TECHNICAL REPORT

ISO/TR 19733

First edition
2019-03

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)

Reference number
ISO/TR 19733:2019(E)

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Published in Switzerland

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Foreword

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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 ISO documents 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).

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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.

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.

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.

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*

3 Terms and definitions, symbols and abbreviated terms

3.1 Terms and definitions

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]

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

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

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

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.

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	O				O					O		O		O	O		O						
	Domain (grain) Size	O								O	O	O			O	O								
	Flake Size	O								O	O		O			O								
	Number of Layers	O							O	O	O	O				O								
	Stacking Angle										O				O	O								
	Surface Area			O																				
	Thickness	O							O							O								
Chemical	Metal Contents				O			O						O					O					
	Non-Graphene Contents and Residue						O				O			O					O	O	O			
	Oxygen Contents				O									O					O	O		O		
Mechanical	Elastic Modulus	O									O													
Thermal	Thermal Conductivity										O													
Optical	Transmittance									O		O												
Electrical/ Electronic	Charge Carrier Concentration		O								O													O
	Mobility																							O
	Sheet Resistance																						O	O
	Work Function		O														O							

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.

[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.

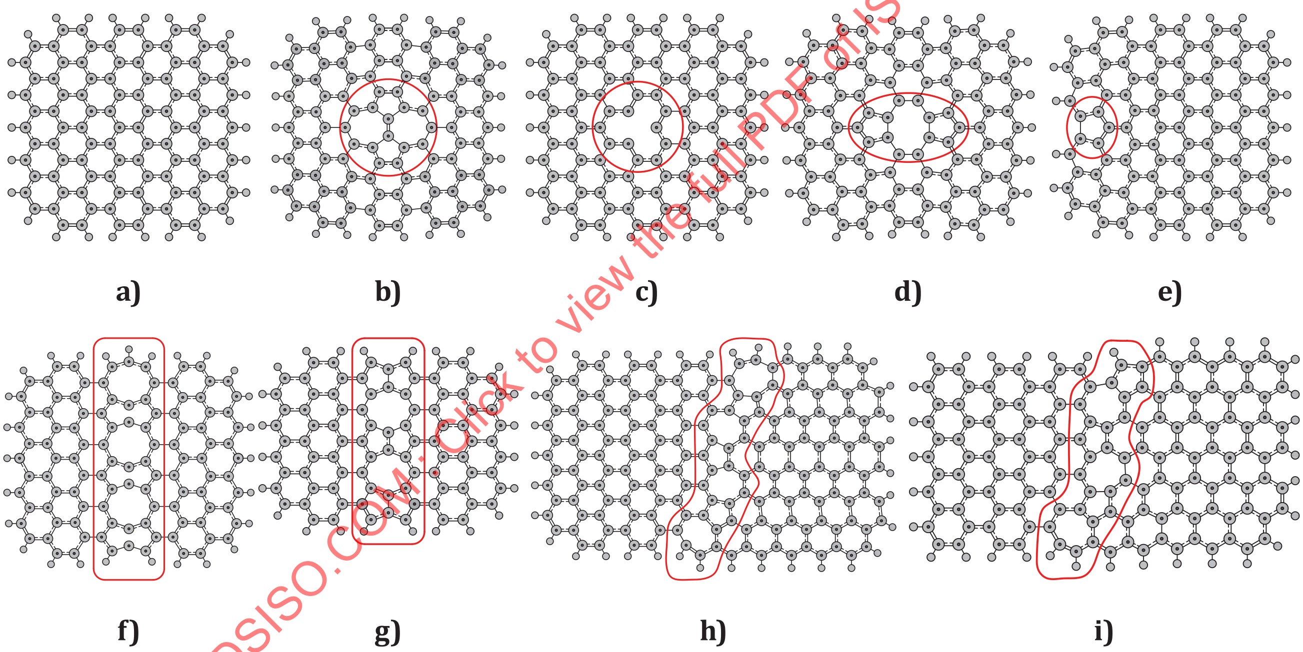

Figure 1 — Examples of various point defects, a) to e) and line defects, f) to i)[5]

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.

[SOURCE: ISO/TS 80004-13:2017, 6.1.14]

NOTE 4 Typical domain size of graphene ranges from tens of µm to hundreds of µm, and even up to mm recently[6].

5.1.3 Flake size

Flake size is lateral dimensions of a 2D material flake.

NOTE 1 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, 6.1.15]

NOTE 2 Flakes in powder or solution are the most common commercially available form of graphene when purchased from graphene manufacturers.

5.1.4 Number of layers

The number of layers is a count of the stacking of each two-dimensional monolayer on the top of the previous one in few layer 2D materials. As the number of layers increases, the distinct characteristics of graphene diminishes and many layer graphenes eventually become graphite when they lose all the features unique to graphene. For this reason, staking of graphene sheets should be limited to few layers, less than 10, to be used in graphene based applications.

NOTE 1 Number of layers could be used as a reasonable estimate of thickness of multi-layer graphene sheets provided the value of 0,335 nm for an interplanar spacing of graphite[7].

5.1.5 Stacking angle

Stacking angle is the angle measured in the horizontal plane between the orientations of two layers of 2D material that are stacked vertically on top of one another. When certain stacking angles are repeated in sequence in multi-layered 2D materials, the term, stacking order, is often used interchangeably.

[SOURCE: ISO/TS 80004-13:2017, 6.1.12]

5.1.6 Surface area

The surface area in 2D material refers to the specific surface area in m^2/kg, or more practically in m^2/g. The theoretical surface area of graphene was reported as 2 630 m^2/g[8], but the measured value can be significantly less than this hypothetical value for a monolayer due to overlapping of sheets and the various surroundings[9].

5.1.7 Thickness

The thickness is the vertical dimension of 2D material.

NOTE 1 The interplanar spacing of graphite is often used to estimate the thickness of few-layer graphene provided the measured value of number of layers.

5.2 Chemical properties

5.2.1 Metal contents

Metal contents are the amount of metals existing on or within graphene and GO. The metals can be introduced as a residue from catalytic process or by intentional impurity doping for better functionality. The typical metals found in graphene are gold (Au), silver (Ag), platinum (Pt), titanium (Ti), chromium (Cr), copper (Cu), Iron (Fe), etc.[10] Metal contents are typically measured in mass fraction as in [mg/kg or µg/g].

5.2.2 Non-graphene contents and residue

Non-graphene contents and residues are substances other than graphene including carbon debris in different crystal structure such as carbon soots, carbon nanotubes (CNT) and fullerene, non-carbon ligands and substituents, oxide derivatives, polymeric ligands and substituents, and other debris left over from processing in synthesis, purification, dispersion and separation of graphene. Metal contents are commonly excluded from this category because different methods are adopted in contents evaluation.

5.2.3 Oxygen content

Oxygen content is the amount of total oxygen in 2D materials. It represents the degree of oxidation in graphene oxide (GO) and reduced graphene oxide (rGO), which are common forms of 2D materials in practical use. The oxygen content of GO or rGO is commonly expressed as the carbon to oxygen ratio (C/O). The ratio increases in rGO through the reduction process (deoxygenation) of GO.

5.3 Mechanical properties, elastic modulus

Elastic modulus is a measure of resistance of graphene or related 2D materials to being deformed elastically under the influence of mechanical force, and defined as the ratio of stress to strain. The unit of modulus is Pa [N/m^2]. The elastic in-plane modulus of graphene monolayer was reported as high as 1,02 TPa[11].

NOTE 1 Young's modulus for tension or compression, shear modulus for shearing, bulk modulus for hydraulic compression.

5.4 Thermal properties, thermal conductivity

The thermal conductivity is areic heat flow rate divided by temperature gradient.

[SOURCE: ISO 80000-5:2007, 5-9]

NOTE 1 Thermal conductivity of graphene is typically measured in [W/m·K] and ranges around 3 000 W/m·K to 5 000 W/m·K[12].

5.5 Optical properties, optical transmittance

Optical transmittance is the ratio of transmitted light to incoming light through a single layer or multiple layers of 2D materials. Despite of having only a single-atomic thickness, a single layer of graphene shows unusually high absorption causing a transmittance drop of greater than 2 %. It was reported that each layer of graphene in few-layer graphene decreased the light transmittance by 2,3 % in red light[13] and 2,6 % in green light[14].

5.6 Electrical and electronic properties

5.6.1 Charge carrier concentration (density)

Charge carriers in graphene or related 2D materials are electrons (negative charge) and holes (positive charge). Charge carrier concentration is the number density of charge carriers per area for graphene and related 2D materials. The unit [m^{-2}] or more commonly [cm^{-2}] is used. Sometimes, the term 'carrier density' is alternately used.

NOTE 1 Charge carrier concentration in semiconductor is the number density per volume [cm^{-3}].

5.6.2 Charge carrier mobility

Charge carrier mobility in graphene or related 2D materials quantifies how fast charge carriers, such as electrons or holes, can move under the influence of external electric field. It is related to electrical conductivity (σ) as follows.

$$\sigma = e\left(n\mu_e + p\mu_h\right)$$

where

n and p	are electron and hole concentrations in unit area for graphene and related 2D materials;
μ_e and μ_h	are electron and hole mobility, respectively.

The electron mobility of suspended graphene was found to be as high as 200,000 $cm^2V^{-1}s^{-1}$[15], which is 10 times greater than copper. The electron mobility of graphene and related 2D materials is highly dependent on charge concentration and temperature. The mobility increases usually with temperature up to 2 540 K, but the dependency on temperature varies widely with different functional groups[16].

5.6.3 Sheet resistance

Sheet resistance [Ω], which is the in-plane resistivity per thickness, is often measured for thin films such as graphene. In three dimensional bulk materials, electrical conductivity [S/m] is an intrinsic property and a measure of a material's ability to conduct an electrical current and a reciprocal of electrical resistivity, which has unit of [Ω·m]. However, it is common to use a sheet resistance in graphene rather than bulk resistivity. The conductivity in 5.6.2 takes the unit of [S] for graphene and related 2D materials.

NOTE Common notation of sheet resistance is conveniently taken in [Ω/sq or Ω/☐], not to be confused with a bulk resistance. In such case, the unit is dimensionally equivalent to ohm[Ω] and refers to resistance of a square sheet regardless of the size.

5.6.4 Work function

Work Function is potential difference for electrons between the Fermi level and the maximum potential just outside a specified surface.

NOTE 1 The work functions of the different crystal facets of a single crystal will, in general, differ from one another. These work functions will also change with the state of cleanness of the crystal surfaces.

NOTE 2 A polycrystalline surface will exhibit an average work function which will depend on the types of exposed constituent single-crystal facets and their areas.

[SOURCE: ISO 18115-1:2013, 4.487]

NOTE 3 The work function is typically measured in [eV].

6 Measurement techniques

6.1 Atomic force microscopy (AFM)

AFM is a method for imaging surfaces by mechanically scanning their surface contours, in which the deflection of a sharp tip sensing the surface forces, mounted on a compliant cantilever is monitored.

NOTE 1 AFM can provide a quantitative height image of both insulating and conducting surfaces.

NOTE 2 Some AFM instruments move the sample in the x-, y- and z-directions while keeping the tip position constant and others move the tip while keeping the sample position constant.

NOTE 3 AFM can be conducted in vacuum, a liquid, a controlled atmosphere, or air. Atomic resolution may be attainable with suitable samples, with sharp tips, and by using an appropriate imaging mode.

NOTE 4 Many types of force can be measured, such as the normal forces or the lateral, friction, or shear force. When the latter is measured, the technique is referred to as lateral, frictional, or shear force microscopy. This generic term encompasses all of the types of force microscopy.

NOTE 5 AFMs can be used to measure surface normal forces at individual points in the pixel array used for imaging.

For typical AFM tips with radii < 100 nm, the normal force should be less than about 0,1 µN, depending on the sample material, or irreversible surface deformation and excessive tip wear occur.

[SOURCE: ISO 18115-2:2013, 3.2, ISO/TS 80004-6:2013, 3.5.2, ISO/TS 80004-13:2017, 5.1.2]

NOTE 6 The properties of graphene and related 2D materials that AFM can be used to measure are thickness, flake size, modulus, etc. The most effective method to measure the modulus of graphene is free standing indentation testing based on AFM[17].

ADVANTAGE: High resolution topographical imaging down to sub-nm[18]; no need for pre-process on samples.

LIMITATION: Lateral resolution sensitive to the sharpness of the tip; slow scanning.

6.2 Brunauer, Emmett and Teller method (BET)

BET is a method for the determination of the total specific external and internal surface area of disperse powders and/or porous solids by measuring the amount of physically adsorbed gas utilizing the model developed by Brunauer, Emmett and Teller for interpreting gas adsorption isotherms.

NOTE 1 Method originates from Brunauer, S., Emmett, P.H. and Teller, E.: Adsorption of gases in multimolecular layers, J. Am. Chem. Soc. 60 (1938) p. 309.

NOTE 2 The BET method is applicable only to adsorption isotherms of type II (disperse, nonporous or macroporous solids) and type IV (mesoporous solids, pore diameter between 2 nm and 50 nm). Inaccessible pores are not detected. The BET method cannot reliably be applied to solids which absorb the measuring gas.

[SOURCE:ISO/TS 80004-6:2013, 3.6.3]

ADVANTAGE: Inexpensive; fast.

LIMITATION: Not applicable to all types of isotherms; showing variation between measurements.

6.3 Combustion analysis

Combustion analysis is a method to determine the elemental composition of an organic compound by combusting the sample where the resulting combustion products can be quantitatively analysed. Carbon (C), hydrogen (H), nitrogen (N), sulfur (S).

ADVANTAGE: Ease of operation; inexpensive.

LIMITATION: Limited variety of elements.

6.4 Electron probe X-ray microanalysis (EPMA)

EPMA is a technique of spatially-resolved elemental analysis based upon electron-excited X-ray spectrometry with a focused electron probe and an electron interaction volume with micrometre to sub-micrometre dimensions.

[SOURCE: ISO 23833:2013, 3.1, ISO/TS 10798:2011, 3.2.9]

EPMA is fundamentally the same as SEM with the added capability of quantitative chemical analysis.

The properties of graphene and related 2D materials that EPMA can be used to measure are quantitative chemical analysis such as metal contents.

NOTE 1 Energy-Dispersive Spectrometry X-ray spectrometry (EDS or EDX) is X-ray spectrometry in which the energy of individual photons are measured by a parallel detector and used to build up a histogram representing the distribution of X-rays with energy.

[SOURCE: ISO/TS 80004-6:2013, 4.21, ISO/TS 80004-13:2017, 5.2.4]

NOTE 2 EDS (EDX or XEDS) is sometimes called Energy Dispersive X-ray Analysis (EDXA) or Energy Dispersive X-ray Microanalysis (EDXMA).

NOTE 3 When a wavelength-dispersive spectrometer for determining X-ray intensity as a function of the wavelength of the radiation[19] is used, the method is called Wavelength-Dispersive Spectroscopy (WDS). WDS uses the diffraction of X-ray on crystal samples and have better spectral resolution than EDS, but detects one element (at a particular wavelength) at a time while EDS measures a spectrum of all elements in the sample.

ADVANTAGE: High resolution as SEM (6.13); high sensitivity; fast compositional analysis; quantitative for 2D samples.

LIMITATION: Difficulty in sample preparation; sample in vacuum; detectors in cryostat temperature.

6.5 Electron spin resonance (ESR)

ESR is a method for studying chemical species that have one or more unpaired electrons through resonant excitation of electron spin.

NOTE 1 Similar to NMR but measuring electron spin.

[SOURCE: ISO/TS 80004-6:2013, 4.27]

The property of graphene and related 2D materials that ESR can be used to measure is crystal defect, especially by magnetic impurity.

NOTE 2 Electron Spin Resonance (ESR) is sometimes called Electron Paramagnetic Resonance (EPR).

ADVANTAGE: An effective method to detect the defects by vacancy or an adatom.

LIMITATION: Sensitive to temperature.

6.6 Fourier transform- infrared spectroscopy (FT-IR)

Analytical chemical technique based on absorption of infrared radiation by chemical moieties in the specimen, used to identify and quantitate the absorbing chemical moieties.

[SOURCE: ISO/TS 14101:2012, 3.3]

The property of graphene, especially GO, and related 2D materials that FT-IR can be used to measure is non-graphene contents and residue.

ADVANTAGE: Simple, easy, fast.

LIMITATION: The broad spectrum should be checked; spectrum overlapping.

6.7 Hall bar measurement

Method utilizing Lorentz force on electric carriers in a sample with appropriate contacts positioned to measure the Hall effect[20].

The property of graphene and related 2D materials that Hall effect measurement can be used to measure is electron mobility.

ADVANTAGE: Simple; relatively inexpensive.

LIMITATION: Sensitive to temperature.

6.8 Inductively coupled plasma — Mass spectrometry (ICP-MS)

ICP-MS is a method in which a high-temperature discharge generated in flowing argon by an alternating magnetic field induced by a radio-frequency (RF) load coil that surrounds the tube carrying the gas is detected using a mass spectrometer.

[SOURCE: ISO/TS 80004-6:2013, 4.22]

The property of graphene and related 2D materials that ICP-MS can be used to measure is metal contents.

ADVANTAGE: Fast; multi-element analysis; high sensitivity on elemental mass fraction; detection in the sub µg/kg range.

LIMITATION: Measurement of total mass fraction requires solubilization of sample, typically by acid digestion.

6.9 Kelvin probe force microscopy (KPFM)

KPFM is a dynamic-mode AFM (6.1) method using a conducting probe tip to measure spatial or temporal changes in the relative electric potentials of the tip and the surface.

[SOURCE: ISO 18115-2:2013, 3.12]

NOTE 1 Changes in the relative potentials reflect changes in the surface work function.

NOTE 2 Kelvin Probe Force Microscopy (KPFM)[21] is often used as a synonymous term to SKPM. SKPM and KPFM are dynamic modes of AFM (6.1).

NOTE 3 Scanning Kelvin Probe Microscopy (SKPM), also known as surface potential microscopy, is a noncontact variation of AFM and measures the contact potential difference (CPD) between a conducting tip and the sample in a noncontact way forming a capacitor (electrostatic force) while AFM measures the atomic force (Van der Waals force)[22].

Non-scanning Kelvin Probe Microscopy may be sometimes used as a rapid and accurate method for measuring work function at local points.

The properties of graphene and related 2D materials that KPFM can be used to measure are the work function and the number of layers.

NOTE 4 The carrier concentration can be estimated from work function if the work function at the Dirac point is known. The difference between the Dirac point and work function (Fermi level) can be used to calculate the carrier concentration. However, this method is only valid for the single layer graphene, or decoupled more than 1 layer graphene. AB-stacked bi-layer graphene features a parabolic E-k relationship, therefore the effective mass needs to be known.

NOTE 6 The surface contamination present from the growth or transfer procedures has great influence on the work function measurement using SKPM. Moreover, SKPM is often qualitative measurement by using commercial instruments which only measure the surface potential, and calibration of the probe against a reference material is needed for quantitative work function measurement.

ADVANTAGE: High spatial resolution same as AFM (6.1); non-contact.

LIMITATION: Slow.

6.10 Low energy electron microscopy (LEEM)

LEEM is a method that examines surfaces where images and/or diffraction patterns of the surfaces are formed by low-energy elastically backscattered electrons generated by a non-scanning electron beam.

NOTE 1 The method is typically used for the imaging and analysis of very flat, clean surfaces.

NOTE 2 Low energy electrons have energies typically in the range 1 eV to 100 eV.

[SOURCE: ISO/TS 80004-6:2013, 3.5.8]

The properties of graphene and related 2D materials that LEEM can be used to measure are thickness and the number of layers.

ADVANTAGE: Minimal electron beam damage; capable of real-time imaging.

LIMITATION: Relatively low resolution compared to SEM (6.13), typically several nm; ultra-high vacuum ($<10^{-6}$ torr) required.

6.11 Optical microscopy

Optical Microscopy is a technique that produces a magnified image of a sample using visible light. The technique is a quick way to observe domain boundaries of 2D materials. Recently, observing domain boundaries of graphene by regular optical microscopy even becomes possible on a copper plate when the copper has been purposefully oxidized[23].

ADVANTAGE: Inexpensive; fast.

LIMITATION: Resolution is generally diffraction limited.

NOTE 1 Super-resolution techniques beyond diffraction limit, such as Stimulated Emission Depletion (STED) or Near-field Scanning Optical Microscopy (NSOM), have been employed for graphene but these are fairly specialized.

6.12 Raman spectroscopy

Raman spectroscopy is spectroscopy in which the Raman effect is used to investigate molecular energy levels.

[SOURCE: ISO 18115-2:2013, 5.129, ISO/TS 80004-6:2013, 4.10]

Raman effect is emitted radiation, associated with molecules illuminated with monochromatic radiation, characterized by an energy loss or gain arising from rotational or vibrational excitations.

[SOURCE: ISO 18115-2:2013, 5.128]

The properties of graphene and related 2D materials that Raman spectroscopy can be used to measure are the number of layers, domain size, crystal defects, thermal conductivity, charge concentration (doping level), etc.

ADVANTAGE: Samples in solid, in liquid or in suspension.

LIMITATION: Small signals; sometimes laser excitation with more than one wavelength needed.

NOTE 1 Raman signal is usually very weak, and surface enhanced Raman spectroscopy (SERS) or tip enhanced Raman spectroscopy (TERS) is often used to increase the signal to noise ratio (S/N).

NOTE 2 The conventional Raman spectroscopy measurement can provide the information of the staking angle only for the AB-stacked or misoriented bilayer graphene. In order to measure the staking angle in general, the polarized (angle dependent) Raman with some additional measurement such as TEM (6.16) is needed[24].

6.13 Scanning electron microscopy (SEM)

SEM is a method that examines and analyses the physical information (such as secondary electron, backscattered electron, absorbed electron and X-ray radiation) obtained by generating electron beams and scanning the surface of the sample in order to determine the structure, composition and topography of the sample.

[SOURCE: ISO/TS 80004-6:2013, 3.5.5]

The properties of graphene and related 2D materials that SEM can be used to measure are flake size, crystal defects, etc.

ADVANTAGE: Relatively simple sample preparation, high resolution typically in few nm.

LIMITATION: Generally lower resolution than TEM (6.16).

6.14 Secondary-ion mass spectrometry (SIMS)

SIMS is a method in which a mass spectrometer is used to measure the mass-to-charge quotient and abundance of secondary ions emitted from a sample as a result of bombardment by energetic ions.

NOTE 1 SIMS is, by convention, generally classified as dynamic, in which the material surface layers are continually removed as they are being measured, and static, in which the ion areic dose during measurement is restricted to less than 10^{16} ions/m^2 in order to retain the surface in an essentially undamaged state.

[SOURCE: ISO/TS 80004-6:2013, 4.23]

ADVANTAGE: High sensitivity; identifying functional groups.

LIMITATION: Qualitative; vacuum; destructive.

6.15 Scanning tunnelling microscopy (STM)

STM is the SPM mode for imaging conductive surfaces by mechanically scanning a sharp, voltage-biased, conducting probe tip over their surface, in which the data of the tunnelling current and the tip-surface separation are used in generating the image.

NOTE 1 STM can be conducted in vacuum, a liquid or air. Atomic resolution can be achieved with suitable samples and sharp probes and can, with ideal samples, provide localized bonding information around surface atoms.

NOTE 2 Images can be formed from the height data at a constant tunnelling current or the tunnelling current at a constant height or other modes at defined relative potentials of the tip and sample.

NOTE 3 STM can be used to map the densities of states at surfaces or, in ideal cases, around individual atoms. The surface images can differ significantly, depending on the tip bias, even for the same topography.

[SOURCE: ISO 18115-2:2013, 3.34, ISO/TS 80004-6:2013, 3.5.3]

The properties of graphene and related 2D materials that STM can be used to measure are stacking angle, defects, and domain (grain) size.

ADVANTAGE: High resolution (0,1 nm lateral, 0,01 nm vertical), generally better than AFM (6.1); can be conducted in vacuum, a liquid or air.

LIMITATION: Slow.

6.16 Transmission electron microscopy (TEM)

TEM is a method 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/TS 80004-6:2013, 3.5.6]