IEC TS 62607-6-2

Edition 1.0 2023-04

TECHNICAL SPECIFICATION

colour inside

Nanomanufacturing – Key control characteristics – Part 6-2: Graphene – Number of layers: atomic force microscopy, optical transmission, Raman spectroscopy

IEC TS 62607-6-2:2023-04(en)

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INTERNATIONAL
ELECTROTECHNICAL
COMMISSION

ICS 07.120 ISBN 978-2-8322-6749-3

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IEC TS 62607-6-2 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
113/676/DTS	113/727/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 http://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- withdrawn,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.

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INTRODUCTION

Graphene has attracted significant interest as a next-generation electronic material due to its good conductivity and mobility. It has been regarded as more advantageous than carbon nanotube (CNT) because of its isotropic and homogeneous electronic properties. For these reasons and many more, a Nobel prize in physics was awarded to A. Geim and C. Novoselov in 2010 for their efforts in discovering graphene when they isolated a single layer of graphene using clear adhesive tape.

Graphene has been widely studied by researchers from academic institutions, research institutes, and industries due to its unique and interesting properties such as conductivity [1][1], mechanical strength and flexibility [2], which are better than other metals or semiconductors. These properties are influenced by the number of layers of graphene and disappear as the number of layers increases. Graphene also shows an unusual reduction in optical transparency even considering a single atomic layer [3]. Therefore, graphene applications need to investigate the precise number of layers of graphene.

Many companies are now providing graphene samples to industries and research communities. These are prepared (or manufactured) by various methods such as CVD or mechanical exfoliation. Defining and evaluating the number of layers of this fabricated graphene is critical both from research and industrial points of view. Unfortunately, there are no commonly accepted standards for this purpose, hindering the reliable production and expansion of graphene applications.

The number of layers of graphene is usually observed by atomic force microscopy (AFM), light transmittance, Raman spectroscopy, transmission electron microscopy (TEM), and ellipsometry. Every analytical method has its own limitations in terms of precisely measuring the number of graphene layers and can also cause ambiguity for providing reliable information. For these reasons, developing an easy, fast, and reliable method for counting the number of graphene layers is needed.

This document describes a combined method to evaluate accurate number of layers of graphene, which includes measurement method.

Description of combined method and case studies illustrating the application of the standard are provided in Annex A and Annex B, respectively.

1 Numbers in square brackets refer to the Bibliography.

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 6-2: Graphene – Number of layers: atomic force microscopy, optical transmission, Raman spectroscopy

1 Scope

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

– number of layers

for graphene flakes by a combination of

– atomic force microscopy,
– optical transmission, and
– Raman spectroscopy

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 terminology databases for use in standardization at the following addresses:

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

3.1 General terms

3.1.1
graphene
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 80004-13:2017 [4], 3.1.2.1]

3.1.2
graphene oxide
GO
chemically modified graphene prepared by oxidation and exfoliation of graphite, causing extensive oxidative modification of the basal plane

Note 1 to entry: Graphene oxide is a single-layer material with a high oxygen content, typically characterized by C/O atomic ratios of approximately 2,0 depending on the method of synthesis.

[SOURCE: ISO/TS 80004-13:2017, 3.1.2.13]

3.1.3
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.

Note 3 to entry: It can take the form of several morphological variations such as platelets and worm-like structures.

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

3.1.4
bilayer graphene
2LG
two-dimensional material consisting of two well-defined stacked graphene layers

Note 1 to entry: If the stacking registry is known it can be specified separately, for example as “Bernal stacked bilayer graphene”.

[SOURCE: ISO/TS 80004-13:2017, 3.1.2.6]

3.1.5
few-layer graphene
FLG
two-dimensional material consisting of three to ten well-defined stacked graphene layers

[SOURCE: ISO/TS 80004-13:2017, 3.1.2.10]

3.1.6
graphite
allotropic form of the element carbon, consisting of graphene layers stacked parallel to each other in a three-dimensional, crystalline, long-range order

Note 1 to entry: Adapted from the definition in the IUPAC Compendium of Chemical Terminology.

Note 2 to entry: There are two allotropic forms with different stacking arrangements: hexagonal and rhombohedral.

[SOURCE: ISO/TS 80004-13:2017, 3.1.2.2]

3.1.7
highly oriented pyrolytic graphite
HOPG
highly pure and ordered form of synthetic graphite

Note 1 to entry: Material often used as reference material for calibration of measurement equipment.

3.1.8
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 ten 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.

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

3.1.9
chemical vapour deposition
CVD
deposition of a solid material onto a substrate by chemical reaction of a gaseous precursor or mixture of precursors, commonly initiated by heat

[SOURCE: ISO/TS 80004-8:2020 [5], 8.2.4]

3.2 Terms related to measurements

3.2.1
atomic force microscopy
AFM
method for imaging surfaces by mechanically scanning their surface contours, in which the deflection of a sharp tip sensing the surface forces, mounted on a compliant cantilever, is monitored

Note 1 to entry: AFM can provide a quantitative height image of both insulating and conducting surfaces.

Note 2 to entry: Some AFM instruments move the sample in the x-, y- and z-directions while keeping the tip position constant, and others move the tip while keeping the sample position constant.

Note 3 to entry: AFM can be conducted in a vacuum, a liquid, a controlled atmosphere or air. Atomic resolution may be attainable with suitable samples, with sharp tips, and by using an appropriate imaging mode.

Note 4 to entry: Many types of force can be measured, such as the normal forces or the lateral, friction or shear force. When the latter is measured, the technique is referred to as lateral, frictional or shear force microscopy. This generic term encompasses all of these types of force microscopy.

Note 5 to entry: AFMs can be used to measure surface normal forces at individual points in the pixel array used for imaging.

[SOURCE: ISO 18115-2:2021 [6], 3.1.2, modified – Note 6 to entry has been deleted.]

3.2.2
offset height
difference between the height of monolayer graphene on the substrate using AFM and the actual height of monolayer graphene

Note 1 to entry: The offset height can be affected by the type of substrate, AFM mode, and environment.

Note 2 to entry: Large thickness variation of monolayer graphene (0,4 nm to 1,7 nm) due to the offset height has been reported [7].