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TECHNICAL SPECIFICATION

colour inside

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

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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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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.

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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.

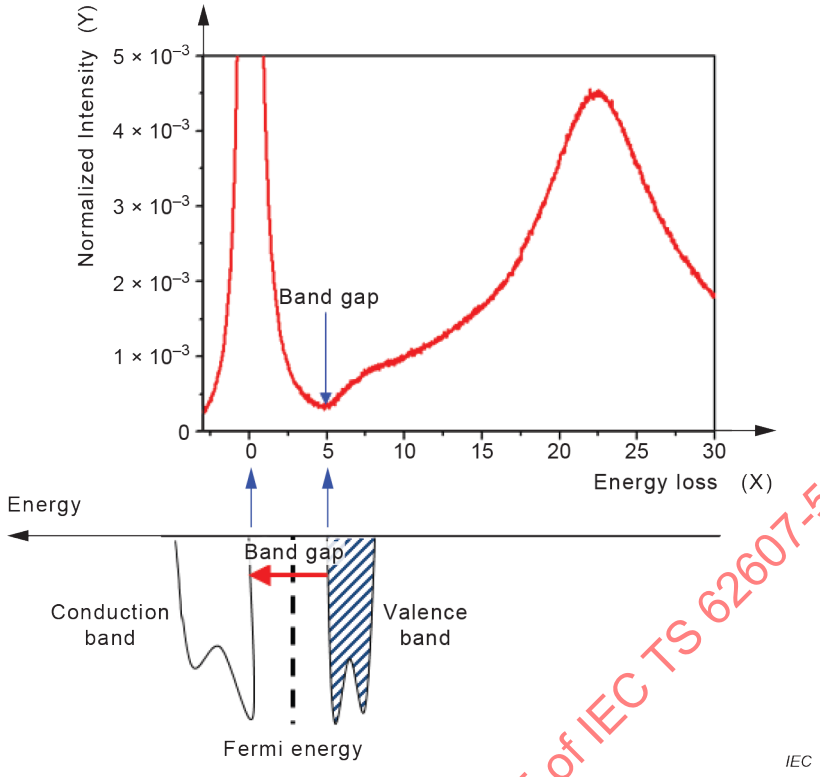


Figure 1 – Definition of band gap energy

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.]

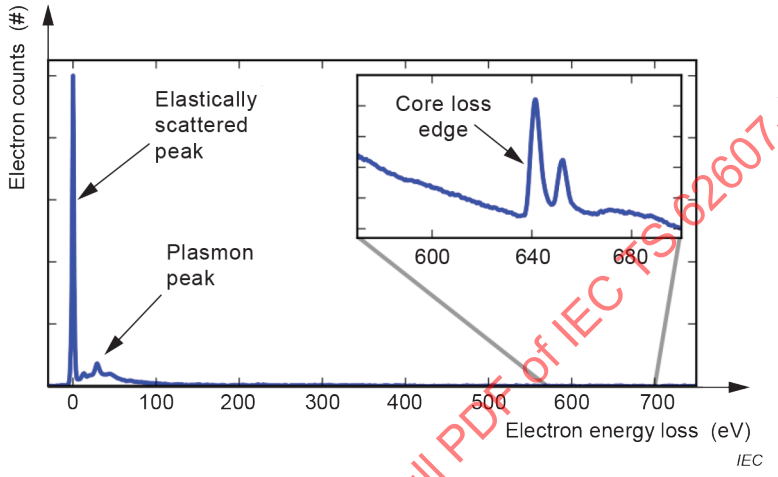
3.4
electron energy loss spectroscopy
method in which an electron spectrometer measures the energy spectrum of electrons from a nominally monoenergetic source emitted after inelastic interactions with the sample, often exhibiting peaks due to specific inelastic loss processes

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

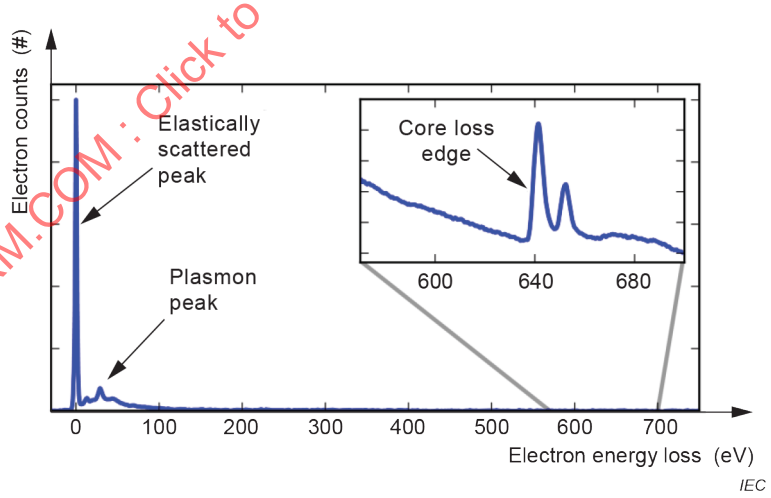
3.5
electron energy loss spectrum
energy spectrum of electrons from a nominally mono-energetic source emitted after inelastic interactions with the sample, often exhibiting peaks due to specific inelastic loss processes

[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.

In TEM, a material is exposed to a beam of electrons with a known, narrow range of kinetic energies. Some of the electrons will undergo inelastic scattering, which means that they lose energy (Figure 2). The higher accelerating voltage would be preferred for this measurement. However, there is an opposite aspect such as Cerenkov effect, which also originates from an inelastic scattering. The lower voltage is preferable in order to avoid the effect. In this experiment the accelerating voltage ranges from 120 keV to 200 keV.

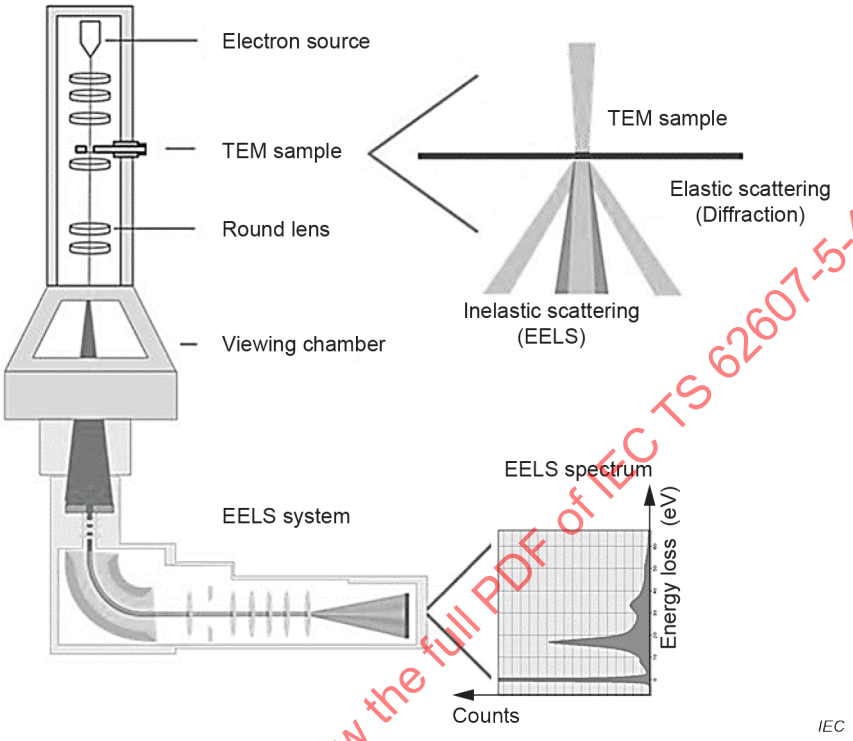


Figure 2 – Schematic showing TEM and EELS system

7.2 Detector

There are two types of detector used in EELS to measure kinetic energy of the energy-loss electrons. The first type, known as magnetic sector prism, is capable of providing energy measurements and microscopic images simultaneously. However, this type of detector is less accurate in detecting energy. The second type of detector, known as electrostatic sector, is mainly used for detecting more precise energy differences up to 10 meV. However, this type of detector is not capable of revealing topographic images simultaneously. Since this document aims at detecting the band gap at the target area, the use of magnetic sector prism is preferred (Figure 3). The EELS spectrum includes some information of band gap energy, plasmon energy and inner shell ionizations.

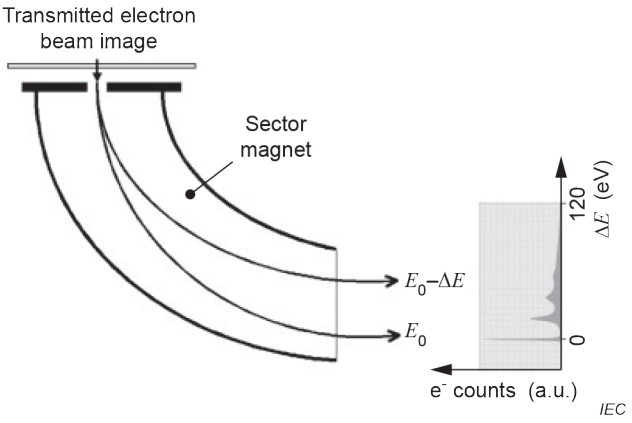


Figure 3 – Apparatus for the measurement of band gap energy by using electron energy loss spectroscopy

7.3 Test procedure

7.3.1 Transmission electron microscope alignment

a) Increase the acceleration voltage to a desired value.

b) Insert the specimen into the holder and insert the holder into the transmission electron microscope.

c) Align the electron beam according to the equipment manual.

d) Place the electron beam at the desired site.

e) Focus the measurement position and obtain the image.

7.3.2 Scanning transmission electron microscope alignment

a) Complete the transmission electron microscope mode alignment according to the equipment manual.

b) Switch to scanning transmission electron microscope mode from transmission electron microscope mode.

c) Find the amorphous region and align the electron beam.

d) Lower the magnification to find the desired sample position, select the desired magnification, and adjust the focus.

7.4 Measurement

The test procedure is listed as follows:

a) Locate the electron beam to a blank area in which there is no sample layer under microscopic mode.

b) Switch to the electron energy loss mode and align the electron energy loss spectrometer to the detected area.

c) Place the elastically scattered peak at zero energy loss.

d) Adjust the half-width of the elastically scattered peak to 0,8 eV or less.

e) Adjust the energy range to include the zero-loss peak and the plasmon peak. (The range of 40 eV to 50 eV is recommended.)

f) Position the electron beam at the target area. A thinner layer is preferred for the target area in order to reduce multiple scattering effects.

g) Obtain a microscopic image of the target area and set the spectrum image area.

h) Set the 2D array and pixel time of the spectral image.

In general, the scan size is greater than 10 × 10 and pixel time is determined to the extent that the specimens are not damaged. To ensure statistical accuracy, maximize the amount of data (Figure 4).

i) Set the spatial drift correction and obtain the spectral image.

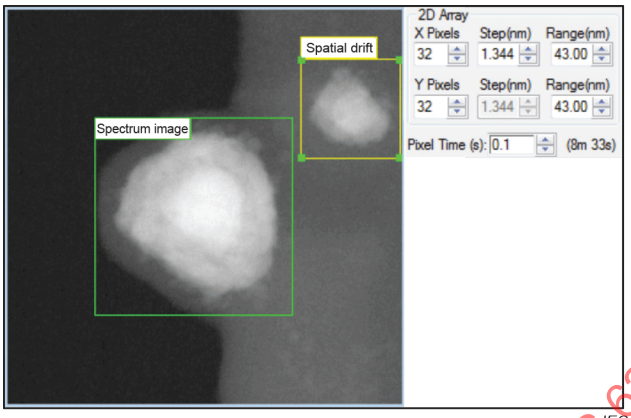


Figure 4 – Setting for spectrum imaging

8 Data analysis

8.1 Band gap determination

a) Align the position where the elastically scattered peak of spectral image data shows the maximum value to zero energy loss.

NOTE 1 This condition is shown in Figure 5.

b) When normalized based on the elastically scattered peak, the intensity value of the band gap measurement area should be 10^{-3} or less. (The value of A shall be smaller than the band gap.)

NOTE 2 This condition is shown in Figure 5.

c) Base line 1 is determined based on the point of the lowest intensity.

NOTE 3 This condition is shown in Figure 6.

d) Determine the straight line 2 so that the linear coefficient of the nearest to the lowest point of the spectrum and the coefficient of determination of the straight line 2 are 0,95 or more.

NOTE 4 This condition is shown in Figure 6.

e) Find the band gap by reading the contact points of the base line 1 and the line 2.

NOTE 5 This condition is shown in Figure 6.

f) Plasmon energy is read at the position where the intensity of the plasmon peak is maximum.

NOTE 6 This condition is shown in Figure 6.

NOTE 7 If the conditions outlined in the procedure are not satisfied by the measured spectra, band gap determination cannot be made.

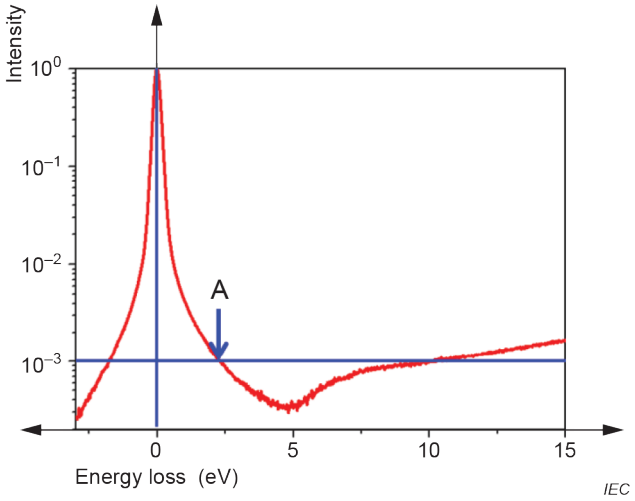


Figure 5 – Normalized spectrum of log scale

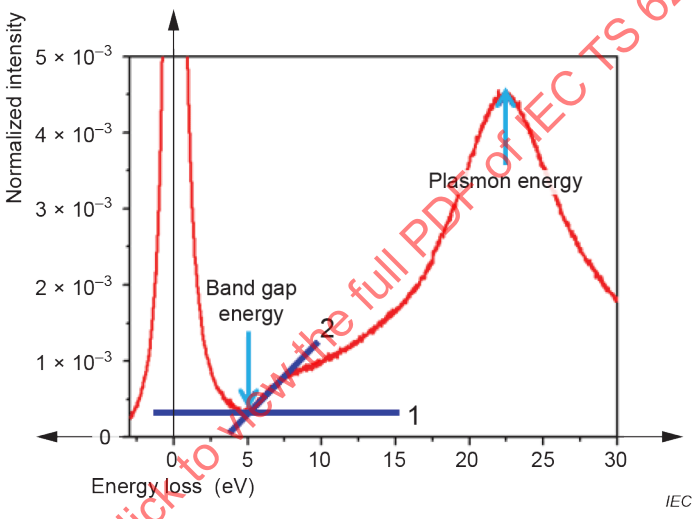


Figure 6 – Band gap measurement method using linear fitting

Examples of band gap measurement on WO_3 on PEF and SiN_x multilayers are exhibited in Annex A and Annex B, respectively.

8.2 Report of the results

The report shall include the following items.

Band gap energy measurements are expressed in electron-volts (eV) and rounded to the first decimal place.

a) Information on test equipment:
   1) model name of test apparatus and spectroscope;
   2) acceleration voltage of the test apparatus;
   3) extraction voltage of the test apparatus.
b) Information on prepared specimen:
   1) composition of specimen;
   2) preparation method and form of specimen;
   3) specimen identification (including film thickness, sample size).

c) Experimental parameters:
   1) energy resolution of the spectroscope (full width at half maximum);
   2) magnification of the used STEM;
   3) scan size;
   4) pixel time.
d) Recording the measurement result in unit [eV]:
   1) band gap energy;
   2) plasmon energy.

Annex A
(informative)

Band gap measurement of WO_3 thin film on PET

It is essential to confirm the thickness of a sample in order to avoid the error sources originated from inelastic scattering during the measurement. The inelastic scattering length is dependent on an accelerating voltage of the electron beam. The typical value is more than 100 nm at the accelerating voltage more than 120 keV. Hence this document is valid on the thin layer less than 100 nm. In case of a sample with more than 100 nm thickness, additional data treatment processes need to be carried out. The thickness of the sample was confirmed by FIB.

The band gap of the insulating layer WO_3 on a PET substrate, which can be used as a flexible substrate in nanoelectronics, is measured with a transmission electron microscope to obtain a similar value of 3,7 eV. Refer to Figure A.1.

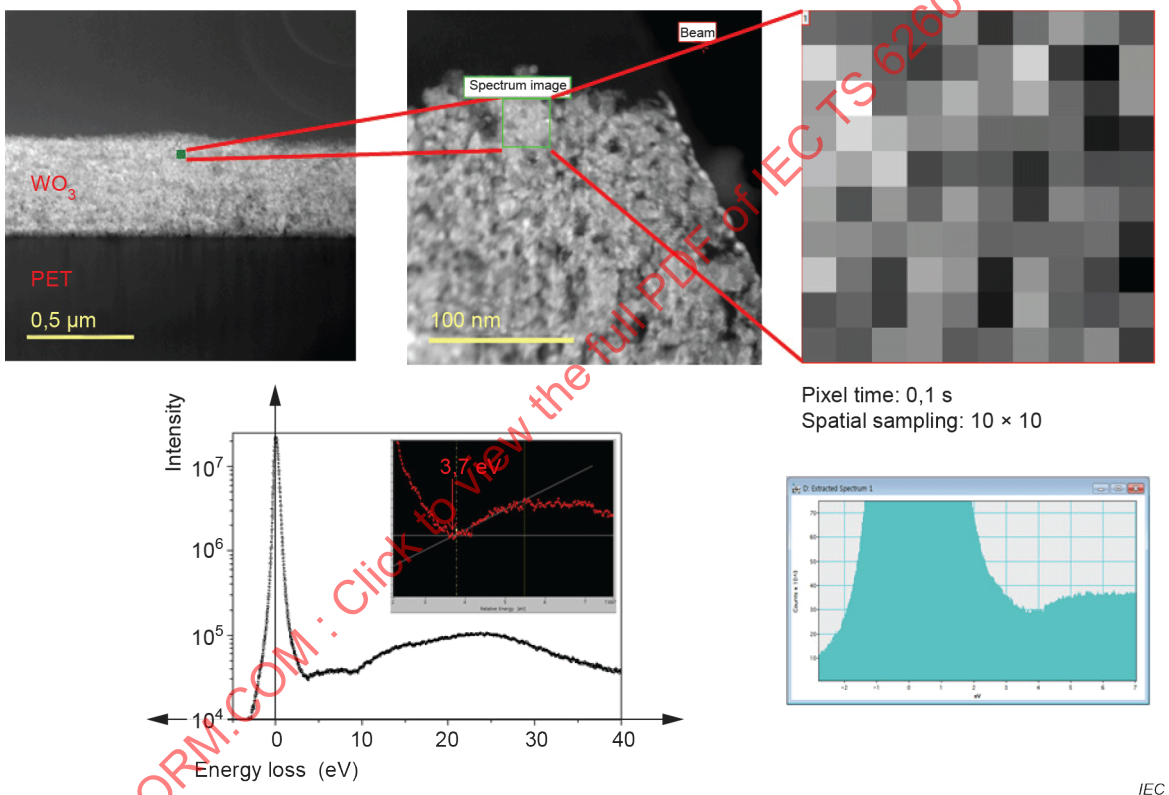


Figure A.1 – Measurement of band gap of WO_3 by STEM-EELS

Table A.1 presents the example of a report for the above sample.