

Potential Substrates for Low-Cost Flexible Supercapacitors

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ABSTRACT

Over the past several years, there has been a growing interest in the application of supercapacitors as replacements for conventional batteries, which are significant contributors of heavy metals into the environment. Flexible supercapacitors, unlike the traditional rigid supercapacitor, can be constructed with more accessible and cheaper flexible substrates. I constructed carbon nanotube (CNT) test samples using three low-cost substrates—polydimethylsiloxane (PDMS), polycarbonate (PC), and Scotch-brand matte adhesive tape—and looked at whether or not stress on the horizontally-aligned CNT affected the supercapacitor's performance with respect to capacitance, resistance, and cost. Using a Teflon tube to achieve horizontally-aligned carbon nanotubes (HA-CNT) prior to delamination in deionized water, the CNT were subjected to bending tests of varying degrees on all three substrates as well as cycling tests to measure specific capacitance in both a concave and convex orientation. There was no significant increase in resistance in any of the substrates to indicate a damaged CNT forest. Specific capacitances for the densified CNT samples were at least twice that of an undensified CNT sample. As for which material is best, because the PDMS, PC, and tape samples all performed well in the resistance tests, the choice depends on the application and which characteristics are required of the substrate. With its already remarkably high power density and long lifetime, flexible supercapacitor can now be manufactured at a lower cost and applied to a wider range of technologies than traditional rigid supercapacitors, providing the necessary foothold for future supercapacitors in the competitive energy storage market.

KEYWORDS

carbon nanotube, energy storage device, bending test, polydimethylsiloxane, polycarbonate

INTRODUCTION

Over the past several years, there has been a quickly growing interest in the application of supercapacitors as replacements for conventional batteries. At the moment, with its cheaper manufacturing costs and higher energy density compared to supercapacitors (Jennings et al. 2009), batteries are the preferred and most widely used form of chemical energy storage system in portable applications such as cellular phones, laptops, and mp3 players. However, rechargeable batteries suffer from low longevity and decreased performance as a result. Rechargeable batteries can only be recharged for an average of 200 to 1000 times before they are rendered unusable. Also, when used batteries are disposed of improperly, they can contribute a significant amount of toxic chemicals into the environment (Bozoglian et al. 2001). For example, urban pavement litter contains a surprisingly large number of consumer batteries that, when ruptured, release heavy metals—such as lead, cadmium, and mercury—that contaminate urban storm water (Jennings et al. 2009), making batteries an unwise option for further widespread applications.

Traditional supercapacitors are a form of chemical energy storage that are known for their long lifecycle and can often go through a million cycles with little to no decrease in its capacitance, meaning that supercapacitors will not have to be replaced as often as batteries. Also, supercapacitors use non-corrosive chemical electrolytes as well as low-toxicity materials that do not involve heavy metals, rendering supercapacitors less of a health and environmental hazard than batteries. But, because supercapacitors hold a charge that is only about a tenth of rechargeable batteries (Chai et al. 2007), this limits supercapacitors to mostly low-energy applications such as the flash application in cameras. Compared to ordinary capacitors, supercapacitors use electrochemical-based reactions that allow for higher energy densities (1000's of times greater) (Jayalakshmi et al. 2008). The electrical potential and high surface area of the carbon nanotubes (CNT) gives supercapacitors extremely fast charge and discharge rates and long lifecycles (Jiang et al. 2009). This makes them ideal for energy buffers and regenerative braking systems in hybrid vehicles (Bravis et al. 2010). With improvements to their energy storage capacity, supercapacitors have the potential to replace batteries (Chou et al. 2008). However, even with significant improvements to energy storage capacity, supercapacitors are still relatively expensive to produce in comparison to batteries (Musolino et al. 2010). Because

traditional rigid supercapacitors rely upon a silicon substrate as the foundation for the supercapacitor, the scope of a supercapacitor's applications are limited to its rigidity and further market cost penetration is difficult.

Flexible supercapacitors, unlike the traditional rigid supercapacitor, can be constructed with accessible and cheaper flexible substrates (Nyholm et al. 2011) such as polydimethylsiloxane (PDMS) or polycarbonate (PC). Although they can be used in a wider range of applications than rigid supercapacitors because of their more adaptable structure, the supercapacitor's flexibility is also physically stressing the entire system (Sansom et al. 2008). These challenges are due to the integrity of the carbon nanotube (CNT) forest being compromised as the entire structure is bent at various angles. Until now, common supercapacitors have not incorporated the use of horizontally-aligned CNT; they have only used vertically-aligned CNT (Tsai et al. 2009). When initially grown, CNT form from the bottom-up, making the orientation of the forest vertical. Horizontally-aligned CNT occurs when the forest is pressed down upon, causing the CNT to bend until the forest is lying horizontally. Despite the current studies of various flexible substrates, there is no conclusive study on a flexible substrate that can provide a strong adhesion for the CNT, high capacitance and stable resistance (Sansom et al. 2008).

My experiment focuses on using the horizontal orientation, which will allow the CNT forest to remain connected even while bent, thus circumventing the issue of increased resistivity (an increase in opposition of the electric current) and decreased capacitance. I am using three low-cost substrates—polydimethylsiloxane (PDMS), polycarbonate (PC), and Scotch-brand matte adhesive tape—and determining whether stress on the horizontally-aligned CNT will affect the supercapacitor's performance with respect to capacitance and resistance, as well as analyzing substrate costs. PDMS has potential due to its linear relationship between resistance and temperature and great stability (Kang et al. 2008). As another possible candidate, PC with an additional horizontally aligned carbon nanotube forest provides a strong adhesive surface for the CNT even while bent (Tsai et al. 2009). Lastly, Scotch tape will be tested due to its applicable thickness, adhesive strength, and low manufacturing cost. Performance will be based on how well the supercapacitor functions after bending tests and how its capacitance changes in a bent orientation. I will also be comparing the construction process between each material as well as their per unit area costs.

METHODS

Carbon nanotube fabrication and densification

In preparation for growing carbon nanotubes (CNT), Alina Kozinda thermally oxidized the initial bare silicon or quartz plate (15mm by 40mm) and evaporated molybdenum, aluminum, and iron with a thickness of 50nm, 10nm, and 5nm, respectively. For this experiment, iron was the common catalyst for CNT growth. I obtained the plates from Alina with permission and grew vertically-aligned CNT using a thermal chemical vapor deposition (CVD) furnace (Linderg/Blue M three-zone tube furnace, Thermo Electron Corp., Asheville, NC). After first purging the furnace with hydrogen gas to get rid of any possible contaminants, I grew the CNT with a mix of ethylene and hydrogen with a 1:3 ratio at 720°C for 10 minutes to a height of approximately 87 μm , well within the required range of 40-100 μm necessary for this experiment. After I extracted the plate from the furnace, I broke it into individual units, each measuring 3mm by 10mm. I pressed down on the CNT with either a thumb or a Teflon rod in order to have the CNT lying horizontally rather than the initial vertical orientation. I placed the individual sections into a dust-free plastic dish filled with de-ionized (DI) water for approximately 20 minutes. This initial process is to ensure CNT densification (from capillary and Van der Waals forces in interacting with water), which decreases the volume of the CNT forest and, therefore, increases capacitance of the supercapacitor.

After the CNT densified, I filled the dishes with DI water once more and the CNT was submerged for approximately 3 to 7 days to allow the CNT lift-off to the substrate and to allow the water to evaporate.

Supercapacitor sample construction

Capacitance test samples

Once the CNT was separated from the initial growth wafer but while it was still suspended in the DI water, I transferred the CNT onto a strip of aluminum tape already bent

convexly or concavely. This was to ensure that the CNT would be able to hold the bent orientation during the drying stage as well as during the test. I used a 0.1M K_2MO_4 solution as the electrolyte with an Ag/AgCl reference electrode and Pt counter electrode.

Resistance test samples

In order to test whether or not a flexible supercapacitor can function after being bent, I constructed three sets of flexible supercapacitors using three different types of substrates to test for resistance: poly-dimethylsiloxane (PDMS), polycarbonate (PC), and Scotch brand matte adhesive tape. The PDMS and tape were from the lab stock while the PC I ordered from McMaster-Carr (McMaster-Carr, Santa Fe Springs). After the CNT was transferred onto the surface of the substrate, I lightly dabbed each end of the CNT strip with silver epoxy paste. After the silver epoxy paste was dry, I covered it with aluminum tape before attached an electrode to each of the ends and covering it with aluminum tape as well.

I constructed a single sample of both the PDMS and the tape, and two samples for the PC. The PDMS and tape samples were subject to both concave and convex bending, alternating between the two orientations. Because I had two PC samples, one sample was subject to convex bending and the other to concave bending.

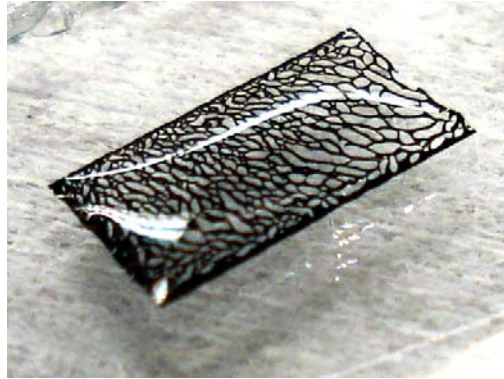
Bending and cycling tests

In order to simulate bending in a real application, after first recording the base resistance value, each sample was bent in sets of either 10, 20, or 30 bends depending on the particular material being tested. I recorded the resistance in the bent state as well as the unbent state in order to determine, if any, the spike in resistance that indicated a damaged CNT forest from the bending. All three samples of PDMS, PC, and tape were subject to first a set of regular bends measuring roughly an angle of approximately 30° and, if no damages occurred, were bent at a more extreme angle of 60° . Due to its pliability, the tape sample was further subject to a set of alternating flexes, where I bent the ends in opposite direction so that the CNT was bent at an angle of approximately 170° .

I used a *Gamry Instruments Reference 600*, a potentiostat, to run a capacitance test (i.e. running a current through it) and obtain a cyclic voltammetry (CV) curve. The samples I tested were a densified convex CNT, a densified concave CNT, an unpressed and undensified (i.e. straight from the furnace) CNT, and an unpressed but densified CNT, which I will refer to as the “zebra” sample due to the zebra-like striations of the CNT forest (Fig. 1). I ran it for an average of 10 cycles per test sample and specific capacitance was calculated using this equation:

$$C_{sp} = \frac{I}{\left(\frac{dV}{dt}\right) \times A} \quad \text{Eq. 1}$$

(a)



(b)

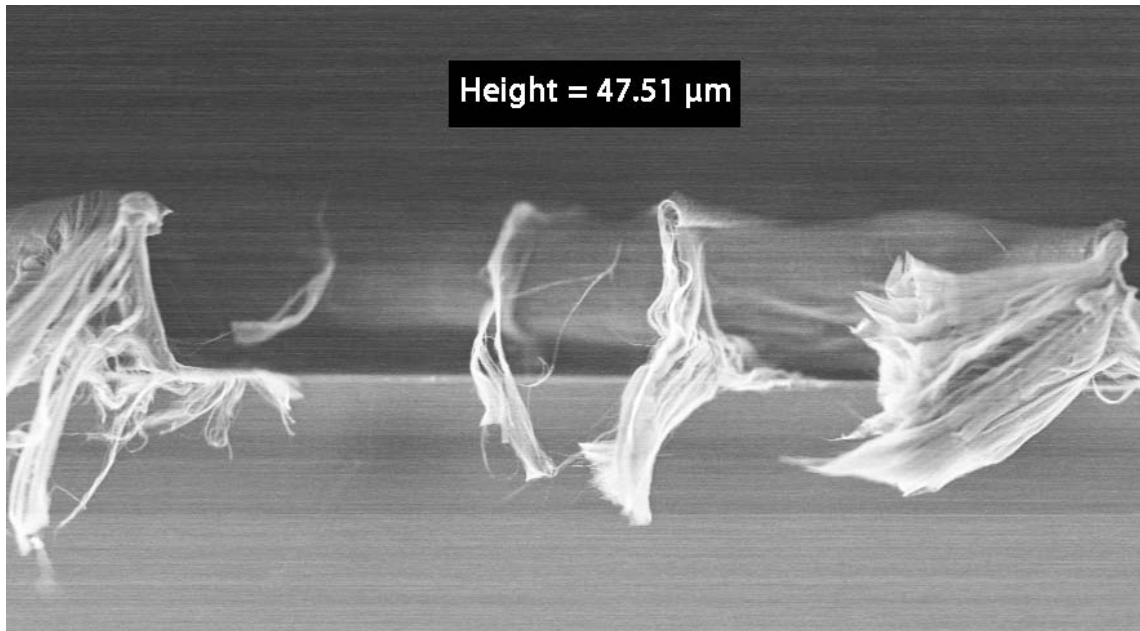


Figure 1. Close-up images of a zebra-pattern CNT sample. (a) Digital camera image of a zebra-pattern CNT on a PDMS substrate. (b) SEM (scanning electron microscope) image of a zebra-pattern CNT forest. The walls in the SEM image are the CNT bunches due to capillary forces, and the spaces between the walls are the areas with no CNT coverage.

RESULTS

Carbon nanotube densification

I found that the samples that were flattened with a thumb, after they densified in DI water, yielded zebra-pattern CNT (Fig. 1), which rendered the CNT unusable for my experiment since they couldn't be bent without compromising the integrity of the CNT forest. However, for the samples that were pressed with a Teflon rod, the batches consistently yielded uniformly pressed CNT forests that densified in DI water perfectly and resulted in a sufficiently dense forest of 987nm (Fig. 2). The pressed and densified CNT lifted off the substrate in approximately 3 days with little to no breakage in the surface.

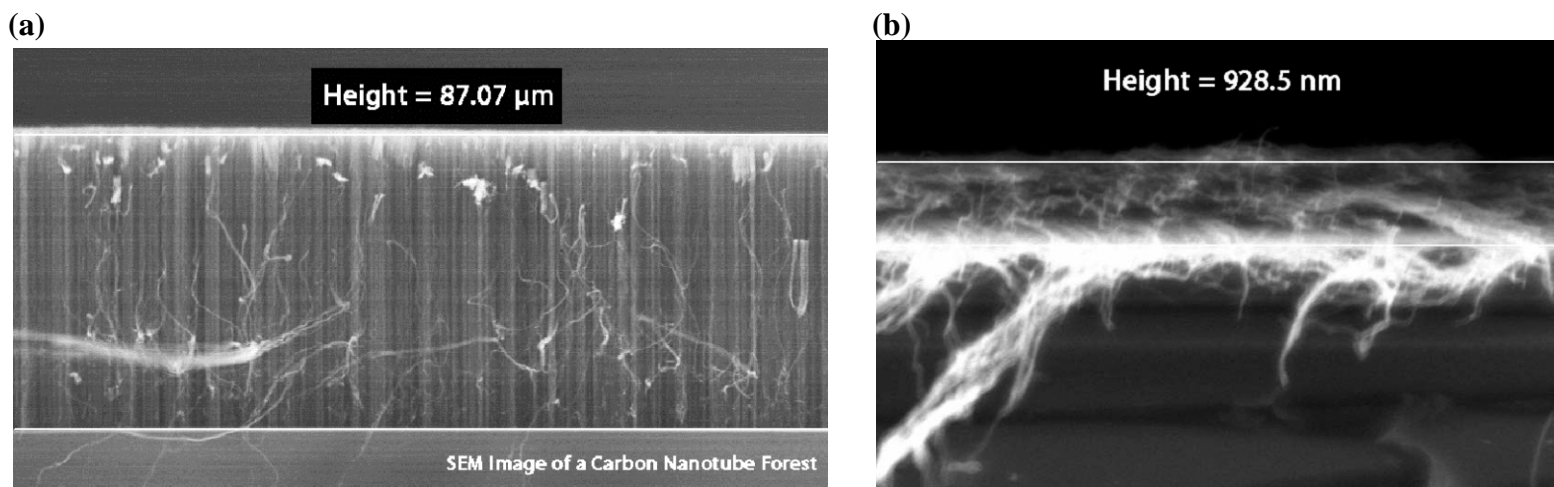


Figure 2. SEM images of a CNT forest. (a) An unpressed and undensified CNT forest with a height of 87.07 μm. (b) A pressed and densified CNT forest using a Teflon rod, with a height of 0.928 μm, a factor of approximately 94 in reduction.

Level of construction difficulty

Out of the three substrates I tested, PDMS was the most difficult to construct a sample for the resistance tests. Although the CNT was relatively simple to transfer onto the surface, the PDMS did not bond well with the silver epoxy paste. For an unknown reason, after I assembled the electrodes and aluminum tape on the sample, the area where the silver epoxy paste ended and where the CNT was exposed had already begun to crack even prior to the bending tests (Fig. 3).

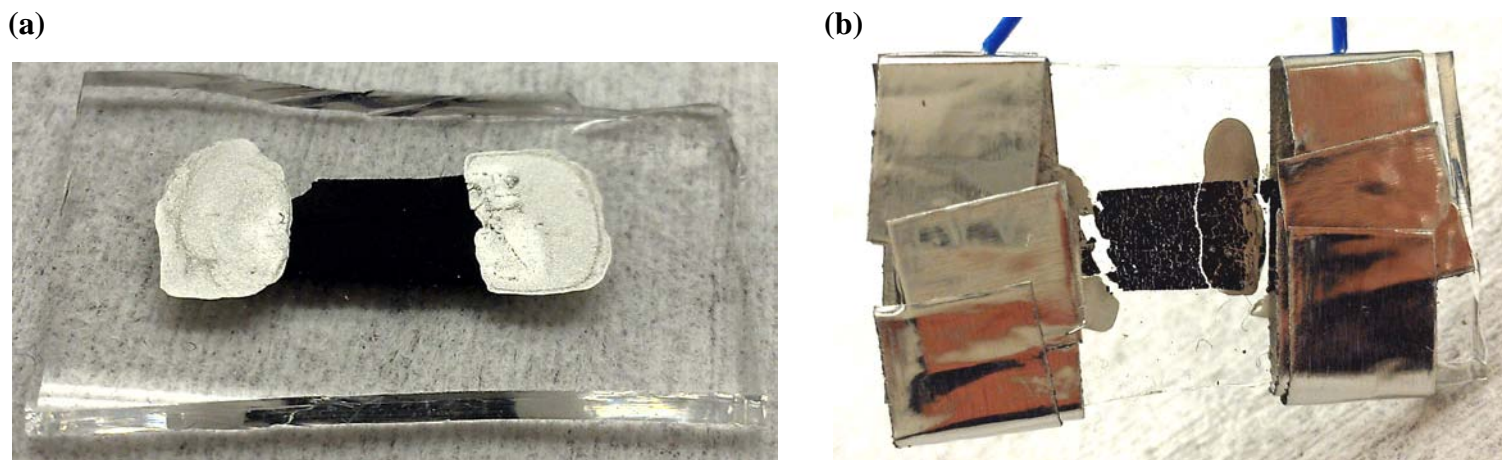


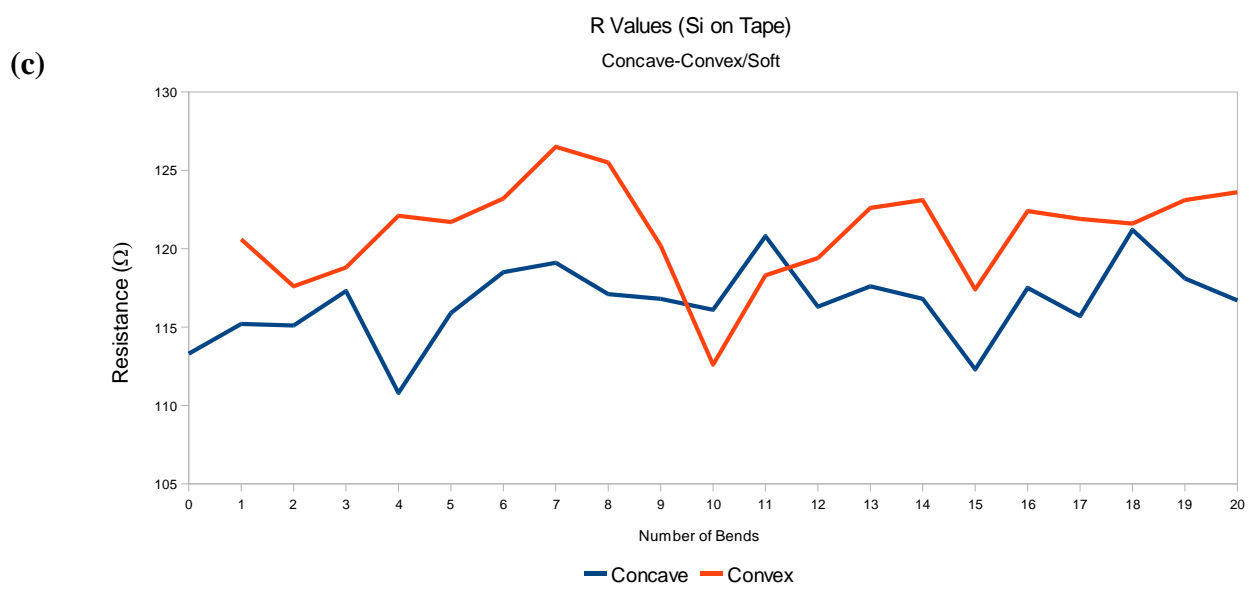
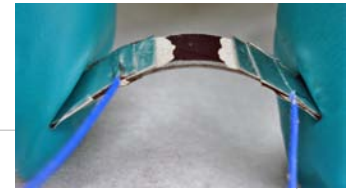
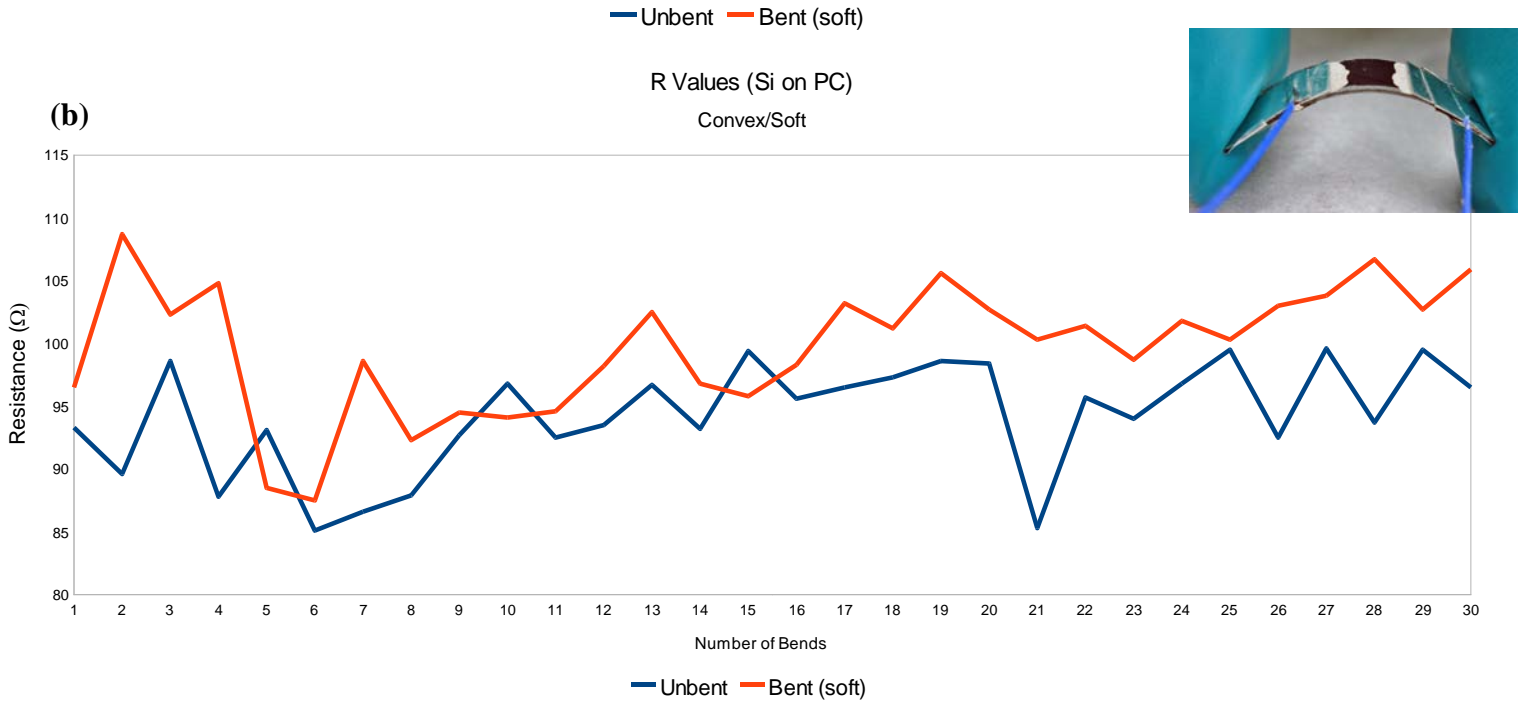
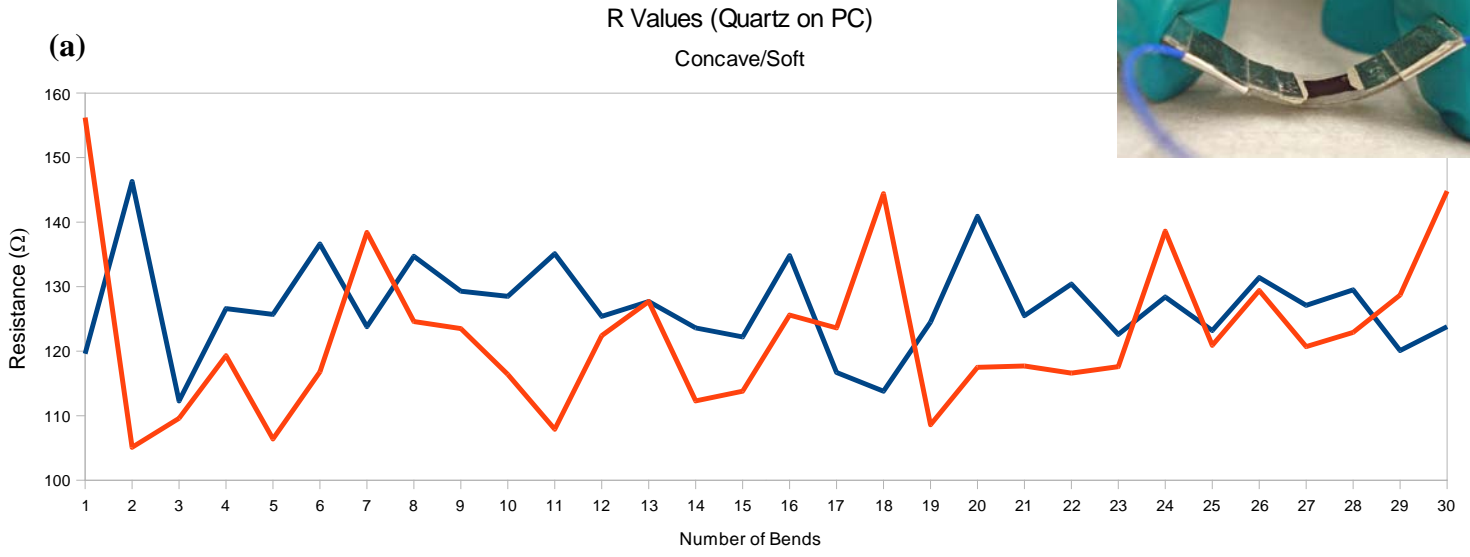
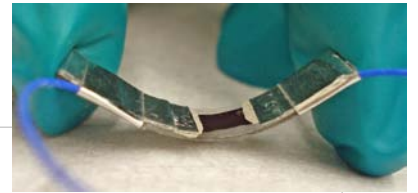
Figure 3. PDMS sample for resistance testing. (a) Topside image of silver epoxy paste and the CNT sample still intact. (b) Underside image of the sample after attaching the electrode and aluminum tape. The CNT has cracked in several places, all where it comes into contact with the silver epoxy paste.

The PC sample, despite having only a thickness of 0.015 inches, was stiffer than the PDMS, which had a thickness of 0.125 inches. I transferred the CNT onto the glossy side of the PC sample to see if the CNT would crack when coming into contact with the silver epoxy paste as it did on the PDMS sample. However, the CNT did not prove difficult to assemble on the PC and no cracks appeared on the surface.

The tape had a natural adhesive on one side, making it more difficult to transfer the CNT onto the surface. Although the adhesive helped with setting the CNT film onto the surface, it also made it difficult for me to position it correctly. Any major adjustment tore the fragile CNT forest, making tape a relatively cheap but nevertheless more troublesome substrate for the initial setup for supercapacitors. I chose to double the tape back onto itself so that both sides were the matte surface of the tape, and the CNT was easily transferred onto the tape. The tape sample had no problems with the silver epoxy paste.

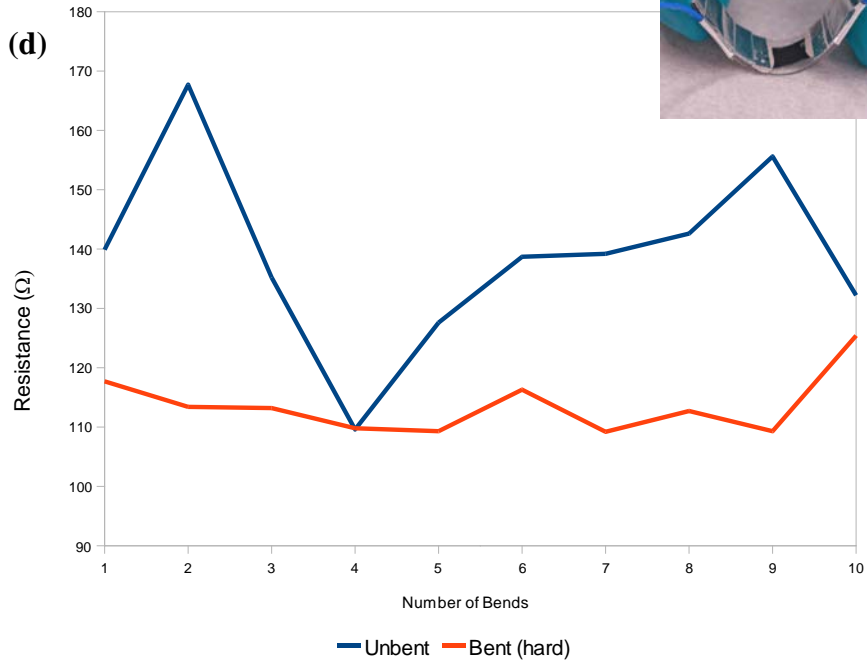
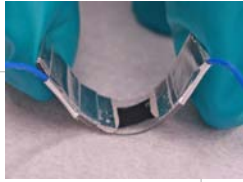
Bending and cycling tests

In the bending tests for the PC and tape samples, the CNT did not exhibit a significant change in resistance in either the convex or concave bending direction (Fig. 4a-e). For the tape sample, I noticed that the resistance levels still did not increase even after bending the sides in an almost 180° orientation (Fig. 4f). However, after introducing artificial cracks in the surface of the tape CNT, the sample ran consistently high values thereafter (Fig. 4g).



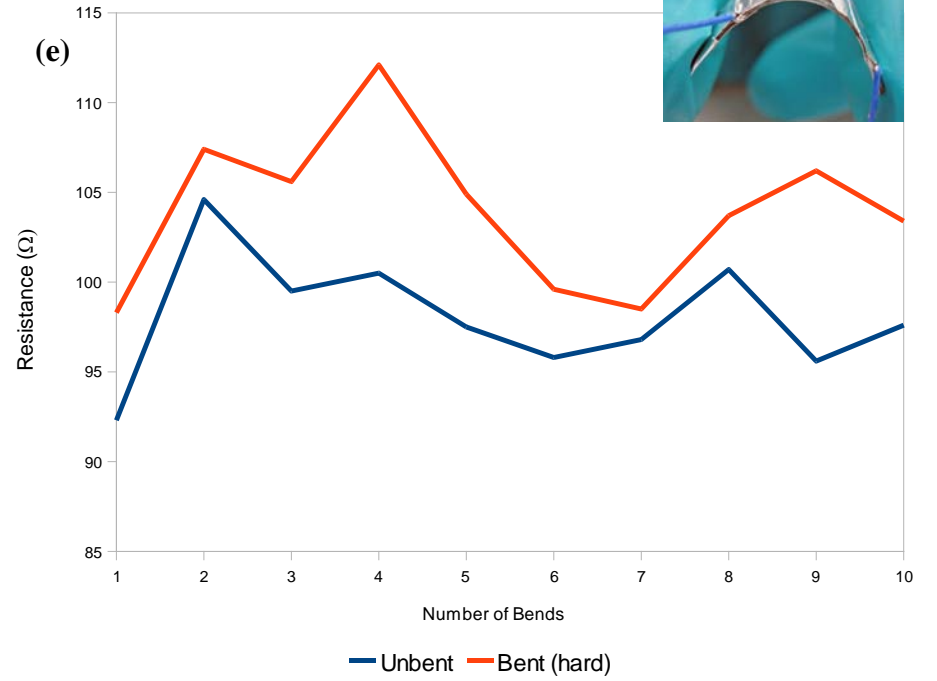
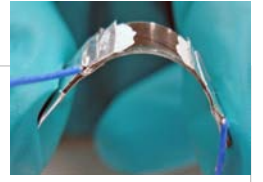
R Values (Quartz on PC)

Concave/Hard



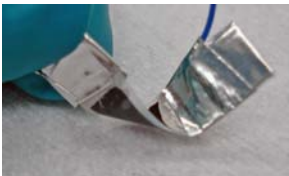
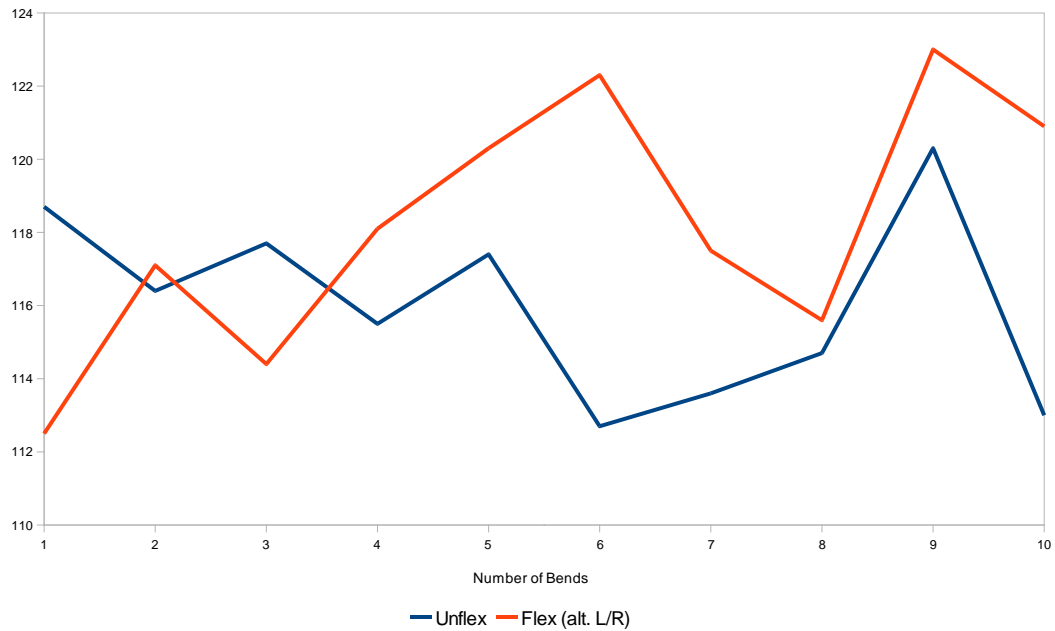
R Values (Si on PC)

Convex/Hard



R Values (Si on Tape)

Flex/Hard



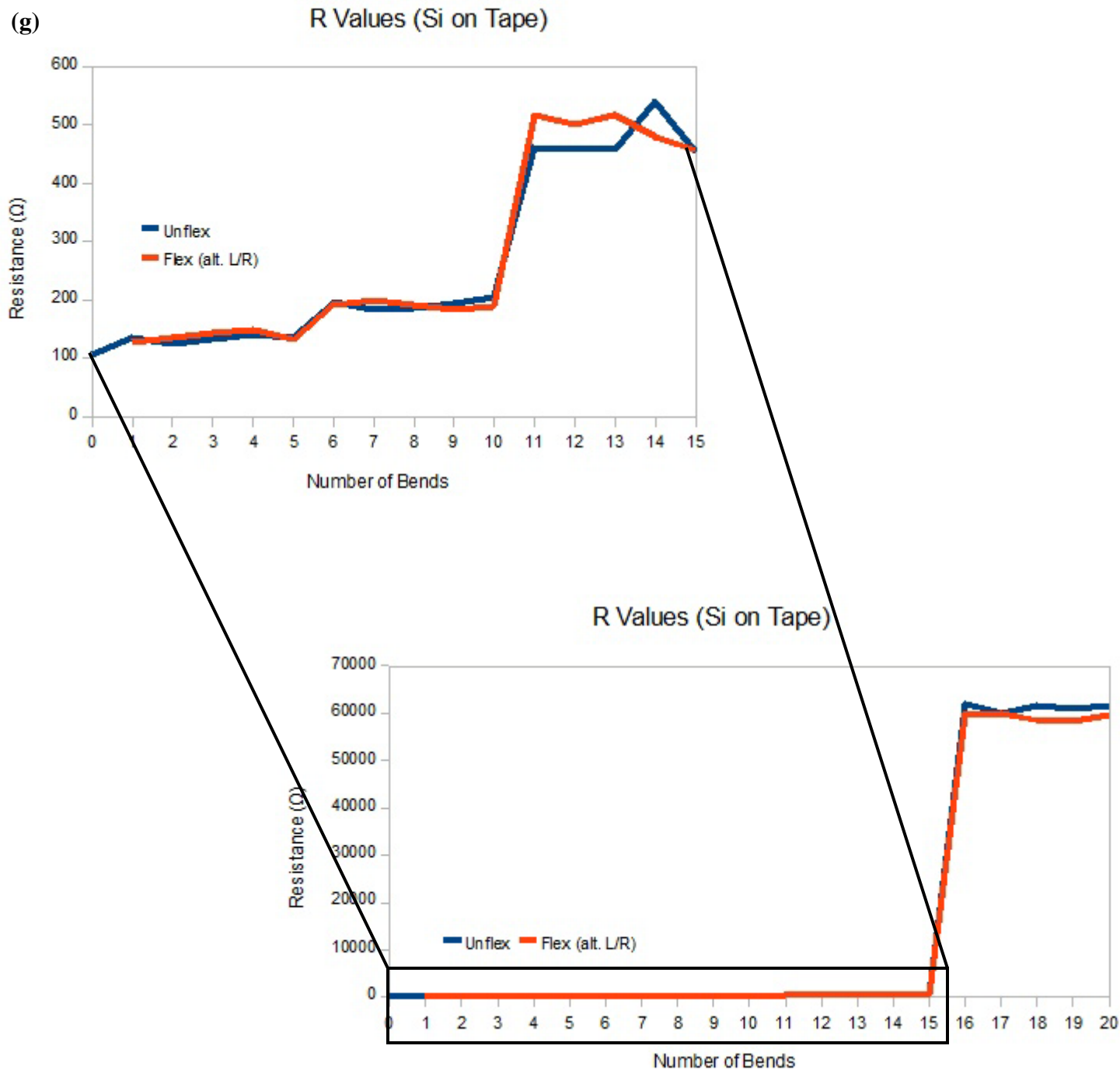


Figure 4. Resistance graphs for PC and Tape samples. (a) Quartz-grown CNT on PC with soft concave bending. (b) Si-grown CNT sample on PC with soft convex bending. (c) Si-grown CNT on tape with soft convex and concave bending. (d) Quartz-grown CNT on PC with hard concave bending. (e) Si-grown CNT on PC with hard convex bending. (f) Si-grown CNT on tape with flex bending. (g) Si-grown CNT on tape with one artificially-created scratch at bends 1, and additional scratches at bends 5, 10, and 15. Resistance significantly increased from ohms to kilohms after bend 15 after an extensive network of scratches appeared on the CNT forest.

Capacitance tests

The results from the cycling test show that both the convex and concave sample exhibited a specific capacitance at least 2.98 times higher than that for the control sample—an undensified CNT forest—and 2.72 times higher than that of the zebra pattern sample (Fig. 5). Using Equation 1 with a scanning rate of voltage (dV/dt) of 50 mV/s, an area A of 0.25 cm^2 , the specific capacitances for the densified concave and convex samples were 4.92 mF/cm^2 and 9.28 mF/cm^2 , respectively. For the undensified and zebra sample, the specific capacitances were 1.65 mF/cm^2 and 1.81 mF/cm^2 , respectively.

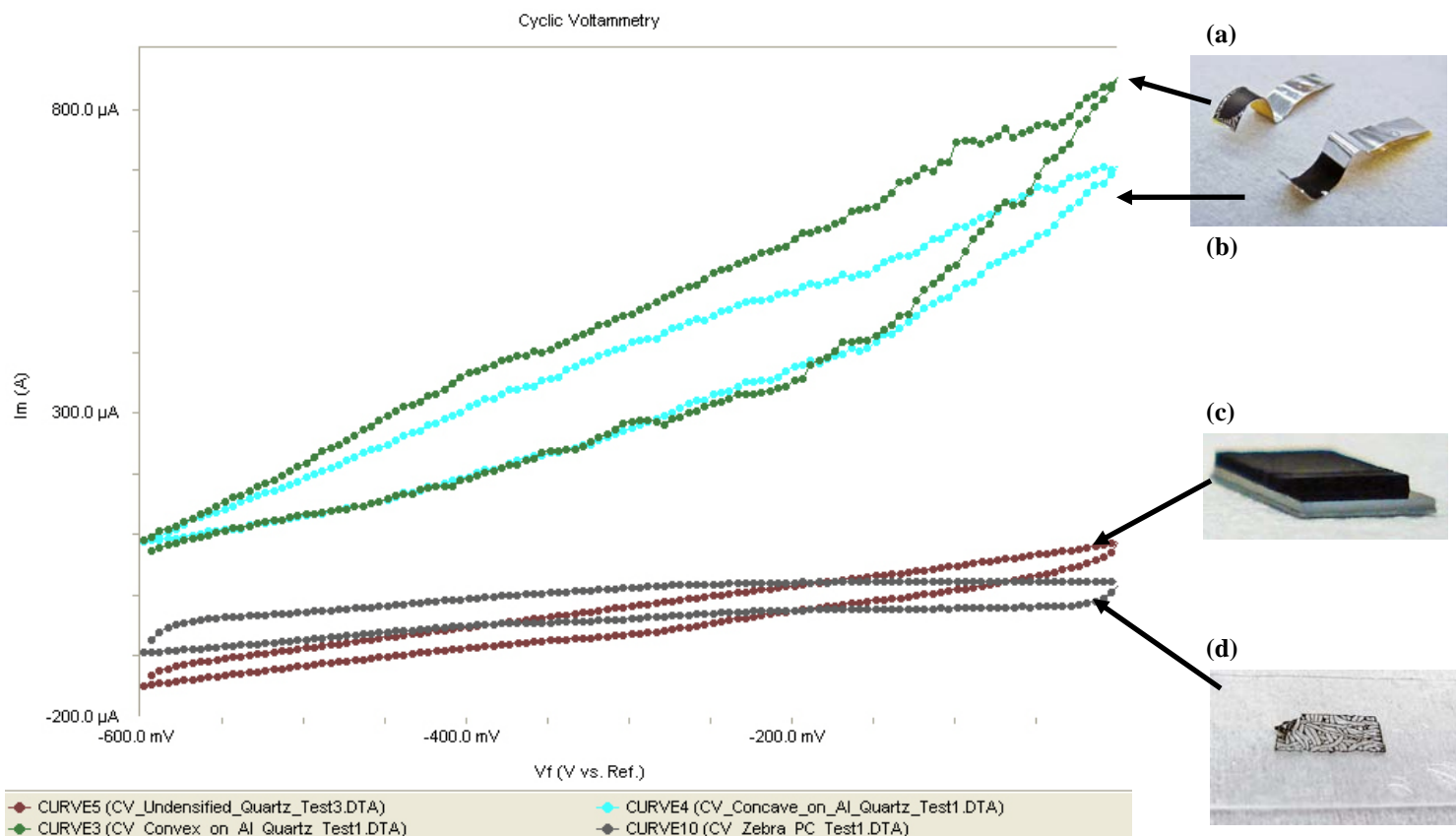


Figure 5. Cyclic voltammetry curves for four different samples. (a) Pressed and densified, quartz-grown CNT on convex Al tape. (b) Pressed and densified, quartz-grown CNT on concave Al tape. (c) Unpressed and undensified, quartz-grown CNT on unben quartz substrate. (d) Unpressed, and densified, zebra CNT on unben PC.

Cost per unit area

In order to compare the cost of materials, I calculated the cost per unit area of the four main substrates in my experiment (Table 1). A typical, pre-treatment P-type silicon wafer from the Marvell Lab (Marvell Lab, University of California, Berkeley) was \$3.49 per square foot (25-wafer case pricing). I indirectly obtained the 0.125 inch thick PDMS, which was made in another lab, at approximately \$1.44 per square foot. Polycarbonate sheets (24" by 24") from McMaster-Carr (McMaster-Carr, Santa Fe Springs), depending on the surface texture—gloss or velvet/matte—were \$0.58 and \$0.52 per square foot, respectively. Lastly, a matte adhesive tape (Scotch Tape #600 partial case pricing for ½" by 36 yards) from McMaster-Carr was \$0.45 per square foot and the cheapest option between all the substrates. However, because bending tests required thicker substrates and I had to increase the thickness of the tape sample by doubling it, the tape could cost as much as \$0.90 per square foot.

Table 1. Material cost per unit area. Costs are calculated using commercial and lab pricing.

<u>Substrate</u>	<u>Cost per Unit Area</u>
P-Type 4" Silicon Plate	\$3.49/ft ²
PDMS (polydimethylsiloxane)	\$1.44/ ft ²
PC (polycarbonate)	\$0.52-0.58/ ft ²
Matte Adhesive Tape (Scotch Tape #600)	\$0.45-0.90/ft ²

DISCUSSION

This project focused on three substrates that could potentially be part of cheaper and flexible supercapacitors commercially viable for mass production to replace expensive silicon substrates. The substrates were chosen for their inexpensive cost, physical and electrical properties. Both polydimethylsiloxane (Kang et al. 2008) and polycarbonate (Tsai et al. 2009) have previously been tested as flexible CNT devices. However, my study aimed at testing these substrates, along with matte adhesive tape, with horizontally-aligned carbon nanotubes (HA-CNT). The PC and tape samples exhibited excellent resistivity values in bending tests of up to

90°. The HA-CNT in both the concave and convex form had a specific capacitance at least twice that of a unpressed, undensified CNT sample.

Fabrication and construction

CNT density levels and uniformity differed between those pressed by hand and those by a Teflon rod (1/4" total diameter). Samples pressed with a Teflon rod delaminated from the silicon wafer more consistently than did those pressed by hand, which tended to form breaks in the surface with the zebra pattern, thus rendering the samples unusable for my experiment. The non-stick surface of the Teflon rod also reduced the amount of damage to the fragile CNT forest during the flattening process, increasing the number of usable samples in a single batch of CNT. Because few other papers have worked with HA-CNT, there isn't much discussion on various other techniques in flattening CNT. Nevertheless, the addition of such a simple yet highly effective tool like the Teflon rod has greatly expedited the flattening process as well as yielded more consistent results. Both methods yielded roughly equal lift-off time from the base. Any variation in lift-off time varied from sample to sample (i.e. a quartz sample versus a silicon sample of CNT).

CNT construction has been reported with highly variable heights. Using the chemical vapor deposition method, my CNT samples were 87.07 μm and 0.928 μm for an unpressed and pressed sample, respectively. De Montsabert et al. (2005) used wet-etched nickel nanoparticles in making vertically-aligned carbon nanotubes (VA-CNT) and reported an average height of 22.3 μm . Hiramatsu et al. (2007) used plasma-enhance chemical vapor CNT deposition and reported a density of 40 μm . Both of these suggest that CNT height is greatly dependent on the method of growth. However, even with methods similar to ours, Zhu et al (2006) has reported a height of 150 μm . Due to the natural adhesion between the CNT and the all three substrates, CNT height orientation had no effect on the level of difficulty in transferring CNT onto the substrate. Therefore, the only difference between VA-CNT and HA-CNT in the pre-liftoff process was the additional step in flattening the CNT, which was made easier with the addition of the Teflon rod, indicating that this simple method could be incorporated in large-scale manufacturing with minimal costs.

Resistance and capacitance analysis

Damages in all samples with HA-CNT—regardless of the substrate with the exception of the PDMS sample—during bending were minimal in comparison to the samples with VA-CNT. In a vertical alignment, concave bending pushes CNTs together. The increased contact between the CNTs and the compressive stress on the substrate surface, shortened the conducting path and subsequently reduced the electrical resistance. Convex bending increased tensile stress on the surface of the substrates, but the HA-CNT were able to undergo convex bending with minimal to zero damage on the nanotube forest for all three substrates. Resistance levels never increased past a 100Ω range for all three samples, indicating that the CNT surface remained intact even with bends of 90° for the PC sample and 170° in a flex test for the tape sample. Only artificially creating cracks in the HA-CNT by scrapping at the surface increased the sample's resistivity, pushing it into the kilohm range, typical of samples exhibiting broken resistance. Tsai et al. (2009) performed both concave and convex bending tests with VA-CNT, which resulted in a sharp increase in electrical resistance as the CNT forest fractured in a convex bend. This suggests that HA-CNT function better than VA-CNT in bending tests due to the ability of HA-CNT to spread the stress over a larger area.

Cost

The cost per unit area of a silicon wafer was higher than the three flexible substrates, and costs per unit area between the PDMS, PC, and tape varied as well. Because PDMS is made from a silicon elastomer kit that involves the mixing of two parts—a base and a curing agent—it can be made in almost any thickness and can be a very useful material in cases where a custom thickness is required. The PC can be purchased in a variety of different coats (i.e. gloss, matte, and velvet) as well as a variety of thickness as well, making it a cheaper option than the PDMS if a specialized texture is required. The Scotch brand matte adhesive tape has the advantage in that it can be obtained in almost any office supply store, whereas the silicon, PDMS, and PC have to be ordered from a specialized seller. However, in a commercial setting, there would be little to no issue in obtaining any of the materials, rendering the matte adhesive tape in applications that require extreme flexibility. All three materials are well below the cost of a silicon wafer and, if it

can be incorporated into a supercapacitor in a commercial setting, can greatly cut down on substrate costs. As for which type is more advantageous, it depends on what application the supercapacitor is used for and what characteristics are required (i.e. flexibility, stiffness, temperature, etc.).

Limitations

Due to time constraints, I was only able to construct three samples for each of the substrates for resistance testing, which meant that any anomalies that might have occurred could not be verified as such due to lack of another similar sample. Although the most important aspect in resistance testing is the range of fluctuation rather than the actual measurement, it would be easier to eliminate possible biases in the data due to errors in readings.

I was unable to obtain any useable resistance readings for the PDMS sample because the silver epoxy paste that I used as the electrical connector between the electrode and the CNT cracked the CNT and rendered the sample unusable. I did not have access to any other electrical connector in the lab that could've been used in place of the silver epoxy paste.

Also, the results for capacitance were difficult to obtain due to the need for aluminum tape as the base and not the three substrates I'm testing. Although the CV (cyclic voltammetry) curves gave the correct specific capacitance, the aluminum tape skewed the curve itself, giving inconsistent readings from cycle to cycle. A more consistent CV curve could be obtained if I were able to run 1000 cycles, but because that would've taken a minimum of 9 consecutive hours and my lab time is limited to around half that, I was unable to obtain more data.

Future Directions

This experiment's results can be used to inform future research regarding flexible supercapacitors. Different types of stress tests such as exposure to more extreme temperatures and substrate flexing, implementing alternative methods of CNT growth, and/or using different flexible materials for the supercapacitor substrate would provide us with even more options of flexible nanotechnology that may potentially be applicable for a wide variety of situations. Alternative methods of CNT growth can include the use of denser nanotubes via plasma-enhanced vapor deposition to see if a higher-density HA-CNT forest can still function on the

same level as a lower-density HA-CNT forest. Other potential substrates may have better electrical properties and lower per unit cost than the three tested in this experiment.

Conclusion

In this experiment, I was able to construct a flexible sample using horizontally-aligned CNT that performed well in capacitance and resistance tests while subject to bending. By using a Teflon rod to flatten the CNT, I was able to consistently obtain dense CNT samples with little to no breakage in the CNT forest. In turn, the HA-CNT samples performed with no significant increase in resistance while subject to extreme bends of up to 170° . The densified, convex and concave CNT samples had specific capacitances at least 2.72 to 2.97 times greater than that of a VA-CNT sample and a zebra sample.

With its remarkably high power density and long lifetime, supercapacitors are already a rapidly growing topic of research as a suitable form of cleaner energy. A simple, yet powerful supercapacitor can potentially be constructed alongside more energy-dense storage devices such as a hydrogen fuel cells resulting in a suitable alternative for current technologies such as the internal combustion engine in modern vehicles. If a flexible supercapacitor can be manufactured at a lower cost and applied to a wider range of technologies than traditional supercapacitors, it may provide the necessary foothold for future supercapacitors in the competitive energy storage market.

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