IEC TS 62607-6-10

Edition 1.0 2021-10

TECHNICAL SPECIFICATION

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

IEC TS 62607-6-10:2021-10(en)

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

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NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

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

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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.

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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

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- withdrawn,
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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.

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[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.

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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:

$\sigma_{\mathrm{s}} = Ne\mu$.

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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.

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[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.

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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.

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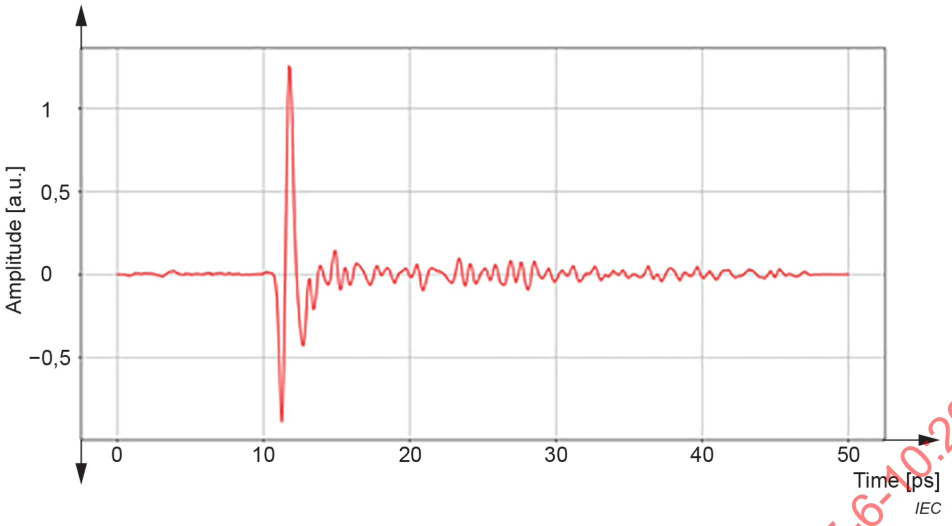

Figure 1 – Time trace of a typical THz waveform

To perform a measurement, the above-mentioned THz pulse is focused on a sample, defined in this document as a thin film of a conducting material (graphene) supported onto a dielectric substrate (see Figure 2). The interaction of the incident THz pulse with the conducting film modifies the amplitude and phase of the transmitted and reflected THz waveforms according to the complex refractive index of the material. The complex refractive index is related to the intrinsic complex resistivity of the material and, in turn, to the sheet resistance. Assuming that the frequency-dependent resistivity is Drude-like [4] or follows another comparable model, the measured resistivity spectrum can be fitted by the given model, and the best-fit values of parameters of the model can be determined.

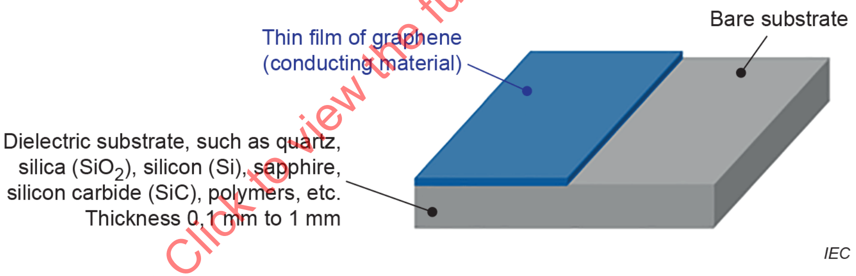

Figure 2 – Sample scheme comprised of a thin film of graphene on a dielectric substrate.

The sheet resistance (R_S) is an extrinsic, generally non-local property of a device and the result of an electrical measurement. The sheet resistivity (ρ_S) is an intrinsic, local property of a material, and is the 2D equivalent of 3D resistivity. For an electrically uniform thin film material, the sheet resistance and sheet resistivity are identical. While for many bulk 3D materials, such as pure metals with uniform conducting properties, a consistent value for the resistivity can be given, this is not generally the case for 2D materials, as these are more vulnerable to environmental conditions, in contact with foreign objects and surfaces, and very frequently contain tears, holes, gaps, cracks and other flaws that leads to a non-uniform current flow. Therefore, the electrical mapping of 2D materials/thin films and following assessment of the uniformity is essential for their correct characterization and THz-TDS provides a suitable method.

THz-TDS technique allows measuring the following electrical parameters:

- sheet resistance (R_S);
- sheet resistivity (ρ_S);
- sheet conductance ($G_S = 1/R_S$);
- sheet conductivity ($\sigma_S = 1/\rho_S$);

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- charge carrier mobility (μ);
- charge carrier density (N);
- substrate refractive index (n_{sub}).

It is important to highlight that the sheet resistance, sheet resistivity and charge carrier mobility can be only defined for diffusive conductors, such as graphene and other 2D materials.

Due to the 300 µm spot size (at 1 THz), THz-TDS measures an average of the local sheet conductivity across the spot, or more precisely, the average sheet conductivity weighted by the spot point spread function, or intensity distribution, of the beam spot.

There are two THz-TDS measurement modes: single-point mode and imaging mode.

– Single-point mode refers to a basic THz-TDS measurement where the THz beam is pointed at a relevant position on the sample. The sample signal is compared to a reference measurement to obtain the resistivity at that point with a resolution determined by the spot size.

– Imaging or mapping mode refers to raster scanning the beam over the sample in the focal plane. The signals are recorded as function of the position (x, y) in the sample and compared to one or several reference measurements. The result is a spatial resistivity or conductivity map [1], [2], [5], [6] .

For graphene or other 2D materials, mapping is required to study the distribution of their electrical parameters ($\rho_{\text{s}}, \sigma_{\text{s}}, N, \mu$) along the surface. The statistical distributions of these parameters are primary descriptors of the sample electrical uniformity and give information about flaws or grain boundaries in the sample. In contrast to electrical measurements, the THz-TDS method is measuring the intrinsic (local) sheet resistivity ρ_{s} averaged over an area corresponding to the THz spot size, weighted by the Gaussian beam spot intensity. THz-TDS can be performed in two measurement configurations: transmission and reflection, described in detail in 4.3.2.

Further information of the theoretical background and the measurement principle of the sheet resistance and conductance can be found in Annex C.

4.2 Sample preparation method

THz-TDS method requires no sample preparation, nor specific sample handling or storage. It can be advantageous for the measurement accuracy if a part of the sample surface is bare substrate (see Figure 2).

4.3 Description of measurement equipment

4.3.1 Principal components of a THz-TDS system

The principal components of a classical THz-TDS system are listed below and schematized in Figure 3, where the dotted area can be substituted by configurations shown in Figure 4 and Figure 5.

– Femtosecond laser (fs LASER): emits an optical femtosecond pulse.

– Beam splitter (BS): splits the laser output into beams used for THz generation and THz detection.

– Time delay stage: one of the arms of the beam splitter contains an optical delay line for the time scan of the THz transient.

– THz emitter (Tx): generates ultrashort THz pulses when excited by the femtosecond laser, typically in the form of a photoconductive antenna (PCA) or a nonlinear electro-optical crystal (EOX).

– THz detector (Rx): gated by the second part of the laser beam for time-resolved detection of the THz transient, typically another PCA or EOX. The detection signal (photocurrent from the PCA or phase retardation of a probe laser beam through the EOX) is detected by, for example, lock-in techniques and recorded as a function of the position of the optical time delay.

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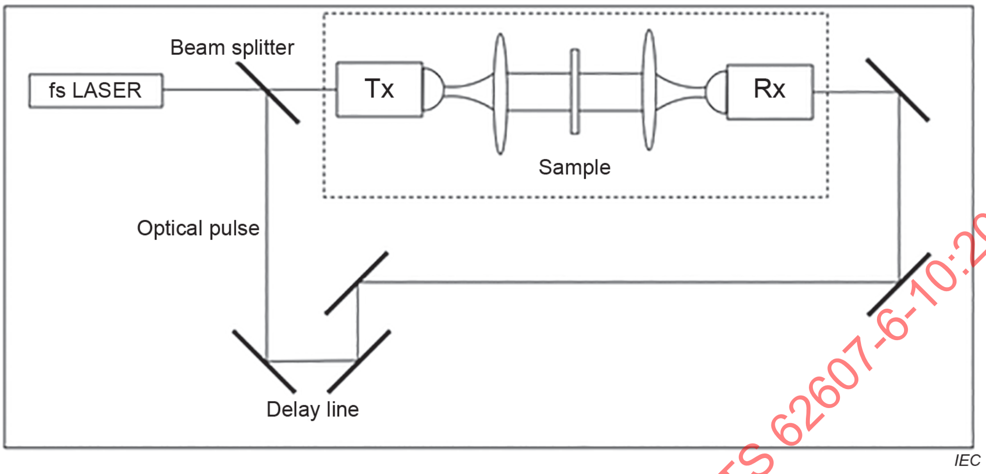
Other THz sources different from femtosecond laser-based sources can be also used in a THz-TDS system, such as electronic generation by microwave up-mixing. THz-TDS systems rely on ultrashort, broadband THz pulses, so the methodology is not compatible with the use of continuous-wave, narrowband THz sources and detectors. Complete THz-TDS systems are available from several commercial suppliers.



NOTE The dotted area can be substituted by any of the configurations shown in Figure 4 and Figure 5.

Figure 3 – Principal components of a classical THz-TDS system

Considerations regarding linearity of custom-made detection systems are described in Annex D, Clause D.1.

4.3.2 Measurement configurations

Two different measurement configurations are possible: transmission and reflection. The method does not require both reflection and transmission measurements to obtain resistance values, just one of them is enough. However, if both measurements are available, then the measurement error might get smaller:

- Transmission geometry. Emitter (Tx) and receiver (Rx) are placed in front of each other along the same optical axis. The sample is placed between them. The THz beam can be either collimated or focused by refractive optics (lenses; Figure 4a,b) or reflective optics (parabolic or elliptical mirrors; Figure 4c,d).
- Standard mathematical analysis of the signals recorded in the experiment assumes plane-wave excitation of the sample, so it is of importance that the sample is thin compared to the Rayleigh range Z_R in order to meet this approximation. In this context, thin substrates fulfil $nd \ll Z_R$, where n is the refractive index of the substrate and d is the thickness of the substrate.

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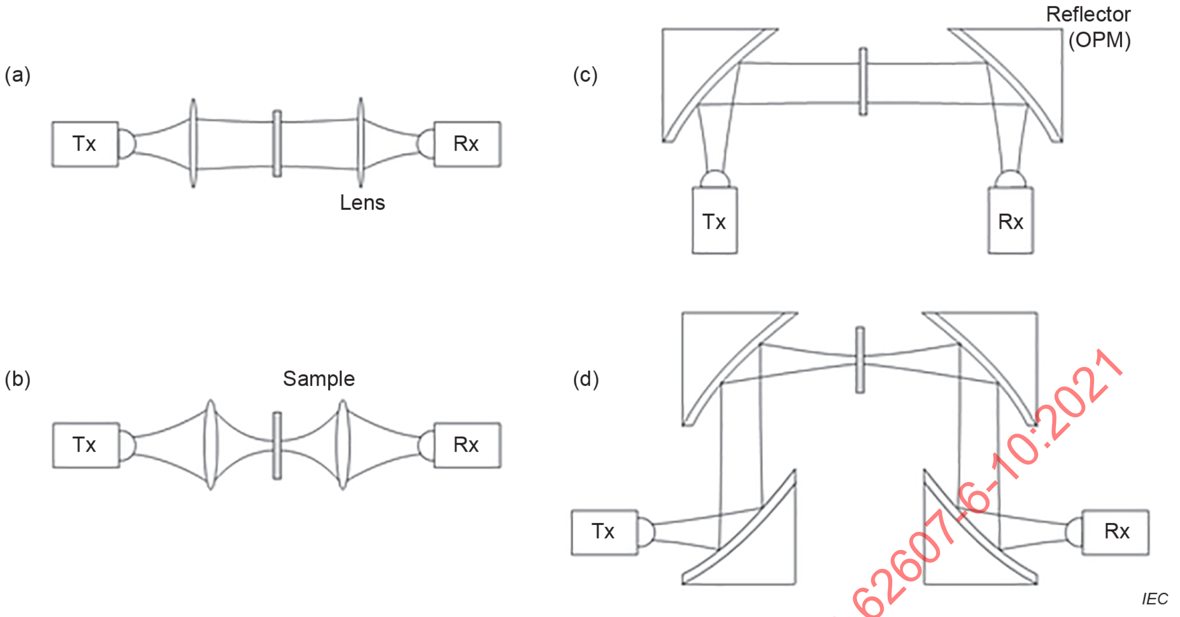

NOTE Collimated (a) and focused (b) geometry with refractive optical elements. Collimated (c) and focused (d) geometry with reflective optical elements.

Figure 4 – Comparison of different transmission geometries

- Reflection geometry. Tx and Rx are placed on the same side of the sample. There are two possible reflection geometries: shallow angle (or pitch-catch) and normal-incidence reflection (Figure 5). In pitch-catch, the THz beam illuminates the sample at an oblique angle and the specular reflection is captured and measured by the detector (Figure 5a). In normal-incidence reflection, the THz beam illuminates the sample perpendicular to the surface; the incident and reflected beams are separated by a beam splitter (Figure 5b).
- The advantages of normal incidence compared to pitch-catch are a tighter focal spot (allowing better spatial resolution) and better beam quality. However, the beam splitter introduces a minimum loss of 6 dB.
- In pitch-catch geometry, the spot size on the sample is enlarged by the factor $1/\cos\theta$, where θ is the incidence angle. Pitch-catch geometry offers a higher signal because losses are minimal, but due to the larger focal spot, spatial resolution is worse than in normal reflection.

Both geometries are similar from the parameter extraction point of view.

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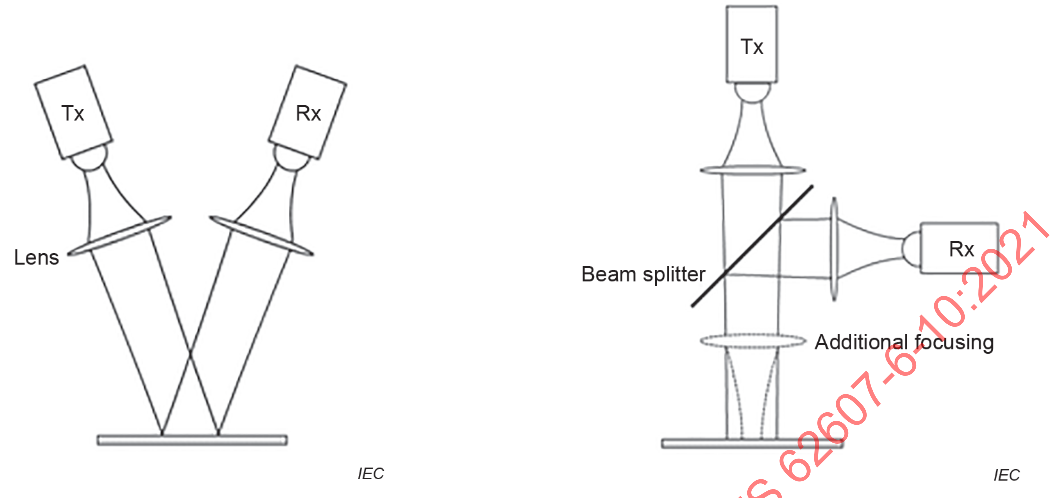

a) Pitch-catch geometry b) Normal-incidence geometry

Figure 5 – Comparison of different reflection geometries

Systems with a focused THz beam are much more sensitive to the exact positioning of the sample with respect to the focal plane. When working with focused beams, it is important to place the sample exactly in the focal plane of the THz beam.

The procedure to extract the parameters is more complex in reflection than in transmission geometry. On the other side, the reflection measurements are less sensitive to the precise thickness of the substrate than transmission measurements and this removes the requirement of a specialized THz-transparent substrate to measure the conductivity [5].

4.4 Supporting materials

Supporting materials are necessary to obtain reference signals, essential to calculate the sheet resistance of the sample. The selection of supporting materials depends on the measurement configuration.

- For transmission measurements, reference signals can be measured:
  - through the bare substrate of the sample to be measured, i.e. at a position on the sample without graphene (see Figure 2). The bare substrate shall be transmissive to THz radiation and the substrate thickness shall be constant along the whole sample surface (see 5.3 for accuracy dependence on substrate thickness);
  - through the air, without any sample in the beam path.
- For reflection measurements, reference signals can be measured on:
  - the bare substrate of the sample to be measured, i.e. at a position on the sample without graphene (see Figure 2). The substrate thickness shall be constant along the whole sample surface (see 5.3 for accuracy dependence on substrate thickness);
  - a reference material, such as a perfect reflector surface (e.g. a mirror or an optically polished metal).

The frequency-dependent index of refraction and the absorption coefficient of the substrate shall be known in order to calculate the resistivity of the graphene film on top of the substrate. For many standard substrates, this information is available in the open literature. However, it is recommended that a separate THz-TDS characterization of the bare substrate is performed. For maximum consistency, the same frequency resolution and bandwidth as for the subsequent graphene conductivity measurements shall be used.

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4.5 Calibration standards

To check the correct performance of the equipment, several standard references can be used.

- For transmission measurements: air or a blank substrate (transmissive to THz radiation) with known refractive index and absorption parameter.
- For reflection measurements: a perfect reflector surface (e.g. a mirror or optically polished metal) or a blank substrate with known refractive index and absorption.

The result of measuring the standard references should be constant over time if the equipment works properly.

4.6 Measurement conditions

For comparison purposes, measurements shall be performed under the same conditions. Water vapour in the atmosphere leads to sharp and strong absorption lines. The influence of these lines is difficult to remove numerically and can be reduced by performing measurements in a dry atmosphere (recommended minimum requirement: RH < 5 %). Lowest possible humidity is thus preferred, but finite relative humidity can be acceptable, given that the relative humidity is constant throughout the experiment and remains below the dew point. The longer the THz beam path of the apparatus is, the stronger the influence of relative humidity is. Temperature variations may impact the stability of the THz spectroscopy equipment for instance by induced variations of the optical path length and alignment of the THz beam.

- Temperature range: 10 °C < T < 37 °C (determined by recommended temperature range for operation of the specific THz spectroscopy equipment used for the measurements).
- Range of relative humidity: RH < 70 % (as low as possible, upper limit is determined by the recommended humidity range for operation of the specific THz spectroscopy equipment used for the measurements).
- Atmosphere: air, nitrogen, argon (at atmospheric pressure). Vacuum is also feasible.

5 Measurement procedure

5.1 Calibration of the measurement equipment

Commercial THz-TDS systems have a factory-calibrated time axis (and thus a calibrated frequency axis), and there is no need for further calibrations by the end-user. Recommendations when using custom-made systems are described in Annex D, Clause D.2.

5.2 Detailed protocol of the measurement procedure

Prior to the measurement it shall be checked that the free sample area is large enough to ensure that the THz beam is not disturbed by the edges of the sample or any metallic structures (e.g. electrodes or contact pads). As a guideline, this is the case 1,2 mm away from any edges or any metallization on the sample at 0,9 THz [5], and data closer to the contacts shall be neglected. Depending on the choice of step-size used during the THz-TDS experiment, samples with dimensions of several millimetres are large enough to obtain hundreds of resistivity pixels. There is no upper sample size limit associated to the THz-TDS method.

On the other side, the path between the emitter and the receiver can be evacuated and/or purged with dry air or nitrogen to reduce or suppress the effects of water vapour adsorption in the measurement, i.e. the introduction of narrow resonances in the spectrum (see 4.6).

The measurement protocol is as follows.

1) Check relative humidity (this step may be omitted under special circumstances):
   - Record the relative humidity relevant for the sample.
   - In case of a purged measurement, wait until the humidity level in the measurement chamber has reached the target value (i.e. RH < 5 %).
2) Set a temporal scan range:

   The temporal scan range of the THz system shall be set to a fixed range wide enough to contain the relevant information. Commercial equipment might have a non-adjustable,

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the scan window should be chosen such that the entire signal shape is captured, with the start of the scan window chosen to be the point at which the THz signal is below the floor noise. The end of the scan window is defined by the substrate thickness (d_{sub}), since the first time-domain echo (t_{echo}) from the substrate will be at

$$t_{\text{echo}} = \frac{2 n_{\text{sub}} d_{\text{sub}}}{c} \tag{1}$$

where n_{sub} is the substrate refractive index of the substrate and c is the speed of light in a vacuum. Formula (2) applies both for reflection and transmission measurement configurations. Longer windows can be used if the subsequent time-domain echoes are included in the data extraction algorithm.

3) Acquire the reference waveform. This waveform is the reference measurement.
   - For transmission configuration: measure the bare substrate of the sample to be measured (it must be transmissive to THz radiation) or the air.
   - For reflection configuration: measure an area of bare substrate in the sample or a perfect reflector surface.
4) Block the THz path to avoid the THz beam reaching the detector. Use:
   - For transmission configuration: a metallic object.
   - For reflection configuration: an absorbing material or a metallic object with such a shape that it ensures the radiation is redirected to any direction different from the detector.
5) Acquire a waveform with the THz path blocked. This waveform is the floor noise measurement. For equipment with adjustable temporal scan range, check that such range has been correctly selected in step 2 and, if not, modify it.
6) Unblock the THz path and place the sample at the measurement location.
7) Check that relative humidity is still in the accepted range (this step may be omitted under special circumstances):
   - Record the relative humidity relevant for the sample.
   - In case of a purged measurement, wait until the humidity level in the measurement chamber has reached the target value (i.e. RH < 5 %).
8) Acquire the sample waveform(s).
   - Define and record the position of the measurement point for single measurements. For imaging mode, identify the area of the sample to be measured by defining and recording the measurement grid ($x_{\text{origin}}, x_{\text{stop}}, y_{\text{origin}}, y_{\text{stop}}$) and the scanning step size in x- and y-axes.
   - Acquire a waveform at the measurement point (for single measurements) or at each point of the measurement grid (for imaging mode) in the same conditions as with the reference. These waveforms are the sample measurements.
9) Transform the first peak of the reference and sample measurements (time-domain windowed waveforms, $E_{\text{ref}}(t)$ and $E_{\text{sample}}(t)$) to spectra (frequency-domain waveforms, $E_{\text{ref}}(\omega)$ and $E_{\text{sample}}(\omega)$) through a Fourier transform.

$$E_{\text{ref}}(\omega) = \text{FT}\left\{E_{\text{ref}}(t)\right\} \tag{3}$$

$$E_{\text{sample}}(\omega) = \text{FT}\left\{E_{\text{sample}}(t)\right\} \tag{4}$$

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10) Calculate the ratio of the spectra by dividing the sample and reference spectra to obtain either the transmission coefficient, $t_1(\omega)$, or the reflection coefficient, $r_1(\omega)$, in accordance with the measurement setup.

$$t_1(\omega) = \frac{E_{\mathrm{sample}}(\omega)}{E_{\mathrm{ref}}(\omega)} \tag{5}$$

$$r_1(\omega) = \frac{E_{\mathrm{sample}}(\omega)}{E_{\mathrm{ref}}(\omega)} \tag{6}$$

11) Use any of the computed ratios, $t_1(\omega)$ or $r_1(\omega)$, to calculate the frequency-dependent sheet conductivity or conductance of the graphene, $\sigma_{\mathrm{s}}(\omega)$. The equations are deduced in Annex C. Formulas (7) and (8) correspond to the ones deduced with reference waveforms acquired onto a perfect reflector (from reflection) or an empty beam path (for transmission). For waveforms acquired onto a bare substrate, the corresponding equations are included in Annex C.

$$\sigma_{\mathrm{s}} = \frac{4n_{\mathrm{sub}}e^{i\omega(n_{\mathrm{sub}}-1)d_{\mathrm{sub}}/c} - t_1(\omega)(1+n_{\mathrm{sub}})^2}{Z_0 t_1(\omega)(1+n_{\mathrm{sub}})} \tag{9}$$

$$\sigma_{\mathrm{s}}(\omega) = \frac{1-n_{\mathrm{sub}} + r_1(\omega)(1+n_{\mathrm{sub}})}{Z_0\left(1-r_1(\omega)\right)} \tag{10}$$

where n_{sub} is the refractive index of the substrate, d_{sub} is the thickness of the substrate, c is the speed of light in vacuum and Z_0 is the free space impedance.

12) To obtain sheet resistivity or resistance $\rho_{\mathrm{s}}(\omega)$ of the graphene, calculate the inverse of the sheet conductivity or conductance,

$$\rho_{\mathrm{s}}(\omega) = \frac{1}{\sigma_{\mathrm{s}}(\omega)} \tag{11}$$

Steps 9) and 10) are discussed in detail in reference [3].

5.3 Measurement accuracy

Measurement accuracy depends on many factors, which are listed below. A list of actions to improve the measurement accuracy is explained for each factor. Some of the actions are addressed to end-users of the equipment; however, some others require technical modifications of the device and are only addressed to THz-TDS qualified technicians.

– Thickness variations in the substrate, Δd. This is applicable to transmission and reflection modes, but it is especially important for transmission measurements, where the full complex-valued thin-film conductivity is extremely sensitive to the precise thickness of the substrate. The error of the frequency-dependent phase of the detected THz pulse is $\phi = 2\pi\nu n\Delta d / c$ for a thickness error Δd. Thus, unknown variations of the substrate thickness directly lead to the same relative uncertainty of the detected phase. The numerical value of the phase uncertainty in silicon (n = 3,417 7) is 0,072 mrad/(THz·nm). For 100 nm thickness error at 1 THz, the phase error is thus 7,2 mrad. The same arguments are valid if different positions on the same wafer are used for sample and reference measurements [8]. Thickness variations of the sample can only be minimized at the fabrication step. It is customary to use substrate thicknesses in the range 0,1 mm to 1 mm.

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- Irregularities in the reflection plane of the sample, Δz. Reflection measurements of the full complex-valued thin-film conductivity are sensitive to the precise location of the reflection plane of the sample along the propagation direction of the THz beam. The error on the frequency-dependent phase of the reflected signal is given by $\delta\phi = 4\pi\nu\Delta z / c$. The numerical value of this phase error is 0,042 mrad/(THz*nm). For 100 nm positioning error at 1 THz, the phase error is thus 4,2 mrad. This error is particularly difficult to avoid if separate materials are used for sample and reference measurement (for instance a smooth metal reflector for reference measurements), but also measurements with sample and reference points on the same substrate can have positioning errors that become significant. Irregularities in the reflection plane of the sample can only be minimized at the fabrication step.
- Phase variations due to the pulse jitter. Some systems show a change in the apparent position of the pulse caused by jitter in the laser or in the delay line [9]. These phase variations increase the error in the thin film conductivity calculation, as explained before in this section. Phase variations shall be minimized especially for thin substrates. Phase variations can be reduced by avoiding vibrations of the laser table and avoiding air fluctuations.
- Time-domain waveforms windowing. Frequency information is obtained through a Fourier transform of the waveform. In order to remove redundant information from the signal, thus facilitating the calculations and reducing uncertainties from them, a windowing technique is used defining a temporal window that cuts away unwanted parts of the full recorded waveform. It is well-known that some window functions tend to affect the magnitude of the peak while enhancing the frequency resolution, whereas some windows tend to preserve the magnitude of the peaks while losing some frequency resolution.
- Signal-to-noise-ratio (SNR) and dynamic range (DNR). The higher the SNR and DNR, the more accurate the measurement. The best way to increase the SNR and DNR in a THz-TDS system is by minimizing the noise, rather than increasing the signal. Noise can be reduced by (1) placing absorbing material close to the THz beam output and around the sample, (2) increasing the number of scans per measurement, (3) increasing the acquisition time and (4) ensuring that the laser power is stable over the duration of the measurement to avoid laser intensity variations. Actions 2 and 3 imply longer exposition of the sample to the THz beam; longer exposition does not heat the sample, nor does it have other detrimental effects on the measurement, as long as the room temperature remains constant.

6 Data analysis / interpretation of results

If multiple measurements on the same position on a graphene surface are available, the standard deviation of the measurement at each frequency point shall be calculated, and thus a complete measurement (value and uncertainty) shall be reported.

To obtain sheet resistance or conductance maps at a given frequency, represent the sheet resistance or conductance values of the measured grid with a colour mapping. With dedicated software tools these maps can be used for standardized analysis of products which ease the interpretation of the results.

Figure 6 shows a CVD monolayer graphene over PET substrate where the analysed area has been indicated with a blue square. Figure 7 shows the sheet conductance and resistance maps at a given frequency of the sample shown in Figure 6.

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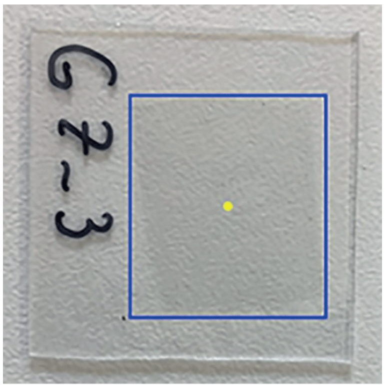

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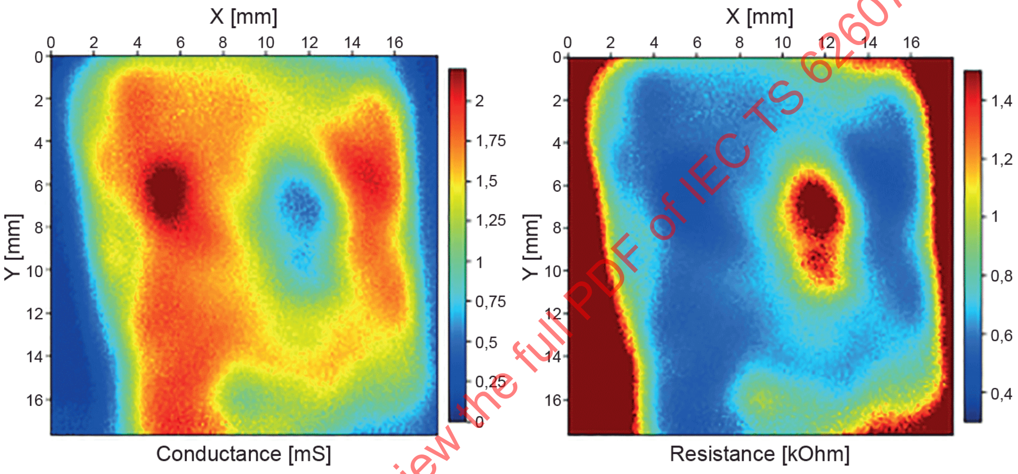
NOTE Size of sample approximately 15 mm × 15 mm. The blue square identifies the analysed area. The slightly darker shaded region within the blue square corresponds to the graphene film.

Figure 6 – Photograph of a CVD monolayer of graphene on PET substrate



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NOTE Frequency 0,5 THz, recorded with a step size of 100 µm. The colour bar shows conductance [mS] and resistance [kΩ]. Reproduced with permission from das-Nano, S.L.

Figure 7 – THz-TDS conductance and resistance maps of the sample

The conductance distribution along the graphene sheet can be analysed to identify the degree of homogeneity or heterogeneity of the material. In Figure 7, a central area with lower conductance than the average value of the film can be clearly identified, suggesting the existence of a defective area.

Figure 8 to Figure 11 are examples of data analysis for the sample in Figure 6. Depending on the software used for analysis, local areas can be inspected by selecting the area of interest with a polygon. Figure 8 shows the local area with the highest conductance. The global and local statistics (average conductance, standard deviation, maximum, minimum and dispersion values) are shown in the right upper corner and the global and local conductance distributions are shown in the "Histogram" graph. As expected, the mean local conductance is higher than the global one (1,711 mS and 1,253 mS, respectively). Furthermore, the local histogram (green bars) is much narrower than the global one (blue bars), also evidenced by the standard deviations (0,181 mS and 0,516 mS, respectively).

Analysis of linear conductance distributions can be also performed along line sections of the conductance map (see Figure 8). The conductance along the line is displayed in the "Line profile" graph.

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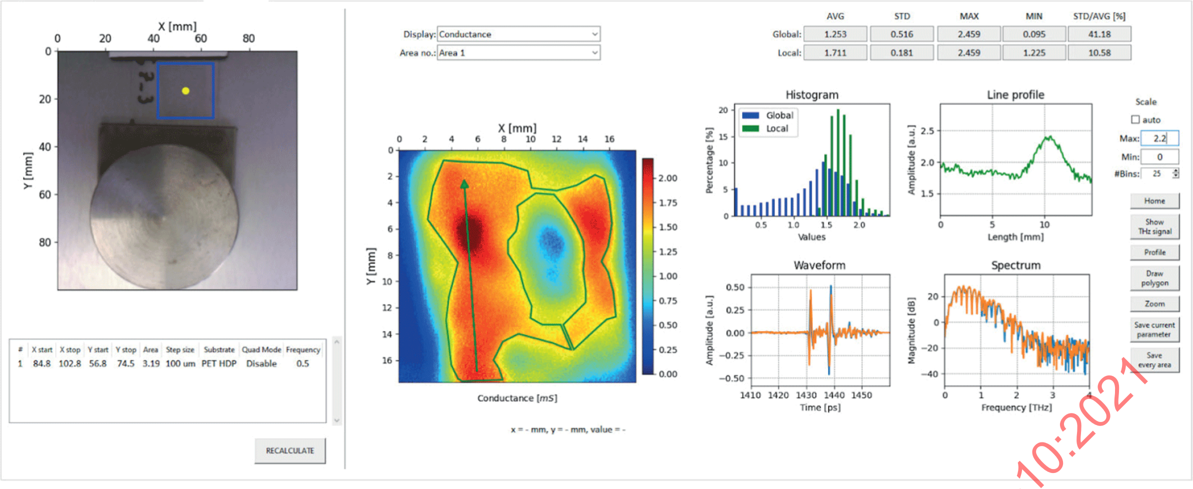

Display: Conductance

Area no.: Area 1

	AVG	STD	MAX	MIN	STD/AVG [%]
Global:	1.253	0.516	2.459	0.095	41.18
Local:	1.711	0.181	2.459	1.225	10.58

#	X start	X stop	Y start	Y stop	Area	Step size	Substrate	Quad Mode	Frequency
1	84.8	102.8	56.8	74.5	3.19	100 um	PET HDP	Disable	0.5

Scale

☐ auto

Max: 2.2

Min: 0

#Bins: 25

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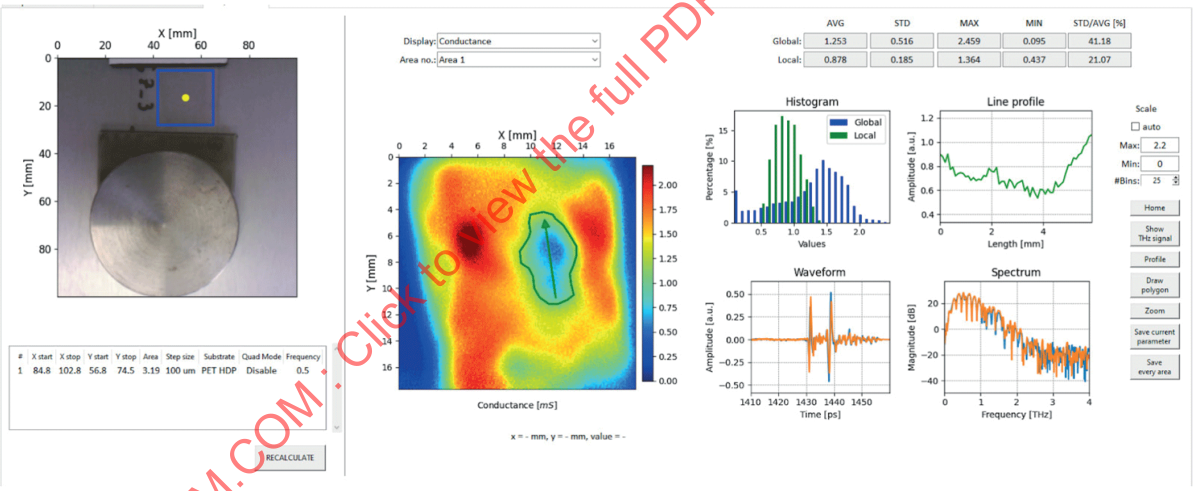
NOTE The polygon in the conductance map selects the high-conductance area. Reproduced with permission from das-Nano, S.L.

Figure 8 – Analysis of high conductance areas in the conductance map

Figure 9 shows the statistics for a selected area with low conductance in the centre of the sample and a linear conductance distribution within. The mean conductance (0,8 mS) is half the mean conductance in the high-conductance area (1,7 mS), suggesting a defective area in the graphene film.



Display: Conductance

Area no.: Area 1

	AVG	STD	MAX	MIN	STD/AVG [%]
Global:	1.253	0.516	2.459	0.095	41.18
Local:	0.878	0.185	1.364	0.437	21.07

#	X start	X stop	Y start	Y stop	Area	Step size	Substrate	Quad Mode	Frequency
1	84.8	102.8	56.8	74.5	3.19	100 um	PET HDP	Disable	0.5

Scale

☐ auto

Max: 2.2

Min: 0

#Bins: 25

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NOTE The polygon in the conductance map selects the low-conductance area. Reproduced with permission from das-Nano, S.L.

Figure 9 – Analysis of low conductance areas in the conductance map

The same analysis can be performed for the sheet resistance of the graphene film, as shown in Figure 10 and Figure 11.

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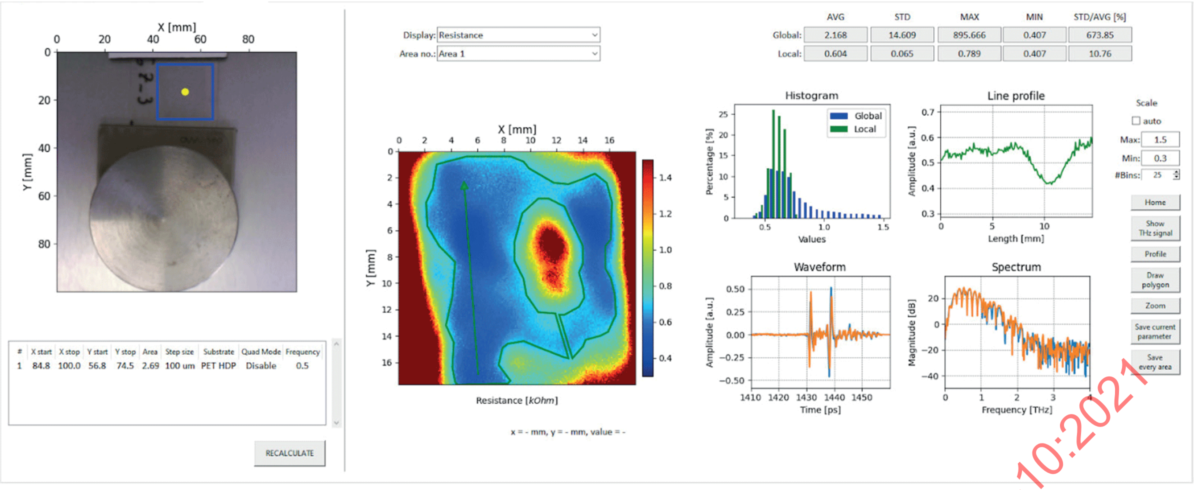

Display: Resistance

Area no.: Area 1

	AVG	STD	MAX	MIN	STD/AVG [%]
Global:	2.168	14.609	895.666	0.407	673.85
Local:	0.604	0.065	0.789	0.407	10.76

#	X start	X stop	Y start	Y stop	Area	Step size	Substrate	Quad Mode	Frequency
1	84.8	100.0	56.8	74.5	2.69	100 um	PET HDP	Disable	0.5

Scale

☐ auto

Max: 1.5

Min: 0.3

#Bins: 25

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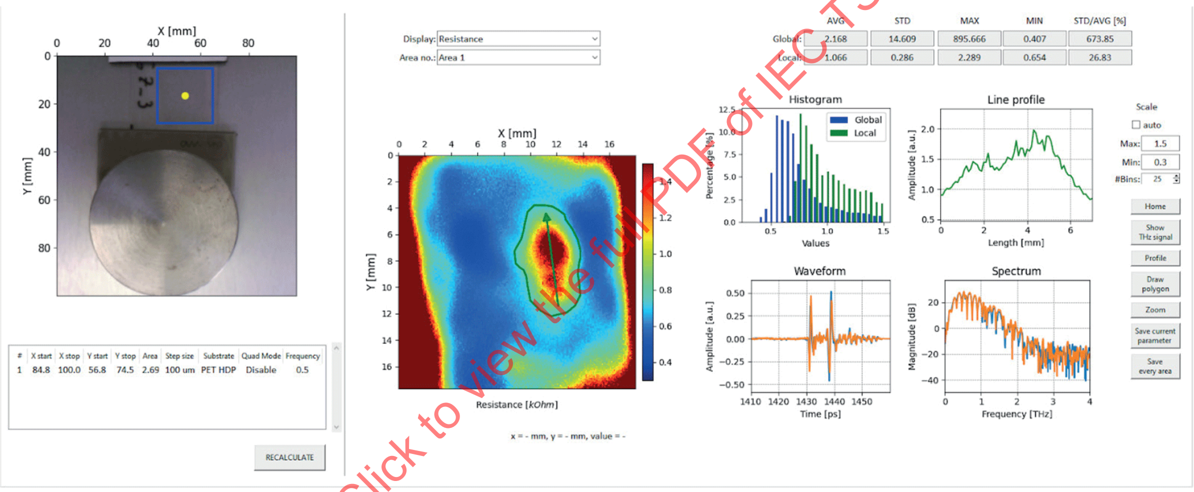
NOTE The polygon in the resistance map selects the low-resistance area. Reproduced with permission from das-Nano, S.L.

Figure 10 – Analysis of low-resistance areas in the resistance map



Display: Resistance

Area no.: Area 1

	AVG	STD	MAX	MIN	STD/AVG [%]
Global:	2.168	14.609	895.666	0.407	673.85
Local:	1.066	0.286	2.289	0.654	26.83

#	X start	X stop	Y start	Y stop	Area	Step size	Substrate	Quad Mode	Frequency
1	84.8	100.0	56.8	74.5	2.69	100 um	PET HDP	Disable	0.5

Scale

☐ auto

Max: 1.5

Min: 0.3

#Bins: 25

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NOTE The polygon in the resistance map selects the high-resistance area. Reproduced with permission from das-Nano, S.L.

Figure 11 – Analysis of high-resistance areas in the resistance map

The sheet conductance and resistance vary with the frequency. The frequency dependence analysis can be performed by plotting the conductance at different frequencies (see Figure 12).

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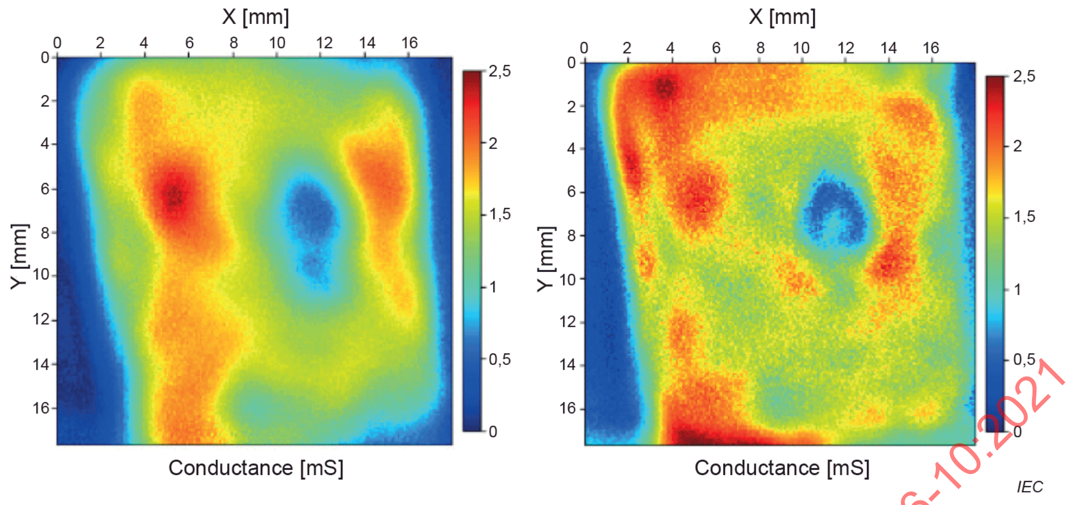

NOTE Reproduced with permission from das-Nano, S.L.

Figure 12 – Conductance maps at two different frequencies: 0,5 THz and 0,75 THz

For patterned devices, the data analysis of the conductance maps allows to determine the electrical quality of the graphene layer, as well as its shape. Application examples are explained in Annex B.

7 Results to be reported

7.1 Cover sheet

The results of the measurement shall be documented in a measurement report, including a reference to this document (IEC 62607-6-10:2021), date and time of the measurement as well as the name and signature of the person responsible for the accuracy of the report. Guidelines are given in Annex A.

7.2 Sample identification

The report shall contain all the information to identify the test sample and trace back the history of the sample:

- General procurement information.
- General material description, including a technical drawing or a picture:
  - top view, indicating the inspected area and location of the measurement positions;
  - cross section, showing the sample layer structure.

7.3 Measurement conditions

The laboratory ambient conditions during the test:

- Atmosphere: air, nitrogen, argon (at atmospheric pressure).
- Temperature range: 10 °C < T < 37 °C.
- Range of relative humidity: RH < 70 %.

7.4 Measurement specific information

- Sampling plan:
  - coordinates of the measured spot in the sample for single point mode;
  - location of the measured area in the sample for imaging mode;
  - scanning step size in x- and y-axes for imaging mode.
- Kind of equipment (commercial or custom-built; when commercial, add manufacturer and model).
- Measurement configuration: reflection, transmission.

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- Bandwidth of the system (units of THz).
- Spot size of the terahertz beam.
- Reference material.

7.5 Measurement results

The following items shall be part of the test report.

- Sampling plan: coordinate system used in the measurement setup in absolute positions with a definition of the origin, so that the measurement locations can be related to the technical drawing or picture of the sample.
- Results of sheet resistance or sheet conductance measured according to this document: table of mean values and standard deviations at the positions and/or areas defined by the sampling plan.
- For imaging mode measurements, graphical visualization of sheet resistance or sheet conductance measured according to this document as colour maps. The colour map shall be scaled in absolute positions in respect of the origin of the coordinate system. The colour code shall be calibrated in absolute values of the measured sheet resistance or sheet conductance.

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Annex A
(informative)

Worked example

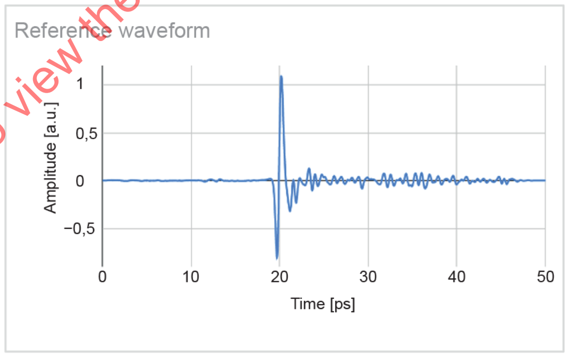
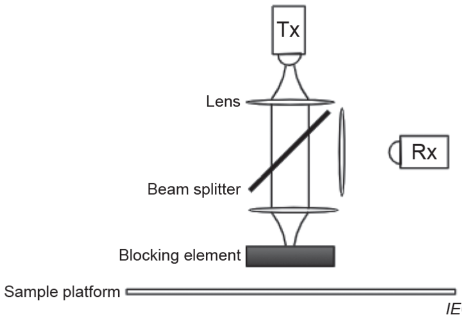
A.1 Background

This worked example is intended to illustrate the use of this document. It shows the measurement of the sheet resistance of a chemical vapour deposited (CVD) monolayer graphene over quartz substrate in reflection mode with Onyx[2], a calibrated commercial THz-TDS spectrometer.

A.2 Measurement protocol

The measurement is performed according to the measurement protocol defined in this document (See Clause 5). The measurement protocol is illustrated in Table A.1. R/O qualifies the step as required (R) or optional (O).

Table A.1 – Illustration of measurement protocol

Step	Action	Illustration	R/O
1	Check relative humidity	Wait until the RH is below 5 %. The range of the RH at the location of the sample is recorded over the duration of the measurement and reported.	O
2	Set the temporal scan range of the THz system	The equipment used to perform the measurement is commercial and the temporal scan range is non-adjustable. Scan range is predetermined to 50 ps.	R
3	Acquire the reference waveform	Reference waveform taken at a point of an area of a perfect reflector surface.  IEC	R
4	Block the THz path with an absorbing material.	 IEC	R

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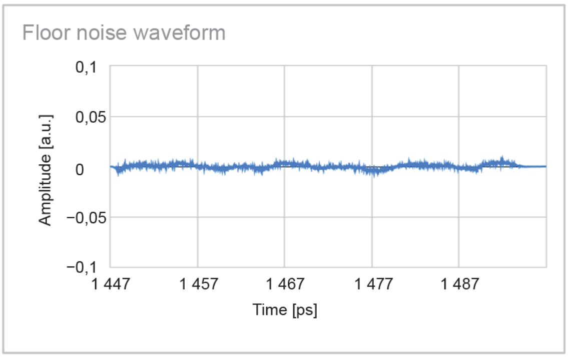
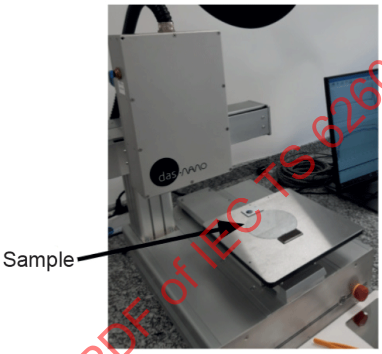
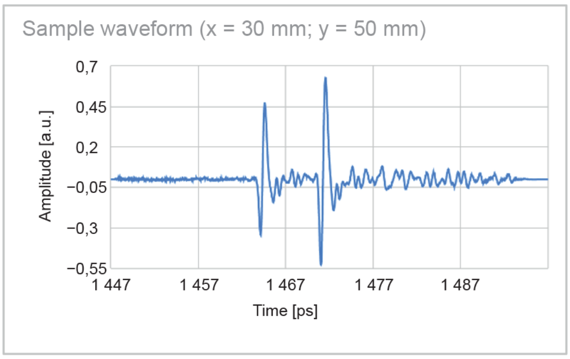
[2] Onyx is the trade name of a product supplied by das-Nano, S.L. This information is given for the convenience of users of this document and does not constitute an endorsement by IEC of the product named.

Step	Action	Illustration	R/O
5	Acquire a waveform with the THz path blocked	 IEC	R
6	Unblock the THz path and place the sample on the sample platform	 IEC	R
7	Check relative humidity	Wait until the RH is below 5 % The range of the RH at the location of the sample is recorded over the duration of the measurement and reported.	O
8	Acquire the waveforms of the sample and record the measurement position of each waveform	Define the measurement grid: - $x_{\text{origin}} = 0$ mm - $x_{\text{stop}} = 100$ mm - $y_{\text{origin}} = 0$ mm - $y_{\text{stop}} = 100$ mm - scanning step size in x-axis = 1 mm - scanning step size in y-axis = 1 mm Acquire a waveform of the sample in each point of the grid (10^4 measurements) at the same measurement conditions as the reference waveform. Measured waveform of the sample at a single point, at the same measurement conditions as the reference waveform.  IEC	R

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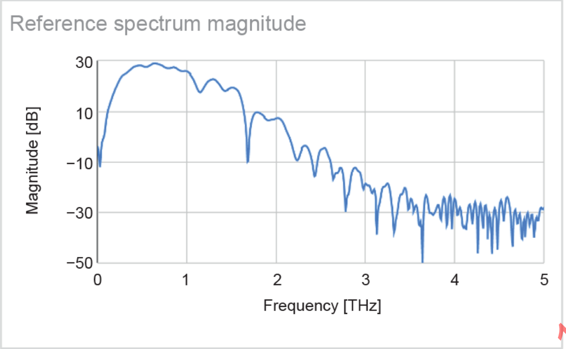
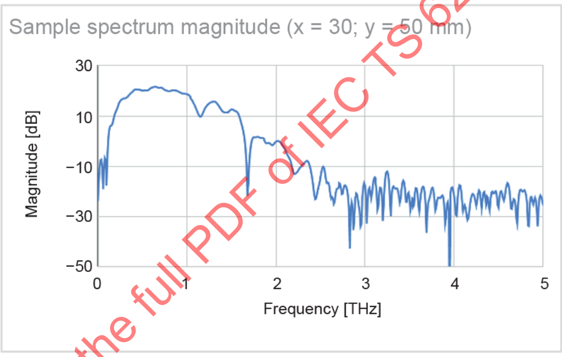
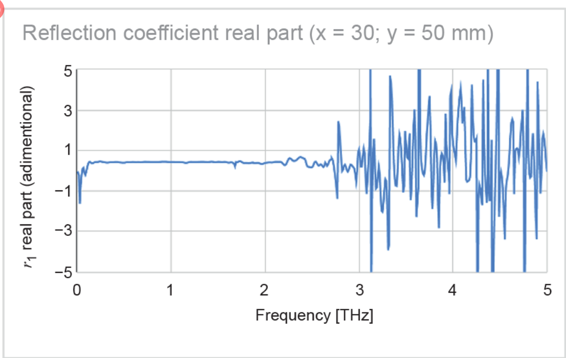
Step	Action	Illustration	R/O
9	Transform the first peak of the reference waveform and all the sample waveforms to spectra.	Fourier transformation of the first peak of the reference waveform to obtain the reference spectrum. Below the reference spectrum magnitude.  IEC Fourier transformation of the first peak of one sample waveform to obtain the corresponding sample spectrum. Below the sample spectrum magnitude.  IEC Transform all the sample waveforms to sample spectra.	R
10	Calculate $r_1(\omega)$	Real part of the reflection coefficient of one sample measurement.  IEC Calculate the reflection coefficient for all the sample spectra.	R

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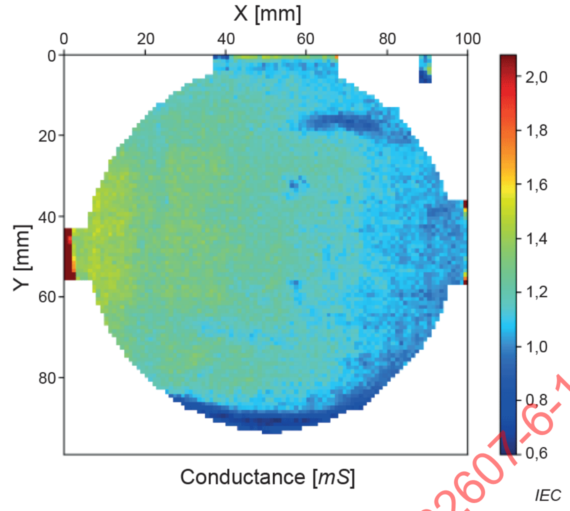
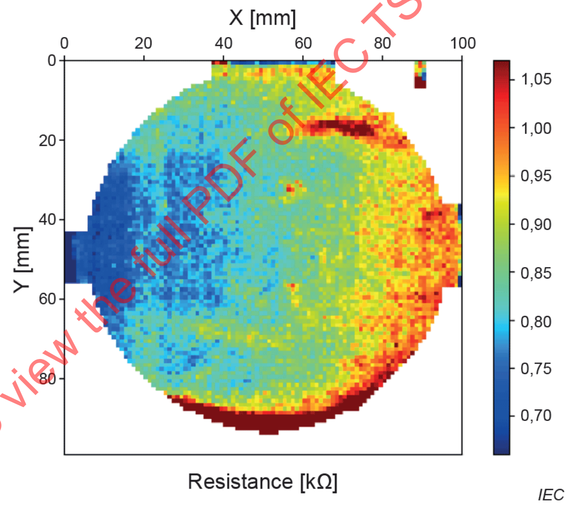
Step	Action	Illustration	R/O
11	Calculate $\sigma_s(\omega)$	Conductance map of the complete measurement grid. 	O
12	Calculate $\rho_s(\omega)$	Resistance map of the complete measurement grid. 	R

A.3 Test report

The format of the report is aligned to the relevant material specification. Table A.2 to Table A.6 are guidelines for writing the report and can be modified to fulfil the particular requirements of the involved parties.

It is recommended to refer in the test report to this document, for example: "The test described in this report and the writing of this report have been performed in accordance with IEC TS 62607-6-10:2021.".

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Table A.2 – Product identification

Item No	Item		Information
1.1	Supplier		Supplier A
1.2	Trade name		Trade name supplier A
1.3	ID number		ID number supplier A
1.4	Typical batch quantity: number of wafers		1
1.5	Traceability requirements	☐ Batch number ☒ Serial number ☐ Others, specify …………………………	
		Manufacturing date	2013-10-03
1.6	Specification	Number	Customer specific
		Revision level	NA
		Date of issue	NA
1.7	Material Safety Data Sheet (MSDS) available	☒ No	
		☐ Yes	Reference

Table A.3 – General material description

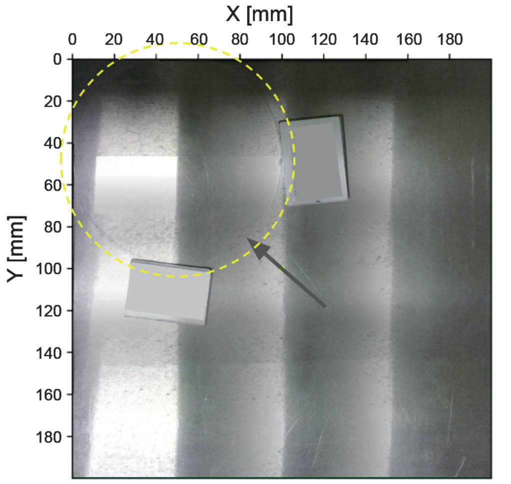
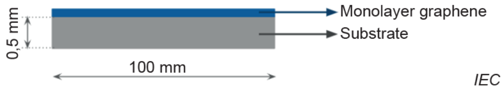
Item No	Item	Information	
2.1	Material type	CVD monolayer graphene over quartz substrate	
2.2	Manufacturing method	CVD grown on copper transferred to quartz	
2.3	Substrate material	Quartz	
2.4	Sample	Technical drawing or picture (top view)	 The sample is a circular colourless wafer, placed in the upper-left quadrant of the image. For ease of visualization, the wafer is surrounded by a yellow dotted circle. Reproduced with permission from das-Nano, S.L.
		Technical drawing (cross section)	
2.5	Shelf life	Nominal [ ] s, d	
2.6	Typical batch quantity	Number of wafers	Nominal [ ]

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Table A.4 – Sampling plan

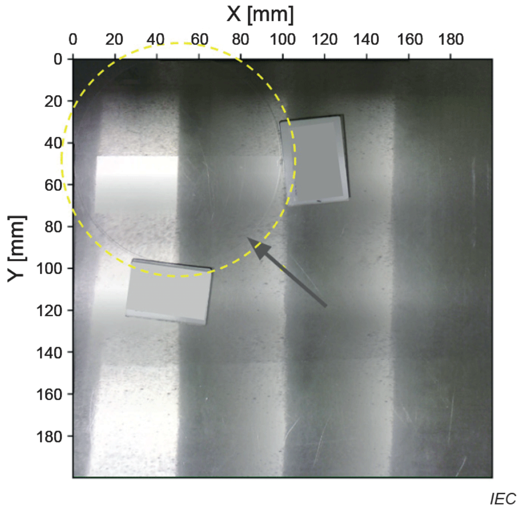
Image	Parameter	Value
 Photograph of the circular wafer placed on the sample platform in Onyx THz-TDS spectrometer. For ease of visualization, the wafer is surrounded by a yellow dotted circle. The grey rectangles on the right and bottom of the wafer are weights to immobilize the wafer during the measurements. Reproduced with permission from das-Nano, S.L.	Measured area (mm^2)	100 × 100
	x_{origin} (mm)	0
	$x_{step\ size}$ (mm)	1
	x_{stop} (mm)	100
	y_{origin} (mm)	0
	$y_{step\ size}$ (mm)	1
	y_{stop} (mm)	100
	Sample height (substrate + graphene film; mm)	0,5
	Number of measured points	10^4

Table A.5 – Measurement related information

Item No	Item	Information
3.1	Date and time of the measurement	[date] [time]
3.2	Person in charge of the measurement (name and signature)	[name] [signature]
3.3	Kind of equipment (commercial or custom-built; when commercial, add brand and model)	Commercial. Manufacturer: das-Nano. Model: Onyx.
3.4	Measurement configuration	☒ Reflection ☐ Transmission
3.5	Bandwidth of the system	0,1 THz to 5 THz
3.6	Spot size of the THz beam	1,4 mm in diameter
3.7	Reference material	Perfect reflector surface
3.8	Sample atmosphere	Dry N_2
3.9	Environmental temperature (mean)	23 °C
3.10	Environmental humidity (mean)	4 % RH

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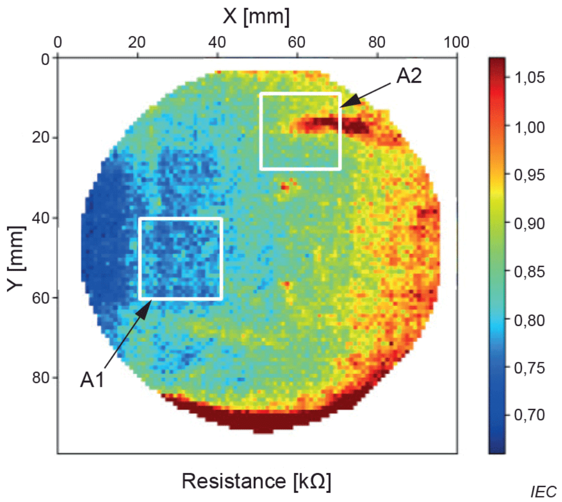

The colour bar shows electrical resistance [kΩ]. The results of the edges of the wafer have been discarded. Two 20 mm × 20 mm subareas have been selected (A1 and A2) to obtain local quantitative statistics. Reproduced with permission from das Nano S.L.

Figure A.1 – Sheet resistance map of the wafer at 0,5 THz, with step size 1 mm

Table A.6 contains statistical results of selected areas and of single points, as examples. Any point or area of the data shown in Figure A.1 can be added to the information in Table A.6.

Table A.6 – Measurement results: Sheet resistance at the selected frequency

Analysed area	Frequency (THz)	Mean sheet resistance (kΩ)	Standard deviation (kΩ)	Variation coefficient (%)	Min sheet resistance (kΩ)	Max sheet resistance (kΩ)
Total area	0,5	0,868	0,117	7,4	0,078	1,646
Subarea A1 ($20 \leq x \leq 40$; $40 \leq y \leq 60$)	0,5	0,776	0,023	38,3	0,722	0,847
Subarea A2 ($50 \leq x \leq 70$; $10 \leq y \leq 30$)	0,5	0,881	0,063	13,9	0,789	1,147
Point $x = 70$, $y = 18$	0,5	1,054	–	–	–	–
Point $x = 18$, $y = 40$	0,5	0,780	–	–	–	–

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Annex B
(informative)

Application examples

B.1 General

Several examples of resistance and conductance mapping of graphene films can be found in literature [1],[2],[5],[6]. This Annex B aims at showing selected examples of the use of THz-TDS for the characterization of sheet resistance and conductance of graphene layers deposited over different materials.

B.2 Conductance map of CVD graphene on quartz

Here there is one example of a CVD graphene layer transferred onto a quartz wafer, divided into 10 mm × 10 mm subsections. Figure B.1 shows a picture of the wafer (left) and its overall conductance map (right), while Figure B.2 shows the conductance maps of four exemplary subdivisions. The colour bar indicates the conductance [mS]. The comparison of the subdivisions shows a significant change in conductance, which could be associated to the fabrication process or to the cutting process.

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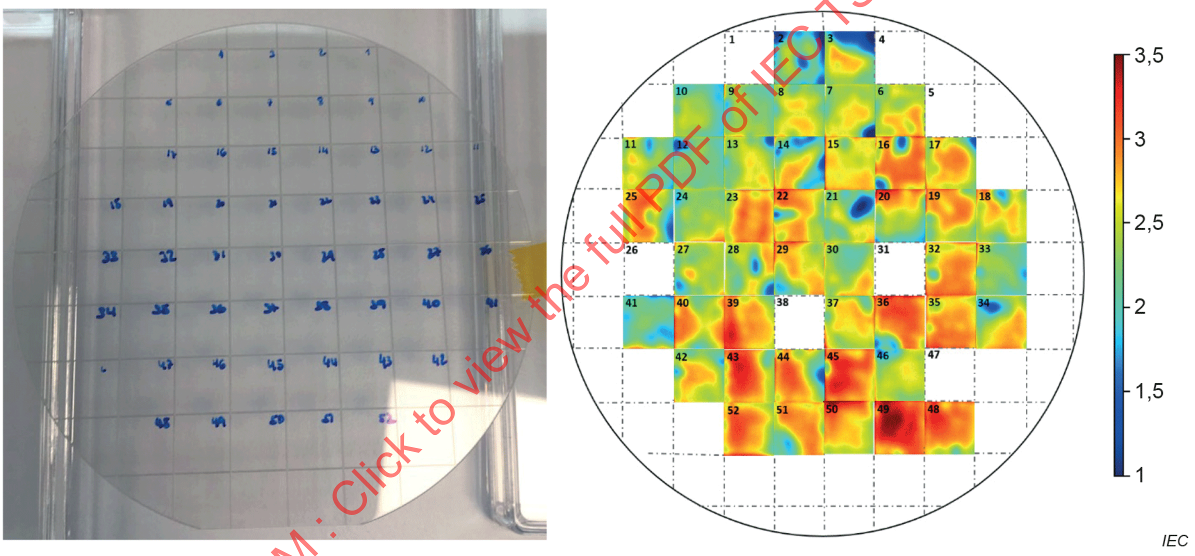
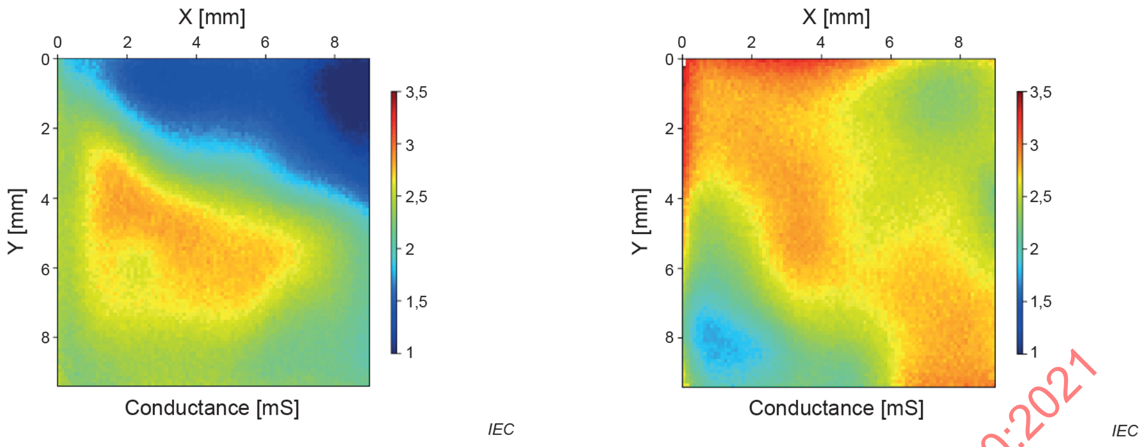
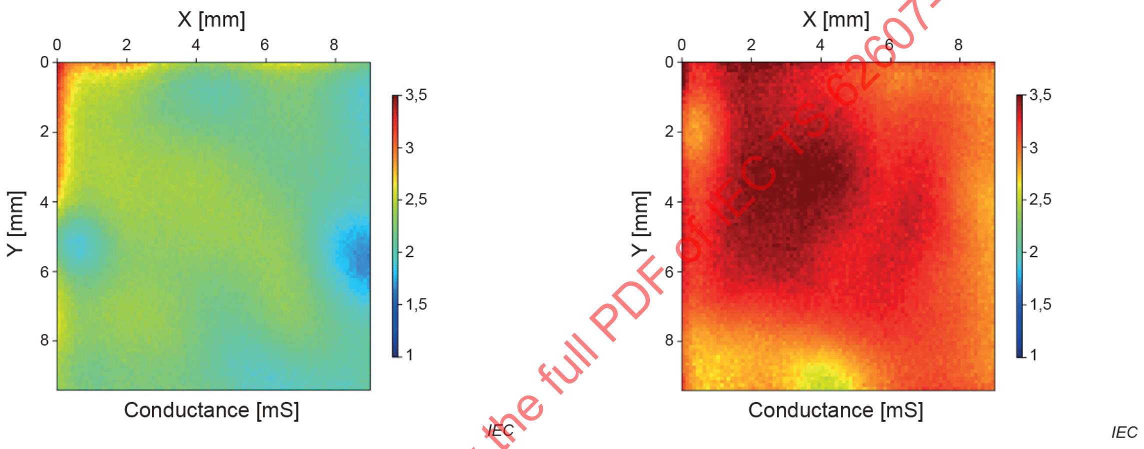


NOTE Each subdivision is 10 mm × 10 mm. Right: THz-TDS conductance maps of the subdivisions at 0,5 THz, recorded with a step size of 100 µm. The colour bar shows conductance [mS]. Reproduced with permission from das-Nano, S.L.

Figure B.1 – Photograph of a graphene film on a 100 mm quartz wafer and related conductance map



a) Subdivision 3 **b) Subdivision 29**



c) Subdivision 33 **d) Subdivision 49**

NOTE Conductance maps of selected subdivisions at 0,5 THz, namely 3, 29, 33 and 49. Reproduced with permission from das-Nano, S.L.

Figure B.2 – Detailed view of subsections from Figure B.1

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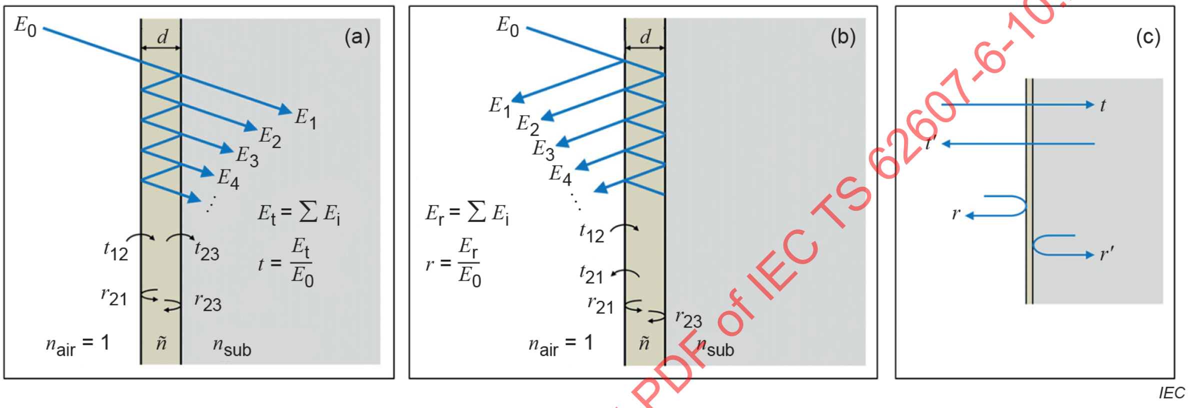
Annex C
(informative)

Theoretical background

C.1 Reflection and transmission of plane electromagnetic waves

The transmission and reflection of electromagnetic waves at a dielectric interface are described by the well-known Fresnel equations, which will not be derived here. See any standard textbook on optics for derivation of these relations. Here, we will consider the effect of a finite thickness of a thin film on top of a dielectric substrate on the effective transmission and reflection coefficients. The cases considered here are illustrated in Figure C.1. In Clause C.2 the expressions in the limiting case of a very thin conductive film, such as graphene, will be derived.



NOTE (a) Transmission and (b) reflection of an electric field at a thin film interface on a substrate, including multiple reflections in the thin film. (c) Definitions of transmission and reflection coefficients through the thin film from the air and substrate side.

Figure C.1 – Reflection and transmission for a thin film on a substrate

The analysis is based on the following basic assumptions.

- The electric fields are plane waves.
- Transmission and reflection at each interface are described by the appropriate Fresnel equations.
- Only normal incidence is considered, but the analysis can be straightforwardly extended to incidence at a given angle.

The plane-wave assumption defines how the field propagates:

$$E(z) = E(0)\exp(i\tilde{n}\omega z/c) \tag{C.1}$$

Where $\tilde{n}$ is the index of refraction, $\omega = 2\pi v$ is the angular frequency, z is the position in the propagation axis and c is the speed of light in vacuum. If the refractive index is complex-valued, then all Fresnel transmission and reflection coefficients are also complex-valued. For the sake of easy notation, the tilde-notation for complex values will be abandoned from here on.

The total transmitted field through a finite-thickness film (thickness d) will be the sum of the directly transmitted portion of the field, E_1, the portion that has bounced one time between the film interfaces, E_2, the portion that has bounced two times, E_3, and so on. Using the transmission and reflection coefficients, this is written as:

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$$\begin{aligned}
E_1 &= E_0 t_{12} e^{i\delta} t_{23} \\
E_2 &= E_0 t_{12} r_{23} r_{21} e^{i2\delta} e^{i\delta} t_{23} \\
E_3 &= E_0 t_{12} (r_{23} r_{21} e^{i2\delta})^2 e^{i\delta} t_{23} \\
&\vdots \\
E_k &= E_0 t_{12} (r_{23} r_{21} e^{i2\delta})^k e^{i\delta} t_{23}
\end{aligned} \tag{C.2}$$

where $\delta = n\omega d/c$ is the phase (and attenuation if n is complex) that the field acquires during one pass through the film.

This means that the total transmitted field will be the sum:

$$E_\mathrm{t} = E_0 t_{12} t_{23} e^{i\delta} \sum_{k'=0}^{\infty} (r_{23} r_{21} e^{2i\delta})^{k'} \tag{C.3}$$

This geometric series is known,

$$-1 < x < 1: \quad 1 + x + x^2 + x^3 + \ldots = \frac{1}{1-x}. \tag{C.4}$$

So, the transmission coefficient then can be written as

$$t \equiv \frac{E_t}{E_0} = \frac{t_{12} t_{23} e^{i\delta}}{1 - r_{23} r_{21} e^{2i\delta}}. \tag{C.5}$$

Similarly, the total reflected field from the film will be given by the sum of the direct and the multiple-bounce reflected portions of the incident field,

$$\begin{aligned}
E_1 &= E_0 r_{12} \\
E_2 &= E_0 t_{12} r_{23} t_{21} e^{2i\delta} \\
E_3 &= E_0 t_{12} r_{23} r_{21} r_{23} t_{21} e^{4i\delta} \\
&\vdots \\
E_k &= E_0 t_{12} t_{21} (r_{23} e^{2i\delta})^{k-1} r_{21}{}^{k-2} = E_0 t_{12} t_{21} r_{23} e^{2i\delta} (r_{23} r_{21} e^{2i\delta})^{k-2}
\end{aligned} \tag{C.6}$$

The sum of all contributions to the reflected field can therefore be written as:

$$E_\mathrm{r} = E_0 r_{12} + E_0 t_{12} t_{23} e^{2i\delta} \sum_{k'=0}^{\infty} (r_{23} r_{21} e^{2i\delta})^{k'} \tag{C.7}$$

Using the same geometric series expression as before, the reflection coefficient is then: