IEC TS 62607-6-11

Edition 1.0 2022-02

TECHNICAL SPECIFICATION

colour inside

Nanomanufacturing – Key control characteristics – Part 6-11: Graphene – Defect density: Raman spectroscopy

IEC TS 62607-6-11:2022-02(en)

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

ICS 07.120

ISBN 978-2-8322-1071-9

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

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

Draft	Report on voting
113/591/DTS	113/626/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

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- withdrawn,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

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INTRODUCTION

Graphene is a single layer of carbon atoms arranged in a honeycomb lattice. Due to its outstanding properties such as high mobility and flexibility, it has high potential for future applications. Structural defects, e.g. anything that changes the regularity of the lattice, have a huge influence on the properties of graphene, especially the mobility. For most electronic applications having high quality, almost defect-free graphene is crucial. Thus the defect density as a measure of the structural quality of graphene is a key control characteristic of graphene.

Raman spectroscopy is one of the most widely used characterization techniques in carbon science and technology. There are two main peaks in the Raman spectrum of graphene, the G peak located around 1 580 cm^{-1} and the 2D peak located around 2 680 cm^{-1} for an excitation wavelength of 514 nm. Raman spectroscopy can be used to extract valuable information about the sample properties such as the number of layers, doping level, amount and type of strain as well as defect density [1][1]. Quantifying defects in graphene is crucial for both gaining insight in fundamental properties and for applications. Defects strongly affect the mobility of graphene. It is thus important for device fabrication and optimization as well as a quality check to know the defect density in a sample.

Disorder of the graphene lattice can be described [2] in a three-stage classification, leading from graphite to amorphous carbons, that allows to simply assess all the Raman spectra of carbons:

- Stage 1: graphene to nanocrystalline graphene.
- Stage 2: nanocrystalline graphene to low-sp^3 amorphous carbon.
- Stage 3: low-sp^3 amorphous carbon to high-sp^3 amorphous carbon.

This classification is illustrated in Figure 1.

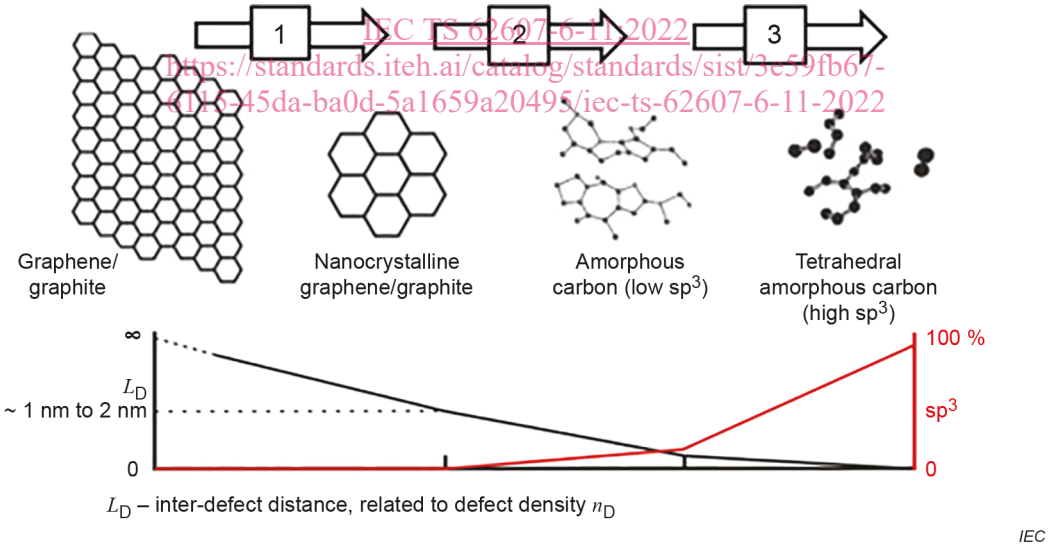

Figure 1 – Three-stage classification to describe graphene lattice disorder

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

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 6-11: Graphene – Defect density: Raman spectroscopy

1 Scope

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

- defect density n_D

of graphene films grown by chemical vapour deposition as well as exfoliated graphene flakes by

- Raman spectroscopy.

The defect density n_D is derived from the intensity ratio of the D-peak and the G-peak I(D)/I(G) in the Raman spectrum based on the three-stage model for amorphization.

- The classification helps manufacturers to classify their material quality and customers to provide an expectation of the electronic performance of the classified graphene and more specifically to decide whether or not the graphene material quality is potentially suitable for various applications.
- The defect density n_D determined in accordance with this document is listed as a key control characteristic in the blank detail specification for graphene IEC 62565-3-1. The inter-defect distance L_D can be calculated from the defect density n_D and is an equivalent measure of defects in the graphene lattice.
- The method is applicable for exfoliated graphene and graphene grown on or transferred to a substrate with I(D)/I(G) in the range of 0,1 to 3, which corresponds to a defect density of 2,46 × 10^{10} cm^{-1} up to 7,39 × 10^{11} cm^{-2} for an excitation energy of 2,41 eV (514 nm), corresponding to stage 1 of the three-stage model for amorphization.
- The spatial resolution is in the order of 1 µm given by the spot size of the exciting laser.
- The method is complementary to the method described in IEC 62607-6-6 and is used if the Raman spectrum shows a visible D-peak with an intensity ratio I(D)/I(G) in the range of 0,1 to around 3.

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
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.

3.1.2
chemical vapour deposition
CVD
deposition of a solid material by chemical reaction of a gaseous precursor or mixture of precursors, commonly initiated by heat on a substrate.

[SOURCE: ISO/TS 80004-13:2017, 3.2.1.1]

3.1.3
defect
local deviation from regularity in the crystal lattice of a 2D material

[SOURCE: ISO/TS 80004-13:2017, 3.4.1.1]

3.1.4
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-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, 3.1.2.1]

3.1.5
point defect
<2D material> defect that occurs only at or around a single lattice point of a 2D material

Note 1 to entry: Point defects generally involve at most a few missing, dislocated or different atoms creating a vacancy or vacancies, extra atoms (interstitial defects) or replaced atoms.

[SOURCE: ISO/TS 80004-13:2017, 3.4.1.2]

3.1.6
roll-to-roll production
R2R production
<2D material> CVD growth of 2D material upon a continuous substrate that is processed as a rolled sheet, including transfer of 2D material to a separate substrate

[SOURCE: ISO 80004-13:2017, 3.2.1.2]

3.2 Key control characteristics measured in accordance with this document

3.2.1
defect density
n_D
number of point defects per area

Note 1 to entry: The unit of the defect density is cm^{-2}.

Note 2 to entry: The defect density is a key control characteristic to describe local deviations from regularity in the crystal lattice of graphene according to stage 1 of the three-stage model for amorphization.

Note 3 to entry: The defect density is related to the mean distance between point defects L_D by $n_D = 10^{-4}/\pi L_D^2$.

3.2.2
inter-defect distance
L_D
mean distance between point defects in the crystal lattice of graphene

Note 1 to entry: The unit of the inter-defect distance is the nanometre (nm).

Note 2 to entry: The inter-defect distance is a key control characteristic to describe local deviations from regularity in the crystal lattice of graphene according to stage 1 of the three-stage model for amorphization.

Note 3 to entry: The defect density n_D is related to the inter-defect distance by $n_D = 10^{-4}/\pi L_D^2$.

3.3 Terms related to the measurement method described in this document

3.3.1
2D peak
second order Raman peak related to a two-phonon process located at approximately twice the frequency of the D peak

Note 1 to entry: As well as the D peak the 2D peak is also dispersive with wavelength. The position of the 2D peak changes strongly with laser energy

Note 2 to entry: The 2D peak is always present in the Raman spectrum of graphene and does not need defects to be activated.

3.3.2
D peak
defect activated Raman peak related to lattice breathing modes in six-carbon rings away from the centre of the Brillouin zone

Note 1 to entry: The D peak is located between 1 270 cm^{-1} and 1 450 cm^{-1} depending on the wavelength of the excitation laser. The dispersion with wavelength is around 50 cm^{-1}/eV.

Note 2 to entry: The D peak is most intense at defective graphene lattices and disappears for perfect monolayer crystals. Therefore it is often called the disorder band.

3.3.3
D' peak
defect activated Raman peak in the spectrum of graphene located around 1 620 cm^{-1} originating from scattering away from the Brillouin zone centre.

3.3.4
G peak
Raman peak related to in-plane motion of the carbon atoms located near 1 580 cm^{-1} originating from scattering at the centre of the Brillouin zone

Note 1 to entry: The G peak can be observed in pristine graphene and does not need lattice defects to occur.

3.3.5
laser spot size
diameter of circular laser spot on sample when sample is in focus

Note 1 to entry: Diameter is measured at the full width at half maximum (FWHM) of the intensity distribution.

3.3.6
Raman spectroscopy
spectroscopy in which the radiation emitted from a sample illuminated with monochromatic radiation is characterized by an energy loss or gain arising from rotational, vibrational or phonon excitations

[SOURCE: ISO/TS 80004-13:2017, 3.3.1.6]

4 General

4.1 Measurement principle

Raman spectroscopy is very sensitive to defects. In addition to the G and 2D peaks, which are always present in the Raman spectrum of graphene, Figure 2 top spectrum, additional defect activated peaks appear in the Raman spectrum of defective graphene. As shown in the bottom spectrum in Figure 2, the Raman spectrum of defective graphene changes as follows: the defect activated D, D′ peaks and their combination D+D′ appear.

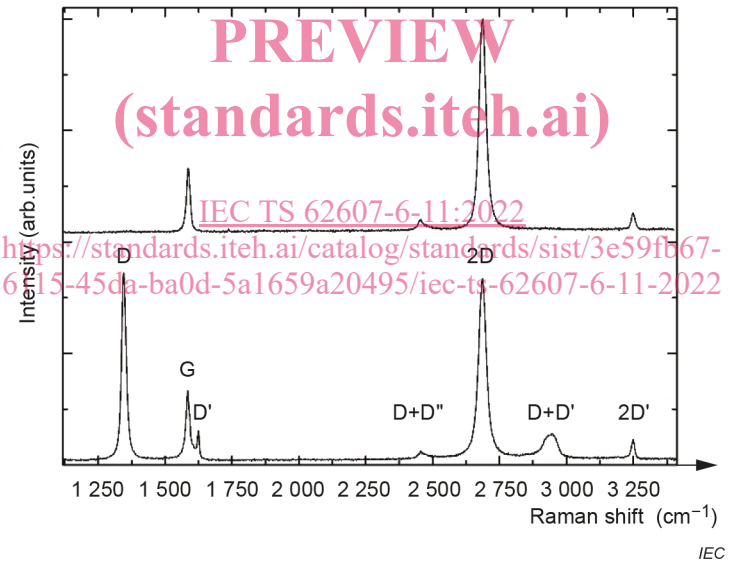

Figure 2 – Raman spectra of pristine (top) and defective graphene (bottom) [1]

In this document we focus on stage 1 of the three-stage model for amorphization, the most relevant when considering the vast majority of publications dealing with graphene production, processing and applications. In stage 1 the intensity of the D peak increases for increasing amount of defects. The intensity ratio of the D peak to the G peak, I(D)/I(G), is then directly linked to the inter-defect distance and the defect density in the sample.

The inter-defect distance L_D can be expressed by [3]:

$$L_D^2 = \frac{4{,}3\times10^3}{E_L^4}\left[\frac{I(D)}{I(G)}\right]^{-1} \quad (1)$$