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TECHNICAL SPECIFICATION

colour inside

Nanomanufacturing – Key control characteristics – Part 6-10: Graphene-based material – Sheet resistance: Terahertz time-domain spectroscopy

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IEC TS 62607-6-10 has been prepared by IEC technical committee 113: Nanotechnology for electrotechnical products and systems. It is a Technical Specification.

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

Draft	Report on voting
113/568/DTS	113/604/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/standardsdev/publications.

A list of all parts of the IEC TS 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- withdrawn,
- replaced by a revised edition, or
- amended.

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INTRODUCTION

Graphene is an important nanomaterial in R&D and industry due to its outstanding electrical properties. It is already present in multiple commercial products, and furthermore, it is a strong candidate as an electrical material in numerous new application areas. However, no established method to characterize its local electrical performance and quality across large areas exists yet. The four-point probe method, either as single point or mapping (scanning) technique, is an industry standard for silicon wafers and conventional thin films, but unavoidably leads to damage, due to the physical contact between the tip and the one atom thin graphene film. The microwave resonant cavity method has been demonstrated as a mapping technique for graphene, but with spatial and sample resolution limited by the cavity size: no attempt has been made to scale this technique to industrially relevant sample sizes. Other methods for providing spatial information relating in some way to electrical quality include optical, Raman and scanning electron microscopies. These ones give local information that only indirectly relates to the electrical properties of interest.

The focus of this document is to provide a method to characterize the electrical performance, quality and uniformity of large-area graphene films with terahertz time-domain spectroscopy (THz-TDS). THz-TDS allows for large-area mapping of graphene films in a non-destructive, fast and robust mode, without contact and with no sample preparation at all. This method has no upper limitations in the size of the graphene film to be analysed. It is applicable for statistical process control, comparison of graphene films produced by different vendors, obtaining information about imperfections on the microscale such as grain boundaries and defects, and uniquely allows process modifications and development to be analysed step by step due to its non-destructive property and ability to access buried conductive layers. THz-TDS has been tested against other methods such as van der Pauw (vdP), electrical resistance tomography and calibrated Kelvin probe force microscopy with good matching of results [1] [2][1].

THz-TDS method provides direct measurements of the sheet resistance, both in transmission and reflection modes [3]. The spatial resolution is related with the diffraction limited THz beam spot size, reaching about 300 µm at 1 THz, and the maximum surface density of measurements is determined by the minimum step-size of the actuator moving the sensor or the sample.

The default sample in this document is monolayer graphene grown by chemical vapour deposition (CVD) on or transferred to a quartz substrate. Nevertheless, the methodology can be extended to graphene on silicon carbide (epitaxial graphene), multilayer graphene, and thin conductors generally, including other 2D materials, on several other dielectric and high resistive substrates including sapphire, silicon coated with silicon dioxide, silicon carbide, polymers and III-V semiconductors, among others. It is noted that for the reflection-mode THz-TDS, the technique tolerates less THz-transparent substrates (e.g. medium to highly doped silicon) than the transmission-mode THz-TDS.

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

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 6-10: Graphene-based material – Sheet resistance: Terahertz time-domain spectroscopy

1 Scope

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

– sheet resistance (R_s)

for films of graphene-based materials by

– terahertz time-domain spectroscopy (THz-TDS).

In this technique, a THz pulse is sent to the graphene-based material. The transmitted or reflected THz waveform is measured in the time domain and transformed to the frequency domain by the fast Fourier transform (FFT). Finally, the spectrum is fitted to the Drude model (or another comparable model) to obtain the sheet resistance.

- This non-contact inspection method is non-destructive, fast and robust for the mapping of large areas of graphene films, with no upper sample size limit.
- The method is applicable for statistical process control, comparison of graphene films produced by different vendors, or to obtain information about imperfections on the microscale such as grain boundaries and defects, etc.
- The method is applicable for graphene grown by chemical vapour deposition (CVD) or other methods on or transferred to dielectric substrates, including but not limited to quartz, silica (SiO_2), silicon (Si), sapphire, silicon carbide (SiC) and polymers.
- The minimum spatial resolution is in the order of 300 μm (at 1 THz) given by the diffraction limited spot size of the THz pulse.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions 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 General terms

3.1.1
graphene
graphene layer
single-layer graphene
monolayer graphene
1LG
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 80004-3:2020, 3.1.13]

3.1.2
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.

3.1.3
thin film
conductive, resistive or dielectric material, usually less than 50 000 Å in thickness, that is deposited onto a substrate by vacuum evaporation, sputtering, or other means

[SOURCE: IEC 60748-23-2:2002, 3.64]

3.1.4
two-dimensional material
2D material
material, consisting of one or several layers with the atoms in each layer strongly bonded to neighbouring atoms in the same layer, which has one dimension, its thickness, in the nanoscale or smaller and the other two dimensions generally at larger scales

Note 1 to entry: The number of layers when a two-dimensional material becomes a bulk material varies depending on both the material being measured and its properties. In the case of graphene layers, it is a two-dimensional material up to 10 layers thick for electrical measurements, beyond which the electrical properties of the material are not distinct from those for the bulk (also known as graphite).

Note 2 to entry: Interlayer bonding is distinct from and weaker than intralayer bonding.

Note 3 to entry: Each layer may contain more than one element.

Note 4 to entry: A two-dimensional material can be a nanoplate.

[SOURCE: ISO/TS 80004-13:2017, 3.1.1.1]

3.1.5
diffusive conductor
conductor where the dimensions in which electron transport takes place are significantly larger than the mean free path

3.1.6
mean free path
product of the momentum relaxation time and the Fermi velocity

Note 1 to entry: Mean free path can be estimated from the Drude-Boltzmann transport theory:

$$L_{\mathrm{mfp}} = \frac{\mu h}{2e}\sqrt{\frac{N}{\mu}}$$

where h is the Planck's constant, e is the electron charge, μ is the carrier mobility and N is the carrier density.

Note 2 to entry: CVD graphene on SiO_2 has mean free paths in the range 1 nm to 200 nm at room temperature for a doping level of 10^{12} cm^{-2} (corresponding to carrier mobility being in the μ = 100 cm^2/Vs to 20 000 cm^2/Vs range) while CVD graphene encapsulated in hexagonal boron nitride can have mean free paths up to 1 µm to 2 µm at room temperature (corresponding to the $\mu = 1 \times 10^6$ cm^2/Vs to 2×10^6 cm^2/Vs range)

Note 3 to entry: The relationship between sheet conductivity σ_s, carrier density N and carrier mobility μ is given by:

$\sigma_s = Ne\mu$.

3.2 Key control characteristics measured according to this document

3.2.1
key control characteristic
KCC
key performance indicator
material property or intermediate product characteristic which can affect safety or compliance with regulations, fit, function, performance, quality, reliability or subsequent processing of the final product

Note 1 to entry: The measurement of a key control characteristic is described in a standardized measurement procedure with known accuracy and precision.

Note 2 to entry: It is possible to define more than one measurement method for a key control characteristic if the correlation of the results is well-defined and known.

3.2.2
sheet resistance
R_S
measure of resistance of thin films that are nominally uniform in thickness

Note 1 to entry: Two-dimensional (x-y) sheet resistance (Rs) can be determined for electrically uniform thin films. In rectangular geometry $R_s = R/(L/w)$, where R is the measured resistance, $R = V/I$, L is the distance between parallel electrodes, between which the voltage drop (V) is measured, and w is the length of these electrodes. The electrical current (I) must flow along the plane of the sheet, not perpendicular to it (see Figure 4). The ratio L/w represents the number of squares of the film specimen.

Note 2 to entry: Sheet resistance is expressed in ohms (Ω). However, for the purpose of this procedure, Ω represents the unit ohm/square (Ω/sq).

3.2.3
resistivity
ρ
resistance per unit length of a material of unit cross-sectional area

Note 1 to entry: For a uniform conductor with a uniform cross-section, the relationship between resistivity and resistance is given by: $R = \rho \frac{l}{A}$

where

A is the cross-sectional area of the conductor;

l is the length of the conductor

The unit of resistivity is the ohm metre (Ω·m).

Note 2 to entry: For a non-uniform conductor, there is in general no simple relationship between resistivity and resistance.

[SOURCE: ISO 15091:2019, 3.2, modified – Note 1 to entry has been slightly reformulated. Note 2 has been added.]

3.2.4
sheet conductance
G_S
inverse of sheet resistance

$$G_S = 1/R_S$$

3.2.5
electrical conductivity
σ
reciprocal of the resistivity

Note 1 to entry: Electrical conductivity is given by $\frac{1}{\rho} = \frac{1}{R} \times \frac{l}{A}$. The unit of electrical conductivity is the siemens reciprocal metre ($S \cdot m^{-1}$).

Note 2 to entry: The reciprocity is only valid for conductors without directional dependence of the conductivity.

[SOURCE: ISO 15091:2019, 3.4, modified – The symbol γ has been replaced by σ. Note 2 to entry has been added.]

3.2.6
mobility
drift mobility
μ
<of a charge carrier> quantity equal to the ratio of the modulus of the mean velocity of the charge carriers in the direction of an electric field by the modulus of the field strength

[SOURCE: IEC 60050-521:2002, 521-02-58]

3.2.7
charge carrier density
N
density of mobile electrons and/or holes in a material

Note 1 to entry: Expressed in cm^{-3}.

[SOURCE: IEC 62341-1-2:2014, 2.3.1]

3.3 Terms related to the measurement method described in this document

3.3.1
terahertz time-domain spectroscopy
THz-TDS
method to measure the complex-valued dielectric function or conductivity of a material in the terahertz (THz) frequency range (typically 0,1 THz to 5 THz) by the measurement of the temporal shape of an electromagnetic pulse with a duration in the range of picosecond, either reflected from or transmitted through the sample

Note 1 to entry: The amplitude and phase of the frequency components of the signal are compared to those of a reference signal, and can be related to the complex refractive index, permittivity or conductivity of the sample.

3.3.2
signal-to-noise ratio
SNR
ratio of the amplitude of the time trace of the terahertz (THz) electric field signal to the root-mean-square of the noise time trace (measured with the THz beam path blocked)

Note 1 to entry: SNR may be expressed as a level difference in decibels.

Note 2 to entry: SNR can be used to estimate the usable bandwidth of the spectrometer.

3.3.3
dynamic range
DNR
ratio of the amplitude of the frequency trace of the terahertz (THz) electric field signal to the amplitude of the noise frequency trace (measured with the THz beam path blocked)

Note 1 to entry: DNR may be expressed as a level difference in decibels.

Note 2 to entry: DNR can be used to estimate the usable bandwidth of the spectrometer.

3.3.4
spot size
size of the terahertz beam spot on the sample

Note 1 to entry: The terahertz (THz) pulse contains a broad band of frequencies typically ranging from GHz up to several THz depending on the pulse duration. Therefore, the spot size of a THz beam can either be measured at a specific frequency within its bandwidth, or as an average value by a superposition of spot sizes at all frequencies, weighted by their spectral amplitude. The spot size is typically given as FWHM (full width at half maximum) of the spatial field distribution.

Note 2 to entry: The effective THz beam spot is typically measured using the knife-edge method. In this method, a knife-edge is introduced in the area illuminated by the THz beam. The size of the THz spot is measured when the power is half-reduced and it corresponds to the length of the knife-edge introduced.

Note 3 to entry: The effective THz beam spot can be measured with a THz camera.

3.3.5
Rayleigh range
distance from the focal plane of a Gaussian optical beam where the beam radius has increased by a factor of 1,41 (square root of 2)

Note 1 to entry: The Rayleigh range is computed as $Z_R = \pi w_0^2 / \lambda$, where w_0 is the beam radius in the focal plane and λ is the wavelength.

3.3.6
four point probe method
method to measure electrical sheet resistance of thin films that uses separate pairs of current-carrying and voltage-sensing electrodes

Note 1 to entry: The method is fast, repositionable and local, compared to using fixed electrodes.

Note 2 to entry: The method requires the probes making direct contact to the sample, as opposed to four-terminal measurements done via lithographically defined electrodes, i.e. in cloverleaf or Hall bar design.

[SOURCE: ISO/TS 80004-13:2017, 3.3.3.1, modified – In the definition, ", impedance or conductivity" has been deleted. Note 1 has been changed and Note 2 has been added.]

3.3.7
microwave resonant cavity method
method to measure surface conductance or equivalently sheet resistance by resonant cavity that 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

Note 1 to entry: The method is fast and non-contacting.

[SOURCE: ISO/TS 80004-13:2017, 3.3.3.7, modified – The term "non-contact microwave method" is replaced with "microwave resonant cavity method". In the definition, "cavity involves" is replaced with "cavity that involves".]

3.3.8
electrical resistance tomography
method to obtain maps of electrical conductivity of the interior of a two- or three-dimensional sample from a set of four-terminal resistance measurements performed at its boundary

4 General

4.1 Measurement principle

The method to measure the intrinsic resistivity and sheet resistance of a thin conducting film as described in this document is based on terahertz time-domain spectroscopy (THz-TDS). In this technique, an electromagnetic pulse with a typical duration of one picosecond (i.e. the terahertz pulse) is generated and its electric field amplitude and temporal shape is measured in the time domain, as the so-called THz waveform. Figure 1 shows the time trace of a typical THz waveform. The Fourier transform of the time-domain THz waveform provides access to the frequency components of the pulse, which typically spans from 0,1 THz up to 5 THz.