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TECHNICAL SPECIFICATION

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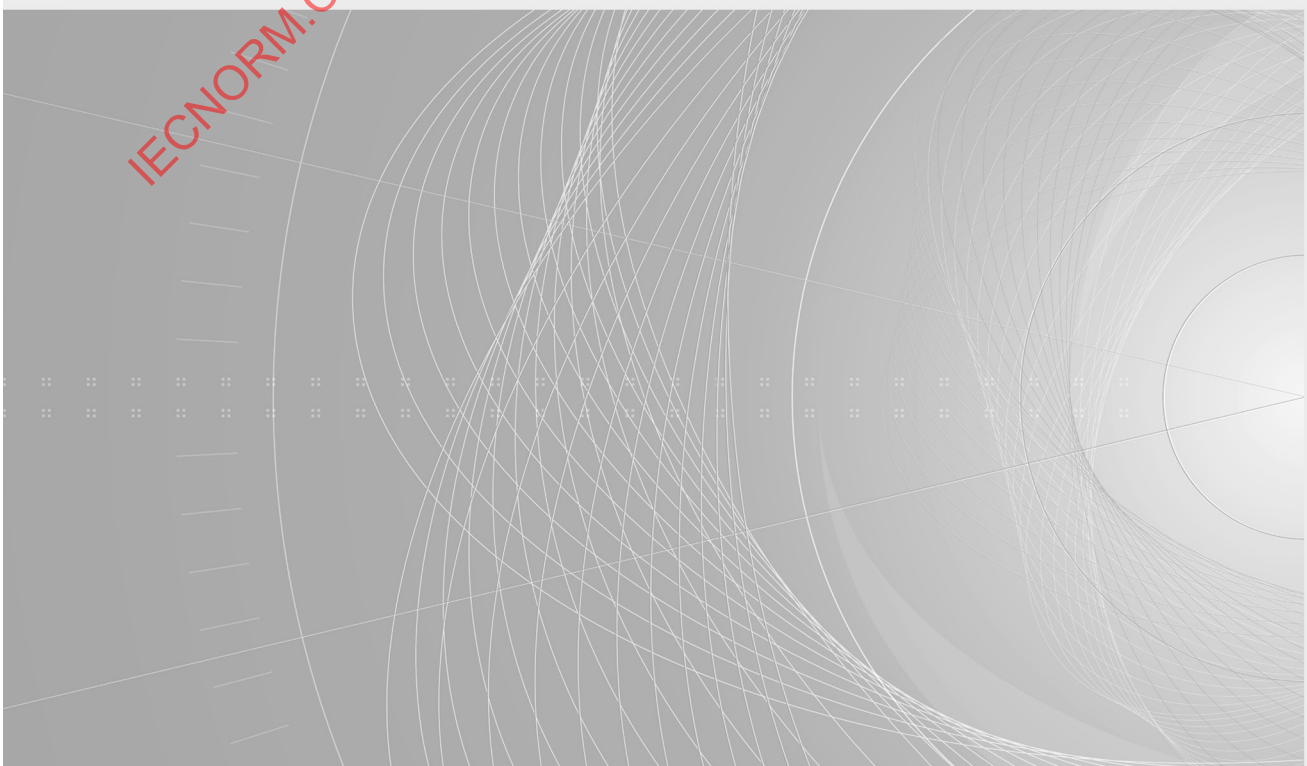
Nanomanufacturing – Key control characteristics – Part 6-4: Graphene-based materials – Surface conductance: non-contact microwave resonant cavity method



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NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 6-4: Graphene-based materials – Surface conductance: non-contact microwave resonant cavity method

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

This second edition cancels and replaces the first edition published in 2016. This edition constitutes a technical revision.

This edition includes the following significant technical changes with respect to the previous edition:

a) changed the document title to better reflect its purpose and application:

old title: Graphene – Surface conductance measurement using resonant cavity

new title: Graphene based materials – Surface conductance: non-contact microwave resonant cavity method.

b) replaced former Figure 1 with new Figure 1 and Figure 2, to better illustrate the method's fundamentals and its implementation for a non-technical reader.

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

Draft	Report on voting
113/756/DTS	113/809/RVDTS

Full information on the voting for its approval can be found in the report on voting indicated in the above table.

The language used for the development of this Technical Specification is English.

This document was drafted in accordance with ISO/IEC Directives, Part 2, and developed in accordance with ISO/IEC Directives, Part 1 and ISO/IEC Directives, IEC Supplement, available at www.iec.ch/members_experts/refdocs. The main document types developed by IEC are described in greater detail at www.iec.ch/publications.

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 webstore.iec.ch in the data related to the specific document. At this date, the document will be

- reconfirmed,
- withdrawn, or
- amended.

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INTRODUCTION

The microwave cavity test method for surface conductance is non-contact, fast, and accurate. It is well suited for standards development, 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. They can be stacked, folded, crumpled, or pillared into a variety of nano-carbon architectures with the vertical dimension limited to a few tenths of a nanometre. Many of these 2D materials, and their derivatives, 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][1], [2], [3].

Depending on particular morphologies, density of states, and structural perfection, the surface conductance of these materials can vary from1 S to about 10^{-5} S. Conventional direct current (DC) surface conductance measurement techniques require a complex test vehicle and interconnections for making electrical contacts to such materials, which affect and distort the measurement, thus, making it difficult to resolve the intrinsic properties of the material from the artifacts associated with the electrical contact formation.

In comparison, the resonant cavity measurement method is non-contact, fast, and avoids the artifacts associated with the electrical contact formation. 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 without the need for establishing electrical contacts or sample thickness.

[1] Numbers in square brackets refer to the Bibliography.

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 6-4: Graphene-based materials – Surface conductance: non-contact microwave resonant cavity method

1 Scope

This part of IEC 62607 establishes a standardized method to determine the key control characteristic

- surface conductance

for films of graphene and graphene-based materials by the

- non-contact microwave resonant cavity method

The non-contact microwave resonant cavity method monitors the microwave resonant frequency shifts and changes in the cavity's quality factor during the insertion of the specimen into the microwave cavity, as a function of the specimen surface area. The empty cavity is an air-filled standard R100 rectangular waveguide operated at one of the resonant frequency modes, typically at 7,5 GHz [4].

– The method is applicable for graphene materials which are synthesized by chemical vapour deposition (CVD) on metal substrates, epitaxial growth on silicon carbide (SiC), obtained from reduced graphene oxide (rGO), or mechanically exfoliated from graphite [5].

– 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 dimensions are uniform over the specimen area.

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

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.

ISO/TS 80004-13, *Nanotechnologies – Vocabulary – Part 13: Graphene and related two-dimensional (2D) materials*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO/TS 80004-13 and the following apply.

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

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

3.1 Graphene layers

3.1.1
graphene
graphene layer
single-layer graphene
monolayer graphene
single layer of carbon atoms with each atom bound to three neighbours in a honeycomb structure

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

Note 2 to entry: As graphene is a single layer, it is also sometimes called monolayer graphene or single-layer graphene and abbreviated as 1LG to distinguish it from bilayer graphene (2LG) and few-layer graphene (FLG).

Note 3 to entry: Graphene has edges and can have defects and grain boundaries where the bonding is disrupted.

[SOURCE: ISO/TS 80004-13:2017, 3.1.2.1]

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".

[SOURCE: ISO/TS 80004-13:2017, 3.1.2.6]

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 "twisted trilayer graphene".

[SOURCE: ISO/TS 80004-13:2017, 3.1.2.9]

3.1.4
few-layer graphene
FLG
two-dimensional material consisting of three to ten well-defined stacked graphene layers.

[SOURCE:ISO/TS 80004-13:2017, 3.1.2.10]

3.1.5
graphene oxide
GO
chemically modified graphene prepared by oxidation and exfoliation of graphite, causing extensive oxidative modification of the basal plane.

Note 1 to entry: Graphene oxide is a single-layer material with a high oxygen content, typically characterized by C/O atomic ratios of approximately 2,0 depending on the method of synthesis.

[SOURCE: ISO/TS 80004-13:2017, 3.1.2.13]

3.1.6
reduced graphene oxide
rGO
reduced oxygen content form of graphene oxide

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.

Note 3 to entry: It can take the form of several morphological variations such as platelets and worm-like structures.

[SOURCE: ISO/TS 80004-13:2017, 3.1.2.14]

3.1.7
graphene-based material
GBM
graphene material
grouping of carbon-based 2D materials that include one or more of graphene, bilayer graphene, few-layer graphene, graphene nanoplate, and functionalized variations thereof as well as graphene oxide and reduced graphene oxide.

Note 1 to entry: "Graphene material" is a short name for graphene-based material.

3.2 Measurement terminology

3.2.1
surface conductance
sheet 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: $G = I/U = \sigma_s \cdot (w/l)$.

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
electrical conductivity
σ_v
characteristic physical property of 3D materials describing the ability to conduct electric current.

Note 1 to entry: The electrical conductivity can be obtained from surface conductance dividing it by the conductor thickness (t), with $\sigma_v = \sigma_s/t$. The unit of measure of σ_v is siemens per metre (S/m).

3.2.3
surface resistance
sheet resistance
ρ_s
reciprocal of surface conductance, σ_s

Note 1 to entry: Sheet resistance measurements are commonly made to characterize the uniformity of conductive or semi-conductive coatings for quality assurance. 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. This is to avoid confusion between surface resistance and electrical resistance (R), which share the same unit of measure.

3.2.4
microwave cavity
radio frequency cavity
RF cavity
special type of 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.

Note 2 to entry: 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.

3.2.5
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)

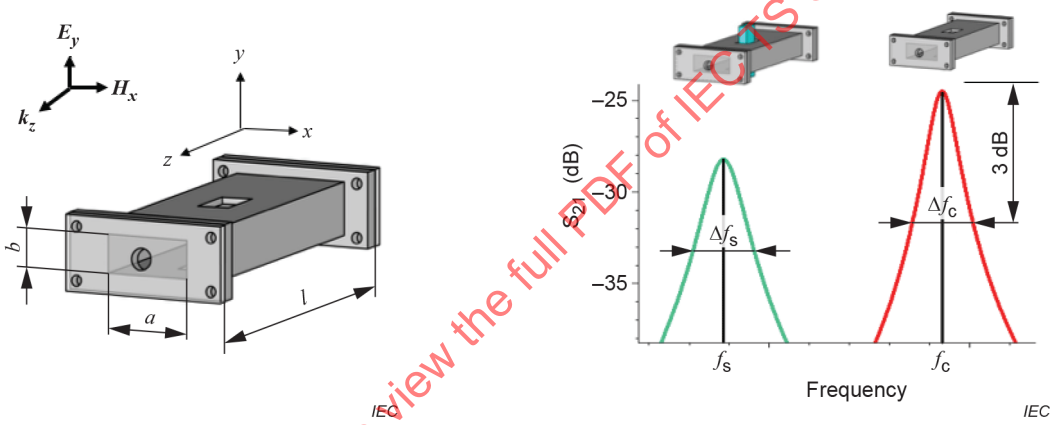
Note 1 to entry: $Q = f_r/\Delta f$

3.2.6
microwave scattering parameter
S-parameter
S_{ij}
factor that quantifies 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 source (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 testing structure

Figure 1 a) illustrates geometry of the waveguide resonating structure (cavity) with the wave propagating along vector k_z. The waveguide is short terminated on both ends with near-zero impedance walls. This allows excitation of standing waves (TE_{10n} modes) at specific resonant frequency, where the subscript n indicates the longitudinal mode number excited along the cavity length. Insertion of a specimen in the centre of the cavity [Figure 1 b)] causes the resonant peak to shift to lower frequencies in proportion to the specimen dielectric constant, while the resonant peak area decreases in proportion to the specimen conductivity.



a) Waveguide terminated with near-zero impedance walls. When implemented using R100 standard waveguide with dimensions a = 24,6 mm, b = 10,16 mm, l = 135 mm, the structure can operate as a resonator in the frequency of 6,5 GHz to 13 GHz.

b) Specimen insertion into cavity causes the resonant peak to shift towards lower frequencies and a decrease in its area, in proportion to specimen conductivity.

Figure 1 – Microwave cavity test structure

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 200 µm to 250 µm thick electronic grade fused silica wafer. The graphene material to be tested should be transferred onto the substrate surface using a process that preserves the structural integrity and purity of the graphene layer, while minimizing the possibility of contamination. The method requires measuring both the coated and uncoated substrate. The typical size of test specimen is 3 mm × 20 mm.

Epitaxial SLG can be grown by heating the SiC single crystal in a high vacuum or in an inert gas atmosphere [6]. The SiC surfaces after graphene growth contain Si- and C-terminated faces. On the Si-face the process stops at the homogeneous SLG. A few-layer graphene (FLG) can be grown with a controlled number of layers on the C-terminated face. Epitaxial graphene grown on silicon carbide (SiC) may be tested directly on the native substrate after removing either the Si- or C-terminated face. The carrier mobility reaches as high as several

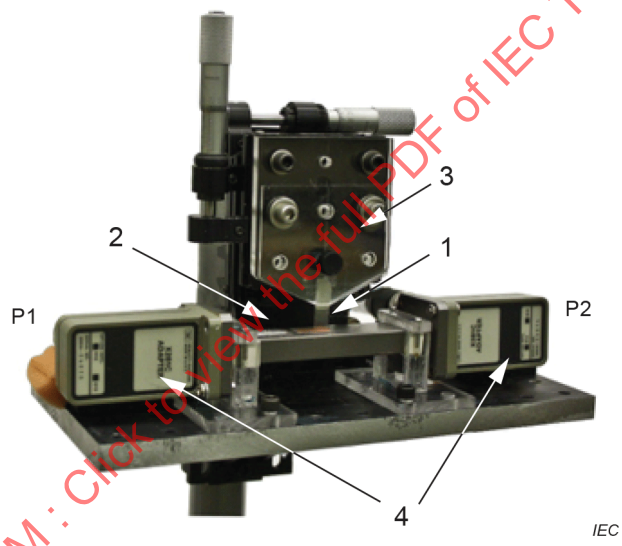
thousand $m^2V^{-1}s^{-1}$. On the C-face, twisted bilayer graphene can be grown. From the application point of view, graphene on SiC can be the platform used to fabricate high-speed electronic devices [7].

NOTE From the application point of view, graphene on SiC will be used as a standard reference graphene material and the platform used to fabricate high-speed electronic devices and dense graphene nanoribbon arrays, which will be used to introduce a bandgap [2], [3], [7].

6 Measurement procedure

6.1 Apparatus

Figure 2 illustrates an example of testing fixture that consists of a R100 waveguide (2), with recommended voltage reflected standing wave ratio (VRSW) of 1,04:1 or less, and the insertion loss 0,5 dB/m or less. The waveguide operates in the microwave frequency range of 6,5 GHz to 13,0 GHz. The walls terminating the cavity on both ends are implemented using coaxial to R100 waveguide couplers (4), which are near-cross-polarized (θ = 87°) 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 (empty) waveguide with the relative permittivity ε_r = 1,0, $l_e \approx l$, which is used as reference in this measurement.



The specimen (1) is inserted into the cavity (2) through a narrow slot of 1 mm × 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 odd modes TE_{10n}, where n = 1, 3, 5, … The specimen is attached to a stage (3) that controls and measures the specimen area inside the cavity. The fixture is connected to ports P1 and P2 of a vector network analyser by using cross-polarized coaxial to R100 waveguide couplets (4).

Figure 2 – Microwave cavity testing fixture

The measurement requires an automatic two-port vector network analyser (VNA) operating in the frequency range that covers the frequency band (6 GHz to 13 GHz) with the capability of measuring the microwave scattering parameters S_{21} or S_{12} transmitted between P1 and P2 of the VNA. Connections between the test fixture (Clause 5) and the VNA should be made using high quality coaxial cables and appropriate adapters. The dynamic range of the measurements should be within 70 dB. The instrument should be equipped with an IEEE 488 I/O interface or equivalent, for transferring data between the VNA and a data collection unit.

6.2 Calibration

Calibration is required only at the coaxial ends connecting the VNA to R100 couplers. Two-port full calibration for S_{21} and S_{12} should be performed in accordance with the VNA 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_{10n} modes, where n = 1, 3, 5, …. The first resonant mode, TE_{101}, can fall below the waveguide cut-off frequency of 6,557 GHz, if the cavity is electrically too long. Therefore, the test method uses the mode 1,0,3, which can be easily identified at frequency of about 7,5 GHz. 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, …) remain intact.

6.3.2 Empty cavity

Set the centre frequency to f_0 and the frequency span to about $2\Delta f$ (10 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).

$$Q_0 = \frac{f_0}{\Delta f} \tag{1}$$

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. Record the area (A_x) of the sample inside the cavity at each step (h_x).

$$A_x = w h_x \tag{2}$$

where w is the width of the test specimen. When the cavity is perturbed by the specimen, the frequency peak 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 6.3.1 if necessary. Record the resonant frequency f_x, half power bandwidth Δf and the corresponding quality factor Q_x (Formula (1)).

NOTE The active length (h_x) perturbing the cavity volume corresponds to the portion of the specimen inside the cavity). The typical value of Q_0 is about 3 200, which can be measured with uncertainty ΔQ ± 5,0 [4]. Therefore, the minimum insertion h_{xo} which causes the quality factor to change from its initial value Q_0 can be determined experimentally from ΔQ.

6.3.4 Repeated procedure

Repeat the steps in 6.3.2 recording Q_x and A_x, until $h_x \leq 10$ mm ($h_x \leq a$) or when in the case of highly conducting samples the value of Q_x decreases to about 100 ($Q_x \approx 100$) or the resonant peak height, $|S_{21}|_{max}$, decreases to about 10 dB above the noise level.

NOTE The operating menu of a typical automatic vector 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 programme for automated calculation of the quality factor after each specimen insertion step.

6.3.5 Substrate

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

7 Calculations of surface conductance

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

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

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 at insertion h_x;

Q_0 is the quality factor and resonant frequency of the empty cavity;

f_0 is the 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.

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

where

$$k = \frac{2}{\pi \varepsilon_0 f_0 V_0}.$$

Thus, σ_s can be solved from the slope of a linear portion of plot $(1/Q_x - 1/Q_0)$ versus $(k w h_x)$ (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 $(1/Q_x - 1/Q_0)$ versus $(k w h_x)$ 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;
- measured surface conductance for specimen of bare substrate, σ_{sub};
- surface conductance, σ_G, for the graphene layer: $\sigma_G = \sigma_s - \sigma_{sub}$;

NOTE The typical surface conductance value of graphene materials can be between 1 S (for a multilayer 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. When the film consists of a not percolated semi-continuous network of graphene flakes, 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, the measurement results can be unreliable. In such cases a correction model ca be used as described in reference [8]. Example measurements are illustrated in Figure A.1 and Figure A.2 for single layer and multi-layer CVD graphene deposited on a fused silica substrate (see Annex A).

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 vector network analyser, about ±0,01 dB for the magnitude and ±05° 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 1 %. 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} [8]. Comparison of calculation techniques for quality factor influenced by the measurement uncertainty of the vector network analyser is given in reference [9].

Additional limitations may arise from the systematic uncertainty of the instrumentation, calibration standards, 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 $\varepsilon_s^* = \varepsilon_s' + \varepsilon_s''$, is given by Formulas (A.1) to (A.3), where the complex integral, C_v, given by Formula (A.3), accounts for non-uniform fields [4], [8].

$$\frac{f_0 - f_s}{f_0} = (\varepsilon_s' - 1)\frac{2V_s}{V_0} - b_x \tag{A.1}$$

$$\frac{1}{Q_s} - \frac{1}{Q_0} = \varepsilon_s'' \frac{4V_s}{V_0} - b_y \tag{A.2}$$

$$C_v = b_x + b_y = \frac{2\int_{V_s} E_s E_0 \mathrm{d}V}{\int_{V_0} E_0^2} \tag{A.3}$$

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_s, is small compared to volume of the cavity, V_0, (V_s << V_0) and the sample permittivity does not depend on V_s. In the range of Q_s, and V_s values where C_v = constant, Formulas (A.1) and (A.2) are linear and can be solved for ε_s' by measuring Q_0, Q_s, f_0 and f_s as a function of V_s [4]. Inserting into cavity, tuned at the resonant frequency f_0, a low loss substrate, having permittivity $\varepsilon_{sub}^* = \varepsilon_{sub}' - j\varepsilon_{sub}''$, thickness t_{sub} and volume V_{sub} causes the resonant frequency shift to lower frequencies from f_0 to f_{sub}, which is proportional to ε_{sub}'. The corresponding quality factor decreases from Q_0 to Q_{sub}, which depends on ε_{sub}''. Similar experiments with nominally the same substrate coated with a conducting layer of graphene material, having thickness t_G and volume V_G causes the quality factor to decrease from Q_0 to Q_G, which includes combined losses of the substrate and the graphene layer. Since the dielectric loss of substrate is much smaller than that of graphene and can be neglected, $\varepsilon_{sub}'' \approx 0$, Formula (A.2) can be re-rearranged into Formula (A.4) as shown in reference [4]:

$$\frac{1}{Q_x} - \frac{1}{Q_0} = \frac{\sigma_G}{\pi \varepsilon_0 f_0} \frac{2 w h_x}{V_0} - 2 b_q \quad \text{(A.4)}$$

where:

σ_G is the surface conductance of the graphene layer;

ε_0 is the permittivity of free space;

$w\,h_x$ 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.4), from which σ_G is determined, does not depend on the thickness of the graphene layer but on its area, $w\,h_x$, which can be determined with much higher accuracy than the graphene thickness t_G [4].

A.3 Experimental procedure

Single-layer graphene (1LG) [6] and few-layer graphene (FLG) from a CVD process [10] were obtained from a commercial source and transferred on to 200 µm thick fused silica wafers, from which 3 mm × 20 mm specimens were extracted by dicing.

The cavity test fixture design shown in Figure 2 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 vector network analyser[2]) with semi-rigid coaxial cables and coaxial-to-R100 adapters. The walls of the cavity are implemented via R100 couplers, which are cross-polarized (φ = 87°) 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 TE_{103} to TE_{109} odd resonant mode, 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 5 Here, f_0 of the TE_{103} peak is 7,319 112 5 GHz, Q_0 = 3 000 and V_0 = 33,491 cm^3.

A.4 Results

Figure A.1 shows the magnitude of S_{21}, at the TE_{103} resonant peak measured for SLG 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.4). 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.

2 Agilent N5225 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.