BS ISO 20263:2024

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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

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This British Standard is the UK implementation of ISO 20263:2024. It supersedes BS ISO 20263:2017, which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee CII/9, Microbeam analysis.

A list of organizations represented on this committee can be obtained on request to its committee manager.

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International Standard

# ISO 20263

Second edition
2024-11

# 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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# 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:

— introduction has been revised;

— terms and sources in Clause 3 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 revised;

— figures have been revised.

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# 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:

  — for very thick single-layered material;

  — for thick multi-layered material.

This document gives the method to determine the averaged interface position using a differential processing of the accumulated intensity profile obtained from the ROI set in the cross-sectional TEM/STEM image of the multi-layered 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.

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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

## 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 material. 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 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 digitized images recorded on an image sensor built into a digital camera, a digital memory set in the PC or an imaging plate, where the digitalized image is obtained by converting an analogue image recorded on photographic film using 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/obp

— IEC Electropedia: available at 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), 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

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**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) 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) 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]

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**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]

**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 29301:2023, 3.36]

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### 3.2 Abbreviated terms

| | |
|---|---|
| AEM | Analytical electron microscope/microscopy |
| CCD | Charge coupled device |
| CMOS | Complementary metal oxide semiconductor |
| CRT | Cathode ray tube |
| EDS/EDX | Energy dispersive X-ray spectrometer/spectroscopy<br>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 |

## 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 %, the cut out angle $\alpha$ [shown in Figure 1, a)] shall be 90° ± 6°.

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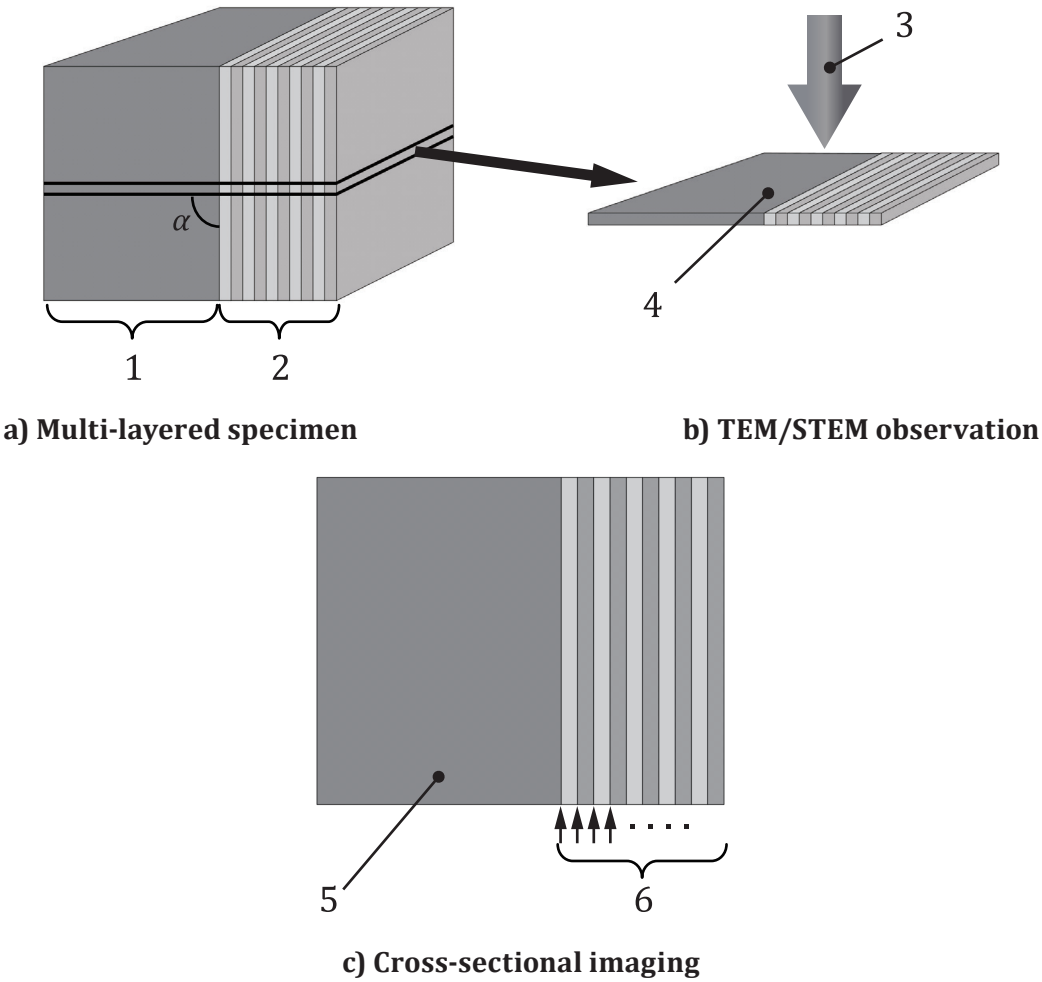


**a) Multi-layered specimen** **b) TEM/STEM observation**

**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 and sharp contrast and clear and straight 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.

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# 5 Determination of an interface position

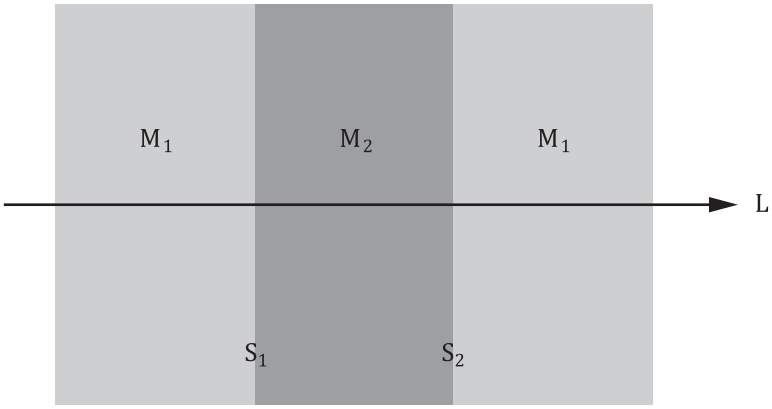
## 5.1 General

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 Clause 6, the main scheme for the determination of the interface position, as prescribed by this document, is explained.

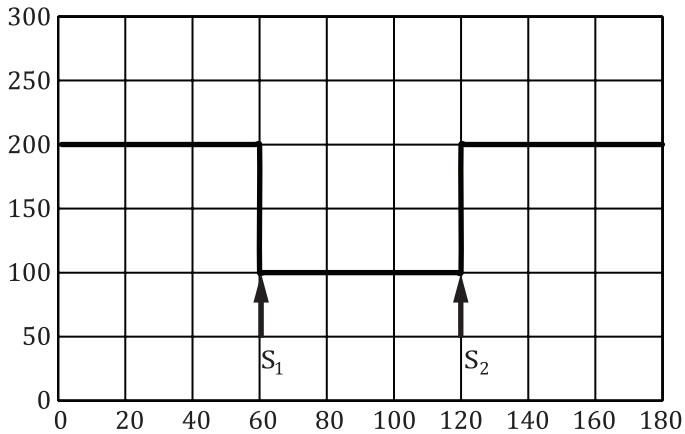
## 5.2 Preliminary considerations

### 5.2.1 Ideal model of an interface

Ideally, the interface between two kinds of materials, $M_1$ and $M_2$, show straight edge [see 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

In general, the interface will not be in a straight line. It typically appears as a region with a 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

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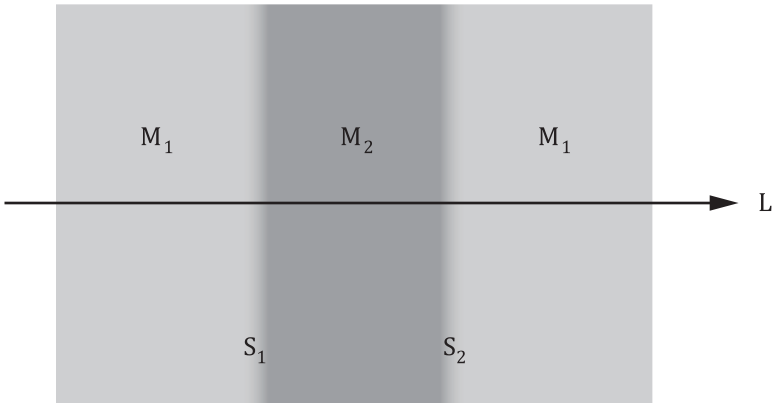
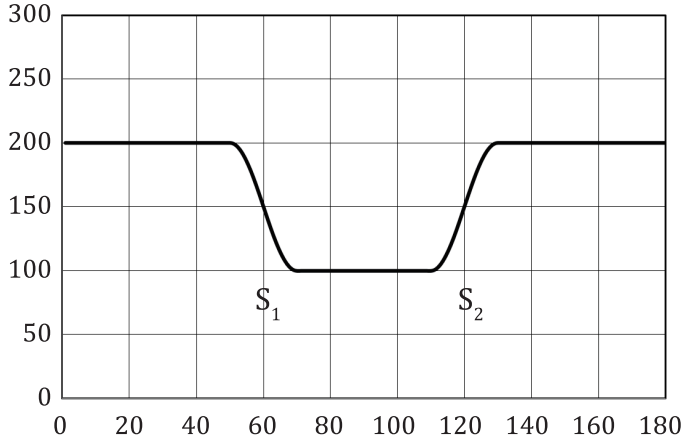
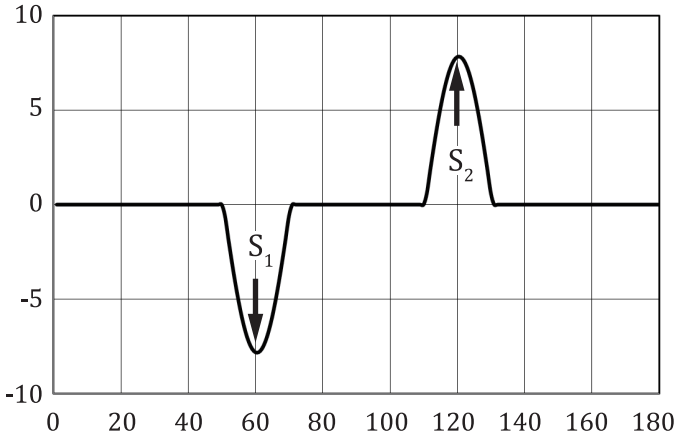
the minimal value and maximal value in the curve show the interface positions ($S_1$ and $S_2$) on both sides of the layer $M_2$.




**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)

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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 g) sequentially for obtaining the desired smooth intensity profile with a plateau and well-defined slope.

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

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

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

c) Set the ROI area in the image.

d) Obtain the averaged intensity profile by adding up and averaging the line profiles in the direction perpendicular to the interface (parallel to the x-axis of the monitor screen) over the entire interface direction (parallel to the y-axis of the monitor screen) in the ROI.

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

f) Apply differential processing to the moving-averaged intensity profile obtained in e).

g) 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.

# 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 an atomic resolution image with an atomic column arrangement along the interface, the oscillated component depends on the atomic column which 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 an 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 Clause 6 details of each procedure will be described.

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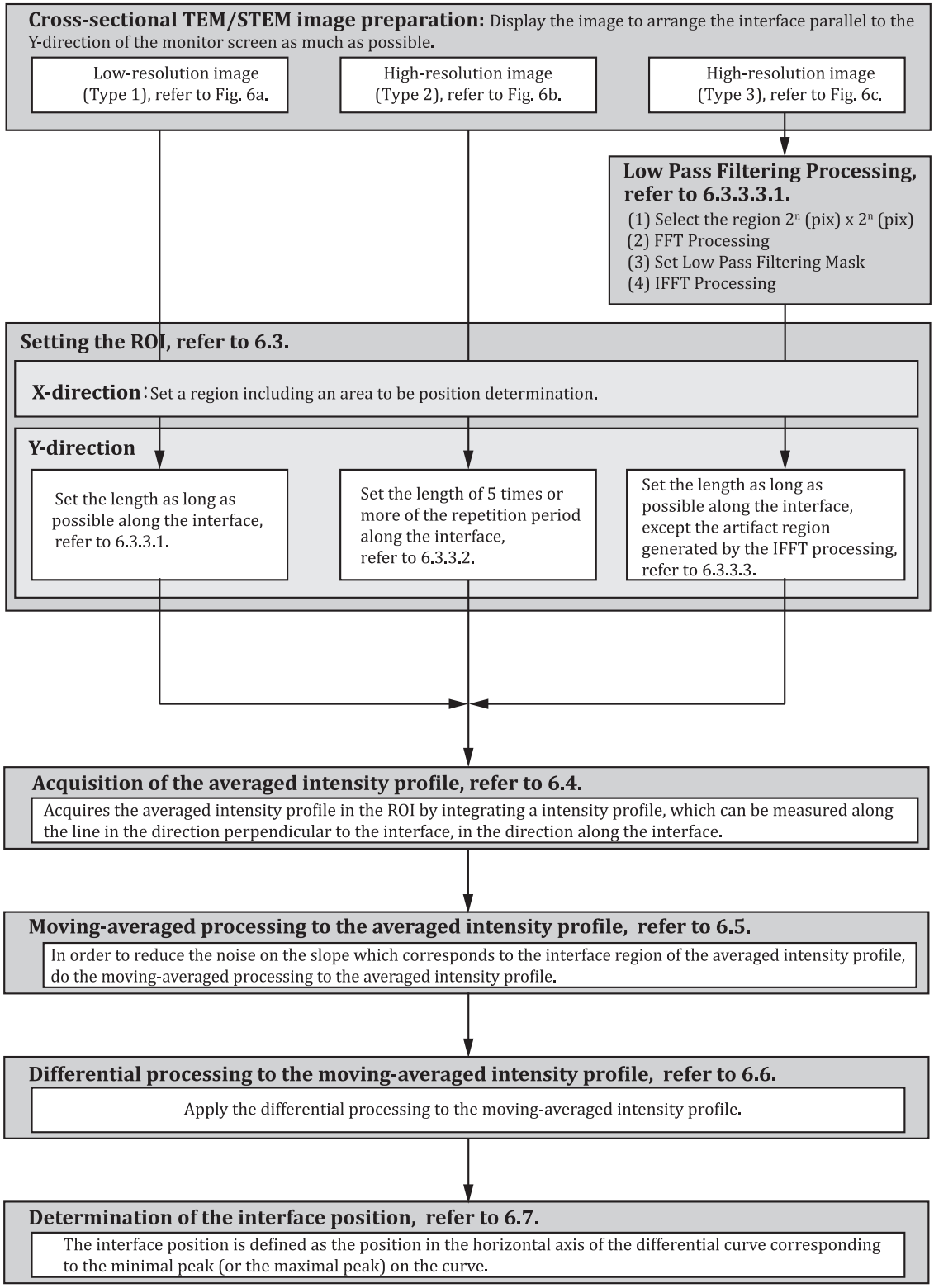


**Figure 4 — Flow chart of interface position determination procedure**

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