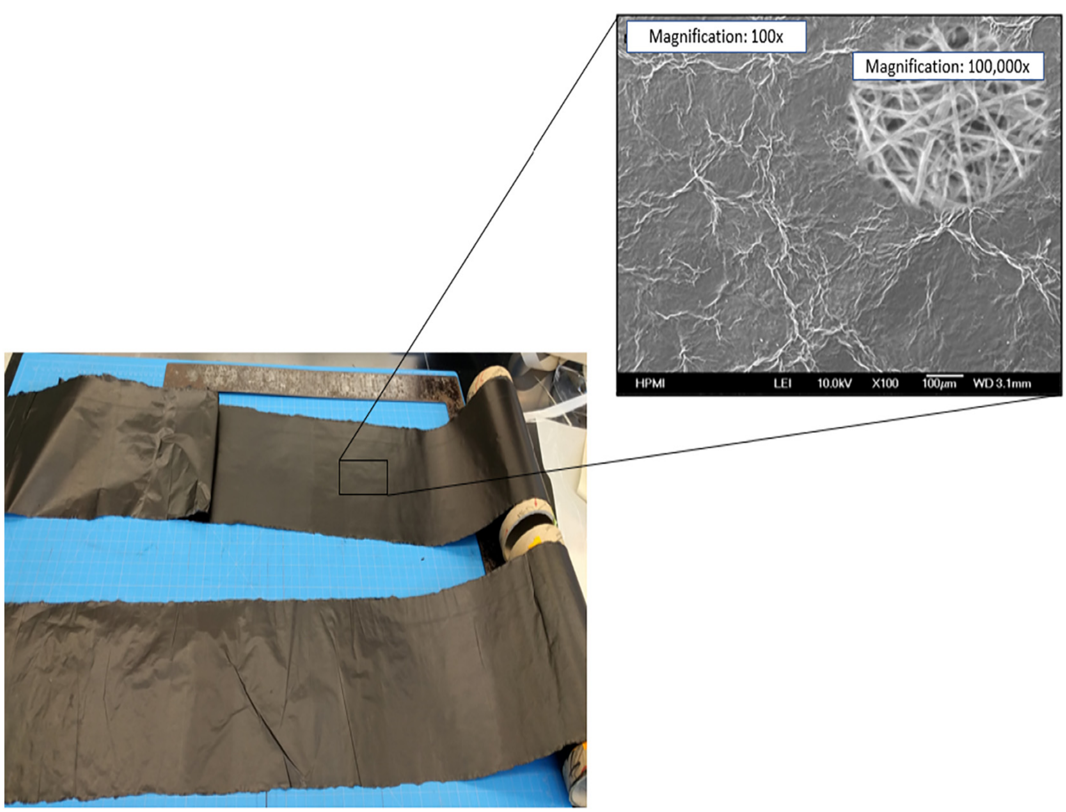

Fig. 1. Multi-wall carbon nanotube buckypaper; a. continuous roll of MWCNT buckypaper, b. scanning electron microscopy images at 100× and 100,000× magnification.

2. Materials and methods

2.1. Multi-walled carbon nanotube buckypaper fabrication

The fabrication process for MWCNT-BP first involves creating suspension of uniformly dispersed CNTs through sonication. The fabrication procedure has been documented in detail by Smalley [31] and Wang [32]; however, Triton-X surfactant has been added to the suspension to further improve the CNT dispersions by reducing the surface energies between CNT bundles. Highly dispersed CNTs promote high quality films in terms of both mechanical and electrical properties. After dispersing the nanotubes, the suspension is then filtrated onto a substrate through a mesh filter and dried. The resultant free-standing films are repeatedly washed with distilled water and heated at 850 °C under argon gas for 4 h to remove residual surfactant and impurities. Fig. 1 presents a 2 m-long continuous roll of buckypaper along with scanning electron microscopy (SEM) images. Buckypaper consists of tightly packed CNT networks compared to solvent-cast CNT/polymer sensors. The dense and conductive network of buckypaper provides a more sensitive response to strain changes. The MWCNT-BPs used in this research were 6 μm thick, which exhibited an electrical conductivity of 200 S/cm and a possessed an elastic modulus of almost 3GPa.

2.2. Ink-jet printed sensor design

The sensor structure includes a strip of buckypaper (20 mm × 3 mm × 0.006 mm) and a PET substrate with printed circuitry on its surface. The structure was laminated with a film to protect the sensor components and hold the buckypaper strips in position. The interaction between the sensing element and polymer is critical. Laminating the structure ensures interfacial binding between the materials to facilitate fast responses and cyclic performance with limited hysteresis [6,10,33].

Fig. 2 presents the manufacturing process, which is low-cost and scalable given the simplicity and commercial availability on ink-jet printing technology. Silver ink electrodes were printed on a thin polyethylene terephthalate (PET) substrate using a one-pass printer. Commercially available, water-based silver ink from NovaCentrix was used to print the electrodes. The composition included 25 wt% silver particles ($\bar{d} \approx$ 60–80 nm), 1–15 wt% ethylene glycol, and 60–75 wt% water. The ink was emission-free and dries quickly. Once the silver electrodes were printed, a buckypaper strip was positioned between the substrate's printed contacts and laminated.

The buckypaper is considerably thin and avoids influencing waviness in the final laminated structure. The printed electrodes provide each end of the buckypaper strips with a fixed electrical contact. The closely paired ends of the printed silver electrodes were crimped with male connectors using a Nicomatic CrimpFlex tool for electrical contacts. Three fundamental principles define this manufacturing strategy. The first exploits an observation in basic mechanics. Polymers that are sufficiently thin will also be flexible. Fig. 3 displays the sensor design's low profile and flexibility. The second idea is based on the piezoresistivity response to stress in buckypaper [7,11,19]. A dense network of conductive CNTs promotes a seamless flow of current; however, under tensile stress, the network stretches and becomes less dense. This reduces the number of percolative pathways and ultimately reduces conductivity. Fig. 4 illustrates the reversible changes in the CNT network due to both tensile and compression strains. As the network stretches, the number of conductive paths reduces. This leads to limited tunneling

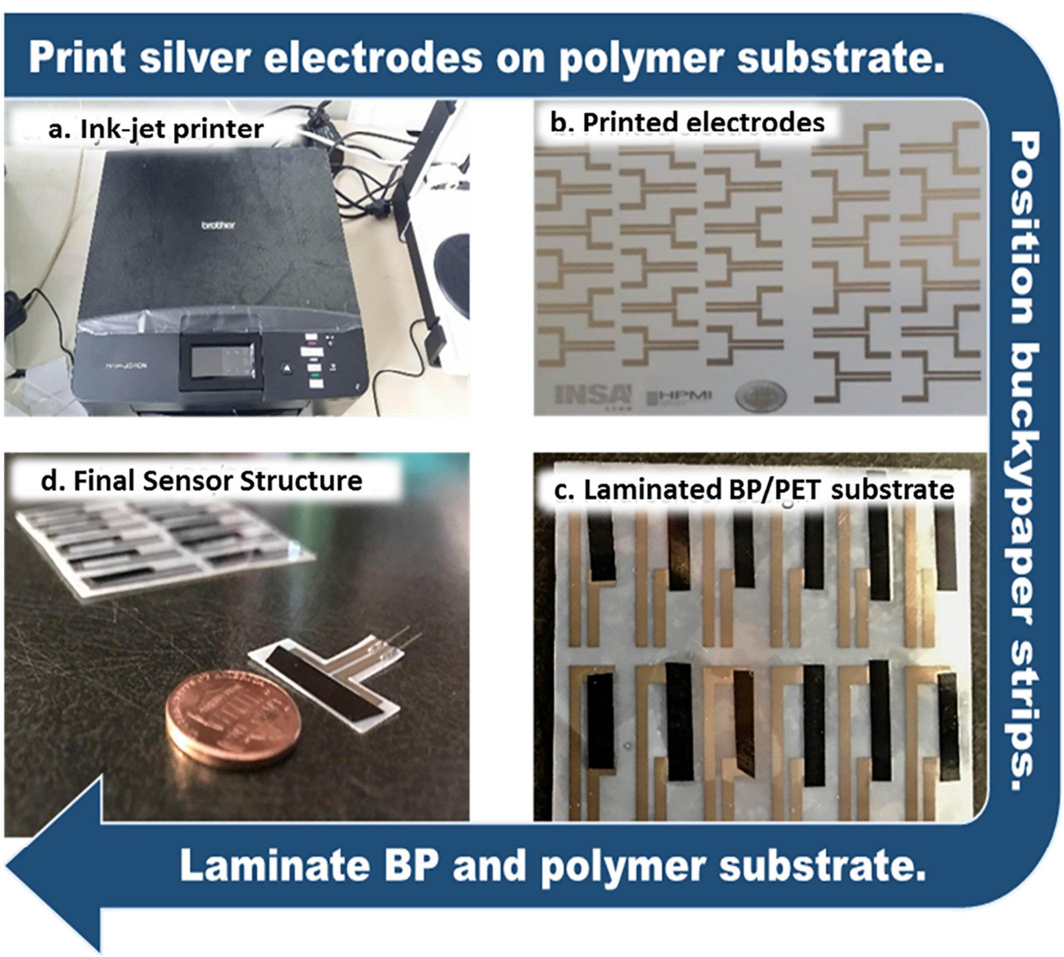

Fig. 2. Manufacturing process flow for buckypaper strain sensors. It includes printing, laminating, and cutting.

Fig. 3. BP strain sensors offer a low-profile and flexible design.

effects and increased resistance [19,23,28]. As the network compresses, the contrary occurs leading to increased conductivity [34]. The third manufacturing principle exploits the recent advances in printed electronics, more specifically inkjet printing (IJP), which has the advantages of short production time, low cost, minimal waste, high spatial resolution, and good reproducibility.

2.3. Strain gauge characterization

Strain gauge measurements were taken using a DEWE-SIRUS-STG-Multi card. The printed buckypaper sensors were fastened to a flexible polyvinyl chloride (PVC) substrate (150 mm × 25 mm × 3 mm) using an epoxy-based adhesive. The PVC substrate was fixed to a linear actuator (NITEK GDI 350ES500S), and the actuator applied periodic (1 Hz) tensile strains of 0.4% with highly accurate positional control. To better access sensor performance, metallic, commercial strain gauges with a gauge factor of 2.5 were fastened to the PVC substrate. The strain gauges were characterized using a Wheatstone bridge configuration, and the measurements were recorded on a DEWESoft data acquisition card.

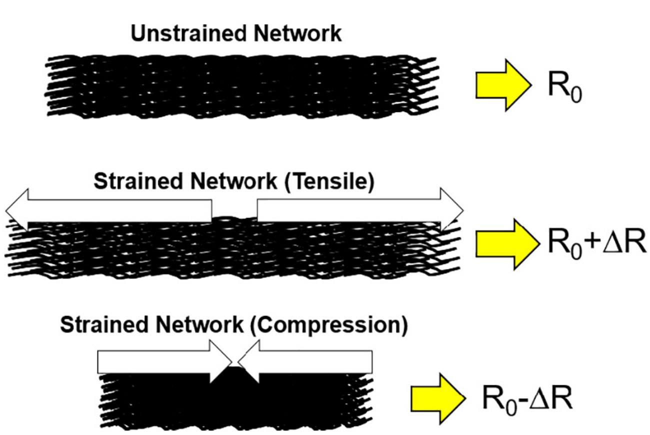

Fig. 4. Schematic of buckypaper before and after being stretched. The induced gaps in the conductive network increases electrical resistance.

3. Results and discussion

Buckypaper has an electrical resistance that encompasses three major components: 1) the intrinsic resistance ($R_{Individual}$) of the individual CNTs, 2) the contact resistance ($R_{Contact}$) between the CNTs, and 3) the tunneling resistance ($R_{Tunneling}$) between the neighboring CNTs [7,10,28]. The increasing number of gaps in the network causes an increase in electrical resistance [7,28]. As percolation pathways are strained and disconnected, less current can flow through the printed sensor altering the conductivity of the structure [7,11,27,28].

$$R_{BP} = R_{Individual} + R_{Contact} + R_{Tunneling} \tag{1}$$

Individual CNTs have an elastic modulus that approaches 1 TPa. This high stiffness indicates that their contributions ($R_{Individual}$) to the global piezoresistivity are negligible in motion sensing. In addition, the intertube resistance is much greater compared to the individual resistance. The weak interactions at the nanotubes' joints influence interfacial sliding and dictate piezoresistivity. As the nanotubes separate from each other, the network becomes less conductive. Therefore, $R_{Contact}$ and $R_{Tunneling}$ dictate global piezoresistivity [28]. To determine the gauge factor, the buckypaper sensors were connected to a Wheatstone Quarter-Bridge circuit, as shown in Fig. 5. A small voltage (V_{in} = 3 V) was applied to the circuit, and the voltage (V_{out}) across the buckypaper sensor was monitored. As presented in Eq. (2), V_{out} can be expressed in terms of the resistors in the circuit (R_x) and V_{in} [35].

$$V_{out} = V_A - V_B = \frac{R_{BP}R_4 - R_2R_3}{(R_{BP}+R_2)(R_3+R_4)} V_{in} \tag{2}$$

R_{2-4} are known values that were chosen with the purpose of balancing the bridge. The bridge is balanced when V_{out} is null when the sensor is at rest. Eq. (3) presents the general rule for balancing a Wheatstone bridge [35].

$$\frac{R_{BP}}{R_2} = \frac{R_3}{R_4} \tag{3}$$

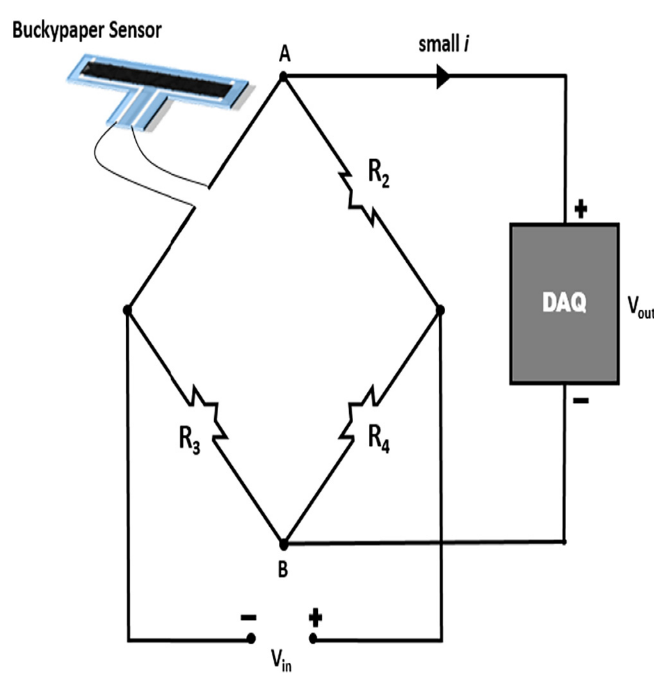

Fig. 5. Schematic of the Wheatstone bridge characterization setup, where $V_{in} = 3$ V and $R_{BP} \approx R_2 = R_3 = R_4$ to balance the bridge.

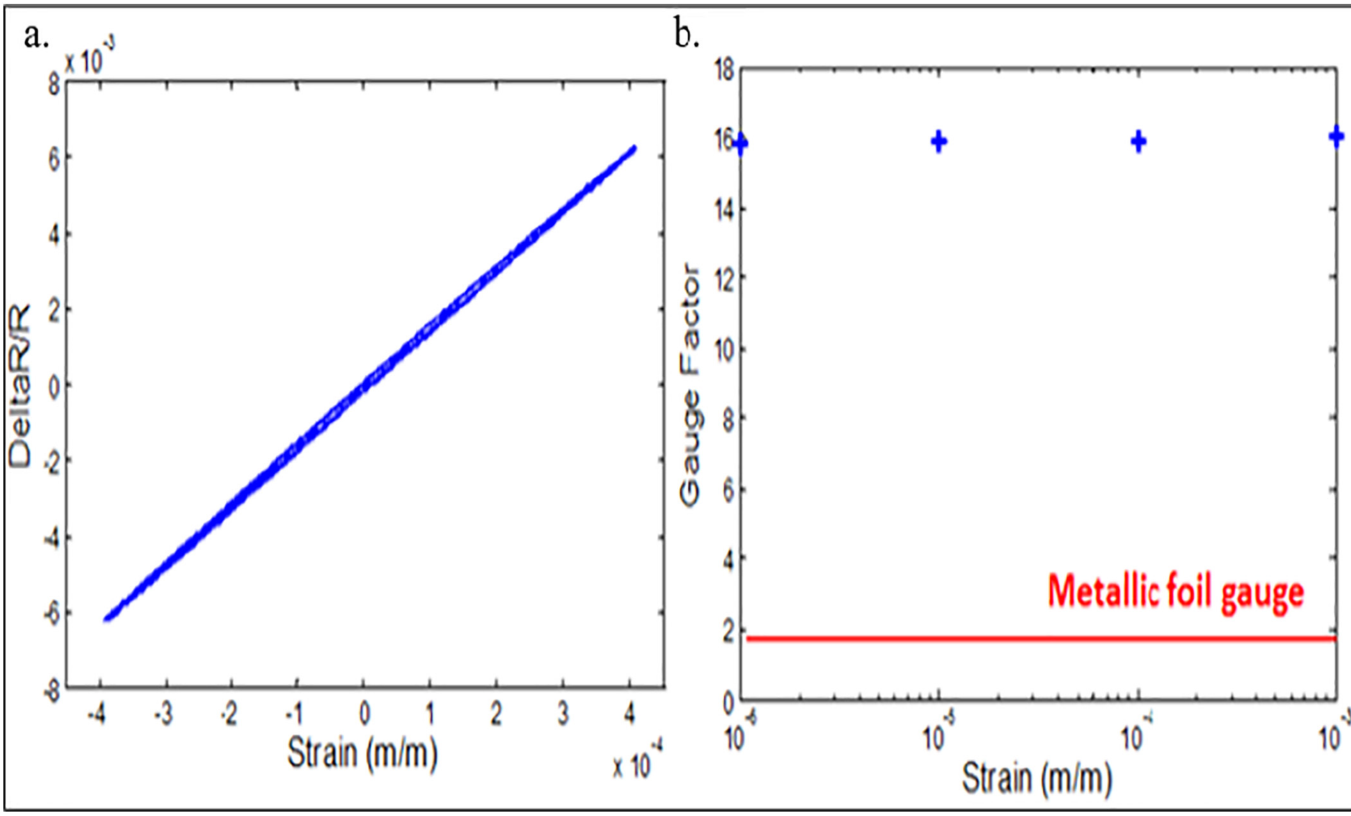

Fig. 6. BP strain sensor's dynamic response: (a) Measured sensitivity based on a Wheatstone bridge configuration. (b) Gauge factor versus strain comparison.

Eq. (4) expresses the change in the output voltage with respect to the induced change in resistance (ΔR_{BP}) due to an applied strain [35].

$$\Delta V_{out} = \frac{[(R_{BP} + \Delta R_{BP})R_4 - R_2R_3]}{[(R_{BP} + \Delta R_{BP}) + R_2)](R_3 + R_4)} V_{in} - 0 \quad (4)$$

By using equivalent resistors for $R_{2–4}$, the three resistors can be represented as a single resistance ($R_{BP} \approx R_2 = R_3 = R_4 = R$). Now, Eq. (4) can be simplified to determine sensor sensitivity, as expressed in Eq. (5).

$$\frac{\Delta V_{out}}{V_{in}} = \frac{\Delta R_{BP}}{4R} \quad (5)$$

By solving for ΔR_{BP}, the gauge factor (GF) can be determined using Eq. (6). The gauge factor expresses the sensitivity of the piezoresistive

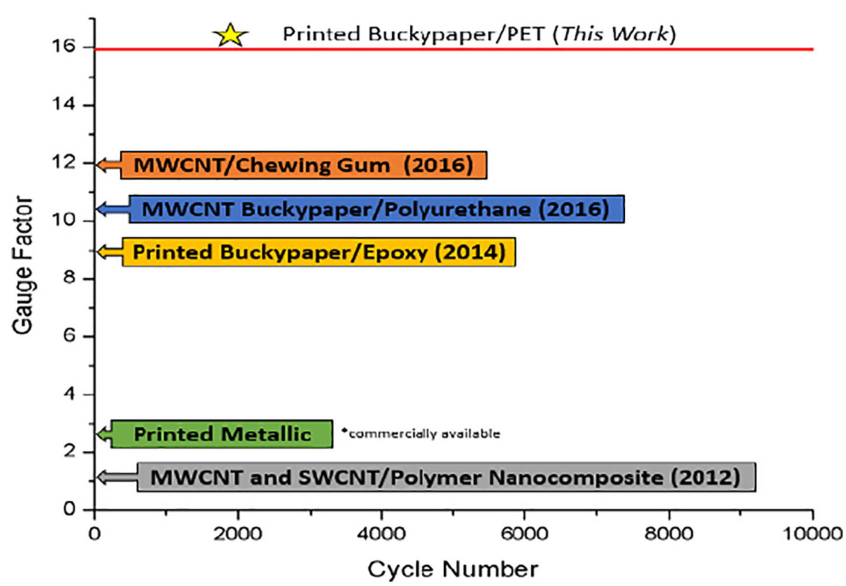

Fig. 7. Comparison of commercial and other CNT-based sensors' gauge factors. The printed BP sensor's gauge factor remains consistent for >10,000 cycles.

response to strain ($\frac{\Delta l}{L}$)

$$GF = \frac{\frac{\Delta R_{BP}}{R_{BP}}}{\frac{\Delta l}{L}} \tag{6}$$

Fig. 6a presents the percent change in resistance as a function of strain, and shows good linearity for calibration. A strong, linear relationship with respect to both extension and compression modes resulted in a large strain window [−0.4%, 0.4%]. The strain range was also rather large and symmetric, as the normalized resistance change reached 6.23×10^{-3} under 380 μm/m deformation in extension mode and reached 6.22×10^{-3} under 380 μm/m in compression mode. Fig. 6b displays steady behavior at various levels of strain, as the gauge factor remains constant up to 0.1% strain.

In contrast, the metallic semiconductor gauge strain range reached ±0.05%. Most notably, the gauge factor of the printed buckypaper sensor was eight times higher than the metallic gauge GF = (2.5). Note that gauge factor of the printed buckypaper sensor was low compared to classical semiconductor strain gauge ($50 < GF < 200$). However, semiconductor strain gauges are fragile, brittle, expensive, temperature-dependent, and possess a limited strain range. The buckypaper sensor offers much higher sensitivity than metallic sensors while promoting the benefits of printing technology and thin polymer substrates for conformability. Fig. 7 provides a comprehensive comparison among the presented CNT-BP sensor and recent CNT-based strain sensors [3,14,28]. The printed buckypaper sensor exhibits good stability as it maintains the same gauge factor for >10,000 cycles (at 1 Hz) of strain. The sensors can detect strains as low as 0.005% within a 10 ms period. The sensors were fastened to a latex glove using an adhesive to demonstrate their ability to detect subtle body movements. Fig. 8 displays the sensor output given obvious and subtle finger bending movements. The buckypaper sensor rapidly and consistently detected both forms of bending.

Monitoring, analyzing, and quantifying subtle, dynamic motions are vital for the advancement of human informatics. The motions detected by the printed buckypaper sensor were impressively small showing promise in measuring critical health data like heartbeat, breathing patterns and recognizing the intended movements of amputees [1,36]. Printed buckypaper sensor offers two main advantages. The first is that the manufacturing methods avoid traditional, low-throughput methods that have limited carbon nanotube-based devices to the laboratory scale. After establishing the printing process, various low profile substrates with high thermal and chemical resistance like polyimide (PI) polydimethylsiloxane (PDMS) can be used to improve performance, provide transparency and impede device degradation [6]. The second advantage is that paper materials are lightweight and deformability [30]. They have become excellent candidates in tactile sensing. Sensing performance can be enhanced by improving the Young's modulus of MWCNT buckypaper [10].

4. Conclusion

The integration of a carbon nanotube buckypaper with affordable printing technology has enabled the manufacturing of flexible tactile sensors. The results demonstrated significant improvement in response sensitivity in comparison to commercial metallic strain gauges. The sensors can detect micro-strains and the responses display good linearity in both extension and compression modes, which makes for easy calibration. The sensors were fastened to human fingers to display their ability to collect biomechanical data with high repeatability. The advances in the materials and processes described here provide several promising engineering options for printing low profile electronic components and integrated systems. Successful outcomes from these efforts have the potential to fundamentally change our conception of electronics, from hard, rigid, planar chips to soft, curvilinear sheets that can decode and store large amounts of biological information. Printable tactile sensors like the buckypaper design will continue to strengthen progress in human/machine interfacing, soft-robotics, and biomedicine.

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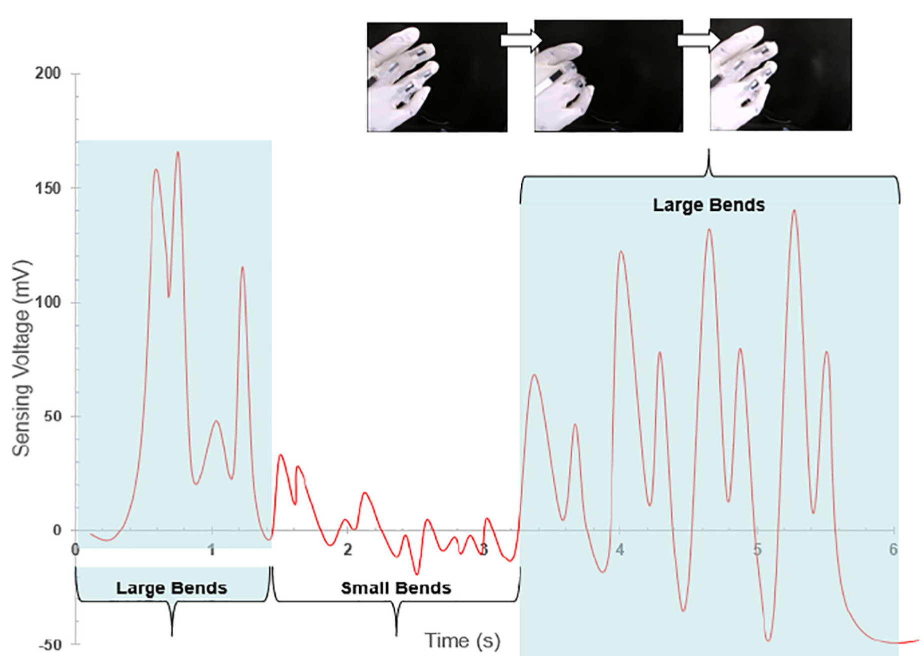

Fig. 8. Experimental results from monitoring fingers using BP strain gauge that was fastened to a glove.

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