2D Imaging of Graphene Oxide Electric Parameters with a 10 GHz Inverted Single-Post Dielectric Resonator

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***Abstract*— Accurate measurement of conductivity and sheet resistance is essential for evaluating conductive materials, such as graphene oxide papers. This study employs a 10 GHz inverted Single-Post Dielectric Resonator (iSiPDR) integrated into a 2D scanning system to detect shifts in resonant frequency and Q- factor for thin films and bulk materials. The experimental setup, controlled by a Scanner Unit Control Application and a Vector Network Analyzer, enables the generation of 2D maps of material properties to identify variations and defects. Data analysis involves retro-modeling with a Body-of-Revolution Finite-Difference Time-Domain (BoR FDTD) algorithm and advanced signal processing techniques, including automated Prony methods, to accurately extract material parameters. This method provides a robust modelling-based characterization framework for material characterization, supporting quality control and optimization in microelectronics and battery technologies.**

***Index Terms*—material measurements, resonant method,**

non destructive testing, 2D imaging , reduced graphene oxide,

I. Introduction

The precise measurement of conductivity σ and sheet resistance R_s is essential for evaluating conductive materials, including advanced materials like graphene. This study leverages the high-resolution capabilities of a 10 GHz inverted Single-Post Dielectric Resonator (iSiPDR) [1] integrated into a 2D scanning system to achieve accurate assessments. The iSiPDR measures shifts in resonant frequency and Q-factor to evaluate both thin films, such as epitaxial layers or graphene, and bulk materials.

Our approach involves scanning the sample with the iSiPDR while controlled by a Scanner Unit Control (SUC) Application and analyzed using a Vector Network Analyzer (VNA). This setup enables the construction of 2D maps of material properties, which are crucial for detecting variations and defects. Data from these scans are processed using retro-modeling with an ultra-fast Body-of-Revolution Finite-Difference Time-Domain (BoR FDTD) algorithm and advanced signal processing, including an automated Prony method, ensuring precise extraction of resonant frequencies and Q-factors. This study is a response to the demand from the I4Bags project [2], which focuses on developing innovative processing and characterization solutions for microelectronics and battery applications. The project emphasizes advanced material analysis to improve the performance and reliability of these technologies. Our research specifically targets reduced graphene oxide (rGO) paper, particularly G-Flake [3], with meticulous sample handling and preparation to avoid measurement artifacts. This comprehensive framework provides detailed insights into material conductivity σ, sheet resistance R_s, and potential defects, aligning with the I4Bags project's goals.

Section II of this manuscript will detail the measurement setup, including the 10 GHz iSiPDR and the preparation of graphene paper. Section III will present the results, including 2D maps of material parameters. Finally, Section IV will summarize the conclusions drawn from the analysis, offering insights into the conductivity σ, sheet resistance R_s, and potential defects in the materials studied.

II. Measurement procedure

Microwave dielectric resonators are particularly effective for rGO characterization as they provide high precision in material characterization. The synthesis and processing of graphene and other conductive materials require specialized methods to maintain the integrity of the samples during measurements. Integrating advanced resonator technology with precise material preparation techniques, we provide a robust framework for detailed analysis of conductive materials, ensuring accurate and comprehensive property evaluation.

A. Measurement Setup - 10 GHz iSiPDR

We utilize a 10 GHz inverted Single-Post Dielectric Resonator (iSiPDR), specifically designed for integration into a 2D scanning system (Fig. 1). This builds on previous work [4][5] with microwave dielectric resonators, which have proven effective for characterizing conductive bulk materials, films, and surface imaging. The iSiPDR detects shifts in resonant frequency and Q-factor when a conductive sample is introduced. These shifts vary based on whether the sample is a thin film, like epitaxial layers or graphene, which are characterized by sheet resistance R_s —the resistance of a square thin film or sheet of material—or a bulk material characterized by resistivity ρ, where thickness matches or exceeds the material's skin depth. The measured Q-factor accounts for losses from the sample, the resonator cavity, and any supporting substrate, requiring two key measurements: one with the resonator empty to set a baseline and another with the sample inserted.

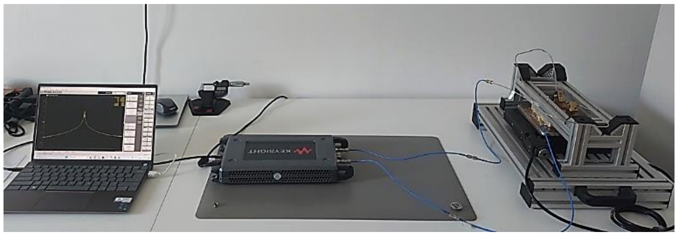

Fig. 1 A measuring station consisting of 10 GHz iSiPDR scanner, VNA and laptop with dedicated software.

Control of the scanning process is managed by SUC, which systematically controls the movement of the iSiPDR head across the sample with defined spatial resolution. At each position, a VNA, such as the Keysight VNA Streamline P5008B used in this work, conducts point-wise measurements that average material parameters over the resonator head. This scanning generates a series of resonant curves, from which resonance frequencies and Q-factors are extracted, reflecting localized variations in material properties.

The extracted data are then translated into material characteristics through retro-modeling [4], a process that matches the observed microwave response of the iSiPDR loaded with an unknown sample to modeled responses of the resonator with various assumed materials. This procedure requires extensive electromagnetic simulations to accurately align the experimental and theoretical data. To achieve this, we employ an ultra-fast conformal BoR FDTD [6] algorithm for modeling the iSiPDR in both its empty and loaded configurations, operating in a grid-search regime to assess a broad range of material properties. An advanced Prony method [7] is used, featuring automatic selection of signal post-processing parameters, allowing precise determination of resonant frequencies and Q-factors. This approach significantly enhances the speed and accuracy of the measurements, facilitating detailed characterization of the sample's electromagnetic properties.

B. Graphene Paper - Material Under Tests

The G-Flake graphene oxide used in this study was synthesized using a modified Hummers method performed by the Flake Graphene Research Group at the Łukasiewicz Research Network—Institute of Microelectronics and Photonics as reported in [8]. To create the G-Flake graphene oxide paper sheet, the purified graphene oxide was deposited onto a flexible substrate according to a method described more precisely in [8]. This method eliminates the need for any binder materials and avoids the use of toxic or costly solvents and produces sheets composed solely of pristine graphene oxide flakes. The reduction of graphene oxide paper to rGO paper involved a stepwise thermal reduction process, including two heating steps: the first at 100°C to evaporate water residues, and the second at 200°C to remove some oxygen-containing functional groups from the surface of the GO sheets, enhancing their reduction. An additional thermal step at 300°C was applied further reducing the material. The reduction was conducted in a dryer, resulting in large rGO paper sheets which were subsequently cut into smaller samples using a scalpel or stainless steel hole punch for further testing. Given the fragile nature of the graphene paper samples, which are prone to tearing and exhibit slight curvature, fused silica (FS) was used to support and flatten the samples during measurements. The FS itself was attached to the base of the resonator table, providing a stable, relatively flat surface for the samples. As a reference, measurements were taken with the resonator containing only the FS, a material known for its very low loss, with a permittivity and loss tangent that minimally impact the measured parameters of the graphene samples. This careful setup ensured that the material properties of the graphene were accurately assessed without significant interference from the support structure. The rGO sample squeezed by FS and additional tape attachment is shown in Fig. 2.

III. Results

In this study, we analyzed two samples with varying thicknesses, differentiated by their reduction methods. Cross-sectional images of the samples were obtained using a Scanning Electron Microscope (Phenom ProX) to measure the thickness of the sheets. These thickness measurements served as input values for material parameter extraction. The initial reference measurement was performed using a FS with a thickness of 0.802 mm and a 2 inch diameter. The dielectric constant ε of the FS was measured as 3.797 and the loss tangent $\tan\delta$ was 9.11×10^{-5} at 10 GHz using

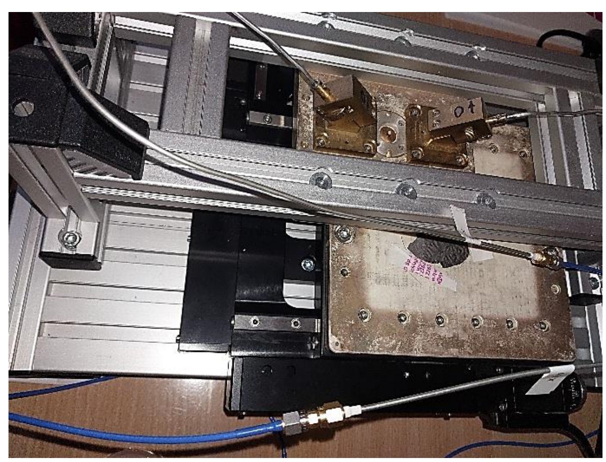

Fig. 2 10 GHz iSiPDR scanner with prepared graphene paper sample pressed with fused silica.

a Split-Post Dielectric Resonator (SPDR)[9]. The FS caused an insignificant frequency shift and changed the Q-factor from 13894.7 (empty resonator) to 13420.6 (with FS), serving as the baseline reference for subsequent measurements. A series of resonance curves were measured over a 62 mm by 62 mm area with a step size of 2 mm. The frequency shifts and Q-factors from each measurement point were utilized for automatic extraction of the R_s. The extracted R_s values were then mapped into a 2D matrix of material parameters, presented in Fig. 3, by using QuickWave software [10], which incorporates the already mentioned BoR FDTD [6] and Prony [7] algorithms.

Each scan clearly shows the outline of the fused silica as a red circle, with the underlying graphene sample visible beneath it. The edges of the graphene paper are blurred due to the averaging effect of the resonator head, which includes both the sample and the surrounding field, resulting in averaged R_s values, but are nevertheless observable and marked with black lines in Fig.3, for the readers' convenience. Across Fig. 3a, regions with significant deviations in R_s values, represented as distinct dots, are visible. These deviations are primarily due to localized defects in the samples, which are often caused by variations in sample thickness and its uniformity. In Fig. 3b, the dark blue areas in the left and right side of the maps indicate the presence of tape used to secure the fused silica, further confirming the influence of non-sample elements on the measurements. To derive meaningful insights into the material parameters, only the central region of the sample was considered for analysis, excluding the edges. The conductivity σ was calculated using the averaged R_s values and the sample thickness (d) according to the formula provided in [11]. The results are summarized in Tab. 1. Based on the calculated conductivities σ, the samples are actually thinner than the penetration depth.

As described in the manufacturing section, the material does not require the use of any binder or spoiler, resulting in a uniform flake concentration across the samples. Since the material consists solely of flakes, the observed differences between these samples are attributed to the reduction methods employed. Graphs of the Raman spectra and SEM images were not prepared for inclusion at this time, but they may be added later if the opportunity arises. The oxygen content analysis revealed that CHL contains 24.4% oxygen, while T300 has 22.4%; these values are quite similar. Structural properties, such as the number of defects, play a crucial role; CHL is expected to have a higher defect density, which theoretically should reduce its conductivity.The observed differences in conductivity between the samples appear to stem from delamination and the presence of air pockets within the graphene paper, which are introduced during the reduction process. These factors hinder good contact between the layers of flakes within the paper sheet. Comparing SEM images of the cross-sections would provide valuable insights into this phenomenon. The results indicate that the material properties are heavily influenced by the reduction method and its parameters. Mild reduction methods typically result in more efficient removal of functional groups and fewer structural defects, especially when thermal methods are employed. However, these methods also tend to cause delamination. In contrast, chemical reduction methods often lead to an increased number of defects but may reduce morphological changes such as air pockets, thereby enhancing the contact between successive layers of flakes in the sheet.

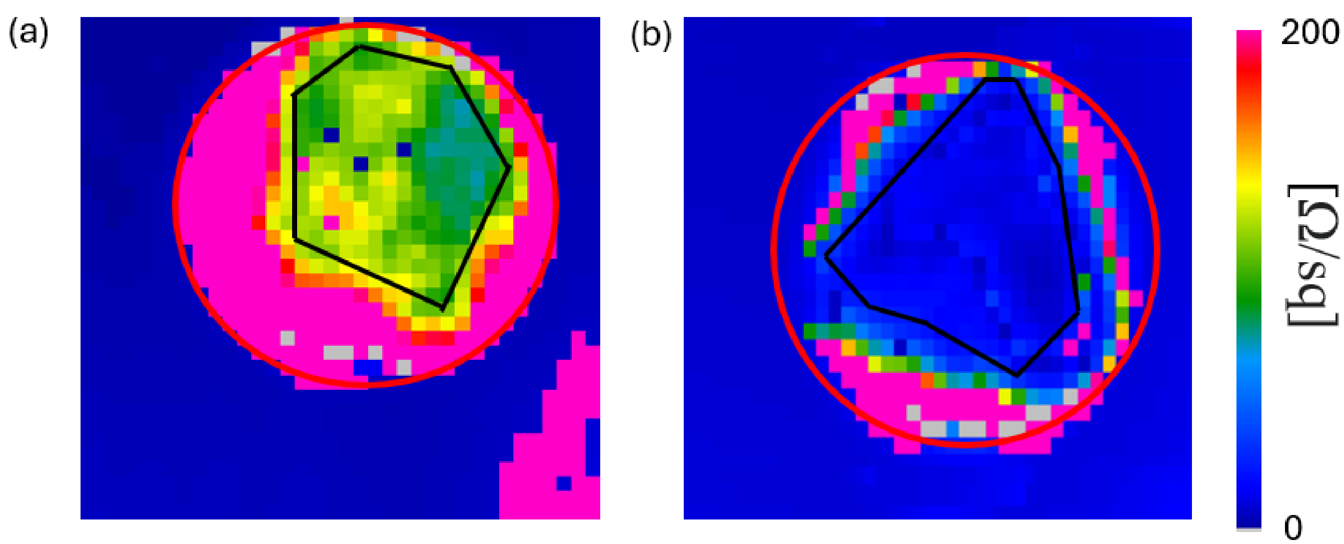

Fig. 3 2D maps of sheet resistance of rGO with different thicknesses and modified process elements: (a) T300 and (b) CHL.

TABLE I. MATERIAL PARAMETERS OF THE GIVEN GRAPHENE PAPER SAMPLES

Sample Name	Average Sheet resistance R_s [Ω/sq]	Thickness d [μm]	Average Conductivity σ [S/m]
T300 (a)	105.96 ± 30.87	4.950	1906.59 ± 554.05
CHL (b)	14.79 ± 2.18	5.085	13295.71 ± 1961.48

IV. CONCLUSION

This study demonstrated that the 10 GHz inverted Single-Post Dielectric Resonator integrated with a 2D scanning system is highly effective for measuring the conductivity σ and sheet resistance R_s of reduced graphene oxide papers. The method provided detailed 2D maps that revealed defects, thickness variations, and non-uniformities, which are crucial for quality control and optimizing material properties. The electrical properties of rGO are primarily influenced by defect concentration, the amount and type of oxygen functionalities, and the morphology of the samples. These factors are determined not only by the chosen reduction methods but also by their specific parameters, which enhance the conductivity of rGO. Improved uniformity and flatter sample surfaces result in more consistent and reliable measurements. The technique also reveals how preparation processes affect material properties, emphasizing the importance of controlled manufacturing conditions.

Our ongoing work in the I4Bags project [2] concerns two steps:

- correlating the microwave resonance-based methodology and results presented herein - to the results of direct-current Van der Pauw technique and quasi-free-space characterization of [8], in order to understand the behavior of graphene paper over a broad frequency range.
- Our method, as well as the one described in [8], demonstrates a general sensitivity to the conductivities σ of graphene paper. This indicates that the high conductivities σ observed under these measurement conditions are within the measurable limits of the employed methods. Future research will focus on expanding these limits, which will be the subject on a LAMC conference.

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