IEC TS 62607-6-14

Edition 1.0 2020-10

TECHNICAL SPECIFICATION

colour inside

Nanomanufacturing – Key control characteristics –
Part 6-14: Graphene-based material – Defect level: Raman spectroscopy

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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 62607-6-14, 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:

Enquiry draft	Report on voting
113/495/DTS	113/536/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- withdrawn,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.

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INTRODUCTION

Graphene has been intensively studied by researchers from both academic and industrial communities due to its unique properties, which include exceptional thermal conductivity, great strength and excellent transparency. Defects in graphene influence its optical and magnetic performance, electronic structure and thermal conductivity, thus influencing its applications. Therefore, defect is a key control characteristic for the fabrication of high-quality graphene for desired applications.

One of the most useful methods to evaluate defect level in graphene is Raman spectroscopy, which is sensitive to the structure of samples. This method is efficient, non-contact and well-understood. The defect states and boundary states of realistic graphene material will induce a series of Raman scattering processes (Figure 1). Some of scattering processes are only associated with defective states, which are used in this document to analyse defect level in graphene powder.

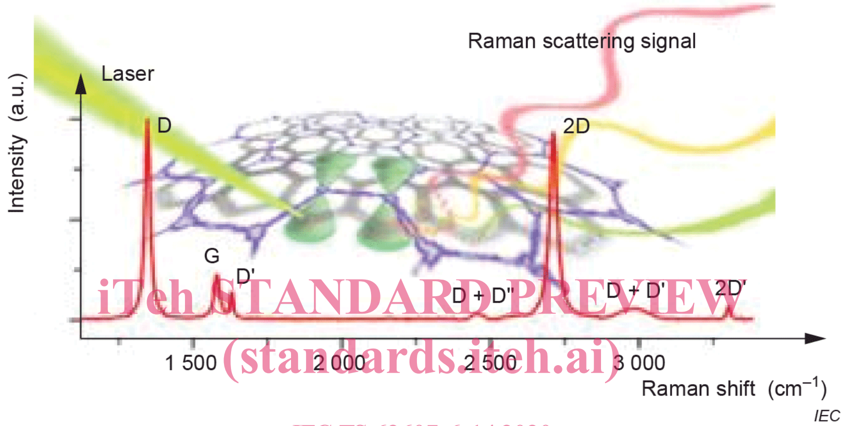

Figure 1 – Schematic diagram of Raman scattering processes in realistic graphene material

Commercialized graphene samples can be classified by their physical forms as graphene film, graphene powder and graphene solution. Figure 2 shows the schematic packing configurations of graphene flakes in graphene film (left side of Figure 2) and graphene powder (right side of Figure 2) and their corresponding SEM images.

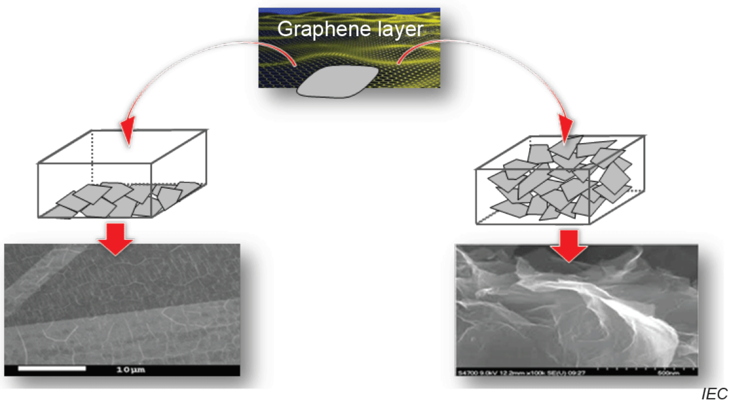

Figure 2 – Different packing configurations of graphene flakes in film (left) and powder (right)

Usually, defects in graphene films are characterized by the intensity ratio of two principle bands – D band and G band – in Raman spectra (symbolized by I_D/I_G) [1],[2]. However, in graphene powders consisting of flakes with sizes below 10 µm there are numerous edges and boundary states, which all contribute to the D-band signal and make its correlation to various defects problematic. The D-band intensity could result from the contribution of edges, boundary states or defects, so it is not appropriate to determine the defect level of graphene powder with the parameter I_D/I_G.

D+D′ band is only relevant with defects in graphene powder, but not with edges and boundary states. Therefore, in order to characterize defect level in graphene powder, the intensity ratio of D+D′ and 2D bands (symbolized by $I_{D+D'}/I_{2D}$) is proposed as a more relevant parameter in this document. Detailed information can be found in Annex D.

NANOMANUFACTURING –
KEY CONTROL CHARACTERISTICS –

Part 6-14: Graphene-based material – Defect level: Raman spectroscopy

1 Scope

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

- defect level

for powders consisting of graphene-based material by

- Raman spectroscopy.

The defect level is derived by the intensity ratio of the D+D′ band and 2D band in Raman spectrum, $I_{D+D'}/I_{2D}$.

- The defect level determined in accordance with this document will be listed as a key control characteristic in the blank detail specification for graphene IEC 62565-3-1 for graphene powder.
- The method is applicable for graphene powder or graphene-based material, e.g. reduced graphene oxide (rGO), bilayer graphene, trilayer graphene and few-layer graphene.
- Typical application areas are quality control and classification for graphene manufacturers, and product selection for downstream users.
- The method described in this document is appropriate if the physical form of graphene is powder.

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
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]

3.1.2
graphene
graphene layer
single-layer graphene
monolayer 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-layered 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, 3.1.2.1]

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.

3.1.4
bilayer graphene
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
trilayer graphene
two-dimensional material consisting of three well-defined stacked graphene layers

Note 1 to entry: If the stacking registry is known, it can be specified separately, for example, as "twisted trilayer graphene".

[SOURCE: ISO/TS 80004-13:2017, 3.1.2.9]

3.1.6
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]

3.1.7
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 primary allotropic forms with different stacking arrangements: hexagonal and rhombohedral.

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

3.1.8
blank detail specification
BDS
structured generic specification of the set of key control characteristics which are needed to describe a specific nano-enabled product without assigning specific values and/or attributes

Note 1 to entry: The templates defined in a blank detail specification list the key control characteristics for the nano-enabled material or product without assigning specific values to it.

Note 2 to entry: Examples of nano-enabled products are: nanomaterials, nanocomposites and nano-subassemblies.

Note 3 to entry: Blank detail specifications are intended to be used by industrial users to prepare their detail specifications used in bilateral procurement contracts. A blank detail specification facilitates the comparison and benchmarking of different materials. Furthermore, a standardized format makes procurement more efficient and more error robust.

3.1.9
sectional blank detail specification
SBDS
specification based on a blank detail specification adapted for a subgroup of the nano-enabled product

Note 1 to entry: In general, the sectional blank detail specification contains a subset of those key control characteristics listed in the blank detail specification. In addition, sectional specific key control characteristics can be added if they are not listed in the blank detail specification.

Note 2 to entry: The templates defined in the sectional blank detail specification can contain key control characteristics with and without assigned values and attributes.

Note 3 to entry: The section can be defined by application, manufacturing method or general material properties.

3.1.10
detail specification
DS
specification based on a blank detail specification with assigned values and attributes

Note 1 to entry: The properties listed in the detail specification are usually a subset of the key control characteristics listed in the relevant blank detail specification. The industrial partners define only those properties which are required for the intended application.

Note 2 to entry: Detail specifications are defined by the industrial partners. SDOs will be involved only if there is a general need for a detail specification in an industrial sector.

Note 3 to entry: The industrial partners can define additional key control characteristics if they are not listed in the blank detail specification.

3.1.11
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.