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Surface conductance of graphene from non-contact resonant cavity

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Abstract

A method is established to reliably determine surface conductance of single-layer or multi-layer atomically thin nano-carbon graphene structures. The measurements are made in an air filled standard R100 rectangular waveguide configuration at one of the resonant frequency modes, typically at TE₁₀₃ mode of 7.4543 GHz. Surface conductance measurement involves monitoring a change in the quality factor of the cavity as the specimen is progressively inserted into the cavity in quantitative correlation with the specimen surface area. The specimen consists of a nano-carbon-layer supported on a low loss dielectric substrate. The thickness of the conducting nano-carbon layer does not need to be explicitly known, but it is assumed that the lateral dimension is uniform over the specimen area. The non-contact surface conductance measurements are illustrated for a typical graphene grown by chemical vapor deposition process, and for a high quality monolayer epitaxial graphene grown on silicon carbide wafers for which we performed non-gated quantum Hall resistance measurements. The sequence of quantized transverse Hall resistance at the Landau filling factors $\nu = \pm 6$ and ± 2 , and the absence of the Hall plateau at $\nu = 4$ indicate that the epitaxially grown graphene is a high quality mono-layer. The resonant microwave cavity measurement is sensitive to the surface and bulk conductivity, and since no additional processing is required, it preserves the integrity of the conductive graphene layer. It allows characterization with high speed, precision and efficiency, compared to transport measurements where sample contacts must be defined and applied in multiple processing steps.

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Keywords

graphene; surface conductance; microwave cavity; noncontact; nondestructive

1. Introduction

Two-dimensional (2D) nano-carbon materials can be assembled into atomically-thin mono-layer or multi-layer graphene materials, which can be stacked, folded, crumpled or pillared into a variety of nano-carbon architectures with the lateral dimension limited to few tenths of a nanometer [1]. These can be synthesized by chemical vapor deposition (CVD), epitaxial growth on silicon carbide (SiC), obtained from reduced graphene oxide (rGO) or mechanically exfoliated from graphite. Many of these materials are new and exhibit extraordinary physical and electrical properties such as optical transparency, anisotropic heat diffusivity, and exceptional charge transport that are of significant interest to science, technology and commercial applications [2]. Depending on particular morphologies, density of states, and structural perfection, the surface conductance of these materials may vary from 10^{-5} S to several siemens [3,4]. For a single-layer graphene, the theoretical surface conductance in the phonon limited mobility of $2 \times 10^5 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$, and the charge carrier concentration of $10^{12}/\text{cm}^2$ may reach the value of 3.2×10^{-2} S, while acquiring value of $e^2/h \approx 4 \times 10^{-5}$ S at the Dirac point [5]. Conventional direct current (DC) surface conductance measurement techniques require complex, typically gated test structures and interconnections for making electrical contacts, which can affect and alter the measurement, making it difficult to decouple the intrinsic properties of the material [3–6].

Microwave cavity perturbation methods are attractive for quantifying the surface conductance (σ_G), because they are non-contact, non-destructive, and experimentally simple, in contrast to electrode-based techniques. In Ref. [7], a contact-less technique based on evanescent field perturbation of a dielectric post resonator has been used to characterize CVD-graphene and epitaxial graphene grown on semi-insulating SiC substrates. At 13.388 GHz, the measured surface resistance of 0.6 nm-thick layers was about 275.6Ω ($\sigma_G = 3.63 \times 10^{-3}$ S). Thicker, 1.2 nm layers showed lower surface resistance, in the range of 168.3Ω ($\sigma_G = 6 \times 10^{-3}$ S). A similar technique operating at 10.5 GHz has been presented in Refs. [8] and [9]. The method measures the frequency shift and the quality factor of a dielectric post resonator, referencing the specimen surface resistance to the electrical characteristics of the substrate. The reported surface resistance for 0.4 nm-thick epitaxial graphene was in the range of 62Ω to 27Ω ($\sigma_G = 3.7 \times 10^{-2}$ S) [8], where the latter is somewhat lower than the lowest theoretical surface resistance value predicted for graphene [5]. Clearly, lack of reliable mono-layer graphene reference material with known density of charge carriers and their mobility makes it difficult to adequately assess diversity of the reported experimental results for graphene.

Here we investigate surface conductance of graphene of epitaxial mono-layer graphene on SiC and CVD-graphene using an air-filled microwave resonant cavity. The specimen is suspended inside the cavity without contacting the cavity walls, which simplifies the cavity perturbation problem and minimizes the effect of charge carrier heating from parasitic

current loops. We compare these non-contact results with the DC longitudinal Hall resistance measurements of epitaxial mono-layer graphene grown on silicon carbide [6,10].

2. Cavity Perturbation Model

In our earlier work [11] we showed that for a small specimen inside a rectangular cavity operating in the TE_{10n} mode Equations (1a, b) from the classical perturbation theory [12] can be simplified to linear analytic expressions (2a) and (2b):

$$\frac{f_0 - f_s}{f_0} \approx 2(\varepsilon_r^* - 1) \frac{V_s'}{V_0} - b^* \quad (1a)$$

$$b^* \equiv \frac{2 \int_{V_s} (\mathbf{E}_c \mathbf{D}_s^{\text{non}}) dV}{|\mathbf{E}_0|^2 V_c} = b' + j b'' \quad (1b)$$

$$y' = (\varepsilon_r' - 1) 2x - b' \quad (2a)$$

$$y'' = \varepsilon_r'' 4x - 2b'' \quad (2b)$$

where, f_0 and f_s are the resonant frequencies of the empty cavity and cavity with the specimen, V_0 the volume of the cavity, V_s is the effective volume of the specimen ($V_0 \gg V_s$), $\varepsilon_r^* = \varepsilon_r' + j\varepsilon_r''$ is the relative complex permittivity of the specimen, the complex integral b^* accounts for the non-uniform field in the specimen, $\mathbf{E}_c$ is the local electric field in the cavity having the integral maximum of $\mathbf{E}_0$, $\mathbf{D}_s^{\text{non}}$ which represents the non-uniform field in the sample due to the depolarization field, and x is the relative volume fraction of the specimen inside the cavity.

Parameters x , y' and y'' in the linear Equations (2) are defined as follows:

$$x \equiv V_s/V_0, y' \equiv (f_0 - f_s)/f_0, \text{ and } y'' = 1/Q_s - 1/Q_0 \quad (3)$$

where, Q_0 is the quality factor of the empty cavity and Q_s is the quality factor of the cavity loaded with the specimen.

The conventional approach to solve (2a) and (2b) for the complex permittivity $\varepsilon_r^* = \varepsilon_r' + j\varepsilon_r''$ is limited to situations where the nonuniform fields can be neglected. In our experiments and theoretical simulations we did not find evidence of conditions for which $b^* \approx 0$. We found, however, that there is a range of x values where b' and b'' are constant and (2a) and (2b) represent linear equations with intercepts b' and b'' respectively.

2.1 Surface conductance

For a cavity tuned at the resonant frequency f_0 , inserting a low-loss substrate having permittivity $\varepsilon_s^* = \varepsilon_s' - j\varepsilon_s''$, thickness t_s , and volume $V_s = w t_s h$, causes the resonant frequency shift to lower frequencies from f_0 to f_s , and this shift is proportional to ε_s' . The corresponding quality factor decreases from Q_0 to Q_s by an amount that depends on the dielectric loss factor, ε_s'' . A similar experiment with the same substrate coated with a conducting layer of graphene, having permittivity $\varepsilon_G^* = \varepsilon_G' - j\varepsilon_G''$, thickness t_G , and volume $V_x = w t_G h_x$, causes the quality factor to decrease from Q_0 to Q_x , due to combined losses of the substrate and the graphene layer, $\varepsilon_G'' + \varepsilon_s''$. Since the dielectric loss of graphene is much larger than that of the substrate, $\varepsilon_s'' + \varepsilon_G'' \approx \varepsilon_G''$. In the case of conducting materials with high mobility of delocalized charge carriers, such as graphene, the energy loss resulting from dipolar polarization and polarization of bound charges can be neglected, and the dielectric loss factor is fundamentally equivalent to conductivity σ_v , $\varepsilon_G'' = \sigma_v / (2\pi\varepsilon_0 f)$ [13]. This relation can be expressed in terms of surface conductance σ_G , $\sigma_v = \sigma_G / t_G$, by (4):

$$\varepsilon_G'' = \frac{\sigma_G}{2\pi\varepsilon_0 f_x t_G} \quad (4)$$

Equation (4) can be substituted in (2b) and then rearranged into Equation (5):

$$\frac{1}{Q_x} - \frac{1}{Q_0} = \sigma_G \frac{2wh_x}{\pi\varepsilon_0 f_x V_0} c_\omega - 2b'' \quad (5)$$

where, σ_G is the surface conductance of graphene, ε_0 is the permittivity of free space, wh_x is the area of the graphene layer in the cavity, Q_x is the quality factor of cavity loaded with the graphene specimen, $c_\omega = (f_x/f_0)^2$ accounts for frequency correction due to substrate polarization, f_x is the resonant frequency of sample-loaded cavity and f_0 , Q_0 , and V_0 are the same as defined in (1) and (3). We note that Equation (5), from which we determine σ_G , does not depend explicitly on the thickness of the graphene layer, t_G , but on its area, wh_x , which can be determined with much higher accuracy than t_G . In the range of h_x where b'' is constant, (5) reduces to a linear equation with slope of σ_G . In the case when the surface conductance is comparable with that of the substrate and cannot be neglected, the conductance of the graphene layer can be extracted using a parallel admittance model of combined conductance described in Ref. [11].

3. Experimental^{b)}

Our cavity test fixture design shown in Figure 1 employs a R100 waveguide ($a=10.16$ mm, $b=22.86$ mm, $l_z = 127.0$ mm) operating in the microwave frequency range of 6.7 GHz to 13 GHz. The fixture is connected to a network analyzer (Agilent N5225A) with semi-rigid coaxial cables and coaxial to R100 coupling adapters. The walls of the cavity are implemented via R100 couplers, which are near cross-polarized in respect to the waveguide

^{b)}The method is subject to future standardization as IEC/TS 62607-6-4.

polarization. The polarization angle of 87° results from the standard dimensions of the R100 flange and is sufficient to achieve optimal power loading into cavity while maximizing the quality factor. We note that compared to typical near short termination and coaxial lumping loops, the near-cross-polarized coupling approach substantially improves uncertainty and reproducibility in f_0 , Q_0 , f_x and Q_x . The resonant frequency, f_x , and the half power bandwidth, Δf_x , are determined for each odd TE_{10n} resonant modes from the measured scattering parameter peaks, which we can detect in our cavity between 6 GHz and 12 GHz. The specimen is inserted into the cavity through a slot machined through both walls of the waveguide in the center of the cavity, where the electric field, E_x , attains a maximum value. The specimen insertion (h_x), and the corresponding area of the material in the cavity (wh_x) are controlled by a stage. During measurements, the specimen is partially inserted in steps, while the scattering parameter, S_{21} , is recorded. In the presented measurement examples, the quality factor is calculated from the conventional half power bandwidth formula as $Q_x = f_x / \Delta f_x$. The measured frequency span is typically $2 \Delta f_x$, recorded with a resolution of ± 50 kHz. The dynamic range of the noise level in the $|S_{21}|$ magnitude is typically below -65 dB. In situation when the resonant peak height decreases below -50 dB, the quality factor can be determined more accurately by fitting both, the S_{21} magnitude and phase of the resonant peak, to the damped resonator model [11]. The cavity test specimen consists of a graphene layer coated on a non-conducting substrate. The substrate provides mechanical support for handling and positioning the graphene materials inside the test fixture. We used $250 \mu\text{m}$ thick electronic grade fused-silica with a low surface conductance as a substrate for graphene obtained from CVD. The CVD single-layer (SL-CVD/G) and multi-layer (ML-CVD/G) graphene materials were obtained from a commercial source [14], and transferred onto the silica substrate surface using a process that preserves the integrity and purity of the graphene layer while minimizing the possibility of contamination [15]. Integrity of the SL-CVD/G was confirmed by optical transmittance of 97 %, Raman spectroscopy, and scanning electron microscopy [14]. Mono-layer epitaxial graphene on the Si-terminated face (SiC/G) was grown on high purity 4H-SiC(0001) $330 \mu\text{m}$ thick wafers. The wafers were diced to obtain specimen size of $7.8 \text{ mm} \times 3.7 \text{ mm}$. The substrates were then annealed at 1900°C in Argon at (101–105) kPa using a controlled Si sublimation process, which stops at one mono-layer on Si-terminated face [10]. After removing the graphene material on the C-terminated face [10], several SiC/G samples were tested directly in the microwave cavity on the native substrate. Hall resistance (ρ_{xy}), longitudinal resistance (ρ_{xx}), carrier concentration (n) and Hall mobility (μ) were measured on the corresponding samples at temperatures from 1.5 K to 30 K [10], using a Hall-bar device having a channel size $100 \mu\text{m} \times 600 \mu\text{m}$.

The implemented non-contact microwave cavity technique is applicable to materials having surface conductance from 10^{-6} S to 10 S. The surface conductance can be determined with the combined relative uncertainty of 1.5 %.

4. Results and Discussion

Figures 2(a) and 2(b) show the S_{21} magnitude of the TE_{103} mode measured for the epitaxial graphene and SL-CVD graphene as a function of the specimen insertion into cavity. The corresponding resonant peaks for SiC and silica substrates are shown in Figures 2(c) and 2(d), respectively. The height and width of the resonant peaks of both substrates remain

relatively unchanged with increasing specimen insertion, indicating low conductivity. From Equation (5), σ_s is in the range of 10^{-8} S for the silica substrate and about 10^{-7} S for the SiC substrate, which confirms the validity of the simplifying assumption in (5). In comparison, Figures 2(a) and 2(b) show that the height of the resonant peak of specimens with graphene decrease and widen considerably with increasing specimen insertion due to much higher conductance of the graphene layer, where the conductance of the SL-CVD/G (Figure 2b) is apparently higher than conductance of SiC/G (Figure 2a). The shift of the resonant peaks to lower frequencies is primarily due to the dielectric polarization of the substrates. From Equation (1), we find ϵ_r' of SiC to be 9.4 while for SiO₂ ϵ_r' is about 3.9. The frequency shift in SiC/G is larger than in SL-CVD/G and agrees well with ϵ_r' values for the SiC and silica substrates. This indicates that the polarization effects from SiC/G and SL-CVD/G layers are not significant, which would otherwise manifest as a frequency shift larger than that observed for the pure substrates.

Figure 3 shows graphical representation of Equation (5), using experimental Q_x and h_x data for SiC/G, SL-CVD/G, and ML-CVD/G materials. The solid lines are linear fits to Equation (5) through the data points. In the presented notation, the surface conductance, σ_G , is obtained directly from the slope of the linear portion of the plots where the parameter b'' in (5) can be neglected. For SiC/G (plot a) $\sigma_G = 2.55 \times 10^{-4}$ S ($\rho_G = 3922 \Omega$), for SL-CVD/G (plot b) $\sigma_G = 1.84 \times 10^{-3}$ S ($\rho_G = 543 \Omega$), and in the case of ML-CVD/G (plot c) $\sigma_G = 7.19 \times 10^{-3}$ S ($\rho_G = 139 \Omega$). The σ_G of SiC/G is almost an order of magnitude smaller than σ_G of SL-CVD/G, and about 28 times smaller than σ_G of ML-CVD/G. Moreover, the presented surface conductance results only qualitatively agree with the previously published data for similar graphene materials [7–9].

In order to evaluate our results in more detail, we performed non-gated quantum Hall resistance (QHR) measurements on our SiC/G samples. The QHR is a macroscopic quantum effect observable in quasi two-dimensional conductors. It relates the resistance quantum h/e^2 (or the complementary conductance quantum, e^2/h) to the fundamental Planck constant, h , and the elementary charge, e . The QHR universality allows assessing the quality of the SiC/G specimens and establishing a reliable reference for evaluating conductance of graphene samples from the CVD process. Figure 4 shows the longitudinal resistivity (ρ_{xx}) and transverse resistance (ρ_{xy}) as a function of magnetic flux density B at several temperatures, from 1.5 K to 30 K. The ρ_{xx} plots in Figure 4(a) show at $B \leq \pm 4$ T the characteristic Shubnikov-de Haas oscillations. At higher B , ρ_{xx} decreases to near zero indicating that the charge transport approaches the fully quantized state. The corresponding ρ_{xy} values in Figure 4(b) indicate the exact quantum Hall resistance, at the Landau filling factors $\nu = \pm 6$ and ± 2 , where at $\nu = 2$, the extended ρ_{xy} plateaux acquire the value of $h/2e^2 \approx 12.906$ k Ω . The sequence of quantized ρ_{xy} at $\nu = \pm 6$ and ± 2 and the clear absence of the quantized ρ_{xy} plateau at $\nu = 4$ in Figure 4(b), provide strong evidence that the SiC/G is a mono-layer [10,16]. We note that the electric field configuration of ρ_{xx} at zero magnetic field corresponds to that of ρ_G measured in the microwave cavity. Therefore we may correlate ρ_{xx} at zero B with ρ_G from the cavity measurements. In the vicinity of small B , ρ_{xx} decreases with increasing temperature from 5.699 k Ω at 1.5 K to about 4.588 k Ω at 30 K. The weak localization peak at zero B in Figure 4(a), and a decrease of ρ_{xx} with increasing temperature

can be explained within the formalism of the Fermi liquid model as to originate from charge disorder within the buffer layer beneath the SiC/G mono-layer [17].

In the conductivity notation the longitudinal conductance σ_{xx} increases in our experiment from 1.755×10^{-4} S at 1.5 K to 2.180×10^{-4} S at 30 K. The corresponding Hall mobility (μ) decreases with increasing temperature from $4688 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$ at 1.5 K to $4575 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$ at 30 K, while the concentration of charge carriers (n) increases in this temperature range, from $2.34 \times 10^{11} \text{ cm}^{-2}$ to $2.98 \times 10^{11} \text{ cm}^{-2}$. Similarly to n , we observe an increase of $\sigma_{xx} = en\mu$ with the temperature, suggesting that the interplay between mobility μ and n , and their contribution to σ_{xx} is primarily due to the carrier concentration. This trend may continue to higher temperatures, where at zero gating voltage, conductance due to intrinsic carriers concentration is evidently non-metallic, and is shown to increase by a factor of 2 with increasing temperature from 4.2 K to 300 K, at either low and high mobilities [18,19]. Thus, in our experiment we may rationalize the similarity between experimental σ_{xx} and σ_G values in terms of impurity scattering from molecular doping [20], and the increase of charge carrier concentration with increasing temperature. Assuming that at 298 K the Hall mobility of SiC/G may decrease to $4000 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$, the measured σ_G would correspond to a moderate increase of n to about $3.5 \times 10^{11} \text{ cm}^{-2}$. We note that the experimental σ_G obtained in the microwave cavity are reproducible to within 2 %. Our SiC/G samples were handled at ambient conditions for several weeks and/or subjected to annealing cycle at 120 °C under nitrogen for several hours without any significant effect on the measured σ_G . In the case of SL-CVD/G, we compare our σ_G results with the AC conductance obtained for CVD graphene on SiO₂ substrate from the THz absorption spectra [21,22]. Accordingly, below the carrier relaxation frequency (f_{cr}) of about 0.4 THz [24], the real part of conductance (σ_1) appears suppressed, and it increases from DC through microwave frequencies according to the universal scaling law [23,24], reaching a maximum value of about $55 (e^2/h) \approx 2.1 \times 10^{-3}$ S [22]. At higher frequencies, above f_{cr} , the relaxation of charge carriers gives rise to imaginary conductance (σ_2), and consequently, the response becomes increasingly dielectric. From the fit to the Drude formula [22], $\mu \approx 2000 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$ and $n \approx 6 \times 10^{12} \text{ cm}^{-2}$, consistent with charge transport in doped graphene on a substrate [19,21]. The corresponding quasi-static conductance of about 1.9×10^{-3} S is in good agreement with $\sigma_G = 1.85 \times 10^{-3}$ S measured for SL-CVD/G. Assuming similar mobility of $2000 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$, and carrier concentration of $6 \times 10^{12} \text{ cm}^{-2}$, the measured σ_G of 7.2×10^{-3} S for ML-CVD/G corresponds to four layers of doped graphene.

5. Summary

We employed a microwave cavity perturbation technique for non-contact surface conductance measurement of graphene materials deposited on dielectric substrate. By acquiring data over a range of the specimen insertion into cavity, surface conductance can be determined with increased accuracy simply from the slope of Equation (5). The thickness of the graphene layer does not need to be known, but it is assumed that its lateral dimension is uniform over the specimen area. The method is illustrated for mono-layer epitaxial graphene SiC/G, single-layer CVD graphene, and multi-layer CVD graphene, having surface conductances of 2.55×10^{-4} S, 1.84×10^{-3} S, and 7.19×10^{-3} S, respectively. The measured QHR, is a macroscopic quantum effect observable in quasi two-dimensional conductors. It

relates the resistance quantum h/e^2 (or the complementary conductance quantum e^2/h) to the fundamental Planck constant, h , and the elementary charge, e . The QHR universality allows to assess the quality of the SiC/G specimens and establish a reliable monolayer graphene reference material for evaluating conductance of graphene samples from the CVD process.

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Figure 1. Microwave cavity test fixture. (1) waveguide of length l_z , (2) cross-polarized couplers, (3) specimen, (4) specimen holder. The waveguide cross-section and orientation of the electric (E_x) and magnetic (H_x) field vectors are shown in the inset.

Figure 2.

Scattering parameters S_{21} of the resonant peak T_{103} at $f_0 = 7.434935$ GHz moving to lower frequencies with increasing specimen insertion (h_x). (a) mono-layer SiC/G. (b) SL-CVD/G. (c) 4H-SiC(0001) substrate. (d) Fused-silica substrate.

Figure 3.

Plots of $(1/Q_x - 1/Q_0)$ as a function of the normalized specimen area, $w h_x$. (a) mono-layer SiC/G, (b) SL-CVD/G and (c)-ML-CVD/G. Symbols – experimental data, the solid lines are linear fits to Equation (5) through the data points. The combined uncertainty of σ_G determined from the slope of the plots is 1.5 %.

Figure 4.
Longitudinal (a) and (b) transverse resistance of SiC/G mono-layer at indicated temperatures.