IEC TS 62607-5-4

Edition 1.0 2022-12

TECHNICAL SPECIFICATION

colour inside

Nanomanufacturing – Key control characteristics –
Part 5-4: Energy band gap measurement of nanomaterials by electron energy loss spectroscopy (EELS)

IEC TS 62607-5-4:2022-12(en)

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INTERNATIONAL
ELECTROTECHNICAL
COMMISSION

ICS 07.030; 07.120 ISBN 978-2-8322-6199-6

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 5-4: Energy band gap measurement of nanomaterials by electron energy loss spectroscopy (EELS)

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IEC TS 62607-5-4 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
113/513/DTS	113/594/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- withdrawn,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

Electronic and electrical devices developed up to now have been fabricated by stacking a series of active and/or passive layers on a specific substrate. The current trend in developing such devices is the miniaturization of product size, whereas the basic structure of multilayers on a substrate has not changed. Accordingly geometrical scales in the inner structure of a device have been decreasing and some of the scales such as thickness of the layers have finally reached a few nanometres. One of the key control characteristics (KCCs) is the band gap of an active layer which enables the electron or hole transportation, excitation and emission of electrons, etc. to be controlled.

The band gap is referred to as an energy gap, which means a difference between an energy level in which electrons exist and an energy level in which electrons do not exist. Even though the band gap of a material is intrinsic, the band gap of a nanomaterial is an extrinsic property which represents its size-dependency. Therefore, the band gap of nanoscale materials needs to be measured locally, in situ or in vitro.

For the band gap measurement application to nanomaterials, a specific region of a nanometre-scale device or a single layer of the multi-layered structure, a transmission electron microscope (TEM), which has atomic-scale image resolution, and electron energy loss spectroscopy (EELS), which can measure energy loss of electrons, have in general been used.

In this document, a method of measuring the band gap energy at a specific location for a nanomaterial by using TEM and EELS is proposed.

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 5-4: Energy band gap measurement of nanomaterials by electron energy loss spectroscopy (EELS)

1 Scope

This part of IEC TS 62607 specifies the measuring method of the band gap energy of a nanomaterial using electron energy loss data of transmission electron microscope.

The method specified in this document is applicable to semiconducting and insulating nanomaterials to estimate the band gap.

The measurement to get reliable data is performed under the consistent conditions of TEM observation and specimen thickness. The applicable measurement range of band gap energy is more than 2 eV.

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
band gap energy
difference between the highest occupied energy level in which electrons exist and the lowest unoccupied energy level in which electrons do not exist

Note 1 to entry: Gap energy is defined in IEC 60050-113:2011, 113-06-16 as the smallest energy difference between two neighbouring allowed bands separated by a forbidden band.

Note 2 to entry: Refer to Figure 1.

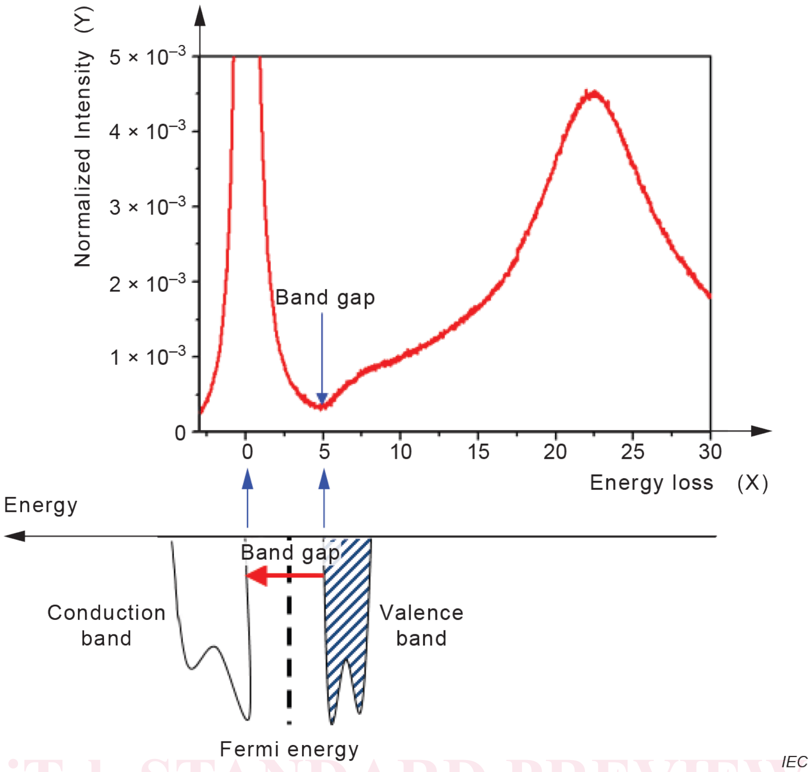


Figure 1 – Definition of band gap energy

3.2
transmission electron microscopy
TEM
method that produces magnified images or diffraction patterns of the sample by an electron beam which passes through the sample and interacts with it

[SOURCE: ISO 29301:2017, 3.34, modified – The term and definition have been modified to refer to the method rather than the instrument. In the definition, "specimen" has been replaced by "sample".]

3.3
scanning transmission electron microscopy
STEM
method that produces magnified images or diffraction patterns of the sample by a finely focused electron beam, scanned over the surface and which passes through the sample and interacts with it

[SOURCE: ISO/TS 10797:2012, 3.10, modified – The term and definition have been modified to refer to the method rather than the instrument.]

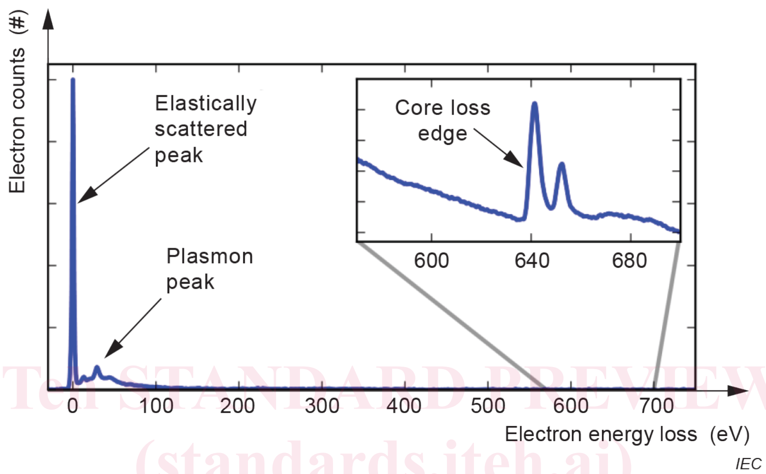
3.4
electron energy loss spectroscopy
method in which an electron spectrometer measures the energy spectrum of electrons from a nominally monoenergetic source emitted after inelastic interactions with the sample, often exhibiting peaks due to specific inelastic loss processes

[SOURCE: ISO/TS 80004-6:2013, 4.14]

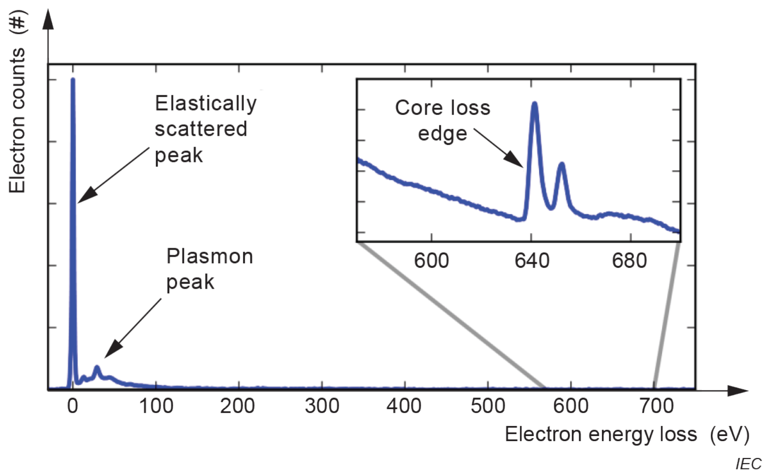
3.5
electron energy loss spectrum
energy spectrum of electrons from a nominally mono-energetic source emitted after inelastic interactions with the sample, often exhibiting peaks due to specific inelastic loss processes

[SOURCE: ISO/TS 10797:2012, 3.5]

3.6
elastically scattered peak
peak of electrons from the electron beam that are elastically scattered by the specimen and have no energy loss



3.7
plasmon peak
peak of electrons from the electron beam that have been inelastically scattered by the specimen resulting in energy transfer to plasmon modes in the specimen



3.8
spectrum imaging
imaging by the pixels which contains their spectra

3.9
determination coefficient
measure of the degree to which the estimated linear model is appropriate for the given data

Note 1 to entry: It refers to the percentage of the variable that can be explained by the applied model among the variation of the response variable.

Note 2 to entry: A typical symbol of the coefficient of determination is R^2.

4 Abbreviated terms

CCD	charge-coupled device
EELS	electron energy loss spectroscopy
FIB	focused-ion beam
PET	polyethylene terephthalate
STEM	scanning transmission electron microscopy
TEM	transmission electron microscopy

5 Environmental conditions

For a specimen to be transparent to electrons, it needs to be thin enough to transmit sufficient electrons such that enough intensity falls on the screen, CCD, or photographic plate to give an interpretable image in a reasonable time. Typically for 100-keV electrons, specimens of aluminium alloys up to ~1 µm would be thin, while steel would be thin up to about several hundred nanometres. However, it is an axiom in TEM that, almost invariably, thinner is better in collecting better interpretable image and specimen thickness should be approximately 100 nm or less. The target nanomaterials should be stable to electron beam.

To increase the mean free path of the electron gas interaction, a standard TEM is evacuated to low pressures, typically on the order of 10^{-4} Pa. High-voltage TEMs require ultra-high vacuums on the range of 10^{-7} Pa to 10^{-9} Pa to prevent the generation of an electric arc, particularly at the TEM cathode.

The storage temperature and humidity conditions of the sample are as follows.

a) Temperature: Target temperature ± 2 °C, recommended temperature 23 °C ± 2 °C.

b) Relative humidity: Target humidity range ± 10 %, recommended humidity 40 % ± 10 %.

6 Test sample

6.1 General

The test sample consists of semiconducting (or insulating) nanomaterials on a flexible substrate. In case of an insulating sample, the sample surface shall be coated with conducting materials in order to avoid a charging effect.

6.2 Size of test sample

The total size of specimens made using various specimen fabrication methods should be less than 3 mm in diameter. Care should be taken not to damage the specimen during specimen production, and the observation area should be made thin enough to transmit the electron beam and specimen thickness should be approximately 100 nm or less.

7 Testing method and test apparatus

7.1 General

The transmission electron microscope used in the measurement shall be capable of obtaining an image by scanning a small electron beam of a nanometre size and an electron energy loss spectrometer (EELS) capable of measuring electron energy.