

TECHNICAL SPECIFICATION



**Nanomanufacturing – Key control characteristics –
Part 6-9: Graphene-based material – Sheet resistance: Eddy current method**

IECNORM.COM : Click to view the full PDF of IEC TS 62607-6-9:2022



THIS PUBLICATION IS COPYRIGHT PROTECTED

Copyright © 2022 IEC, Geneva, Switzerland

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either IEC or IEC's member National Committee in the country of the requester. If you have any questions about IEC copyright or have an enquiry about obtaining additional rights to this publication, please contact the address below or your local IEC member National Committee for further information.

IEC Secretariat
3, rue de Varembe
CH-1211 Geneva 20
Switzerland

Tel.: +41 22 919 02 11
info@iec.ch
www.iec.ch

About the IEC

The International Electrotechnical Commission (IEC) is the leading global organization that prepares and publishes International Standards for all electrical, electronic and related technologies.

About IEC publications

The technical content of IEC publications is kept under constant review by the IEC. Please make sure that you have the latest edition, a corrigendum or an amendment might have been published.

IEC publications search - webstore.iec.ch/advsearchform

The advanced search enables to find IEC publications by a variety of criteria (reference number, text, technical committee, ...). It also gives information on projects, replaced and withdrawn publications.

IEC Just Published - webstore.iec.ch/justpublished

Stay up to date on all new IEC publications. Just Published details all new publications released. Available online and once a month by email.

IEC Customer Service Centre - webstore.iec.ch/csc

If you wish to give us your feedback on this publication or need further assistance, please contact the Customer Service Centre: sales@iec.ch.

IEC Products & Services Portal - products.iec.ch

Discover our powerful search engine and read freely all the publications previews. With a subscription you will always have access to up to date content tailored to your needs.

Electropedia - www.electropedia.org

The world's leading online dictionary on electrotechnology, containing more than 22 300 terminological entries in English and French, with equivalent terms in 19 additional languages. Also known as the International Electrotechnical Vocabulary (IEV) online.

IECNORM.COM : Click to view the full PDF of IEC 60169:2022

TECHNICAL SPECIFICATION



**Nanomanufacturing – Key control characteristics –
Part 6-9: Graphene-based material – Sheet resistance: Eddy current method**

INTERNATIONAL
ELECTROTECHNICAL
COMMISSION

ICS 07.120

ISBN 978-2-8322-1073-5

Warning! Make sure that you obtained this publication from an authorized distributor.

CONTENTS

FOREWORD.....	4
INTRODUCTION.....	6
1 Scope.....	7
2 Normative references	7
3 Terms and definitions	7
3.1 General terms	8
3.2 Terms related to the measurement method	8
3.3 Key control characteristics	9
4 Abbreviated terms	10
5 General	10
5.1 Measurement principle.....	10
5.2 Measurement configuration.....	10
5.3 Measurement mode	11
5.3.1 Single point mode.....	11
5.3.2 Imaging scanning mode.....	11
5.4 Measurement system.....	11
5.4.1 Measurement equipment	11
5.4.2 Calibration standards.....	12
5.4.3 Ambient conditions	12
5.4.4 Sample preparation method.....	13
6 Measurement procedure	13
6.1 Calibration of test equipment	13
6.2 Verifying calibration	14
6.3 Description of the measurement procedure	14
7 Data analysis / interpretation of results	14
7.1 Single point sheet resistance measurement	14
7.2 Sheet resistance imaging.....	15
7.2.1 General	15
7.2.2 Line profile analysis.....	15
7.2.3 Histogram analysis	15
7.2.4 Statistical analysis.....	15
7.3 Individual homogeneity assessment.....	15
7.4 Defect identification	15
8 Test report.....	16
8.1 General.....	16
8.2 Sample identification.....	16
8.3 Test conditions	16
8.4 Measurement specific information.....	16
8.5 Test results.....	16
Annex A (informative) Worked example and test report.....	17
A.1 General.....	17
A.2 Sample identification.....	17
A.3 Geometry of the sample and scanning method.....	18
A.4 Measurement related information.....	18
Annex B (informative) Comparison between eddy current and four point probe measurement.....	20

B.1	General.....	20
B.2	Results	20
Annex C (informative)	Use case quality inspection	22
C.1	General.....	22
C.2	Analysis of the sheet resistance distribution for quality inspection.....	22
C.3	Analysis example	22
Annex D (informative)	Reference sample sets.....	25
Bibliography		26
Figure 1	– Scheme of measurement setup	12
Figure A.1	– Sheet resistance map with histogram.....	19
Figure B.1	– Correlation of the results of sheet resistance measured by the eddy current (EC) method and by the four point probe (4PP) method	21
Figure C.1	– Sheet resistance image of 200 mm × 200 mm graphene sheet.....	22
Figure C.2	– Line profile analysis	23
Figure C.3	– Histogram view of the whole sample	23
Figure C.4	– Histogram analysis (high sheet resistance area)	23
Figure C.5	– Histogram analysis (good area)	24
Figure C.6	– Selective area analysis (used specified shape).....	24
Figure C.7	– Selective area analysis (regular shape).....	24
Figure D.1	– Sheet resistance reference sample set 300 mm × 300 mm	25
Table A.1	– Product identification	17
Table A.2	– General material description.....	17
Table A.3	– Geometry of the sample and scanning parameter.....	18
Table A.4	– Measurement related information	18
Table A.5	– Test results: Sheet resistance	18
Table B.1	– Comparison of the results of sheet resistance measured by the eddy current method and by the four point probe method	20
Table B.2	– Measurement accuracy	21

INTERNATIONAL ELECTROTECHNICAL COMMISSION

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –**Part 6-9: Graphene-based material – Sheet resistance:
Eddy current method**

FOREWORD

- 1) The International Electrotechnical Commission (IEC) is a worldwide organization for standardization comprising all national electrotechnical committees (IEC National Committees). The object of IEC is to promote international co-operation on all questions concerning standardization in the electrical and electronic fields. To this end and in addition to other activities, IEC publishes International Standards, Technical Specifications, Technical Reports, Publicly Available Specifications (PAS) and Guides (hereafter referred to as "IEC Publication(s)"). Their preparation is entrusted to technical committees; any IEC National Committee interested in the subject dealt with may participate in this preparatory work. International, governmental and non-governmental organizations liaising with the IEC also participate in this preparation. IEC collaborates closely with the International Organization for Standardization (ISO) in accordance with conditions determined by agreement between the two organizations.
- 2) The formal decisions or agreements of IEC on technical matters express, as nearly as possible, an international consensus of opinion on the relevant subjects since each technical committee has representation from all interested IEC National Committees.
- 3) IEC Publications have the form of recommendations for international use and are accepted by IEC National Committees in that sense. While all reasonable efforts are made to ensure that the technical content of IEC Publications is accurate, IEC cannot be held responsible for the way in which they are used or for any misinterpretation by any end user.
- 4) In order to promote international uniformity, IEC National Committees undertake to apply IEC Publications transparently to the maximum extent possible in their national and regional publications. Any divergence between any IEC Publication and the corresponding national or regional publication shall be clearly indicated in the latter.
- 5) IEC itself does not provide any attestation of conformity. Independent certification bodies provide conformity assessment services and, in some areas, access to IEC marks of conformity. IEC is not responsible for any services carried out by independent certification bodies.
- 6) All users should ensure that they have the latest edition of this publication.
- 7) No liability shall attach to IEC or its directors, employees, servants or agents including individual experts and members of its technical committees and IEC National Committees for any personal injury, property damage or other damage of any nature whatsoever, whether direct or indirect, or for costs (including legal fees) and expenses arising out of the publication, use of, or reliance upon, this IEC Publication or any other IEC Publications.
- 8) Attention is drawn to the Normative references cited in this publication. Use of the referenced publications is indispensable for the correct application of this publication.
- 9) Attention is drawn to the possibility that some of the elements of this IEC Publication may be the subject of patent rights. IEC shall not be held responsible for identifying any or all such patent rights.

IEC TS 62607-6-9 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
113/569/DTS	113/625/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 in 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.

IMPORTANT – The "colour inside" logo on the cover page of this document indicates that it contains colours which are considered to be useful for the correct understanding of its contents. Users should therefore print this document using a colour printer.

IECNORM.COM : Click to view the full PDF of IEC TS 62607-6-9:2022

INTRODUCTION

The application of graphene and graphene-related materials as a conductive large-area electrode material has become of rising interest during recent years. Especially in the application of graphene as a replacement material for indium tin oxide (ITO), graphene combines low sheet resistance and high optical transparency. In particular, the application of optically transparent large-area graphene layers has become more important. Hence, the electrical characterization of large-area graphene layers is essential.

However, contacting methods, such as four-probe measurements, can cause damage to the graphene and deteriorate its quality.

Non-contact methods have advantages for measurement of the sheet resistance since damage to the layers is avoided and it is possible to readily scan the film to examine homogeneity.

The sheet resistance can serve as a measure for the electrical characterization due to its direct dependence on conductivity and graphene quality for electrical applications.

IECNORM.COM : Click to view the full PDF of IEC TS 62607-6-9:2022

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 6-9: Graphene-based material – Sheet resistance: Eddy current method

1 Scope

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

- sheet resistance

for films of graphene-based materials by

- eddy current method.

With this method a coil-generated primary alternating electromagnetic field induces eddy currents in the conducting layer to be measured. The superposition of the primary field with the secondary field induced by the eddy currents is a function of the sheet resistance of the layer.

- The method is applicable for the contactless measurement of the sheet resistance of large area graphene layers on non-conductive substrates. As the method avoids any physical contact, it prevents any mechanical damage to the sensitive graphene layer. Therefore, the method is suitable for electrical characterization and quality control in an industrial fabrication environment.
- Due to the use of two detectors – one above the substrate and one below the substrate – the method is insensitive regarding small deviations from perfect flatness of the substrate.
- The range of graphene layers to be characterized comprises any quality, size and morphology of graphene crystallites. Hence, the applicability of this method spans from high quality, defect-free graphene layers to layers of dried graphene ink.
- The size of the graphene layers to be characterized includes layers larger than 25 mm × 25 mm for single point testing and 50 mm × 50 mm for imaging testing.
- The method can be used for layers of graphene-based material with a sheet resistance in the nominal range of 10 Ω/sq to 5 000 Ω/sq.

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-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.

3.1.2

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 Terms related to the measurement method

3.2.1

eddy current

induced currents circulating along closed paths within a substance

[SOURCE: IEC 60050-121:1998, 121-12-32]

3.2.2

four point probe method

four terminal sensing

method to measure electrical sheet resistance or conductivity 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.

Note 2 to entry: There is a comparison of *eddy current* (3.2.1) and four point probe methods in Annex B.

[SOURCE: ISO 80004-13:2017, 5.3.1, modified – Note 2 to entry has been added.]

3.2.3

inline four point probe method

type of four point probe measurement where four point electrodes are aligned in a row

Note 1 to entry: In this method, four probes contact the test sample in a linear arrangement. A voltage drop is measured between the two inner probes while a current source supplies current through the outer probes.

Note 2 to entry: The thickness of the layer needs to be small compared to its lateral dimensions so that the sample is approximately two-dimensional.

Note 3 to entry: The distance between the probes shall be small compared to the lateral dimensions of the sample so that edge effects on the electric field in the sample can be neglected.

Note 4 to entry: The resistance of the sample can be calculated by Ohm's law. Geometrical factors can be used for corrections if the sample is too small in size or if the measurement is performed close to the edges of the sample.

3.2.4**van der Pauw method**

type of four probe measurement for samples of arbitrary shape

Note 1 to entry: The thickness of the layer needs to be small compared to its lateral dimensions so that the sample is approximately two-dimensional.

Note 2 to entry: The van der Pauw method requires four probes placed arbitrarily around the perimeter of the sample, in contrast to the linear four point probe which is placed on the top of the sample.

Note 3 to entry: The van der Pauw method provides an average sheet resistivity of the sample.

Note 4 to entry: The van der Pauw method relies on the assumption that the sample is homogeneous and continuous.

3.2.5**surface conductance measurement using resonant cavity**

method to measure the local surface conductance

Note 1 to entry: The measurements are made in an air filled standard R100 rectangular waveguide configuration, at one of the resonant frequency modes, typically at 7 GHz.

Note 2 to entry: Surface conductance measurement by resonant cavity involves monitoring the resonant frequency shift and change in the quality factor before and after insertion of the specimen into the cavity in a quantitative correlation with the specimen surface area.

Note 3 to entry: The thickness of the specimen does not need to be known, but it is assumed that the lateral dimension is uniform over the specimen area. This measurement is suited for atomically thin materials. Only resonant frequency and the area of the specimen are measured. No calibration standards are required. Details are shown in IEC TS 62607-6-4:2016.

Note 4 to entry: Resonance cavity methods and sheet resistance measurement methods do not deliver the same information and therefore do not compete with each other. Resonance cavity methods are mainly used to assess local defects and local properties whereas the sheet resistance measurements describe the electrical performance of a film.

Note 5 to entry: Surface conductance measurement by resonance cavity method is obtained on a very small area only. This technology is applied for the assessment of the individual performance, e.g. of a graphene flake.

Note 6 to entry: The above discussed sheet resistance measurement methods are measuring the ability of a larger area to transport currents through an area requiring the currents to run through various flakes or areas. Here, the number of grain boundaries, their electrical interconnectivity and the individual flake's electrical property matter.

Note 7 to entry: Graphene films can have a good surface conductivity but can be unable to transport currents through an area which is relevant for all applications where graphene is used for its electrical properties.

3.3 Key control characteristics**3.3.1****surface conductance**

σ_S

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/sq) is commonly used when referring to surface conductance. "S/sq" is dimensionally equal to "S", but is exclusively used for surface conductance to avoid confusion with electric conductance (G), which shares the same unit of measure: $G = I/U = \sigma_S \cdot (w/l)$.

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

3.3.2**surface resistance**

ρ_S

sheet resistance

reciprocal of surface conductance, σ_S

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

4 Abbreviated terms

4PP	four point probe, test method
EC	eddy current, test method
ITO	
NIST	National Institute of Standards and Technology
PET	polyethylene terephthalate
SEMI	Semiconductor Equipment and Materials International
VLSI	

5 General

5.1 Measurement principle

The eddy current method is a method applied for the characterization of electrical properties such as sheet resistance, conductivity, and local magnetization. Typically, an alternating electromagnetic field (primary field) is inducing eddy currents in the flat, electrically conductive sample of interest. According to Lenz' Law, the induced eddy currents generate a secondary electromagnetic field which is opposed to the primary field. The interaction of the primary field with the secondary field is a function of the sheet resistance of the present conductive layers. This principle is applied to electrically characterize layers without establishing an electrical contact. Generally, there are variants of measurements in physical contact and without physical contact of an electrically isolated eddy current sensor. The non-contacting mode allows the investigation of specimens without any mechanical contact as a potential source of damage or artefacts. The primary field induction and the resulting field measurement can be at different positions. The detecting sensor measures the complex impedance, which picks up the electric power absorption. The sheet resistance is directly correlated with this absorption. The industry is using various probe types and sizes for eddy current testing (see DIN 54140, Section 1-3 - Representation and general characteristics of test coil assemblies).

5.2 Measurement configuration

The measurement is conducted in a transmittance setup, where the sensor parts for field generation and for field measurement are separated by the specimen. The distance between both sensor parts is fixed. The recommended distance is 5 mm to 25 mm. The vertical position of the graphene layer should not vary for more than 10 % of the gap between the upper and lower sensor elements in accordance with the vertical position where the system was calibrated. The gap is fixed and shall not to be changed after system calibration and validation.

This setup supports the measurement on various substrate thicknesses that fit into the measurement gap. The tolerated variation of the thickness within one measurement is 10 % of the gap size. The measurement on different substrate thicknesses and hence different vertical positions in the measurement gap can be achieved by system calibration on these levels. Before each measurement, the user selects a suitable calibration dedicated to the substrate thickness in the tool software.

Reflective measurement setup is not supported by this document since the position tolerance for vertical variation is only given in transmittance mode. This enables robust measurement on all relevant substrates including non-conductive foils. Reflective mode measurements can be done on wafer since those are very flat. In this case, refer to conventional sheet resistance measurement SEMI standards.

5.3 Measurement mode

5.3.1 Single point mode

The single point measurement mode uses a static sensor, whereas the graphene sheet is moved below the upper sensor. Measurements are taken in the inner areas of the graphene material sample.

5.3.2 Imaging scanning mode

The imaging mode uses a moving sensor in the X direction and a moving substrate in the Y direction. Alternative modes where the substrate is moving in X and Y, with the sensor being static, or vice versa, are also common. This allows scanning the entire sample line by line. Such scanning systems are equipped with edge effect compensation mechanisms for measurements close to edges: typically, 3 mm cut away on each side.

The edge effect correction coefficients depend on the distance between the sensor elements and the sensor type and setup. Generic tables should not be used; instead, commercial manufacturers usually supply a correction table specific to the individual setup used. Software integrated edge effect correction solutions are more user friendly and are therefore preferred.

5.4 Measurement system

5.4.1 Measurement equipment

5.4.1.1 Description of test equipment

The measurement setup consists of a sample stage and two sensor probes (see Figure 1), which are described in 5.4.1.2 and 5.4.1.3, respectively.

5.4.1.2 Sample stage

The sample stage shall be flat and mechanically stable in order to carry the specimen safely. The sample stage shall be free of vibrations and horizontally aligned.

5.4.1.3 Sensor probes

Two sensor parts are applied for the measurement: the sending probe element and the receiving probe element. Each part consists of a coil and core setup in order to generate and detect an alternating electromagnetic field. The sending and receiving probes are opposing each other. Typically, one sensor probe is above the sample stage, while the other probe is located below the sample stage. It is essential that the sensor probes are in fixed position and cannot be moved relative to each other after calibration. The sample stage with the specimen to measure is positioned between the two sensor probes at the height of calibration and measurement.

Graphene on wafer can also be measured by using reflected eddy current tools. Here, standard SEMI MF673 is applicable. All other measurements such as graphene films on foil are measured in the transitive eddy current approach since it excels with a significantly higher tolerance to height variations. Additionally, the application of graphene as large area electrode, e.g. on foil, requires a higher vertical sample position tolerance and a higher flexibility in spot size, which can only be achieved in transmission by using different probe-to-probe distances.

5.4.1.4 Sensor gap

The measurement gap (distance between the upper probe and lower probe) should comfortably accommodate both the sample stage and sample, without touching the sample surface. The sensor gap shall be larger than 5 mm and smaller than 25 mm. This enables a sufficient tolerance for vertical substrate height variation and even tolerates inline setups that require a larger gap, without sacrificing too much of the spatial resolution.

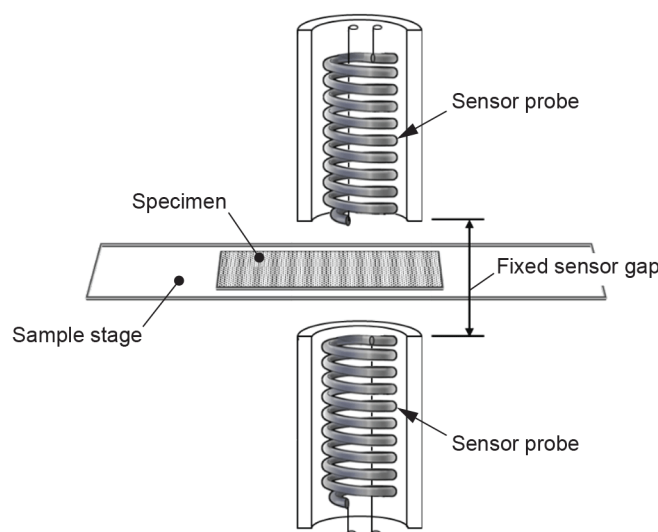


Figure 1 – Scheme of measurement setup

5.4.1.5 Measurement spot size

The high sensitivity zone of the measurement spot shall be larger than 5 mm and typically smaller than 25 mm. This ensures that isolated graphene areas with poor electrical contact to neighbouring graphene respond with higher sheet resistances in the measurement, which is essential for the qualitative measurement of a graphene sheet.

NOTE If the measurement spot size is too small, individual graphene flakes with very good sheet resistance would result in an assessment of high-quality graphene, which would still fail to be a good electrode, as it fails to transport current through the graphene sheet. Much larger spot sizes, however, would reduce the resolution by averaging over the spot size so that defect clusters and changes in grain boundaries, etc. cannot be resolved properly.

5.4.2 Calibration standards

Calibration standards are used to calibrate the eddy current measurement systems. As reference specimen, one can use homogeneous conductive films or homogeneously doped silicon wafers. The homogeneity is required to be better than 2 % (at 2 sigma) over an area of 100 mm × 100 mm measured with a measurement pitch of 1 mm (10 000 measurement points). The reference specimens are validated by non-contact eddy current sheet resistance mapping systems with a maximum measurement point pitch in X and Y of 1 mm to ensure the integrity of the calibration standard. The reference values of the calibration specimen can be defined by inline four point probe systems or eddy current sheet resistance testing systems that are validated by accepted standards like VLSI / NIST standards.

The measurement system is required to be checked every six months using calibration standards. The calibration of the reference system is required to be conducted with a traceable reference specimen (see Annex D).

5.4.3 Ambient conditions

The measurement shall be performed at constant temperature and after sufficient thermal equilibration of the measurement setup, typically at least one hour after powering up the measurement system. The relative humidity shall be less than 70 % and change in humidity should not be more than 5 % during the measurement.

5.4.4 Sample preparation method

5.4.4.1 Specimen

The specimen to be characterized can be doped or undoped graphene mono- or multi-layers, films, or sheets, including but not limited to those formed from a graphene containing ink or dispersion.

The graphene layer shall be continuous and not showing any isolated structures such as lines or squares. The graphene layer shall not be covered with any other conductive layer. The graphene layer shall cover an area of at least 25 mm × 25 mm for single point measurement and 50 mm × 50 mm for imaging applications.

Wet films should completely dry before applying this test method as the sheet resistance of, for example, wet graphene ink or dispersion based films changes during desiccation. Wet films based on inks or dispersion will not provide the same sheet resistance as dry films. Note that this eddy current method is also used as monitoring method for drying processes.

5.4.4.2 Substrate

Eddy current measurement setups induce currents in all present conductive materials. This applies to both the specimen and its substrate. A measurement on non-conductive substrates can be done directly since the measured sheet resistance is arising from the graphene layer alone.

The substrate shall be flat. There are no special requirements in terms of roughness since this technology is robust against height variation within tolerance. If the roughness however affects the ability of current to travel through the film, then this is a real result and not a measurement artefact.

When applying the direct measurement, there shall not be any further electrically conductive structures, other conductive layers or materials present in the gap between the probes.

A measurement on a conductive substrate, such as highly doped Si/SiO₂ wafers, requires the knowledge of the sheet resistance of the substrate. The sheet resistance of the substrate needs to be considered by applying the parallel resistance formula:

$$1/R_{\text{total}} = 1/R_{\text{substrate}} + 1/R_{\text{graphene}}$$

This formula can be well applied if the sheet resistance of the specimen is not more than one order of magnitude smaller than the sheet resistance of the substrate. Hence, a measurement of graphene on copper substrate is not possible as the graphene's sheet resistance is several orders of magnitude lower.

6 Measurement procedure

6.1 Calibration of test equipment

The sheet resistance calibration of the measurement device shall be conducted in accordance with the following procedure.

- 1) Take a self-referencing (= zeroing) measurement with an empty sample stage.

NOTE 1 A self-reference compares the measurement result in air at time of calibration and time of measurement. This is a common way to detect system changes and to compensate for such changes.

- 2) Take at least three calibration samples with known and homogeneous sheet resistances for the calibration. Calibration samples on wafer, glass or foils certified reference values shall be used (see Annex D).

The calibration samples should ideally differ in sheet resistance from each other by one order of magnitude (e.g. 2; 20; 200; 2 000; 20 000 Ω/sq). The calibration samples should ideally be of identical size, substrate type, substrate thickness and geometry, as the specimen to be measured. For a valid calibration, the span of sheet resistance of the calibration samples shall comprise the sheet resistance of any unknown specimen to be measured.

If the measurement tool supports zeroing, then this is required.

NOTE 2 The here described procedure is common for eddy current based measurement tools but can slightly vary between tool manufacturers. The here derived procedure was obtained from SURAGUS, Germany (see user manuals EddyCus® TF lab, map or inline¹).

- 3) Start the calibration by placing one calibration sample onto the sample stage in central position between the two probe elements.
- 4) Remove hands or other conducting items from the sample and take the measurement.
- 5) Save the measurement into the calibration file of the sheet resistance measurement device including the respective absolute sheet resistance value of the calibration sample (e.g. 0,1 Ω/sq).
- 6) Remove the sample from the sample stage.
- 7) Repeat steps 2) to 6) with each calibration sample and save the calibration file.

6.2 Verifying calibration

One reference sample should remain with the measurement tool to be used for testing the stability of the measurement tool before measurement. Typically, this reference sample is roughly in the middle of the measurement range or at the targeted sheet resistance. Every day before measurement, this reference sample needs to be tested. In case the deviation is larger than the stated accuracy, following measurements are not valid until the system has been recalibrated and has passed this test.

6.3 Description of the measurement procedure

The sheet resistance measurement of the graphene layer needs to be conducted in accordance with the following procedure.

- 1) Prepare the measurement by choosing the correct settings of the measurement device. The pre-set sample size, sample thickness and geometry should match the properties of the unknown specimen to be measured.
- 2) If the measurement tool supports zeroing, take a self-referencing (= zeroing) measurement with an empty sample stage.
- 3) Place the specimen with graphene layer face up onto the sample stage in central position on the measurement field between the probe elements. Then, take a measurement or initiate a scan.
- 4) Remove any other items contacting the specimen or in vicinity of the probe gap and take the measurement.
- 5) Remove the sample from the sample stage.

7 Data analysis / interpretation of results

7.1 Single point sheet resistance measurement

The initial test is a centred measurement of a graphene sample. This enables the assessment and qualification of graphene samples for certain applications. A single point measurement is typically taken in the inner area of the graphene sheet to avoid edge effects. Single point

¹ EddyCus® is the trademark of a product supplied by SURAGUS GmbH. This information is given for the convenience of users of this document and does not constitute an endorsement by IEC of the product named. Equivalent products may be used if they can be shown to lead to the same results.

measurements near edges require an edge effect compensation using an automated imaging device that comprises an edge effect correction mechanism (see EddyCus® TF map series).

7.2 Sheet resistance imaging

7.2.1 General

Sheet resistance images provide a profound understanding of the electrical performance across the graphene area. Graphene can contain small defective areas that are often missed by selective single point measurement. Hence, comprehensive and detailed measurements across the entire area with a measurement pitch of 1 mm are required to ensure the entire integrity of the sample (see Annex C).

7.2.2 Line profile analysis

Line profiles describe the sheet resistance across a line. Typically, a line contains 50 to 500 measurement points depending on the covered distance. A standard line profile analysis is done on centred horizontal and vertical line profiles. The maximum variance of both lines is an important indicator for the specimen's homogeneity (see Figure C.2).

7.2.3 Histogram analysis

All data points are depicted in a histogram. A histogram of large area graphene characterization should contain 10 to 100 points per square centimetre, which results in 10 000 measurements for larger samples. The sheet resistance distribution typically follows a Gaussian curve with one peak. If the curve contains several peaks, then the graphene sample is to be considered as largely inhomogeneous (e.g. mono-layer and double-layer areas).

7.2.4 Statistical analysis

The scanned graphene sheet resistance can be statistically analysed (average, variance, minimum, maximum).

7.3 Individual homogeneity assessment

Individual data analysis can be conducted according to the device structure. Suitable homogeneous areas can be selected within larger inhomogeneous areas.

7.4 Defect identification

Defective areas show up in sheet resistance images as higher sheet resistance areas. Defects are typically smaller than the high resistance areas. Defective zones should not be used as electrode material.

8 Test report

8.1 General

The results of the measurement shall be documented in a measurement report, providing the background of the test, date and time of the measurement as well as names of the persons in charge of performing the measurements, and name and signature of the person responsible for the accuracy of the report (see Annex A).

8.2 Sample identification

The report shall contain all information to identify the test sample and trace back its history.

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

8.3 Test conditions

The ambient conditions during the test.

- Temperature range: $15\text{ °C} < T < 25\text{ °C}$.
- Range of relative humidity: $20\% < RH < 70\%$.

8.4 Measurement specific information

Documentation on the applied setup should comprise information on the applied measurement device (type, manufacturer), especially the following:

- calibration status of equipment;
- measurement configuration: transmission shall be noted explicitly, even if reflection does not conform to this document;
- spot size;
- scanning step size for imaging scanning mode;
- sampling plan for single point mode.

8.5 Test results

- 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 of the sample.
- Table of mean values and standard deviation of sheet resistance and/or sheet conductance at the positions defined by the sampling plan.
- Maps for sheet resistance and optionally sheet conductance. The colour map shall be scaled in absolute positions in respect of the origin of the coordinate system. The colour code should be calibrated in absolute values of the measured KCC.

Annex A (informative)

Worked example and test report

A.1 General

A.2 Sample identification

Table A.1 provides the information required to trace back the sample to fabrication conditions.

Table A.1 – 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 samples	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.2 provides the general material description.

Table A.2 – General material description

Item No	Item	Information	
2.1	Material type	CVD monolayer graphene over silicon substrate	
2.2	Manufacturing method	CVD grown on copper transferred to Si	
2.3	Substrate	Material	Si
		Technical drawing (Top view)	
		Technical drawing (Cross section)	
2.4	Shelf life	Nominal [] s, d	
2.11	Typical batch quantity	Number of wafers	Nominal []

A.3 Geometry of the sample and scanning method

Table A.3 provides the geometry of the sample and scanning parameter.

Table A.3 – Geometry of the sample and scanning parameter

	Measured area	100 mm × 100 mm
	X pitch	1 mm
	X stop	continuous
	Y pitch	1 mm
	Y stop	continuous
	Sample height	200 µm
Figure 1: sample photograph	Flat	none

A.4 Measurement related information

Table A.4 provides measurement related information.

Table A.4 – Measurement related information

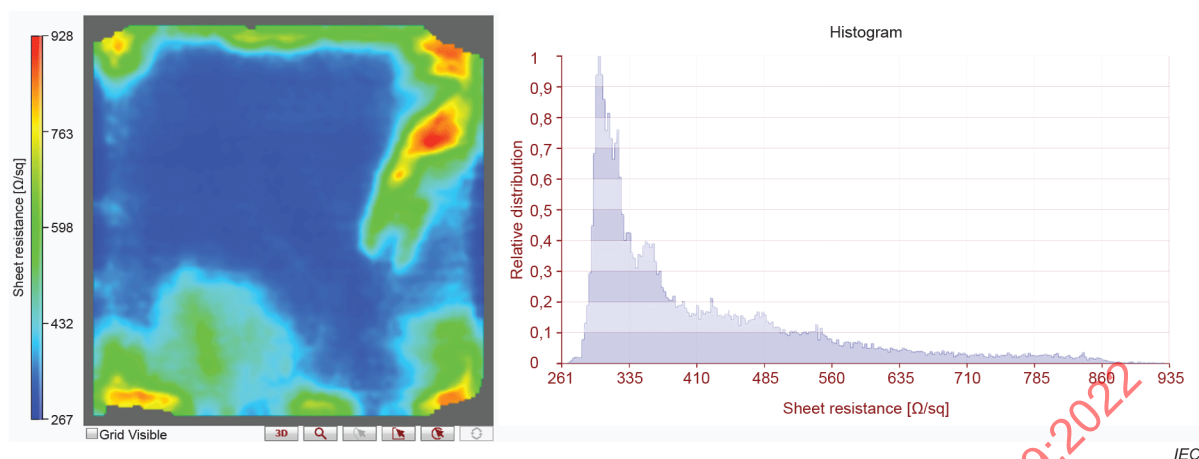
Item No	Item	Information
3.1	Measurement configuration	☒ Transmission
3.2	Measurement upper sensor element to lower sensor element	5 mm
3.3	Tool verification	Reference sample 512 Ω/sq
3.4	Results frequency	100 measurements per line (100 mm)
3.5	Environmental humidity (mean)	40 %
3.6	Environmental temperature (mean)	22 °C
3.7	Sample atmosphere	Air

Table A.5 provides test results for sheet resistance.

Table A.5 – Test results: Sheet resistance

Sub-area	Mean value	Standard deviation	Variation coefficient (%)
A1			

The following example shows a sheet resistance mapping of a monolayer on PET with a size of 200 mm × 200 mm. The utilized instrument was EddyCus® TF map 5050 with a scanning area of 500 mm × 500 mm. The probe gap was 8 mm. The measurement pitch was 1 mm in X and Y. The result shows a large homogenous area in the centre and defective areas around the edges and at the right.



IEC

Figure A.1 – Sheet resistance map with histogram

Annex B (informative)

Comparison between eddy current and four point probe measurement

B.1 General

The four point probe method is widely used as a reference for contact and contactless measurement of the key control characteristic sheet resistance. Such a comparison requires samples which have a sufficiently good sheet resistance uniformity to bridge the difference of the probing area of both methods. Care was taken so that the unavoidable damage caused by the direct contact in case of the four point probe method does not falsify the results.

B.2 Results

A comparison of repeated sheet resistance measurements obtained by non-contact eddy current and contacting four point probe method is shown in Table B.1.

**Table B.1 – Comparison of the results of sheet resistance measured by
the eddy current method and by the four point probe method**

Sample number	Four point probe method				Eddy current method			
	Mean	Standard deviation	Min.	Max.	Mean	Standard deviation	Min.	Max.
	[Ω/sq]	[Ω/sq]	[Ω/sq]	[Ω/sq]	[Ω/sq]	[Ω/sq]	[Ω/sq]	[Ω/sq]
1	0,496	0,052	0,467	0,64	0,485	0,000 2	0,484 2	0,484 7
2	1,120	0,022	1,079	1,16	1,120	0,000 1	1,120 3	1,120 6
3	1,759	0,032	1,720	1,81	1,772	0,000 2	1,771 5	1,772 1
4	4,430	0,100	4,300	4,61	4,425	0,000 6	4,424 4	4,426 3
5	11,840	0,200	11,350	12,09	11,622	0,010 2	11,605 5	11,642 1
6	30,400	0,500	29,800	31,30	30,498	0,024 1	30,454 4	30,536 0
7	82,500	0,700	81,500	83,40	81,359	0,114 5	81,229 4	81,497 2

The correlation of the results is shown in Figure B.1.

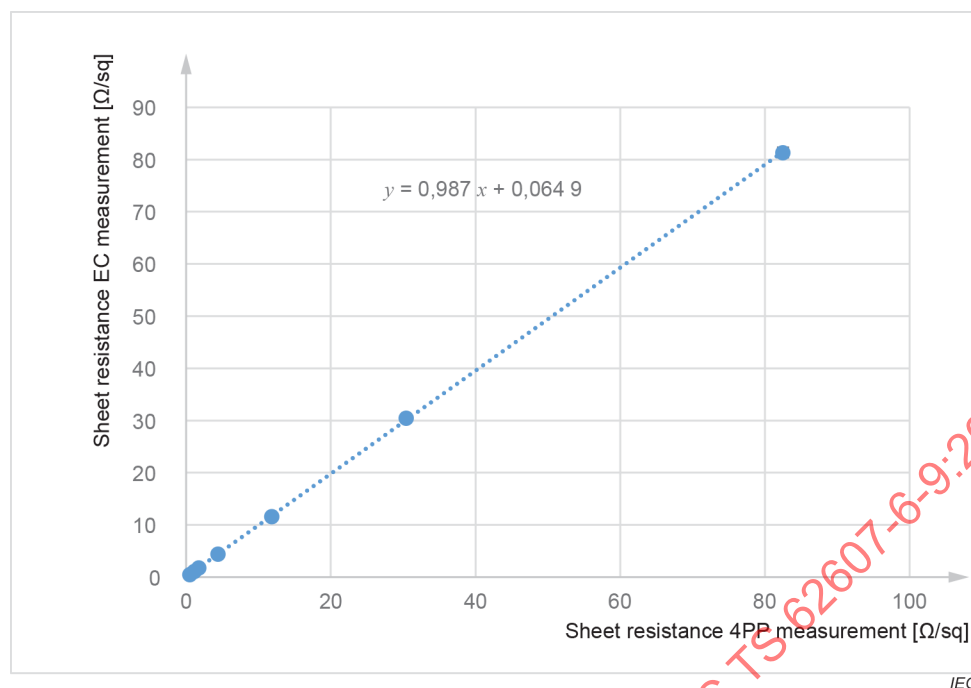


Figure B.1 – Correlation of the results of sheet resistance measured by the eddy current (EC) method and by the four point probe (4PP) method

If the measurements are conducted following the standard testing procedure, the level of performance shown in Table B.2 can be achieved.

Table B.2 – Measurement accuracy

Accuracy	0,96 %
Repeatability	0,21 %
Repeatability and reproducibility (R&R)	0,21 %

Annex C (informative)

Use case quality inspection

C.1 General

Annex C illustrates the application of the method for quality inspection which takes advantage especially of the fast acquisition of sheet resistance. As a contactless method which can be implemented into the fabrication process, the eddy current method is perfect choice for high-throughput applications.

C.2 Analysis of the sheet resistance distribution for quality inspection

The measurement of the sheet resistance distribution provides an excellent tool for the inspection of product quality under fabrication conditions. Figure C.1 shows a sheet resistance image of 200 mm × 200 mm graphene sheet with 1 mm measurement pitch in X and Y and an edge effect exclusion of 3 mm.

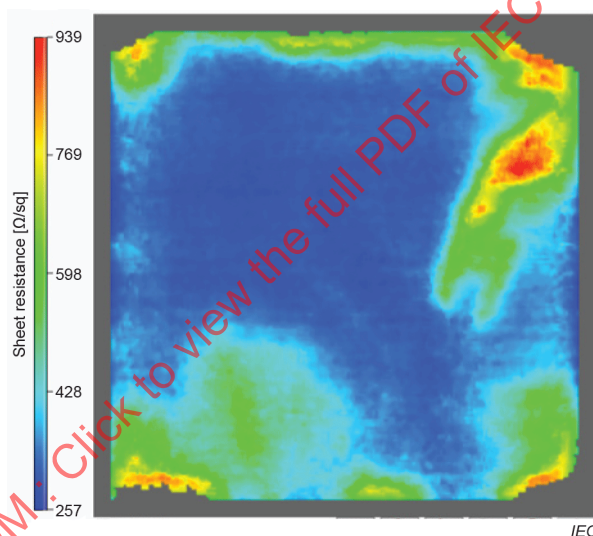


Figure C.1 – Sheet resistance image of 200 mm × 200 mm graphene sheet

C.3 Analysis example

If the method is combined with image inspection software tools, it is possible to generate customer-relevant information during the fabrication process. The examples given in Figure C.2 to Figure C.7 illustrate line scans and histogram analysis of selected areas.