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

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IEC TS 62607-6-17 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
113/700/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.

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- withdrawn,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

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.

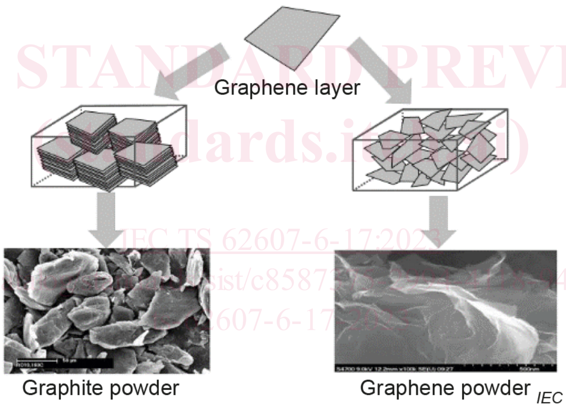

Figure 1 – Different packing configurations of graphene layers in graphite powder and graphene powder

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.