SIST

SLOVENSKI STANDARD
SIST-TS CEN ISO/TS 21356-1:2022

01-maj-2022

Nanotehnologije - Strukturne značilnosti grafena - 1. del: Grafen iz prahu in disperzije (ISO/TS 21356-1:2021)

Nanotechnologies - Structural characterization of graphene - Part 1: Graphene from powders and dispersions (ISO/TS 21356-1:2021)

Nanotechnologien - Strukturelle Charakterisierung von Graphen - Teil 1: Graphen aus Pulvern und Dispersionen (ISO/TS 21356-1:2021)

Nanotechnologies - Caractérisation structurelle du graphène - Partie 1: Graphène issu de poudres et de dispersions (ISO/TS 21356-1:2021)

Ta slovenski standard je istoveten z: **CEN ISO/TS 21356-1:2022**

ICS:

07.120	Nanotehnologije	Nanotechnologies

SIST-TS CEN ISO/TS 21356-1:2022 **en,fr,de**

TECHNICAL SPECIFICATION

SPÉCIFICATION TECHNIQUE

TECHNISCHE SPEZIFIKATION

CEN ISO/TS 21356-1

March 2022

ICS 07.120

English Version

Nanotechnologies - Structural characterization of graphene - Part 1: Graphene from powders and dispersions (ISO/TS 21356-1:2021)

Nanotechnologies - Caractérisation structurelle du graphène - Partie 1: Graphène issu de poudres et de dispersions (ISO/TS 21356-1:2021)

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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Ref. No. CEN ISO/TS 21356-1:2022 E

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European foreword

The text of ISO/TS 21356-1:2021 has been prepared by Technical Committee ISO/TC 229 "Nanotechnologies" of the International Organization for Standardization (ISO) and has been taken over as CEN ISO/TS 21356-1:2022 by Technical Committee CEN/TC 352 "Nanotechnologies" the secretariat of which is held by AFNOR.

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Endorsement notice

The text of ISO/TS 21356-1:2021 has been approved by CEN as CEN ISO/TS 21356-1:2022 without any modification.

TECHNICAL SPECIFICATION

ISO/TS 21356-1

First edition
2021-03

Nanotechnologies — Structural characterization of graphene —

Part 1: Graphene from powders and dispersions

Nanotechnologies — Caractérisation structurelle du graphène —

Partie 1: Graphène issu de poudres et de dispersions

Reference number
ISO/TS 21356-1:2021(E)

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Foreword

ISO (the International Organization for Standardization) and IEC (the International Electrotechnical Commission) form the specialized system for worldwide standardization. National bodies that are members of ISO or IEC participate in the development of International Standards through technical committees established by the respective organization to deal with particular fields of technical activity. ISO and IEC technical committees collaborate in fields of mutual interest. Other international organizations, governmental and non-governmental, in liaison with ISO and IEC, also take part in the work.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives or www.iec.ch/members_experts/refdocs).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html. In the IEC, see www.iec.ch/understanding-standards.

This document was prepared jointly by Technical Committee ISO/TC 229, *Nanotechnologies*, and Technical Committee IEC/TC 113, *Nanotechnology for electrotechnical products and systems*.

A list of all parts in the ISO/IEC 21356 series can be found on the ISO and IEC websites.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html and www.iec.ch/national-committees.

Introduction

Due to the many superlative properties of graphene and related 2D materials, there are many application areas where these nanomaterials could be disruptive, areas such as flexible electronics, nanocomposites, sensing, filtration membranes and energy storage.

There are barriers to commercialisation that are impeding the progress of products containing graphene, which need to be overcome. One of these crucial barriers is answering the question “What is my material?”. End-users of the raw materials containing graphene should be able to rely on the advertised properties of the commercial graphene on the global market, instilling trust and allowing worldwide trade. Reliable and repeatable measurement protocols are required to address this challenge.

This document provides a set of flow-charts for analysts to follow in order to determine the structural properties of graphene from powders and liquid dispersions (suspensions). Initially, a quick check should be undertaken to determine if graphene and/or graphitic material is present. If it is, then further detailed analysis is required to determine if the samples contain a mixture of single-layer graphene, bilayer graphene, few-layer graphene, graphene nanoplatelets and graphite particles. Depending on the methods used, the samples are typically analysed after deposition on a substrate. The document describes how to assess what measurements are required depending on the type of sample and includes decision trees and flow diagrams to aid the user. This document describes a selected set of measurands that are needed, namely:

a) the number of layers/thickness of the flakes;

b) the lateral dimensions of flakes;

c) layer alignment;

d) the level of disorder;

e) the estimated number fraction of graphene or few-layer graphene;

f) the specific surface area of the powder containing graphene.

The above physical properties of the material can change during its processing and lifetime, for example, the samples can become more agglomerated, obtain different surface functionalities. The above measurand list for the initial material defines their inherent characteristics that, along with the chosen manufacturing processes, will determine the performance of real-world products. Generally, different material properties can be important in different application areas, depending on the functional role of the material.

The document provides methods for structural characterization of individual flakes of graphene, bilayer graphene, graphene nanoplatelets and graphite particles isolated from powders and/or liquid dispersions. It does not provide methods for determination of whether the powders and/or dispersions are composed solely of these materials. No recommendation is provided as to when or how often to measure samples, although it is not expected this would be for every batch of the same material. It is up to the user to determine when, how often and which characterization routes described in this document to take. As with all microscopical investigations, care is needed in drawing statistical conclusions dependant on representative sampling.

A set of annexes provide example protocols on how to prepare and analyse the samples, sources of uncertainty and how to analyse the data. The methods used are Raman spectroscopy, scanning electron microscopy (SEM), atomic force microscopy (AFM), transmission electron microscopy (TEM) and the BET (Brunauer–Emmett–Teller) method.

Nanotechnologies — Structural characterization of graphene —

Part 1: Graphene from powders and dispersions

1 Scope

This document specifies the sequence of methods for characterizing the structural properties of graphene, bilayer graphene and graphene nanoplatelets from powders and liquid dispersions using a range of measurement techniques typically after the isolation of individual flakes on a substrate. The properties covered are the number of layers/thickness, the lateral flake size, the level of disorder, layer alignment and the specific surface area. Suggested measurement protocols, sample preparation routines and data analysis for the characterization of graphene from powders and dispersions are given.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO/TS 80004-1:2015, *Nanotechnologies — Vocabulary — Part 1: Core terms*

ISO/TS 80004-2:2015, *Nanotechnologies — Vocabulary — Part 2: Nano-objects*

ISO/TS 80004-6:2021, *Nanotechnologies — Vocabulary — Part 6: Nano-object characterization*

ISO/TS 80004-13:2017, *Nanotechnologies — Vocabulary — Part 13: Graphene and related two-dimensional (2D) materials*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO/TS 80004-1:2015, ISO/TS 80004-2:2015, ISO/TS 80004-6:2021, ISO/TS 80004-13:2017 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
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) (3.3) and *few-layer graphene* (FLG) (3.4).

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]

3.2
graphite
allotropic form of the element carbon, consisting of *graphene layers* (3.1) 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]

3.3
bilayer graphene
2LG
two-dimensional material consisting of two well-defined stacked *graphene layers* (3.1)

Note 1 to entry: If the stacking registry is known, it can be specified separately, for example, as "Bernal stacked bilayer graphene".

[SOURCE: ISO/TS 80004-13:2017, 3.1.2.6]

3.4
few-layer graphene
FLG
two-dimensional material consisting of three to ten well-defined stacked *graphene layers* (3.1)

[SOURCE: ISO/TS 80004-13:2017, 3.1.2.10]

3.5
graphene nanoplate
graphene nanoplatelet
GNP
nanoplate consisting of *graphene layers* (3.1)

Note 1 to entry: GNPs typically have thickness of between 1 nm to 3 nm and lateral dimensions ranging from approximately 100 nm to 100 μm.

[SOURCE: ISO/TS 80004-13:2017, 3.1.2.11]

3.6
lateral size
flake 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 80004-13:2017, 3.4.1.15]

3.7
graphene oxide
GO
chemically modified *graphene* (3.1) prepared by oxidation and exfoliation of *graphite* (3.2), causing extensive oxidative modification of the basal plane

Note 1 to entry: Graphene oxide is a single-layer material with a high oxygen content, typically characterized by C/O atomic ratios of approximately 2,0 depending on the method of synthesis.

[SOURCE: ISO/TS 80004-13:2017, 3.1.2.13]

3.8
reduced graphene oxide
rGO
reduced oxygen content form of *graphene oxide* (3.7)

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* (3.1) 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]

4 Abbreviated terms

For the purposes of this document, the following abbreviated terms apply.

NOTE The final “M”, given as “microscopy”, can be taken equally as “microscope” depending on the context.

1LG	single-layer graphene
2D	two dimensional
2LG	bilayer graphene
AFM	atomic force microscopy
BET method	Brunauer–Emmett–Teller method
CVD	chemical vapour deposition
FLG	few-layer graphene
FWHM	full width at half maximum
GNP	graphene nanoplate or graphene nanoplatelet
GO	graphene oxide
NMP	1-methyl-2-pyrrolidinone also known as N-methylpyrrolidone
rGO	reduced graphene oxide
SAED	selected area electron diffraction
SEM	scanning electron microscopy
TEM	transmission electron microscopy

5 Sequence of measurement methods

This clause presents the sequence of measurement methods necessary to most efficiently characterize graphene, bilayer graphene, few-layer graphene and graphene nanoplatelets from powders and liquid dispersions (suspensions). In this document, graphene, bilayer graphene, few-layer graphene and

