Determining Carbon Fiber Composite Loading with Flip-Chip Measurements to 110 GHz

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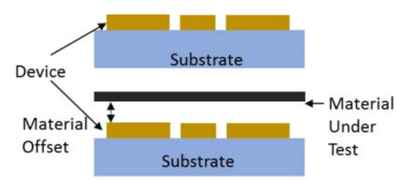
Abstract — Precise knowledge of electrical properties of materials are a necessary input to any circuit design. As applications at millimeter-wave frequencies increase, there is a need to develop new materials with low loss and multiple functionalities. Most material characterization techniques are destructive to the original sample. For example, most resonant cavity techniques require a sample to be diced into a sub-millimeter geometry. Alternatively, broadband on-wafer techniques require lithographically patterned devices on the material under test. Here, we demonstrate a technique that combines on-wafer calibration with a flip-chip transmission-line test fixture. This single-transmission-line fixture enables quantitative measurement of effective permittivity from 40 kHz to 110 GHz. The resulting materials characterization approach is non-destructive and directly applicable to measurements of dielectric, magnetic, and nonlinear properties. The broad applicability of the technique makes it well-suited for characterizing the next generation of materials, including tunable materials and complex structural composites.

***Keywords* — millimeter waves, calibration, flip-chip, coplanar waveguide, dielectric spectroscopy, composites**

I. Introduction

Both electrical and mechanical material properties are important for the design of electronic devices. In order to optimize these properties, multifunctional materials often require very specific micro- and nanostructures. For example, a multifunctional composite containing conducting fibers might provide both mechanical strength and electrical shielding. Understanding the mechanical and electrical properties and their relationships is paramount for identifying the possible applications for materials. The goal of this paper is to show a relationship between the measured electrical properties (distributed circuit parameters) and the physical properties (mass fraction) of a composite carbon fiber material. The data in this paper is the first step in understanding this relationship, however there are still many questions to answer and more measurements to be taken.

Unlike typical characterization techniques, the flip-chip technique discussed in this paper provides broadband, non-destructive, and quantitative measurements of distributed circuit parameters of a coplanar waveguide loaded with a material. The new method leverages a combination of broadband measurement techniques [1], [2] and low-frequency electrical property analysis to discern electrical characteristics [3]. As an example, we explore the range of dielectric properties and frequency dependence of a multi-functional material consisting of carbon-fibers embedded in a polymer matrix. As a structural material, carbon fiber serves as a mechanical reinforcement that finds applications ranging from sporting goods to aerospace and reducing the weight of automobiles. As an electrical material, it has been employed for both electrostatic discharge protection and suppression of electromagnetic interference [4].



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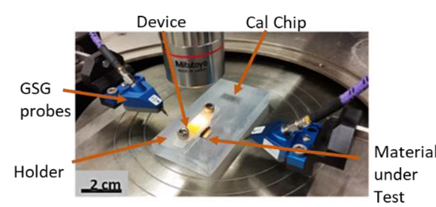

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Fig. 1. The flip-chip transmission line for materials characterization from 40 kHz to 110 GHz. (a) A top view schematic of the coplanar waveguide device. (b) The cross-section of the two topologies needed to extract material properties. The chip is 500 µm thick fused silica with relative permittivity of 3.8, and the material offset is 100 µm.

The single-line flip-chip technique (Fig. 1), is inspired by on-wafer material characterization techniques designed for thin-film dielectric materials [2]. These techniques are based on the multiline thru-reflect-line (multiline TRL) calibration [5] in a coplanar waveguide topology. Broadly speaking, the previous work employs a two-tiered measurement procedure: first, a rigorous calibration sets the reference impedance and translates the measurement reference plane onto a test wafer; second, a set of transmission lines loaded by a material under test is characterized with multiline TRL and the properties of the material under test are extracted.

In this work, we follow a similar two-tiered approach, though in lieu of the more conventional multiline TRL technique in the second tier, we used a single coplanar waveguide device (Fig. 1a). We measure this single waveguide both with and without a material under test (Fig. 1b), and extract distributed circuit parameters.

The coplanar waveguide (CPW) device has electric (**E**) and magnetic (**H**) fields that penetrate above and below the metallization of the coplanar waveguide. These fields allow the waveguide to interrogate the electromagnetic properties of a material placed near the waveguide. To control the coupling between the fields and material under test, the measurements require a holder to elevate the sample a precisely-known distance above the waveguide (Fig. 1b).

From the single-line measurements, the distributed circuit parameters of the waveguide were extracted. While we expect that this single-line technique will not be as accurate as a multiline technique, it has two key advantages. First, the test fixture can be reused many times for different samples, and second, it does not require lithographic processing of each sample. This latter point is especially valuable for samples that are too rough or fragile for coplanar waveguides to be directly printed on the material's surface. These advantages open the door to fast, inexpensive, broadband material characterization.

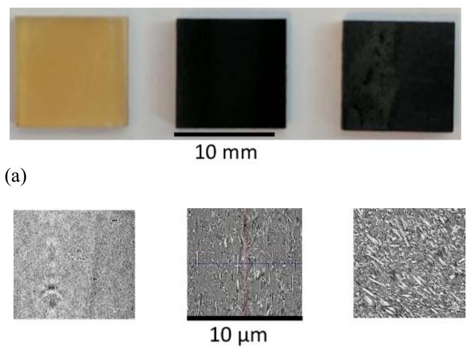
In what follows, we first provide a description of the methodology behind the materials characterization technique. Next, we discuss the algorithm implemented to extract distributed circuit parameters of an unloaded and loaded transmission line. In Section IV, we analyze measured data and correlations between the electrical and physical parameters of the material.

II. Description of Methodology

As an example of a multifunctional material, we chose to study a carbon fiber composite. Neat carbon fibers (Mitsubishi, DIALEAD K223HE[1,2]) were first ball milled, then combined with a polyetherimide polymer matrix (Sabic, ULTEM 1010) in a twin-screw extruder. The milled fibers up were up to 60 μm long, and had a nominal diameter of 5 μm. After mixing, the samples were injection molded into a rectangular blank, then diced to dimensions of approximately 10 mm × 10 mm × 2 mm. Here, we study samples with carbon fiber mass fractions of approximately 0.0 %, 8.4 %, and 27.3 % (Fig. 2), as defined by a precision scale.

The microstructure of the samples was characterized by X-ray computed tomography (CT) (Fig. 2b), which creates a 3-dimensional (3D) image of each sample. We qualitatively analyzed the 3D images, showing that the fibers were oriented in many different directions and appeared to be uniformly distributed throughout the polymer matrix. Based on these qualitative results, we treat the materials as an effective medium with an isotropic effective permittivity.

The surface roughness of the samples, as found with the X-ray CT analysis, is on the order of a few hundred nanometers and must be considered in the measurement setup. To minimize the effect of the surface roughness of the samples on electrical measurements, the material is separated from the surface of the coplanar waveguide with a sample holder (Fig. 3). The separation between the waveguide surface and the sample surface is chosen to be large compared to the sample's surface roughness, but small enough that the fields still interact with the sample. In this work, the gap width of the coplanar waveguide was 50 μm, and the sample was elevated 100 μm (Fig. 3b) above the device. Finite element simulations using ANSYS Q3D were used to determine the maximal distance between the coplanar waveguide and sample (Fig. 3c). Since the sample thickness (about 2 mm) is much larger than the gap width of the coplanar waveguide, it appears infinite and does not need to be simulated. It is clear from the simulation that a substantial amount of the incident wave propagates through the sample material. With the physical setup (Fig. 3), we can now take measurements to determine the electrical properties of the composite.



(b)

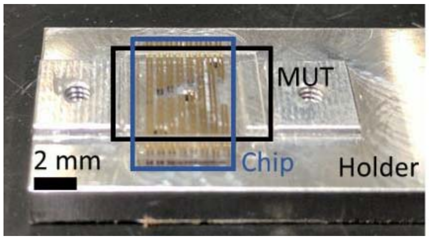
Fig. 2. Samples of milled carbon fiber in a polymer matrix with mass fractions of 0.0 %, 8.4 %, and 27.3 % as seen in (a) photograph and (b) X-ray CT.

To test the electrical properties of the composite, we measured the scattering-parameters (S-parameters) of coplanar waveguide devices with a vector network analyzer (VNA). The first-tier calibration was based on multiline TRL, as described elsewhere [2]. The first-tier calibration was used to solve for the error boxes associated with the VNA, cables, and probes. We also use it to solve for the characteristic impedance (Z) and propagation constant (γ) of the waveguide without loading by the material under test. Because these complex quantities are used for the second-tier calibration and they are related to the geometry of the CPW, the first- and second-tier CPWs must have the same geometry. The CPW has a nominal center conductor width of 20 μm, a gap width of 50 μm, a ground plane width of 200 μm, and a conductor thickness of 0.5 μm. We fabricated all devices on the same wafer using an in-house cleanroom and conventional i-line stepper lithography. The

1 Mention of commercial products in this manuscript is for information purposes only; it does not imply recommendation or endorsement by NIST.

CPW lines were made by electron-beam vapor deposition, depositing 0.5 μm thick gold with a 10 nm Ti adhesion layer on a fused silica substrate. For more details about the fabrication, see [6].

In the next section, we discuss how we measure the per-unit-length quantities of series resistance (*R*), series inductance (*L*), shunt conductance (*G*), and shunt capacitance (*C*).



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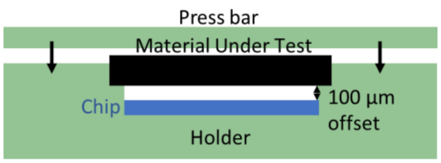

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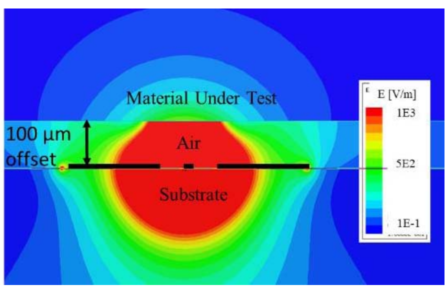


(c)

Fig. 3. (a) A photograph of the chip and a fused silica MUT on the holder (press bar not pictured). (b) A not-to-scale schematic of the cross-section of the holder, that elevates the sample 100 μm away from the surface of the chip. (c) A finite element simulation illustrating the change in **E**-field magnitude used to determine the offset. The black bars are a not-to-scale depiction of the coplanar waveguide.

III. Extracting Distributed Circuit Parameters

To extract the distributed circuit parameters (*R, L, G*, and *C*) of the material-loaded line, we modeled the S-parameters using a T-matrix formalism. In this case, the model for a uniform, non-loaded transmission line in a 50 Ω system is

$$\boldsymbol{T} = Q_Z^{50}\begin{pmatrix} e^{-\gamma\ell} & 0 \\ 0 & e^{\gamma\ell} \end{pmatrix} Q_{50}^{Z}, \tag{1}$$

and the model for a uniform, loaded transmission line in a 50 Ω system is given by:

$$T = Q_Z^{50}\begin{pmatrix} e^{-\gamma\ell} & 0 \\ 0 & e^{\gamma\ell} \end{pmatrix} Q_{Z_l}^{Z}\begin{pmatrix} e^{-\gamma_l l_l} & 0 \\ 0 & e^{\gamma_l l_l} \end{pmatrix} Q_Z^{Z_l}\begin{pmatrix} e^{-\gamma\ell} & 0 \\ 0 & e^{\gamma\ell} \end{pmatrix} Q_{50}^{Z}, \tag{2}$$

where the impedance transformer Q between any impedances $\mathrm{Z_n}$ and $\mathrm{Z_m}$ is given by

$$\boldsymbol{Q}_{\boldsymbol{Z_m}}^{\boldsymbol{Z_n}} = \frac{1}{2\sqrt{Z_n \cdot Z_m}}\begin{pmatrix} Z_m + Z_n & Z_m - Z_n \\ Z_m - Z_n & Z_m + Z_n \end{pmatrix}, \tag{3}$$

where γ is the complex propagation constant, Z is the characteristic impedance, ℓ is the length of the line, and the ℓ subscript indicates parameters of the loaded transmission line. The standard definitions of γ and Z are used here:

$$\gamma \equiv \sqrt{(R + j\omega L)(G + j\omega C)}, \text{ and} \tag{4}$$

$$Z \equiv \sqrt{\frac{(R+j\omega L)}{(G+j\omega C)}}. \tag{5}$$

We note that expressions (1), (2), and (3) make several assumptions. Specifically, they assume that the system is linear, that there is only a single quasi-TEM mode of propagation in the coplanar waveguide, and that the interface between the unloaded segment of the waveguide and the material-loaded segment of the line can be modeled as an impedance transformer. These assumptions are discussed in the next section in the context of experimental data.

The distributed circuit parameters for each sample are extracted by a non-linear least-squares fitting algorithm that minimizes the error between the theoretical S-parameters expressions and the measured S-parameters [7], [8]. We tested our approach on three samples of milled carbon fiber in a polymer matrix (Fig. 2). These results are discussed in the following section.

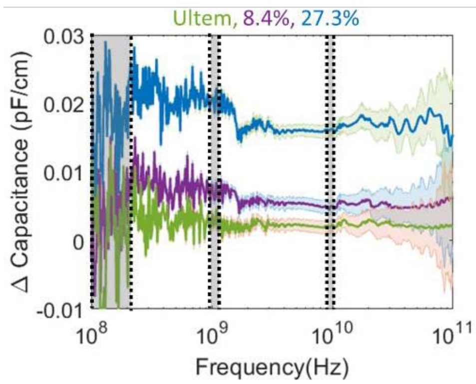
IV. Results

After extracting the distributed circuit parameters for the three samples shown in Fig. 2, we analyzed the data to identify how these parameters change in the presence of the material-under-test. Although not shown here, *R* and *L* were unaffected by our material-under-test. Therefore, we hold these parameters fixed as frequency-dependent values obtained after analyzing the CPW without the material-under-test. Holding *R* and *L* to these values allowed for a more robust extraction of the values of *C* and *G*. We computed the difference between *C* and *G* with and without the material-under-test (Fig. 4), computing *ΔC* as a function of frequency.

At this point, we can do a short analysis of the validity of the single quasi-TEM mode of propagation assumption made earlier. We expect the single quasi-TEM approximation to be valid as long as the guided wavelength is small compared to the characteristic lengths of the waveguide [9]. This assumption breaks down at some frequency and is quantified as

$$\frac{\lambda_0}{10\sqrt{\varepsilon_r}} = w + 2s, \tag{6}$$

where relative permittivity is 2.5, w is the width of the center conductor, and s is the gap width between center conductor and ground plane. From this analysis, we estimated that the assumption is not valid starting at $f = 158$ GHz, well above the measurements presented here.



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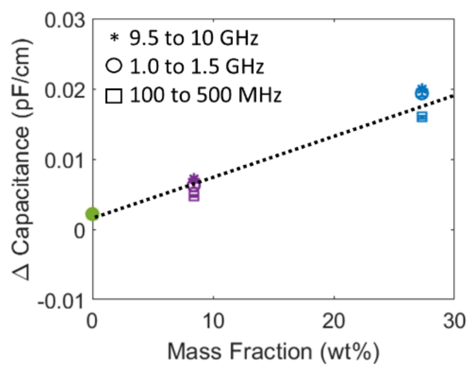

(b)

Fig. 4. Change in capacitance with uncertainty of three samples as a function of (a) frequency plotted from 100 MHz to 100 GHz and (b) sample mass fraction at frequency ranges of 100 MHz to 500 MHz, 1.0 GHz to 1.5 GHz, and 9.5 GHz to 10 GHz. The standard uncertainty for the frequency sweep is propagated through the analysis and added in quadrature for the normalization. The uncertainty for frequency slices is a single standard deviation in the mean. Where the error bars are not visible, they are smaller than the plot marker.

Inside the frequency range where a single quasi-TEM mode propagates, ΔC did not change significantly as a function of frequency (Fig. 4a). However, ΔC shows a systematic increase as a function of fiber mass fraction (Fig. 4b). This data means that as the mass fraction increased, the dielectric constant (real part of the permittivity) also changed. Interestingly, by taking the mean over frequency slices at 100 MHz to 500 MHz, 1.0 GHz to 1.5 GHz, and 9.5 GHz to 10 GHz, a roughly linear dependence of ΔC on mass fraction is determined. These points were chosen to highlight the extremes of the frequency spectrum; other points show a similar data point spread.

Future measurements and simulations are underway to help us understand how the change in capacitance should depend on the presence of the material-under-test. The relative change in capacitance related to the mass fraction is consistent with what was expected, as the capacitance change for the 8.4 % sample was roughly a third of the change for the 27 % sample.

V. Conclusions

In this paper, we demonstrated a flip-chip approach for determining the effect on electrical properties based on changes in physical properties. Specifically, carbon fiber composite material loading of a coplanar waveguide from 100 MHz to 110 GHz. The distributed circuit parameters were correlated with the carbon fiber mass fraction. For future works, the change in extracted capacitance and conductance per-unit-length can be mapped to electrical properties (e.g., permittivity, permeability, conductivity) via finite element simulations (FEM) of the cross-sectional geometry. More generally, our approach demonstrates a quantitative nondestructive materials characterization that can be applied to material systems beyond those explored here.

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References

[1] N. Orloff, J. Mateu, M. Murakami, I. Takeuchi, and J. C. Booth, “Broadband characterization of multilayer dielectric thin-films,” in *2007 IEEE/MTT-S International Microwave Symposium*, 2007, pp. 1177–1180.

[2] C.-H. Lee *et al.*, “Exploiting dimensionality and defect mitigation to create tunable microwave dielectrics,” *Nature*, vol. 502, no. 7472, pp. 532–536, Oct. 2013.

[3] J. M. Torrents, T. O. Mason, and E. J. Garboczi, “Impedance spectra of fiber-reinforced cement-based composites: a modeling approach,” *Cem. Concr. Res.*, vol. 30, no. 4, pp. 585–592, Apr. 2000.

[4] D. D. L. Chung, “Electromagnetic interference shielding effectiveness of carbon materials,” *Carbon*, vol. 39, no. 2, pp. 279–285, Feb. 2001.

[5] R. B. Marks, “A multiline method of network analyzer calibration,” *IEEE Trans. Microw. Theory Tech.*, vol. 39, no. 7, pp. 1205–1215, Jul. 1991.

[6] C. A. E. Little, N. D. Orloff, I. E. Hanemann, C. J. Long, V. M. Bright, and J. C. Booth, “Modeling electrical double-layer effects for microfluidic impedance spectroscopy from 100 kHz to 110 GHz,” *Lab. Chip*, vol. 17, no. 15, pp. 2674–2681, Jul. 2017.

[7] W. R. Eisenstadt and Y. Eo, “S-parameter-based IC interconnect transmission line characterization,” *IEEE Trans. Compon. Hybrids Manuf. Technol.*, vol. 15, no. 4, pp. 483–490, Aug. 1992.

[8] F. Mirri *et al.*, “Lightweight, Flexible, High-Performance Carbon Nanotube Cables Made by Scalable Flow Coating,” *ACS Appl. Mater. Interfaces*, vol. 8, no. 7, pp. 4903–4910, Feb. 2016.

[9] W. Heinrich, “Quasi-TEM description of MMIC coplanar lines including conductor-loss effects,” *IEEE Trans. Microw. Theory Tech.*, vol. 41, no. 1, pp. 45–52, Jan. 1993.