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Microbeam analysis — Analytical electron microscopy — Method for the determination of interface position in the cross-sectional image of the layered materials

Analyse par microfaisceaux — Microscopie électronique analytique — Méthode de détermination de la position d'interface dans l'image de coupe transversale des matériaux en couches

FDIS stage

ISO ~~Copyright Office~~copyright office
CP ~~56 -~~ 401 • Ch. de Blandonnet 8
CH-~~1211~~1214 Vernier, Geneva ~~20~~
Phone: + 41 22 749 01 11

~~Fax: + 41 22 749 09 47~~

~~Email~~E-mail: copyright@iso.org
Website: ~~www.iso.org~~www.iso.org

Published in Switzerland~~.~~

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Foreword

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This document was prepared by Technical Committee ISO/TC 202, *Microbeam analysis*, Subcommittee SC 3, A*nalytical electron microscopy*.

This second edition cancels and replaces the first edition (ISO 20263:2017), which has been technically revised.

The main changes are as follows:

- ~~the foreword has been revised;~~
- ~~the~~ introduction has been revised;
- ~~the normative references~~terms and sources in ~~the subclauses~~Clause 3.~~1.4, 3.1.11, 3.1.16, 3.1.21, 7.2~~ have been revised;
- subclauses 5.2.2, 5.2.3, 6.1, 6.2.1, 6.7, 7.1, 7.2, A.4.1, B.2.3 and B.2.4 have been ~~updated~~revised;
- ~~the terms and definitions of terminological entry 3.1.1 and 3.1.23 have been deleted;~~
- ~~the terms and definitions of terminological entry 3.1.4 has been added;~~
- ~~the terms and definitions of terminological entry 3.1.19 has been revised.~~
- ~~the subclauses 3.1, 3.2, 5.2.2, 5.2.3, 6.1, 6.2.1, 6.7, 7.1, 7.2, A.4.1, B.2.3 and B.2.4 has been revised;~~
- ~~the~~ figures ~~3, 9, 10, 11, 12, A2, A3, A4, A5, A7, A8, A9, A10, A12, A16, A17, A18, B2, B3, B4, B6, B7, B8 and C1~~ have been revised.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at ~~www.iso.org/members.html~~www.iso.org/members.html.

Introduction

Multi-layered materials are widely used in the production of semiconductor devices, various kinds of sensors, coating films for optical element, new functional materials, etc. One of the factors used to determine the characteristics of multi-layered materials is the layer thickness, for evaluation of products and verification of the production process. In practice, measuring the total thickness and/or the thickness of each layer and checking the uniformity of thickness and/or flatness of the interface are often done using recorded images of the materials. Evaluations can be made from the cross-sectional TEM/STEM images by accurately determining the averaged interface position between two different layered materials.

In relation to the determination of the interface position in the HR atomic imaging, analysis by the multi-slice simulation (MSS) method can be applied for the target measurement, if the atomic structural models can be constructed. However, in real materials, there are a lot of cases when they cannot, such as:

— ~~—~~the interface between amorphous layers, or layers of amorphous substance and crystal;

— ~~—~~the interface recorded in low-resolution image in which the atomic columns cannot be identified:

  — ~~1)~~for very thick single-layered material;

  — ~~2)~~for thick multi-layered material.

This document ~~relates~~gives the method to determine the averaged interface position~~,~~ using a differential processing of the accumulated intensity profile ~~getting~~obtained from the ROI set in the cross-sectional TEM/STEM image of the multi-layered ~~materials~~material. The thickness of the layer that can be applied ranges from a few nanometers to a few micrometers. Thus, this document is not intended for the determination of the simulated position of the layer interface analysed by the MSS method.

Microbeam analysis — Analytical electron microscopy — Method for the determination of interface position in the cross-sectional image of the layered materials

1 Scope

This document specifies a procedure for the determination of the averaged interface position between two different layered materials recorded in the cross-sectional image of the multi-layered materials. It is not intended to determinematerial. This document does not apply for determining the simulated interface of the multi-layered materials expected through the multi-slice simulation (MSS) method.

This document is applicable to the cross-sectional images of the multi-layered materials recorded using a transmission electron microscope (TEM) or a scanning transmission electron microscope (STEM) and cross-sectional elemental mapping images using an energy dispersive X-ray spectrometer (EDS) or an electron energy loss spectrometer (EELS). This document is also applicable to the digitized imageimages recorded on an image sensor built into a digital camera, a digital memory set in the PC or an imaging plate and, where the digitalized image converted fromis obtained by converting an analogue image recorded on the photographic film byusing an image scanner.

2 Normative references

There are no normative references in this document.

3 Terms, definitions and abbreviated terms

3.1 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obphttps://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/https://www.electropedia.org/

3.1.1
cross-sectional image
TEM/STEM image of the multi-layered materials along a plane perpendicular to the stacking direction

3.1.2
differential processing
calculation of the difference between the values of adjacent pixel data in the intensity profile

3.1.3
digital camera
device that detects the image using a chip-arrayed *image sensor* ~~(3.1.12),~~(3.1.12), such as a charge-coupled device (CCD) or complementary metal-oxide semiconductor (CMOS), which converts a visual image to an electric signal

[SOURCE: ISO 29301:2023, 3.7]

3.1.4
dot pitch
distance between adjacent pixels in pixel-based devices

3.1.5
elemental mapping image
image produced by the selected signal which is attributed to a particular element, from the EDS/EELS spectrum

3.1.6
FIB thinning
site-specific thinning technique by using focused ion beam to thin a particular region of the specimen

3.1.7
filtering mask
mask to define the cut–off frequency in the reciprocal space

3.1.8
fast Fourier transformation
FFT
efficient algorithm to compute the discrete Fourier transform

[SOURCE: ISO 15932:2013, 5.4.1.1]

3.1.9
inverse fast Fourier transformation
IFFT
efficient algorithm to compute the inverse of the discrete Fourier transform

[SOURCE: ISO 15932:2013, 5.4.1.2]

3.1.10
image file format
format for saving an image as a computer file according to a predetermined rule

3.1.11
image scanner
device that converts an analogue image into a digitized image with the desired resolution

EXAMPLE There are mainly two different types of scanners: flatbed type and drum type.

[SOURCE: ISO 29301:2023, 3.17, modified — example has been added.]

3.1.12
image sensor
device, such as a charge-coupled device (CCD) array or complementary metal-oxide semiconductor (CMOS) sensor, which converts visual image information to an electric signal, built-in *digital camera* (3.1.3)(3.1.3) or other imaging devices

3.1.13
intensity profile
signal intensity distribution along a line specified in the image

3.1.14
interface
boundary surface at the junction of two different layers of materials recorded in the *cross-sectional image* (3.1.1)(3.1.1) of the multi-layered materials

3.1.15
ion-milling
thinning technique of sputtering the specimen with an inert gas

[SOURCE: ISO 15932:2013, 4.1]

3.1.16
imaging plate
IP
electron image detector consisting of a film with a thin active layer embedded with specifically designed phosphors

[SOURCE: ISO 29301:2023, 3.16, ISO 29301:2010, 3.23]

3.1.17
low pass filter
filter to pass signals of frequencies lower than the cut-off frequency

3.1.18
moving average
calculation for averaging the selected dataset which is picked out from equal number of dataset on either side of a central data

3.1.19
multi-slice simulation
MSS
computer simulation method of high-resolution TEM images, which treats electrons as incoming waves and treats the interactions with matter as occurring on multiple successive single slices of the specimen

[SOURCE: ISO 15932:2013, 6.4.1, modified — “algorithm for the simulation” has been replaced by “computer simulation method”.]

3.1.20
multi-layered material
laminated material which is fabricated by alternating layers of at least two kinds of materials on the substrate

3.1.21
photographic film
sheet or a roll of thin plastic coated by photographic emulsion for recording an image

[SOURCE: ISO 29301:2023, 3.23]

3.1.22
pixel
smallest unit element that makes up the digital image

3.1.23
region of interest
ROI
sub-dataset picked out from the entire dataset for a specific purpose

3.1.24
ultra-microtome
thin sectioning instrument to prepare the specimen thin enough for TEM observation by using glass or diamond knives

3.1.25
zone axis
crystallographic direction, designated [*u v w*], defined by the intersection of a number of crystal planes (h_1,k_1,l_1…….h_i,k_i,l_i) such that all of the planes satisfy the so-called Weiss zone law; $hu + kv + lw = 0$

[SOURCE: ISO 29301:20102023, 3.3836]

3.2 Abbreviated terms

AEM	Analytical electron microscope/microscopy
CCD	Charge coupled device
CMOS	Complementary Metal Oxide Semiconductormetal oxide semiconductor
CRT	Cathode ray tube
EDS/EDX	Energy dispersive X-ray spectrometer/spectroscopy Although "EDX" and "EDS" are interchangeable, this document uses "EDS".
EELS	Electron energy loss spectrometer/spectroscopy
FFT	Fast Fourier transformation
FIB	Focused ion beam
HREM	High-resolution transmission electron microscope/microscopy
IFFT	Inverse fast Fourier transformation
MSS	Multi-slice simulation
ROI	Region of interest
STEM	Scanning transmission electron microscope/microscopy
TEM	Transmission electron microscope/microscopy