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TECHNICAL SPECIFICATION

colour inside

Nanomanufacturing – Key control characteristics – Part 6-6: Graphene – Strain uniformity: Raman spectroscopy

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IEC Central Office
3, rue de Varembé
CH-1211 Geneva 20
Switzerland

Tel.: +41 22 919 02 11
info@iec.ch
www.iec.ch

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NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 6-6: Graphene – Strain uniformity: Raman spectroscopy

FOREWORD

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The text of this Technical Specification is based on the following documents:

Draft	Report on voting
113/579/DTS	113/605/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/standardsdev/publications.

A list of all parts of 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

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- withdrawn,
- replaced by a revised edition, or
- amended.

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INTRODUCTION

Graphene, a single layer of carbon atoms arranged in a honeycomb lattice, has a high potential for future nanoelectronic applications thanks to the excellent conductivity and high flexibility of the material. As there is a strong connection between nanoscale lattice deformations and carrier mobility, the uniformity of strain and flatness of the graphene lattice is a key control characteristic for the fabrication of high-quality graphene layers for electronic devices.

One of the most useful methods to evaluate the structural properties of graphene is Raman spectroscopy (see, for example, [1][1]). The method is simple, fast, non-destructive and well understood so that the Raman spectrum can be used as a fingerprint for graphene especially if the sample under evaluation consists of single-layer graphene not too far away from perfection. Things become more complicated if the sample consists of more than one layer, perhaps with different stacking angles and many lattice defects. As this document is intended to support the fabrication of nearly defect-free high-quality single-layer graphene, the interpretation of the Raman spectrum remains relatively simple.

As recently reported [2], nanometre-scale strain variations in graphene give rise to a pseudo-vector disorder potential which allows the pseudo-spin in graphene to flip and thus enables intra-valley backscattering. This scattering mechanism has been identified to be the responsible mechanism for limiting the carrier mobility in high-quality graphene [2]. Interestingly these nanometre-scale strain variations are directly connected to the experimentally observed linewidth of the Raman 2D-peak [3], making this quantity a very interesting measure for estimating the possibility of getting very high mobility graphene devices.

It is important to note that although graphene is a truly two-dimensional material, consisting exclusively of surface atoms, it is embedded in our three-dimensional world. This has the consequence that the properties of graphene are in all cases intrinsically influenced by its intimate surrounding. Thus, substrates or contact gases (in the case of suspended graphene) play a very crucial role when fabricating, transferring and characterizing graphene. Most crucially, substrates, contact gases and moisture are actually becoming part of the graphene system under investigation and there is no way (in practice) of eliminating their influence on the two-dimensional graphene layer.

1 Numbers in sqaure brackets refer to the Bibliography.

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 6-6: Graphene – Strain uniformity: Raman spectroscopy

1 Scope

This part of IEC 62607 establishes a standardized method to determine the structural key control characteristic

- strain uniformity

for single-layer graphene by

- Raman spectroscopy.

The width of the 2D-peak in the Raman spectrum is analysed to calculate the strain uniformity parameter which is a figure of merit to quantify the influence of nano-scale strain variations on the electronic properties of the layer. The classification will help manufacturers to classify their material quality to provide an upper limit of the electronic performance of the characterized graphene, to decide whether or not the graphene material quality is potentially suitable for various applications.

- The uniformity of strain measured by this method is applicable for nearly defect free, high-quality single-layer graphene, e.g. synthesized by chemical vapour deposition or graphene integrated into 2D-material heterostructures.

- The method is used if the Raman spectrum shows a visible D-peak with an integrated intensity ratio A(D)/A(G) < 0,1.
- Confocal Raman spectroscopy is used to consistently evaluate the graphene layer according to strain variations on the nanoscale.

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.

IEC TS 62607-6-11, *Nanomanufacturing – Key control characteristics – Part 6-11: Graphene film – Defect density: Raman spectroscopy* [2]

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/

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2 Under preparation. Stage at the time of publication: IEC DTS 62607-6-11.

- ISO Online browsing platform: available at http://www.iso.org/obp

3.1 General terms

3.1.1
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.

3.1.2
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]

3.1.3
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.

3.1.4
chemical vapour deposition
CVD
deposition of a solid material by chemical reaction of a gaseous precursor or mixture of precursors, commonly initiated by heat on a substrate

[SOURCE: ISO/TS 80004-13:2017, 3.2.1.1]

3.2 Key control characteristics

3.2.1
strain uniformity
Γ_{80}
quality parameter describing the uniformity of the strain distribution in the graphene layer

Note 1 to entry: Γ_{80} is the 80 % value of the 2D-peak width distribution, $FWHM(2D)_{80}$.

Note 2 to entry: The strain uniformity is a figure of merit describing the quality of graphene layers in respect of the uniformity of strain in the layer. Even if Γ_{80} can be calculated from basic physical principles, this is out of the scope of this document.

Note 3 to entry: The lower the value of Γ_{80}, the higher is the strain uniformity in the graphene layer. Low values of Γ_{80} are a necessary but not sufficient condition for high carrier mobility and high conductivity.

3.3 Measurement related terms

3.3.1
2D-peak
second order Raman peak related to a two-phonon process located at approximately twice the frequency of the D-peak

Note 1 to entry: As well as the D-peak, the 2D-peak is also dispersive with wavelength. The position of the 2D-peak changes strongly with laser energy.

Note 2 to entry: The 2D-peak is always present in the Raman spectrum of graphene and does not need defects to be activated.

3.3.2
D-peak
defect activated Raman peak related to lattice breathing modes in six-carbon rings away from the centre of the Brillouin zone

Note 1 to entry: The D-peak is located between 1 270 cm^{-1} and 1 450 cm^{-1} depending on the wavelength of the excitation laser. The dispersion with wavelength is approximately 50 cm^{-1}/nm.

Note 2 to entry: The D-peak is most intense at defective graphene lattices and disappears for perfect monolayer crystals. Therefore it is often called the disorder band.

3.3.3
D'-peak

defect activated Raman peak in the spectrum of graphene located around 1 620 cm^{-1} originating from scattering away from the Brillouin zone centre

3.3.4
G-peak
Raman peak related to in-plane motion of the carbon atoms located near 1 580 cm^{-1} originating from scattering at the centre of the Brillouin zone

Note 1 to entry: The G-peak can be observed in pristine graphene and does not need lattice defects to occur.

3.3.5
Raman spectroscopy
spectroscopy in which the radiation emitted from a sample illuminated with monochromatic radiation is characterized by an energy loss or gain arising from rotational, vibrational or phonon excitations

[SOURCE: ISO/TS 80004-13:2017, 3.3.1.6]

4 General introduction

4.1 Measurement principle

Raman spectroscopy is a very prominent tool for the investigation of carbon-based material systems [1][4]. In particular, scanning confocal Raman spectroscopy [5] has various beneficial features as a characterization tool for graphene and graphene-related materials such as a particularly high spatial resolution of up to 0,5 µm. If operated with carefully chosen parameters., it is generally a non-destructive method for investigating graphene, Other positive aspects include it being rather fast, and the possibility to analyse graphene that is buried underneath protecting layers of insulating materials, provided they are thin.

In fact, Raman spectroscopy has been shown to be particularly useful for graphene research right from the first isolation of graphene in 2004. The first milestone was made by identifying that the Raman spectrum of single- and bi-layer graphene shows severe differences, so that the technique can be used to distinguish between the exact thickness of graphene and few-layer graphene crystals in their natural crystal stacking order [5][6]. In later years, it was demonstrated that the charge carrier doping of a graphene sample, the amount of mechanical strain in the lattice, as well as nanometre-scaled strain variations [3] and the amount of lattice defects can be determined from a Raman spectrum. All these parameters are essential for the fabrication of graphene-based devices and for the analysis and improvement of synthesis and patterning processes.

It is important to note that strain variations in the graphene lattice are limiting the carrier mobility in "defect-free" graphene samples and allow for an estimate of the achievable carrier mobility in both exfoliated and CVD graphene layers on various substrates.

In a more general sense, the method of scanning confocal Raman mapping can be used to compare graphene layers on different substrates or graphene transfer processes under different fabrication conditions. The experimentally observed linewidth of the 2D-peak is known to contain information about the nanometre-scaled strain variations in graphene [3]. The lower the linewidth, the lower the nanometre-scaled strain variations. This document provides a quantitative method to measure the linewidth including a statistical interpretation to deliver a single value which acts as a key control characteristic of graphene on substrates.

A typical Raman spectrum of defect-free graphene is shown in Figure 1. Raman analysis concentrates on the three most dominating Raman peaks for graphene, the D-, G- and 2D-peaks. Note the absence of the defect-induced D-peak located between 1 270 cm^{-1} and 1 450 cm^{-1}.

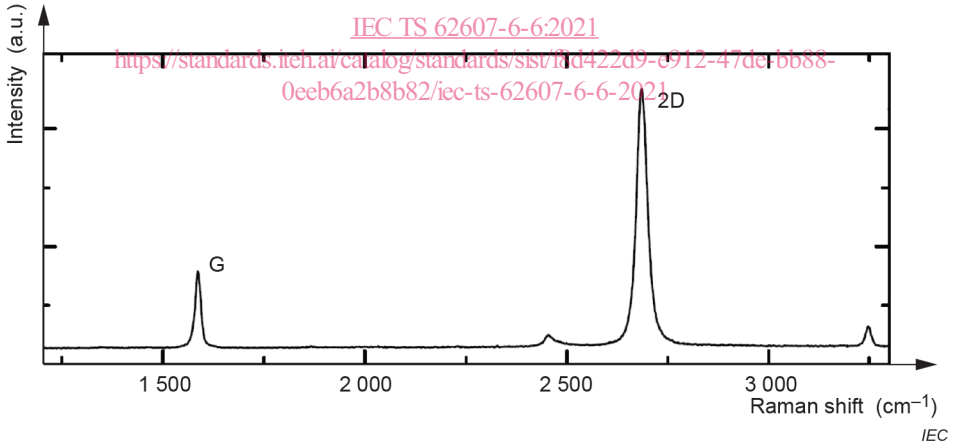

Figure 1 – Typical Raman spectra of an exfoliated graphene flake adopted from [6]

This document addresses pristine or nearly defect-free graphene, similar to that shown in Figure 1. Due to the absence of the D-peak in defect-free samples, it cannot be used to further characterize the quality of the graphene. However, for such defect-free graphene, the 2D-peak, and in particular its linewidth, can be used for the further evaluation of the uniformity of strain which has been proven to be crucial for high carrier mobilities [2][3]. Note that, while strain variations were found to be the dominating limiting factor of the charge carrier mobilities [2], other imperfections can also limit the mobility to a lower value. Therefore, the measured small linewidth is a necessary, but not a sufficient condition to achieve a high mobility value.

4.2 Sample preparation method

Graphene on insulating substrates and certain metals, such as copper, can be reliably characterized by Raman spectroscopy. The sample needs to be sufficiently larger than the