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Nanomanufacturing - Key control characteristics -
Part 6-33: Graphene-related products - Defect density of graphene: electron energy loss spectroscopy

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Nanomanufacturing - Key control characteristics - Part 6-33: Graphene-related products - Defect density of graphene: electron energy loss spectroscopy

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IEC TS 62607-6-33 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
113/913/DTS	113/932/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 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– withdrawn, or

– revised.

INTRODUCTION

Quantitative and qualitative measurement of graphene defect is one of the most important factors to determine the quality of graphene. This document presents an analytical method for measuring local defect of graphene or graphene-related materials using scanning transmission electron microscopy (STEM) with electron energy loss spectroscopy (EELS). The scanning capability used in this method enables the defect distribution to be analysed, which can be used in determination of graphene quality. This document will be strengthened and compensated by other methods, such as Raman spectroscopy, X-ray photoelectron spectroscopy (XPS), optical microscopy, etc., which have already been standardized in IEC TC 113.

STEM-based EELS uses electron beams with sub-nanometre scale, which allows structural observations within a highly localized area. Therefore, it is suitable for tracking important defect information that occurs at the atomic scale. Unlike other macroscopic analytical tools (e.g. Raman and XPS), the EELS method sets a clear boundary between graphene and defects that are induced by lattice transformation, doping, and adatoms. Moreover, the method determines the classification and quantity of defects including the number of holes and amorphous regions. These characteristics would complement the existing standards for measuring the defect density in graphene. Particularly, the advancements in STEM-EELS instruments, combined with large-scale data processing technologies, can significantly enhance their complementarity with other macroscopic analysis tools.

The electron energy structure of carbon atoms varies according to the irradiation direction of the electron beam or the sample orientation with respect to electron beam. It can be detected mainly through the change of π* and σ* peaks at the carbon K-edge. This tendency is apparent in the π* peak, which also presents the sp^2 bonding information. Since graphene is a single-layer sp^2 hybrid structure, the intensity ratio between π* and σ* peaks for an ideal graphene is 1:3, which is the same for the π/σ bond ratio. Thus, graphene defects show different electron energy structure compared to pristine graphene. The defect structure of graphene greatly affects the electrical and chemical properties of graphene. Therefore, measuring the defect density of graphene is the key to improving and optimizing its quality for the advancing industrial applications.

1 Scope

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

– defect density (%, nm^2)

of single layer graphene films by

– electron energy loss spectroscopy (EELS in transmission electron microscopy (TEM)).

This document outlines a method for quantitative measurement of defects in graphene at the nanoscale.

The method specified in this document is applicable to single layer graphene acquired via chemical vapour deposition (CVD), roll-to-roll production and exfoliated graphene flakes to estimate the defect density.

In order to obtain reliable data, it is essential that the procedure is consistent for each specified condition from the preparation of the TEM specimen to its observation. It is essential to maintain the spatial resolution below 1 nm by alignment of the beam. The dispersion value, which covers the entire energy loss near edge structure (ELNES) region of the carbon-K edge and maintains the highest energy resolution corresponds to 0,1 eV/ch. Defects in graphene are determined by measuring the spectral differences between sp^2 hybridized and sp^2/sp^3 hybridized atoms, which are obtained by calculating the amplitude ratio of the π* and σ* orbital spectra.

2 Normative references

There are no normative references in this document.