

On the Performance of Carbon Nanotubes in Extreme Conditions and in the Presence of Microwaves

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14. ABSTRACT Using van der Pauw and microwave surface resistance measurements, a series of temperature-dependent data sets from carbon nanotube (CNT) thin films have been measured. The test structures were fabricated using photolithography, E-beam evaporation, and a novel CNT network deposition technique. The sheet resistance and resistivity of each sample were recorded at temperatures ranging from 0–60 °C with test currents ranging from 100 nA to 100 µA. These values demonstrated excellent linearity, with no dependence on currents. At room temperature, the sheet resistance yielded a negative temperature coefficient (of approximately –900 ppm/°C). Our objective is to analyze the effect that DC and microwave currents have on CNT thin films using a two-point measurement technique on Corbino discs and in the presence of microwaves (8–12 GHz) to determine at which temperatures a CNT thin film performs best and identify the ideal temperature range in which a CNT microchip yields the maximized sheet resistance. The results counter the trend found by the four-point measurements; a positive temperature coefficient was observed. This observation indicates the temperature coefficient of the material is actually dependent on the size of the sample tested.					
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1. Introduction

There is currently a strong military interest in the use of transparent conductors for communication applications. Materials that have been considered for use as transparent conductors include: transparent conducting oxides (TCOs), intrinsically conducting polymers (ICPs), graphene, elemental metal nanowires, and carbon nanotubes (CNTs). All of these materials offer advantages and disadvantages. TCOs offer excellent conduction and optical transmission characteristics but tend to be very brittle and cannot be fabricated on flexible substrates. ICPs have good conduction and optical transmission properties, but are extremely sensitive to environmental conditions (such as temperature and humidity). Graphene has recently attracted much attention within the research community because of its exotic physical properties. While it is very compatible with flexible substrates, it is very challenging to manufacture and its electrical properties for communication applications are still inadequate at this point in time. Elemental metal nanowires offer excellent electrical properties and are also very compatible with flexible substrates; however, they easily oxidize and require passivation coatings. In this study, we have tested the properties of CNT thin films (1–3) for the purpose of evaluating their performance in communication electronics applications.

2. Four-point DC Measurements

2.1 Setup

Four-point test structures (4, 5) were fabricated on top of sapphire substrates using photolithography (6) and electron-beam evaporation processes. A layer of chrome was applied as an adhesive between the substrate and the metal conduction layer. Copper was applied to the chrome layer to reduce the resistance of the film. A gold cap layer was required to protect the copper layer from becoming oxidized and damaged. A thin film composed of a CNT network was deposited by an independent company called Nano-C[®] and returned almost test-ready. When returned, the wafers were diced by hand using a diamond scribe. Because two of the contacts were damaged during the dicing procedure, silver paint was applied to the sample to serve as improvised contact/probe-landing points. Figure 1 shows the CNT thin-film structure.

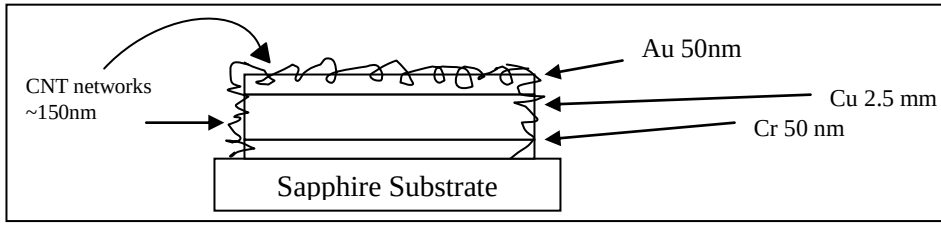


Figure 1. CNT thin film.

Nine temperatures were tested in this experiment. Due to the seemingly strange behavior of the first set, the focus of the second set was shifted to the range of 0–10 °C (typical freezing range). The final set was defined by the temperatures 0, 1, 2, 3, 6, 10, 20, 30, and 60 °C (commercial product test ceiling). Test currents were sourced from 100 nA to 100 μ A. Room temperature was roughly 24 °C and relative humidity was at 65%. The results were as followed.

2.2 Results

The calculated temperature coefficient for each temperature was negative, meaning that as the sample temperature increases, the sheet resistance decreases. This trend is shown in figure 2.

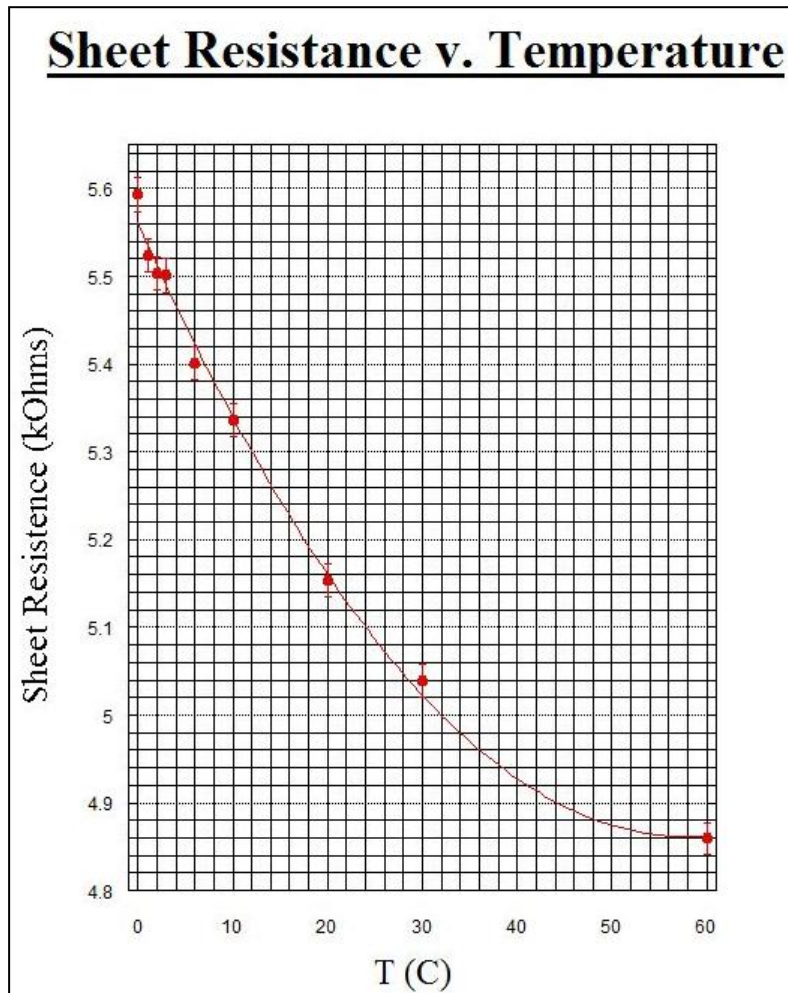


Figure 2. Overall decrease in sheet resistance as temperature increases.

Figure 3 and table 1 show the temperature-dependent data of sheet resistance measured using the four-point technique. In figure 3, the sheet resistance was measured using test currents ranging in value from 100 nA to 100 μ A. These data show exceptional temperature stability. There is little self-heating present; the test current does not greatly influence the measured sheet resistance. Table 1 lists the sheet resistance data points, measured using the limits of the test current range (100 nA and 100 μ A), as a function of temperature, which ranged from 0 to 60 $^{\circ}$ C.

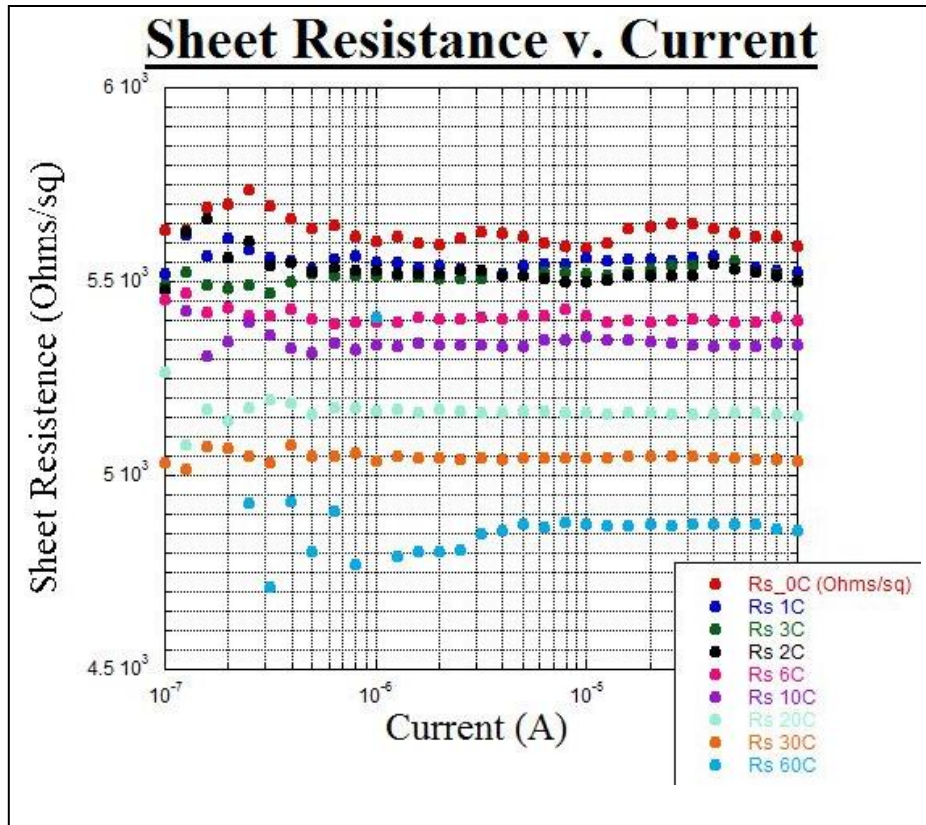


Figure 3. Temperature dependence of DC sheet resistance measured with test currents ranging from 100 nA to 100 μ A.

Table 1. Temperature dependence of DC sheet resistance measured with test currents of 100 nA and 100 μ A.

T(C)	Input	Output (R_s) in ohms/sq	Input	Output (R_s) in ohms/sq
0 degrees	100 nA	5943.33	100 μ A	5745.65
1 degree	100 nA	5523.30	100 μ A	5529.14
2 degrees	100 nA	5478.57	100 μ A	5502.93
3 degrees	100 nA	5490.78	100 μ A	5501.04
6 degrees	100 nA	5455.97	100 μ A	5400.80
10 degrees	100 nA	5267.03	100 μ A	5336.04
20 degrees	100 nA	5265.23	100 μ A	5153.57
30 degrees	100 nA	5034.37	100 μ A	5038.89
60 degrees	100 nA	3295.08	100 μ A	4859.40

These data were collected on the program LabView v.8.5 (using a code which performs a van der Pauw algorithm) and formatted into graphs on the program KaleidaGraph 4.1 (table 2).

The temperature coefficient represents the deviation of the sheet resistance due to a change in temperature. This value is calculated by the following equation but then converted into percentages for the purposes of this study:

$$T_c = (R_s/R_s(T)) * 100\%. \quad (1)$$

Table 2. Temperature coefficient, from four-point measurement versus temperature.

	Temperature Coefficient
at 20 °C	~0.09%
at 30 °C	~0.1%
at 60 °C	~0.15%

3. Corbino Disc Microwave Measurements

Corbino discs are a supplemental test structure used for sheet resistance characterization. A single Corbino disc is composed of an inner conducting circular contact and an outer conducting ring contact. In this study, the space between the two was occupied by a CNT mesh network.

An unexpected occurrence was observed when the testing was first initiated. Due to the high temperatures (approaching 310 °C) of the data set, the titanium-nitride cap began to melt with the other layers and started to smear. It was decided that the sample integrity was comprised, and these resistance measurements were regarded as invalid.

As a replacement film, a gold cap was used for testing, rather than titanium nitride, and the CNTs were installed underneath the metallic layers. Room temperature was 21.1 °C, and the relative humidity was at 65%. The test temperatures ranged from 296 to 400 K.

For the DC sheet resistance measurement, the voltages were sourced from 0.4 V to –0.4 V and from –0.4 V back to 0.4 V. The reason for this selection in voltages is to determine if there were any detectable effects of hysteresis on the sweep downward. A DC current-versus-voltage measurement (taken at room temperature) of a Corbino disc with a diameter of 300 µm and a spacing of 45 µm is shown in figure 4. Resistance data were extracted from the slope of this curve, and the sheet resistance information was calculated by taking into consideration the geometric dimensions. Figure 5 shows a plot of sheet resistance values for Corbino discs of different diameters (d) and spacings (g). These measurements seemed to converge to approximately 800 ohms per square as a function of spacing.

The purpose of the microwave measurement was to gather further insight about how the thin films perform in a variety of communication scenarios. Similar temperatures were used when testing with microwaves (296 to 400 K). Microwaves ranged from 8 to 12 GHz (X band) and the signal power level was held constant at 1 mW. Figure 6 demonstrates the dependence of sheet resistance on microwave frequency. The temperature dependence of both the DC and microwave sheet resistance is shown in figure 7.

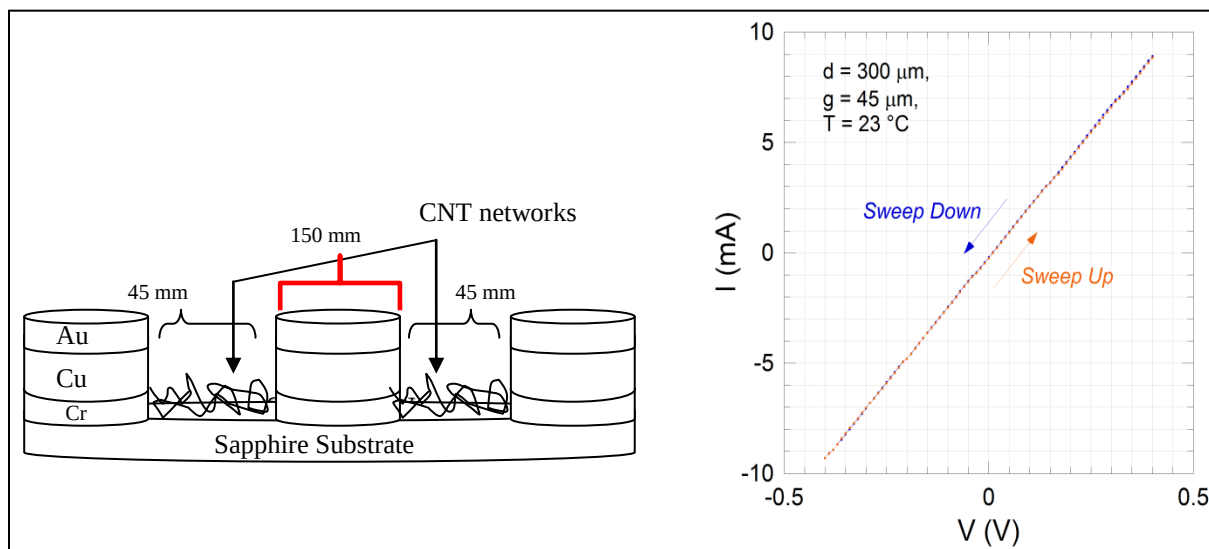


Figure 4. The graph shows there is no detectable effect of hysteresis on the downwards sweep and the contacts experienced very ohmic behavior.

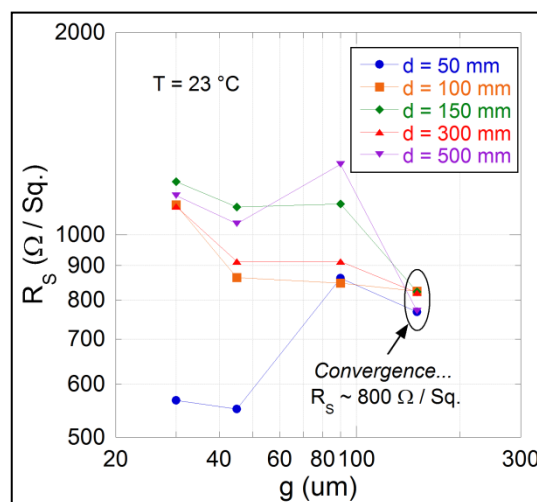


Figure 5. DC sheet resistance versus Corbino disc gap. The convergence of the different diameters at the sheet resistance of ~ 800 ohms/sq.

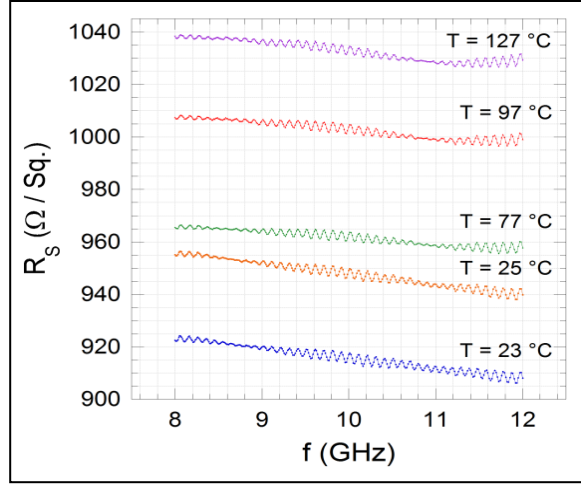


Figure 6. Microwave sheet resistance versus frequency.

From the cross-correlation values, we can assume there is some noticeable relationship between the DC and microwave (8 and 12 GHz) sheet resistance functions. The relationship is not absolute, but is noteworthy. This relationship reveals the amount of effect the microwaves have on the current (figure 7). This coefficient was calculated by the following equation (7):

$$(f \star g)[n] \stackrel{\text{def}}{=} \sum_{m=-\infty}^{\infty} f^*[m] g[n + m]. \quad (2)$$

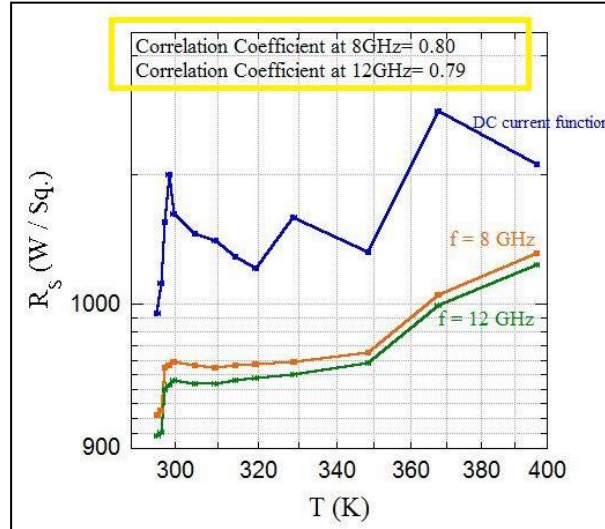


Figure 7. DC and microwave sheet resistance versus temperature.

4. Conclusion

In summary, the CNT thin films performed well in all of the conditions of this experiment. In the four-point measurement, there was some temperature-dependence when measuring sheet resistance, but the sample remained linear, displaying its stability and versatility in the presence of a range of currents. Although the Corbino disc two-point measurements were unpredictable, and at times incoherent, from those measurements, we were able to construct better contacts with the first two failures in mind. The obscured sheet resistances resulting from the first few sets of measurements could have been due to a native copper oxide that accumulated under the cap layer resulting in poor contact connection. The two-point tests actually yielded a positive temperature coefficient compared to the negative temperature coefficient of the four-point measurements. Most likely this discrepancy is a size effect resulting from heat-induced expansion of the CNTs. An additional microwave test was performed using 1 to 8 GHz (the L, S, and C bands) and similar behavior was observed, these data, however, were been omitted from this report due to time restraints. But the method of probing on-chip Corbino disc test structures placed in direct contact with pre-deposited CNT networks is an entirely new technique that can be credited to this study.

This body of work resulted from an eight-week summer internship sponsored by the George Washington University/Department of Defense (DoD) Science and Engineering Apprenticeship Program (SEAP).

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