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TECHNICAL SPECIFICATION

colour inside

Nanomanufacturing – Key control characteristics – Part 6-17: Graphene-based material – Order parameter: X-ray diffraction and transmission electron microscopy

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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
113/700/DTS	113/746/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/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.

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- withdrawn,
- replaced by a revised edition, or
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INTRODUCTION

Graphite is composed of layers of carbon atoms just a single atom in thickness, known as graphene layers, to which it owes many of its remarkable properties. When the thickness of graphite flakes is reduced to just a few graphene layers, some of the material's technologically most important characteristics are greatly enhanced. In other words, graphene is more than just graphite. Although graphene has a vast number of potential applications, a survey of commercially available graphene samples reveals that research could be undermined by the poor quality of the available material [1][1]. Many highly priced graphene products from 60 producers consist mostly of graphite powder [2]. Therefore, a lack of classification standards is creating a situation that downstream users are afraid to use graphene because they do not know whether the graphene is fake.

Figure 1 shows the schematic packing configurations of graphene layers in graphite powder (left side of Figure 1) and graphene powder (right side of Figure 1) and their corresponding SEM images. It can be seen that graphite can be formed regularly in the z-axis, but graphene powder is assembled like house-of-card-type stacking, which is formed by graphene layers in a disorderly way in 3D space. For other carbon-related materials – for example, amorphous carbon, glassy carbon, expanded graphite – their packing configurations differ from those of graphite and graphene. An order parameter which indicates the order degree of a system can be employed to classify different carbon-related materials.

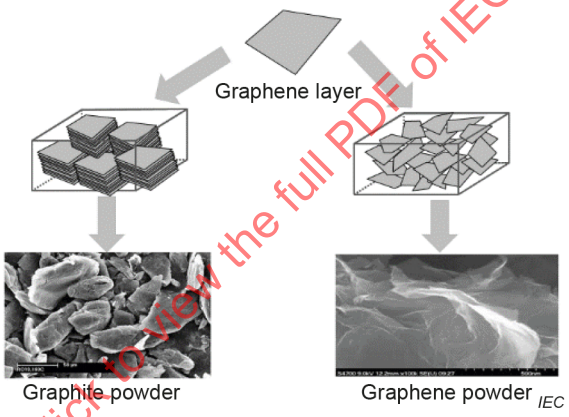

Figure 1 – Different packing configurations of graphene layers in graphite powder and graphene powder

This document establishes a method for determining the order parameter of graphene-based material and carbon material. The order parameter can be analysed from the z-axis and x-y-axis, respectively. The former can be derived from X-ray diffraction (XRD) spectra based on Bragg diffraction, and the latter can be derived from the diffraction patterns by selected area electron diffraction (SAED) technique, which is performed on a transmission electron microscope (TEM) with very high-resolution imaging. Since thermal temperature can lead to re-graphitization, the FWHM of peak (002) in the XRD spectrum indicates the quality of thermally reduced graphene powder [3]. Therefore, the order parameter can be an index of production uniformity of graphene-based materials, and also relates the materials' application with respect to heat dissipation.

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

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 6-17: Graphene-based material – Order parameter: X-ray diffraction and transmission electron microscopy

1 Scope

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

– order parameter

for graphene-based material and layered carbon material by

– X-ray diffraction (XRD) and transmission electron microscopy.

The order parameter is analysed from two perspectives: z-axis and x-y-axis. In the z-axis the order parameter is derived from the full width at half maximum (FWHM) of peak (002) in the XRD spectrum. In the x-y-axis, it is derived from the FWHM of peak (100) corresponding to diffraction patterns obtained by SAED (selected area electron diffraction) technique, which is routinely performed on most transmission electron microscopes in the world.

– The method is applicable for graphene-based material and layered carbon material including graphite, expanded graphite, amorphous carbon, vitreous carbon or glassy carbon, the structures of which are clarified by other characterization techniques.
– The method is applicable for differentiating few-layer graphene or reduced graphene oxide from layered carbon material.
– Typical application area is quality control in manufacturing to ensure batch-to-batch reproducibility.

NOTE Graphene oxide, one type of graphene-based material, is not within the scope of this document.

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
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]

3.1.2
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.

3.1.3
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]

3.1.4
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]

3.1.5
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]

3.1.6
highly oriented pyrolytic graphite
HOPG
highly pure and ordered form of synthetic graphite

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

3.1.7
amorphous carbon
carbon material without long-range crystalline order

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

Note 2 to entry: Short-range order exists, but with deviations of the interatomic distances or interbonding angles with respect to the graphite lattice as well as to the diamond.

3.1.8
expanded graphite
modified graphite that has a layered structure with interlayer spacing

3.1.9
vitreous carbon
form of carbon derived through solid-phase carbonization from a preform comprising an appropriate highly cross-linked polymer

Note 1 to entry: Vitreous carbon is characterized by a pseudo-amorphous, isotropic structure with low density, and non-permeability for gases.

[SOURCE: ISO 20507:2022, 3.2.79]

3.1.10
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.

3.2 Key control characteristics measured in accordance with this document

3.2.1
order parameter
normalized parameter that indicates the degree of order of a system

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

Note 2 to entry: An order parameter of 0 indicates disorder; the absolute value in the ordered state is 1.

Note 3 to entry: The order parameter includes z-axis order parameter and x-y-axis order parameter.

3.3 Terms related to the measurement method

3.3.1
X-ray diffraction
XRD
method to obtain crystallographic information about a sample by observing the diffraction pattern due to an X-ray beam hitting a sample

Note 1 to entry: The method can be used to estimate the size of coherent scattering regions.

[SOURCE: ISO 80004-6:2013, 5.2.1]

3.3.2
transmission electron microscope
TEM
instrument that produces magnified images or diffraction patterns of the sample by an electron beam which passes through the sample and interacts with it

[SOURCE: ISO 29301:2017, 3.34, modified – In the definition, "specimen" has been replaced by "sample".]

3.3.3
selected area electron diffraction
SAED
technique in which the crystalline structure of a sample area selected by an aperture is examined by the diffraction of transmitted electrons resulting in a diffraction pattern

Note 1 to entry: The electrons used typically have energies of 10 keV to 200 keV.

Note 2 to entry: The diffraction pattern represents an image of the reciprocal lattice and therefore contains information about crystal structure.

[SOURCE: ISO 80004-6:2021, 6.3.3]

3.3.4
full width at half maximum
FWHM
measure of the width of an X-ray peak in which the background is first removed to reveal the complete peak profile

Note 1 to entry: FWHM is determined by measuring the width at half the maximum height.

[SOURCE: ISO 22309:2011, 3.16]

3.3.5
lattice spacing
d-spacing
lattice plane spacing
distance between adjacent parallel crystallographic lattice planes

[SOURCE: ISO 21432:2019, 3.20]

3.3.6
Bragg diffraction
width between the wavelength of light and the width of crystal structure, or relationship between the reflecting surface and the angle formed by the ray

Note 1 to entry: The formula is $2d\sin\theta = n\lambda$

where

d is the width of periodic structure;

θ is the angle between the crystal plane and incident light;

λ is the wavelength of light;

n is the constant.

[SOURCE: ISO 22278:2020, 3.4]

3.3.7
2 theta
2θ
angle of the detected X-ray beam with respect to the incident X-ray beam direction

[SOURCE: ISO 22278:2020, 3.9]

3.3.8
diffraction pattern
distribution of light due to diffraction, which depends on the geometrical and optical properties of the object, the aberrations of the lens and the shape of its exit pupil, and the wavelength of the light

Note 1 to entry: Also misused as the angle between the direction of an incident optical radiation beam to a diffractive optical element and the direction of any resulting diffracted optical radiation beam.

[SOURCE: ISO 10934:2020, 3.1.41.1, modified – Note 1 to entry has been added.]

4 General

4.1 Measurement principle

XRD works by irradiating a material with incident X-rays and then measuring the intensities and scattering angles of the X-rays that leave the material. With the obtained spectrum, one can get information on the FWHM corresponding to peak (002) in the XRD spectrum. Using HOPG as a reference sample, the order parameter of tested sample in z-axis can be derived from the FWHM ratio of HOPG and tested sample.

When an energetic electron beam is incident upon a thin sample in a TEM, a diffraction pattern, (e.g. diffraction spot, diffraction ring) will be produced in the back focal plane of the objective lens. This pattern is magnified by the intermediate and projector lenses, then displayed on a viewing screen and recorded [4]. This pattern can also be displayed on a monitor if the TEM is equipped with a digital camera system. One can obtain information on corresponding interplanar spacing and diffraction intensity from the diffraction patterns. Also the FWHM corresponding to peak (100) is available. Using amorphous carbon as a reference sample, the order parameter of tested sample in x-y-axis can be derived from one minus the FWHM ratio of tested sample and amorphous carbon.

4.2 Sample preparation method

As for XRD measurement, take an appropriate amount of powder sample with a spoon and deposit it in the groove of a sample loader. Gently press the sample with a glass slide so that the sample surface is as flat as possible. The centre of the groove in the sample loader should be covered by powder sample.

As for TEM measurement, disperse an appropriate amount of tested sample in absolute ethanol, and shake the mixture for 15 minutes with an ultrasonic machine at a power of 120 W and frequency of 40 Hz. Then suck 2 µl dispersed mixture with a micropipette and drop it onto a support grid. Finally leave it to dry naturally for one hour prior to TEM measurement.

4.3 Description of measurement equipment and apparatus

4.3.1 XRD equipment

4.3.1.1 X-ray generator

Device which generates an X-ray beam of fixed intensity. Different target materials (i.e. Cu, Cr, Co, Fe, Mo) correspond to different wavelengths in Bragg diffraction.

4.3.1.2 Monochromator

When X-rays having diverse wavelengths are incident on a sample, a diffraction peak broadening occurs, disrupting interpretation of the diffraction peak. For this reason, a monochromator that takes only one ray from incident X-rays to make a single wavelength shall be used for accurate diffraction tests with high resolution.

4.3.1.3 Goniometer

Device designed for the sample to move in the x-axis, y-axis and z-axis. A mechanically well-aligned and state X-ray goniometer is required.

4.3.2 TEM equipment

4.3.2.1 Facilities for sample preparation

Ultrasonic machine can be used to completely disperse and mix the samples.

4.3.2.2 Acquisition of SAED patterns and images

The SAED patterns and images obtained on the TEM shall be recorded on the photographic films or imaging plates or an image sensor built in the digital camera.

4.4 Supporting materials

During the measurement, reagents and materials are needed. Unless otherwise stated, use only reagents of recognized analytical grade.

4.4.1 Appropriate solvent, suitable for dispersing powder samples or cleaning sample holders, e.g. absolute ethanol.

4.4.2 Spoon, suitable for taking powder samples from the containers.

4.4.3 Glass slide, suitable for pressing the sample to ensure the flatness of the surface.

4.4.4 Centrifugal tube, suitable for dispersing powder samples and being vibrated in an ultrasonic oscillator.

4.4.5 Micropipette, suitable for sucking dispersed mixtures.

4.5 Ambient conditions during measurement

All measurements shall be carried out at room temperature and relative humidity below 60 %. Specific temperature and relative humidity are not required.

5 Measurement procedure

5.1 Calibration of measurement equipment

Calibration on a regular basis shall be conducted in accordance with the procedures and methods in the manual provided by the TEM and XRD manufacturer.

5.2 Detailed protocol of the measurement procedure

For XRD measurement, firstly prepare the HOPG sample in accordance with the method described in 4.2, and place the sample loader on the flat sample stage. Set measurement parameters through the programme panel, including voltage, current, scan mode, range of scan angle, scan step and scan speed, etc. Afterwards, start the measurement programme until the automatic measurement ends. Then prepare the tested samples in accordance with the method described in 4.2, and follow the above steps without changing the operation conditions. Finally obtain the XRD spectrum of all samples for analysis.

For TEM measurement, firstly prepare the amorphous sample in accordance with the method described in 4.2, and place the sample loader into the chamber. Select an appropriate accelerating voltage so that the incident electron beam can penetrate through the sample. After setting the appropriate instrument parameters and attaining the optimal beam conditions for high resolution, it is important to start the sample investigation at low magnification, i.e. at large field-of-view. A quick survey of the sample can determine whether the quality of sample preparation is acceptable. Adjust the magnification until the sample can be observed clearly. A suitable magnification for SAED analysis is usually from several thousands to tens of thousands of times. Insert the selected area aperture available and switch the microscope from image mode to diffraction mode. Remove the objective aperture and obtain a diffraction pattern on the viewing screen. In one sample, three to five examination regions can be chosen during measurements. Then prepare the tested samples in accordance with the method described in 4.2, and follow the above steps without changing the operation conditions. Finally obtain the SAED images of all samples for analysis.

For an unknown sample, XRD measurement and z-axis order parameter analysis should be performed first. If the z-axis order parameter is larger than that of expanded graphite, the sample is graphite. Otherwise, TEM measurement and x-y-axis order parameter analysis should be performed to further determine the type of the sample. If the x-y-axis order parameter is smaller than that of glassy carbon, the sample is amorphous carbon. Otherwise, the sample is graphene-based material. A flow chart in Figure 2 illustrates when XRD measurement alone is sufficient to determine the sample type.

NOTE For users like manufacturers who have basic recognition of products, it is likely that costly TEM measurement can be avoided. XRD measurement plus other characterization techniques like Raman spectroscopy can possibly be sufficient.

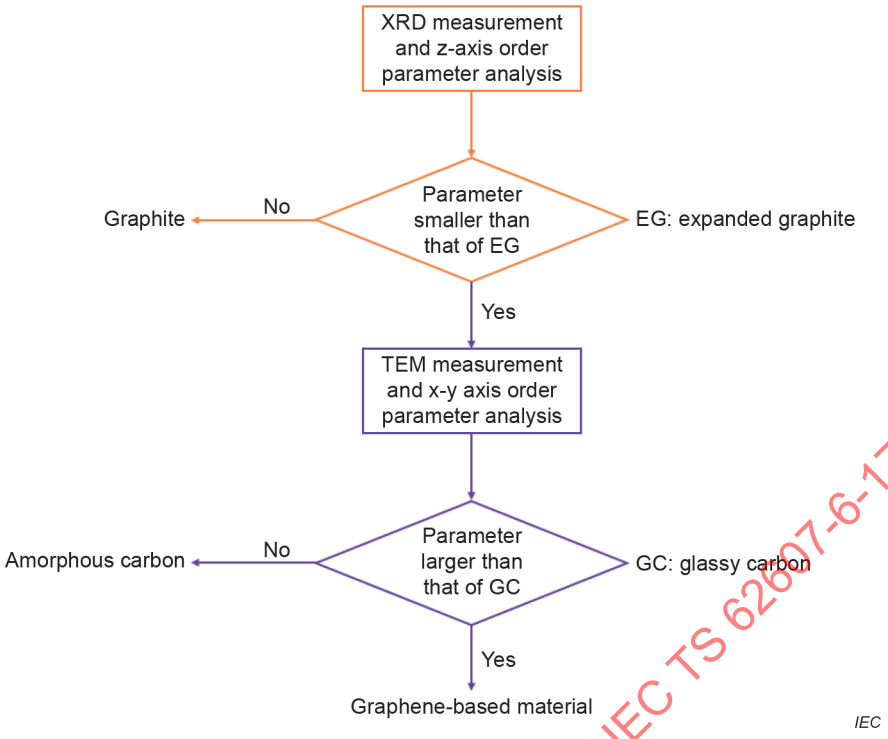

Figure 2 – A flow chart illustrating the cases when TEM measurement is needed

5.3 Measurement uncertainty source

The XRD measurement is affected by several factors, for instance, slit, scan step, and the flatness of the tested sample. The camera constant will change if the high voltage supply to the TEM fluctuates. Normally, stabilization ratios are 10^5 to 1 or better and thus, this effect is minimal. Meanwhile, the spherical aberration of the objective lens also influences the SAED acquisition.

6 Data analysis

For XRD measurements, firstly analyse the XRD spectrum of HOPG by commercially available software. Plot the curve of d-spacing versus intensity for analysis and fit the peak (002) by Gaussian algorithm. The horizontal axis is plotted as d-spacing for two reasons. One is that d-spacing is independent of the target (e.g. Cu, Co) used in XRD measurements. Another is that the d-spacing versus intensity can directly reflect the distribution of interplanar spacing. Measure the FWHM value of HOPG corresponding to peak (002) in XRD spectrum, denoting it as FWHM (002) of HOPG. Then analyse the XRD spectrum of other samples with the same procedure. The FWHM value of tested samples corresponding to peak (002) is denoted as FWHM (002) of tested sample. The order parameter in z-axis, a dimensionless value between 0 and 1, can be calculated by the ratio between FWHM (002) of HOPG and FWHM (002) of tested sample.

For TEM measurements, firstly analyse the diffraction pattern of amorphous carbon using commercially available software. Extract information on corresponding interplanar spacing and diffraction intensity. Plot the data in the same way as the XRD spectrum and fit the peak (100) by Gaussian algorithm. Measure the FWHM value of amorphous carbon corresponding to peak (100), denoting it as FWHM (100) of amorphous carbon. Then analyse the diffraction patterns of other samples with the same procedure. The FWHM value of tested samples corresponding to peak (100) is denoted as FWHM (100) of tested sample. The order parameter in x-y-axis, a dimensionless value between 0 and 1, can be calculated by subtracting the ratio between FWHM (100) of tested samples and FWHM (100) of amorphous carbon HOPG from 1.

With calculated order parameters in x-y-axis and z-axis, a spatial order system can be established. It helps to classify whether an unknown sample is graphene or other layered carbon material, like glassy carbon, amorphous carbon, graphite or expanded graphite.

For graphene powders consisting of single layer sheets, the sheets tend to randomly form a non face-to-face stacking with certain angles, which is described as "house-of-card stacking" in some work [5], [6]. Diffraction plane (002) can appear due to accidental repetition of the random stacking. In this case, the corresponding FWHM is close to infinite, leading to a z-axis order parameter of 0.

7 Results to be reported

7.1 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.

7.2 Product or 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 the relevant blank detail specification;
– general material description in accordance with the relevant blank detail specification.

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

7.3 Test conditions

The laboratory ambient conditions during the test:

– Temperature range: 20 °C < T < 30 °C.
– Range of relative humidity: 40 % < RH < 60 %.

7.4 Measurement specific information

7.4.1 Detailed explanation of the XRD measurement

– manufacturer's name and model number of XRD equipment
– X-ray source type;
– X-ray beam size and shape;
– incident full beam intensity and detector background;
– details of incident and diffracted beam conditioning (monochromator, slits, collimator and other optics);
– detector type;
– scan parameters: step size, scan speed, start/stop range;
– FWHM calculation method;

  The report should briefly specify the calculation approach used and what type of software was employed; if a commercially available or custom-made software was employed, it should be indicated in the text.

– order parameter in z-axis.

7.4.2 Detailed explanation of the TEM measurement

– manufacturer's name and model number of TEM equipment;

– operation parameters: accelerating voltage, magnification, exposure time;

– powder dispersion method;

– image processing method;

– FWHM calculation method;

The report should briefly specify the calculation approach used and what type of software was employed; if a commercially available or custom-made software was employed, it should be indicated in the text.

– order parameter in x-y-axis.

7.5 Test results

– Results of order parameter measured according to this document.

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 to Table A.5 are guidelines to write the report and can be customized to fulfil the requirements of the involved parties.

Table A.1 – Product identification (in accordance with the relevant blank detail specification)

Item No.	Item		Information
1.1	Supplier		
1.2	Trade name		
1.3	ID number		
1.4	Typical batch quantity	Mass [g]	
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 the relevant blank detail specification)

Item No.	Item	Information
2.1	Material type	
2.2	Manufacturing method	
2.3	Physical form	
2.5	Shelf life	
2.6	Typical batch size	

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

Table A.3 – Information related to XRD test

Item No.	Item		Information
3.1	Instruments	Name	
		Manufacturer	
		Model	
3.2	X-ray source type		
3.3	X-ray beam size and shape		
3.4	Incident full beam intensity and detector background		
3.5	Details of incident and diffracted beam conditioning	Monochromator	
		Slits	
		Collimator	
		Other optics	
3.6	Detector type		
3.7	Scan parameters	Step size	
		Scan speed	
		Start/stop range	
3.8	Environmental temperature		
3.9	Environmental relative humidity		
3.10	FWHW calculation method		
3.11	z-axis order parameter		

Table A.4 – Information related to TEM test

Item No.	Item		Information
3.1	Instruments	Name	
		Manufacturer	
		Model	
3.2	Operation parameters	Accelerating voltage	
		Magnification	
		Exposure time	
3.3	Powder dispersion method		
3.4	Image processing method		
3.5	Environmental temperature		
3.6	Environmental relative humidity		
3.7	FWHW calculation method		
3.8	x-y-axis order parameter		

Table A.5 – Measurement results

Item		sample 1	sample 2	sample 3	···
Order parameter	x-y-axis				
	z-axis				

Annex B
(informative)

Case study: Measurement and data analysis

B.1 Data analysis of XRD measurements

Firstly, analyse the XRD data of HOPG. The intensity versus d-spacing can directly reflect the distribution of different layer spacing, which is key for the order degree. Therefore, for the data analysis of XRD, convert the 2 theta versus intensity curve to d-spacing versus intensity by using the Bragg's equation. Then remove the baseline and noise, and fit the target range of the curve by Gaussian fitting. Here the target range represents the surrounding range of peak (002). Then obtain the FWHM (002) value of HOPG as 0,013 3, as shown in Figure B.1.

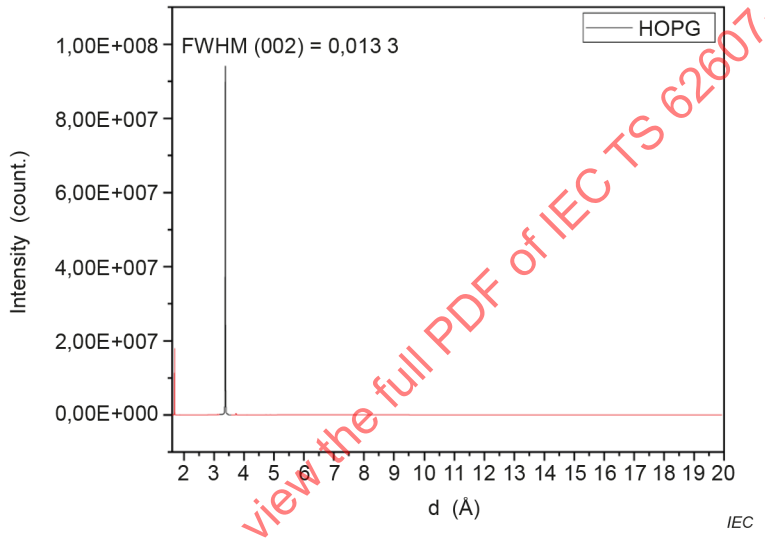

Figure B.1 – d-spacing versus intensity of HOPG and the fitting result of peak (002)

Then conduct data analysis for other samples, like glassy carbon, amorphous carbon, expanded graphite, graphene, etc. The corresponding results are presented in Figure B.2. With the FWHM (002) of different samples, one can derive the corresponding z-axis order parameter.