PD IEC TS 62607-6-10:2021



BSI Standards Publication

Nanomanufacturing — Key control characteristics

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

bsi.

National foreword

This Published Document is the UK implementation of IEC TS 62607-6-10:2021.

The UK participation in its preparation was entrusted to Technical Committee NTI/1, Nanotechnologies.

A list of organizations represented on this committee can be obtained on request to its committee manager.

Contractual and legal considerations

This publication has been prepared in good faith, however no representation, warranty, assurance or undertaking (express or implied) is or will be made, and no responsibility or liability is or will be accepted by BSI in relation to the adequacy, accuracy, completeness or reasonableness of this publication. All and any such responsibility and liability is expressly disclaimed to the full extent permitted by the law.

This publication is provided as is, and is to be used at the recipient's own risk.

The recipient is advised to consider seeking professional guidance with respect to its use of this publication.

This publication is not intended to constitute a contract. Users are responsible for its correct application.

This publication is not to be regarded as a British Standard.

© The British Standards Institution 2023
Published by BSI Standards Limited 2023

ISBN 978 0 539 14153 5

ICS 07.120

Compliance with a Published Document cannot confer immunity from legal obligations.

This Published Document was published under the authority of the Standards Policy and Strategy Committee on 31 March 2023.

Amendments/corrigenda issued since publication

Date	Text affected

IEC TS 62607-6-10

Edition 1.0 2021-10

TECHNICAL SPECIFICATION

colour inside

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

INTERNATIONAL ELECTROTECHNICAL COMMISSION

ICS 07.120

ISBN 978-2-8322-1033-3

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

® Registered trademark of the International Electrotechnical Commission

CONTENTS

FOREWORD 4
INTRODUCTION 6
1 Scope 7
2 Normative references 7
3 Terms and definitions 7
3.1 General terms 7
3.2 Key control characteristics measured according to this document 9
3.3 Terms related to the measurement method described in this document 10
4 General 11
4.1 Measurement principle 11
4.2 Sample preparation method 13
4.3 Description of measurement equipment 13
4.3.1 Principal components of a THz-TDS system 13
4.3.2 Measurement configurations 14
4.4 Supporting materials 16
4.5 Calibration standards 17
4.6 Measurement conditions 17
5 Measurement procedure 17
5.1 Calibration of the measurement equipment 17
5.2 Detailed protocol of the measurement procedure 17
5.3 Measurement accuracy 19
6 Data analysis / interpretation of results 20
7 Results to be reported 24
7.1 Cover sheet 24
7.2 Measurement conditions 24
7.3 Measurement specific information 24
7.4 Measurement results 25
Annex A (informative) Worked example 26
A.1 Background 26
A.2 Measurement protocol 26
A.3 Test report 29
Annex B (informative) Application examples 33
B.1 General 33
B.2 Conductance map of CVD graphene on quartz 33
Annex C (informative) Theoretical background 35
C.1 Reflection and transmission of plane electromagnetic waves 35
C.2 Transmission coefficient through a thin, conductive film 37
C.3 Sheet conductivity of a thin, conductive film 39
C.3.1 Reflection-mode geometry 40
C.3.2 Transmission-mode geometry 41
Annex D (informative) Considerations for custom-built systems 43
D.1 Linearity of the THz-TDS detection system 43
D.2 Calibration of custom-built equipment 43
Bibliography 44

Figure 1 – Time trace of a typical THz waveform.....12
Figure 2 – Sample scheme comprised of a thin film of graphene on a dielectric substrate. 12
Figure 3 – Principal components of a classical THz-TDS system.....14
Figure 4 – Comparison of different transmission geometries. 15
Figure 5 – Comparison of different reflection geometries 16
Figure 6 – Photograph of a CVD monolayer of graphene on PET substrate.....21
Figure 7 – THz-TDS conductance and resistance maps of the sample 21
Figure 8 – Analysis of high conductive areas in the conductivity map.....22
Figure 9 – Analysis of low conductive areas in the conductivity map 22
Figure 10 – Analysis of low resistance areas in the resistance map 23
Figure 11 – Analysis of high resistance areas in the resistance map.....23
Figure 12 – Conductance maps at two different frequencies: 0,5 THz and 0,75 THz 24
Figure A.1 – Colour map of sheet resistance results at the selected frequency. Sheet resistance map of the wafer at 0.5 THz, with step size 1 mm.. 32
Figure B.1 – Photograph of a graphene film on a 100 mm quartz wafer and related conductivity map.....33
Figure B.2 – Detailed view of subsections.....34
Figure C.1 – Reflection and transmission for a thin film on a substrate 35
Figure C.2 – Reflection and transmission for a thin film on a substrate. 40

Table A.1 – Illustration of measurement protocol 26
Table A.2 – Product identification 30
Table A.3 – General material description 30
Table A.4 – Sampling plan.....31
Table A.5 – Measurement related information.....31
Table A.6 – Measurement results: Sheet resistance at the selected frequency.....32

INTERNATIONAL ELECTROTECHNICAL COMMISSION

_____

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

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

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

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

Draft	Report on voting
113/568/DTS	113/604/RVDTS

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

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

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

A list of all parts of the IEC TS 62607 series, published under the general title *Nanomanufacturing – Key control characteristics*, can be found on the IEC website.

The committee has decided that the contents of this document will remain unchanged until the stability date indicated on the IEC website under webstore.iec.ch in the data related to the specific document. At this date, the document will be

- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

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.

INTRODUCTION

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

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

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

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

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

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

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

1 Scope

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

– sheet resistance (R_s)

for films of graphene-based materials by

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

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

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

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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

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

3.1 General terms

3.1.1
graphene
graphene layer
single-layer graphene
monolayer graphene
1LG
single layer of carbon atoms with each atom bound to three neighbours in a honeycomb structure