

Non-contact Conductance Measurement of Nanosize Objects using Resonant Cavity

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Abstract — A cavity perturbation method is used to determine conductance of small volume nano-carbon materials. These are the building blocks of nanostructured materials and devices, and therefore their electrical characteristics are important in the materials development and production. Non-contact measurement is performed in WR-90 waveguide configuration in the X-band frequency band from 6 GHz to 12 GHz. The presented semi-empirical perturbation model correlates the experimental quality factor and the specimen volume with the specimen imaginary part of permittivity and conductance. The measurement is illustrated on conducting carbon nanotube films having volumes in the range of 10^{-5} cm³ and conductivity of 50 S/cm.

Keywords— non contact measurement, resonant cavity, microwave conductivity, carbon nanotubes.

I. INTRODUCTION

A non-contact measurement technique that can characterize the nanoscale conducting reinforcement in composite materials is needed to allow for process and quality control during manufacturing. We have shown that the microwave conductance of thin film of nanoparticles can be determined from transmitted and reflected waves in a coplanar waveguide configuration [1, 2]. Such measurement techniques require, however, a complex test vehicle and interconnections for making electrical contacts to nano-size objects.

In comparison, resonant cavity measurements are fast and non-contact and are thus well suited for use in a manufacturing environment. The measurement involves monitoring the resonant frequency shift and change in the quality factor before and after insertion of the specimen into the cavity. [3]. The parameters of the cavity depend on the volume, geometry, mode of operation, shape, dimensions and location of the object inside the cavity [4]. The field perturbation relations are simple when the electromagnetic field distribution can be described by first order approximation. Therefore, the resonant perturbation methods have been widely used in the characterization of low loss dielectric materials, which cause a small perturbation to the electromagnetic field distribution inside the cavity. Materials with higher conducting losses cause a large field perturbation, which may lead to considerable errors if the linear perturbation technique is

used [5]. Our proposed solution is to include the higher order perturbation modes through semi-empirical correcting factors and use specimens having a small volume compared to the volume of the cavity.

In this presentation we examine the applicability a cavity perturbation method for non-contact microwave conductance measurements of small volume nano-carbon materials, for which we have established suitable correlations between the microwave data and the material characteristics. We illustrate the measurement results on small bundles of conducting carbon nanotube films using a WR-90 waveguide cavity operating in the X-band frequency band.

II. EXPERIMENTAL PROCEDURE AND METHODS

Our prototype design shown in figure 1 employs a 134 mm long WR-90 waveguide operating in the microwave X-band, 6 GHz to 12 GHz. The walls of the cavity are implemented via WR-90 couplers, which are cross-polarized ($\theta = 87.75^\circ$) in respect to the waveguide polarization. In comparison to

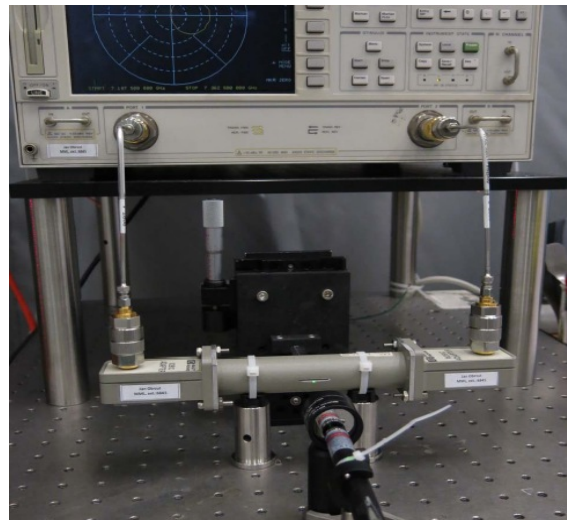


Figure 1. Resonant cavity for non contact electrical measurement of nanosize objects.

standard end-metal plates (ASTM D-2520), our cavity arrangement with cross-polarized couplers minimizes

conducting losses, improving the quality factor (Q) of the resonator and thus considerably improving the accuracy of the method.

The specimen is inserted into the cavity through a narrow slot (1mm x 19 mm) precisely machined through both walls of the waveguide in the center of the cavity, where the electric field attains a maximum value. The specimen position is controlled by a motorized stage and guided by a laser. In Fig. 1 the motorized stage with the specimen mounted horizontally is located behind the waveguide, while the laser guide is assembled at the front. We measured thin films of doped multi-walled carbon nanotubes (MWCNT) [6], typically 0.5 μm thick and 500 μm wide, which were deposited on a polyterephthalate (PET) substrate. The length of the MWCNT film specimen inserted into the cavity is controlled by the stage, which allows us to change the MWCNT volume from (3 to 30) $\times 10^{-6} \text{ cm}^3$.

Measurements of the S_{21} parameter were performed using an HP 8720D network analyzer connected to the cavity test structure with semi-rigid coaxial cables and coaxial to WR-90 adapters. The system was calibrated at the coaxial ends using a Short-Open-Load calibration kit. The resonant frequency f_c and half power bandwidth Δf is determined for each $\text{TE}_{103} - \text{TE}_{109}$ resonant modes, from S_{21} peaks which we can detect in our cavity between 6 GHz and 12 GHz. The quality factor is calculated as $Q = f_c / \Delta f$ [4].

Cavity perturbation model

The classical treatment of a cavity resonator involves solving Maxwell's equations for resonance frequencies with the appropriate cavity boundary conditions [7]. For the case of small perturbation, a simplified solution for the quality factor [8-10] is given by equations (1):

$$\frac{1}{Q_s} - \frac{1}{Q_0} = C_v \varepsilon'' \quad (1a)$$

$$C_v = \frac{\int_{V_s} E_s E_0 dV}{\int_{V_c} E_0^2 dV} \quad (1b)$$

where, C_v is the relative field distribution in the specimen, $\varepsilon'' = \sigma/\varepsilon_0\omega$ is the imaginary part of permittivity, σ is the sample conductivity, ε_0 is the permittivity of free space, $\omega = 2\pi f$, E_0 and E_s denote the electric fields in the cavity and in the specimen, and Q_0 and Q_s are the quality factors of the empty cavity and cavity with the sample respectively. Here the volume of the empty cavity, $V_c = 31.122 \text{ cm}^3$, and V_s is the volume of the sample. When a low loss dielectric sample is located at the electric field maximum, (1b) simplifies to $C_v = 4V_s/V_c$ [9]. In the case of conducting samples and stronger perturbation, the quality factor is no longer a simple linear function of V_s [10, 11]; Equation (1b) has no simple solution for C_v even if the sample volume is small [10].

A semi-empirical approach can be a useful alternative to (1b). We examine a semi-empirical extension of equations (1), where the nonlinear character of the higher perturbation order is approximated by the exponent n :

$$\frac{1}{Q_s} - \frac{1}{Q_0} = \left(C_v \frac{\sigma_s}{\varepsilon_0\omega} \frac{V_s}{V_c} \right)^n \quad (2a)$$

On the log-log scaled plane (2a) represents a straight line:

$$\log\left(\frac{1}{Q_s} - \frac{1}{Q_0}\right) = n\log(C_v) + n\log\left(\frac{\sigma_s}{\varepsilon_0\omega}\right) + n\log\left(\frac{V_s}{V_c}\right) \quad (2b)$$

with the slope of n . The intercept $A = n\log(C_v) + n\log(\sigma_s/\varepsilon_0\omega)$ depends on C_v , which in (2b) represents an adjustable parameter. The second parameter in A is the specimen conductivity σ_s , which we generally assume to be frequency independent in the frequency range where Q_s is measured. The validity of the perturbation range for (1b) can be evaluated by inspecting the uncertainty of fitting (2b) to experimental data for a specimen with known electrical conductivity.

III. RESULTS AND DISCUSSION

Figure 2 illustrates example measurements of the magnitude of the scattering parameters S_{21} , showing a peak value at the TE_{103} resonant frequency for a 0.5 μm thick film composed of MWCNT [6]. The height of the resonant peak decreases significantly and its position shifts to lower

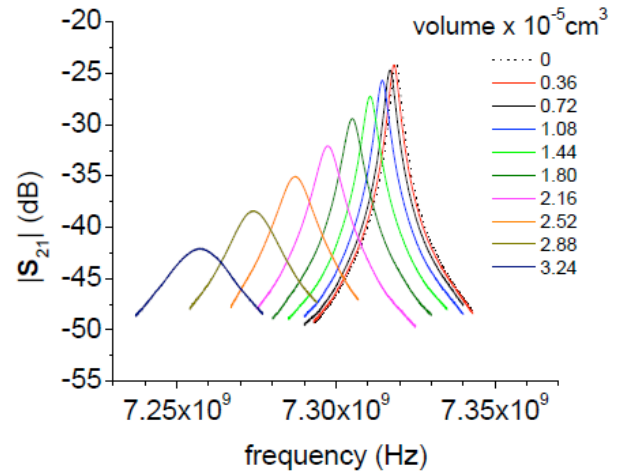


Figure 2. Resonant peak TE_{103} as a function of MWCNT volume in the cavity. The ac conductivity of the MWCNT sample measured at 1 MHz is 50 S/cm.

frequency when the volume of the MWCNTs in the cavity increases. In Fig. 2 the specimen volume increases in steps of $0.36 \times 10^{-5} \text{ cm}^3$, from zero for an empty cavity to $3.24 \times 10^{-5} \text{ cm}^3$ for our largest sample. The corresponding Q

factor decreases from $Q_0 = 2600$ to $Q_s = 341$ when the cavity is loaded with MWCNT sample having volume of $3.24 \times 10^{-5} \text{ cm}^3$.

In order to examine the applicability of the semi-empirical perturbation equation (2), we plotted in Figure 3 the experimental quality factor against the relative specimen volume using double log-log scales. The solid line in Fig. 3 represents a linear fit to the first five resonant data points, from which we obtain $n = 2.22$ and $\sigma_s = 45 \text{ S/cm}$ with the relative standard uncertainty of 2 %. The dash line in Fig. 3 represent a linear fit through all the points from which the average exponent $n_{av} = 2.32$ and average $\sigma_{s-av} = 55 \text{ S/cm}$ with the relative standard uncertainty of 5%. We consider both of these results to be in good agreement with the ac conductivity values

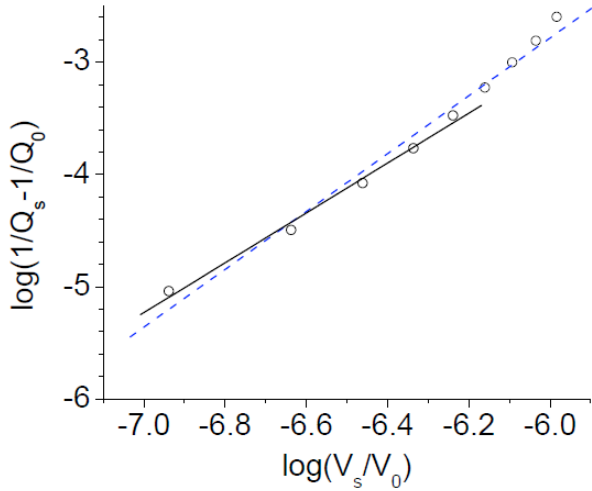


Figure. 3. Plot of equation (2a) using experimental Q_0 and Q_s data and the corresponding specimen volume sample volume in the resonant cavity. The solid line is a linear fit to the first five resonant data points while the dash line represents a linear fit to the all resonant data points, shown in Fig.2.

determined from impedance of electroded MWCNTs specimens at 1 MHz, $\sigma_{ac} = 50.2 \text{ S/cm}$. Taking σ_{ac} as a nominal reference, the cavity resonance conductivity results are within 10 % of that reference. A clear departure from linearity in Fig. 3 is seen when $\log(1/Q_s - 1/Q_0) > -3$ indicating that equations (2a) and (2b) become invalid when $Q_s < 1000$.

IV. CONCLUSION

We employ a semi-empirical approach to the cavity perturbation method to determine conductance of small volume nano-carbon materials. These are the building blocks

of nanostructured materials and devices, and therefore their electrical characteristics are important in the materials development and production. The proposed formula (2b) correlates the quality factor of the loaded cavity with the specimen conductivity and extends the applicability of the conventional perturbation method to a non-linear higher perturbation range. The experimental results are illustrated on thin films of conducting MWCNT specimens having volumes in the range of $0.3 \times 10^{-5} \text{ cm}^3$ to $2.5 \times 10^{-5} \text{ cm}^3$ and the volume conductivity of 50 S/cm. Our results indicate that the presented non contact cavity perturbation methodology is fast and accurate with high sensitivity that is unobtainable by other techniques.

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