# A Fast Modelling-Based Technique for the Characterization of Graphene-Based Polymer Composites

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## ABSTRACT

**This work is dedicated to a non-destructive technique that allows modeling-based extraction of the surface resistance, resistivity and conductivity maps of graphene-based polymer composites. The technique is based on the use of a 10 GHz inverted Single-Post Dielectric Resonator (iSiPDR) incorporated into a 2D scanner. Retro-modeling is then applied to convert the measured microwave signal parameters to material characteristics. Retro-modeling requires the actual modeling to be performed may times, over a grid of parameters (material parameters and sample dimensions), and therefore, an ultra-fast conformal FDTD Bodies-of-Revolution (BoR) formulation of the FDTD method is utilized here. The maps of material parameters allow non-destructive testing of the developed composites and will be used for their assessment for different applications, such as electromagnetic shielding.**

***Index Terms* — graphene nanoplatelets, polymer composites, EMI shielding, material measurements, resonant method, non-destructive testing, 2D imaging, CEM, retro-modeling, FDTD, BoR FDTD**

## I. INTRODUCTION

New functional materials are key enablers for today's industries. This is recognized by e.g. many European research programmes, where the focus is made on materials' developments as well as characterization, from the viewpoint of target applications and technological needs. Due to the proliferation of the materials' research and the use of new materials in various industry segments, of special interest are low-cost, easy-to-use, and non-destructive characterization techniques. It has been demonstrated that microwave resonator techniques meet such requirements [1-5], provided however that the actually measured microwave signals are converted to the material parameters of interest to the user, in a way that remains "transparent" to the user, i.e., such that in-depth knowledge of the underlying microwave technologies is not required on the user's side. Such a seamless integration of microwave measurements with materials' science is achieved by computational modeling.

Our work is concerned with the application of the dielectric resonator method to the characterization of graphene-based polymer composites. In Section II, the basis of the technique are summarized, on the microwave measurement and computation modeling sides. Section II presents our initial results and Section IV – the summary and planned worked.

## II. ISIPDR MEASUREMENT OF GRAPHENE-BASED POLYMER COMPOSITES AND RETRO-MODELING

Graphene based polymer composites are conductive at microwave frequencies. Our interest is in measuring absolute values of resistivity or surface resistance of a composite sample, but also on constructing 2D maps of such quantities, for evaluating sample homogeneities and detecting fabrication defects. As such, we shall build upon the past works on microwave dielectric resonators applicable to conductive bulk materials and films, and those surface imaging. In particular, the theoretical bases for measuring conductive materials with Single-Post Dielectric Resonators (SiPDR) have been described in [1][2]. The concept of surface scanning with dielectric resonators has been introduced in [3]. The inverted SiPDR (iSiPDR) of nominal frequency 10GHz and suitable for incorporation into a 2D scanner has been presented in [4]. A portable device for microwave signal generation and scalar measurements of transmission through a resonator is presented in [5][6].

When a conductive sample is inserted into an iSiPDR, the resonant frequency and quality factor (Q-factor) of the resonator change. The measurement conditions are different for thin samples (characterized by surface resistance), such as epitaxial layers or graphene, and for bulk samples (characterized by resistivity), whose thickness is of the same order or greater than the skin depth. In both cases, the total Q-factor that is measured depends on the losses in the sample and the losses in the remaining part of the cavity (and the substrate, in the case of thin sheets deposited on a substrate). Thus, to determine either resistivity or surface resistance, two measurements must be made. In the first one, the iSiPDR is measured without a sample. Then the sample is inserted into the scanner and Master Unit Control Application [4] initiates scanning the surface of the composite with a given spatial resolution. At each position over the sample, a dedicated Q-Meter [5][6] performs a point-wise measurement - note that "point-wise" means a measurement that reflects the material parameters averaged over the resonator head [3]. The scanning process produces a family of resonant curves, from which the values of the resonance frequency and Q-factors are extracted. At the final step of the procedure these are converted to the scanned material parameters by the so-called retro-modeling.

Our conference talk will focus on the challenges that have to be overcome in the retro-modelling approach. Essentially, retro-modelling means matching the measured microwave response (of the resonator loaded with an unknown material) to the modeled response (of the resonator loaded with some assumed material). This requires performing many electromagnetic simulations of the iSiPDR loaded with different assumed materials. Therefore, the simulations need to be fast and augmented with signal-processing techniques for accurate extraction of resonant frequencies and Q-factors. Our method comprises:

- using an ultra-fast conformal BoR FDTD algorithm for the modeling of empty and loaded iSiPDR, in a grid-search regime [7],
- applying an advanced Prony method [8] with automatic selection of signal post-processing parameters for the extraction of the poles of the simulated microwave signals.

## III. CHARACTERIZATION AND RETRO-MODELLING RESULTS

The technique has been applied to a set of samples of graphene-based polymers such as those in [9]. Commercially available acrylonitrile butadiene styrene (ABS) and graphene nanoplatelets (GNP) with an average lateral dimension of 25 µm and a surface area of 120–150 $m^2/g$ were mixed using a Turbula T2F mixer, where the filler content reaches 10wt%. Then the mixture of powders was pressed on a hydraulic press using the hot press method, where the material was subjected to the appropriate pressure and temperature. In this way, samples of the nanocomposite were obtained for further research

The scanning setup based on the 10 GHz iSiPDR head is shown in Fig.1a, and an example ABS/GNP sample – in Fig.1b. The scan was taken over an area of 100 mm x 100 mm with a step of 2 mm. Figure 2 shows a map of Q-factors of the iSiPDR placed over a grid of points over the sample and Fig. 3 a map of resistivity values extracted from the Q-factor map (by modeling-based correlation to the Q-factor of the empty iSiPDR).

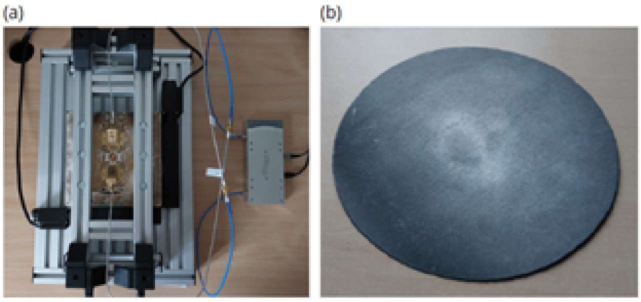

Fig. 1. (a) Measurement setup for 2D imaging of graphene-based polymer composites [4], (b) acrylonitrile–butadiene–styrene with graphene nanoplatelet (ABS/GNP) [8].

Note that the conversion from Q-factors to resistivities is obtained assuming the average thickness of the sample, 429.9 µm. Red areas in Fig.2 and Fig.3 are those within the 100mm x 100mm scanning rectangle, but beyond the circular area of the sample. Over the sample (bluish circular area in Fig.2 and Fig.3), the extracted resistivity values are in the range from 0.849 to 21.79 [Ωcm], implying substantial non-uniformities of the sample. It should be noted however that these non-uniformities result from two factors: the actual inhomogeneity of the applied graphene-based polymer powders and non-uniformity of the deposited material thickness. Internal stresses cause the sample to deform over time so that it begins to float above the substrate in some places. In areas where the sample is thicker, a bigger volume of the material interacts with the iSiPDR, which is therefore more detuned from its unloaded state, which is in turn interpreted as higher material losses.

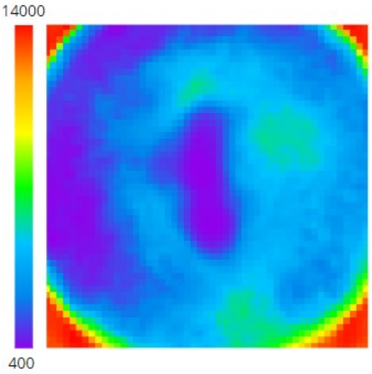

Fig. 2.2D maps of Q-factors of iSiPDR scanned over the ABS/GNP sample.

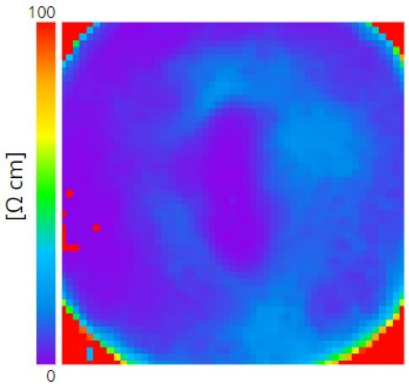

Fig. 3. 2D maps of resistivity of ABS/GNP sample for iSiPDR scanning.

The Q-Factor and resistivity maps are based on the scattering matrix transmission parameters |S21| obtained at each position of the iSiPDR head in the scanner. After each step, the resonance curve is analyzed (Fig. 4), its maximum indicating the resonant frequency (of the iSiPDR loaded with the material currently within its head), and 3dB bandwidth indicating the losses (of the iSiPDR head loaded with the materials). For reference, a single measurement is first taken of the empty iSiPDR. Its frequency was 10.1635 GHz and its Q-factor 13948.868. Inserting a sample shifts the resonant frequency (in Fig.4 - by 0.0026 GHz toward lower frequencies) and decreases the Q-factor (as seen by the broadening of the resonance curve). For fast and accurate extraction of resonant frequencies and Q-factors at many scanning steps, the measured transmission curves are considered only in a limited frequency range close to the resonance, so that the extraction not burden the speed of data acquisition from the scanner and their post- processing.

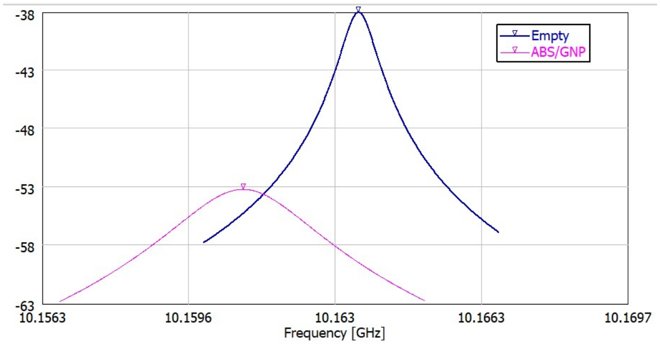

Fig. 4. Transmission (abs (S21)) through the 10 GHz iSiPDR mounted in the scanner and placed at two selected positions: over an empty region (blue) and at a selected point over the ABS/GNP sample (pink).

Figure 5 shows a set of, individual measurements obtained from the scanner, with the iSiPDR head passing through the center of the sample, from one sample extreme edge to the other. Since one step is 2 mm, in one scanning line 51 curves are obtained. In order to avoid overlapping curves only 25 of them were selected and shown in Fig. 5. After analyzing all the results, it can be seen that the resonant frequency varies up to 10 MHz up and down, for consecutive steps. At the very center of the sample we obtain the curves below the red straight line drawn in Fig. 5. These resonance curves are much wider thus indicating lower Q-factors calculated for them. Thus, the algorithm used returned very low resistivity values (Fig 3.).

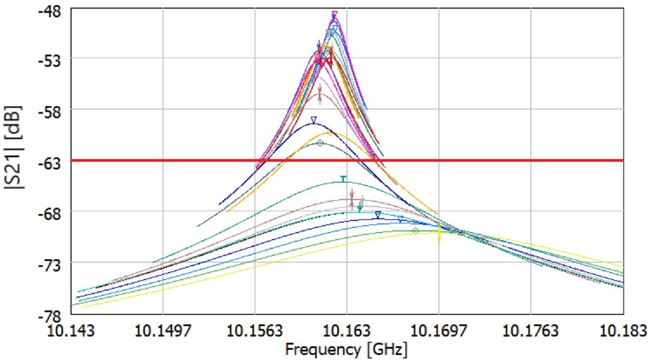

Fig. 5 Transmission in a 10 GHz scanner depending on the position of the head along the middle of ABS/GNP sample.

## III. CONCLUSIONS

We have presented a recent application of the microwave dielectric resonator technique to the characterisation of graphene-based composites. A 10GHz Inverted Single-Post Dielectric-Resonator (iSiPDR) mounted into a 2D scanner allows us to construct 2D Q-factor maps (of the iSiPDR loaded with different fragments of the scanned material). The Q-factor maps are converted to the materials resistivity maps with retro-modeling, utilizing an ultra-fast BoR FDTD electromagnetic simulations and an advanced Prony post-processing. For the initial samples ABS/GNP, significant surface inhomogeneities have been detected, indicating the need and directions for the improvement of the material fabrication process.

It should be noted that the iSiPDR-based technique provides effective de-tuning of the resonator, which is a combined effect of the constitutive parameters of the scanned material as well as its deposited thickness. Since the thickness map is unknown at this stage of our research, the thicker areas appear to be more lossy (i.e., as if built of a lower resistivity material than the deposited material really was). This does not limit the practical sense of the performed measurement, as from the viewpoint of EMI applications the overall losses of the sample are relevant. Yet for the monitoring of possible inhomogeneities of the graphene powders themselves, a better control of the deposited layer thickness will be ensured in future work. Also, the modeling-based extraction procedure will be enhanced to take into account pre-measured maps of sample thickness.

## ACKNOWLEDGEMENT

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