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TECHNICAL SPECIFICATION

colour inside

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



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CONTENTS

FOREWORD 3
INTRODUCTION 5
1 Scope 6
2 Normative references 6
3 Terms and definitions 6
3.1 Graphene layers 7
3.2 Measurement terminology 8
4 Microwave cavity testing structure 9
5 Test specimen 9
6 Measurement procedure 10
6.1 Apparatus 10
6.2 Calibration 10
6.3 Measurements 11
6.3.1 General 11
6.3.2 Empty cavity 11
6.3.3 Specimen 11
6.3.4 Repeated procedure 11
6.3.5 Substrate 12
7 Calculations of surface conductance 12
8 Report 12
9 Accuracy consideration 13
Annex A (informative) Case study of surface conductance measurement of single-layer and few-layer graphene 14
A.1 General 14
A.2 Cavity perturbation procedure 14
A.3 Experimental procedure 15
A.4 Results 15
A.5 Surface conductance of single-layer graphene and few-layer graphene 16
A.6 Summary 17
Bibliography 18

Figure 1 – Microwave cavity test structure 9
Figure 2 – Microwave cavity testing fixture 10
Figure A.1 – S_{21} magnitude of the resonant peak TE_{103} as a function of frequency at several specimen insertions (h_x) 16
Figure A.2 – Plots of $1/Q_x - 1/Q_0$ as a function of the normalized specimen area (wh_x). 16

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NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

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

FOREWORD

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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)

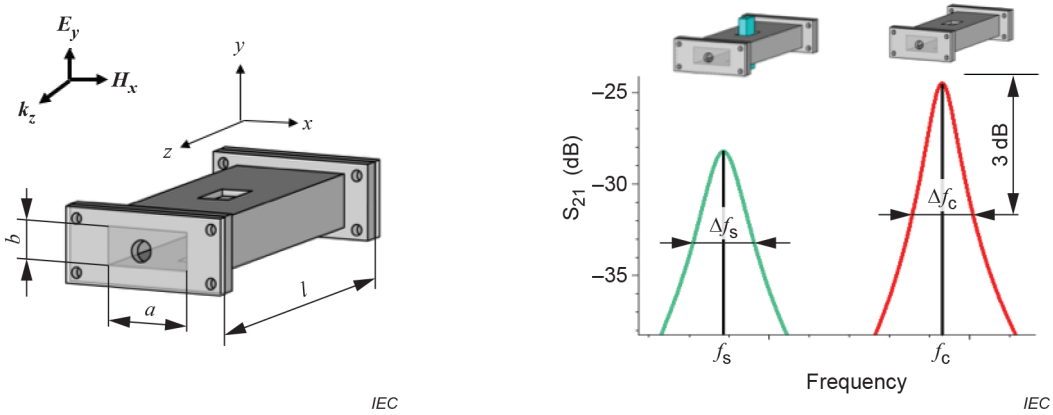
Note 1 to entry: $Q = f_r/\Delta f$

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