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TECHNICAL SPECIFICATION

Nanomanufacturing – Key control characteristics –
Part 6-18: Graphene-based material – Functional groups: TGA-FTIR

colour inside

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NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

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FOREWORD

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IEC TS 62607-6-18 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
113/680/DTS	113/706/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/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- withdrawn,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.

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INTRODUCTION

One of the most well-studied routes for the preparation of graphene is the oxidation and reduction process. The most cost-effective process to obtain graphene is the exfoliation of natural graphite layers after oxidation to get individual oxidized layers and then de-oxygenation (reduction) of these individual layers [1], [2] [1]. During the oxidation process, various functionalized groups (-OH, -O-, -COOH, C=O, etc.) go into the graphene skeleton, breaking the π bond of graphene structure [3]. Oxygen attachment to graphene in any chemical form (epoxide, hydroxyl, carboxyl and ketonic-type functional groups) both on the basal plane and at the edges reduces electronic states at the Fermi level [4], [5], [6]. The type and content of functional groups affect the physiochemical properties of graphene. Therefore, the identification and quantification of functional groups on graphene powder is believed to be a key control characteristic for its production and application.

Coupling thermal gravimetric analysis (TGA) and Fourier transform infrared spectroscopy (FTIR) is an excellent solution to identify and quantify functional groups on graphene powder. In TGA-FTIR, while mass changes such as sample pyrolysis and vaporization that accompany changes in temperature are measured quantitatively by the TGA, qualitative analysis of the gaseous components can be conducted simultaneously by FTIR measurement of the obtained spectra. This document focuses on determining the type and content of functional groups (e.g. hydroxyl, amino, carboxyl, alkyl, carbonyl, sulfonic acid group) on graphene powder by coupling TGA and FTIR.

[1] Numbers in square brackets refer to the Bibliography.

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 6-18: Graphene-based material – Functional groups: TGA-FTIR

1 Scope

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

- functional groups

for functionalized graphene-based material and graphene oxide by

- thermogravimetry analysis (TGA) coupled with Fourier transform infrared spectroscopy (FTIR), referred to as TGA-FTIR.

The content of functional groups is derived by changes in mass of the sample as a function of temperature using TGA. Materials evolved during these mass changes are then analysed using coupled FTIR to identify functional groups.

- The functional groups determined according to 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 functionalized graphene powder and graphene oxide that can be pyrolysed and gasified with elevated temperature during TGA.
- Typical application areas are quality control for graphene manufacturers, and product selection for downstream users.

2 Normative references

There are no normative references in this document.

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

3.1 General terms

3.1.1
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]

3.1.2
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]

3.1.3
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
blank detail specification
BDS
structured generic specification of the set of key control characteristics which are needed to describe a specific nano-enabled product without assigning specific values and/or attributes

Note 1 to entry: The templates defined in a blank detail specification list the key control characteristics for the nano-enabled material or product without assigning specific values to it.

Note 2 to entry: Examples of nano-enabled products are: nanomaterials, nanocomposites and nano-subassemblies.

Note 3 to entry: Blank detail specifications are intended to be used by industrial users to prepare their detail specifications used in bilateral procurement contracts. A blank detail specification facilitates the comparison and benchmarking of different materials. Furthermore, a standardized format makes procurement more efficient and more error robust.

3.1.5
sectional blank detail specification
SBDS
specification based on a blank detail specification adapted for a subgroup of the nano-enabled product

Note 1 to entry: In general the sectional blank detail specification contains a subset of those KCCs listed in the blank detail specification. In addition, sectional specific key control characteristics may be added if they are not listed in the blank detail specification.

Note 2 to entry: The templates defined in the sectional blank detail specification may contain key control characteristics with and without assigned values and attributes.

Note 3 to entry: The section can be defined by application, manufacturing method or general material properties.

3.1.6
detail specification
DS
specification based on a blank detail specification with assigned values and attributes

Note 1 to entry: The properties listed in the detail specification are usually a subset of the key control characteristics listed in the relevant blank detail specification. The industrial partners define only those properties which are required for the intended application.

Note 2 to entry: Detail specifications are defined by the industrial partners. SDOs will be involved only if there is a general need for a detail specification in an industrial sector.

Note 3 to entry: The industrial partners may define additional key control characteristics if they are not listed in the blank detail specification.

3.1.7
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.

3.2 Key control characteristics measured in accordance with this document

3.2.1
functional group
atom or group of atoms that has similar chemical properties whenever it occurs in different compounds, which defines the characteristic physical and chemical properties of families of organic compounds

3.3 Terms related to the measurement method

3.3.1
thermogravimetry analysis
TGA
method in which the change in mass of a sample is measured as a function of temperature while the sample is subjected to a controlled temperature programme.

[SOURCE: ISO/TS 80004-6:2021, 6.1.2, modified – The term has been changed from "thermogravimetry" to "thermogravimetry analysis".]

3.3.2
Fourier transform infrared spectroscopy
FTIR
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]

3.3.3
attenuated total reflection mode
ATR Mode
instrumental mode of operation in which the incident angle of IR light on the crystal is adjusted to be higher than the critical angle

Note 1 to entry: The light is completely reflected by the upper surface of the crystal, and the intensity of the light is attenuated through absorption by materials covering the upper surface of the crystal. The frequency of IR light absorbed is used to identify the absorbed chemical moiety, and the fraction of light that is absorbed is used to quantitate the amount of that moiety present.

[SOURCE: ISO/TS 14101:2012, 3.1]

3.3.4
evolved gas analysis
method in which the nature and/or amount of volatile product(s) released by a substance is (are) measured as a function of temperature while the substance is subjected to a controlled temperature programme.

[SOURCE: ISO/TS 80004-6:2021, 5.26]

4 General

4.1 Measurement principle

In TGA-FTIR, TGA is connected via heated and temperature-controlled transfer line. The samples are heated at a given heating rate from room temperature to the desired temperature in an inert gas such as ultra-purity nitrogen or helium. While mass changes such as sample pyrolysis and vaporization that accompany changes in temperature are measured quantitatively by the TGA, qualitative analysis of the evolved gases from pyrolysed materials can be monitored simultaneously by FTIR measurement of the obtained spectra.

4.2 Sample preparation method

For TGA measurements, tablet at least 5 mg of graphene powder for 1 min with pressure of 3 MPa to 4 MPa. The size of the sample tablet is controlled to be suitable for the pan of TGA.

For ATR measurements, gently press the graphene powder with a glass slide to ensure the sample contacting with ATR base is as smooth as possible, so that satisfactory spectral signals can be produced from ATR tests.

4.3 Measurement system

The heated gas cell is placed in the FTIR sample compartment for detection of the decomposition products. One end of the transfer line is interfaced to the inlet port of the gas cell and the other end to the TGA. TGA monitors sample weight loss caused by volatilization and pyrolysis often as a function of temperature ramping using a high-precision balance and furnace. Evolved sample gases originating from the TGA pass through the heated transfer line into the heated gas cell in the FTIR sample compartment. As these evolved gases travel through the gas cell, FTIR spectra are collected and stored for further processing to obtain qualitative and quantitative information.

4.4 Description of measurement equipment

The transfer line from the TGA to the gas cell needs to present an inert, nonporous surface to the evolved gas. Evolved gas transfer lines shall be heated to temperatures sufficient to prevent condensation of the evolved gas species. The temperature of the transfer line is normally held constant during an experiment at a level chosen to avoid both condensation and degradation of the evolved gases. Typical working temperatures have a range of 150 °C to 300 °C.

The gas cell usually is heated to a constant temperature at or slightly higher than the temperature of the transfer line, approximately 10 °C higher, to avoid condensation of the evolved gas. However, the maximum temperature recommended by the manufacturer should not be exceeded.

The ends of the gas cell are sealed with infrared transmitting windows or window and mirror combinations.

4.5 Supporting materials

During the analysis, reagents and materials are needed. Unless otherwise stated, use only reagents of recognized analytical grade.