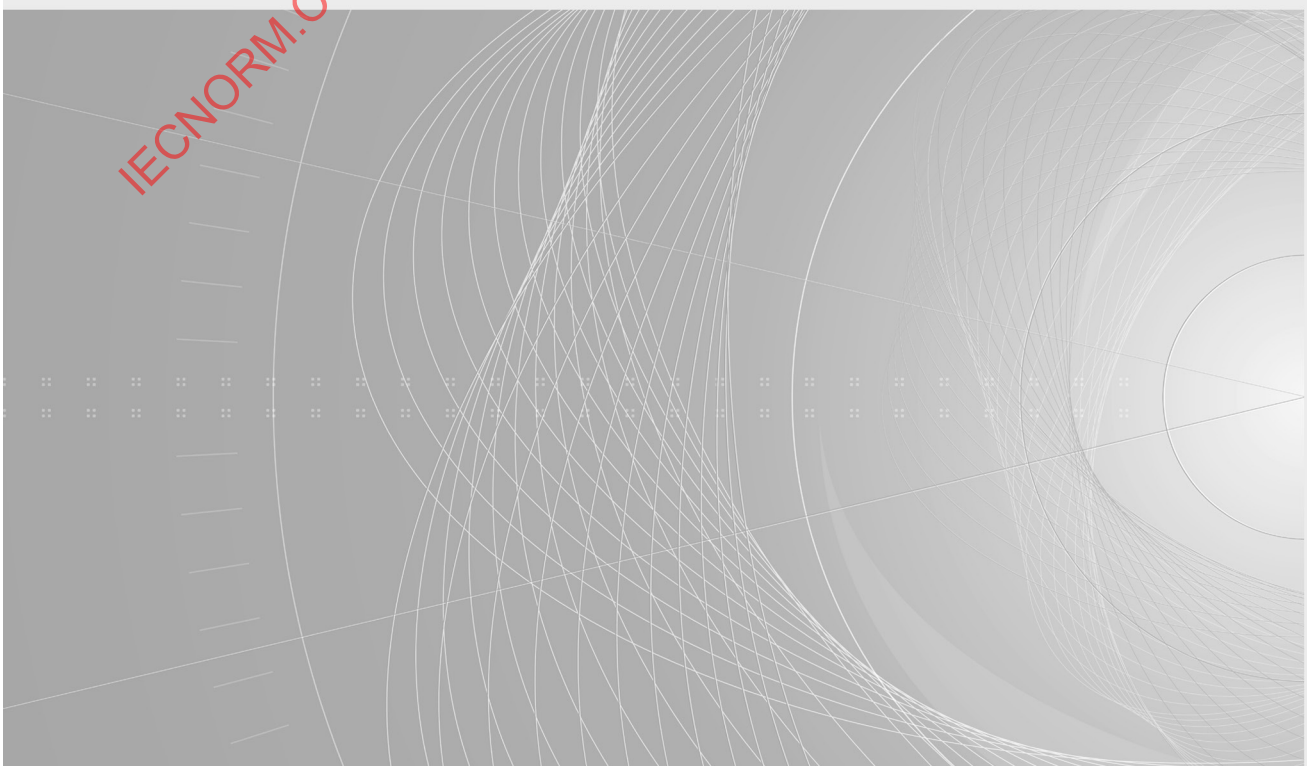
IEC TS 62607-6-11

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

INTERNATIONAL ELECTROTECHNICAL COMMISSION

ICS 07.120

ISBN 978-2-8322-1071-9

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NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

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

FOREWORD

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IEC TS 62607-6-11 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
113/591/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

- reconfirmed,
- withdrawn,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.

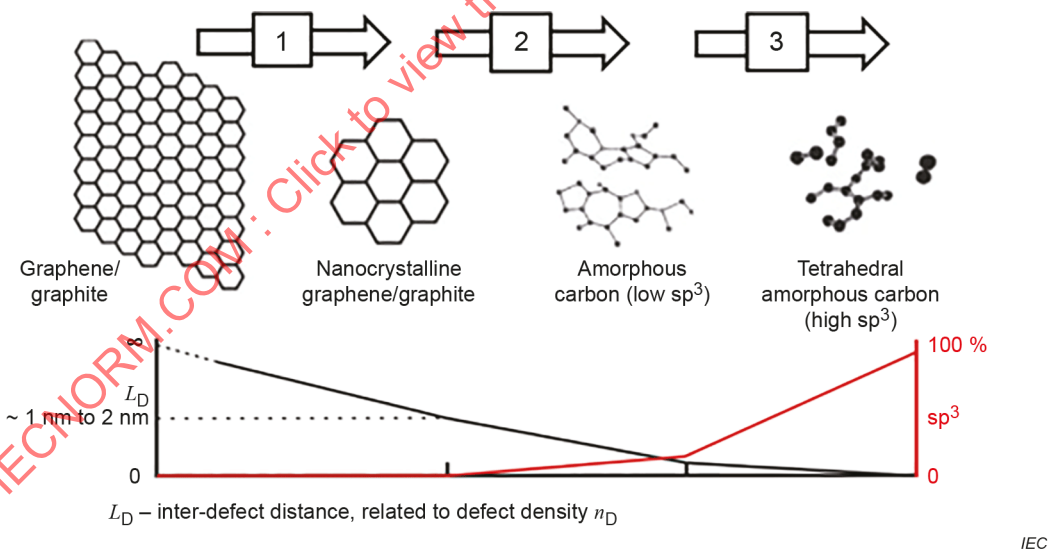


Figure 1 – Three-stage classification to describe graphene lattice disorder

[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 $2{,}46 \times 10^{10}$ cm^{-1} up to $7{,}39 \times 10^{11}$ 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.

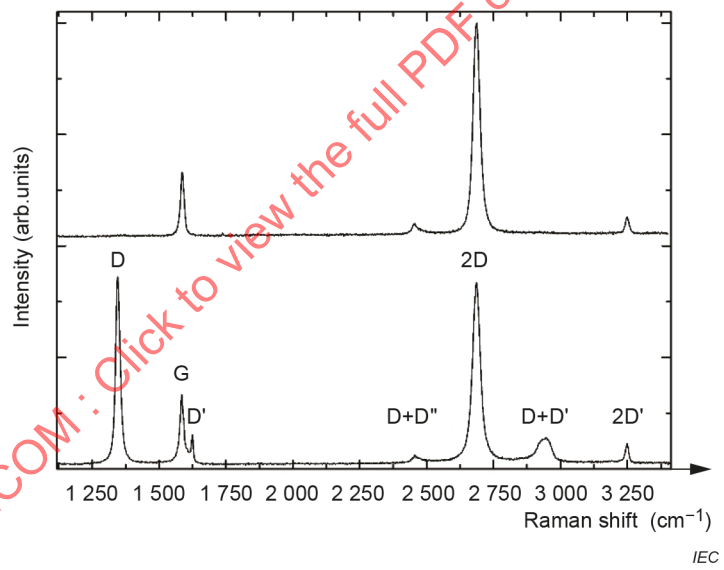


Figure 2 – Raman spectra of pristine (top) and defective graphene (bottom) [1]

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]:

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

E_L is the laser excitation energy in electronvolts (eV) (E_L = 2,41 eV at λ = 514 nm). By considering point defects, separated from each other by L_D in nm, Formula (1) can be restated in terms of defect density, n_D, in cm^{-2}, given by $n_D = 10^{14}/\pi L_D^2$, as [3]:

$$n_D = 7{,}3 \times 10^9 E_L^4 \frac{I(D)}{I(G)} \quad (2)$$

This relation is valid for $L_D \geq 10$ nm corresponding to $n_D \leq 3{,}2 \times 10^{11}$ cm^{-2} (stage 1). Note that Formula (1) and Formula (2) are limited to Raman-active defects. Perfect zig-zag edges [4][5], charged impurities [6][7], intercalants [8], and uniaxial and biaxial strain [9][10] do not generate a D peak in the Raman spectrum of graphene [1].

Examples for Raman spectra for graphene with defects are given in Annex D. Clause D.1 shows spectra for undoped graphene and an inter-defect distance between 2 nm and 24 nm. The influence of doping is described in Clauses D.2 and D.3. Clause D.2 also provides the modification of Formula (1) and Formula (2) for doped graphene.

4.2 Sample preparation method

The method described in this document can be reliably applied to graphene on any substrate, e.g. transferred to Si/SiO_2, or grown on copper or nickel by chemical vapour deposition, direct exfoliation or transferred from another substrate. A special preparation of the sample is not necessary.

Before the measurement, it shall be checked that there are no substrate generated Raman peaks overlapping with the D, G, D′, 2D and D+D′ peaks in the Raman spectrum of graphene. Suitable substrates should have a flat background in the Raman spectrum in the range where these peaks are located, e.g. 1 200 cm^{-1} to 1 700 cm^{-1} and 2 300 cm^{-1} to 3 500 cm^{-1}.

4.3 Description of test equipment

To analyse samples by Raman spectroscopy a micro-Raman setup is used, where the laser is focused with a microscope objective to a laser spot size ≤ 1 µm. The power on the sample should be kept well below 1 mW to avoid laser-induced heating effects and burning of the sample. This corresponds to a laser power density of 1,27 $mW/\mu m^2$. Recommendations for wavelengths depending on the substrate are summarized in Annex C.

Raman measurements are carried out using a micro-spectrometer equipped with a high magnification objective (numerical aperture > 0,8).

The laser beam can be either delivered with a standard free beam path directed by mirrors or via fibres. Figure 3 shows a schematic of a micro-Raman setup. The beam is delivered from a laser. The light beam shall be cleaned by a laser line filter to filter the plasma lines and the laser power is controlled by a neutral density (ND) filter to attenuate the power. The beam is then delivered by either mirrors or fibres to the microscope and directed through an objective and focused on the sample. The sample sits on a (in most cases motorized) stage, which controls its position and allows the sample to be moved with respect to the laser beam. The scattered light is then collected in backscattering geometry through the objective and guided by mirrors to the spectrometer. The scattered light is a mixture of Rayleigh (elastically) and Raman (inelastically) scattered light. Due to its orders of magnitude higher intensity compared to the Raman scattered light, an additional filter (Raman filter with optical density of at least 9) is needed to attenuate the Rayleigh scattered light before the signal is going to the spectrometer, where the signal is dispersed by a grating and then collected by a detector, usually a charged coupled device (CCD). Setups also have a camera attached to the microscope, to enable the user to view the sample via the camera on a computer screen.

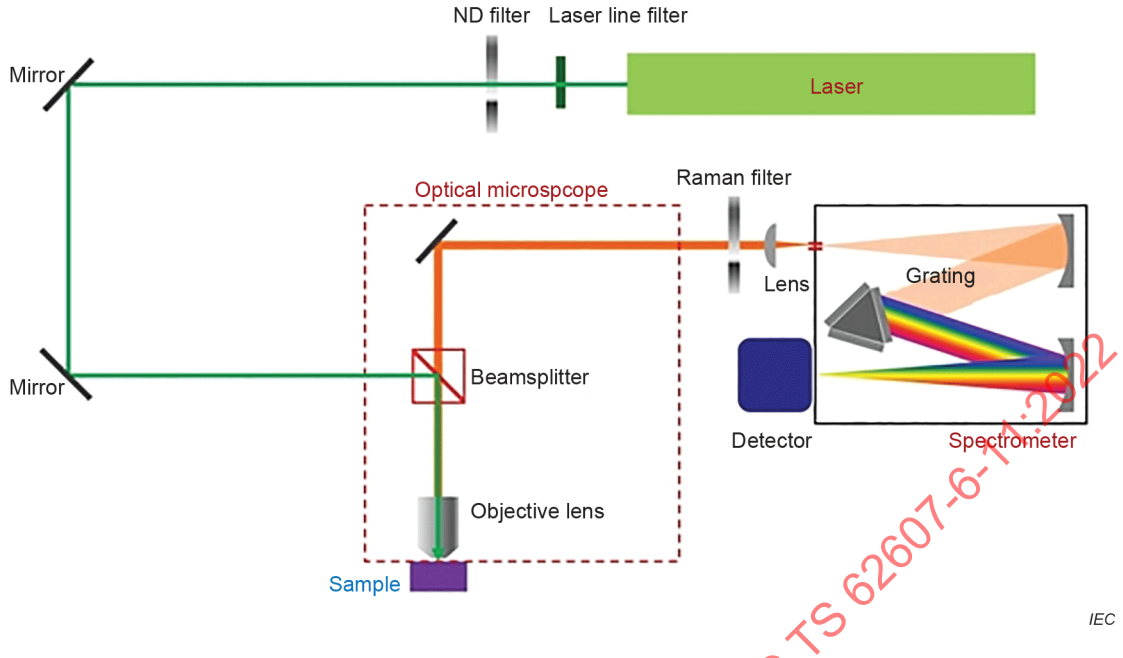


Figure 3 – Schematic of micro-Raman setup used for graphene characterization

4.4 Calibration standards, alignment and peak fitting

The calibration should be done in accordance with the manufacturer's requirements and needs to be performed at the beginning of a measurement. Typical ways to calibrate the spectrometer are either using calibration lamps with discrete emission lines, e.g. neon lamps, or a silicon substrate with a well-defined sharp peak at 520,7 cm^{-1}. Both the emission lines of the calibration lamps as well as the silicon sample have sharp well-defined peaks with well-known peak positions and are used as reference to calibrate the Raman shift, i.e. the x-axis of the spectrum.

The Raman D and G peaks should be fitted with single Lorentzian lines. To cross-check a good alignment the Gaussian component of the peak fit for the silicon peak should be checked regularly by using a Voigt function, i.e. a convolution of a Gaussian and a Lorentzian. The Gaussian component should be below 10 %.

4.5 Ambient conditions

The measurements can be performed under regular laboratory conditions without precise temperature and humidity control.

5 Measurement procedure

5.1 Description of the measurement procedure

Raman measurements are carried out under ambient conditions. The incident power is kept well below 1 mW corresponding to a laser power density of 1,27 $mW/\mu m^2$ to avoid heating effects. This is valid for any substrate, also for metals such as copper and nickel. To avoid an artificial background in the spectrum, the measurements are usually done in a dark room, i.e. a spectroscopy laboratory. This is highly recommended as good practice, but not strictly required by this document as long as stable measurement conditions without any change in the environment, such as change of the light intensity in the room, are ensured throughout the measurements.

Before starting the measurements, the system shall be calibrated in accordance with the manufacturer's requirements. After placing the sample on the microscope stage, the sample

shall be brought in focus. Focusing the sample can be done by optimizing the optical focus of the sample as viewed under the microscope. Fine-focusing can be done by adjusting the focus with the laser on the sample, i.e. minimizing the laser spot size on the camera image, as sometimes the optical focus is not exactly the same as the laser focus. Here it is essential that when using the laser spot to focus, the laser power is decreased to a few microwatts (μW). The same focusing procedure shall be done for each measurement when measuring different points on a sample. When spectra at different points are collected, it is important that all the spectra are collected under the same conditions with the same settings for laser power, spectral range, number of accumulations and acquisition time, to be able to compare the spectra with each other.

5.2 Measurement accuracy

To obtain analysable data, the acquisition time and number of accumulations (repetitions and averaging of spectra) need to be optimized such that a signal-to-noise ratio (S/N) of at least 10 for the D peak is obtained. The S/N is defined as follow:

$$\mathrm{S/N} = \frac{I_\mathrm{p} - I_\mathrm{bg}}{\sqrt{I_\mathrm{bg}}}$$

where I_p is the peak signal intensity and I_bg is the mean background signal intensity. For example, we find a maximum peak signal of 15 000 counts (or arbitrary units) for the 2D peak, while the mean background signal is around 2 500 counts. The signal-to-noise ratio would then be: S/N = (15 000 − 2 500)/√2 500 = 250.

6 Data analysis and interpretation of results

The data is processed by standard peak fitting techniques. In particular, the Raman peaks of graphene are fitted using a single Lorentzian lineshape. The following notation is used in this document: I for peak height, A for peak area, Pos for peak position, FWHM for the full-width at half-maximum. So, for example, I(G) is the height of the G peak, A(G) its area, FWHM(G) the full-width at half-maximum and Pos(G) its position.

The intensity, i.e. height, ratio as obtained from the single Lorentzian fits of the Raman D and G peaks, I(D)/I(G), can be used to estimate the defect density and inter-defect distance using Formula (1) and Formula (2). As the use of Formula (1) and Formula (2) requires that L_D ≥ 10 nm (stage 1 of the graphene lattice disorder classification), see black line in Figure 4, only values with L_D ≥ 10 nm are valid with respect to this document.

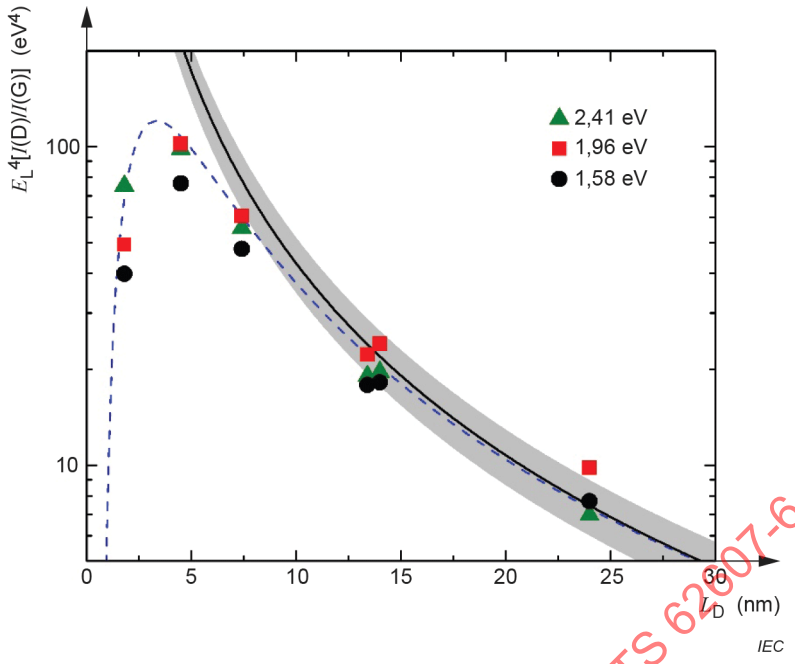


Figure 4 – E_L^4 [I(D)/I(G)] as a function of L_D

NOTE The black line in Figure 4 is obtained by using Formula (1), while the grey shaded region accounts for the upper and lower limits given by the ±30 % experimental error [3].

7 Sampling plan

To gather information about the distribution of the structural quality of the graphene layer over the whole wafer, one of the sampling plans in Annex B shall be used. Which one is chosen can depend on the maturity of the fabrication process, the cost related to testing or agreements between manufacturer and customer. Three Raman spectra should be measured at each location for statistical reasons.

8 Test report

8.1 General

The results of the measurement shall be documented in a measurement report, including the date and time of the measurement as well as the name and signature of the person responsible for the accuracy of the report. Guidelines are given in Annex A.

8.2 Sample identification

The report shall contain all information to identify the test sample and trace back the history of the sample:

– General procurement information, in accordance with IEC 62565-3-1.
– General material description in accordance with IEC 62565-3-1, including a technical drawing:
  - top view, indicating the inspected area and location of the measurement positions;
  - cross section, showing the layer structure.

8.3 Test conditions

The laboratory ambient conditions during the test:

– Atmosphere.
– Temperature range: 17 °C < T < 24 °C.
– Range of relative humidity: 40 % < RH < 60 %.
– Laser power density should be below 1,27 mW/μm^2.

8.4 Measurement specific information

– Calibration status of equipment.
– Spectral resolution of the spectrometer.
– Wavelength, spot size and power of the laser used.
– Signal-to-noise ratio for the Raman spectra based on the G peak.
– Typical measured Raman spectrum.
– Intensity ratio of D peak and G peak, I(D)/I(G). If the D peak is not visible, this shall be noted and the maximum value of the ratio shall be estimated based on the signal-to-noise ratio of the spectra.

8.5 Test results

– Coordinate system used in the measurement setup in absolute positions with a definition of the origin so that the measurement locations can be related to the technical drawing of the sample.
– Table of mean values and standard deviation of the KCC defect density and the corresponding inter-defect distance at the positions defined by the sampling plan.
– Colour maps for defect density and optionally inter-defect distance. The colour map shall be scaled in absolute positions in respect of the origin of the coordinate system. The colour code should be calibrated in absolute values of the measured KCC.

Annex A
(informative)

Format of the test report

The form of the report is oriented on the relevant material specification,[2] a related sectional blank detail specification or detail specification. Table A.1, Table A.2, Table A.3, Table A.4 and Table A.5 are guidelines to write the report and can be modified to fulfil the requirements of the involved parties.

Table A.1 – Product identification (in accordance with IEC 62565-3-1)

Item No	Item		Information
1.1	Supplier		
1.2	Trade name		
1.3	ID number		
1.4	Typical batch quantity	Number of wafers	
1.5	Traceability requirements	☐ Batch number ☐ Serial number ☐ Others, specify ……	
		Manufacturing date	
1.6	Specification	Number	
		Revision level	
		Date of issue	
1.7	Material Safety Data Sheet (MSDS) available	☐ No	
		☐ Yes Reference	

Table A.2 – General material description (in accordance with IEC 62565-3-1)

Item No	Item		Information
2.1	Material type		
2.2	Manufacturing method		
2.3	Substrate	Material	
		Technical drawing (top view)	
		Technical drawing (cross section)	
2.4	Shelf life		
2.5	Typical batch size		

2 A blank detail specification for graphene is under development (IEC 62565-3-1).

Table A.3 – Measurement related information

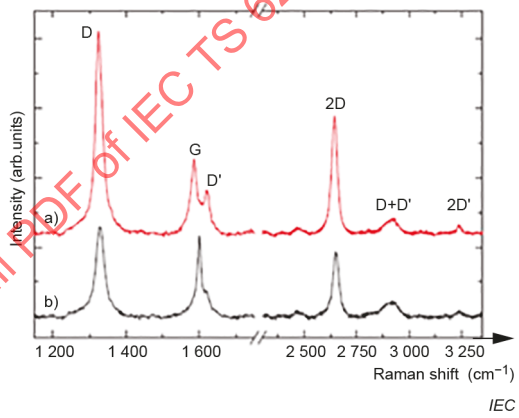
Item No	Item	Information
3.1	Sampling plan	☐ Circular, specify C-…. ☐ Square, specify S-….
		☐ drawing attached
3.2	Number of spectra per location	☐ 3 ☐ others, specify
3.3	Excitation wavelength	
3.4	Laser power on sample	
3.5	Laser spot size on sample	
3.6	I(D)/I(G)	☐ not visible
		I(D)/I(G) =
	corresponding defect density [cm^{-2}]	n_D =
3.7	Typical Raman spectrum	
3.8	Signal-to-noise ratio	
3.9	Raman data	☐ attached ☐ available on request
3.10	Environmental humidity (mean)	
3.11	Environmental temperature (mean)	

Table A.4 – Measurement results

Measurement point in accordance with sampling plan	Mean I(D)/I(G)	Mean defect density	Standard deviation	Mean inter-defect distance	Standard deviation
1					
2					
3					
4					
5					
6					
7					
8					
9					

Table A.5 – Colour map of KCC

Item No	Location of the mapped area on the sample	Map of the defect density
5.1		

Annex B
(normative)

Sampling plan

B.1 General

Annex B provides recommendations of a sampling plan for circular, rectangular and irregular substrate geometries to identify the exact location of the individual measurement locations. They are marked by red crosses. At each location several Raman spectra shall be measured and averaged to achieve a reasonable signal-to noise ratio (SNR). The achieved SNR shall be reported in the test report.

Which sampling plan is chosen will depend on the required information and measurement time which is directly related to cost. During the development of a process, it might be necessary to accept a high amount of effort to optimize the process. Nevertheless, over time with increasing knowledge regarding the process and increasing maturity of the fabrication this effort can be reduced. While at the beginning all points of a sampling plan need to be measured, over time the measurement effort might be reduced and a sampling plan with a reduced number of points might be chosen. Finally, it might be enough to perform measurements only on a fraction of wafers produced.

The strategy to reduce measurement effort should always be driven by the aim to reduce total quality cost. In this context quality cost includes the cost to perform the measurements but also the additional processing cost if a not identified defective product remains in the production line.

The sampling procedure shall be described in the test report.

B.2 Sampling plan depending on substrate geometry

B.2.1 Circular substrates

Dimensions in millimetres

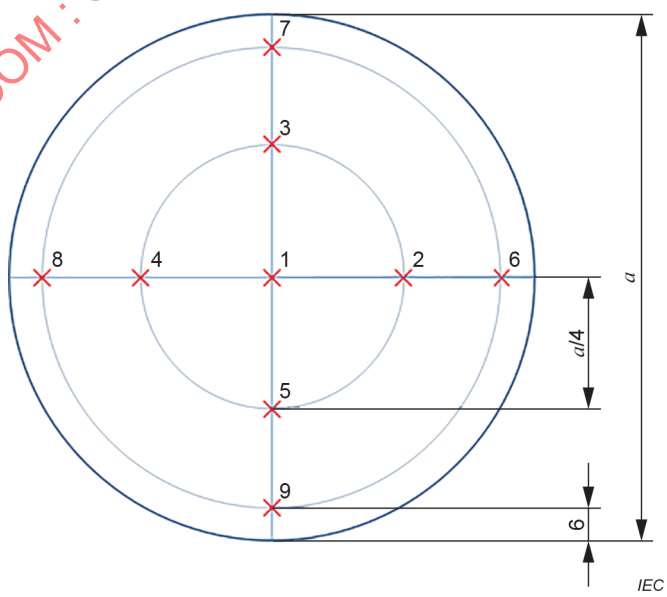


Figure B.1 – Schematic of sample plan for circular substrates

A schematic of a circular substrate with diameter a, such as a wafer, is shown in Figure B.1. Spectra need to be recorded in accordance with the sampling plan such that the whole area of the wafer is covered. Table B.1 summarizes the sampling plan for a circular substrate. As discussed above in the beginning all nine locations shall be covered for a full characterization of the wafer. Later on in the case of a mature technology that guarantees sample uniformity, the number of locations where spectra are measured can be reduced in accordance with the sampling plan in Table B.1.

Table B.1 – Sampling plan for circular substrates

Sampling plan	Locations								
	1	2	3	4	5	6	7	8	9
C-A	x	x	x	x	x	x	x	x	x
C-B	x	x	x	x	x				
C-C	x					x	x	x	x
C-D	x								

B.2.2 Square substrates

Similar to circular substrates, the whole area needs to be covered in a reasonable manner. Figure B.2 shows a schematic of the sampling plan used for square substrates with site length a. At each location three spectra should be recorded at slightly different positions around the same location. For small substrates that are around 1 cm × 1 cm in size, a 3 × 3 grid of locations is sufficient to characterize the uniformity of the sample.

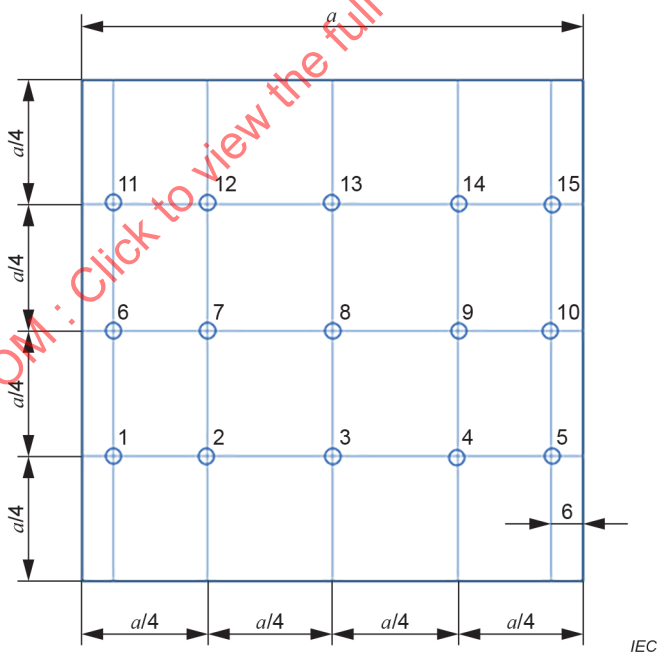


Figure B.2 – Schematic of sample plan for square substrates

The locations for taking Raman spectra on a square substrate are summarized in Table B.2. Similar to circular substrates, the number of locations can be reduced when the technology has matured to such a standard that produced samples show very uniform quality.

Table B.2 – Sampling plan for square sample

Sampling plan	Locations														
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
S-A	x	x	x	x	x	x	x	x	x	x	x	x	x	x	x
S-B	x	x	x	x	x										
S-C	x		x		x						x		x		x
S-D	x		x		x										
S-E			x												

For the special case of roll-to-roll production, one of the plans S-B, S-D or S-E shall be used with the information about the location on the roll.

B.2.3 Irregular shaped substrates

A sketch shall be provided showing the locations of the measurement points (See Figure B.3) and the positions shall be related to the absolute coordinate system.

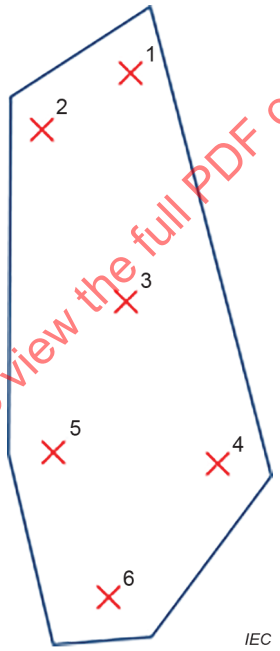
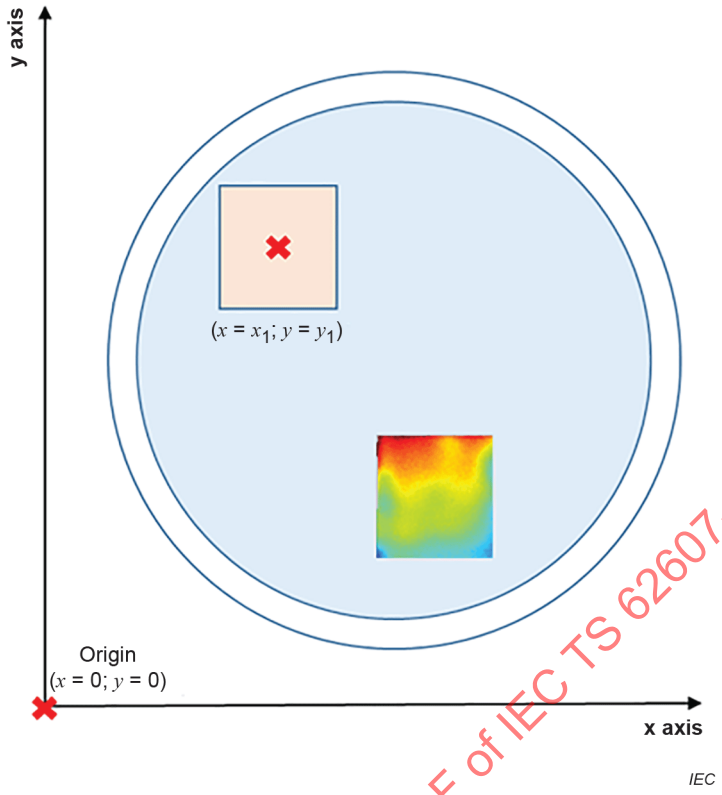


Figure B.3 – Example sampling plan for irregular sample

B.2.4 Coordinate system

For the purpose of measurement interlaboratory comparisons or the analysis of the film quality using different KCCs, a standardized Cartesian coordinate system as shown in Figure B.4 shall be defined. The drawing is generic and can be applied to all shapes of substrates.



Origin: The origin of the coordinate system shall be defined in such a way that the measurement positions can be reproduced reliably. The best way is to have a mark on the substrate.

Usable area: Due to the manufacturing process not the whole substrate surface might be usable. The supplier shall define the usable area of the substrate in which the specifications are guaranteed (blue coloured area in the figure).

Measurement locations: Measurement locations (Example: red cross located on the substrate) shall be provided in absolute coordinates $(x_1;y_1)$. If requested, the reported value of the KCC might be averaged over a defined area (red coloured area in the figure) around the measurement position, $[x_1 - \Delta x/2;x_1 + \Delta x/2];[y_1 - \Delta y/2;y_1 + \Delta y/2]$.

Positions of colour maps and pictures shall be given in absolute coordinates.

Figure B.4 – Coordinate system applied to the measurement results in the test report

Annex C
(informative)

Recommendations for wavelengths depending on substrate

The optimal wavelength of the excitation laser strongly depends on the type of substrate. Otherwise, Raman peaks originating from the substrate material itself or interferences caused by the layer structure of the sample, e.g. the thickness of the SiO_2-layer of Si/SiO_2 substrates, will affect the measurements. In particular, the described method can be well applied to the types of sample configurations given in Table C.1, among others which are not specified in Table C.1.

Table C.1 – Recommended laser wavelength depending on used substrate

Substrate type	Laser wavelength (nm)
SiO_2	514, 532
Hexagonal boron nitride	514, 532
Hafnium oxide	514, 532
Aluminium oxide	514, 532
Copper	457, 514, 532
SiO_2 on silicon	514, 532
Platinum	514,532