IEC TS 62607-6-21

Edition 1.0 2022-09

TECHNICAL SPECIFICATION

colour inside

Nanomanufacturing – Key control characteristics – Part 6-21: Graphene-based material – Elemental composition, C/O ratio: X-ray photoelectron spectroscopy

INTERNATIONAL ELECTROTECHNICAL COMMISSION

ICS 07.120; 07.030

ISBN 978-2-8322-5700-5

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 Key control characteristics measured in accordance with this document 9
4 General 10
4.1 Measurement instrument 10
4.2 Calibration of measurement instrument 10
4.3 Charge control 10
4.4 Quantitative analysis 11
5 Measurement procedure 12
5.1 Sample preparation 12
5.2 Measurement conditions 12
5.3 Measurement procedure 12
6 Data analysis 13
7 Uncertainty estimation 13
8 Measurement report 13
8.1 General 13
8.2 Test sample identification 13
8.3 Ambient conditions 13
8.4 Instrumental information 14
8.5 Measurement specific information 14
8.6 Measurement results 14
Annex A (informative) Relative sensitivity factors 15
A.1 Overview 15
A.2 Elemental relative sensitivity factors 16
A.2.1 General 16
A.2.2 Pure-element relative sensitivity factors 16
A.2.3 Elemental relative sensitivity factors from measurements with compounds 16
A.2.4 Set of elemental relative sensitivity factors 16
Annex B (informative) Discussion about the influence of surface contamination 17
B.1 Sampling depth 17
B.2 Test study of surface contamination 18
Annex C (informative) Case study for GNP 20
C.1 Test sample 20
C.2 Measurement results using XPS 20
Annex D (informative) Case study for GO 24
D.1 Test sample 24
D.2 Measurement results 24
Bibliography 26

Figure 1 – Illustration of XPS peak measured 11

Figure 2 – Digital photos of test samples as pressed pellet of graphene powder on different substrates 12
Figure B.1 – Schematic diagram of sampling depth and XPS spectra 17
Figure B.2 – Data distribution of relative abundance of C 1s at five test positions through long-time vacuum desorption treatment 18
Figure B.3 – XPS spectra of C 1s through thermal desorption of different temperatures for 1 h 19
Figure C.1 – Morphologies of GNP sample 20
Figure C.2 – Survey spectrum of GNP test sample 21
Figure C.3 – XPS spectra of the main elements of C 1s, O 1s, N 1s, Cl 2p and S 2p peaks in GNP test sample 22
Figure C.4 – Data distribution of relative abundance of C, O, and N elements and C/O ratio of eleven test positions in GNP test sample 23
Figure D.1 – Data distribution of relative abundance of C, O, S and N elements and C/O ratio of twelve test positions in GO test sample 25

Table 1 – Reference values for the peak positions on the binding-energy scale, $E_{\mathrm{ref}\ n}$ 10
Table B.1 – Relative abundance of C 1s at different times of vacuum desorption 19
Table B.2 – Relative abundance of C 1s through vacuum desorption under different temperatures for 1 h 19
Table C.1 – Relative abundance of main elements and C/O ratio in GNP test sample 23
Table D.1 – Relative abundance of main elements and C/O ratio in GO test sample 24

INTERNATIONAL ELECTROTECHNICAL COMMISSION

_____

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 6-21: Graphene-based material – Elemental composition, C/O ratio: X-ray photoelectron 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 62607-6-21 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
113/607/DTS	113/630/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

- 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 unique electrical, thermal and mechanical properties and has wide potential industrial application, especially in the electronics industry: batteries, integrated circuits, high-frequency electronics, displays, etc. [1], [2], [3], [4], [5][1]. The content of main elements, especially oxygen element and the ratio of carbon to oxygen are the significant parameters influencing the electronic and thermal application performance of graphene materials [3]. The main elements in graphene materials include carbon (C), oxygen (O), nitrogen (N), sulfur (S), chloride (Cl), and silicon (Si). The C/O ratio is a key parameter to identify the type of graphene or graphene-oxide (GO), and reflects directly the degree of reduction and product quality of reduced graphene oxide (rGO). Because of multiple different production processes and manufacturers for graphene powder, the main elemental composition and C/O ratio are also different. For the development of industrial application, a standard measurement method with reliability, accuracy and reproducibility needs to be established. The X-ray photoelectron spectroscopy (XPS) technique can measure multiple elements simultaneously and obtain accurately the relative abundance of each element in a test sample [6], [7].

The purpose of this document is to provide an optimized preparation, measurement and analysis procedure for graphene powder, to enable accurate and quantitative determination of the C, O, N, S, Cl, Si elements and C/O ratio using the XPS technique.

This document has been developed based on study in VAMAS Technical Working Area 41 (TWA 41).

---

1 Numbers in square brackets refer to the Bibliography.

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 6-21: Graphene-based material – Elemental composition, C/O ratio: X-ray photoelectron spectroscopy

1 Scope

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

– elemental composition, and
– C/O ratio

for powders of graphene-based materials by

– X-ray photoelectron spectroscopy (XPS).

The elemental composition (species and relative abundance) is derived by the elemental binding energy and integral peak area at corresponding portion of XPS spectrum.

– The elemental composition refers to main elements in graphene powders, typically including carbon (C), oxygen (O), nitrogen (N), sulfur (S) , chloride (Cl) and silicon (Si).
– This document is applicable to graphene powders consisting of graphene, bilayer graphene (2LG), trilayer graphene (3LG), few-layer graphene (FLG), graphene nanoplate (GNP), reduced graphene oxide (rGO), graphene oxide (GO), and functionalized graphene powders.
– Typical application areas are the microelectronics and thermal management industries, e.g. batteries, integrated circuits, high-frequency electronics. This document can be used by manufacturers in research and development and by downstream users for product selection.

2 Normative references

There are no normative references in this document.