International Standard

ISO 20263

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

Second edition
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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

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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

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.

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.

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

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]

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]

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
            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 α [shown in Figure 1, a)] shall be 90° ± 6°.

a) Multi-layered specimen

b) TEM/STEM observation

c) Cross-sectional imaging
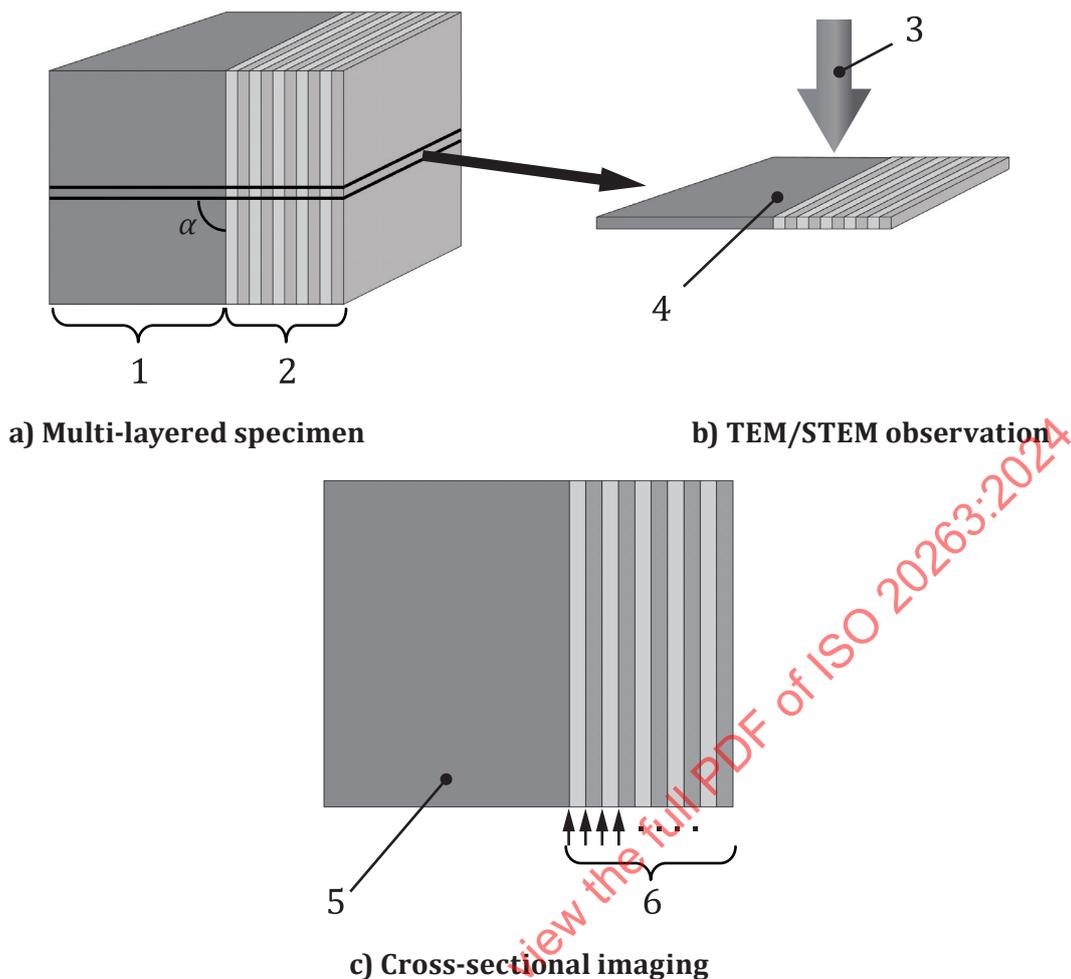
Key

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.

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.
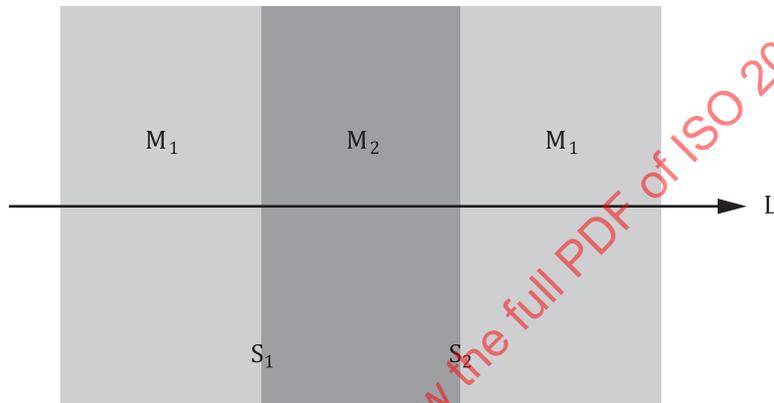
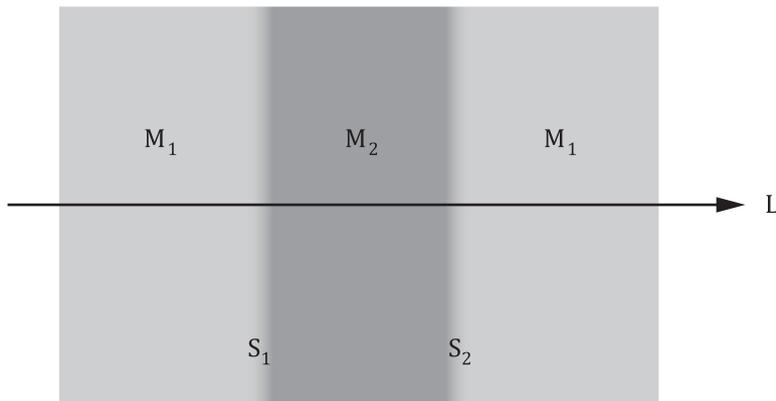a) Ideal interface (S_1 and S_2) model between two kinds of layers, M_1 and M_2
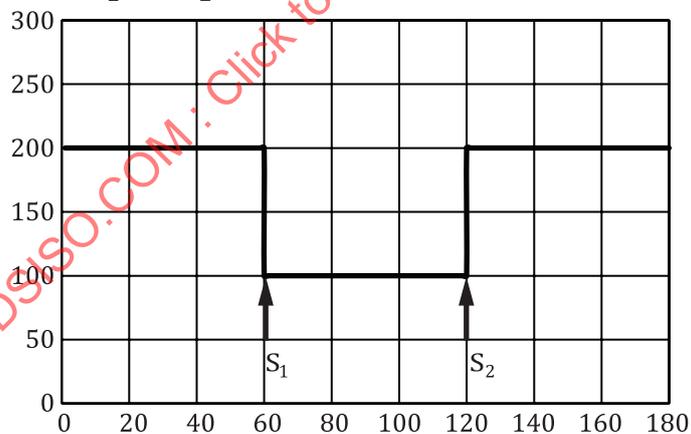
b) Intensity profile along an arrow line, L, in a), perpendicular to the interfaces (S_1 and S_2)

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

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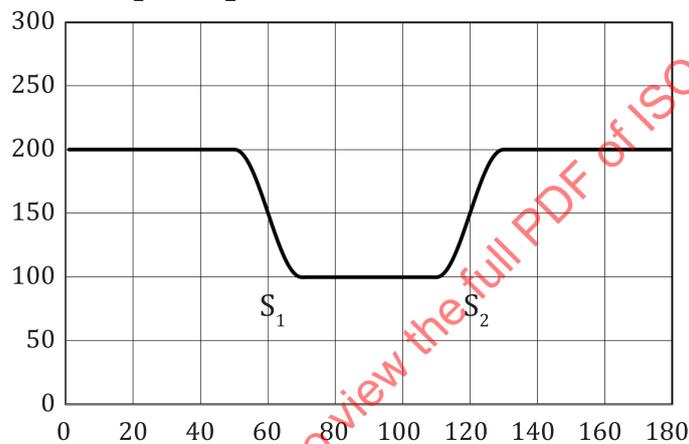
b) Intensity profile along an arrow line, L, in a), perpendicular to the interfaces (S_1 and S_2)
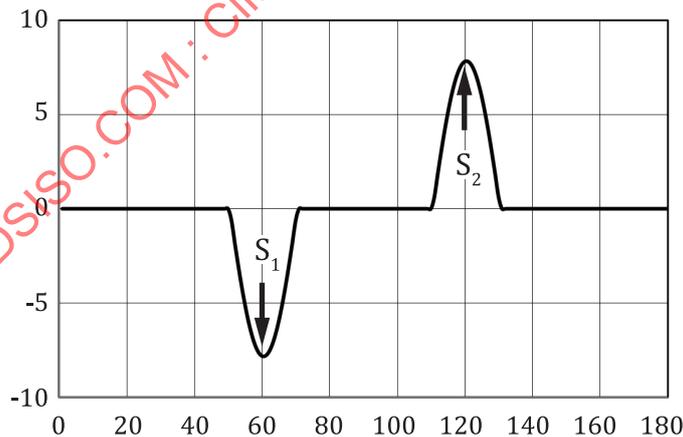
c) Differential curve of intensity profile

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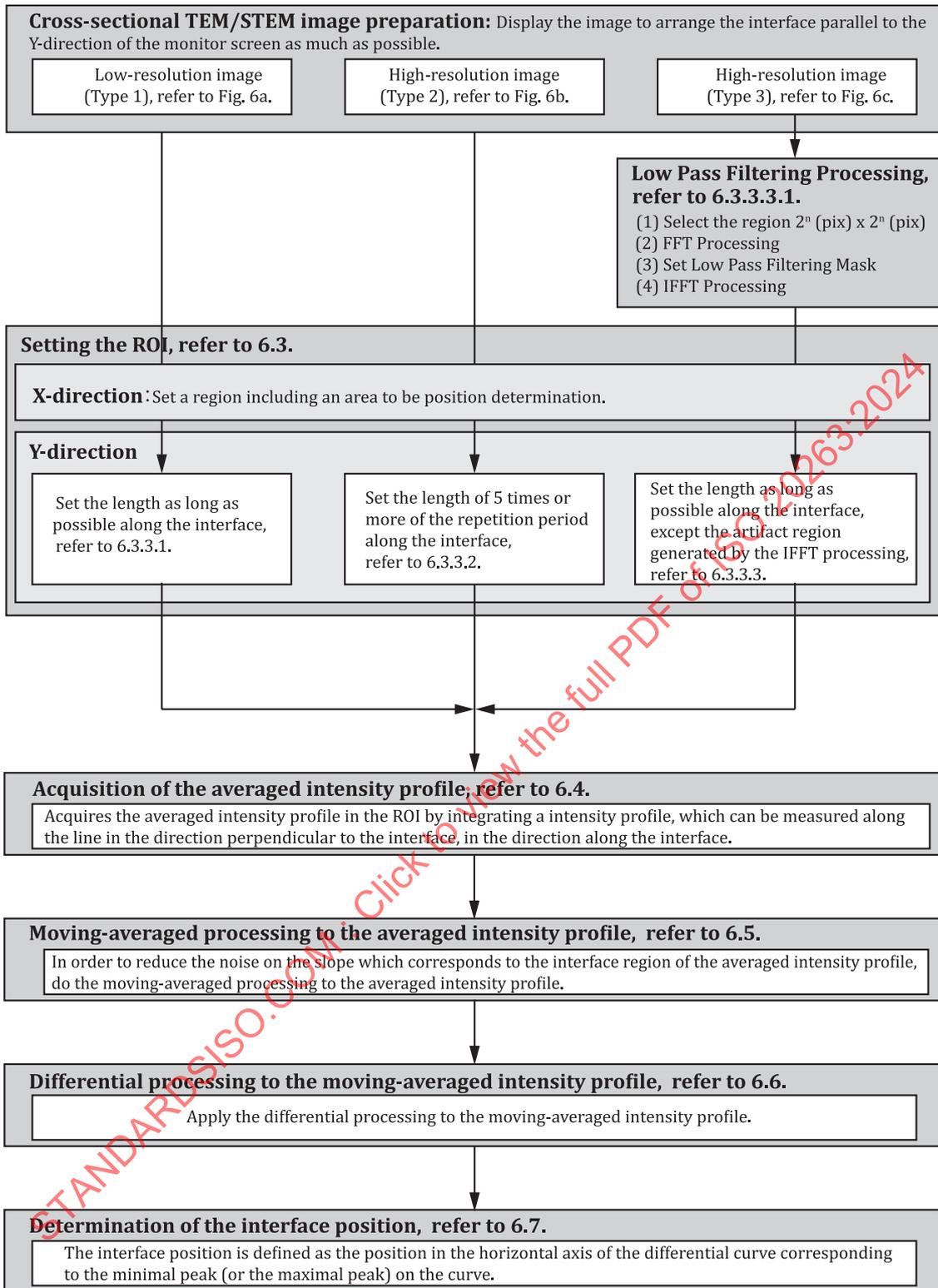
Cross-sectional TEM/STEM image preparation: Display the image to arrange the interface parallel to the Y-direction of the monitor screen as much as possible.

Low-resolution image (Type 1), refer to Fig. 6a.	High-resolution image (Type 2), refer to Fig. 6b.	High-resolution image (Type 3), refer to Fig. 6c.

Low Pass Filtering Processing, refer to 6.3.3.3.1.
(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

6.2 Preparing cross-sectional TEM/STEM image

6.2.1 Preparing digitized Image

It is necessary to prepare a digitized cross-sectional image to determine the interface position. The bit depth of digitization of the image shall be 8 bits or more. There are four ways of digitizing the image corresponding to each image detection system shown in Table 1.

Table 1 — Comparison table for image detection

Image detection system	Apparatus for digitization	Pixel size
Photographic film	Flatbed image scanner	Determined by dot pitch applied to image scanner
Imaging plate	Dedicated scanner	Determined by LASER beam diameter for readout
Image sensor (Digital camera)	Built in the digital camera	Same size as that of the image sensor
Digital memory	Built in the PC connected to the scanning device	Determined by scanning condition of electron beam

a) Photographic film: Analogue image recorded on the photographic negative film shall be converted to a digitized image using an image scanner with dot pitch less than 20 μm.

   NOTE 1   Use a flatbed image scanner because it is easy to set the transparent scale in it for pixel size calibration (Refer to ISO 29301).

b) Imaging plate (IP): The recorded image shall be read out with a dedicated image digitizer (IP reader) connected to a PC.

c) Image sensor: The digitized image, captured by a digital camera, shall be saved on the memory in the PC system as an image file with a reversible format.

   NOTE 2   Ensure that the procedure for normalization of gain is performed to have a uniform background of the image.

d) Digital memory: STEM and elemental mapping image, captured by the digital memory built into a PC, shall be saved as an image file with a reversible format.

Before and during the execution of the digitization procedure, ensure the following.

— The correct sensitivity setting is used for the photographic film used, to generate the negative image with proper density and contrast.

— Keep the exposure time to minimize the drift and reduce blurring in the recorded image.

— Do not use "binning" in the readout process of the magnified digital image from the digital camera.

— When using an image montage function, ensure that the seams of the image do not overlap with any of the interface of the specimen.

— For saving the digitized images, an uncompressed image file format (ESP, PICT, TIFF or Windows bitmap), or reversible (lossless) compressed image file format (GIF or PING) shall be used.

— Ethical digital imaging requires that the original uncompressed image file be stored on archival media, e.g. CD-R, without any image manipulation or processing operation. All parameters of the production and acquisition of this file and any subsequent processing steps shall be documented and reported to ensure reproducibility.

Generally, acceptable imaging operations include gamma correction, histogram stretching and brightness and contrast adjustments need not be reported. All other operations (such as Unsharp-Masking, Gaussian Blur, etc.) shall be directly identified by the author as part of the experimental methodology. However, for

diffraction data or any other image data that is used for subsequent quantification, all imaging operations shall be reported.

6.2.2 Displaying the digitized image

The digitized image used for further processing shall be orientated so that the interface is, as far as possible, parallel to the y-axis of the monitor screen. If the interface is tilted with an angle, α, to the y-axis of the monitor screen, it is necessary to measure and to record the tilting angle, α (see Figure 5).
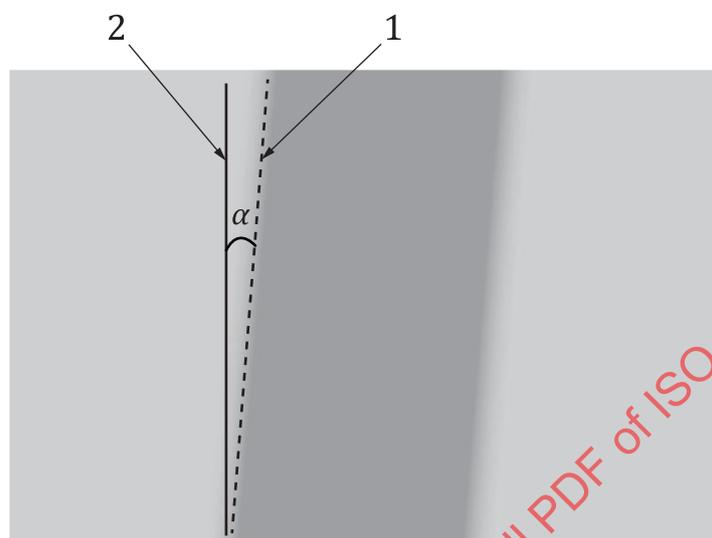
Key

1   interface
2   y-axis of monitor screen

Figure 5 — Example of a tilted target image

6.3 Setting the ROI

6.3.1 General

This depends on the type of the image classified in 6.3.2.

6.3.2 Classification of image

Firstly, it is necessary to obtain an intensity profile in a direction across the interface. Ensure that the intensity profile is as smooth as possible. To do this, set the ROI in the target image in advance. Then, integrate the line profile measured across the interface, pixel by pixel, to the appropriate range along the interface. In practice, the setting procedure depends on the image type classified by image resolution and the atomic column arrangement recorded in high resolution image, as follows.

— Type 1: Low resolution images in which atomic column arrangements cannot be recognized. Figure 6 a) shows an example.

— Type 2: High resolution images in which atomic column arranged at an angle θ to the interface [see Figure 6 b)].

— Type 3: High resolution images in which atomic column is parallel to the interface [see Figure 6 c)].

a) Type 1 image

b) Type 2 image

c) Type 3 image

Key
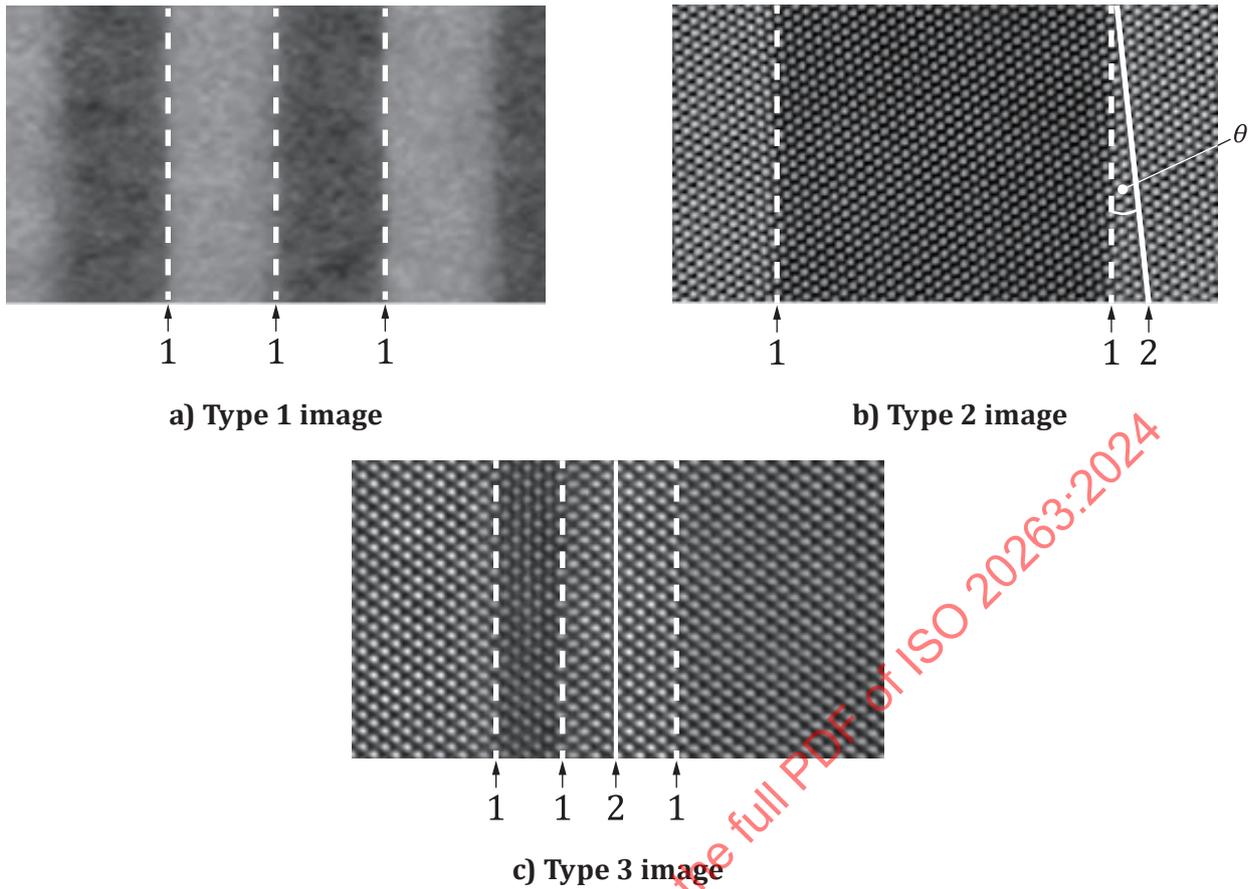
1   interface position

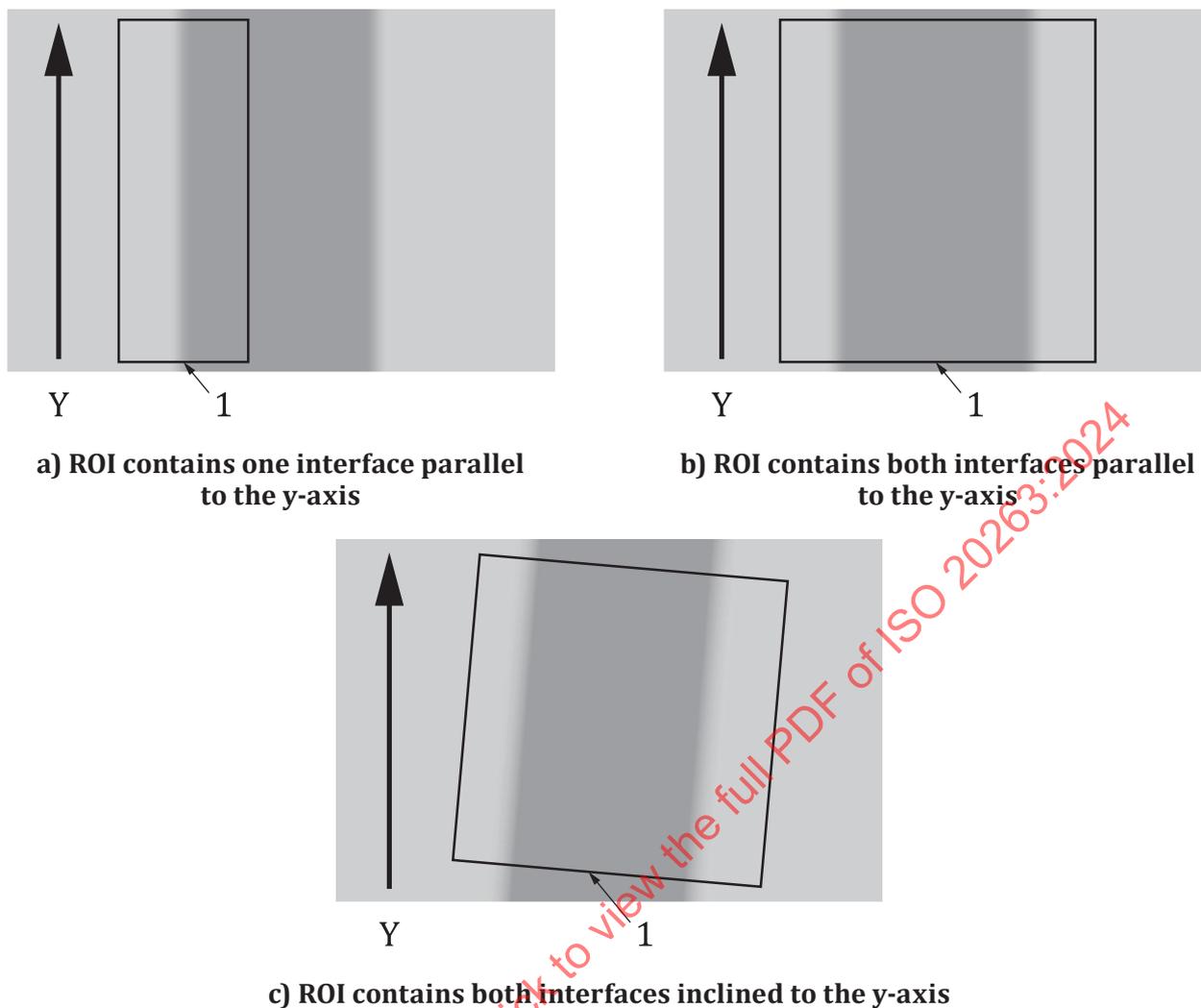2   atomic column arrangement direction

Figure 6 — Examples of classified target image

6.3.3   Procedure of setting the ROI

6.3.3.1   ROI for type 1 image

The ROI shall cover the widest possible area in the image, including the interface.

Figure 7 shows examples of ROI settings for the type 1 image model. In a very low magnification image, image distortion is sometimes observed at the edges. In such case, exclude the area containing distortions from the ROI setting. Figure 7 a) and b) show examples of setting the ROI when the interface is displayed in parallel to the y-axis of the monitor screen. In these cases, each side of the ROI area is parallel to the x-axis or y-axis. Figure 7 c) shows an example of setting the ROI when the interface is inclined to the y-axis of the monitor screen. In this case, the ROI shall be set; one side of the ROI is parallel to the direction of the interface.

a) ROI contains one interface parallel to the y-axis

b) ROI contains both interfaces parallel to the y-axis

c) ROI contains both interfaces inclined to the y-axis

Key

1   region of interest (ROI)

Y   y-axis of monitor screen

Figure 7 — Examples of ROI setting for type 1 image model

6.3.3.2   ROI for type 2 image

A schematic model of the type 2 image is shown in Figure 8. Repetition period, R, of the atomic column arrangement along the interface can be calculated by using Formula (1):

$$R = s/\sin \theta = (s_x \times \cos \theta)/\sin \theta \tag{1}$$

where

s	is the atomic column spacing in layer B;
s_x	is the projected length of the atomic column spacing, s, in layer B onto the x-axis of the monitor screen;
θ (°)	is the off-angle between the interface and the atomic column arrangement in layer B.

For the type 2 image, ROI length shall be at least $(5 \times R)$ where R is the periodic length along the y-axis. However, for an image with a wider field of view, it is better to set the ROI as wide as possible. If the interface

is inclined to the y-axis, set one side of the ROI parallel to the direction of the interface (the same setting as that for the type 1 image).
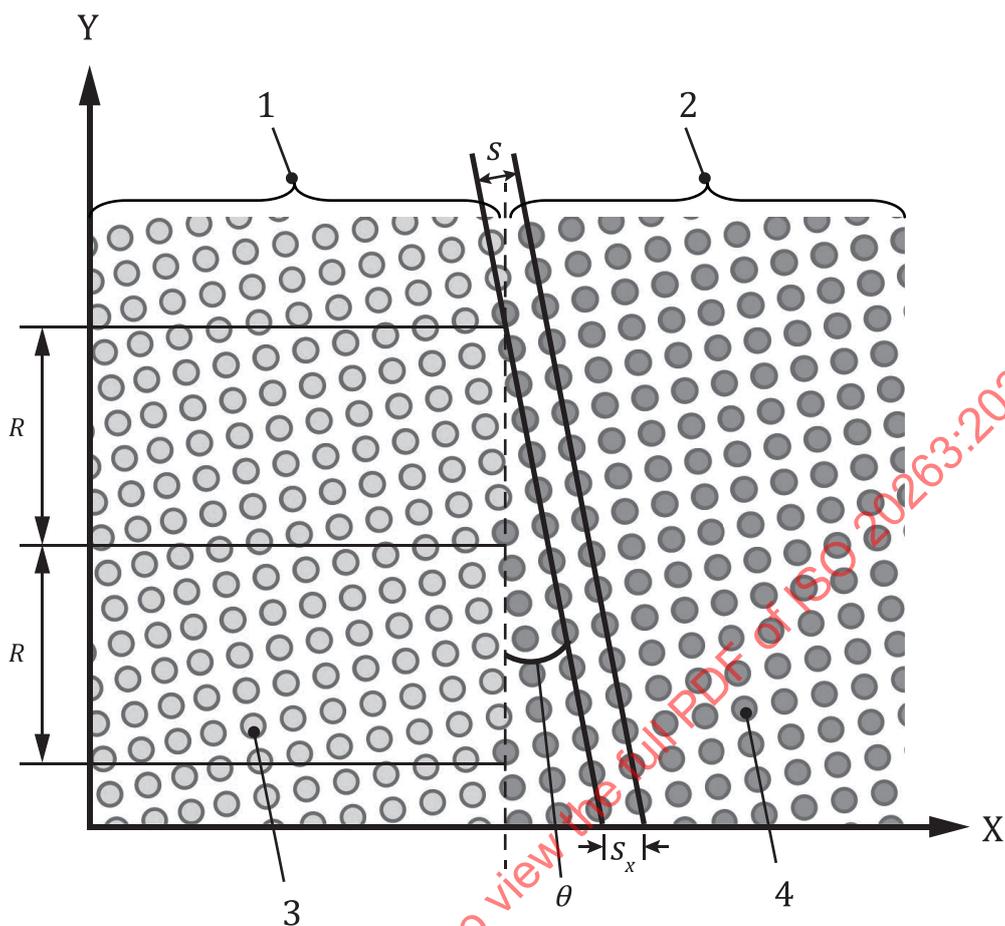
Key

[broken line]	interface position
[thick solid line]	atomic column arrangement direction
[thin solid line]	auxiliary line for repetition period, R
1	layer A
2	layer B
3	atomic column in layer A
4	atomic column in layer B

Figure 8 — Examples of atomic column arrangement in the type 2 image model

6.3.3.3 ROI for type 3 image

A schematic model of the type 3 image is shown in Figure 9 a), keys 1 and 2. In this case, it is impossible to eliminate the oscillation of the intensity in the integrated intensity profile because the atomic column is aligned in the direction perpendicular to the interface. Therefore, for such an oscillatory intensity profile, it is impossible to determine the interface position because one cannot apply the last step of this procedure. In such cases, therefore, a low-pass filter shall be applied as a pre-treatment for the ROI setting.

6.3.3.3.1 Low pass filter treatment

Apply the low pass filter treatment by following the procedure below.

a) Select 2^n (pix) × 2^n (pix) area (with edges parallel to the x-axis and y-axis) in the target image for FFT processing [region F indicated in Figure 9 a), key 3].

In order to get a clear FFT pattern, it is desirable to set $n \geq 8$ to include the largest possible area.

b) Apply FFT processing to the region F [key 5 in Figure 9 b) shows the result of FFT processing].

c) Set low pass filtering mask [Figure 9 c), key 6] onto the pattern generated by FFT processing to the region F. The mask has a circular opening having diameter $d_m = (0,9 \sim 0,95)*d_f$ (where, d_f is the distance between primary spots) centred on the central spot, to set the circumference as close as possible to the inside of the primary spots of the FFT pattern.

NOTE   Image resolution after IFFT processing is determined by diameter, d_m, of the low-pass filtering mask. Application of smaller radius makes image resolution lower than that with bigger radius.

d) Apply an IFFT processing to the FFT pattern with low pass filtering mask. [Figure 9 d), key 7 shows the result of IFFT processing.]
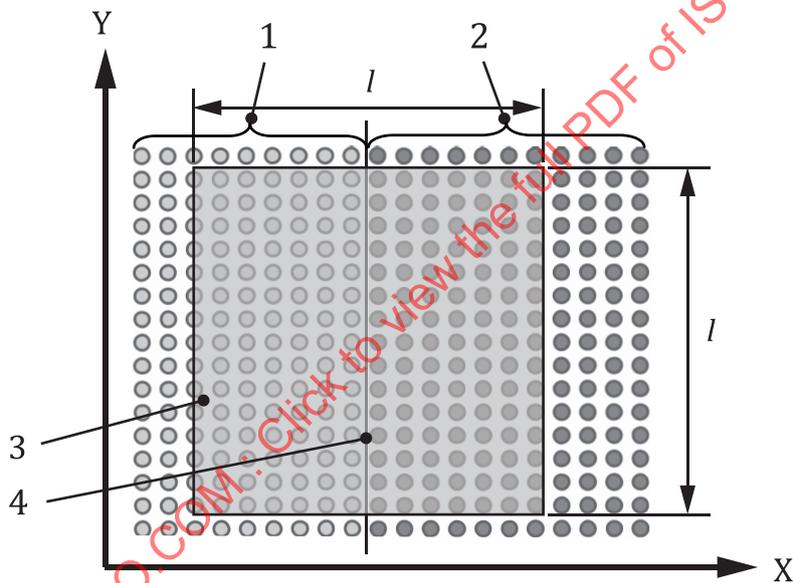
a) Type 3 image model
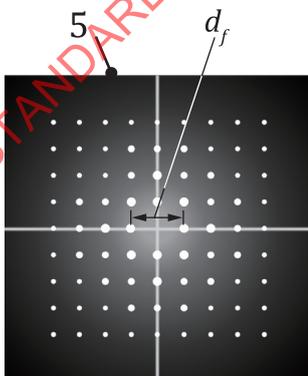
b) FFT pattern model
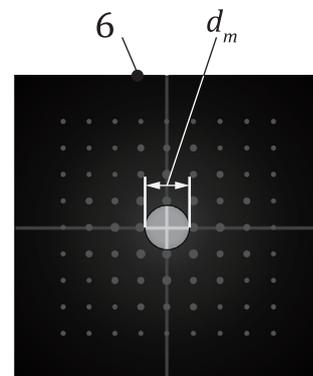
c) Low pass filtering mask superimposed with FFT pattern
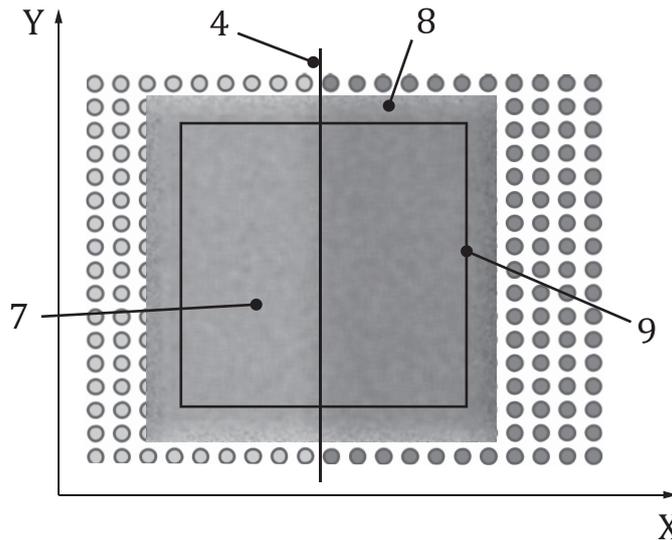
d) IFFT image model

Key

1   layer A in the type 3 image model
2   layer B in the type 3 image model
3   selected square area F for FFT processing
4   interface position
5   FFT pattern from area 3
6   low-pass filtering mask superimposed with FFT pattern from area 3
7   IFFT image
8   artefact region
9   ROI frame

The side length (l) should be 2^n pixels.

Figure 9 — Examples of type 3 image model in which atomic columns are aligned in direction perpendicular to the interface

6.3.3.3.2   Setting the ROI

Set ROI [Figure 9 d), key 9] into the IFFT image [Figure 9 d), key 7], except the artefact region [Figure 9 d), key 8] observed at the edge of the IFFT image [Figure 9 d), key 7]. If the interface is inclined to the y-axis, set the ROI so that one side of it is parallel to the direction of the interface (the same setting as that for the type 1 image).

6.4   Acquisition of the averaged intensity profile

After setting the ROI into the target image, apply the same processing for all types of images. Herein, as an example, the process is applied to the type 1 (low resolution) image [Figure 10 a)].
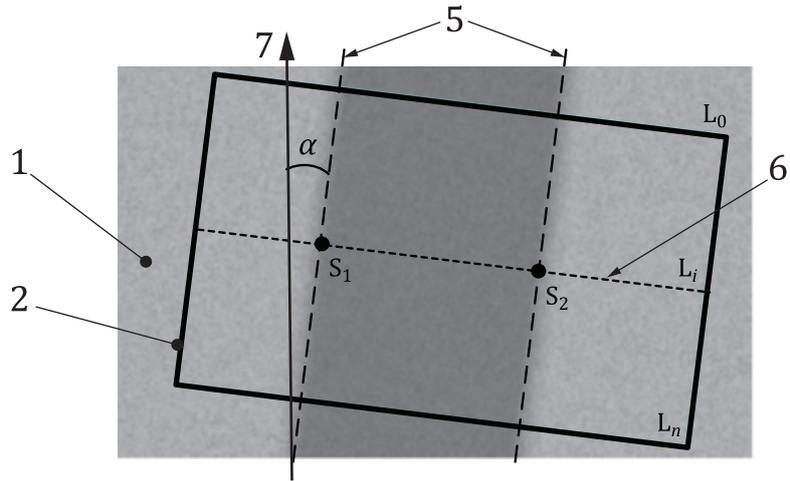
a) Type 1 image model having an inclined interface
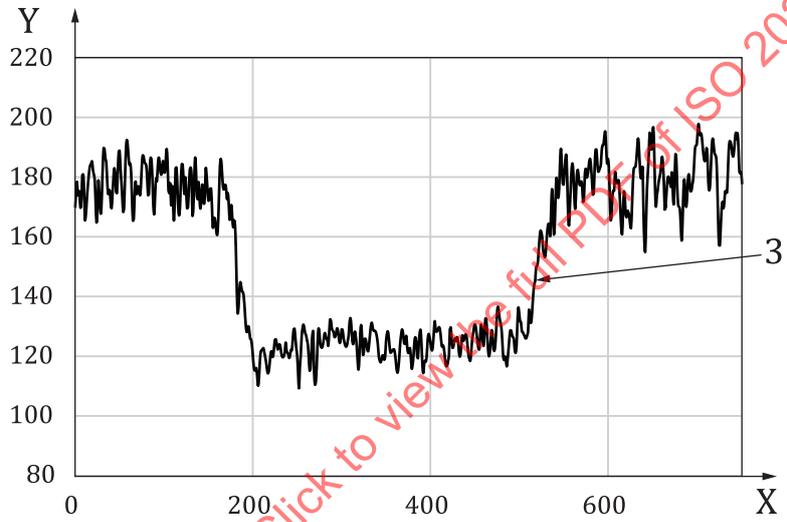
b) Intensity profile
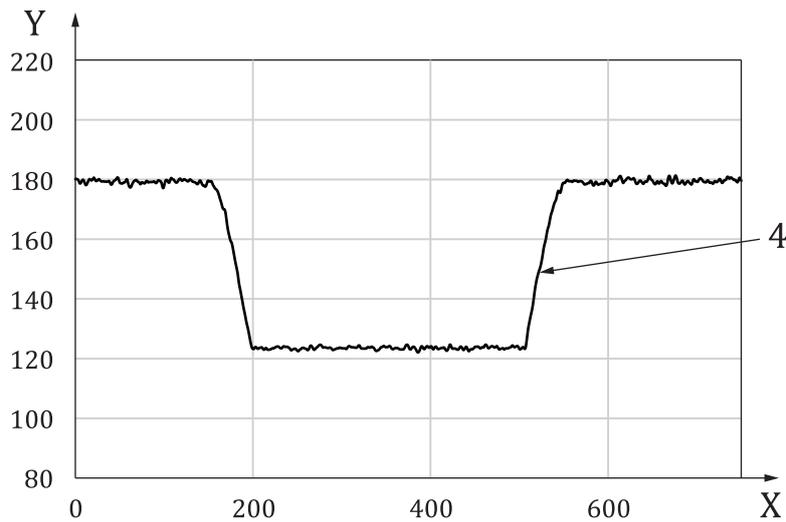
c) Averaged intensity profile

Key

X   position (pix)

Y   intensity

1   type 1 image model

2   ROI frame

3   intensity profile *Ii* along line L_i in key 2

4   averaged intensity profile, *Ia*, calculated by averaging *Ii* from L_o to L_n in key 2

5   interface

6   line for intensity profile measurement

7   y-axis of the monitor screen

The fine broken line should be perpendicular to the interface.

Figure 10 — Examples of type 1 image model having an inclined interface to the y-axis of the monitor screen

a) Measure and record the angle α between the interface and the y-axis of the monitor screen, shown in Figure 10 a).

b) Draw the straight line [L_i in Figure 10 a)] in the direction perpendicular to the interface, in the ROI; the intensity profile, I_i, along the line L_i contains many noise components in it [Figure 10 b), key 3].

    NOTE   The level of the intensity profile is equivalent to the grey level of the image acquired. The grey level is dependent on the image detection system, in most cases, with 8-bit, 12-bit or 16-bit grey scale. It is possible to obtain the intensity profile by reading the grey level of each pixel of the measurement line by using the image processing software.

c) Acquire the averaged intensity profile, I_a, [Figure 10 c), key 4] using Formula (2) through integrating procedure of the intensity profile, from I_0 to I_n, which is measured parallel to the line L_i, from L_0 to L_n in the ROI.

$$I_a \equiv \frac{\sum_{i=0}^{n} I_i}{n+1}$$

(2)

where

I_i is ith intensity profile along the straight line, L_i, in the ROI;

$n + 1$ is total number of pixels contained from straight line L_0 to L_n;

I_a is the intensity profile averaged the profile from I_0 to I_n corresponding to the line $L_0 \sim L_n$ in the whole region of the ROI.

6.5 Moving-averaged processing

After the application of the procedure in 6.4, residual noise still remains in the slope of the averaged intensity profile [Figure 11 a), key 1]. To reduce the residual noise further, apply the moving-averaged processing by Formula (3).

$$I_p \equiv \frac{\sum\limits_{j=p-n}^{p+n} I_j}{2n+1} \tag{3}$$

where

I_p is the calculated moving-averaged intensity of pth pixel in the horizontal axis of the averaged intensity profile;

I_j is the intensity around the pth pixel in the horizontal axis of the averaged intensity profile, to be used to calculate the moving-averaged intensity;

n is a numerical value, a positive integer, to define the range of the I_j to be used in the calculation.

NOTE   Generally, the range $2 \leq n \leq 7$ is suitable.

Key 2 in Figure 11 b) shows the moving-averaged intensity profile of the averaged profile shown in key 4 in Figure 10 c), with $n = 4$ in Formula (3).
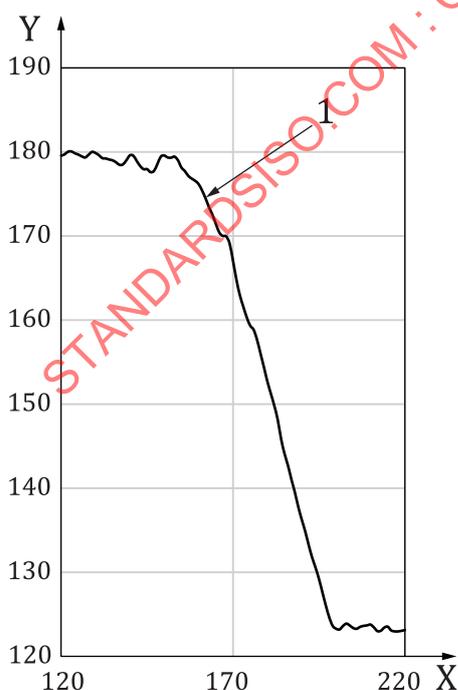
a) Slope before moving-averaged processing   **b) Slope after moving-averaged processing**

Key

X   position (pix)

Y   intensity

1   slope having residual noise in the averaged intensity profile

2   smooth slope after moving-averaged processing applied n = 4 to Formula (3)

Figure 11 — Noise reduction effect by moving-averaged processing

6.6   Differential processing

Apply differential processing to the moving-averaged intensity profile [see key 1 in Figure 12 a)], by using Formula (4) and Formula (5).

$$\Delta I_k \equiv I_{k+1} - I_k \tag{4}$$

here,

$$1 \le k \le n_p - 1 \tag{5}$$

where

ΔI_k is the rate of intensity change of kth pixel position in the horizontal axis of the moving-averaged intensity profile;

I_k is the intensity of the kth pixel position in the horizontal axis of the moving-averaged intensity profile;

I_{k+1} is the intensity of the $(k+1)^{\text{th}}$ pixel position in the horizontal axis of the moving-averaged intensity profile;

n_p is the total number of pixels contained in the horizontal axis of the moving-averaged intensity profile.

By differential processing, the maximum gradient positions of the intensity change at the interface appear as distinct peaks [see key 2 in Fig. 12 b)].
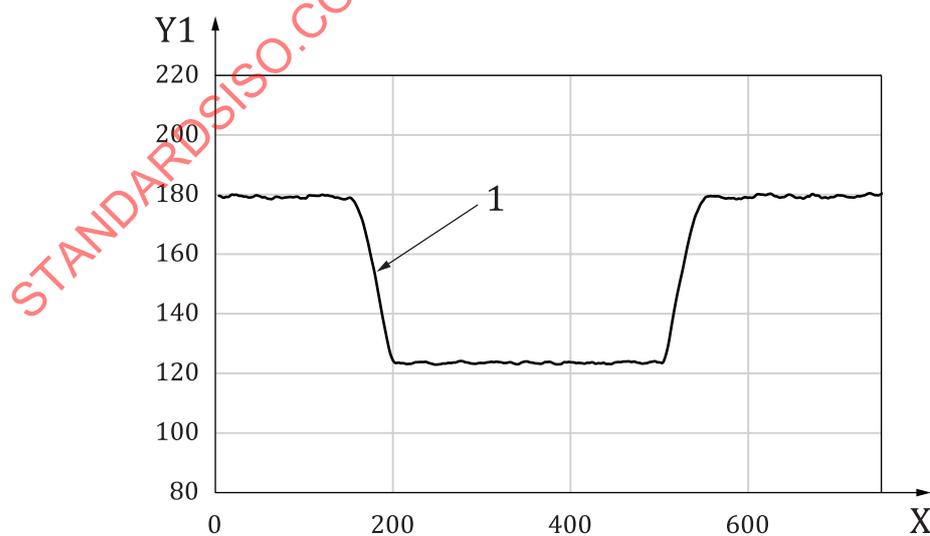
a) Intensity profile after moving-averaged processing
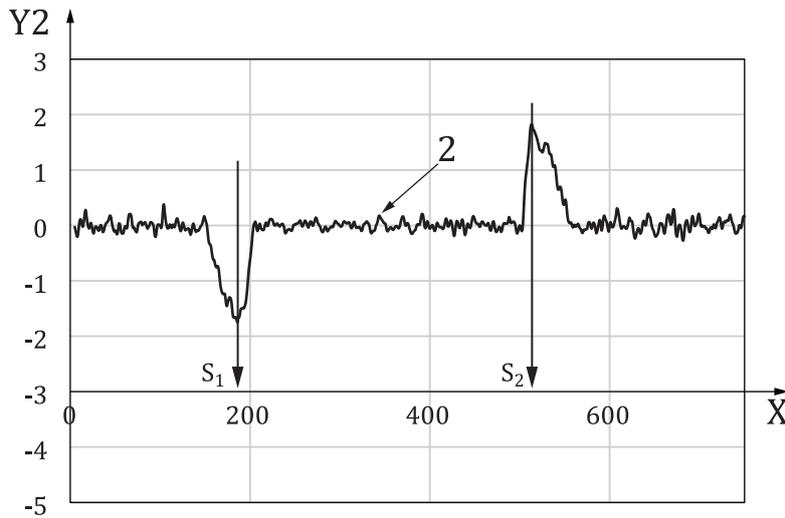
b) Location of interface position

Key

X   position (pix)

Y1   intensity

Y2   rate of change of intensity (ΔI)

1   moving-averaged result applied n = 4 to Formula (3) for key 4 in Figure 10

2   differential processing result applied to key 1

Figure 12 — Detection of interface position based on differential processing on moving-averaged result

6.7   Determination of interface position

As defined in 5.2.2, the interface position is indicated by the pixel position on the horizontal axis corresponding to the steepest slope of the moving-averaged intensity profile. This means that in the resulting graph of the differential processing of the moving-averaged intensity profile, the interface position can be indicated by the position corresponding to the maximum or minimum intensity peak. For example, in key 2 in Figure 12 b), S_1 and S_2 are both interface positions of the layered materials showing in the key 1 Figure 10 a).

If it is difficult to decide the interface position from the differential curve because of many sub-peaks appears around the interface position, it is necessary to try again by changing the n-condition for Formula (3), to get smoother slope of the moving-averaged intensity profile.

Examples of processing this interface position determination procedure to the real TEM/STEM image corresponding to each of three types as defined in 6.3.2 are described in Annex A. Two main applications using this procedure are described in Annex B.

7   Uncertainty

7.1   Uncertainty accumulating from each step of the procedure

There are the following three factors that cause uncertainty accumulated from each step of the procedure described in this document.

σ_{ROI} Uncertainty associated with the ROI setting; ROI size and its location [refer to Figure 7 and key 9 in Figure 9 d)].

σ_{LPF} Uncertainty associated with the low-pass filter setting; mask size [refer to key 6 in Figure 9 c)].

σ_{MA} Uncertainty associated with the moving-averaging setting; pixel value for moving [refer to Formula (3)].

These three factors are independent. Therefore, it is necessary to measure the data under plural conditions setting for each factor. For example, repeat the measurement *l* times at different ROI settings, *m* times at different LPF settings and *n* times for MA settings, totally $R = l \times m \times n$ data set can be obtained. The uncertainty of each factor is expressed as follows in Formulae (6) to (8):

$$\sigma_{\text{ROI}} = \sqrt{\frac{1}{l} \sum_{i=1}^{l} \left(D_i - D_{lavg} \right)^2} \ , \tag{6}$$

where

    D_i is the *i*-th data in the different ROI settings;

    D_{lavg} is the averaged value of the *l*-data set.

$$\sigma_{\text{LPF}} = \sqrt{\frac{1}{m} \sum_{j=1}^{m} \left(D_j - D_{mavg} \right)^2} \tag{7}$$

where

    D_j is the *j*-th data in the different LPF settings;

    D_{mavg} is the averaged value of the *m*-data set.

$$\sigma_{\text{MA}} = \sqrt{\frac{1}{n} \sum_{k=1}^{n} \left(D_k - D_{navg} \right)^2} \tag{8}$$

where

    D_k is the *k*-th data in the different LPF settings;

    D_{navg} is the averaged value of the *n*-data set.

The accumulated uncertainty (σ_{ACC}) of these three factors is represented as follows in Formula (9):

$$\sigma_{\text{ACC}}^2 = \sigma_{\text{ROI}}^2 + \sigma_{\text{LPF}}^2 + \sigma_{\text{MA}}^2 \tag{9}$$

Generally, $\sigma_{\text{ROI}} >> \sigma_{\text{LPF}}, \sigma_{\text{MA}}$, then the $\sigma_{\text{ACC}} = \sigma_{\text{ROI}}$, in many cases.

7.2 Uncertainty of measurement result on image analysis

There are two kinds of factors that influence the measurement result for the interface position analysis.

σ_α Uncertainty of the tilt angle α between the interface of the layer and y-axis of the monitor screen (Refer to Figure 5).

σ_θ Uncertainty of the off-angle θ between the interface of the layer and the atomic column arrangement in the type 2 image (Refer to Figure 8).

According to ISO/IEC Guide 98-3 (GUM)[3], these factors are classified into the category of type A uncertainty (U_A).

The uncertainty σ_α and σ_θ shall be calculated from the results by N_1 and N_2 replicate measurements. To do this, the angle α and θ measurement described in 6.2.2 and 6.3.3.2 shall be repeated N_1 and N_2 times (three times or more), respectively.

The combined standard uncertainty, u_{c1}, of the interface position for N_1 and N_2 independent measurement can be calculated by Formula (10).

$$u_{c1} = \sqrt{\left(\frac{\sigma_\alpha}{\sqrt{N_1}}\right)^2 + \left(\frac{\sigma_\theta}{\sqrt{N_2}}\right)^2} \tag{10}$$

Furthermore, it is necessary to consider the uncertainty, σ_u, of the pixel size, u, when the distance between two interfaces will be measured (refer to ISO 29301:2023, 6.8.2 for pixel size measurement). When a photographic film or imaging plate is used as a recording medium, pixel size, u, can be measured referring to the method described in Annex C. To get the σ_u, the pixel size, u, shall be repeatedly measured N_3 times (three times or more). Also, it is necessary to consider the uncertainty of the reference glass scale, σ_g, which is classified into the category of type B uncertainty (U_B).

In case of the distance measurement, the combined standard uncertainty u_{c2} can be calculated by Formula (11).

$$u_{c2} = \sqrt{\left(\frac{\sigma_\alpha}{\sqrt{N_1}}\right)^2 + \left(\frac{\sigma_\theta}{\sqrt{N_2}}\right)^2 + \left(\frac{\sigma_u}{\sqrt{N_3}}\right)^2 + \sigma_g^2} \tag{11}$$

The expanded uncertainty $U_{(P)}$ of the interface position or distance measurement for a series of measurements can be defined by Formula (12) and Formula (13), respectively.

$$U_{(1)} = k \times u_{c1} \tag{12}$$

$$U_{(2)} = k \times u_{c2} \tag{13}$$

where k is the coverage factor.

NOTE   For a confidence limit of approximately 95 %, k is set to 2 and for a confidence limit of approximately 99 %, k is set to 3.

Annex A
(informative)

# Examples of processing the real TEM/STEM images for three image types

A.1 General

The following examples are given for the convenience of users to understand correctly the processing procedure indicated in this document.

A.2 Processing example to type 1 image

A.2.1 Image used in a series of the processes

The low-resolution image shown in Figure A.1, in which atomic column arrangements cannot be recognized, is used in a series of interface determination processes. Information on this image is as follows.

— Specimen: GaAs (10 nm)/AlGaAs (10 nm) multi-layered thin film

— Observation apparatus: TEM equipped with a CCD camera (2k × 2k pixels)

Key
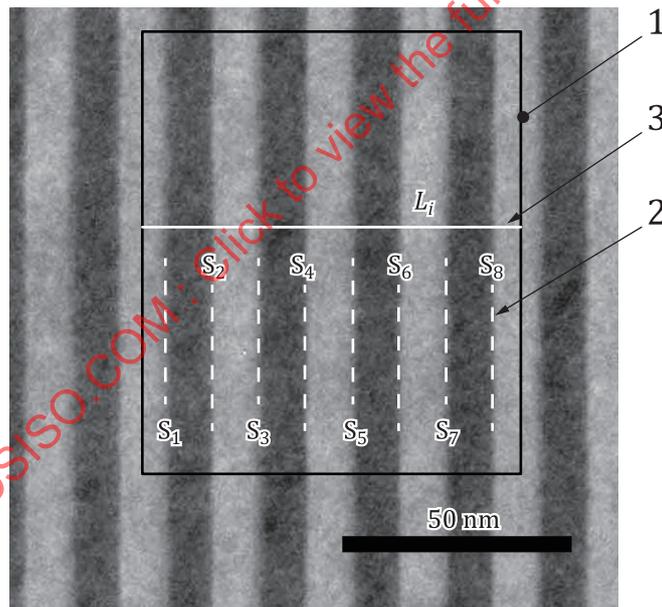
1   ROI frame
2   Interface position (S_1 to S_8)
3   line for intensity profile measurement

The solid white line should be perpendicular to the interface.

Figure A.1 — Example of type 1 image

A.2.2 Acquisition of the averaged intensity profile

Figure A.2 shows the intensity profile along line, *Li*, indicated in Figure A.1. Figure A.3 shows the averaged intensity profile for 450 lines in the ROI frame shown as key 1 in Figure A.1.

Key
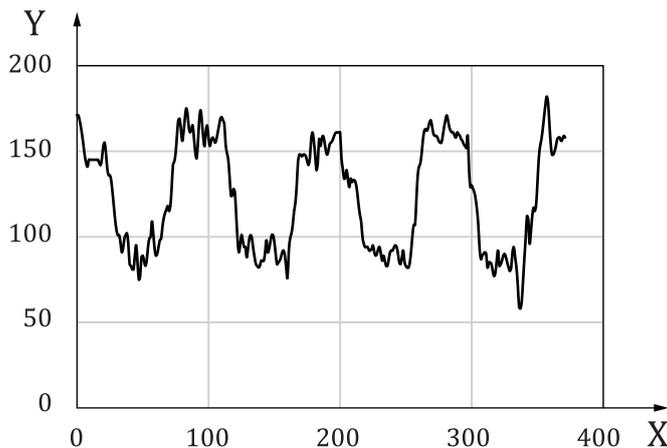
X   position (pix)

Y   intensity

Figure A.2 — Intensity profile for line, *Li*

Key
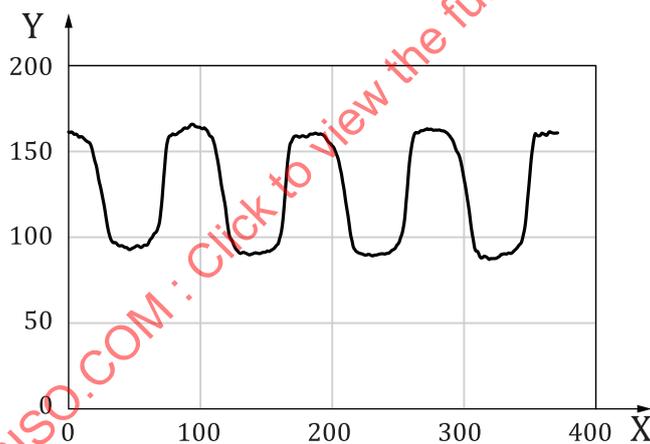
X   position (pix)

Y   averaged intensity

Figure A.3 — Averaged intensity profile for 450 lines in ROI

A.2.3 Acquisition of the moving-averaged processing

Figure A.4 shows the moving-averaged intensity profile of the averaged intensity profile as shown in Figure A.3. To get this profile, a numerical value of 4 was applied as the moving range, *n*, used in Formula (3).

Key
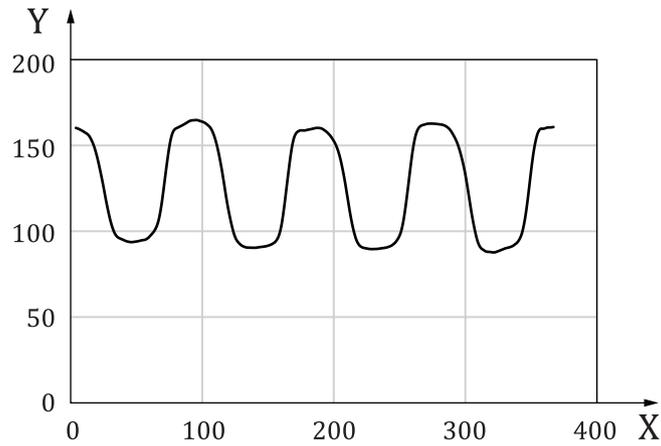
X   position (pix)

Y   moving averaged intensity

Figure A.4 — Moving averaged intensity profile, *n* = 4 applied to Formula (3)

A.2.4 Differential processing

Figure A.5 shows the result of the differential processing on the moving-averaged intensity profile as shown in Figure A.4.

Key
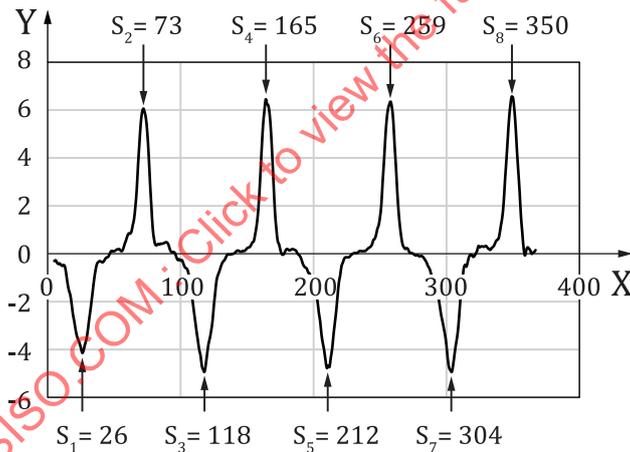
X   position (pix)

Y   rate of intensity change (ΔI)

Figure A.5 — Differential curve

A.2.5 Determination of interface position

Interface positions (S_1 to S_8) are determined as the pixel positions on the x-axis corresponding to the maximal or minimal peaks of the differential curve as shown in Figure A.5. The interface positions obtained are shown below.

$S_1 =$	26 pix		$S_5 =$	212 pix
$S_2 =$	73 pix		$S_6 =$	259 pix
$S_3 =$	118 pix		$S_7 =$	304 pix
$S_4 =$	165 pix		$S_8 =$	350 pix

A.3 Processing example to type 2 image

A.3.1 Image used in a series of the processes

The high-resolution image shown in Figure A.6 in which the atomic column arranged at an angle, θ, to the interface is used in a series of interface determination processes. Information on this image is as follows.

— Specimen: GaAs (10 nm)/AlGaAs (10 nm) multi-layered thin film

— Observation apparatus: STEM with digital scan

— Distance for 10 lines of atomic column: S = 2,83 nm

— Atomic column spacing: a = 2,83 nm/10 lines = 0,283 nm

— Off-angle: θ = 4,35°

— Repetition period: $R = a/\sin \theta$ = 4,18 nm

— ROI setting: 25 nm × 25 nm

Key
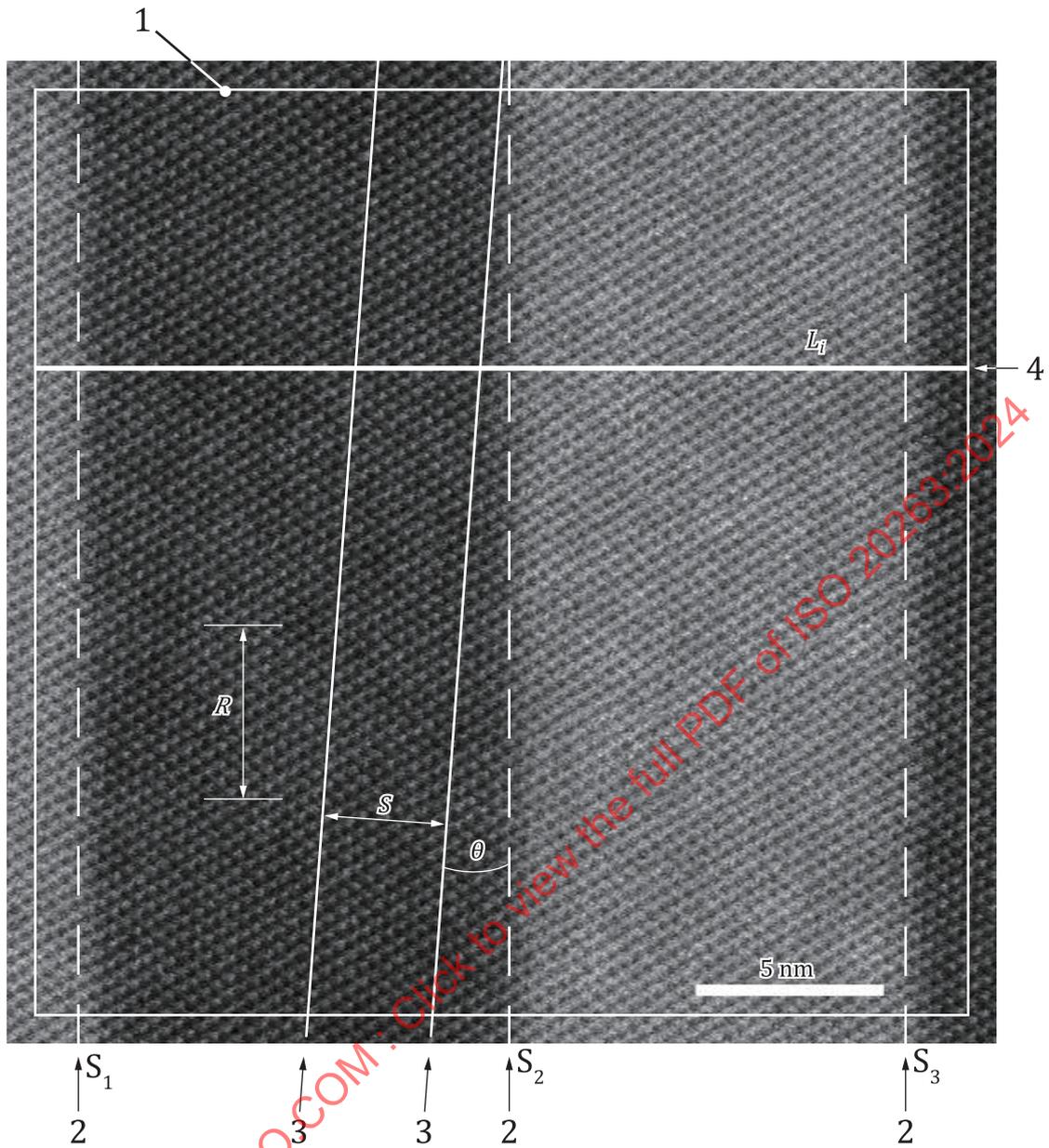
1   ROI frame
2   interface position (S_1 to S_3)
3   atomic column arrangement direction
4   line for intensity profile measurement

This line should be perpendicular to the interface.

Figure A.6 — Example of Type 2 image

A.3.2   Acquisition of the averaged intensity profile

Figure A.7 shows the intensity profile along line, Li, indicated in Figure A.6. Figure A.8 shows the averaged intensity profile for 570 lines in the ROI frame shown as Key 1 in Figure A.6.

Key
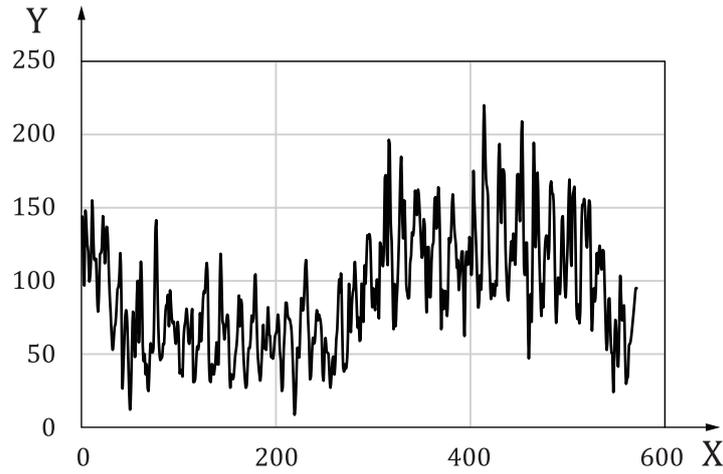
X   position (pix)

Y   intensity

Figure A.7 — Intensity profile for line, *Li*, indicated in Figure A.6

Key
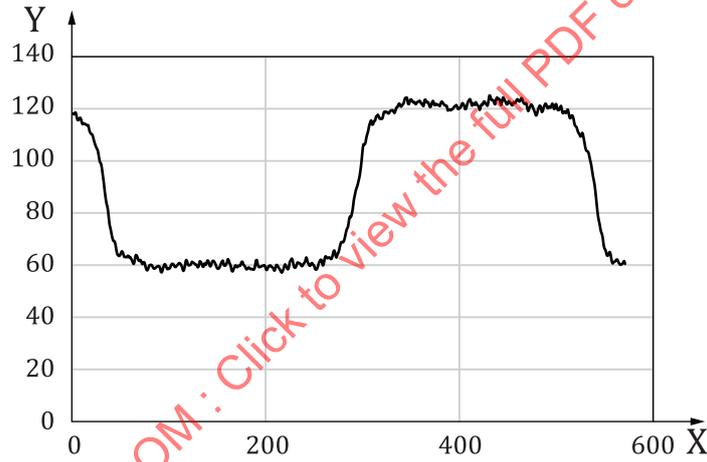
X   position (pix)

Y   averaged intensity

Figure A.8 — Averaged intensity profile for 570 lines in ROI

A.3.3   Acquisition of the moving-averaged processing

Figure A.9 shows the moving-averaged intensity profile of the averaged intensity profile as shown in Figure A.8. To get this profile, a numerical value of 4 was applied as the moving range, *n*, in Formula (3).

Key
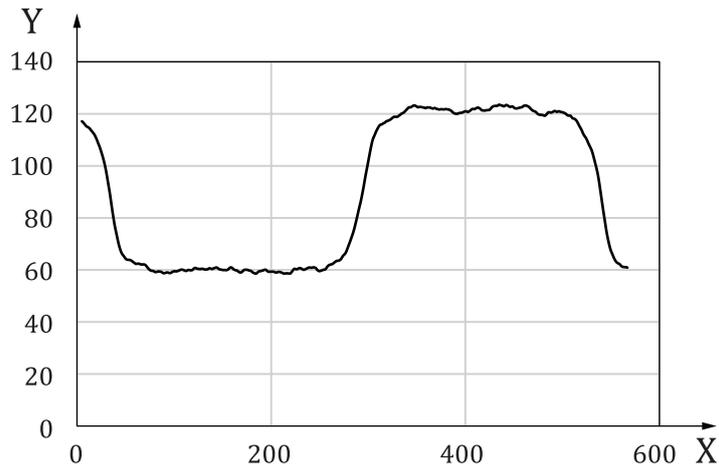
X   position (pix)

Y   moving averaged intensity

Figure A.9 — Moving-averaged intensity profile, *n* = 4 applied to Formula (3)

A.3.4   Differential processing

Figure A.10 shows the result of the differential processing on the moving-averaged intensity profile as shown in Figure A.9.

Key
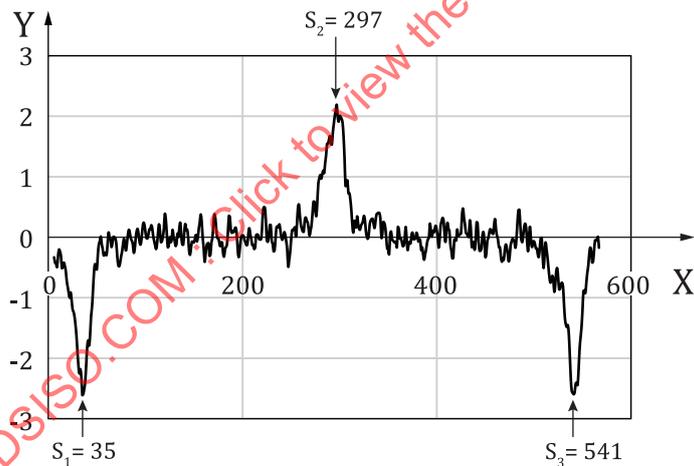
X   position (pix)

Y   rate of intensity change (ΔI)

Figure A.10 — Differential curve

A.3.5   Determination of interface position

Interface positions (S_1 to S_3) are determined as the pixel positions on the x-axis corresponding to the maximal or minimal peaks of the differential curve as shown in Figure A.10. The interface positions obtained are shown below.

S_1 = 35 pix

S_2 = 297 pix

S_3 = 541 pix

A.4 Processing example to type 3 image

A.4.1 Image used in a series of the processes

The high-resolution image shown in Figure A.11, in which an atomic column arranged parallel to the interface, is used in a series of interface determination processes. Information on this image is as follows.

— Specimen: Unknown A/B/C/D/E multi-layered thin film

— Observation apparatus: TEM equipped with a CCD camera (2k × 2k pixels)

— Off-angle: θ = 0° (parallel to the interface)

— FFT area setting: 2 048 pix × 2 048 pix (whole area of the image)

Key
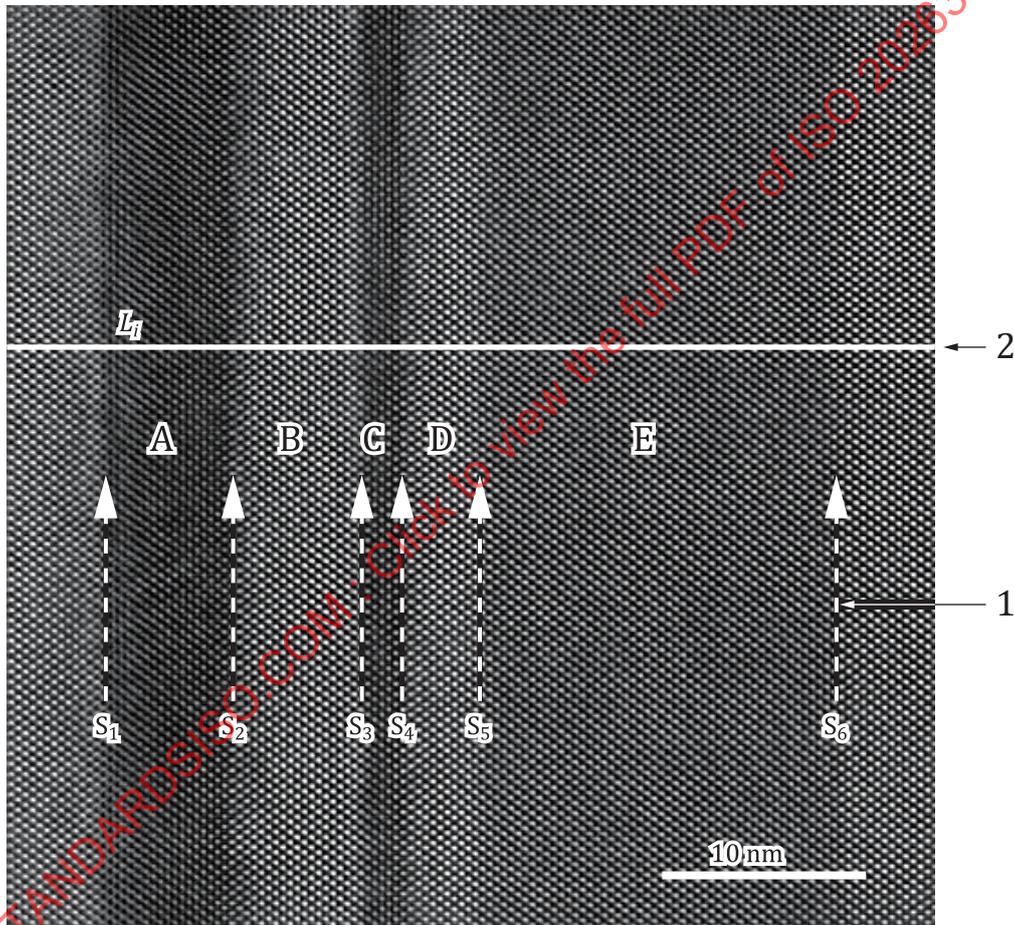
1 interface position (S_1 to S_6)

2 line for intensity profile measurement

The solid line should be perpendicular to the interface.

Figure A.11 — Example of type 3 image

Figure A.12, in which intensity oscillation due to the atomic column appeared, shows the intensity profile along line, *Li*, indicated in Figure A.11. This intensity oscillation cannot be eliminated, even if the averaging processing over several hundred lines is done. Therefore, low-pass filtering processing is required so that there is no influence by the atomic column.

Key
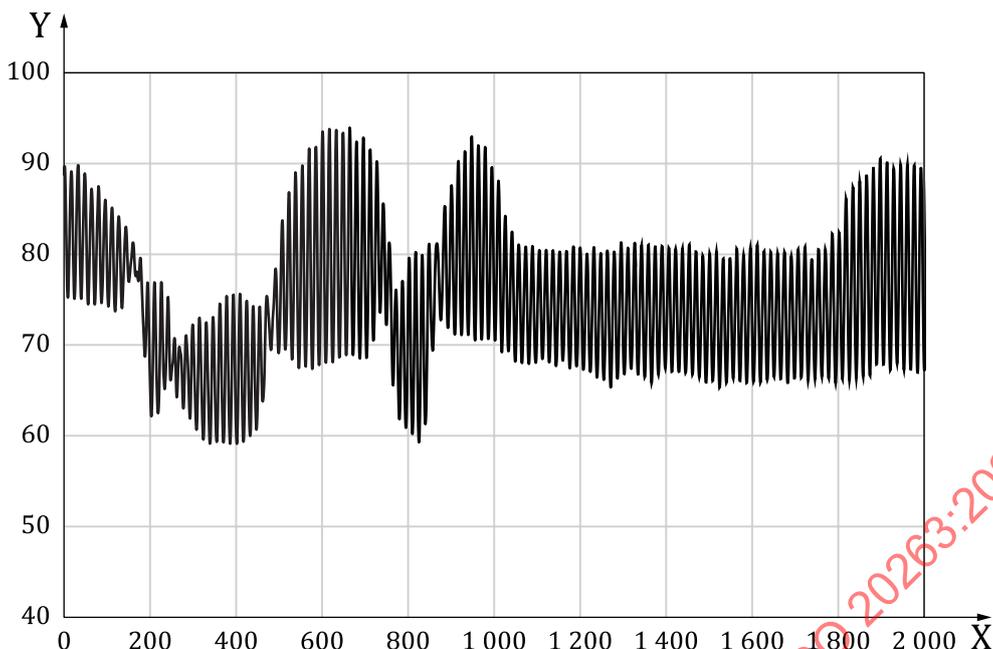
X   position (pix)

Y   intensity

Figure A.12 — Intensity profile for line, *Li*, indicated in Figure A.11

A.4.2   Acquisition of the low pass filtering image

Figure A.13 shows the FFT pattern from the whole area of the image shown in Figure A.11. Figure A.14 shows low pass filtering mask, superimposed to the FFT pattern, to cover clear spots in the FFT pattern. In the IFFT processing, to get the low pass filtering image shown in Figure A.15, only information passing through the window at the centre of the low pass filtering mask is used, a high-frequency component related to the formation of the atomic column arrangement is interrupted.
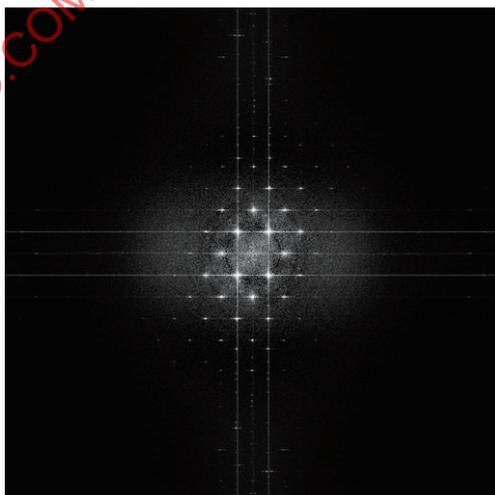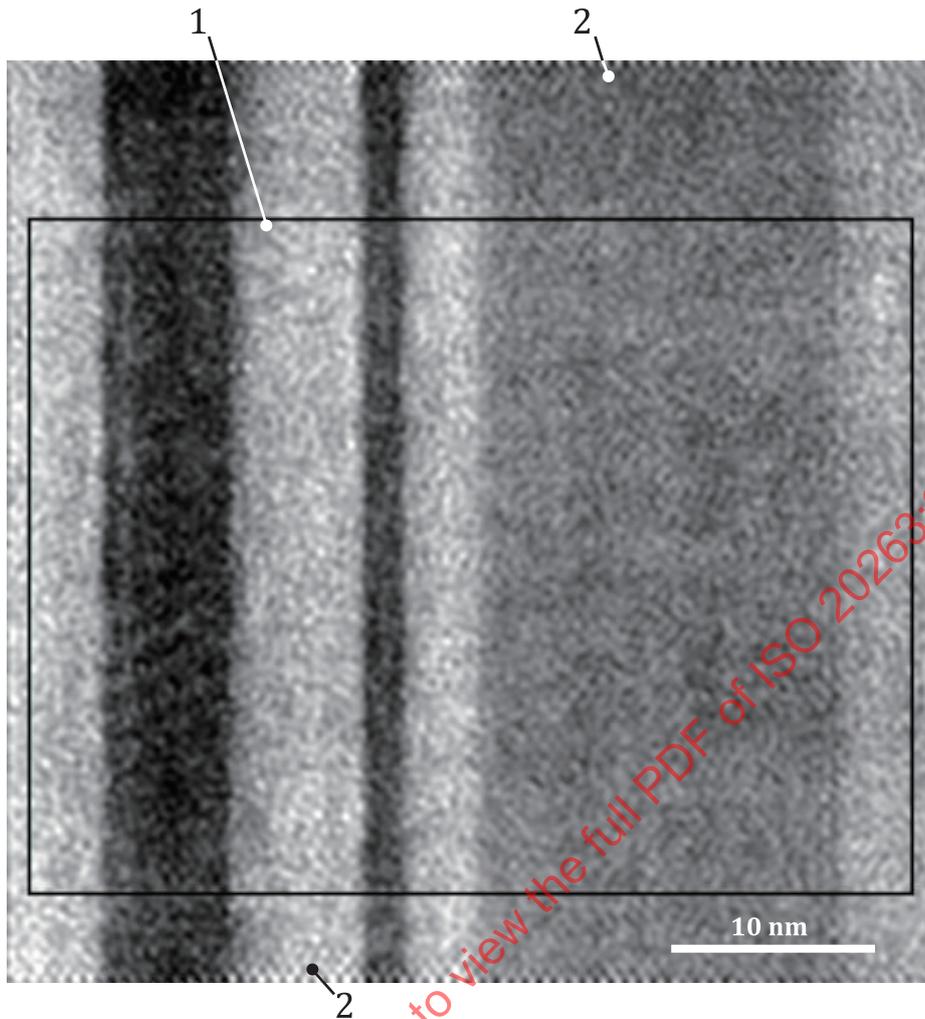

Figure A.13 — FFT pattern processed from Figure A.11
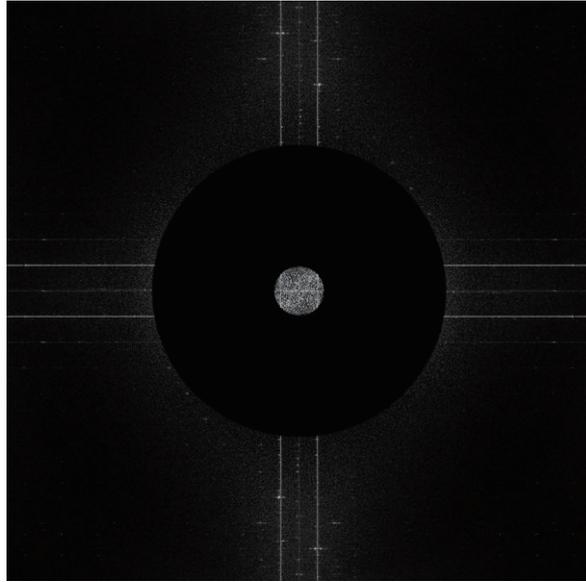
Figure A.14 — Low-pass filtering mask superimposed to the FFT pattern
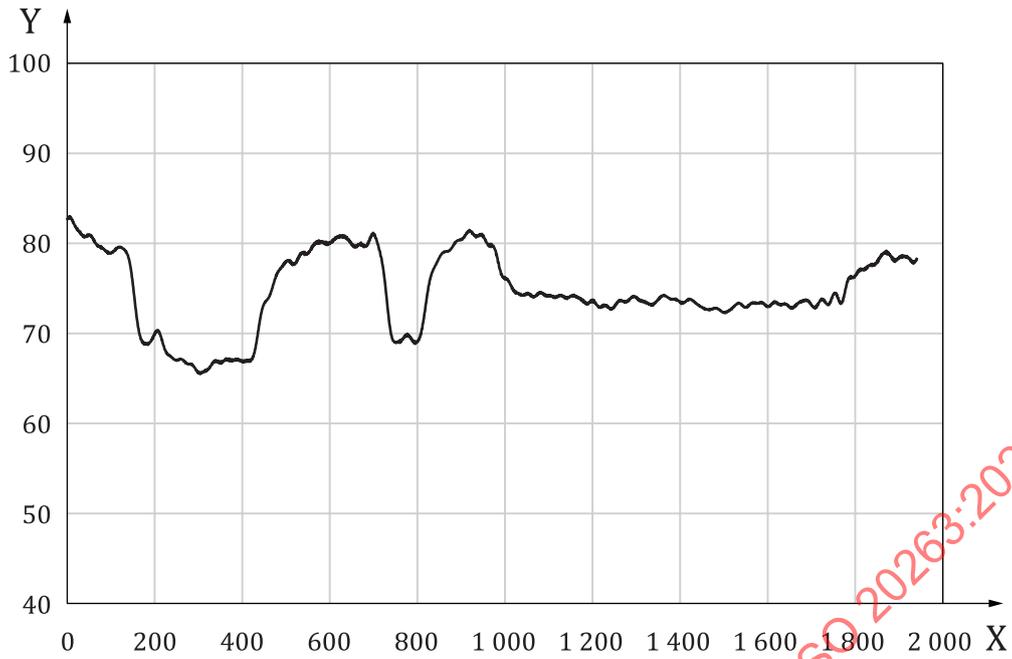
Key

1   ROI frame; 40 nmW × 30 nmH
2   artefact region

Figure A.15 — IFFT image processed with low pass filtering to the FFT pattern

A.4.3 Acquisition of the averaged intensity profile

Figure A.16 shows the averaged intensity profile for 1 940 lines in the ROI frame shown as key 1 in Figure A.15.
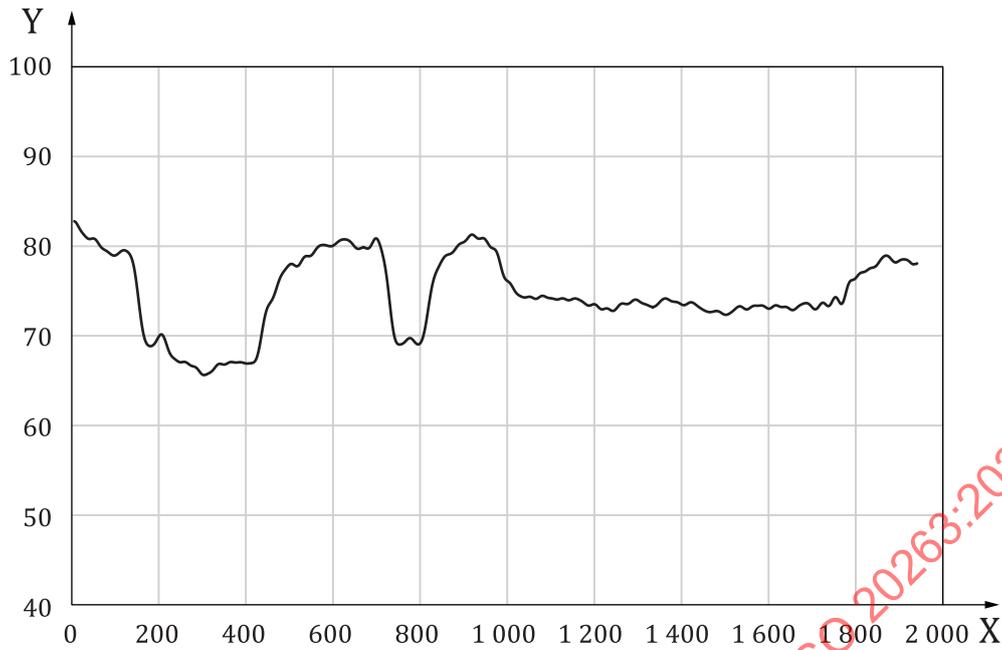
Key

X   position (pix)

Y   averaged intensity

Figure A.16 — Averaged intensity profile for 1 940 lines in ROI

A.4.4   Acquisition of the moving-averaged processing

Figure A.17 shows the moving-averaged intensity profile of the averaged intensity profile as shown in Figure A.16. To get this profile, a numerical value of 6 was applied as the moving range, n, in Formula (3).
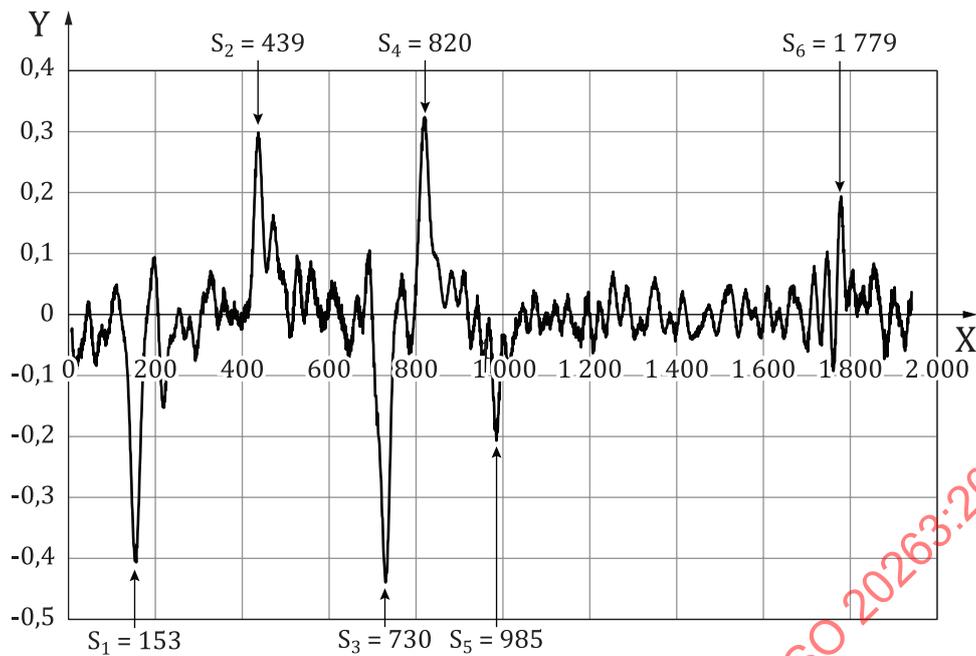
Key

X   position (pix)

Y   moving averaged intensity

Figure A.17 — Moving-averaged intensity profile, n = 6, applied to Formula (3)

A.4.5   Differential processing

Figure A.18 shows the result of the differential processing on the moving-averaged intensity profile as shown in Figure A.17.

Key

X   position (pix)

Y   rate of intensity change (ΔI)

Figure A.18 — Differential curve

A.4.6 Determination of interface position

Interface positions (S_1 to S_6) are determined as the pixel positions on the x-axis corresponding to the maximal or minimal peaks of the differential curve as shown in Figure A.18. The interface positions obtained are shown below.

$S_1 =$ 153 pix $S_4 =$ 820 pix

$S_2 =$ 439 pix $S_5 =$ 985 pix

$S_3 =$ 730 pix $S_6 =$ 1 779 pix