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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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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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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 documents 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*.

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 $(h_1,k_1,l_1 \ldots\ldots h_i,k_i,l_i)$ 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

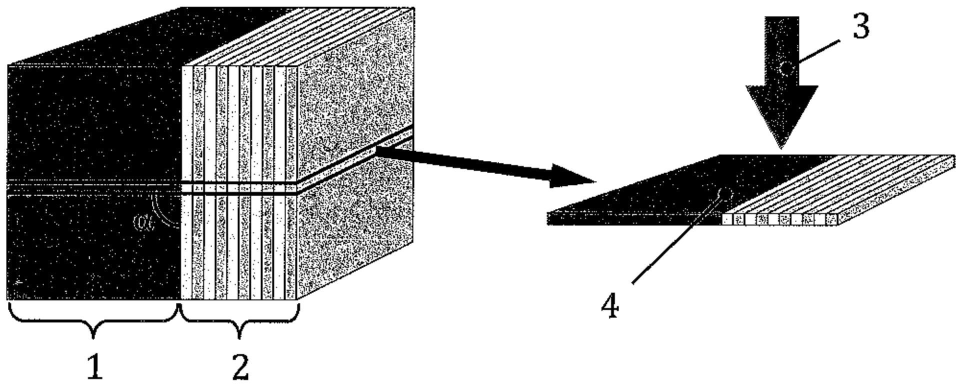
STEM Scanning transmission electron microscope/microscopy

TEM Transmission electron microscope/microscopy

4 Specimen preparation for cross-sectional imaging

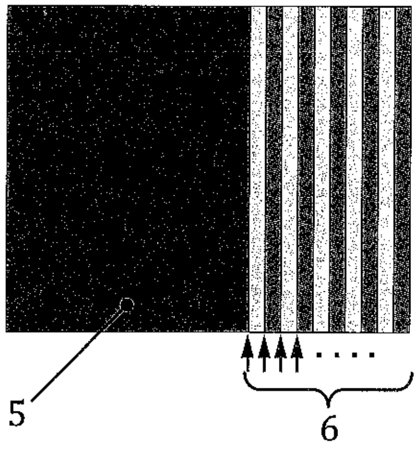
4.1 General

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

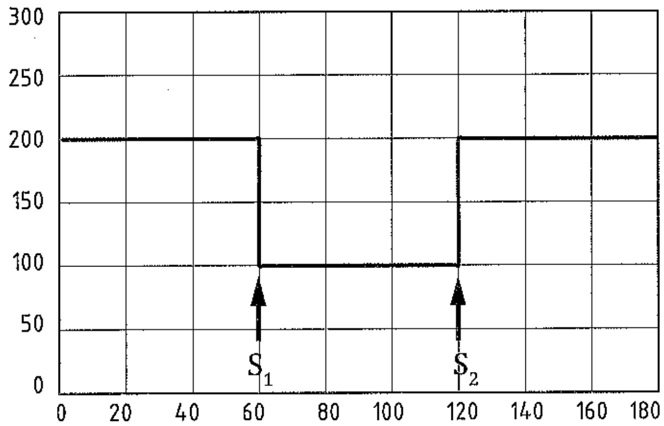
It is important to determine the position of an interface in a layered material from its cross-sectional TEM/STEM/elemental mapping image, objectively and uniquely. In this clause, the main scheme for the determination of the interface position, as prescribed by this document, is explained.

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

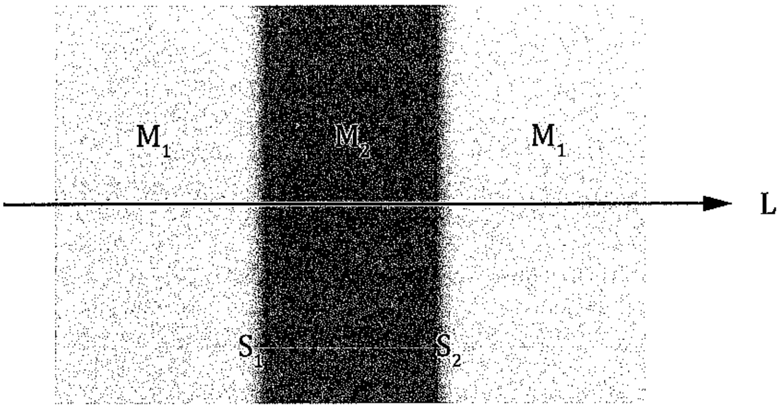


b) Intensity profile along an arrow line, L, in a), perpendicular to the interfaces (S_1 and S_2)

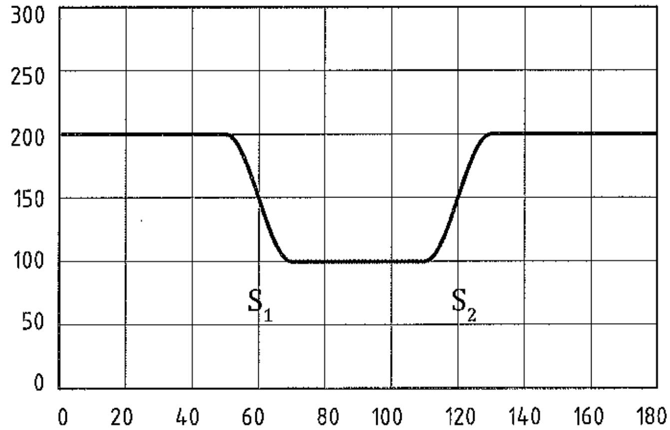
Figure 2 — Ideal interface model

5.2.2 More realistic model of an interface

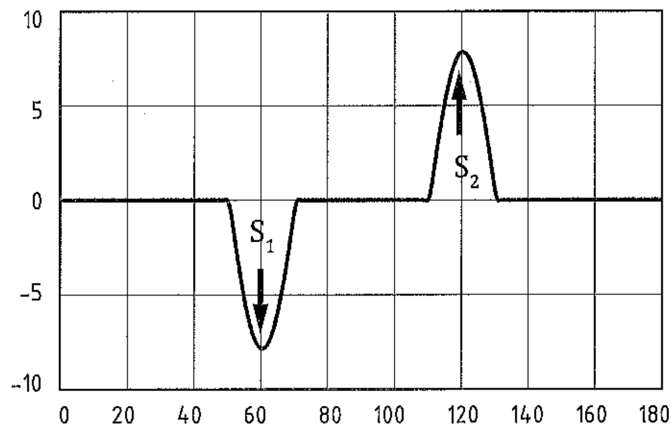
However, in general, the interface will not be in a straight line. It is a region with gradated intensity distribution between layers M_1 and M_2 [see Figure 3 a)]. In this case, it is not easy to find the accurate interface position from its intensity profile which is normally s-shaped [see Figure 3 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.

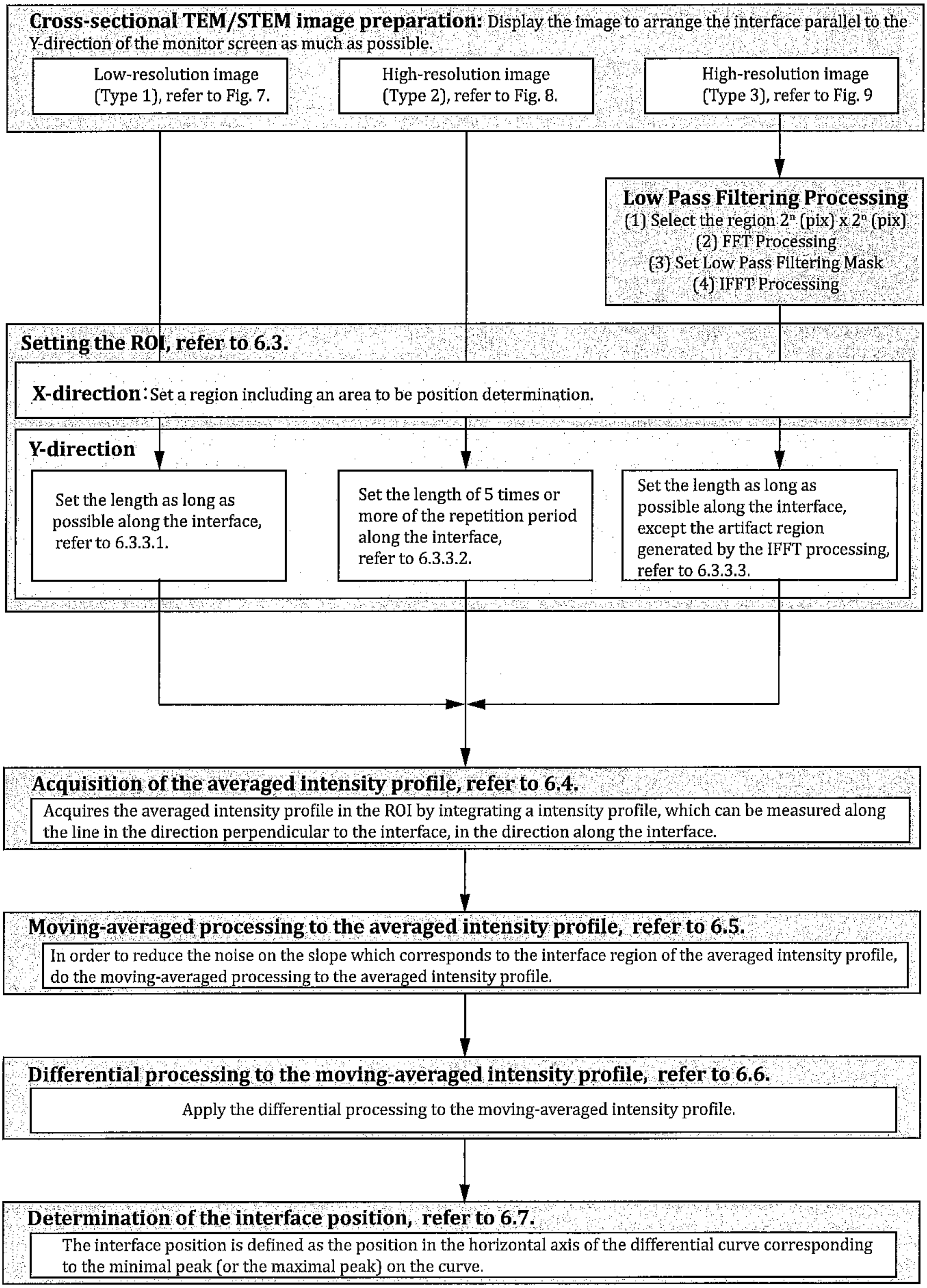


Figure 4 — Flow chart of interface position determination procedure

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 larger than 8 bits. 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 resolution 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 by using image scanner with resolution more than 1 200 dpi.

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 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 to 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.

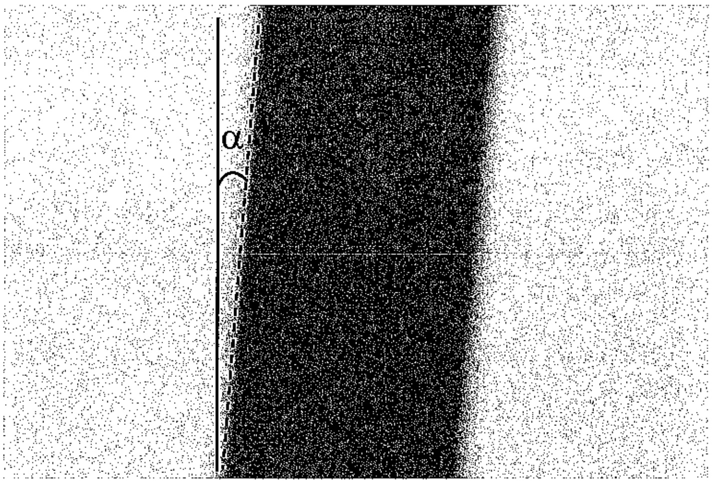
— For saving the digitized images, an uncompressed image file format (ESP, PICT, TIFF or Windows bitmap), or reversible (lossless) compressed image file format (GIF or PING) shall be used.

— 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

[broken line] interface

[solid line] 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.

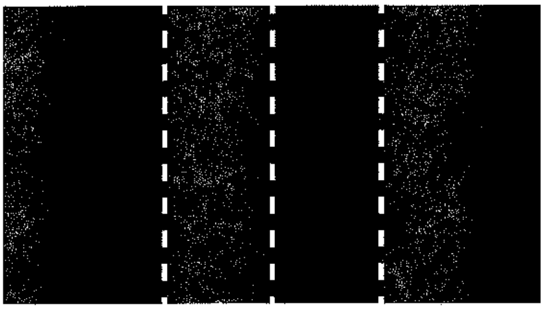
6.3.2 Classification of image

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.

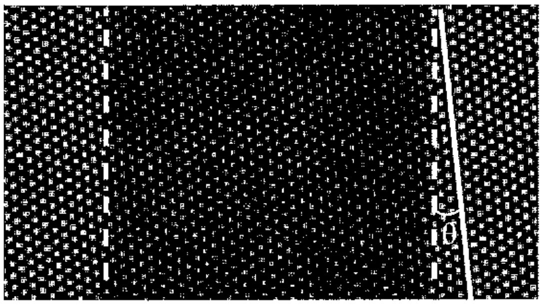
Type 1: Low resolution images in which atomic column arrangements cannot be recognized. Figure 6 a) shows an example.

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

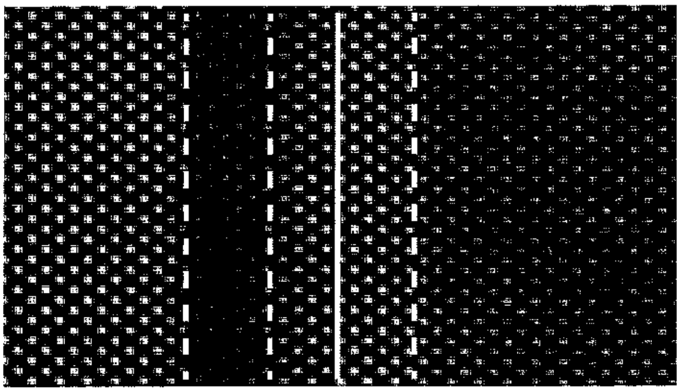
Type 3: High resolution images in which atomic column is parallel to the interface [see Figure 6 c)].



a) Type 1 image



b) Type 2 image



c) Type 3 image

Key

[broken line] interface direction

[solid line] atomic column arrangement direction

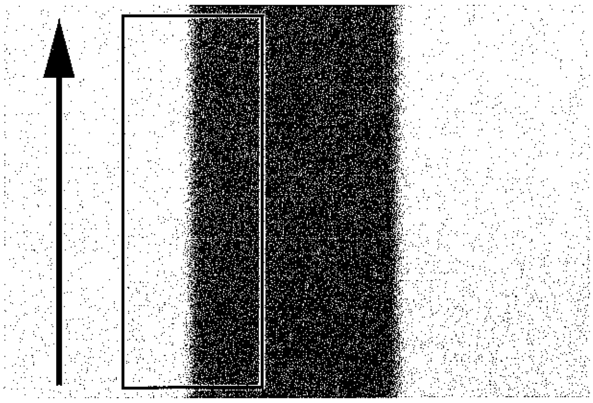
Figure 6 — Examples of classified target image

6.3.3 Procedure of setting the ROI

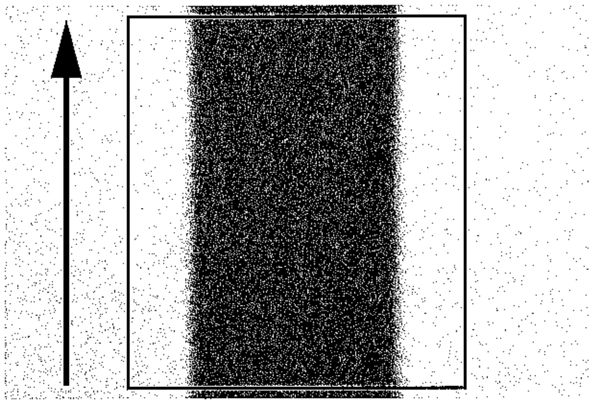
6.3.3.1 ROI for type 1 image

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

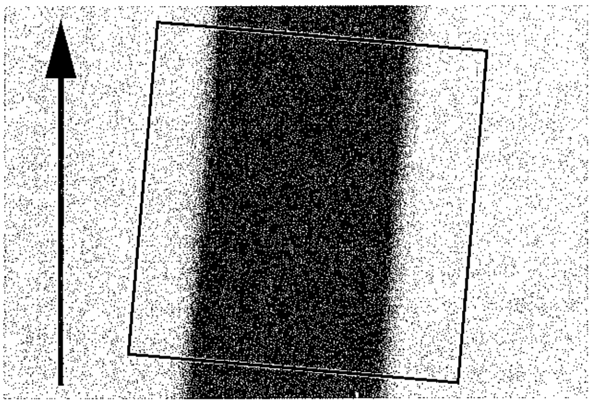
Figure 7 shows the examples of ROI settings for the type 1 image model. In a very low magnification image, image distortion is sometimes observed at the edges. In such case, exclude the area containing distortions from the ROI setting. Figure 7 a) and b) show examples of setting the ROI when the interface 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; its one side 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

[square frame] region of interest (ROI)

[solid arrow line] 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

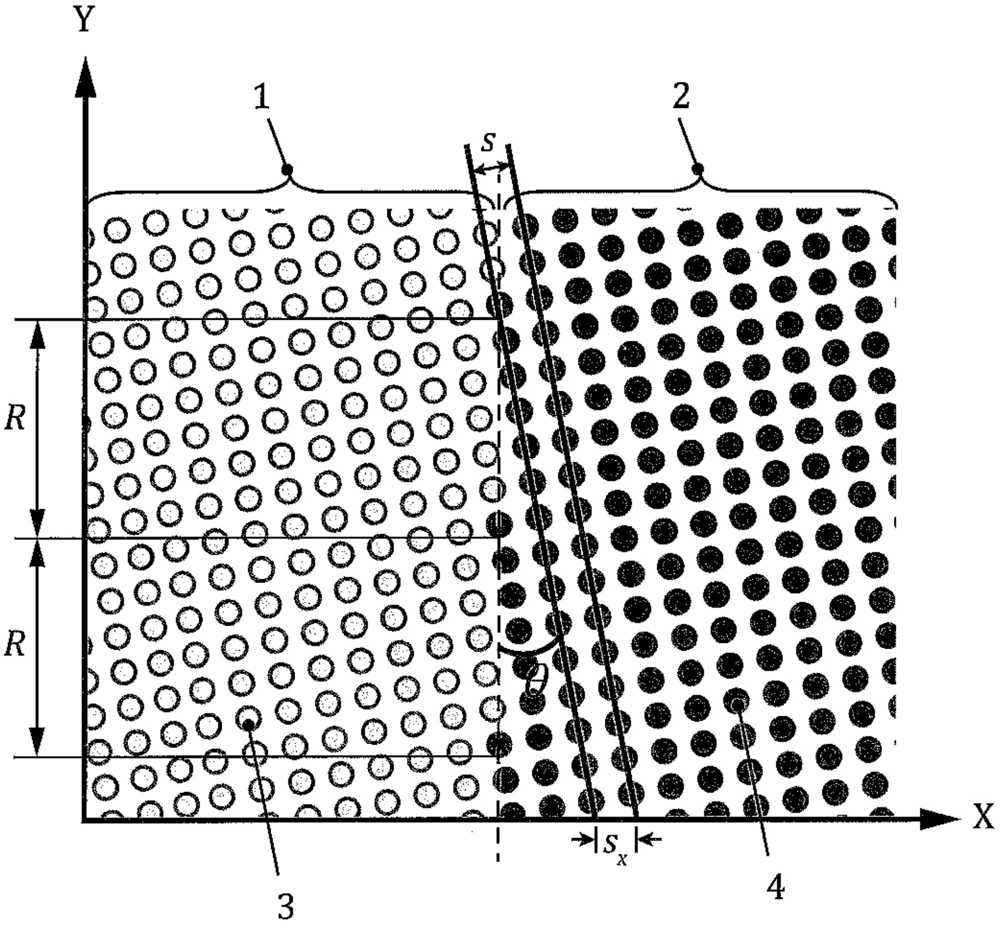
A schematic model of the type 2 image is shown in Figure 8. Repetition period, R, of the atomic column arrangement along the interface can be calculated by using Formula (1):

$$R = s/\sin\theta = (s_X \times \cos\theta)/\sin\theta \qquad (1)$$

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 (degree)	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 direction

[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].

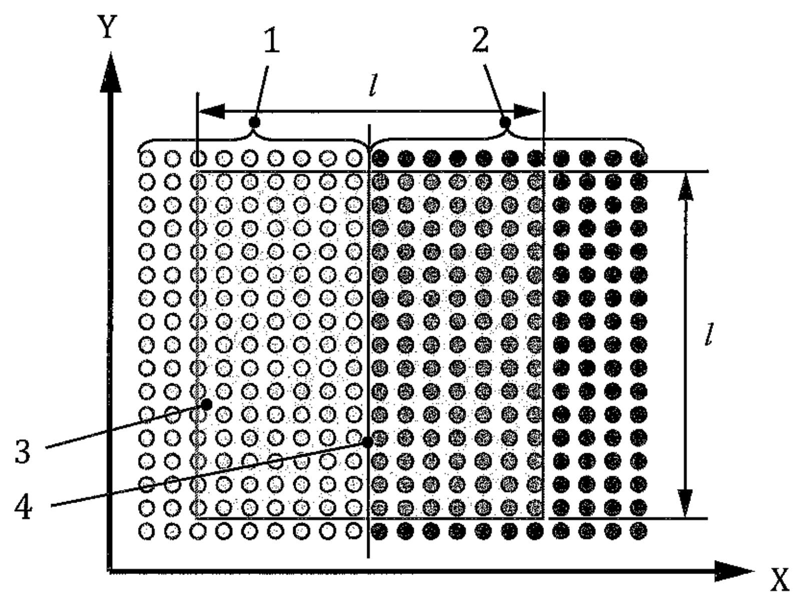
In order to get a clear FFT pattern, it is desirable to set n ≥8 to include the largest possible area.

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

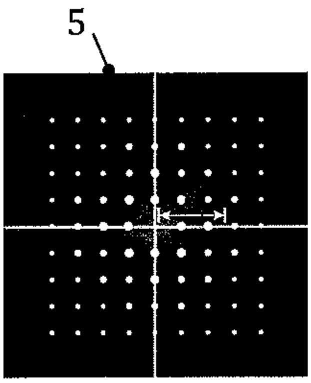
c) Set low pass filtering mask [Figure 9 c), key 6] onto the pattern generated by FFT processing to the region F. The mask has a circular opening having diameter d_m = (0,9~0,95)*d_f (where, d_f is the distance between primary spots) centred on the central spot, to set the circumference as close as possible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

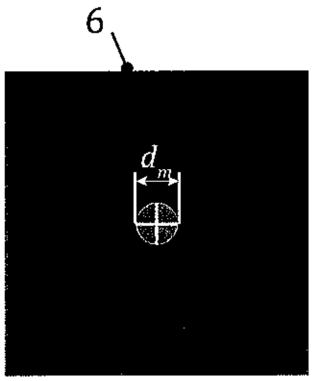
d) Apply an IFFT processing to the FFT pattern with low pass filtering mask. [Figure 9 d), key 7 shows the result of IFFT processing.]



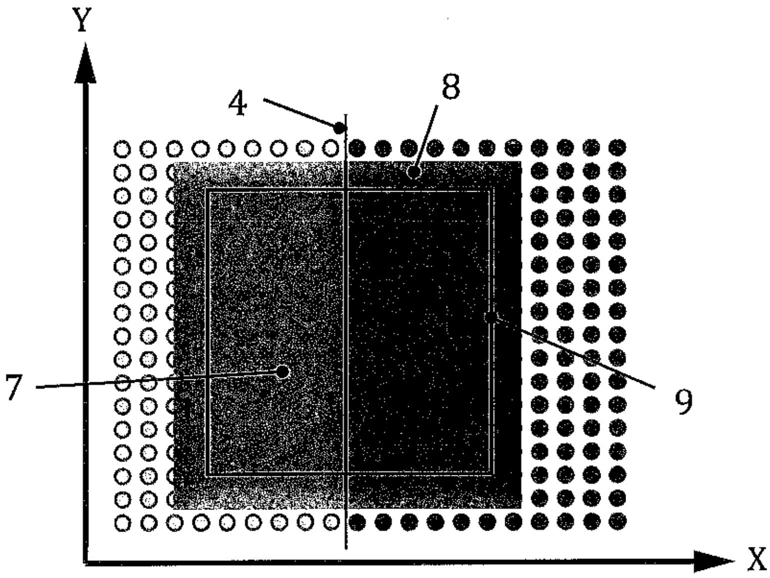
a) Type 3 image model



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 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

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

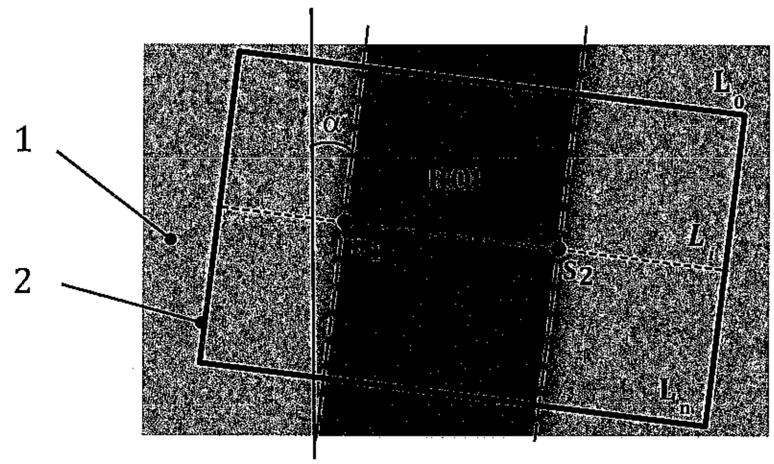
Figure 9 — Examples of type 3 image model in which atomic columns are aligned in direction perpendicular to the interface

6.3.3.3.2 Setting the ROI

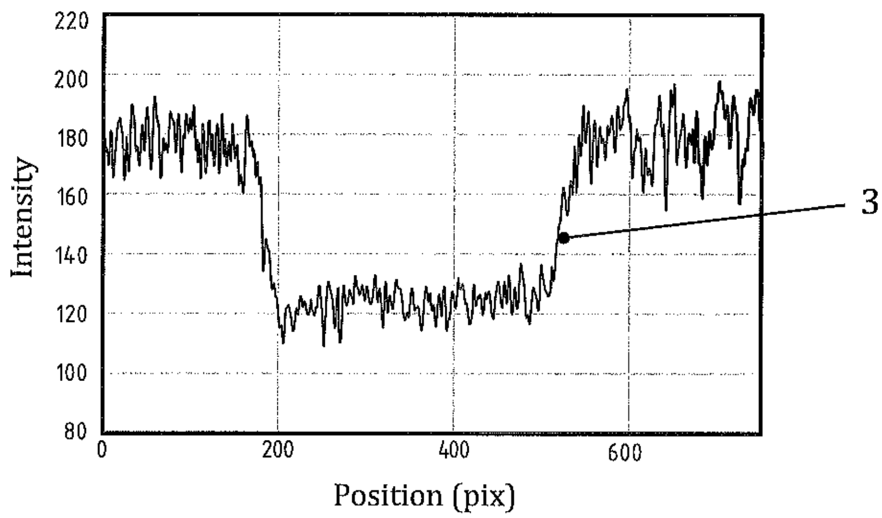
Set ROI [Figure 9 d), key 9] into the IFFT image [Figure 9 d), key 7], except the artefact region [Figure 9 d), key 8] observed at the edge of the IFFT image [Figure 9 d), key 7]. If the interface is inclined to the y-axis, set the ROI so that one side of it is parallel to the direction of the interface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

6.4 Acquisition of the averaged intensity profile

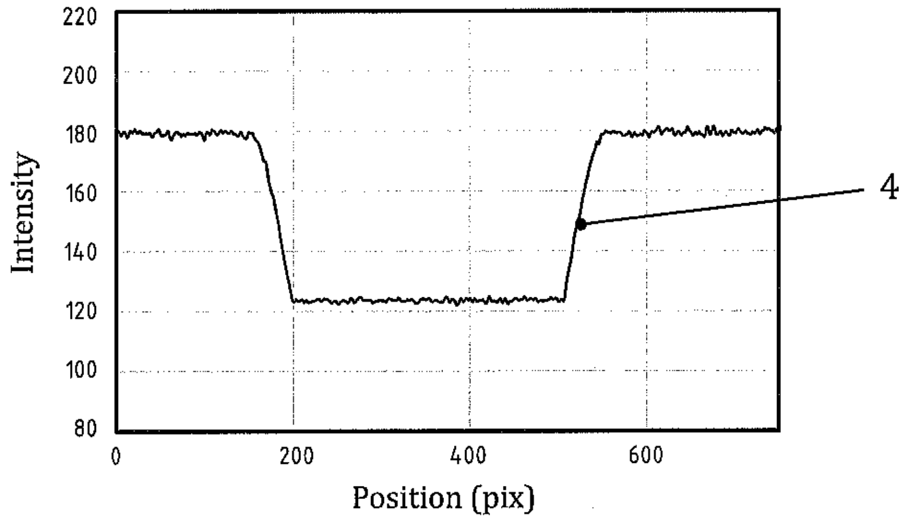
After setting the ROI into the target image, apply the same processing for all types of images. Herein, as an example, the process is applied to the type 1 (low resolution) image [Figure 10 a)].



a) Type 1 image model having an inclined interface



b) Intensity profile



c) Averaged intensity profile

Key

[broken line]	interface
[fine broken line]	line for intensity profile measurement
[solid line]	y-axis of the monitor screen
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_0 to L_n in key 2

NOTE 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 gray level of the image acquired. The gray level is dependent on the image detection system, in most cases, with 8-bit, 12-bit or 16-bit gray scale. It is possible to obtain the intensity profile by reading the gray 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.

$$I_a \equiv \frac{\sum_{i=0}^{n} I_i}{n+1} \tag{2}$$

where

I_i is i^{th} 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 [Figure 11 b), key 2] apply the moving-averaged processing by Formula (3).

$$I_p \equiv \frac{\sum_{j=p-n}^{p+n} I_j}{2n+1} \quad (3)$$

where

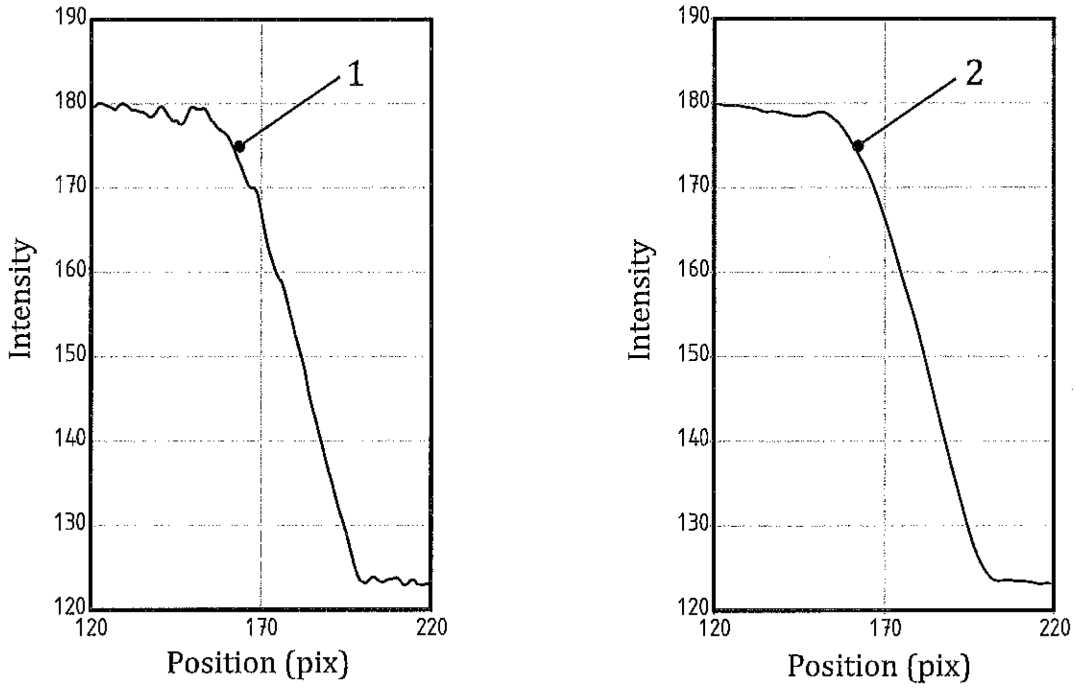
I_p is the calculated moving-averaged intensity of p^{th} pixel in the horizontal axis of the averaged intensity profile;

I_j is the intensity around the p^{th} 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 $2 \le n \le 7$ is suitable.

Key 1 Figure 12 a) 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

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) and key 2 in Figure 12 b)], by using Formula (4) and Formula (5).

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

$$\text{here, } 1 \le k \le n_p - 1 \tag{5}$$

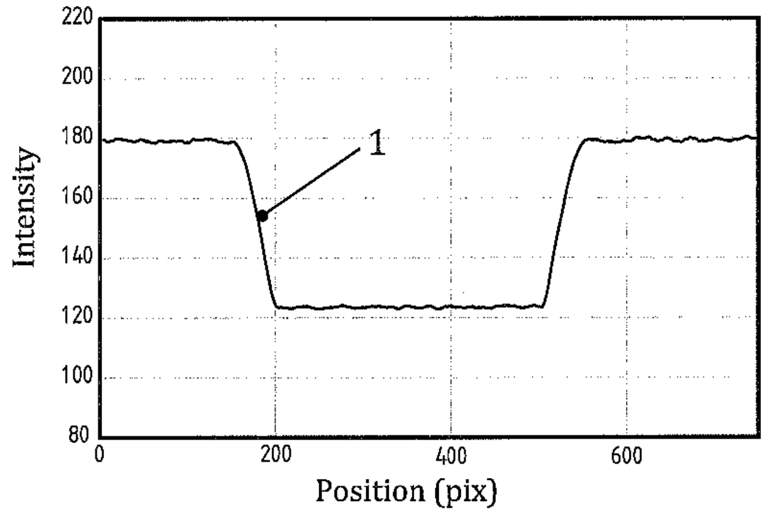
where

ΔI_k is the rate of intensity change of k^{th} pixel position in the horizontal axis of the moving-averaged intensity profile;

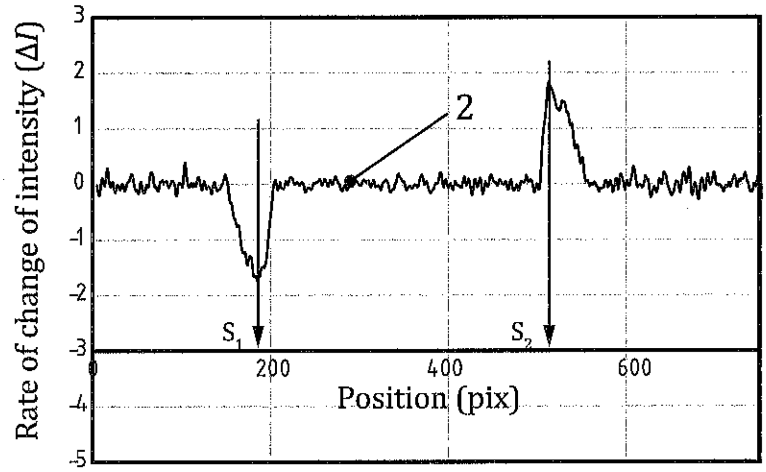
I_k is the intensity of the k^{th} pixel position in the horizontal axis of the moving-averaged intensity profile;

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



a) Intensity profile after moving-averaged processing



b) Location of interface position

Key

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 Final location of the interface

Throughout the determination of the position of the interface described in Clause 6, the interface position is identifiable by the pixel position in the horizontal axis of the moving-averaged intensity profile at the steepest slope in the profile. Therefore, the pixel position in the horizontal axis of the differential curve corresponding to the maximal peak or the minimal peak appearing on the curve indicates the interface position. 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 R_l times at different ROI settings, R_m times at different LPF settings and R_n times for MA settings, totally $R = R_l \times R_m \times R_n$ data set can be obtained. The uncertainty of each factor is expressed as follows in Formulae (6) to (8):

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

where D_i is the i-th data in the different ROI settings and D_{lavg} is the averaged value of the l-data set.

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

where D_j is the j-th data in the different LPF settings,and D_{mavg} is the averaged value of the m-data set.

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

where D_k is the k-th data in the different MA settings and 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):

$$\sigma_{\mathrm{ACC}} = \sigma_{\mathrm{ROI}} \times \sigma_{\mathrm{LPF}} \times \sigma_{\mathrm{MA}}, \quad (9)$$

Generally, $\sigma_{ROI} >> \sigma_{LPF}, \sigma_{MA}$, then the $\sigma_{ACC} = \sigma_{ROI}$, 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 the Guide to the Expression of Uncertainty in Measurement (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).

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

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 29301:2010, 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).

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

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.

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

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

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.

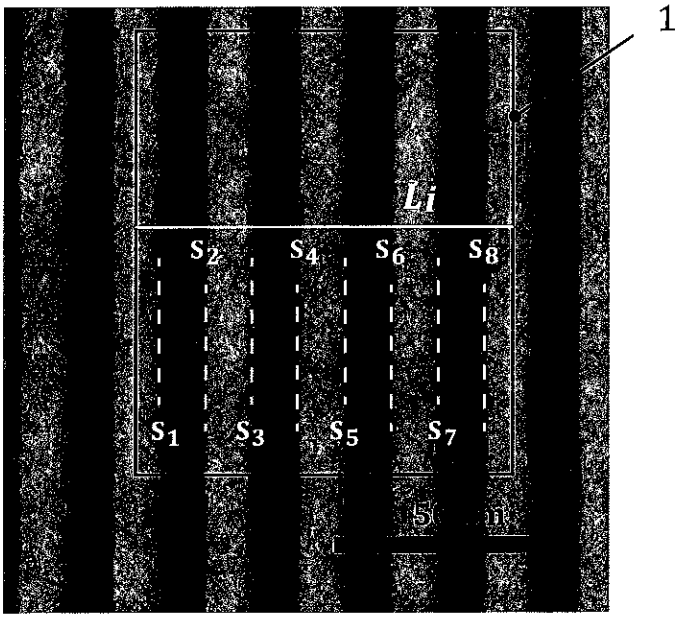
A.2 Processing example to type 1 image

A.2.1 Image used in a series of the processes

A 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

[broken white line] interface

[solid white line] line for intensity profile measurement

1 ROI frame

NOTE 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.

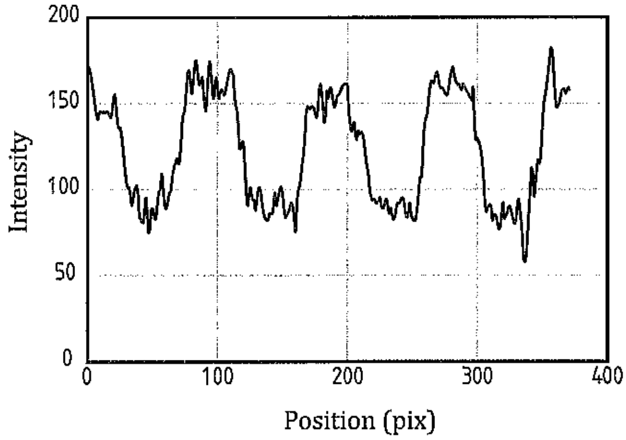


Figure A.2 — Intensity profile for line, *Li*

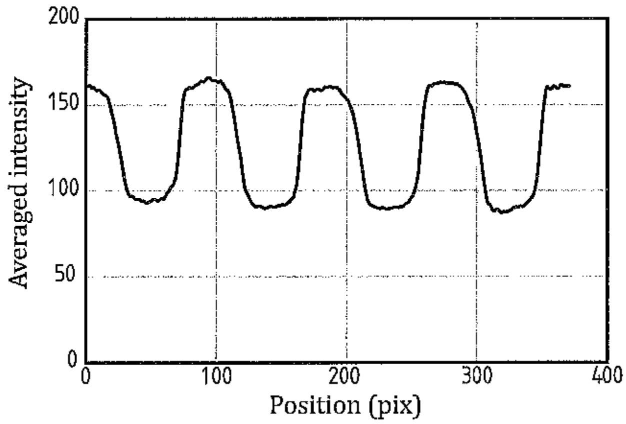


Figure A.3 — Averaged intensity profile for 450 lines in ROI

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).

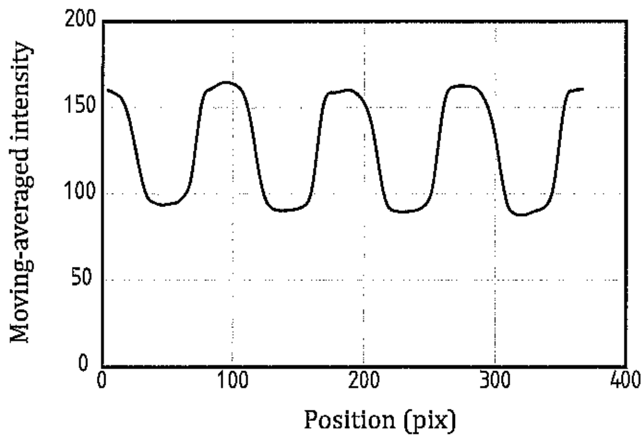


Figure A.4 — Moving averaged intensity profile, *n* = 4 applied to Formula (3)

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.

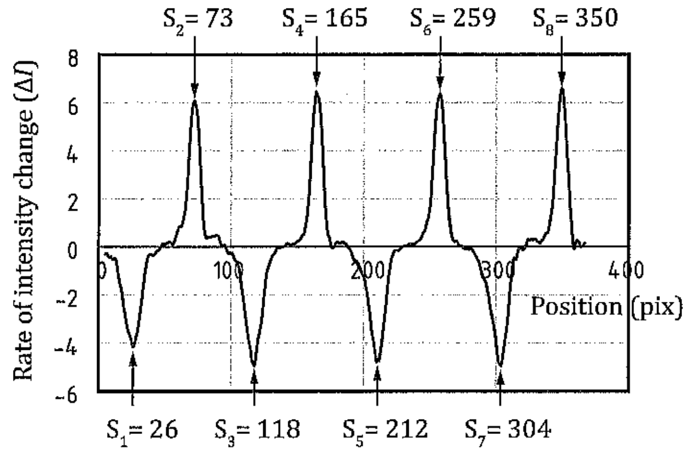


Figure A.5 — Differential curve

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

A 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

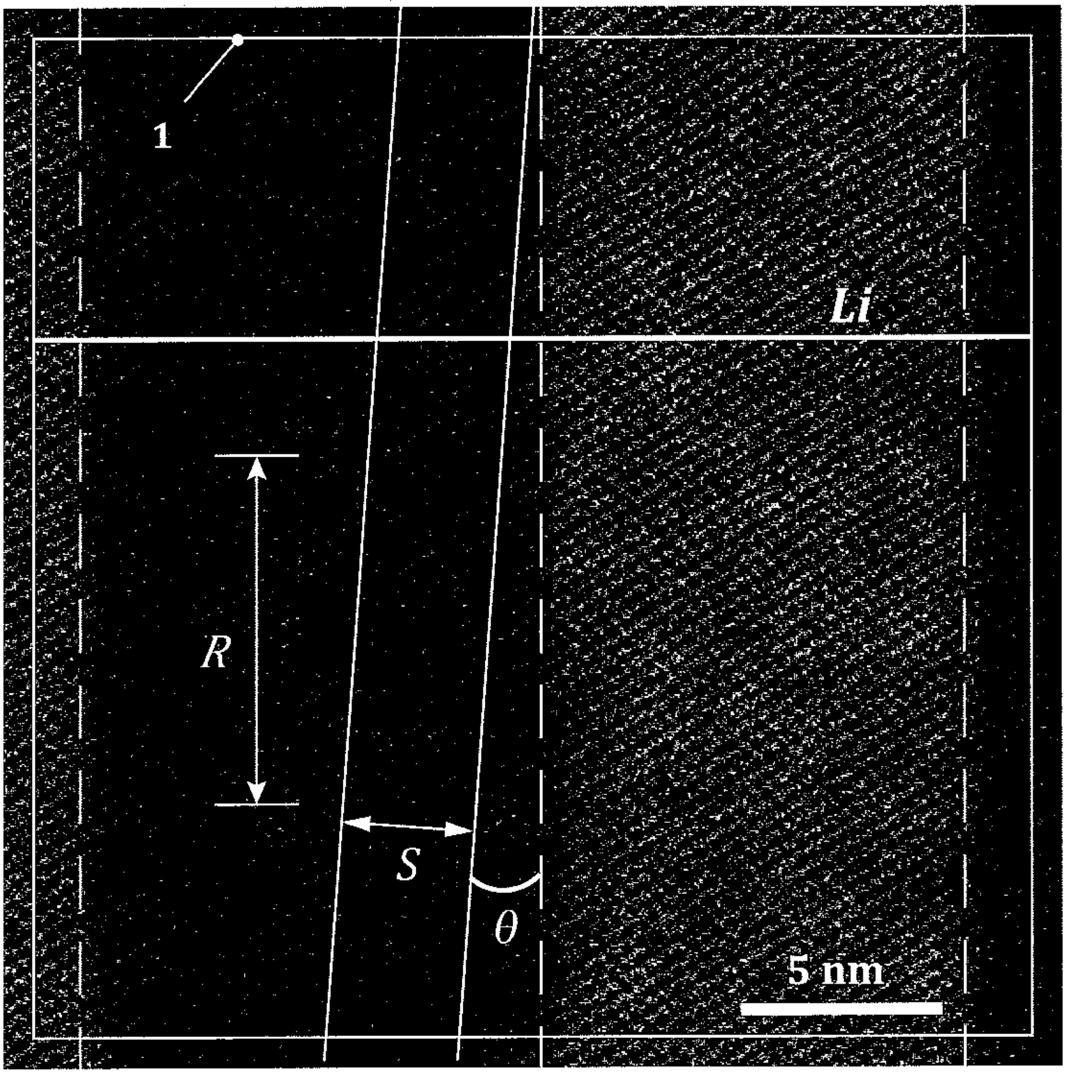
— Distance for 10 lines of atomic column: S = 2,83 nm

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

— Off-angle: θ = 4,35 degrees

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

— ROI setting: 25 nm × 25 nm



Key

[broken white line] interface direction
[thin solid line] atomic column arrangement direction
[thick white line] line for intensity profile measurement
1 ROI frame

NOTE The thick white 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.

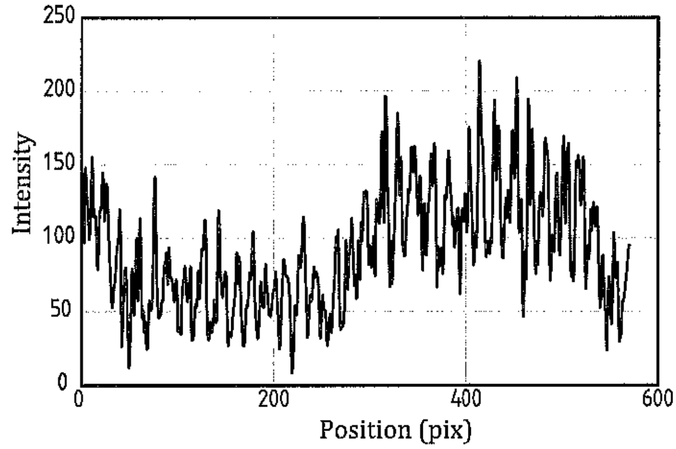


Figure A.7 — Intensity profile for line, *Li*, indicated in Figure A.6

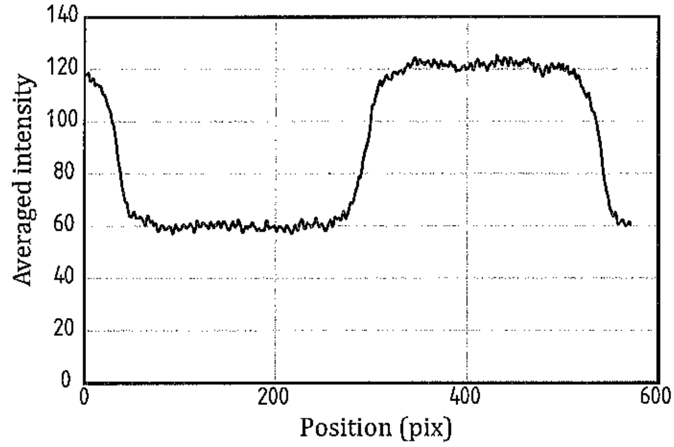


Figure A.8 — Averaged intensity profile for 570 lines in ROI

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).

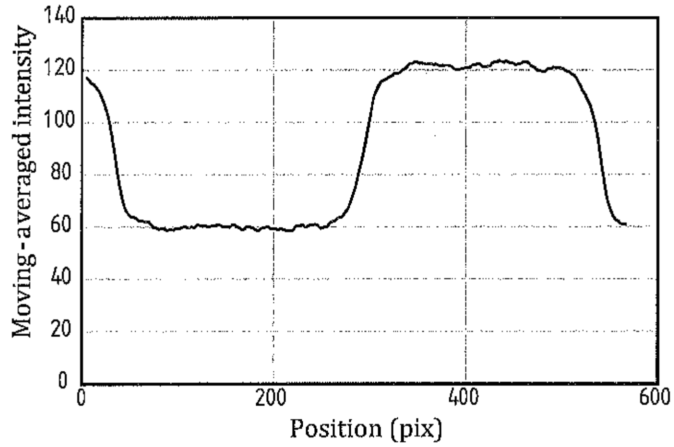


Figure A.9 — Moving-averaged intensity profile, n = 4 applied to Formula (3)

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.

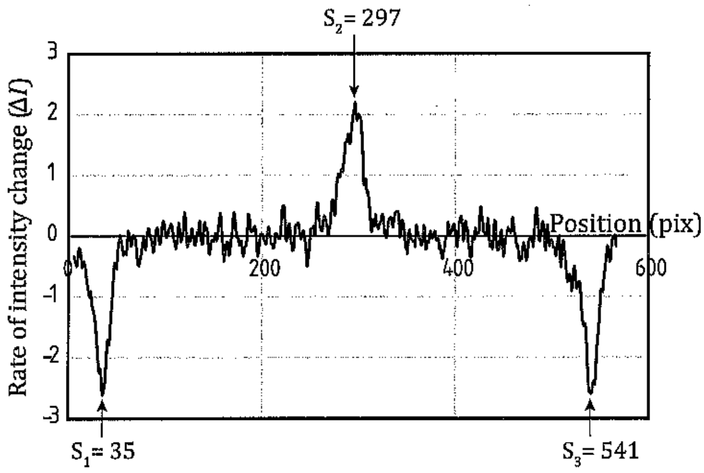


Figure A.10 — Differential curve

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

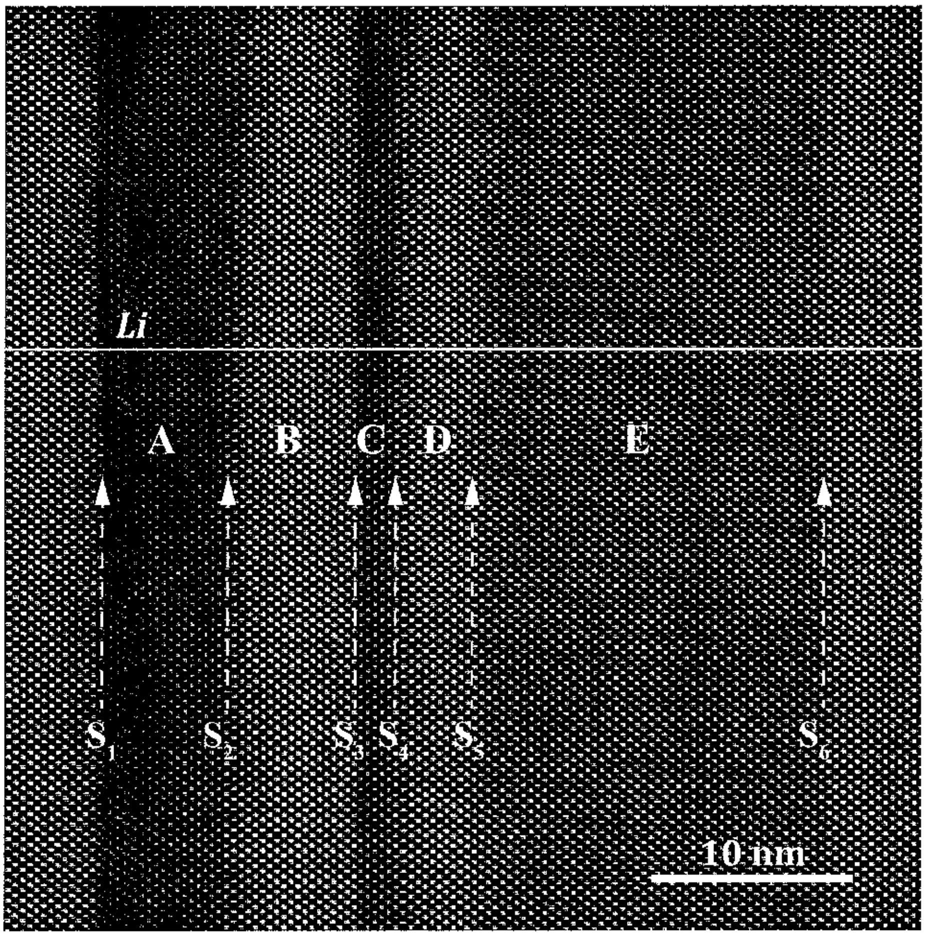
A high resolution image shown in Figure A.11 in which an atomic column arranged parallel to the interface is used in a series of interface determination processes. Information on this image is as follows.

— 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

[broken arrow line] interface position

[solid line] line for intensity profile measurement

NOTE 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 by the averaging process over than few hundreds lines. Therefore, low-pass filtering processing is required so that there is no influence of atomic column.

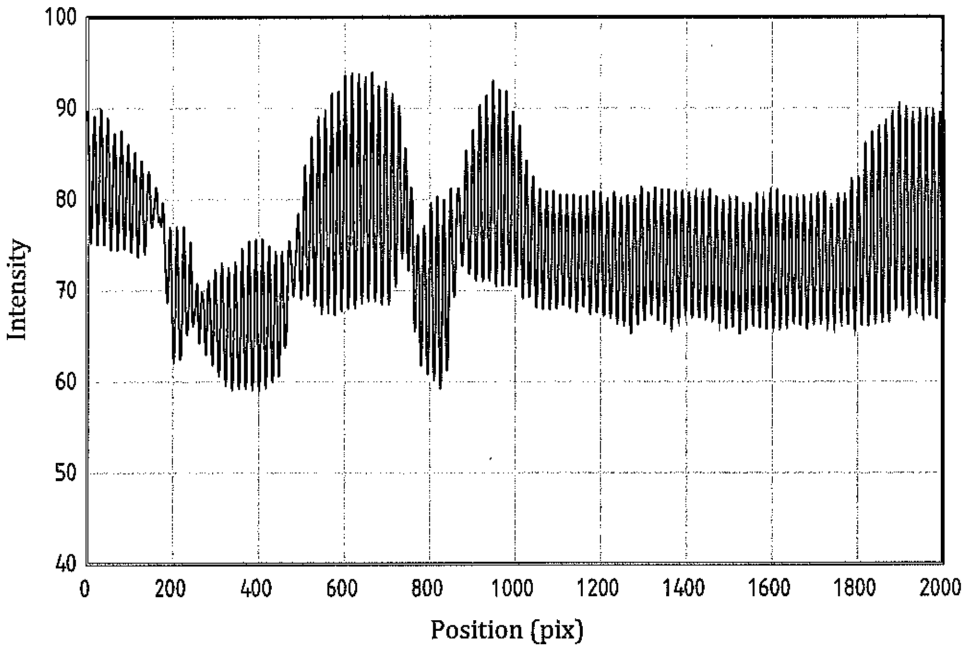


Figure A.12 — Intensity profile for line, *Ll*, indicated in Figure A.11

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 are interrupted.

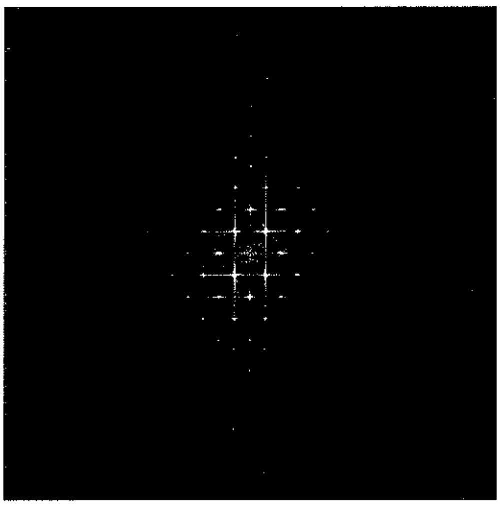


Figure A.13 — FFT pattern processed from Figure A.11

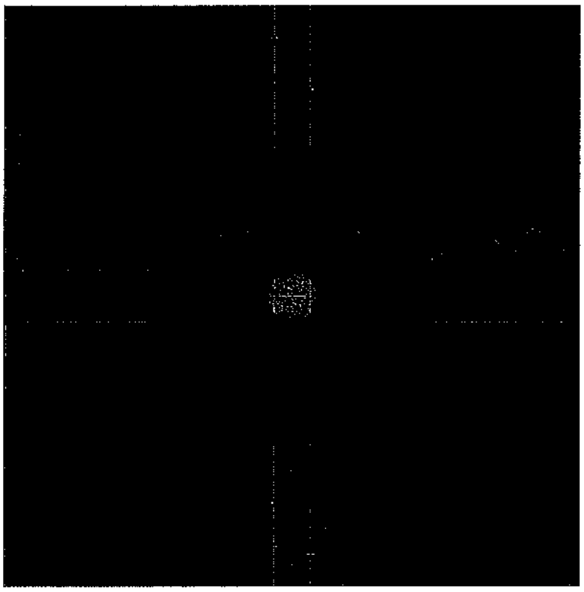
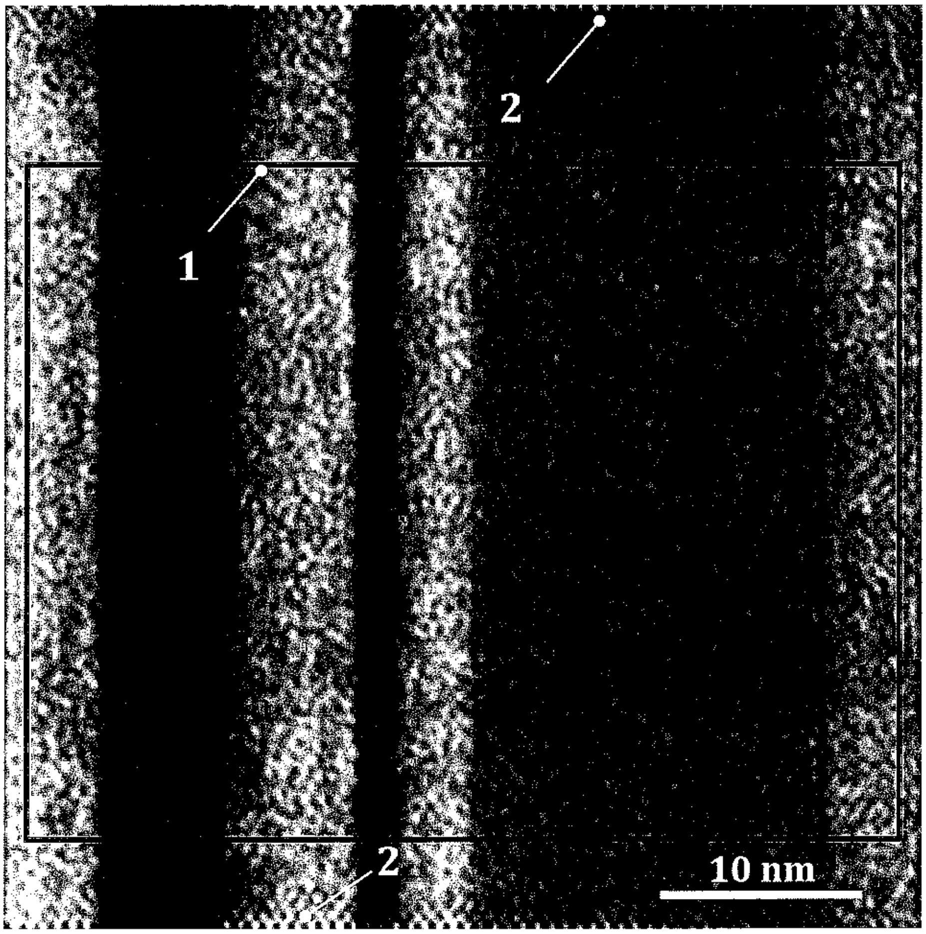


Figure A.14 — Low pass filtering mask superimposed to the FFT pattern



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.

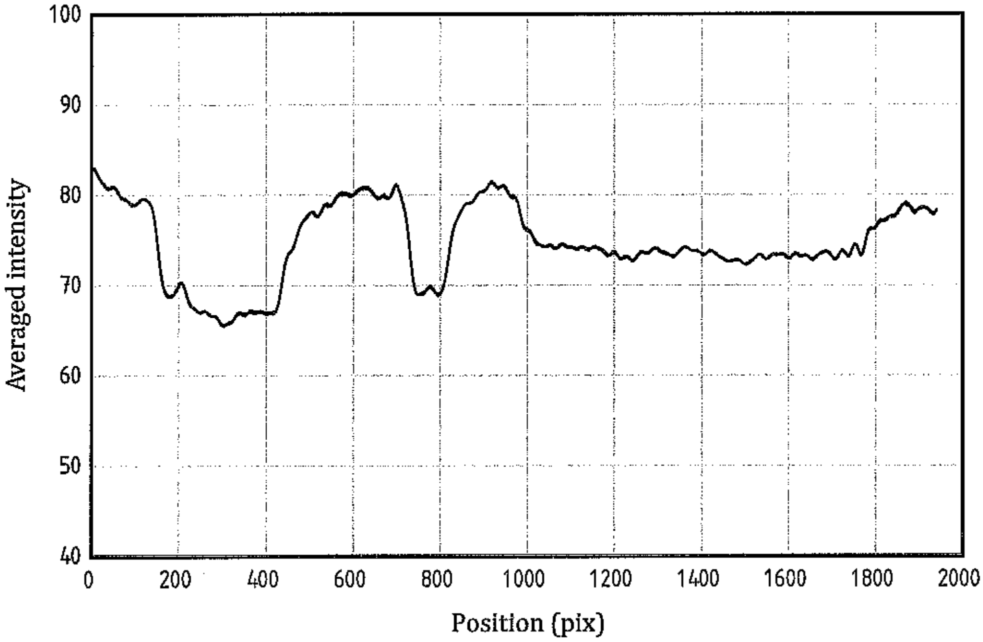


Figure A.16 — Averaged intensity profile for 1 940 lines in ROI

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).

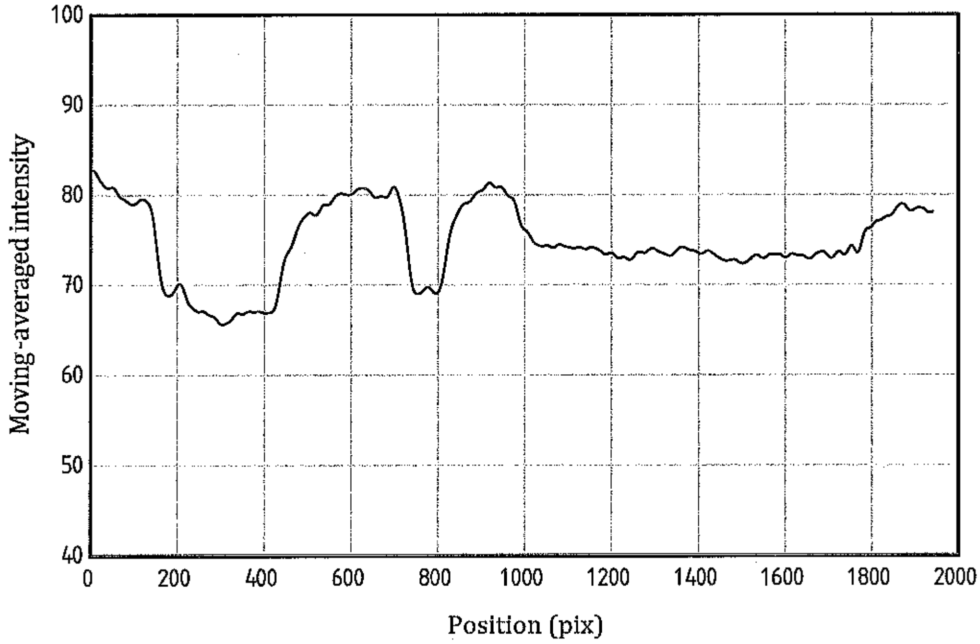


Figure A.17 — Moving-averaged intensity profile, n = 6, applied to Formula (3)

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.

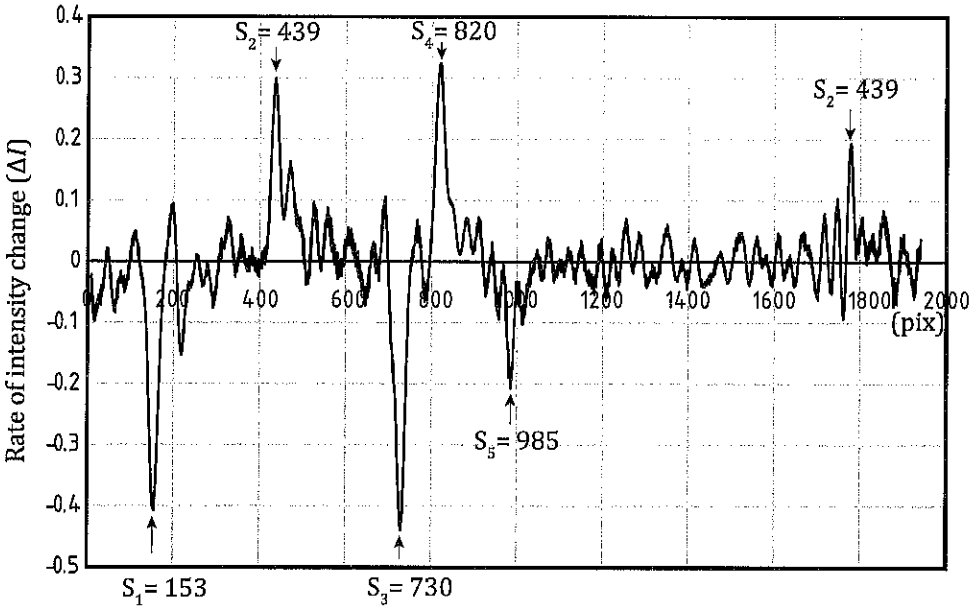


Figure A.18 — Differential curve

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

Annex B
(informative)

Two main applications for this method

B.1 General

There are two main applications using the processing indicated in this document; 1) measurement of layer thickness and 2) calibration of image magnification. The following examples for these applications are given for the convenience of users to understand how to use this method on them.

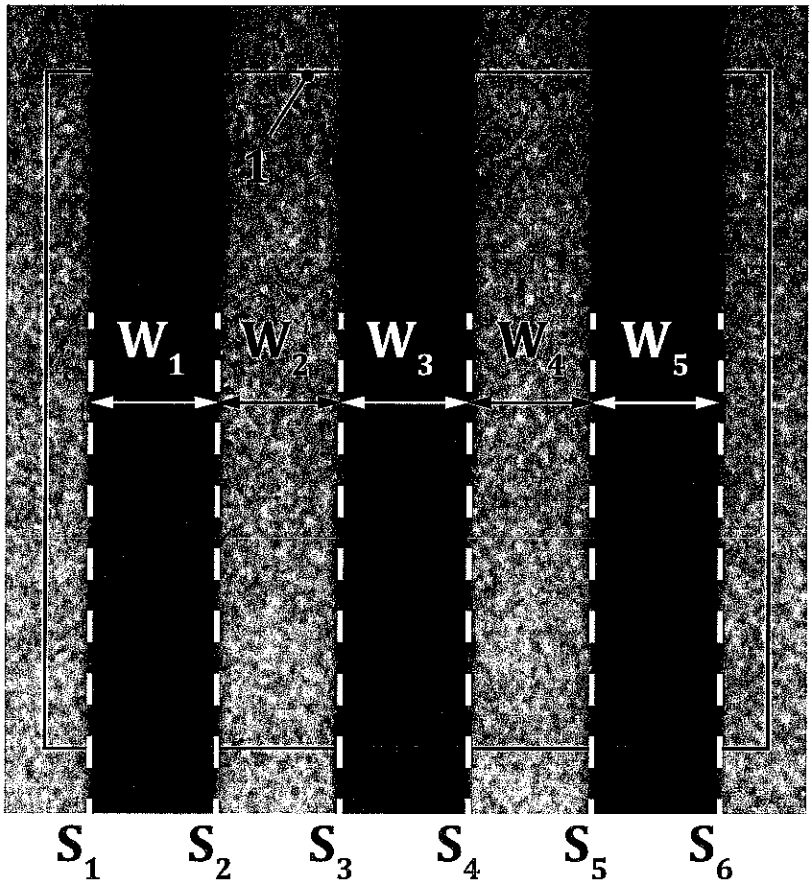
B.2 Application 1: Measurement of layer thickness

B.2.1 General

If the magnification of the TEM/STEM used is known, it is possible to measure the layer thickness objectively from the cross-sectional TEM/STEM image of the multi-layer thin film by using the method indicated in this document.

B.2.2 Image used for this application

Figure B.1 shows an example cross-sectional image of multi-layer thin film for this application. The procedure for determination of the layer thickness (W_1 to W_5) of each layer from this low resolution image (classified as a type 1 image) shown in Figure B.1 is explained in the next section



Key

[broken line] interface position (S_1 to S_6)

1 ROI frame (180 pixW × 150 pixH)

Figure B.1 — Example image of a multi-layered thin film having the width W_1 to W_5

B.2.3 Determination of interface position

It is necessary to determine the interface positions (S_1 to S_6) shown in Figure B.1, following the procedure written in Figure 4 (Flow chart, type 1 image).

Firstly, to get the averaged intensity profile, the ROI frame is set in the image (see key 1 in Figure B.1). Figure B.2 shows the averaged intensity profile for 150 lines in the ROI.

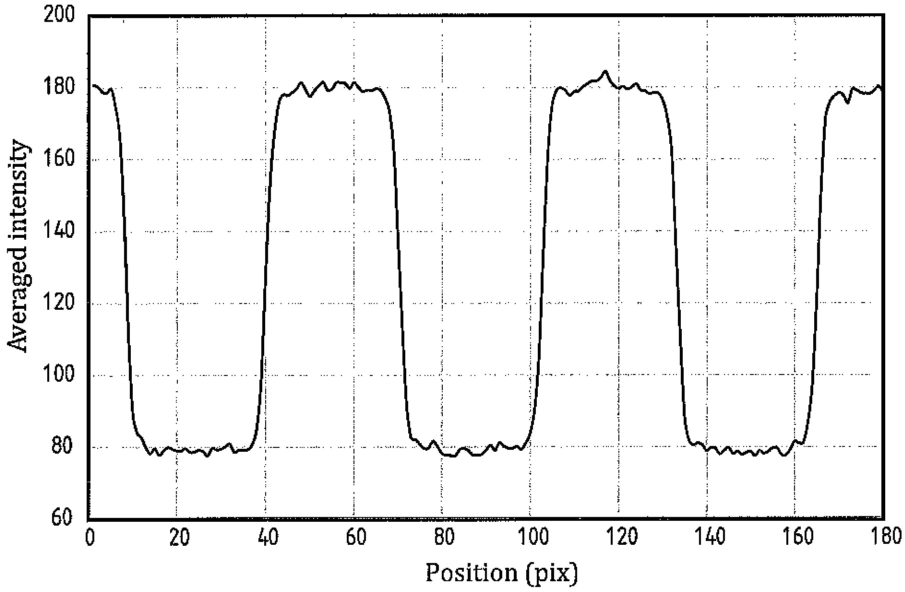


Figure B.2 — Average intensity profile for 150 lines in ROI

Next, moving-averaged processing of the averaged intensity profile is performed to remove residual noise in the slope of the profile. Figure B.3 is the processing result applied a numerical value of 2 as the moving range, *n*, in Formula (3).

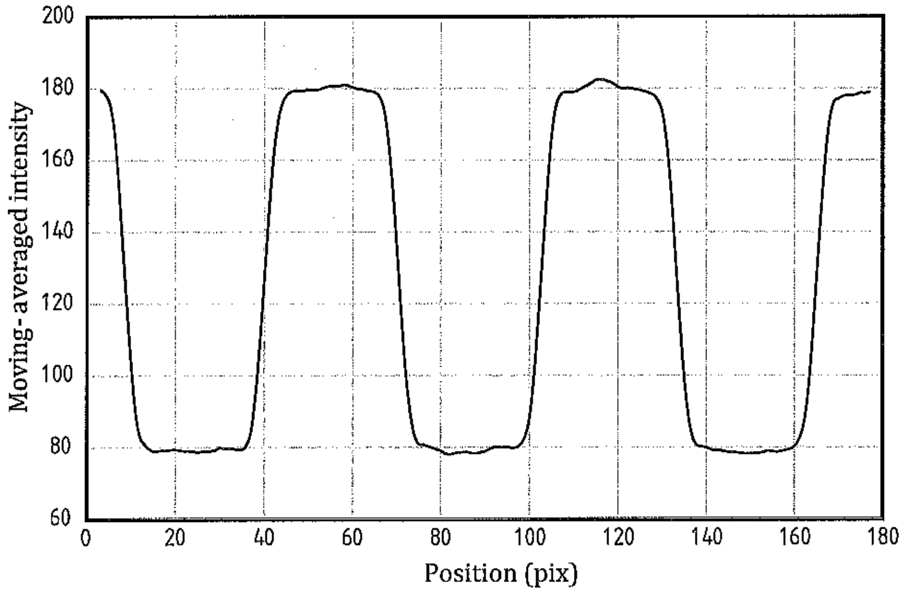


Figure B.3 — Moving-averaged intensity profile, *n* = 2, applied to Formula (3)

Then differential processing on the moving-averaged intensity profile is performed to clarify the interface position defined as the maximal or minimal position in the differential curve. Figure B.4 shows the result of differential processing.

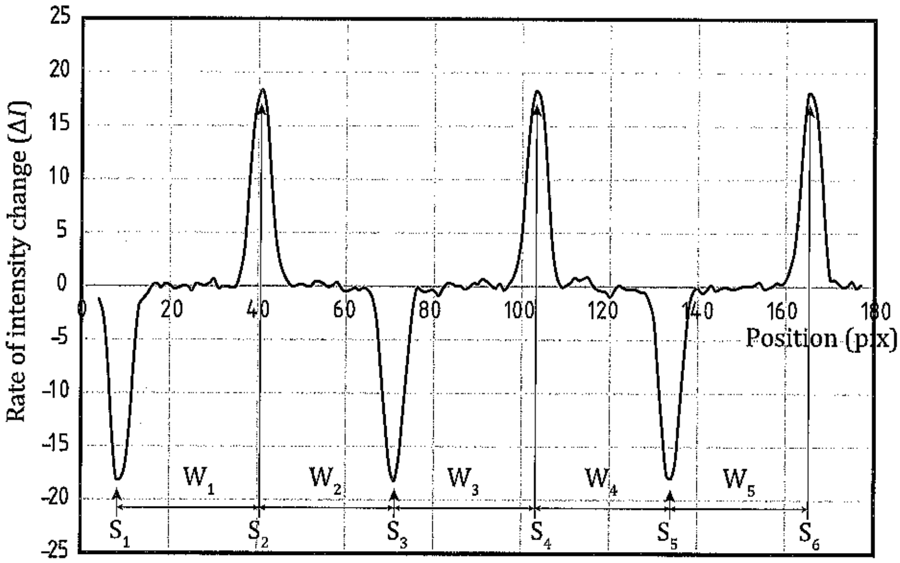


Figure B.4 — Differential curve

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 B.4.

B.2.4 Calculation of layer thickness

The layer thickness (W_1 to W_5) can be calculated by Formula (B.1).

$$W_n \equiv \frac{\left(S_{n+1} - S_n\right)}{M} \times u \text{ here, } n = 1 \sim 5 \tag{B.1}$$

where

- W_n is layer thickness (W_1 to W_5) of the thin film recorded in the image (See Figure B.1);
- S_n is the pixel position of the interface (S_1 to S_6) measured from the differential curve (see Figure B.4);
- M is the magnification of the image shown in Figure B.1;
- u is the pixel size of the image.

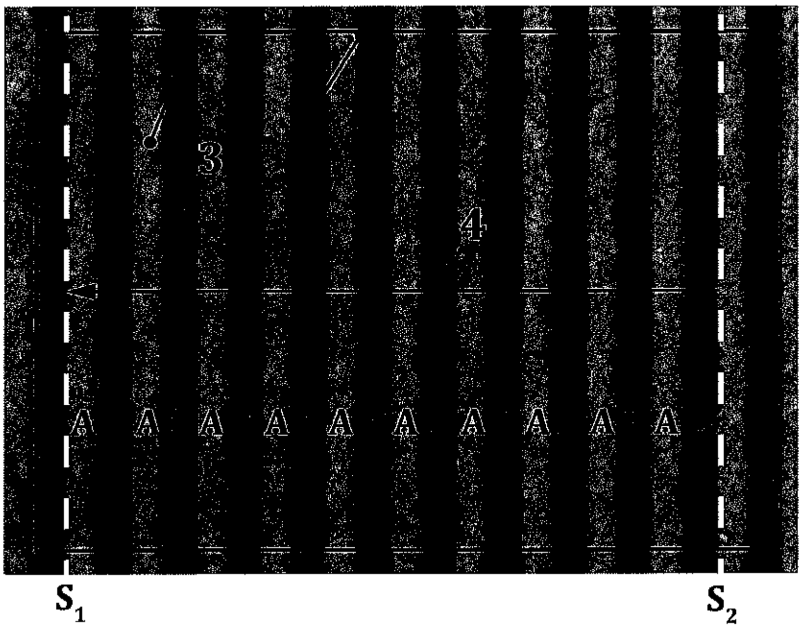
B.3 Application 2: Calibration of image magnification

B.3.1 General

If RM/CRM with known layer thickness is used as a specimen, the image magnification can be calibrated from the cross-sectional TEM/STEM image by applying the method indicated in this document.

B.3.2 Image used for this application

Figure B.5 shows an example cross-sectional image of RM using for this application. The specimen consists of two heterogeneous layers of A and B, having calibrated thickness of each layer being *Ma* and *Mb*, respectively.



Key

[broken line] interface position (S_1, S_2)

1 ROI frame (260 pixW × 180 pixH)

2 bright layer indicates the image of layer A having calibrated thickness, *Ma*

3 dark layer indicates the image of layer B having calibrated thickness, *Mb*

4 measurement width (W) in which 10 pairs of layer A and B are included

Figure B.5 — Example image of multi-layered thin film of RM/CRM having calibrated width *Ma* (μm) and *Mb* (μm)

The procedure for calibration of the image magnification is explained below.

B.3.3 Determination of interface position

When calibrating the image magnification, it is necessary to set the ROI as wide as possible in the recorded image. In this application example, ROI which is shown as key 1 in Figure B.5 is set for the region including 10 pairs consecutive stacked layers. Then, the image magnification is calibrated from the total thickness (W) of 10 pairs consecutive stacked layer, as shown in the key 4 in Figure B.5.

To measure W, it is necessary to determine the interface position of both ends (S_1 and S_2, shown in Figure B.5) of a 10 pairs consecutive stacked layer.

Firstly, it is necessary to get the averaged intensity profile in the ROI. Figure B.6 shows an example of the averaged intensity profile for 180 lines.

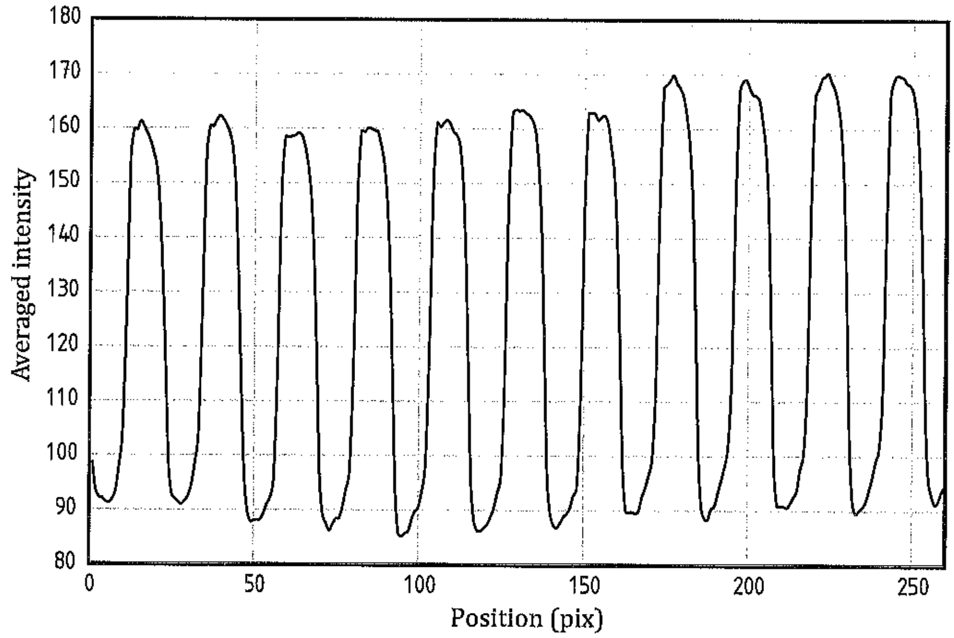


Figure B.6 — Averaged intensity profile for 180 lines in ROI

Next, moving-averaged processing of the averaged intensity profile is performed to remove residual noise in the slope of the profile. Figure B.7 is the processing result applied 2 as the moving range, *n*, in Formula (3).

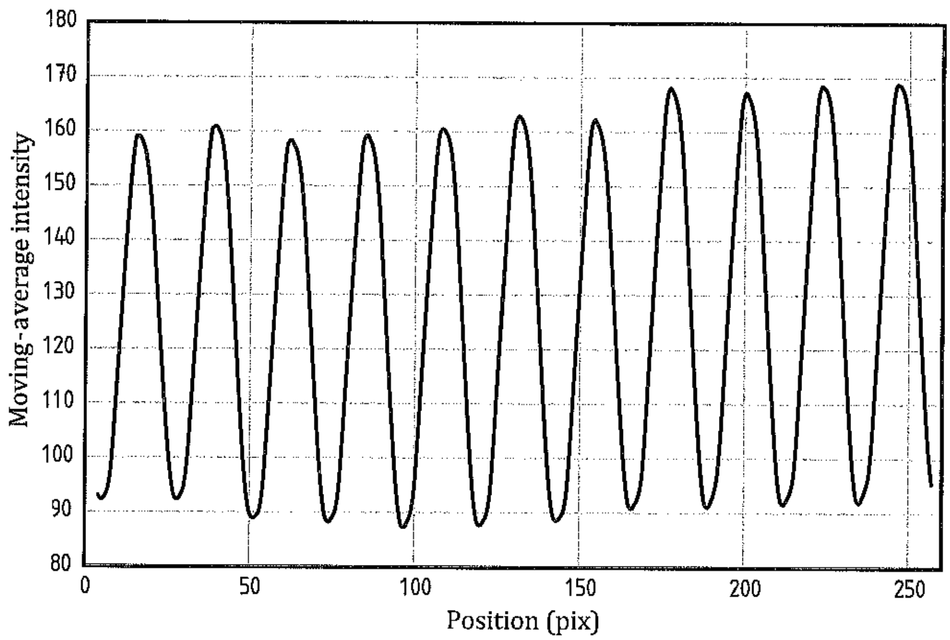


Figure B.7 — Moving-averaged intensity profile, $n = 2$, applied to Formula (3)

Then differential processing on the moving-averaged intensity profile is performed to clarify the interface position of both ends (S_1 and S_2, shown in Figure B.5) of the 10 pairs consecutive stacked layer. Figure B.8 shows the result of differential processing.

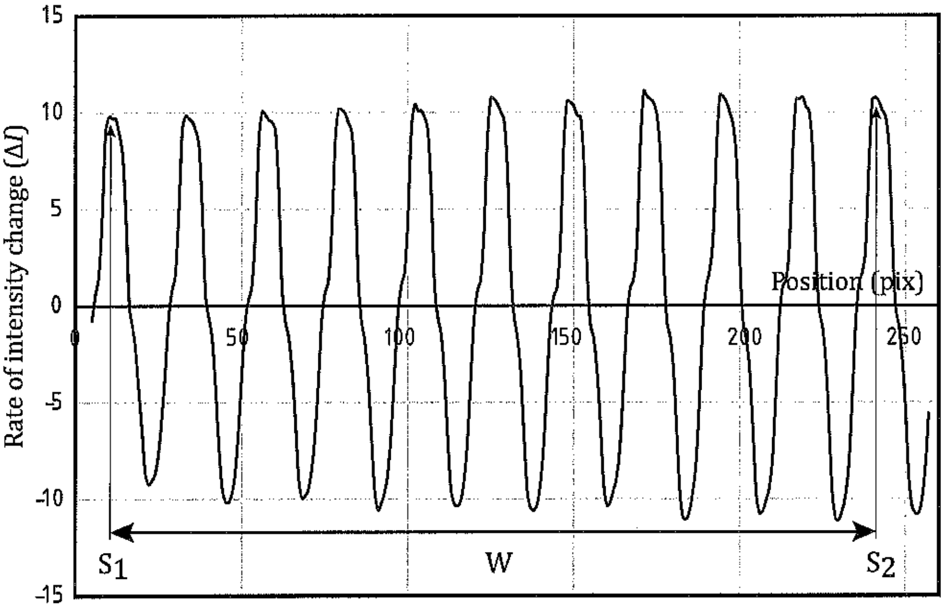


Figure B.8 — Differential curve

Interface positions (S_1 and S_2, shown in Figures B.5 and B.8) of both ends of a 10 pairs consecutive stacked layer are determined as the pixel positions on the x-axis corresponding to the maximal peaks of the differential curve as shown in Figure B.8.

B.3.4 Calibration of image magnification

The width (W), determined in pixel unit, between both interfaces (S_1 and S_2) of the 10 pairs consecutive stacked layer is expressed by the Formula (B.2).

$$W = (S_2 - S_1) \tag{B.2}$$

Then, the image magnification (M) can be calibrated from Formula (B.3).

$$M = \frac{W}{10 \times (Ma + Mb)} = \frac{(S_2 - S_1)}{10 \times (Ma + Mb)} \times u \tag{B.3}$$

where

Ma is calibrated thickness of layer A (See Figure B.5);

Mb is calibrated thickness of layer B (See Figure B.5);

u is the pixel size of the image.

Annex C
(informative)

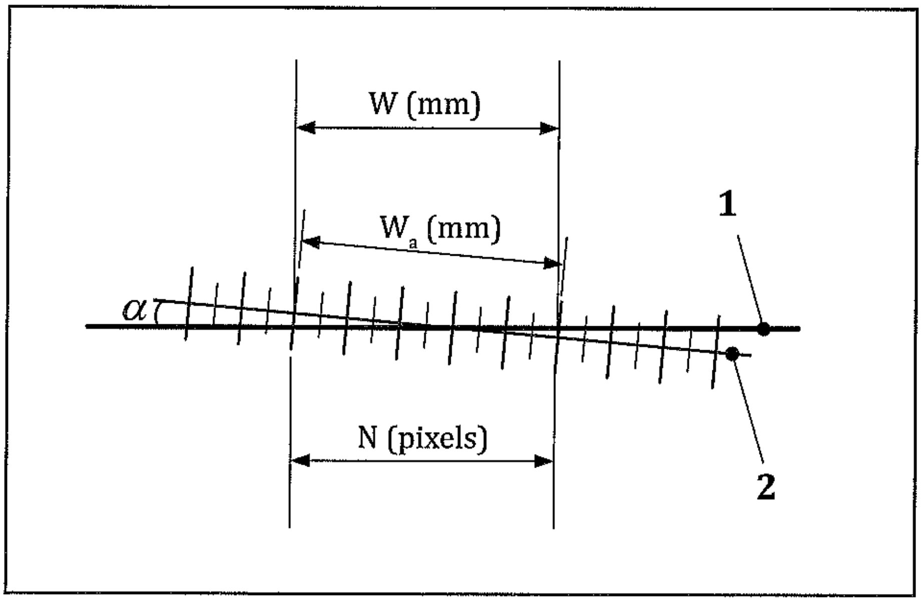
Calibration of scale unit: Pixel size calibration

C.1 General

When using a photographic film or an imaging plate, the pixel size of the image shall be calibrated to calculate the real dimension of the digitized distance, using the digitized reference glass scale.

C.2 Calibration procedure

Measure and record the number of pixels, N, along the x-axis of the PC display, corresponding to the arbitrary readout length, W_a (mm), and determine α. Use Formula (C.1) to calculate W as shown in Figure C.1.



Key

1 x-axis of the PC display

2 reference scale axis

Figure C.1 — Reference glass scale inclined with tilt angle of α to the x-axis of the PC display

NOTE 1 The W_a (mm) is the observed value from the digitized reference scale, not the measured value obtained by using other scales applied to the PC display.

$$W = W_a \times \cos\alpha \qquad \text{(C.1)}$$

where

α is the tilt angle between the scale axis and the x-axis of the PC display.

Then, the pixel size u (mm) can be calibrated by Formula (C.2). Record the calculated values in the data sheet.

$$u=\frac{\mathrm{W}}{N}=\frac{\mathrm{W_a}\times\cos\alpha}{N} \tag{C.2}$$

NOTE 2 If the digital camera is used to record the TEM/STEM image, the individual image sensor size defined in the specification, guaranteed by manufacturer, can be used as the pixel size, u.

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