IEC TS 62607-6-2

Edition 1.0 2023-04

TECHNICAL SPECIFICATION

colour inside

Nanomanufacturing – Key control characteristics – Part 6-2: Graphene – Number of layers: atomic force microscopy, optical transmission, Raman spectroscopy

INTERNATIONAL ELECTROTECHNICAL COMMISSION

ICS 07.120

ISBN 978-2-8322-6749-3

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

® Registered trademark of the International Electrotechnical Commission

CONTENTS

FOREWORD 4
INTRODUCTION 6
1 Scope 7
2 Normative references 7
3 Terms and definitions 7
3.1 General terms 7
3.2 Terms related to measurements 9
4 Method for preparation of graphene flake sample 11
4.1 Micromechanical cleavage 11
4.2 Sonication 11
4.3 Ball milling 11
4.4 Fluid dynamics 11
5 Measurement of the number of graphene layers using combined method 12
5.1 Basic concept of combined method 12
5.2 General protocol 12
5.2.1 Sample preparation 12
5.2.2 AFM calibration 12
5.2.3 Raman calibration 12
5.2.4 Optical reflectance calibration 12
5.3 Measurement procedure 12
6 Data analysis and interpretation of results 13
6.1 General protocol for data analysis 13
6.2 Analysis of number of layers of graphene using Raman spectroscopy 13
6.3 Analysis of number of layers of graphene using AFM topography 13
6.4 Analysis of number of layers of graphene using reflectance (Rayleigh scattering) 14
7 Report 14
Annex A (informative) Summary of three simultaneous measurements and their analysis 15
A.1 Flowchart for determining the number of layers of graphene 15
A.2 Summary table of analysis process 16
Annex B (informative) Interpretation of the simultaneous measurement for Raman scattering, AFM, and reflectance – case studies 17
Annex C (informative) Description of the measurement apparatus 20
C.1 General 20
C.2 AFM system 20
C.3 Spectroscopy system 20
Annex D (informative) Measurement using currently available equipment 21
D.1 Atomic force microscopy 21
D.2 Optical transmittance and reflectance 22
D.3 Raman scattering 23
D.4 Reflection and optical contrast (Rayleigh scattering) 23
Bibliography 24

Figure 1 – Schematic of simultaneous measurement of AFM, Raman scattering, and light reflectance 13

Figure A.1 – Flowchart for determining the number of layers of graphene 15
Figure B.1 – Simultaneous measurement of Raman scattering, AFM, and reflectance images to determine the number of graphene layers 17
Figure B.2 – Confocal Raman spectrum, AFM line profile and reflectance intensity profile of graphene flake extracted along red line in Figure B.1 18
Figure B.3 – Confocal Raman spectrum, AFM line profile and reflectance intensity profile of graphene flake extracted along cyan line in Figure B.1 19
Figure B.4 – Confocal Raman spectrum, AFM line profile and reflectance intensity profile of graphene flake extracted along black line in Figure B.1 19

Table A.1 – Summary table of analysis process 16
Table D.1 – Summary of selected results for monolayer graphene thickness measured by AFM with preparation method, AFM method, substrate, and whether monolayer graphene was confirmed by Raman spectroscopy [7] 22

INTERNATIONAL ELECTROTECHNICAL COMMISSION

_____

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 6-2: Graphene – Number of layers: atomic force microscopy, optical transmission, 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.

IEC TS 62607-6-2 has been prepared by IEC technical committee 113 Nanotechnology for electrotechnical products and systems. It is a Technical Specification.

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

Draft	Report on voting
113/676/DTS	113/727/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 http://www.iec.ch/standardsdev/publications.

A list of all parts in 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

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

IMPORTANT – The "colour inside" logo on the cover page of this document 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 attracted significant interest as a next-generation electronic material due to its good conductivity and mobility. It has been regarded as more advantageous than carbon nanotube (CNT) because of its isotropic and homogeneous electronic properties. For these reasons and many more, a Nobel prize in physics was awarded to A. Geim and C. Novoselov in 2010 for their efforts in discovering graphene when they isolated a single layer of graphene using clear adhesive tape.

Graphene has been widely studied by researchers from academic institutions, research institutes, and industries due to its unique and interesting properties such as conductivity [1][1], mechanical strength and flexibility [2], which are better than other metals or semiconductors. These properties are influenced by the number of layers of graphene and disappear as the number of layers increases. Graphene also shows an unusual reduction in optical transparency even considering a single atomic layer [3]. Therefore, graphene applications need to investigate the precise number of layers of graphene.

Many companies are now providing graphene samples to industries and research communities. These are prepared (or manufactured) by various methods such as CVD or mechanical exfoliation. Defining and evaluating the number of layers of this fabricated graphene is critical both from research and industrial points of view. Unfortunately, there are no commonly accepted standards for this purpose, hindering the reliable production and expansion of graphene applications.

The number of layers of graphene is usually observed by atomic force microscopy (AFM), light transmittance, Raman spectroscopy, transmission electron microscopy (TEM), and ellipsometry. Every analytical method has its own limitations in terms of precisely measuring the number of graphene layers and can also cause ambiguity for providing reliable information. For these reasons, developing an easy, fast, and reliable method for counting the number of graphene layers is needed.

This document describes a combined method to evaluate accurate number of layers of graphene, which includes measurement method.

Description of combined method and case studies illustrating the application of the standard are provided in Annex A and Annex B, respectively.

---

1 Numbers in square brackets refer to the Bibliography.

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 6-2: Graphene – Number of layers: atomic force microscopy, optical transmission, Raman spectroscopy

1 Scope

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

– number of layers

for graphene flakes by a combination of

– atomic force microscopy,
– optical transmission, and
– Raman spectroscopy

2 Normative references

There are no normative references in this document.