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TECHNICAL SPECIFICATION

colour inside

Nanomanufacturing – Key control characteristics –
Part 6-1: Graphene-based material – Volume resistivity: four probe method

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FOREWORD

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- the subject is still under technical development or where, for any other reason, there is the future but no immediate possibility of an agreement on an International Standard.

Technical Specifications are subject to review within three years of publication to decide whether they can be transformed into International Standards.

IEC TS 62607-6-1, which is a Technical Specification, has been prepared by IEC technical committee 113: Nanotechnology for electrotechnical products and systems.

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

Draft TS	Report on voting
113/454/DTS	113/511/RVDTS

Full information on the voting for the approval of this Technical Specification can be found in the report on voting indicated in the above table.

This document has been drafted in accordance with the ISO/IEC Directives, Part 2.

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 "http://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- withdrawn,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 publication 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 has attracted great interest as a next generation electronic material due to its extremely high mobility and ballistic transport of electrons [1] [1]. The unique properties of graphene enable it to be an effective candidate used in electronic products such as transparent conducting films, sensors, field emitters, supercapacitors, etc.

Characterization of the electrical properties of graphene itself is essential to both manufacturers and users in order to develop innovative electronic devices or to improve existing electronic devices using it.

Commercialized graphene products can be categorized by their manufacturing methods as follows: (i) graphene flakes and/or nanoplatelets in powder form (hereinafter called GNP), (ii) graphene sheets suspended in liquids or (iii) graphene films grown by chemical vapour deposition (CVD).

Many electrical properties of a powder-type graphene product are significantly affected by its geometric and electronic parameters [2]. First, it is required to select the parameter that best represents the quality of the graphene products. Second, a suitable sample preparation for the purpose of electrical measurements is also elucidated and described. Finally, measurement conditions are also crucial factors to determine the representative value of the powder product in terms of its electrical properties.

Among the measurands in determining electronic properties of powder-type graphene – sheet resistance (or conductance), resistivity (or conductivity), volume resistivity (or volume conductivity), and so on – this document selects volume resistivity (or volume conductivity) for the representative measurand which reveals the electrical properties of powder-type graphene through a series of experiments.

This document describes a simple method to evaluate the volume resistivity (or volume conductivity) of powder-type graphene, which includes preparation of its pellet and a measurement method.

Case studies illustrating the application of the standard are provided in Annex A.

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

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 6-1: Graphene-based material – Volume resistivity: four probe method

1 Scope

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

– volume resistivity

  for powder consisting of graphene-based material like flakes of graphene, few layer graphene and/or reduced graphene oxide after preparation of a sample in pellet form by

– four probe method

  using powder resistivity measurement system.

The volume resistivity is a measure of the quality of powder-type graphene products in terms of electrical property and reflects the density-dependency shown in a pellet of powder-type graphene.

The volume conductivity can directly be derived from the volume resistivity.

Typical application areas are industries that use powder-type graphene products for graphene manufacture, potential developers, and users who produce graphene-based products. As the volume resistivity measured according to this document requires the preparation of a sample in the form of a pellet, this document describes in detail

– an apparatus to prepare consistently a test sample, the pellet,
– the preparation of the pellet starting from powder-type graphene,
– the measurement procedure to measure the volume resistivity (or volume conductivity) of the pellet, and
– the data analysis, the interpretation and reporting of the results.

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

NOTE A comprehensive nanotechnology vocabulary is under ongoing development in IEC TC 113 and ISO/TC 229, in cooperation with ISO/TC 229. The vocabulary is being published as different parts of IEC TS 80004 and ISO/TS 80004. The terms and definitions in this document are harmonized with the terms and definitions of IEC 80004 (all parts) and ISO 80004 (all parts). They will remain harmonized during the maintenance of the document. Definitions not yet specified are taken from scientific literature.

3.1 General terms

3.1.1
graphene pellet
disc formed by compression of graphene powder

3.1.2
reduced graphene oxide
rGO
reduced oxygen content form of graphene oxide

Note 1 to entry This can be produced by chemical, thermal, microwave, photo-chemical, photo-thermal or microbial/bacterial methods or by exfoliating reduced graphite oxide.

Note 2 to entry: If graphene oxide was fully reduced then graphene would be the product. However, in practice, some oxygen containing functional groups will remain and not all sp^3 bonds will return back to sp^2 configuration. Different reducing agents will lead to different carbon to oxygen ratios and different chemical compositions in reduced graphene oxide.

[SOURCE: ISO/TS 80004-13:2017 [3], 3.1.2.14]

3.1.3
graphene-based material
GBM
graphene material
grouping of carbon-based 2D materials that include one or more of graphene, bilayer graphene, few-layer graphene, graphene nanoplate, and functionalized variations thereof as well as graphene oxide and reduced graphene oxide

Note 1 to entry: "Graphene material" is a short name for graphene-based material.

3.2 Key control characteristics

3.2.1
volume resistivity
<of pellet formed from graphene-based material> key control characteristic to describe the quality of powder-type graphene products

Note 1 to entry The measured volume resistivity of powder-type graphene products mainly depends on geometric factors such as flake size and layer numbers of a flake. As the applied pressure into a pellet increases, electrical contact among flakes is improved. The measurand "volume resistivity" reflects the density-dependency shown in a pellet of powder-type graphene-based material.

3.3 Terms related to measurements

3.3.1
volume conductivity
electrical conductivity
conductivity
σ
characteristic physical property of three-dimensional materials describing the ability to conduct electric current

Note 1 to entry The volume conductivity is defined by Ohm's law, $j = \sigma \times E$, where j is the current density measured in amperes per square metre (A/m^2) and E the electric field in volts per metre (V/m).

Note 2 to entry The volume conductivity can be obtained by dividing the surface conductance by the conductor thickness (t): $\sigma = \sigma_S/t$. The unit of measure of σ is siemens per metre (S/m).

[SOURCE: IEC TS 62607-6-4:2016, 3.2.3]

3.3.2
volume resistivity
electrical resistivity
resistivity
ρ
reciprocal of the volume conductivity

Note 1 to entry The volume resistivity is defined by Ohm's law, $j = \rho^{-1} \times E$, where j is the current density measured in amperes per square metre (A/m^2) and E the electric field in volts per metre (V/m).

Note 2 to entry The volume resistivity can be obtained by multiplying the surface resistance (ρ_s) by the conductor thickness (t): $\rho = \rho_s \times t$. The SI unit of ρ is the ohm metre (Ω·m).

3.3.3
surface conductance
sheet conductance
σ_s
characteristic physical property of two-dimensional materials describing the ability to conduct electric current

Note 1 to entry The SI unit of measure of σ_s is siemens (S). In the trade and industrial literature, however, siemens per square (S/square) is commonly used when referring to surface conductance. This is to avoid confusion between surface conductance and electric conductance (G), which share the same unit of measure:

$$G = I/U = \sigma_s\ (w/l).$$

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

[SOURCE: IEC TS 62607-6-4:2016, 3.2.1]

3.3.4
surface resistance
sheet resistance
ρ_s
reciprocal of surface conductance

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

[SOURCE: IEC TS 62607-6-4:2016, 3.2.4]

3.3.5
conductance
electrical conductance
G
reciprocal of the resistance

Note 1 to entry The conductance is defined by Ohm's law, $I = G \cdot U$, where I is the current through the conductor measured in amperes and U the potential difference along the conductor measured in volts.

Note 2 to entry The conductance is related to the material property conductivity σ by $G = A \times \sigma/L$, where A is the cross-section of the conductor in square metres and L its length in metres. The SI unit of G is siemens (S).

3.3.6
resistance
electrical resistance
R
ratio of the potential difference along a conductor and the current through the conductor

Note 1 to entry The resistance is defined by Ohm's law, $U = R \times I$, where I is the current through the conductor measured in amperes and U the potential difference along the conductor measured in volts.

Note 2 to entry The resistance is related to the material property resistivity ρ by $R = \rho \times L/A$, where A is the cross section of the conductor in square metres and L its length in metres. The SI unit of R is the ohm (Ω).

[SOURCE: ISO 15091:2012, 3.1]

3.3.7
four probe method
method to measure electrical sheet resistance, impedance 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.

[SOURCE: ISO/TS 80004-13:2017, 3.3.3.1, modified – The term "four-terminal sensing" has been deleted and the term "four point probe method" has been replaced by "four probe method".]

4 Sample preparation

For measurement of graphene powders using the four probe method, the sample should be prepared with uniform surface over a large area [4]. Two types of sample are suitable for this measurement: pellet or film. The sample shall be made in the form of a pellet because generating a uniform film from powder-type graphene is difficult due to its low solubility in any solvent.

Graphene powder obtained from commercial sources is pelletized after vacuum drying at 80 °C for 24 h in order to remove any remnant of water content in the sample.

5 Measurement of volume resistivity of graphene pellet

5.1 Description of the measurement apparatus

The powder resistivity measurement system consists of two components [5],[6]:

1) a pelletizer and electrode unit;
2) an electrical measurement system.

The details of the measurement system are shown in Figure 1.

A pelletizer is composed of a four-probe head, a piston, and a cylinder. The head of the piston and the inside of the cylinder are covered with a non-conducting material, zirconia, so that the specimen is electrically isolated from all sides. The pelletizer is electrically connected to a four-probe unit through a probe connector. The four-probe setup consists of four equally-spaced copper or gold rods with identical radius. The inner diameter of the cylinder is 20,4 mm, the probe spacing is 1,6 mm, and the diameter of each of the four electrodes is 1,4 mm. The applied pressure is measured by a pressure gauge with high precision for a maximum pressure of 52 MPa. A height gauge is used to measure the height of the piston at a given pressure. The height difference before and after pressurizing is referred to as the pellet thickness.