

IEC TS 62607-6-14

Edition 1.0 2020-10

TECHNICAL SPECIFICATION



Nanomanufacturing – Key control characteristics – Part 6-14: Graphene-based material – Defect level: Raman spectroscopy

INTERNATIONAL ELECTROTECHNICAL COMMISSION

ICS 07.120 ISBN 978-2-8322-8940-2

Warning! Make sure that you obtained this publication from an authorized distributor.

CONTENTS

FC	DREWO	RD	4
IN	TRODU	CTION	6
1	Scop	e	8
2	Norm	native references	8
3	Term	s and definitions	8
	3.1	General terms	8
	3.2	Key control characteristics measured in accordance with this document	
4	Gene	, eral	
	4.1	Measurement principle	11
	4.2	Sample preparation method	
	4.3	Description of measurement equipment/apparatus	
	4.4	Supporting materials	
	4.5	Ambient conditions during measurement	
5		surement procedure	
	5.1	Calibration of measurement equipment	
	5.2	Detailed protocol of the measurement procedure	
	5.3	Measurement accuracy	
	5.4	Measurement uncertainty source	
6		oling plan	
7		analysis / interpretation of results	
8		Its to be reported	
	8.1	General	
	8.2	Product/sample identification	
	8.3	Test conditions	
	8.4	Measurement specific information	
	8.5	Test results	
		informative) Recommended format of the test report	
Ar	nnex B (informative) Sampling plan	17
Ar	nex C	informative) Case study: measurement and data analysis	18
	C.1	Step 1: sample preparation	18
	C.2	Step 2: Raman test	18
	C.3	Step 3: Raman spectra processing	18
	C.4	Step 4: Data analysis	19
Ar	nex D (informative) Why use the intensity ratio $I_{D+D'}/I_{2D}$ for defect level	
ch	aracter	zation of graphene powder?	22
	D.1	Interpretation of characteristic bands in the Raman spectrum of graphene and Raman scattering mechanism	22
	D.2	Example – Influence of edges in the Raman spectrum of graphene	
	D.3	Example – Influence of defect in the Raman spectrum of single layer graphene	
	D.4	Example – Raman characteristics of reduced graphene sheet	
	D.5	Conclusion	
Ril		phy	
ייכ	znograp	······································	20
Fig	gure 1 -	- Schematic diagram of Raman scattering processes in realistic graphene	e

Figure 2 – Different packing configurations of graphene flakes in film (left) and powder (right)	6
Figure 3 – Schematic drawing of Raman spectra of defective graphene (upper) and pristine graphene (bottom)	12
Figure 4 – Schematic drawing of sample preparation method	12
Figure B.1 – Schematic drawing of five-point-sampling method	17
Figure B.2 – Location of measurement points	17
Figure C.1 – The field view of graphene sample beneath Raman microscope	18
Figure C.2 – The procedure of Raman spectrum processing	19
Figure C.3 – Typical Raman spectrum after processing	20
Figure C.4 – The overall defect level of one test sample	21
Figure C.5 – Measurement results of different testing organizations	21
Figure D.1 – Characteristic bands in the Raman spectrum of graphene and Raman processes [6]	22
Figure D.2 – Raman spectra from the edges of a monolayer graphene sample [9]	23
Figure D.3 – Raman spectra obtained from monolayer graphene samples with hexagonal and circular holes [10]	23
Figure D.4 – Raman spectra for four different ion doses in graphene [1]	24
Figure D.5 – E_{L}^4 [I_{D}/I_{G}] as a function of L_{D} [2]	24
Figure D.6 – (a) Definition of the activated A-region (green) and structurally- disordered S-region (red). (b-e) Snapshots of the structural evolution of the graphene sheet for different defect concentrations [1]	25
Figure D.7 – Evolution of 2D and other second-order bands with increasing ion doses [5]	25
Figure D.8 – Raman characteristics of as-made graphene sheet and different types of reduced graphene sheet film samples: (a) Raman spectra; (b) D/G intensity ratios; (c) S3/2D intensity ratios [7]	26
Figure D.9 – Resistivity of as-made graphene sheet and different types of reduced graphene sheet [7]	26
Table A.1 – Product identification (in accordance with IEC 62565-3-1)	15
Table A.2 – General material description (in accordance with IEC 62565-3-1)	15
Table A.3 – Information related with test	16
Table A.4 – Measurement results	16
Table C.1 – Average I_{D+D}'/I_{2D} of each test point	20

INTERNATIONAL ELECTROTECHNICAL COMMISSION

NANOMANUFACTURING - KEY CONTROL CHARACTERISTICS -

Part 6-14: Graphene-based material – Defect level: Raman spectroscopy

FOREWORD

- 1) The International Electrotechnical Commission (IEC) is a worldwide organization for standardization comprising all national electrotechnical committees (IEC National Committees). The object of IEC is to promote international co-operation on all questions concerning standardization in the electrical and electronic fields. To this end and in addition to other activities, IEC publishes International Standards, Technical Specifications, Technical Reports, Publicly Available Specifications (PAS) and Guides (hereafter referred to as "IEC Publication(s)"). Their preparation is entrusted to technical committees; any IEC National Committee interested in the subject dealt with may participate in this preparatory work. International, governmental and non-governmental organizations liaising with the IEC also participate in this preparation. IEC collaborates closely with the International Organization for Standardization (ISO) in accordance with conditions determined by agreement between the two organizations.
- 2) The formal decisions or agreements of IEC on technical matters express, as nearly as possible, an international consensus of opinion on the relevant subjects since each technical committee has representation from all interested IEC National Committees.
- 3) IEC Publications have the form of recommendations for international use and are accepted by IEC National Committees in that sense. While all reasonable efforts are made to ensure that the technical content of IEC Publications is accurate, IEC cannot be held responsible for the way in which they are used or for any misinterpretation by any end user.
- 4) In order to promote international uniformity, IEC National Committees undertake to apply IEC Publications transparently to the maximum extent possible in their national and regional publications. Any divergence between any IEC Publication and the corresponding national or regional publication shall be clearly indicated in the latter.
- 5) IEC itself does not provide any attestation of conformity. Independent certification bodies provide conformity assessment services and, in some areas, access to IEC marks of conformity. IEC is not responsible for any services carried out by independent certification bodies.
- 6) All users should ensure that they have the latest edition of this publication.
- 7) No liability shall attach to IEC or its directors, employees, servants or agents including individual experts and members of its technical committees and IEC National Committees for any personal injury, property damage or other damage of any nature whatsoever, whether direct or indirect, or for costs (including legal fees) and expenses arising out of the publication, use of, or reliance upon, this IEC Publication or any other IEC Publications.
- 8) Attention is drawn to the Normative references cited in this publication. Use of the referenced publications is indispensable for the correct application of this publication.
- 9) Attention is drawn to the possibility that some of the elements of this IEC Publication may be the subject of patent rights. IEC shall not be held responsible for identifying any or all such patent rights.

The main task of IEC technical committees is to prepare International Standards. In exceptional circumstances, a technical committee may propose the publication of a Technical Specification when

- the required support cannot be obtained for the publication of an International Standard, despite repeated efforts, or
- the subject is still under technical development or where, for any other reason, there is the future but no immediate possibility of an agreement on an International Standard.

Technical Specifications are subject to review within three years of publication to decide whether they can be transformed into International Standards.

IEC TS 62607-6-14, which is a Technical Specification, has been prepared by IEC technical committee 113: Nanotechnology for electrotechnical products and systems.

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

Enquiry draft	Report on voting
113/495/DTS	113/536/RVDTS

Full information on the voting for the approval of this Technical Specification can be found in the report on voting indicated in the above table.

This document has been drafted in accordance with the ISO/IEC Directives, Part 2.

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 "http://webstore.iec.ch" in the data related to the specific document. At this date, the document will be

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

IMPORTANT – The 'colour inside' logo on the cover page of this publication indicates that it contains colours which are considered to be useful for the correct understanding of its contents. Users should therefore print this document using a colour printer.

INTRODUCTION

Graphene has been intensively studied by researchers from both academic and industrial communities due to its unique properties, which include exceptional thermal conductivity, great strength and excellent transparency. Defects in graphene influence its optical and magnetic performance, electronic structure and thermal conductivity, thus influencing its applications. Therefore, defect is a key control characteristic for the fabrication of high-quality graphene for desired applications.

One of the most useful methods to evaluate defect level in graphene is Raman spectroscopy, which is sensitive to the structure of samples. This method is efficient, non-contact and well-understood. The defect states and boundary states of realistic graphene material will induce a series of Raman scattering processes (Figure 1). Some of scattering processes are only associated with defective states, which are used in this document to analyse defect level in graphene powder.

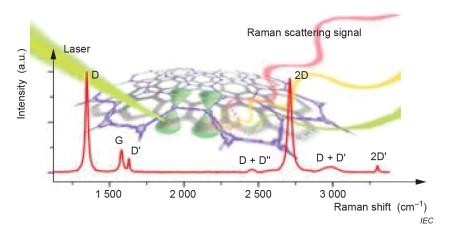


Figure 1 – Schematic diagram of Raman scattering processes in realistic graphene material

Commercialized graphene samples can be classified by their physical forms as graphene film, graphene powder and graphene solution. Figure 2 shows the schematic packing configurations of graphene flakes in graphene film (left side of Figure 2) and graphene powder (right side of Figure 2) and their corresponding SEM images.

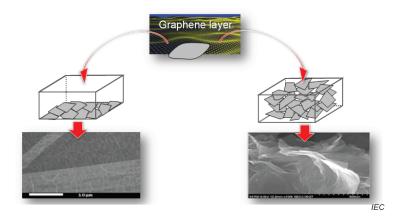


Figure 2 – Different packing configurations of graphene flakes in film (left) and powder (right)

Usually, defects in graphene films are characterized by the intensity ratio of two principle bands – D band and G band – in Raman spectra (symbolized by I_D/I_G) [1],[2]. However, in graphene powders consisting of flakes with sizes below 10 µm there are numerous edges and boundary states, which all contribute to the D-band signal and make its correlation to various defects problematic. The D-band intensity could result from the contribution of edges, boundary states or defects, so it is not appropriate to determine the defect level of graphene powder with the parameter I_D/I_G .

D+D' band is only relevant with defects in graphene powder, but not with edges and boundary states. Therefore, in order to characterize defect level in graphene powder, the intensity ratio of D+D' and 2D bands (symbolized by $I_{D+D'}/I_{2D}$) is proposed as a more relevant parameter in this document. Detailed information can be found in Annex D.

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 6-14: Graphene-based material - Defect level: Raman spectroscopy

1 Scope

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

defect level

for powders consisting of graphene-based material by

Raman spectroscopy.

The defect level is derived by the intensity ratio of the D+D' band and 2D band in Raman spectrum, $I_{\rm D+D'}/I_{\rm 2D}$.

- The defect level determined in accordance with this document will be listed as a key control characteristic in the blank detail specification for graphene IEC 62565-3-1 for graphene powder.
- The method is applicable for graphene powder or graphene-based material, e.g. reduced graphene oxide (rGO), bilayer graphene, trilayer graphene and few-layer graphene.
- Typical application areas are quality control and classification for graphene manufacturers, and product selection for downstream users.
- The method described in this document is appropriate if the physical form of graphene is powder.

2 Normative references

There are no normative references in this document.