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Nanotechnologies — Matrix of properties and measurement techniques for graphene and related two-dimensional (2D) materials

Nanotechnologies — Matrice des propriétés et des techniques de mesure pour le graphène et autres matériaux bidimensionnels (2D)

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Contents

Page

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ISO/TR 19733:2019
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Foreword

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This document was prepared jointly by Technical Committee ISO/TC 229, *Nanotechnologies* and Technical Committee IEC/TC 113, *Nanotechnology for electrotechnical products and systems*. The draft was circulated for voting to the national bodies of both ISO and IEC.

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Introduction

Graphene is a single layer of carbon atoms with each atom bound to three neighbours in a honeycomb structure[1]. Since its discovery in 2004[2], graphene has become one of the most attractive materials in application research and device industry due to its supreme material properties such as mechanical strength, stiffness and elasticity, high electrical and thermal conductivity, optical transparency, etc. It is expected that applications of graphene could replace many of current device development technology in flexible touch panel, organic light emitting diode (OLED), solar cell, supercapacitor, and electromagnetic shielding. To gain deeper understanding of the material properties and to find the ways of mass producing with fine quality, much research on graphene, and similarly on related two-dimensional (2D) materials is being done in universities, research institutes, and laboratories around the globe. However, to lead these revolutionary materials to full commercialization, it is essentially demanded that characterization and measurement techniques for important material properties need to be standardized and globally recognized. In this document, characterization and measurement techniques for particular properties of graphene and related 2D materials which need to be standardized are organized in a form of a matrix. The matrix could serve as an initial guide for developing the necessary international standards in characterization and measurements of graphene and related 2D materials.

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Nanotechnologies — Matrix of properties and measurement techniques for graphene and related two-dimensional (2D) materials

1 Scope

This document provides a matrix which links key properties of graphene and related two-dimensional (2D) materials to commercially available measurement techniques. The matrix includes measurement techniques to characterize chemical, physical, electrical, optical, thermal and mechanical properties of graphene and related 2D materials.

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

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3 Terms and definitions, symbols and abbreviated terms

3.1 Terms and definitions

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For the purposes of this document, the terms and definitions given in ISO/TS 80004-13 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/

3.1.1
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-layered 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]

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3.1.2
two-dimensional material
2D material
material, consisting of one or several layers with the atoms in each layer strongly bonded to neighbouring atoms in the same layer, which has one dimension, its thickness, in the nanoscale or smaller, and the other two dimensions generally at larger scales

Note 1 to entry: The number of layers when a two-dimensional material becomes a bulk material varies depending on both the material being measured and its properties. In the case of graphene layers, it is a two dimensional material up to 10 layers thick for electrical measurements[3][4], beyond which the electrical properties of the material are not distinct from those for the bulk (also known as graphite).

Note 2 to entry: Interlayer bonding is distinct from and weaker than intralayer bonding.

Note 3 to entry: Each layer may contain more than one element.

Note 4 to entry: A two-dimensional material can be a nanoplate.

[SOURCE: ISO/TS 80004-13:2017, 3.1.1.1]

Note 5 to entry: The related 2D materials in this document refer to the graphene –derived materials such as graphene oxide and reduced graphene oxide and other 2D materials with a structure similar to that of graphene showing promising properties including but not limited to monolayer and few-layer versions of hexagonal boron nitride (hBN), molybdenum disulphide (MoS_2), tungsten diselenide (WSe_2), silicene and germanene and layered assemblies of mixtures of these materials.

3.1.3
graphene oxide
GO
chemically modified graphene prepared by oxidation and exfoliation of graphite, causing extensive oxidative modification of the basal plane

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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]

3.1.4
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]

3.2 Symbols and abbreviated terms

AFM	atomic force microscopy
BET	Brunauer, Emmet and Teller method
EDS	energy-dispersive spectroscopy

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EPMA	electron probe X-ray microanalysis
ESR	electron spin resonance
FT-IR	fourier transform infrared spectroscopy
ICP-MS	inductively coupled plasma - mass spectrometry
KPFM	kelvin probe force microscopy
LEEM	low energy electron microscopy
SEM	scanning electron microscopy
SIMS	secondary-ion mass spectrometry
SKPM	scanning kelvin probe microscopy
STM	scanning tunnelling microscopy
TEM	transmission electron microscopy
TGA	thermogravimetric analysis
UPS	ultraviolet photoelectron microscopy
UV-VIS-NIR SPECTROSCOPY	ultraviolet, visible, near infrared spectroscopy
WDS	wavelength-dispersive spectroscopy
XRD	X-ray diffraction
XPS	X-ray photoelectron spectroscopy

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4 Matrix of properties and measurement techniques for graphene and related 2D materials

Table 1 is a matrix that links the key properties of graphene and related two-dimensional (2D) materials to commercially available measurement techniques. The matrix includes measurement techniques to characterize chemical, physical, electrical, optical, thermal and mechanical properties of graphene and related 2D materials. There are many other techniques that are being used to study graphene and related 2D materials but here we include only those that are widely used and widely commercially available.

Some of techniques in the matrix may not be suitable to all forms of graphene and related 2D materials but can be applied only to a certain form, such as in sheets, powder, or dispersion. It is also possible to produce different measurement results using these techniques depending on the synthesizing methods of graphene and related 2D materials to be characterized, such as chemical vapour deposition (CVD), mechanical exfoliation, or others. The appropriate forms, synthesizing method and sample preparation of graphene or related 2D materials that each technique is applicable to will be specified in individual standards to be developed in future in accordance with this document.

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Table 1 — Matrix of properties and measurement techniques for graphene and related 2D materials

Properties		Techniques																						
		AFM	KPFM	BET	EPMA	ESR (EPR)	FT-IR	ICP-MS	LEEM	Optical Microscopy	Raman	UV-VIS-NIR Spectroscopy	SEM	SIMS	STM	TEM	UPS	XRD	XPS	TGA	Combustion	Titration	4-point Probe	Hall Bar
Structural	Crystal Defect	○				○					○		○		○	○		○						
	Domain (grain) Size	○								○	○	○			○	○								
	Flake Size	○								○	○		○			○								
	Number of Layers	○							○	○	○	○				○								
	Stacking Angle										○				○	○								
	Surface Area			○																				
	Thickness	○							○							○								
Chemical	Metal Contents				○			○						○					○					
	Non-Graphene Contents and Residue						○				○			○					○	○	○			
	Oxygen Contents				○									○					○	○		○		
Mechanical	Elastic Modulus	○									○													
Thermal	Thermal Conductivity										○													
Optical	Transmittance									○		○												
Electrical/ Electronic	Charge Carrier Concentration		○								○													○
	Mobility																							○
	Sheet Resistance																						○	○
	Work Function		○														○							

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The properties and measurands are described in more detail in Clause 5. In Clause 6, the measurement techniques are described. The texts for these descriptions are mostly taken from ISO definitions of the techniques. Advantages and limitations of each method as applied to graphene and related 2D materials characterization are also briefly listed.

5 Properties and measurands

5.1 Structural properties

5.1.1 Crystal defect

The crystal defect is a local deviation from regularity in the crystal lattice of graphene or related 2D materials.

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[SOURCE: ISO/TS 80004-13:2017, 3.4.1.1]

Possible defects are point defects, line defects, or planar defects. Some examples of crystal defects are illustrated in Figure 1.

5.1.1.1 Point defect

The point defect is a defect that occurs only at or around a single lattice point of a 2D material.

NOTE 1 Point defects generally involve at most a few missing, dislocated or different atoms creating a vacancies, extra atoms (interstitial defects) or replaced atoms.

5.1.1.2 Line defect

The line defect is a defect that occurs along an atomic line causing a dislocation of a row in a 2D material.

5.1.1.3 Planar defect

The planar defect is a defect occurring in the stacking sequence of the layers of a 2D material.

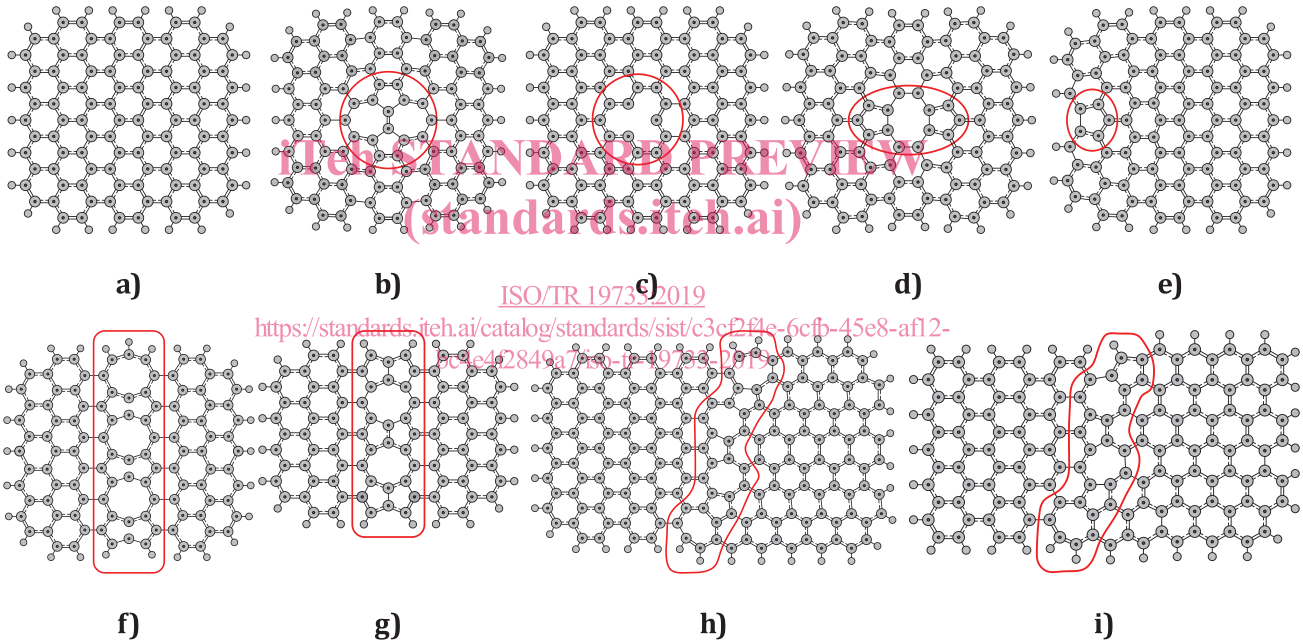

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Figure 1 — Examples of various point defects, a) to e) and line defects, f) to i)[5]

5.1.2 Domain (grain) size

Domain size is lateral dimensions of a single coherent crystalline region within a layer of a 2D material.

NOTE 1 The terms grain size and crystallite size are synonymous with the term domain size.

NOTE 2 If the domain 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.

NOTE 3 If an equivalent circular diameter is used then the term is similar to the crystallite diameter (L_a) which describes the lateral size of a crystal or crystallite region for example as measured by X-ray diffraction or Raman spectroscopy.

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