High Frequency Characterization of Carbon Nanotube Films

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Abstract— Carbon nanotube films are characterized using Vector Network Analyzer (VNA) and Terahertz Time-Domain Spectroscopy (THz-TDS). Both transmission and reflection experiments are performed in order to measure the complex refractive index and the wave impedance. This method allows simultaneous extraction of both the permittivity and permeability without any assumptions. Experimental results are obtained from microwave to THz for various multi-walled and single-walled nanotube samples.

I. Introduction And Background

Carbon nanotubes (CNT) are rolled-up graphene sheets with hollow cylindrical structures. A carbon nanotube may be single-walled if the tube has only one layer, or multi-walled if the tube consists of more than one layer of graphene sheets. CNTs have intrigued many research interests and efforts since they were discovered and are considered as potential building blocks for nano-scale circuits in virtue of their unique mechanical and electrical properties [1]. Many microwave and THz applications have been suggested as well recently, including nano-antennas and nano-interconnects [2].

Although extensive studies have been done on CNTs at DC, low frequencies and optical frequencies, over the microwave and THz regimes, the electrical properties of CNTs have not yet been well studied. The most intuitive method to study the properties of carbon nanotubes is to characterize individual tubes. However, it is very difficult to conduct this kind of measurements due to nanotubes' high intrinsic impedance (~10 KΩ to MΩ), which is incompatible with typical 50Ω microwave testing systems. In addition, at microwave to THz frequencies, parasitics of testing structures often dominate over the intrinsic properties of the devices under test. An alternative approach is to characterize a large ensemble of nanotubes. When a high frequency signal impinges upon a nanotube ensemble, at the interface, part of the signal is reflected and part of it is transmitted. The magnitude and phase of the reflection and transmission depend on the sample's properties and can be measured and used to extract the complex permittivity and permeability of the sample.

In this work, vector network analyzer (VNA) and THz time-domain spectrometer (THz-TDS) have been utilized to characterize ensembles of MWNTs and SWNTs in thin film format from microwave [3] to THz [4].

II. Results

The samples used in our experiments are a sheet form of carbon nanotubes with a thickness of 88.9 μm (~3.5 mil), made by NanoLab Inc. Carbon nanotubes are first suspended in a fluid, then filtered onto a membrane with support underneath. After drying, the membrane is removed from the support, leaving a thin and free-standing nanotube film as shown in Figure 1(a). From the scanning electron microscopy (SEM) image of a nanotube film shown in Figure 1(b), it can be seen that the nanotubes are randomly entangled to form the film.

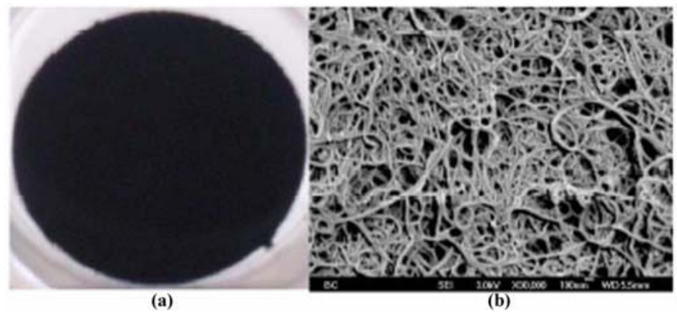

Figure 1. (a) Photo and (b) SEM image of the nanotube film.

At microwave frequency, we measure the S-parameters of a nanotube sheet sandwiched between two rectangular waveguides so that it is perpendicular to the direction of the electromagnetic wave propagation. The experiment setup is as shown in Figure 2. The responses of the sample are measured at X-band, Ku-band, Ka-band and Q-band, which requires four different waveguides WR-90, WR-62, WR-28 and WR-22 to be used respectively. The waveguides are connected to the two ports of an Agilent E8361A VNA. Before measurement, two-port waveguide calibration is performed using the Agilent XI 1644A cal kit so that the reference planes of the measured S-parameters are exactly located at the two surfaces of the MWNT paper under test.

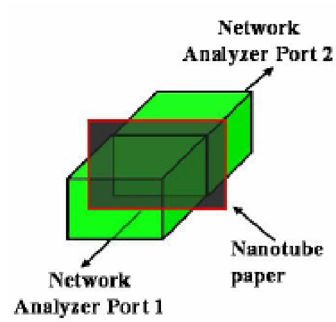

Figure 2. Carbon nanotube paper S-parameter measurement setup with VNA and rectangular waveguides.

For THz frequency, free space transmission and reflection coefficients of the sample are measured with setups shown in Figure 3 (a) and (b), respectively, using a photoconductive THz-TDS system from Advanced Photonix Inc. In the normal incidence transmission configuration, the calibrated complex transmission coefficient is obtained by dividing the sample

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transmission spectrum with a reference spectrum taken without the sample in the beam path (as a lossless through reference). In the reflection configuration, the reference spectrum is obtained by placing a shining metal plate at the same position with the sample (as a perfect reflector with a reflection coefficient of -1). The reflection spectrum of the MWNT film is then calibrated by comparing the reference and sample spectra. Since the THz emitter and detector of the TDS cannot be co-located, the reflection measurement has an oblique incidence with an incidence angle of 26°. Collimating mirrors are applied in both configurations to confine the beam waist completely within the sample cross section area. Multiple numbers of transmission and reflection measurements are performed and good repeatability of the measured spectra is observed.

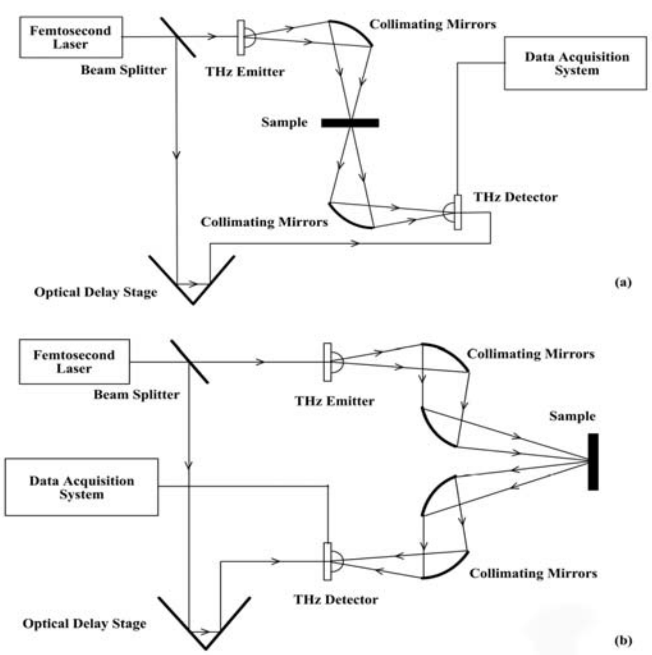

Figure 3. (a) THz-TDS transmission characterization setup; (b) THz-TDS reflection characterization setup. The incident E field is S-polarized in both measurements.

The complex refractive index n and wave impedance z are extracted by the Nicolson-Ross-Weir approach [5], from S-parameters obtained with VNA. For THz frequency, n and z are determined by getting a best simultaneous fitting of both the transmission and reflection coefficients to the measured values [4]. In the refractive index extraction, a phase wrapping (or ambiguity) of $2m\pi$ (m is any integer) will lead to different values of the real part of n. Multiple branches of possible real part of n solutions are obtained in our calculation due to this phase ambiguity. Because the MWNT films are very thin (88.9 μm), the $m = 0$ branch should be the physical solution. The complex permittivity and permeability of the sample throughout the frequency range are then calculated simultaneously without imposing the assumption of unity permeability, with the following equations:

$$\varepsilon = \frac{n}{z}, \quad \mu = n \cdot z \qquad (1).$$

Figure 4 plots the extracted μ', μ'', ε' and ε''. The measured μ' varies from -1 to 1.8 and μ'' varies from ~ 0 to 2.1, showing a weak magnetic response of the MWNT film. The measured ε' decreases with frequency from 650 to 20, and ε'' also decreases with frequency from 3400 to less than 10. Reasonable agreement between the microwave (8 to 50 GHz) and THz (50 to 370 GHz) results can be seen. A Drude-Lorentz model is applied to fit both the real and imaginary parts of the permittivity from 8 to 370 GHz (solid line in Figure 4 (c) and (d)). The measured material properties provide useful data for potential microwave and THz applications based on carbon nanotubes.

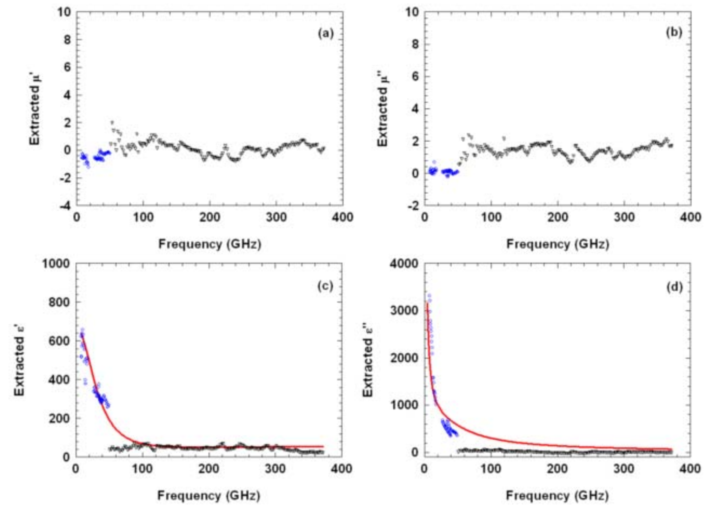

Figure 4. Extracted complex permittivity and permeability: (a) μ', (b) μ'', (c) ε', and (d) ε''. VNA (open diamonds, from 8 to 50 GHz) and THz-TDS (open triangles, from 50 to 370 GHz) results are plotted together. The extracted real and imaginary parts of the permittivity are also fitted by a Drude-Lorentz model (solid lines in (c) and (d)).

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