



BSI Standards Publication

Nanomanufacturing — Key control characteristics

Part 6-4: Graphene — Surface conductance measurement using resonant cavity

National foreword

This Published Document is the UK implementation of IEC/TS 62607-6-4:2016.

The UK participation in its preparation was entrusted to Technical Committee NTI/1, Nanotechnologies.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Published by BSI Standards Limited 2016

ISBN 978 0 580 91019 7

ICS 07.030

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This Published Document was published under the authority of the Standards Policy and Strategy Committee on 31 October 2016.

Amendments/corrigenda issued since publication

Date	Text affected
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TECHNICAL SPECIFICATION



**Nanomanufacturing – Key control characteristics –
Part 6-4: Graphene – Surface conductance measurement using resonant cavity**

INTERNATIONAL
ELECTROTECHNICAL
COMMISSION

ICS 07.030

ISBN 978-2-8322-3667-3

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

**NANOMANUFACTURING –
KEY CONTROL CHARACTERISTICS –****Part 6-4: Graphene – Surface conductance
measurement using resonant cavity**

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Technical Specifications are subject to review within three years of publication to decide whether they can be transformed into International Standards.

IEC TS 62607-6-4, which is a Technical Specification, has been prepared by IEC technical committee 113: Nanotechnology for electrotechnical products and systems.

The text of this Technical Specification is based on the following documents:

Enquiry draft	Report on voting
113/295/DTS	113/324/RVC

Full information on the voting for the approval of this Technical Specification can be found in the report on voting indicated in the above table.

This document has been drafted in accordance with the ISO/IEC Directives, Part 2.

A list of all parts in the IEC 62607 series, published under the general title *Nanomanufacturing – Key control characteristics*, can be found on the IEC website.

The committee has decided that the contents of this document will remain unchanged until the stability date indicated on the IEC website under "<http://webstore.iec.ch>" in the data related to the specific document. At this date, the document will be

- transformed into an International Standard,
- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

A bilingual version of this publication may be issued at a later date.

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INTRODUCTION

The microwave resonant cavity test method for surface conductance is non-contact, fast, sensitive and accurate. It is well suited for standards, research and development (R&D), and for quality control in the manufacturing of two-dimensional (2D) nano-carbon materials. These sheet-like or flake-like carbon forms can be assembled into atomically-thin monolayer or multilayer graphene materials, which can be stacked, folded, crumpled or pillared into a variety of nano-carbon architectures with the lateral dimension limited to a few tenths of a nanometre. Many of these materials are new and exhibit extraordinary physical and electrical properties such as optical transparency, anisotropic heat diffusivity and charge transport that are of significant interest to science, technology and commercial applications [1, 2]¹.

Depending on particular morphologies, density of states and structural perfection, the surface conductance of these materials may vary from 1 S to about 10^{-4} S. Conventional direct current (DC) surface conductance measurement techniques require a complex test vehicle and interconnections for making electrical contacts, which affect and alter the measurement, making it difficult to decouple the intrinsic properties of the material.

In comparison, the resonant cavity measurement method is fast and non-contact. Thus, it is well suited for use in R&D and manufacturing environments where the surface conductance is a critical functional parameter. Moreover, it can be employed to measure electrical characteristics of other nano-size structures.

¹ Numbers in square brackets refer to the Bibliography

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 6-4: Graphene – Surface conductance measurement using resonant cavity

1 Scope

This part of IEC 62607 establishes a method for determining the surface conductance of two-dimensional (2D) single-layer or multi-layer atomically thin nano-carbon graphene structures. These are synthesized by chemical vapour deposition (CVD), epitaxial growth on silicon carbide (SiC), obtained from reduced graphene oxide (rGO) or mechanically exfoliated from graphite [3]. The measurements are made in an air filled standard R100 rectangular waveguide configuration, at one of the resonant frequency modes, typically at 7 GHz [4].

Surface conductance measurement by resonant cavity involves monitoring the resonant frequency shift and change in the quality factor before and after insertion of the specimen into the cavity in a quantitative correlation with the specimen surface area. This measurement does not explicitly depend on the thickness of the nano-carbon layer. The thickness of the specimen does not need to be known, but it is assumed that the lateral dimension is uniform over the specimen area.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

IEC 60153-2, *Hollow metallic waveguides – Part 2: Relevant specifications for ordinary rectangular waveguides*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in IEC 60153-2 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <http://www.iso.org/obp>

3.1 Graphene layers

3.1.1

graphene
single-layer graphene
1LG

single layer of carbon atoms with sp^2 -electronic hybridization bound to three neighbours in a honeycomb structure

Note 1 to entry: It is an important building block of many carbon nano-objects.

[SOURCE: ISO/TS 80004-3:2010, 2.11, modified.]

3.1.2

bilayer graphene

2LG

two-dimensional material consisting of two well-defined stacked graphene layers

Note 1 to entry: If the stacking registry is known it can be specified separately, for example as "Bernal stacked bilayer graphene".

3.1.3

trilayer graphene

3LG

two-dimensional material consisting of three well-defined stacked graphene layers

Note 1 to entry: If the stacking registry is known it can be specified separately, for example as "Bernal stacked trilayer graphene" or "twisted trilayer graphene".

3.1.4

few-layer graphene

FLG

two-dimensional material consisting of three to ten well-defined stacked graphene layers

3.1.5

nanoplate

nano-object with one external dimension in the nanoscale and the other two external dimensions significantly larger

[SOURCE: ISO/TS 80004-2:2015, 4.6]

3.1.6

nanosheet

nanoplate with extended lateral dimensions

3.1.7

graphene oxide

GO

chemically modified graphene prepared by oxidation and exfoliation of graphite that is accompanied by extensive oxidative modification of the basal plane

Note 1 to entry: Graphene oxide is a single material with a high oxygen content, typically characterized by C/O atomic ratios less than 3.0 and typically closer to 2.0.

3.1.8

reduced graphene oxide

rGO

graphene oxide that has been processed to reduce its oxygen content

Note 1 to entry: This can be produced by chemical, thermal, microwave, photo-chemical, photo-thermal or microbial/bacterial methods or by exfoliating reduced graphite oxide.

Note 2 to entry: If graphene oxide was fully reduced then graphene would be the product, however in practice some oxygen containing functional groups will remain and not all sp^3 bonds will return back to sp^2 configuration. Different reducing agents will lead to different carbon to oxygen ratios and different chemical compositions in reduced graphene oxide

3.1.9**graphene material**

nanomaterial based on graphene

Note 1 to entry: Examples of graphene material are multilayered graphene (less than about 10 layers), chemically modified forms (GO, rGO), and materials made via another precursor material or process such as chemical vapour deposition (CVD) [3].

3.2 Measurement terminology**3.2.1** σ_s **surface conductance**

characteristic physical property of two-dimensional materials describing the ability to conduct electric current

Note 1 to entry The SI unit of measure of σ_s is siemens (S). In the trade and industrial literature, however, siemens per square (S/square) is commonly used when referring to surface conductance. This is to avoid confusion between surface conductance and electric conductance (G), which share the same unit of measure:

$$G = IU = \sigma_s (w/l).$$

Note 2 to entry: The surface conductance (σ_s) can be obtained by normalizing conductance G to the specimen width (w) and length (l).

3.2.2 G **electric conductance**

measure of how easily electric current flows along a certain path

Note 1 to entry: The SI unit of electric conductance is siemens (S).

3.2.3 σ_v **volume conductivity**

characteristic physical property of three-dimensional materials describing the ability to conduct electric current

Note 1 to entry: The volume conductivity can be obtained by dividing the surface conductance by the conductor thickness (t):

$$\sigma_v = \sigma_s/t.$$

The unit of measure of σ_v is siemens per metre (S/m).

3.2.4 ρ_s **surface resistance****sheet resistance**reciprocal of σ_s

Note 1 to entry: ρ_s is a characteristic property of two-dimensional materials. The SI unit of measure of ρ_s is ohm (Ω). In the trade and industrial literature, however, ohm per square (Ω /square) is commonly used when referring to surface resistance or sheet resistance.

3.2.5**microwave cavity****radio frequency cavity****RF cavity**

resonator consisting of a closed metal structure that confines electromagnetic fields in the microwave region of the spectrum

Note 1 to entry: The structure can be filled with air or other dielectric material. A cavity acts similarly to a resonant circuit with extremely low loss at its frequency of operation. Microwave cavities are typically made from

closed (or short-circuited) sections of a waveguide. Every cavity has numerous resonant frequencies (f_r) that correspond to electromagnetic field modes satisfying the necessary boundary conditions, i.e. the cavity length is an integer multiple of half-wavelength at resonance.

3.2.6

Q

quality factor

dimension-less parameter describing the ratio of energy stored in the resonant circuit to time-averaged power loss of the cavity, or equivalently, a resonator's half power bandwidth, (Δf) relative to the resonant frequency (f_r):

$$Q = f_r / \Delta f$$

3.2.7

S_{ij}

microwave scattering parameters

S-parameters

factors that quantify how RF energy propagates through a microwave multi-port network.

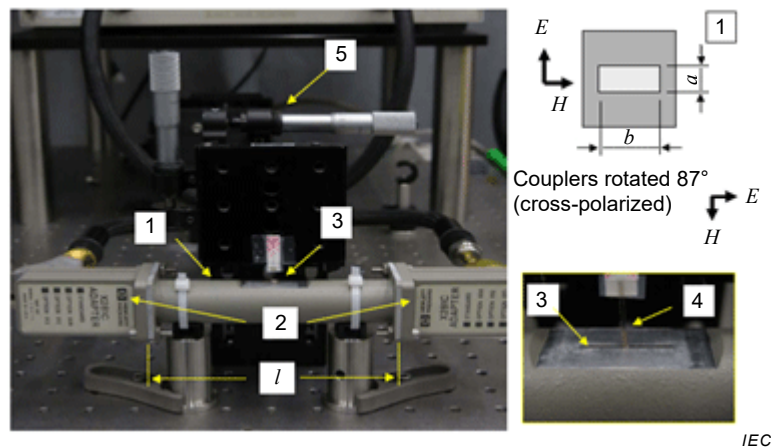
Note 1 to entry: Subscript (i) indicates the detecting port. Subscript (j) refers to the sourcing (input) port. Accordingly, S_{21} quantifies microwave energy that is transmitted from port_1 to port_2. In comparison, S_{11} quantifies microwave energy that is sourced and detected at port_1 and it is often referred to as a reflection coefficient.

4 Microwave cavity test fixture

The test fixture is shown in Figure 1. It consists of a R100 (WR-90²) waveguide (1), having the nominal load impedance of 50 Ω , such that the Voltage Reflected Standing Wave ratio (VRSW) is 1,04:1 or less, and the insertion loss 0,5 dB/m or less. The waveguide operates in the microwave frequency range of 6,6 GHz to 13,0 GHz. The waveguide dimensions are $a = 10,16$ mm, $b = 24,6$ mm and its length $l = 134$ mm. The waveguide is short terminated on both ends. The walls of the cavity are implemented via coax to R100 (WR-90) waveguide couplers (2), which are cross-polarized ($\theta = 87^\circ$) with respect to the waveguide electromagnetic field polarization, E and H . The resonant frequency of the fundamental modes excited along the propagation direction is determined by the electrical length (l_e) of the cavity. For an air filled waveguide with the relative permittivity $\epsilon_r = 1,0$, $l_e \approx l$.

The specimen is inserted into the cavity through a narrow slot (3) (1 mm \times 10 mm), precisely machined through both walls of the waveguide in the centre of the cavity, where the electric field (E) attains a maximum value. The specimen (4) is attached to a stage (5). The stage is used to control and measure the specimen area inside the cavity. In the case of liquid samples the entire test fixture in Figure 1 may be reoriented to perform the measurement of the specimen in horizontal position.

² In some countries the R100 standard waveguide is referenced as WR-90.



Key

- 1 waveguide
- 2 couplers
- 3 slot for specimen insertion
- 4 specimen
- 5 specimen holder

Figure 1 – Microwave cavity test fixture

5 Test specimen

The test specimen consists of a graphene layer coated on or bonded to a non-conducting substrate. The substrate provides mechanical support for handling and positioning the graphene materials inside the test fixture. In order to minimize effects of the substrate on the measurement, the substrate material should exhibit low conductivity and low dielectric permittivity. The recommended substrate for graphene obtained from CVD, exfoliation or other synthetic routes is electronic grade fused silica wafer, 200 μm to 250 μm thick. The graphene material to be tested should be transferred onto the substrate surface using a process that preserves the integrity and purity of the graphene layer, while minimizing the possibility of contamination. The method requires both the coated and uncoated substrate. The recommended size of test specimen is 3 mm \times 20 mm. Epitaxial graphene grown on silicon carbide (SiC) may be tested directly on the native substrate after removing either the Si- or C-terminated face.

NOTE Epitaxial graphene can easily be grown by heating the SiC single crystal in a high vacuum or in an inert gas atmosphere. The SiC surfaces used for graphene growth contain Si- and C-terminated faces. On the Si-face, homogeneous and clean graphene can be grown with a controlled number of layers. The carrier mobility reaches as high as several $\text{m}^2\text{V}^{-1}\text{s}^{-1}$, although this is reduced by the presence of the substrate steps. On the C-face, although the number of layers is not homogeneous, twisted bilayer graphene can be grown, which is expected to be the technique of choice to modify the electronic structure of graphene. From the application point of view, graphene on SiC will be the platform used to fabricate high-speed electronic devices and dense graphene nano-ribbon arrays, which will be used to introduce a bandgap [5].

6 Measurement procedure

6.1 Apparatus

The measurement requires an automatic two-port vector network analyser (VNA) operating in the frequency range that covers the frequency band (6,6 GHz to 13 GHz) with the capability of measuring the scattering wave parameters S_{21} or S_{12} transmitted between ports 1 and 2 of the VNA. Connections between the test fixture (Clause 4) and the network analyser should be made using high quality coaxial cables and appropriate adapters. The dynamic range of the measurements should be within 70 dB or greater. The instrument should be equipped with an

IEEE 488 I/O interface or equivalent, for transferring data between the network analyser and a data collection unit.

6.2 Calibration

Calibration is required only at the coaxial ends. Two-port full calibration for S_{21} and S_{12} should be performed in accordance with the network analyser manufacturer's specification using an appropriate Short-Open-Load calibration kit.

6.3 Measurements

6.3.1 General

Connect the empty test fixture to the vector network analyser (VNA). Set the VNA to measure S_{21} magnitude with 800 data points or more. Select the frequency span to 2 GHz and the centre frequency to 8,5 GHz. Several resonant peaks should appear on the VNA screen, each with the S_{21} peak value of about –20 dB and the S_{21} minimum value (noise floor) in the range of –60 dB or less. Identify the resonant frequency (f_0) of the third resonant peak TE_{103} , for which the electric field of the standing wave attains its maximum value in the middle of the cavity.

NOTE The electric field inside the cavity attains a maximum value in the middle of the cavity where the specimen is inserted for odd fundamental $TE_{1,0,n}$ modes, where $n = 1, 3, 5, \dots$. The first resonant mode, $TE_{1,0,1}$, may fall within the waveguide cut-off frequency, below 6,5 GHz, if the cavity is electrically too long. Therefore, the test method recommends to use modes 3, 5, 7, These modes can be easily identified. Inserting the specimen into the cavity causes all the resonant peaks corresponding to odd modes to decrease in magnitude and shift to lower frequencies, while the resonant peaks corresponding to even modes ($n = 2, 4, 6, \dots$) remain intact.

6.3.2 Empty cavity

Set the centre frequency to f_0 and the frequency span to about $2\Delta f$ (4 MHz or less), such that $|S_{21}|$ peak height is about 5 dB. Determine the half power bandwidth, $\Delta f = |f_2 - f_1|$, where f_2 and f_1 are frequencies of the resonant peak, 3 dB below the $|S_{21}|$ maximum. Determine the quality factor Q_0 of the empty cavity from Formula (1).

$$Q_0 = \frac{f_0}{\Delta f} \quad (1)$$

6.3.3 Specimen

Insert the specimen into the cavity in steps (x), while measuring the length of the insertion (h_x) perturbing the cavity volume (see note 1 to 6.3.3). Record the area (A_x) of the sample inside the cavity at each step (h_x).

$$A_x = w \cdot h_x \quad (2)$$

where w is the width of the test specimen. When the cavity is perturbed by the specimen, the position (f_x) of the resonant peak should move to lower frequencies and the $|S_{21}|_{\max}$ value should decrease when A_x increases. Adjust the centre frequency and the frequency span as in 6.3.2, if necessary. Record the resonant frequency f_x , half power bandwidth Δf and the corresponding quality factor Q_x (Formula (1)). (See note 2 to 6.3.3).

NOTE 1 The active length (h_x) perturbing the cavity volume corresponds to the portion of the specimen inside the cavity (see Figure 1). The typical value of Q_0 is about 3 000, which can be measured with uncertainty $\Delta Q \approx 10$ [4,6]. Therefore, the minimum insertion h_{x0} which causes the quality factor to change from its initial value Q_0 can be determined experimentally from ΔQ .

NOTE 2 The operating menu of a typical automatic network analyser includes functions for finding the peak maximum (resonant frequency), calculating the half-power bandwidth, quality factor, and for adjusting the centre frequency and the frequency span. These functions can be executed either manually or invoked from a stored macro program for automated calculation of Q after each specimen insertion step.

6.3.4 Repeated procedure

Repeat the steps in 6.3.2 recording Q_x and A_x , until $h_x \geq 10$ mm ($h_x \geq a$) or when the resonant peak height decreases to about 10 dB above the noise level.

6.3.5 Substrate

Optionally perform the steps in 6.3.2 and 6.3.3 for bare substrate.

7 Calculations of surface conductance

Equation (3) correlates the specimen surface conductivity with the measured quality factor and the specimen surface area [4]:

$$\frac{1}{Q_x} - \frac{1}{Q_0} = \sigma_s \frac{2}{\pi \varepsilon_0 f_0 V_0} (w h_x) \quad (3)$$

where:

σ_s is the specimen surface conductance;

$w h_x$ is the specimen surface area inside the cavity;

Q_x is the quality factor of the specimen;

Q_0, f_0 , are the quality factor and resonant frequency of the empty cavity;

V_0 is the volume of the empty cavity, $V_0 = a b l$ (see Figure 1);

ε_0 is the permittivity of free space.

Since for a given cavity V_0 , Q_0 and f_0 are constant parameters, Formula (3) can be rearranged into Formula (4), which can be fitted to a straight line.

$$\frac{1}{Q_x} - \frac{1}{Q_0} = \sigma_s \cdot k (w h_x) \quad (4)$$

where:

$$k = 2 / (\pi \varepsilon_0 f_0 V_0).$$

Thus, σ_s can be solved from the slope of a linear portion of plot $(1/Q_x - 1/Q_0)$ vs $(k w h_x)$ (see Figure A.2).

8 Report

The report shall include the following:

- Preparation procedure and dimensions of the specimen.
- Values of V_0 , Q_0 and f_0 .
- Table of f_x , Q_x and h_x for the sample specimen with graphene layer.
- Plot of $1/Q_x - 1/Q_0$ vs $(k w h_x)$ for the sample specimen containing graphene layer on the substrate.
- Table of f_x , Q_x and h_x for the specimen of bare substrate having nominal width w same as the sample specimen.
- Plot of $1/Q_x - 1/Q_0$ as a function of $(k w h_x)$ for the bare substrate.

- Measured surface conductance for specimen containing graphene layer on the substrate, σ_{s_m} .
- Measured surface conductance for specimen of bare substrate, σ_{s_sub} .
- Surface conductance, σ_G , for the graphene layer: $\sigma_G = \sigma_s - \sigma_{s_sub}$.

The typical surface conductance value of graphene materials can be between 1 S (for FLG and/or doped graphene) and 10^{-5} S (for SGL). Such conductance values are much larger than the typical surface conductance of fused silica substrate and therefore the effect of substrate can be neglected in most cases, that is $\sigma_G \approx \sigma_s$. If the σ_s value is comparable to σ_{s_sub} , but σ_s is larger than σ_{s_sub} by at least one order of magnitude, subtract σ_{s_sub} from σ_s to obtain σ_G . When the film consists of a semi-continuous network of graphene nanoplates, σ_s can be much smaller than 10^{-5} S. Similarly, σ_s can decrease rapidly with increasing concentration of carbon sp^3 defects, for example, after chemical functionalization or incomplete reduction of rGO. In the case when the measured surface conductance is comparable to surface conductance of the substrate, results from the simple subtraction formula may be unreliable. In such cases it is recommended to use the data correction model described in reference [6].

9 Accuracy consideration

Several uncertainty factors such as instrumentation, dimensional uncertainty of the test specimen geometry, roughness and impurities contribute to the combined uncertainty of the measurements. Adequate analysis can be performed, however, by using the partial derivative technique for Formula (3) or Formula (4) and considering the instrumentation and the experimental errors. The standard uncertainty of S_{21} can be assumed to be within the manufacturer's specification for the network analyser, about $\pm 0,01$ dB for the magnitude and $\pm 0,5^\circ$ for the phase. The resonant frequency can be determined to within a few kilohertz when collecting 800 data points over the frequency span of $2\Delta f$. The corresponding relative uncertainty of the Q_x factor is then typically below 0,5 %. The combined relative standard uncertainty of the measurements is typically better than 6 %. Further improvement in accuracy can be achieved by fitting the resonant peak data to a damped oscillator model, which utilizes both the magnitude and phase of the measured complex scattering parameter S_{21} [6]. Comparison of calculation techniques for quality factor influenced by the measurement uncertainty of the network analyser is given in reference [7].

Additional limitations may arise from the systematic uncertainty of the particular instrumentation, calibration standards and the dimensional and structural imperfections of the actually implemented test specimen.

Annex A (informative)

Case study of surface conductance measurement of single-layer and few-layer graphene

A.1 General

In this case study, surface conductance of commercially available single-layer and few-layer graphene from the CVD process was measured using non-contact resonant cavity. The implemented partial specimen insertion allows precise control of the sample area in the cavity, more data points for fitting and overall better accuracy. The measurements are referenced to resonant frequency in air.

A.2 Cavity perturbation procedure

The cavity perturbation by a specimen, having relative complex permittivity $\varepsilon_r^* = \varepsilon_r' - j\varepsilon_r''$, is given by Formulas (A.1) to (A.3), where (A.3) accounts for non-uniform fields [4, 6].

$$\frac{f_0 - f_r}{f_0} = (\varepsilon_r' - 1) \cdot 2 \frac{V_r}{V_0} - b_r \quad (\text{A.1})$$

$$\frac{1}{Q_r} - \frac{1}{Q_0} = \varepsilon_r'' \cdot 4 \frac{V_r}{V_0} - 2b_q \quad (\text{A.2})$$

$$C_V = \frac{V_r}{\int_{V_0} E_r E_0 dV} = b_r + b_q \quad (\text{A.3})$$

High frequency structure simulator (HFSS) software was employed to solve Formulas (A.1) to (A.3) numerically under the assumption that the specimen volume, V_r , is small compared to volume of the cavity, V_0 , $V_r \ll V_0$, and the sample permittivity does not depend on V_r . In the range of Q_r , and V_r values where $(b_r + b_q) = \text{constant}$, Formulas (A.1) and (A.2) are linear and can be solved for ε_r^* by measuring Q_0 , Q_s , f_0 and f_r as a function of V_r [4]. Inserting into cavity, tuned at the resonant frequency f_0 , a low loss substrate, having permittivity $\varepsilon_{\text{sub}}^* = \varepsilon'_{\text{sub}} - j\varepsilon''_{\text{sub}}$, thickness t_{sub} and volume $V_{\text{sub}} = w t_{\text{sub}} h$, causes the resonant frequency shift to lower frequencies from f_0 to f_{sub} , which is proportional to $\varepsilon'_{\text{sub}}$. The corresponding quality factor decreases from Q_0 to Q_{sub} , which depends on $\varepsilon''_{\text{sub}}$. Similar experiments with nominally the same substrate coated with a conducting layer of graphene material, having thickness t_G and volume $V_G = w t_G h$, causes the quality factor to decrease from Q_0 to $Q_{G\text{-sub}}$, due to combined losses of the substrate and the graphene layer, $\varepsilon''_G + \varepsilon''_{\text{sub}}$. The dielectric loss and surface conductance are related by Formula (A.4):

$$\varepsilon''_G = \frac{\sigma_G}{2\pi\varepsilon_0 f_0 t_G} \quad (\text{A.4})$$

Assuming that the dielectric loss of graphene is much larger than that of the substrate $\varepsilon''_{\text{sub}} + \varepsilon''_G \approx \varepsilon''_G$, Formula (A.2) can be simplified and rearranged into Formula (A.5) as shown in reference [4]:

$$\frac{1}{Q_x} - \frac{1}{Q_0} = \frac{\sigma_G}{\pi \varepsilon_0 f_0} \frac{2wh_x}{V_0} - 2b_q \quad (\text{A.5})$$

where

σ_G is the surface conductance of the graphene layer;

ε_0 is the permittivity of free space;

wh_x is the area of the graphene layer specimen inserted into the cavity;

Q_x is the quality factor at insertion h_x ;

f_0 , Q_0 and V_0 are the resonant frequency, quality factor and volume of the empty cavity, respectively.

NOTE Formula (A.5), from which σ_G is determined, does not depend on the thickness of the graphene layer but on its area, wh_x , which can be determined with much higher accuracy than t_G [4].

A.3 Experimental procedure

Single-layer graphene (1LG) and few-layer graphene (FLG) from a CVD process were obtained from a commercial source and transferred on to 200 μm thick fused silica wafers [8], from which 3 mm \times 20 mm specimens were extracted by dicing.

The cavity test fixture design shown in Figure 1 employs a 134 mm long R100 waveguide operating in the frequency range of 6,6 GHz to 13 GHz [4]. The specimen area inside the cavity is controlled by partial insertion. The fixture is connected to a network analyser (Agilent 8720D³) with semi-rigid coaxial cables and coaxial to R100 adapters. The walls of the cavity are implemented via R100 couplers, which are cross-polarized ($\varphi = 87^\circ$) with respect to the waveguide polarization. The system is calibrated at the coaxial ends using a Short-Open-Load calibration kit. The resonant frequency f_x and half power bandwidth Δf_x are determined for each TE₁₀₃ to TE₁₀₉ odd resonant modes, from the measured scattering parameter peaks between 6 GHz and 13 GHz. The test fixture, instrumentation and the measurement procedure are described in Clauses 4 to 8. Here, f_0 of the TE₁₀₃ peak = 7,319 1125 GHz, $Q_0 = 3\,000$ and $V_0 = 33,491\text{ cm}^3$.

A.4 Results

Figure A.1 shows the magnitude of S_{21} , at the TE₁₀₃ resonant peak measured for 1LG and FLG, as a function of the specimen insertion into cavity. Figure A.1 a) shows that with increasing insertion, the height of the resonant peak of fused silica substrate remains relatively unchanged indicating a low conductivity of the substrate, therefore confirming validity of simplifying assumption in Formula (A.5). In comparison, Figure A.1 b) shows that the height of the resonant peak of 1LG decreases considerably with increasing specimen insertion due to much higher conductance of the graphene layer. The conductance of FLG is larger than that of 1LG, as evidenced by the rapid decrease in the height of the resonant peak shown in Figure A.1 c). In addition to the decrease in height, this resonant peak shifts to lower frequency considerably, indicating a large dielectric polarization, likely at the boundaries between grains in the FLG sample.

³ Agilent 8720D is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by IEC of this product.

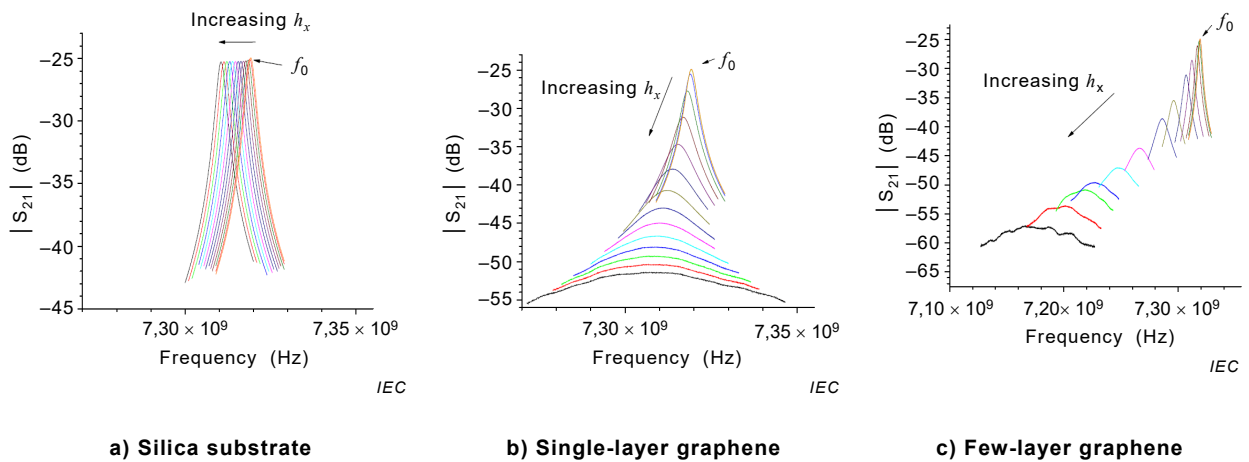
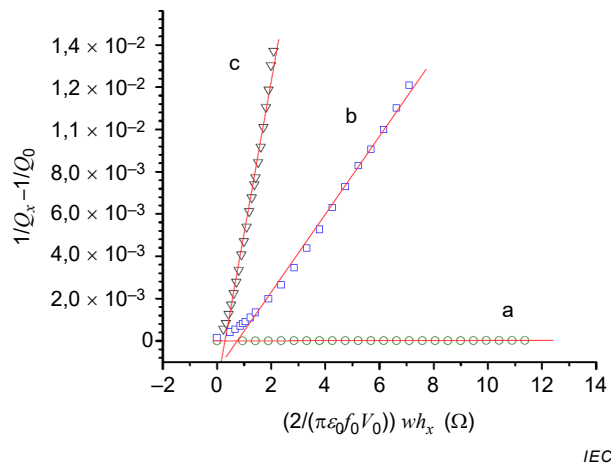


Figure A.1 – S_{21} magnitude of the resonant peak TE_{103} as a function of frequency at several specimen insertions (h_x)

A.5 Surface conductance of single-layer graphene and few-layer graphene

Figure A.2 shows graphical representation of Formula (A.5), using experimental Q_x and h_x data for 1LG and FLG, shown in Figure A.1. The solid lines are linear fits to Formula (A.5) through the data points.



Key

- a silica substrate
- b single-layer graphene
- c few-layer graphene

Lines are linear fits to Formula (A.5) through data points.

Figure A.2 – Plots of $1/Q_x - 1/Q_0$ as a function of the normalized specimen area (wh_x).

The surface conductance, σ_G , is obtained from the slope of the linear portion of the plots where the parameter b_q in Formula (A.5) can be neglected.

For single-layer graphene, $\sigma_G = 1,84 \times 10^{-3} \text{ S}$ ($\rho_G = 543 \text{ }\Omega$).

In the case of few-layer graphene, $\sigma_G = 7,19 \times 10^{-3} \text{ S}$ ($\rho_G = 139 \text{ }\Omega$).

Surface conductance of the silica substrate is about $5,1 \times 10^{-7} \text{ S}$.

The combined relative uncertainty in σ_G is approximately 1,5 %.

A.6 Summary

The presented surface conductance results qualitatively agree with the published data for similar graphene materials [9,10], though they may reflect the particular morphologies, density of states and structural imperfections resulting from the particular CVD process. Graphene material (up to 10 layers) is considered to be a two-dimensional conductor, for which the surface conductance σ_s is the appropriate measure of its electrical conduction. Volume conductance (σ_v) of thicker layers can be obtained by dividing σ_s by the layer thickness.

The effect of conductivity of the substrate on the measurement of conductivity of graphene can be corrected using the procedure described in the Note of Clause 8 and in reference [6].

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