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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

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Contents

Page

Foreword iv
Introduction v
1 Scope 1
2 Normative references 1
3 Terms, definitions and abbreviated terms 1
  3.1 Terms and definitions 1
  3.2 Abbreviated terms 4
4 Specimen preparation for cross-sectional imaging 4
  4.1 General 4
  4.2 Requirements for the cross-sectional specimen 5
5 Determination of an interface position 6
  5.1 General 6
  5.2 Preliminary considerations 6
    5.2.1 Ideal model of an interface 6
    5.2.2 More realistic model of an interface 6
    5.2.3 Dealing with intensity fluctuations in the image 8
6 Detailed procedure for determining the position of the interface 8
  6.1 General 8
  6.2 Preparing cross-sectional TEM/STEM image 10
    6.2.1 Preparing digitized Image 10
    6.2.2 Displaying the digitized image 11
  6.3 Setting the ROI 11
    6.3.1 General 11
    6.3.2 Classification of image 11
    6.3.3 Procedure of setting the ROI 12
  6.4 Acquisition of the averaged intensity profile 17
  6.5 Moving-averaged processing 19
  6.6 Differential processing 20
  6.7 Final location of the interface 21
7 Uncertainty 22
  7.1 Uncertainty accumulating from each step of the procedure 22
  7.2 Uncertainty of measurement result on image analysis 22
Annex A (informative) **Examples of processing the real TEM/STEM images for three image types** 24
Annex B (informative) **Two main applications for this method** 36
Annex C (informative) **Calibration of scale unit: Pixel size calibration** 43
Bibliography 45

Foreword

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

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, as follows:

— 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) very thick single-layered material, 2) thick multi-layered material.

This document relates the method to determine the averaged interface position, using a differential processing of the accumulated intensity profile getting from the ROI set in the cross-sectional TEM/STEM image of the multi-layered materials. 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 averaged interface position between two different layered materials recorded in the cross-sectional image of the multi-layered materials. It is not intended to determine 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 by using a transmission electron microscope (TEM) or a scanning transmission electron microscope (STEM) and the cross-sectional elemental mapping images by using an energy dispersive X-ray spectrometer (EDS) or an electron energy loss spectrometer (EELS). This document is also applicable to the digitized image recorded on an image sensor built into a digital camera, a digital memory set in the PC or an imaging plate and the digitalized image converted from an analogue image recorded on the photographic film by 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 terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at http://www.electropedia.org/

3.1.1
atomic column image
TEM/STEM image recorded at atomic-resolution from a specimen along a high-symmetry crystalline orientation

Note 1 to entry: Crystalline orientation is the direction of crystal which is represented by Miller indices. During TEM imaging, it is often useful to have a crystalline specimen aligned so that a specific (low index) *zone axis* (3.1.26) is parallel, or near parallel, to the beam direction (optical axis).

3.1.2
cross-sectional image
TEM/STEM image of the multi-layered materials along a plane perpendicular to the stacking direction

3.1.3
differential processing
calculation of the difference between the values of adjacent pixel data in the intensity profile

3.1.4
digital camera
device that detects the image using a chip-arrayed *image sensor* (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 29301:2010, 3.8]

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 using abrasion by focused field-emitted gallium atoms accelerated to an energy of 1 keV to 40 keV 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

Note 1 to entry: There are mainly two different types of scanners: flatbed type and drum type.

[SOURCE: ISO 29301:2010, 3.18, modified — the 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* (3.1.4) 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* (3.1.2) 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 29301:2010, 3.17]

[SOURCE: 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
multi-slice method
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 29301:2010, 3.26]

3.1.22
pixel
smallest unit element that makes up the digital image

3.1.23
pixel-resolution
number of imaging *pixels* (3.1.22) per unit distance of the detector

Note 1 to entry: Typical unit of measurement is “pixels per unit distance”, e.g. dots per inch (dpi).

[SOURCE: ISO 29301:2010, 3.27, modified — Note 1 to entry has been added.]

3.1.24
region of interest
ROI
sub-dataset picked out from the entire dataset for a specific purpose

3.1.25

ultra-microtome

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

3.1.26

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; $hu + kv + lx = 0$

[SOURCE: ISO 29301:2010, 3.38]

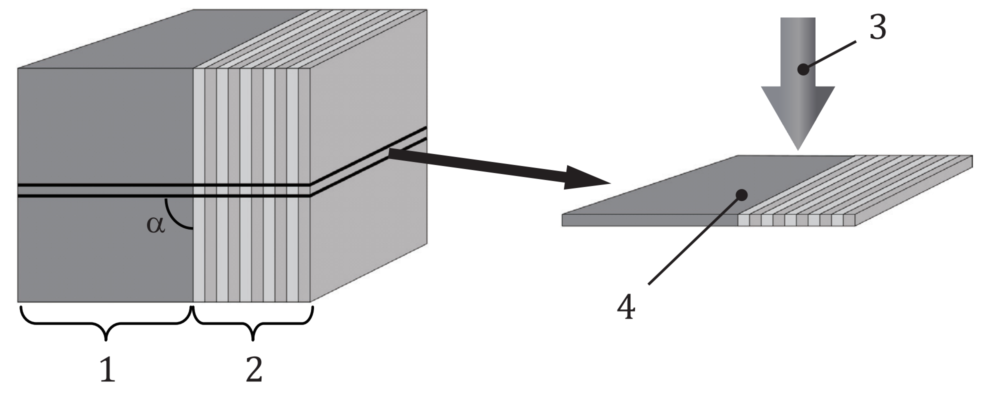
3.2 Abbreviated terms

AEM	Analytical electron microscope/microscopy
CCD	Charge coupled device
CRT	Cathode ray tube
EDS	Energy dispersive X-ray spectrometer/spectroscopy
EDX	Energy dispersive X-ray spectrometer/spectroscopy
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

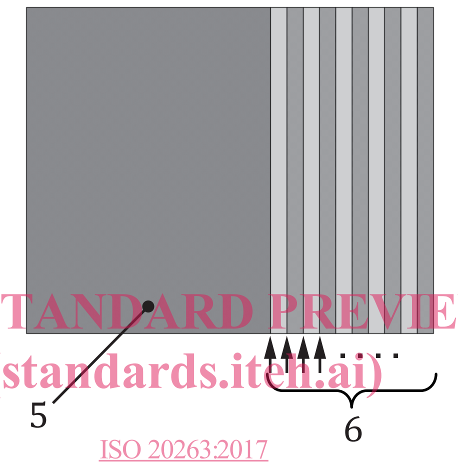
4 Specimen preparation for cross-sectional imaging

4.1 General

To determine the interface potion of the multi-layered materials stacked on a substrate, the specimen observed by TEM/STEM shall be cut into a cross-sectional thin slice perpendicular to the stacking direction of the multi-layered thin film, using the techniques of ultra-microtome, ion-milling, FIB thinning, chemical etching and so on. In order to keep the thickness information of the layered materials with an accuracy of 1 %, cut out angle α [shown in Figure 1, a)] shall be 90 ± 6 degrees.



a) Multi-layered specimen **b) TEM/STEM observation**



c) Cross-sectional imaging

Key

1	substrate
2	multi-layered materials
3	direction of electron beam
4	thin slice of the specimen
5	cross-sectional TEM/STEM image
6	arrows indicate interface positions

Figure 1 — Specimen preparation for cross-sectional imaging

4.2 Requirements for the cross-sectional specimen

Ensure that the specimen

- provides a good contrast and clear interface for the multi-layered materials in the TEM/STEM/elemental mapping image,
- can be cleaned to remove contamination without causing mechanical/electrical damage or distortion,
- has a smooth surface on both sides and identical thickness, at least within the area used for the determination process of interface position,
- is aligned to a low-index zone axis along the electron optical axis, if the specimen region is a single crystal.

5 Determination of an interface position

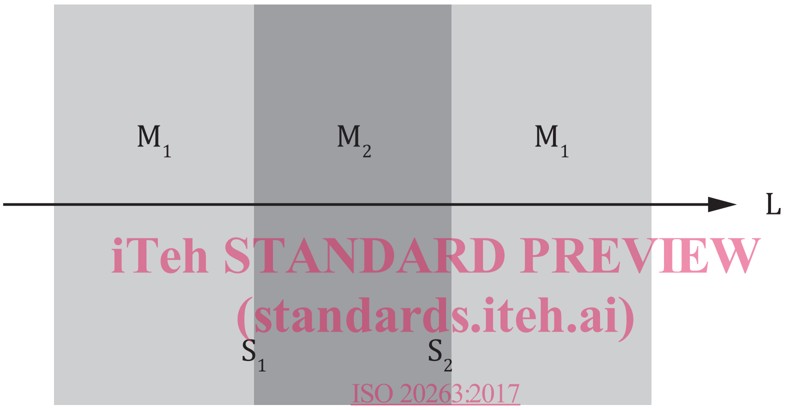
5.1 General

It is important to determine the position of an interface in a layered material from its cross-sectional TEM/STEM/elemental mapping image, objectively and uniquely. In this clause, the main scheme for the determination of the interface position, as prescribed by this document, is explained.

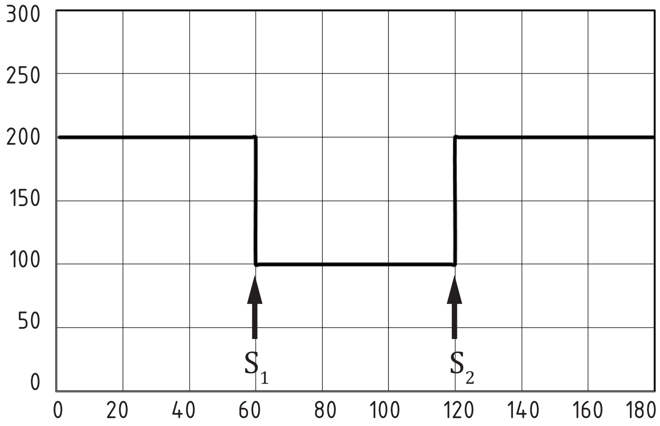
5.2 Preliminary considerations

5.2.1 Ideal model of an interface

Ideally, the interface between two kinds of materials, M_1 and M_2, show straight edge [Figure 2 a)]. In this case, it is easy to find the interface positions (S_1 and S_2) uniquely from the intensity profile [see Figure 2 b)] along a line (L) perpendicular to the interface.



a) Ideal interface (S_1 and S_2) model between two kinds of layers, M_1 and M_2

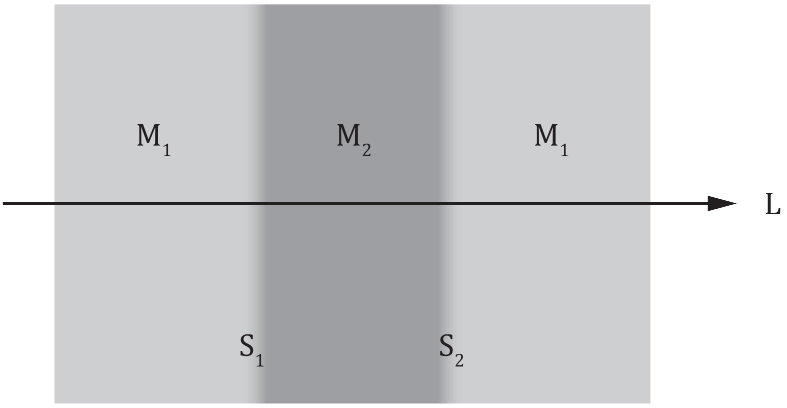


b) Intensity profile along an arrow line, L, in a), perpendicular to the interfaces (S_1 and S_2)

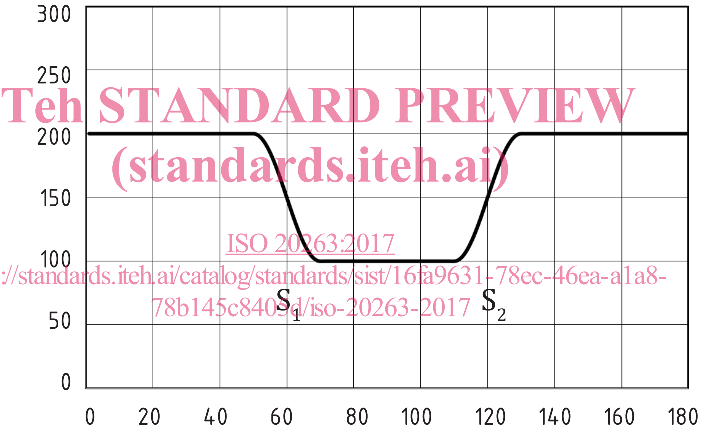
Figure 2 — Ideal interface model

5.2.2 More realistic model of an interface

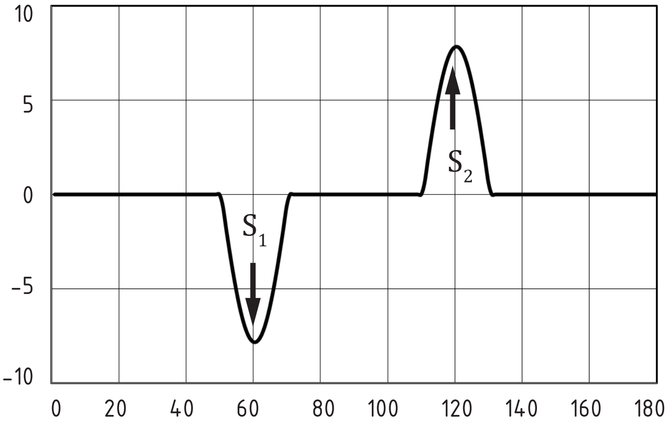
However, in general, the interface will not be in a straight line. It is a region with gradated intensity distribution between layers M_1 and M_2 [see Figure 3 a)]. In this case, it is not easy to find the accurate interface position from its intensity profile which is normally s-shaped [see Figure 3 b)]. This document defines the interface position at the steepest tilt angle in the slope. Differential processing of the intensity profile is the most suitable method to determine the interface position as defined above. Figure 3 c) shows the differential curve of the intensity profile on in Figure 3 b). Pixel positions on the x-axis corresponding to the minimal value and maximal value in the curve show the interface positions (S_1 and S_2) on both sides of the layer M_2.



a) Realistic interface (S_1 and S_2) model between two kinds of layers, M_1 and M_2



b) Intensity profile along an arrow line, L, in a), perpendicular to the interfaces (S_1 and S_2)



c) Differential curve of intensity profile

NOTE Positions of minimal and maximal value correspond to the interfaces (S_1 and S_2) defined in this document.

Figure 3 — Realistic interface model

5.2.3 Dealing with intensity fluctuations in the image

Unlike models described in 5.2.1 and 5.2.2, the actual cross-sectional TEM/STEM/elemental mapping image has a domain-like intensity fluctuation, background noise and sometimes (in the high-resolution images) periodic modulation of the intensity due to atomic column structures. Because of this non-uniformity in the intensity in the image, follow the steps a) to f) sequentially for obtaining the desired smooth intensity profile with a plateau and well-defined slope.

NOTE 1 Details of the actual procedure are described in Clause 6.

a) Prepare the cross-sectional TEM/STEM/elemental mapping digital image.

Set the direction of the interface parallel to the y-axis of the monitor screen.

b) Set the ROI area in the image.

c) Average the intensity line profile, perpendicular to the interface (parallel to the x-axis of the monitor screen) along the interface (parallel to the y-axis of the monitor screen) in the ROI area.

d) Apply "moving-average" processing to the averaged intensity profile produced by the previous step, c). This will remove small noise from the boundary region contributing to the slope of the interface.

e) Apply differential processing to the resulting intensity profile obtained in d) by the moving-average process.

f) Determine the interface position as the pixel coordinate on the x-axis which either corresponds to the maximal or minimal value in the differential curve.

NOTE 2 More details follow in Clause 6.

6 Detailed procedure for determining the position of the interface

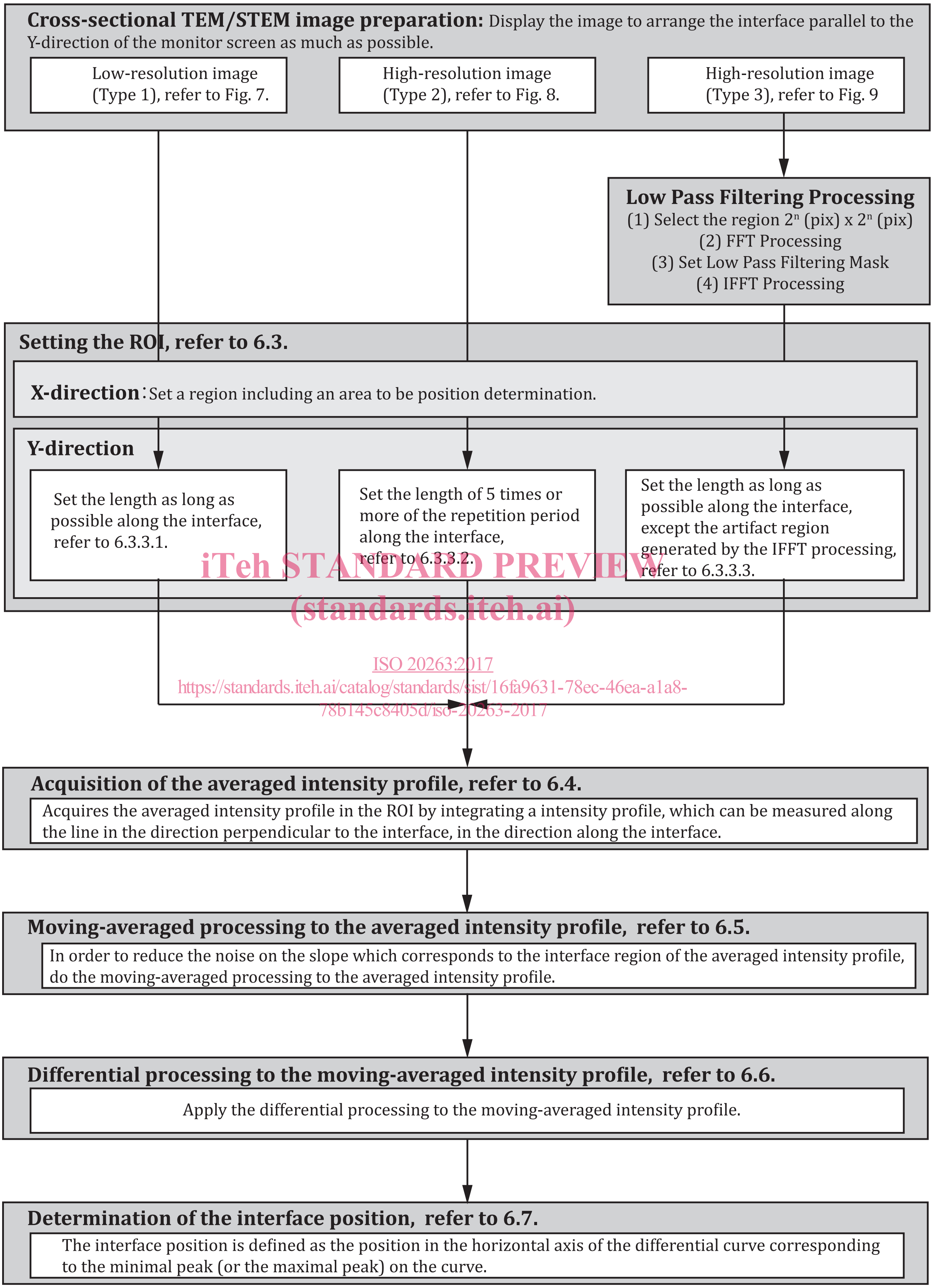
6.1 General

As described in the previous clause, the interface position can be determined through the differential processing of the intensity profile in the ROI.

In the differential processing, a noise component existing in the intensity profile becomes an obstacle to find the correct interface position. Therefore, it is necessary to remove the noise components in advance through the average processing and the moving-averaged processing.

Also, in atomic resolution image with an atomic column arrangement along the interface, the oscillated component depends on the atomic column cannot be eliminated even in the averaged intensity profile. This is an obstacle to the extraction of the correct interface position by differential processing. Therefore, for such image, pre-processing for obscuring the atomic column structure is essential through the processing of FFT/low pass filtering/IFFT.

Figure 4 shows a flow chart of the interface position determination procedure described in 5.2.3. In this clause, details of each procedure will be described.



Cross-sectional TEM/STEM image preparation: Display the image to arrange the interface parallel to the Y-direction of the monitor screen as much as possible.

Low-resolution image (Type 1), refer to Fig. 7.

High-resolution image (Type 2), refer to Fig. 8.

High-resolution image (Type 3), refer to Fig. 9

Low Pass Filtering Processing

(1) Select the region 2^n (pix) x 2^n (pix)
(2) FFT Processing
(3) Set Low Pass Filtering Mask
(4) IFFT Processing

Setting the ROI, refer to 6.3.

X-direction: Set a region including an area to be position determination.

Y-direction

Set the length as long as possible along the interface, refer to 6.3.3.1.

Set the length of 5 times or more of the repetition period along the interface, refer to 6.3.3.2.

Set the length as long as possible along the interface, except the artifact region generated by the IFFT processing, refer to 6.3.3.3.

Acquisition of the averaged intensity profile, refer to 6.4.

Acquires the averaged intensity profile in the ROI by integrating a intensity profile, which can be measured along the line in the direction perpendicular to the interface, in the direction along the interface.

Moving-averaged processing to the averaged intensity profile, refer to 6.5.

In order to reduce the noise on the slope which corresponds to the interface region of the averaged intensity profile, do the moving-averaged processing to the averaged intensity profile.

Differential processing to the moving-averaged intensity profile, refer to 6.6.

Apply the differential processing to the moving-averaged intensity profile.

Determination of the interface position, refer to 6.7.

The interface position is defined as the position in the horizontal axis of the differential curve corresponding to the minimal peak (or the maximal peak) on the curve.

Figure 4 — Flow chart of interface position determination procedure