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Surface chemical analysis — Scanning probe microscopy — Standards on the definition and calibration of spatial resolution of electrical scanning probe microscopes (ESPMs) such as SSRM and SCM for 2D-dopant imaging and other purposes

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National foreword

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Surface chemical analysis — Scanning probe microscopy — Standards on the definition and calibration of spatial resolution of electrical scanning probe microscopes (ESPMs) such as SSRM and SCM for 2D-dopant imaging and other purposes

Analyse chimique des surfaces - Microscopie à sonde à balayage - Normes sur la définition et l'étalonnage de la résolution spatiale des microscopes électriques à sonde à balayage (ESPMs) comme SSRM et SCM pour l'imagerie 2D-dopant et d'autres fins

Reference number
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Foreword

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The committee responsible for this document is ISO/TC 201, *Surface chemical analysis,* Subcommittee SC 9, *Scanning probe microscopy*.

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Introduction

Electrical scanning probe microscopy (ESPM) is a branch of scanning probe microscopy (SPM) with the capability of electrical imaging at nanometre spatial resolution. ESPM includes electrostatic force microscopy (EFM), scanning capacitance microscopy (SCM), scanning spreading resistance microscopy (SSRM), etc. Because ESPM can observe electrical or electronic properties with molecule-scale resolution, it is applied to many fields such as semiconductors, displays, etc. However, there has been no standard measurement method for the spatial resolution.

In this International Standard, standardized procedures to determine the spatial (lateral) resolution of SSRM and SCM, which are widely used to image the distribution of carrier and other electrical properties in semiconductor devices, are provided with the use of suitable reference materials. This International Standard uses the sharp-edge method to measure the lateral resolution of ESPM in a similar manner to that already used in measuring the resolution in micro-beam spectroscopy and in depth-profiling measurements with Auger electron spectroscopy and X-ray photoelectron spectroscopy (refer to ISO 18516).

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Surface chemical analysis — Scanning probe microscopy — Standards on the definition and calibration of spatial resolution of electrical scanning probe microscopes (ESPMs) such as SSRM and SCM for 2D-dopant imaging and other purposes

1 Scope

This International Standard describes a method for measuring the spatial (lateral) resolution of scanning capacitance microscopes (SCMs) or scanning spreading resistance microscopes (SSRMs), which are widely used in imaging the distribution of carriers and other electrical properties in semiconductor devices. The method involves the use of a sharp-edged artefact.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 18115-2, *Surface chemical analysis – Vocabulary – Part 2: Terms used in scanning-probe microscopy*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18115-2 and the following apply.

3.1
electrical scanning probe microscopy
ESPM
SPM mode in which a conductive tip is used to measure electrical properties such as capacitance, resistance, electrical field, etc.

3.2
contact mode
mode of scanning the probe tip over the sample surface, adjusting the relative heights of the probe and sample, in which there is always a repulsive force between the probe and the sample

Note 1 to entry: This mode can be, for example, either the constant-height or constant-force mode.

[SOURCE: ISO 18115-2:2013, 6.35]

4 Symbols and abbreviated terms

AC	alternating current
DC	direct current
ESPM	electrical scanning probe microscopy
SPM	scanning probe microscopy
AFM	atomic force microscopy

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MIS metal-insulator-semiconductor

MOS metal-oxide-semiconductor

SCM scanning capacitance microscopy

SIMS secondary ion mass spectroscopy

S/N signal to noise ratio

SSRM scanning spreading resistance microscopy

TEM transmission electron microscope

2D two dimension

Δx spatial resolution of ESPM

5 General information

5.1 Background information

ESPM is a branch of scanning probe microscope that can be used to image an electrical or electronic property of a sample surface using an electrically conducting probe. Since this conductive probe is scanned over the sample surface in the contact mode, its lateral resolution is strongly related to the size and shape of the probe apex. Currently, this can be as small as a few nanometres, enabling sub-10 nanometre spatial resolution to be achieved. Such a high resolution, shown in ESPM images, allows the investigation of the two-dimensional distribution of carriers in nanoscale semiconductor devices.

5.2 Target

There are a number of types of ESPM categorized by the methods of electrical characterization. Among these ESPMs, this International Standard is for SCM and SSRM.

5.2.1 Scanning capacitance microscope

Scanning capacitance microscopy (SCM) is a modification of scanning probe microscopy in which a conductive probe is in contact with the surface of a sample, with an applied AC bias, and scanned across it. SCM characterizes the change in electrostatic capacitance between the sample and the probe on the surface of the sample. SCM uses an ultra-sharp conducting probe made from etched silicon (often coated with Pt/Ir or Co/Cr alloy) to form a metal-insulator-semiconductor (MIS/MOS) capacitor with a semiconductor sample if a native oxide exists on the sample. When the conducting probe is in contact to the surface under an AC bias, generated capacitance variations on the surface can be detected using a GHz resonant capacitance sensor. The probe is then scanned across the semiconductor's surface in *x*- and *y*-axes while the probe is operated under the contact mode.

By applying an alternating bias to the metal-coated probe or the sample, carriers are alternately accumulated and depleted within the semiconductor's surface layers under the probe, changing the tip-sample capacitance. The magnitude of this change in capacitance with the applied voltage gives information about the concentration of carriers (SCM amplitude data), whereas the difference in phase between the capacitance change and the applied, alternating bias carries information about the sign of the charge carriers (SCM phase data).[2]

5.2.2 Scanning spreading resistance microscope

A very challenging task as the size of the semiconductor components shrinks towards sub-100 nm level is the development of new tools allowing two-dimensional (2D) carrier profiling with very high spatial resolution. One of the promising new tools is scanning spreading resistance microscopy (SSRM).

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SSRM is based on atomic force microscopy (AFM) and has been developed in recent years to probe the 2D resistivity and carrier distribution in semiconductor devices. In SSRM, a very small conductive tip is contacted on the sample surface to be used to measure the local spreading resistance, which is intimately linked to the local resistivity. Scanning a cross section of the sample provides a 2D map of the local spreading resistance with a spatial resolution set by the tip radius (typically 5 nm ~ 15 nm). The main advantages of SSRM lie in its relative robustness, as it is less sensitive to surface preparation than, for instance, scanning capacitance microscopy (SCM) leading to excellent reproducibility. SSRM also benefits from an excellent dynamic range covering the entire dopant range of interest (10^{14} ~ 10^{20}) cm^{-3} with constant sensitivity and from a high spatial resolution (set by the tip radius only) combined with very accurate junction delineation capabilities.[3]

5.3 Measurement method for lateral resolution in SCM and SSRM

The spatial resolution is not only influenced by geometric factors of the conductive probe. Other factors that affect spatial resolution include surface roughness of the sample, contrast of the electrical image from difference in carrier density, pixilation, noise and sensitivity of the detector. The spatial resolution of the ESPM instrument or the image has been determined by a few methods: imaging a regular pattern and measuring the smallest feature and imaging across an electrically abrupt interface, etc.

It is very difficult to fabricate electrically separated layers with two different carrier density. Also, it is crucial to connect, electrically, each plane of the repetitive pattern or the smallest feature. Therefore, the method chosen here is the sharp-edge method based on consideration of ease of use. This method of resolution definition is widely applied for depth-profiling of micro-beam spectroscopy such as secondary ion mass spectroscopy (SIMS). An electrically abrupt interface is line-scanned perpendicularly across the interface by a conductive probe and the detected profile of electrical characteristics is inspected. In the micro-beam spectroscopy, so called 16 % to 84 % width or some other criterion may be applied as the spatial resolution of SCM or SSRM as shown in Figure 1.[1][4] However, the definition of the resolution as 10 % to 90 % width is adopted as a standard method for SCM and SSRM since it has been well agreed academically.[5][6]

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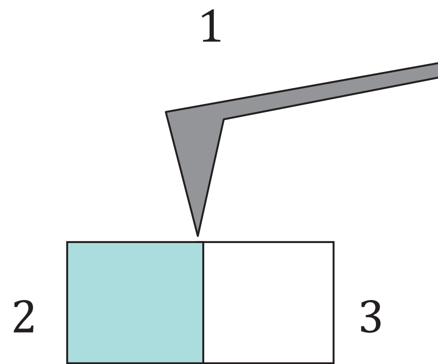
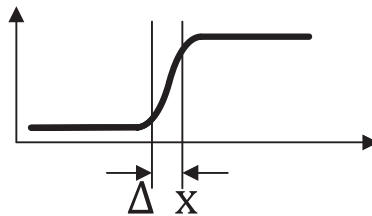


a) Side view of the sample and probe



b) Electric output as a function of the scan position

Key

1 conductive probe

2 coating

3 silicon

Δx spatial resolution of ESPM

Figure 1 — Schematic of sharp-edge method applied to SCM and SSRM

In applying this method and to obtain high-resolution data, the following should be noted.

a) It is recognized that in SSRM, the resolution depends on the contrast. Therefore, the resolution should be compared at the same contrast level. A discussion of contrast levels is given in 7.3.

b) The resolution is also a result of mechanical probe-sample contact between the sample and probe. The mechanical probe condition and the mechanical contact may change continuously even for a good contact mode. In general, the probe apex shape, outer metal coating condition and the contact area continuously change, causing the signal to fluctuate. Also, the electrical property of the silicon or its coating will not be perfectly uniform. These inhomogeneities may increase the noise and uncertainty levels. Therefore, the property of calibration sample, the experimental condition, and the S/N ratio should be considered carefully to obtain the optimum result.

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5.4 Key parameters in determining the lateral resolution

The measurement of lateral resolution can depend upon a number of experimental factors. They could be physical properties of the SCM or SSRM instrument and the sample surface, such as the conductive probe condition and the surface roughness. The following are the most important:

a) apex size of conductive probe: The apex size of the conductive probe is the most determining factor. Smaller probe sizes result in better spatial resolutions;

b) sample surface roughness: Sample surface roughness has a big effect on the measurement of spatial resolution in SCM or SSRM because the rougher surface changes more the contact between the probe and sample, both mechanically and electrically, generating apparent noise that affects the measured data;

c) contact force: A higher contact force makes the contact area wider and the resolution poorer;

d) signal contrast across the interface: A greater contrast gives a higher resolution value in SSRM; therefore, a signal contrast which exhibits at least 10 times of background noise level should be used in the experiment. If any signal saturates, an erroneously better value of the resolution could result.

6 Measurement of lateral resolution of SCM with the sharp-edge method

6.1 Background information

The sharp-edge method is frequently used in micro-beam techniques such as scanning electron microscopy (SEM) and SIMS to estimate the size of the focused beam on the sample surface.[1][4] An electrically abrupt interface is scanned by a conductive probe and the electrical signal profile across the interface is inspected. As the two regions have different electrical conductivities, the resultant SCM image can exhibit the interface region with spatially varying contrast. If a sectional analysis is applied to the interface region, a line profile with two plateau regions and a slope region between them is obtained. After the signal levels of the upper plateau and the lower plateau are normalized to be 1 and 0 as intensity values, respectively, the horizontal distance between the locations corresponding to intensity level of 0,1 and 0,9 is measured as the lateral resolution. In scientific publications, this criterion was generally used for the definition of spatial resolution of SCM or SSRM.[5][6] The concept of this method is straightforward and easy to implement. However, a difficulty is expected in preparing the sample having such abrupt interface without diffusion of dopants.

6.2 Selection of the sample

In this sharp-edge method, one needs a calibration sample with an electrically abrupt interface where the electrical contrast ideally shall change instantaneously as the interface is scanned by the conductive probe. In practice, there will be an "intermediate region" with a spatial gradient in electrical contrast between two regions of different electrical contrasts. This should be as small as possible and should be a fraction of the probe size.

In this International Standard, the fabrication procedure of the calibration sample is not specified because there may be a number of ways having their pros and cons. Therefore, this International Standard only describes the minimum requirements for the specification of the calibration sample.

Usually, a sample with the carrier density changing across an interface is used as an SCM or SSRM resolution calibration sample. For the best resolution, it is best to make the sample without diffusion. One way to do this is to use a doped silicon wafer on which 200 nm thick amorphous silicon layer is sputter-coated without diffusion, as shown in Figure 2, to make an electrically abrupt interface as fine as 1 nm or thinner. The capacitance or the AC capacitance variation of the coating of the reference sample is different from that of the substrate. The sample is cross-sectioned and polished to a roughness below 1 nm. The roughly polished backside of the polished sample is Pt-coated and later scratched deeper than the native oxide. The scratched surface is glued with GaIn and bonded to a metallic sample mount with silver paste. Then, both sides of the interface are electrically well connected to the sample mount.

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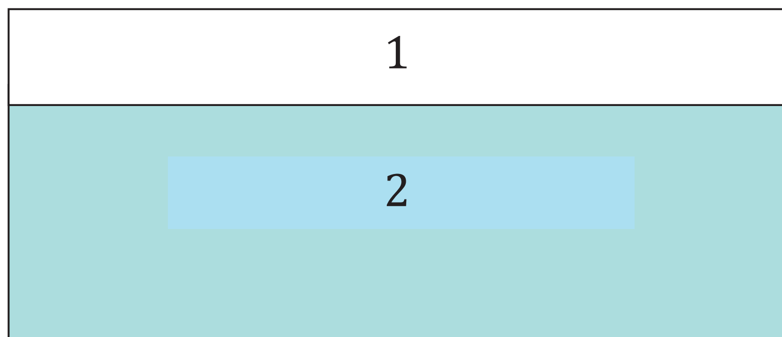
The cross section of the interface region was characterized by TEM. Interfaces thinner than 1 nm may be observed in TEM images. Therefore, this sample is suggested as a reference material which can be applied to determine the spatial resolution of SCM and SSRM.



Key

1 amorphous Si

2 doped silicon wafer

Figure 2 — Cross-sectional view of 200 nm thick amorphous silicon deposited on a doped silicon wafer without diffusion

6.3 Setting the parameters before the operation of the instrument

Operate the instrument in accordance with the manufacturer's or in-house documented procedures using the operating conditions for which the spatial resolution needs to be measured. The spatial resolution could be sensitive to these conditions and it may be useful to determine the resolution as a function of these settings in order to obtain different settings for optimal spatial resolution or adequate spatial resolution but faster imaging. The optimization of instrumental parameters for different experiments is not included in this International Standard.

6.4 Data collection

Using the reference sample from 6.2, obtain an SCM image that covers a region either side of the interface of more than five times the expected resolution. Extract a cross-sectional profile from a freely selected line perpendicular to the interface that divides two different contrasted regions. The interface shall be set perpendicular to the fast scanning axis to exclude the effect of thermal drift. When the line profile has a poor signal to noise ratio, instead of one line, multiple line profiles across the boundary may be selected and the line average of these line profiles may be used to determine the lateral resolution. An example of SCM data are presented in Annex A. As many as possible, pixel points between the 10 % and 90 % spots are recommended to define the resolution more clearly. In the example, there are only seven points and, in general, doubling this number is recommended.

6.5 Data analysis

6.5.1 Obtaining the resolution

From the image, select a single line profile or using the software an averaged line profile for many lines, normalize the intensity levels of upper plateau and the lower plateau to be 1 and 0, respectively, and the distance between the locations corresponding to intensity level of 0,1 and 0,9 is measured as the lateral resolution. The levels and resolution may be judged by eye or fitted with an analytical function. Using an analytical function can provide a more precise and more accurate value of the spatial resolution. Examples of both methods are given in Annex A.

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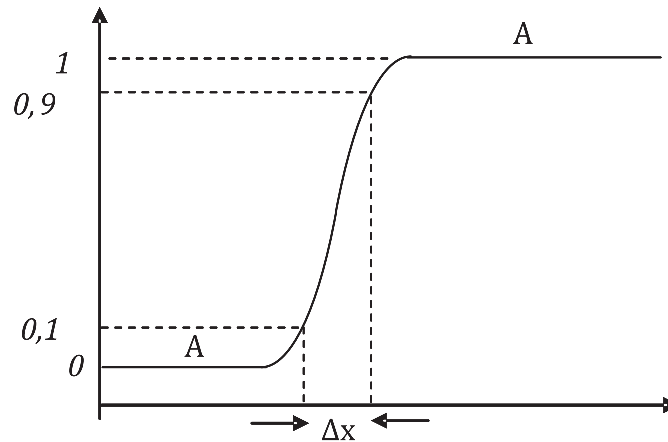

Key

A plateau

Figure 3 — Variation of electrical signal as a function of distance across the interface

6.5.2 Random contributions to the resolution value

From the image, select a number of line profiles, preferably more than seven, determine the spatial resolution of each profile and calculate both the average and standard deviation of the set of profiles. The best estimate of the spatial resolution is then this average with a standard deviation of the mean given by the standard deviation divided by the square root of the number of line profiles used. This gives a measure of the Type A uncertainty. For a full uncertainty, the uncertainty contribution from the reference sample and the scan direction calibration are required. For optimizing the instrument settings, this full uncertainty may not necessarily be required.

6.6 Recording of the parameters

The following parameters shall be recorded:

a) types and modes of SCM operation: DC mode or AC mode;

b) contrast across the interface: the signal contrast across the interface as the ratio of the mean upper plateau signal to the mean lower plateau signal;

c) S/N ratio: the S/N ratio of cross-sectional profile used to estimate the lateral resolution. Both the S/N value for one line scan and the number of scans averaged to form the scan to be used for the resolution measurement. The signal represents the difference between the mean upper plateau signal and the mean lower plateau signal and the noise represents the average of the standard deviations of the mean upper and lower plateau signals;

d) sample description: brief description of sample including the materials of the pattern and the substrate;

e) scanning speed: the speed of scanning in lines/s;

f) set point: the set point to determine the contact force in V;

g) number of pixels in x and y directions;

h) image size in μm;

i) bias voltage: the DC and AC voltage applied to the sample or tip in V;

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j) method, software, number of profiles and analytical function used to evaluate the spatial resolution;

k) $I_{0,1}$: x coordinate corresponding to signal level of 0,1, with the intensity levels of upper plateau and the lower plateau normalized to be 1 and 0, respectively;

l) $I_{0,9}$: x coordinate corresponding to intensity level of 0,9, with the intensity levels of upper plateau and the lower plateau normalized to be 1 and 0, respectively;

m) Δx: measured distance between $I_{0,1}$ and $I_{0,9}$;

n) standard deviation of the mean of the resolution values obtained together with the number of those results or whatever measure of the uncertainty is available from any analysing software, together with a statement of the type of uncertainty recorded.

7 Measurement of lateral resolution of SSRM with the sharp-edge method

7.1 Background information

The background information is same as that of SCM in 6.1.

7.2 Selection of the sample

The same sample as that of SCM in 6.2 can be used. The resistance of the coating is different from that of the substrate. This reference sample gives a resistance contrast across the interface in an SSRM image. The recommended resistance ratio across the interface is around 100 approximately 1 000 times.

7.3 Setting the parameters before the operation of the instrument

Since the operation of the instrument and adjustment of parameters are differed by manufacturers, the details cannot be described in this International Standard. As a higher contrast produces, an adequate signal contrast should be used in the experiment. The recommended minimum signal contrast is 10 times of background noise level.

7.4 Data collection

Using the reference sample from 6.2, obtain an SSRM image that covers a region either side of the interface of more than five times the expected resolution. Extract a cross-sectional profile from arbitrarily-selected lines perpendicular to the interface that divides two different contrasted regions. The interface shall be set perpendicular to the fast scanning axis to exclude the effect of thermal drift. When the line profile has a poor signal to noise ratio, instead of one line, multiple line profiles across the boundary may be selected and the line average of these line profiles may be used to determine the lateral resolution. An example of SSRM data are presented in Annex B. The example gives a resolution for the SSRM and has many pixel points between the 10 % and 90 % spots. As many as possible scanning points between the two points are recommended to define the resolution clearly.

7.5 Data analysis

7.5.1 Obtaining the resolution

From the image, select a single line profile or using the software an averaged line profile for many lines, normalize the intensity levels of upper plateau and the lower plateau to be 1 and 0, respectively, and the distance between the locations corresponding to intensity level of 0,1 and 0,9 is measured as the lateral resolution. The levels and resolution may be judged by eye or fitted with an analytical function. Using an analytical function can provide a more precise and more accurate value of the spatial resolution. Examples of both methods are given in Annex B.

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7.5.2 Random contributions to the resolution value

From the image, select a number of line profiles, preferably more than seven, determine the spatial resolution of each profile and calculate both the average and standard deviation of the set of profiles. The best estimate of the spatial resolution is then this average with a standard deviation of the mean given by the standard deviation divided by the square root of the number of line profiles used. This gives a measure of the Type A uncertainty. For a full uncertainty, the uncertainty contribution from the reference sample and the scan direction calibration are required. For optimizing the instrument settings, this full uncertainty may not necessarily be required.

7.6 Recording of the parameters

The following parameters shall be recorded:

a) contrast across the interface: the signal contrast across the interface as the ratio of the mean upper plateau signal to the mean lower plateau signal;

b) S/N ratio: the S/N ratio of cross-sectional profile used to estimate the lateral resolution. Both the S/N value for one line scan and the number of scans averaged to form the scan to be used for the resolution measurement. The signal represents the difference between the mean upper plateau signal and the lower plateau signal and the noise represents the average of the standard deviations of the mean upper and lower plateau signals;

c) sample description: brief description of sample including the materials of the pattern and the substrate;

d) scanning speed: the speed of scanning in lines/s;

e) set point: the set point to determine the contact force in V;

f) number of pixels in x and y directions;

g) image size in µm;

h) bias voltage: the DC voltage applied to the sample or tip in V;

i) method, software, number of profiles and analytical function used to evaluate the spatial resolution;

j) $I_{0,1}$: x coordinate corresponding to signal level of 0,1, with the intensity levels of upper plateau and the lower plateau normalized to be 1 and 0, respectively;

k) $I_{0,9}$: x coordinate corresponding to intensity level of 0,9, with the intensity levels of upper plateau and the lower plateau normalized to be 1 and 0, respectively;

l) Δx: measured distance between $I_{0,1}$ and $I_{0,9}$;

m) standard deviation of the mean of the resolution values obtained together with the number of those results or whatever measure of the uncertainty is available from any analysing software, together with a statement of the type of uncertainty recorded.

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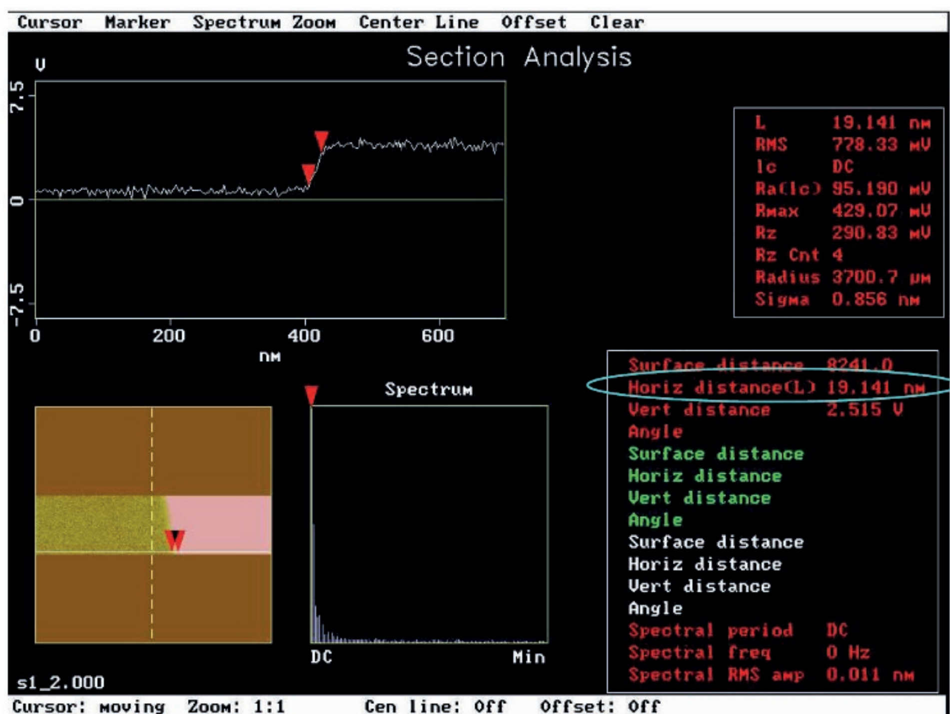
Annex A
(informative)

An example of the measurement of SCM resolution

A.1 Approximate estimation

Figure A.1 is an example of the measurement of SCM resolution from a single line profile from an SCM image of the reference sample in 6.2. The central zone of the image is shown in the lower left part and the line profile from the lowest line in this region in the upper left part. In this software, the two red arrows are moved along the line profile and are set at the nearest pixels to those indicating 10 % and 90 % of the SCM signal change at the interface. The distance between the arrows, 19,1 nm, is displayed in the lower right part as shown in the ellipse. According to this procedure, the resolution is 19,1 nm. Note that there are only seven pixels across this resolution and so the precision of this value is, at best, 2,7 nm.



NOTE The approximate spatial resolution is 19,1 nm.

Figure A.1 — Measurement of SCM resolution

A.2 Data analysis

The above is an approximate method with measurements made by eye. A better method is to use a least squares fitting of a suitable analytical function. Here, the Logistic Function Profile Fitting (LFPF) program is exampled. This program is software for estimating the position, width and asymmetry of the interface between dissimilar materials.[7] It was successfully used to fit Auger sputter-depth-profile data. The SCM data of the sectioned graph is fitted well to give a good result as shown in Figure A.2. The SCM value of lower plateau is 0,65 V and that of upper plateau is 3,98 in the lower left box of the figure. The position $I_{0,1}$ is 401,6 nm and the position $I_{0,9}$ is 428,7 nm. Therefore, the resolution, the distance between 10 % and 90 % of the SCM signal change at the interface, is 27,1 nm ± 5,2 nm in the lower right

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box of the figure where the uncertainty is given at a 95 % confidence level. The fitting provides a more precise value for the spatial resolution.

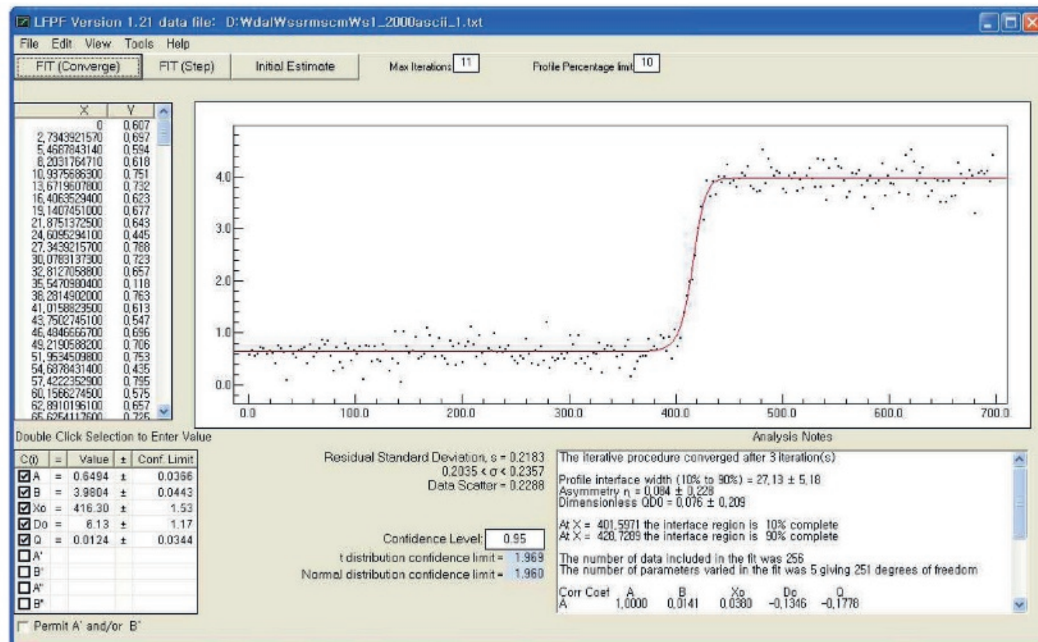

Figure A.2 — LFPF fitting of SCM to determine the SCM resolution

The analytical function used in the LFPF program is

Signal = XYZ (A.1)

where

X is the profile width given by the separation of the 10 % and 90 % points is given by

Interface width = ZZZ ± CCC (A.2)

where the uncertainty at 95 % confidence is evaluated.

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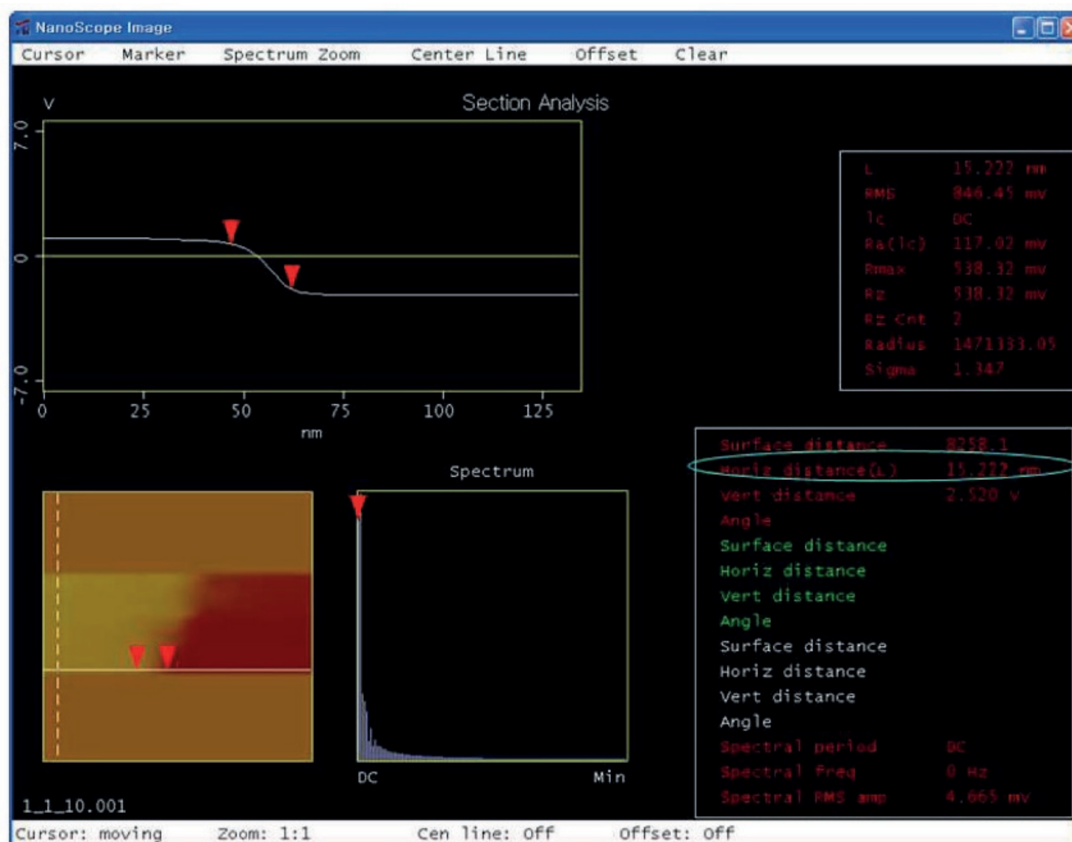
Annex B
(informative)

An example of the measurement of SSRM resolution

B.1 Approximate estimation

Figure B.1 is an example of the measurement of SSRM resolution from a single line profile from the SSRM image of the reference sample in 7.2. The central zone of the image is shown in the lower left part. The line profile from the lowest line in this region is shown in the upper left part. The SSRM signal is not resistance but a signal proportional to log (10^6/resistance) in volts. In this software, the two red arrows indicate the points of which the SCM data are nearest to 10 % and 90 % of the SSRM signal change at the interface and the distance between the two arrows is 15,2 nm in the lower right part in the eclipse. According to this procedure, the resolution is 15,2 nm.



NOTE The approximate spatial resolution is 15,2 nm.

Figure B.1 — Measurement of SSRM resolution

B.2 Data analysis

The above is an approximate method with measurements made by eye. A better method is to use a least squares fitting of a suitable analytical function. Here, the LFPF program is again used. The SSRM data are fitted very well with this asymmetric function to give the result shown in Figure B.2. The SSRM value of lower plateau is –2,13 V and that of upper plateau is 1,00 V in the lower left box of the figure.

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The position $I_{0,1}$ is 61,9 nm and the position $I_{0.9}$ is 47,026 73 nm. Therefore, the resolution, the distance between 10 % and 90 % of the SSRM signal change at the interface is 14,9 nm ± 0,1 nm in the lower right box of the figure where the uncertainty is given at a 95 % confidence level. The fitting provides a more precise value for the spatial resolution.

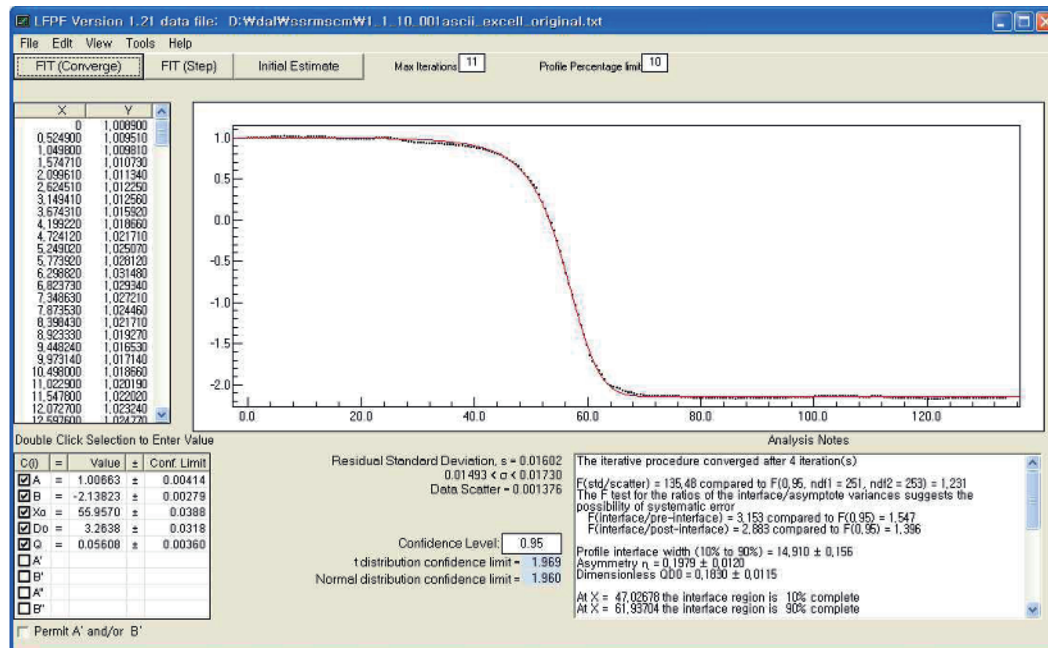

Figure B.2 — LFPF fitting of SSRM to determine the SSRM resolution

The analytical function used in the LFPF program is

$$\text{Signal} = XYZ \quad \text{(B.1)}$$

where

X is the profile width given by the separation of the 10 % and 90 % points is given by

$$\text{Interface width} = ZZZ \pm CCC \quad \text{(B.2)}$$

where the uncertainty at 95 % confidence is evaluated.

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