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Nanotechnologies — Classification framework for graphene-related 2D materials

*Nanotechnologies — Cadre de classification pour les matériaux
bidimensionnels similaires au graphène*

ISO/TC 229

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Foreword

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This document was prepared by Technical Committee ISO/TC 229 *Nanotechnologies*.

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Introduction

This document aims to provide commercial graphene producers and users with a standardized approach to classifying graphene-related 2D materials. It outlines a transparent methodology for categorizing these materials in any form, regardless of production methods or source materials. This approach facilitates accurate and straightforward comparisons between materials from different suppliers. Additionally, the document serves as a foundation for material specification data sheets, proposing a minimum set of relevant data that can be consistently used by producers, users, and regulators. Material characteristics deemed most critical for commercial applications—such as layer count, thickness, lateral flake size, disorder level, and specific surface area—can be identified using standard testing methods and documented in these data sheets.

The framework is primarily intended to support the creation and use of technical data sheets for specific forms of graphene-related 2D materials. It is not designed to prescribe production procedures or quality control testing processes, although some of the described test methods can serve as supporting techniques for those purposes.

Establishing a systematic classification framework for graphene-related 2D materials is essential for several key reasons. First, it ensures consistent and reliable regulation and registration processes, fostering transparency and trust in the market for both producers and users. Second, it enables a thorough understanding and quantification of material properties, forms, and contaminants. Finally, the adoption of universal standardized testing methods is critical for enabling easy, quantitative comparisons of data generated by different laboratories and users globally. This systematic approach will significantly advance the responsible development and use of graphene-related 2D materials.

It is also important to note that certain test methods are possibly not suitable for all forms of graphene-related 2D materials. When employing standardized testing methods, it is beneficial to provide multiple measurement techniques for evaluating different properties. However, these methods can yield varying results for the same sample due to differences in how each technique assesses specific aspects of the material.

NOTE This document is derived from Reference [25] use of which has been licensed to ISO by the Graphene Council (<https://www.thegraphenecouncil.org>).

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Nanotechnologies — Classification framework for graphene-related 2D materials

1 Scope

This document specifies the characteristics and their respective measurement methods of graphene-related 2D materials in sheet and particle forms for commercial applications with the aim of classification of the materials. The classification framework consists of the following elements:

- a) relevant material characteristics for commercial use;
- b) identification of applicable measurement methods;
- c) a range of the characteristic measured values when applicable;
- d) syntax to guide consistent naming and descriptions;
- e) an applicable technical data sheet template.

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 terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

3.1

graphene-related 2D material

GR2M

DEPRECATED: graphene-based material, graphene-material
carbon-based two-dimensional material consisting of one to 10 layers, including graphene, graphene oxide, reduced graphene oxide, and functionalized variations thereof

Note 1 to entry: This includes bilayer graphene, trilayer graphene and few-layer graphene.

Note 2 to entry: The terms graphene-based material and graphene-material are deprecated here. They have been used to describe materials other than graphene, such as graphene oxide.

Note 3 to entry: "Graphene-related 2D material" is defined in contrast with graphene-based and GR2M-based.

[SOURCE: ISO/TS 80004-13, 3.1.1.2]

3.2

sheet

2D material typically situated upon a substrate, with extended lateral dimensions at the micro to macroscale

[SOURCE: ISO/TS 80004-13, 3.1.1.4]

3.3

flake

distinct particle of planar morphology, consisting of 1 or more layers, with a nanoscale thickness that is significantly less than its lateral dimensions.

[SOURCE: ISO/TS 80004-13, 3.1.1.3]

3.4

particle

minute piece of matter with defined physical boundaries

Note 1 to entry: A physical boundary can also be described as an interface.

Note 2 to entry: This general particle definition applies to nano-objects.

[SOURCE: ISO 80004-1, 3.1.1]

3.5

coating

adherent surface *layer*

Note 1 to entry: A coating can consist of multiple layers.

Note 2 to entry: A coating is always attached to a substrate

[SOURCE: ISO 4618:2023, 2.244]

3.6

film

supported or unsupported thin material that is laterally continuously connected

Note 1 to entry: The attribute "thin" is used to emphasize that the thickness of the film is much smaller than the other two dimensions.

Note 2 to entry: A film can be freestanding.

Note 3 to entry: A film can be made of solids or liquids (e.g. liquid film).

Note 4 to entry: A film can be composed of a monomolecular layer (e.g. Langmuir-Blodgett film).

[SOURCE: ISO/TS 80004-1, 3.1.1]

3.7

functionalization

process that intentionally alters the surface chemical properties through a distinct chemical

Note 1 to entry: functionalized material should then be referred to as "functionalized X", where X refers to the material such as graphene, graphene nanoplatelet, etc.

[SOURCE: ISO/TS 80004-13, 3.1.1.17]

3.8

lateral size

<2D material> lateral dimensions of a 2D material flake

Note 1 to entry: If the flake is approximately circular, then this is typically measured using an equivalent circular diameter or if not via x, y measurements along and perpendicular to the longest side.

[SOURCE: ISO/TS 80004-13:2024, 3.4.1.15]

3.9 orientation

direction of crystal which is represented by crystal index

Note 1 to entry: During TEM imaging, it is often useful to have a crystalline specimen aligned such that a specific (low index) zone axis is parallel, or nearly parallel, to the beam direction optical axis.

[SOURCE: ISO 29301:2023, 3.5]

3.10 level of disorder

<GR2M, Raman spectroscopy> quantification of the disorder present in a GR2M given by the ratio of the Raman D peak and G peak intensities as measured by Raman spectroscopy, representing a combination of the amount, size and type of defects

Note 1 to entry: This can be method dependent where different instrument configurations can give different results.

Note 2 to entry: The peak intensities can be measured using area or height depending on which is most relevant.

[SOURCE: ISO 80004-13, 3.4.1.10]

4 Sheet-form graphene-related 2D materials

4.1 General

4.1.1 Production method

The GR2M producer should report the production method in general terms without disclosing confidential information. This is a "self-reported and labelled" item for which there is no independent testing. Examples include chemical vapor deposition (CVD), spin coating, spin on layer, etc.

4.1.2 Source material

The GR2M producer should report the source material used in production, without disclosing confidential information. Examples include, from methane, acetylene, etc. Therefore, the test method is "self-reported and labelled".

4.1.3 Material form and substrate

The form of GR2M should be assessed by visual observation. The form of material defined in this section is categorized as sheets. The producer may specify whether the material provided is on a substrate.

The transfer process used, if any, from the production substrate to the substrate delivered to the customer should be categorized. Examples include chemical etching, oxidative decoupling, electrochemical delamination, direct growth, etc.

This is a self-reported value from the producer.

This section should also describe the substrate material (copper, silicon, nickel, PET, etc.) Example: monolayer graphene film on a silicon wafer.

4.2 Characteristics and measurement methods

4.2.1 Sheet size

4.2.1.1 Definition

The dimensions of the sheet are measured from edge to edge along the x and y axes to determine the length and width. Materials are typically reported as a quadrangle but can be circular or irregular in shape. The applicable standard is ISO/TS 21356-2.^[10]

4.2.1.2 Measurement methods

Measurement methods include optical microscopy, scanning electron microscopy (SEM), atomic force microscopy (AFM) and Raman spectroscopy; however optical microscopy is the recommended method, when measuring GR2M.

4.2.1.3 Measurement results

The measurement results shall be expressed as the average or distribution of sheet sizes, as required. The units of the results shall be cm, mm, μm , or nm. If the flake is approximately circular then this is typically measured using an equivalent circular diameter or if not via x, y measurements along and perpendicular to the longest side. The oxide thickness is crucial for optical contrast.

4.2.2 Covered area

4.2.2.1 Definition

The substrate's coverage in lateral dimensions is expressed as the percent of the area covered by a single layer, the percent of the area covered by a multi-layer (including the number of layers), and the percent of the area comprised of defect.

4.2.2.2 Measurement methods

The determination of the sample containing graphene or graphitic material, that is bilayer graphene, FLG, GNPs or graphite, is done using Raman spectroscopy. Other measurement methods include optical microscopy, SEM, and AFM. For powder samples, disperse them in a solvent concentration of approximately 0,1 mg/ml. If the sample is already in dispersion, dilute it to approximately 0,1 mg/ml using the same solvent or mixture. To decide which methods to use, refer to the applicable standards ISO/TS 21356-1^[9] and ISO/TS 21356-2^[10].

4.2.2.3 Measurement results

The measurements results shall be expressed as the required average or distribution of sheet sizes, and the size of the sample used for testing must also be reported. The measurement results shall be expressed as a percentage. To confirm the presence of graphene or graphite, a sharp [$< 30 \text{ cm}^{-1}$ full width at half maximum (FWHM)] G-peak at $\sim 1580 \text{ cm}^{-1}$ and a 2D-peak at $\sim 2700 \text{ cm}^{-1}$ should be consistently observed in the Raman spectra.

NOTE Expected values for commercial-grade material are usually more than 95 % monolayer coverage (i.e., 5 % or less defective or multilayer material). Similar values are usually expected for materials intentionally produced as bilayer or trilayer films.

4.2.3 Surface residue

4.2.3.1 Definition

Surface residue is defined as any particles or substances that are observed on the surface of the graphene that are not intentionally placed or desired and are possibly the result of a transfer or other processing step.

It can also be considered as a contaminant. The amount of surface residue present on the material should be expressed as a percent of the total area that is covered by residue.

4.2.3.2 Measurement methods

Measurement methods are optical microscopy and Raman spectroscopy. For powder samples, disperse them in a solvent concentration of approximately 0,1 mg/ml. If the sample is already in dispersion, dilute it to approximately 0,1 mg/ml using the same solvent or mixture. A thin layer of powder is required for this rapid Raman spectroscopy analysis step. If a powder has been provided, this should be analyzed with a significant amount of the sample secured on adhesive tape such that only the flakes rather than the substrate are analyzed. The applicable standards are ISO/TS 21356-1^[9] and ISO/TS 21356-2^[10].

4.2.3.3 Measurement results

The values expected are <5 % of the area covered with residue. The percentage of the area covered by residue should be reported.

4.2.4 Flake grain size

4.2.4.1 Definition

The flake grain size refers to the size of domains with the same crystallographic orientation. This measurement is typically determined through microscopic analysis.

4.2.4.2 Measurement methods

Measurement methods are AFM, scanning tunnelling microscopy (STM), optical microscopy, SEM, and transmission electron microscopy (TEM). The applicable standard is ISO/TS 21356-1^[9].

4.2.4.3 Measurement results

Values should be reported with values at the nm or μm scale.

NOTE Expected values range widely (from 100 nm to 100 μm) and are highly dependent on the intended application. Values of >50 micron are expected.

4.2.5 A_D/A_G peak ratio

4.2.5.1 Definition

The ratio of the integrated area of the *D* peak to the integrated area of the *G* peak in Raman spectroscopy can be a metric to assess the degree of disorder or defects in carbon-based materials. A larger A_D/A_G peak ratio can indicate a higher degree of disorder. In comparison, a lower ratio suggests a more ordered structure.

4.2.5.2 Measurement methods

The measurement method is Raman spectroscopy.

4.2.5.3 Measurement results

The values of a Raman spectroscopy measurement of the material should be reported as the ratio of the integrated area of the D to G peaks. Substrates and laser conditions can affect these values and therefore should be listed.