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Microbeam analysis — Scanning electron microscopy — Qualification of the scanning electron microscope for quantitative measurements

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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This document was prepared by Technical Committee ISO/TC 202, *Microbeam analysis*, Subcommittee SC 4, *Scanning electron microscopy*.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The scanning electron microscope (SEM) is a very versatile instrument, which is widely used in production, development and scientific research across the world. While they are easy to operate and provide results quickly, there are a number of notorious problems, which hinder operating them at their best performance. These are the reasons for lack of excellent repeatability in SEM imaging and measurements. The most bothersome ones among these are unintended motions of the sample stage and the primary electron beam, geometry distortions, wrong image magnification, image blur (lack of sharp focus), noise and electron beam-induced contamination. Quantification of these essential performance parameters is very useful to ensure that all SEMs perform at manufacturers specifications and at users' own purpose. Quantified knowledge helps in the evaluation of measurement uncertainties, and necessary repairs.

This document pertains to measurement methods for the following SEM performance parameters:

— Image sharpness (spatial resolution, primary electron beam focusing ability).

— Drifts (the sample stage, the electron beam and the electron-optical column).

— Cleanliness (lack of beam-induced contamination).

— Image magnification and linearity (both in X and Y directions).

— Background noise.

— Primary electron beam current.

These parameters will also be influenced by the SEM conditions such as the lifetime of source (emitter conditions), lifetime of liner tube and apertures (contamination of the electron optical parts), time and intensity of last cleaning of vacuum chamber by the plasma cleaning or Ultra Violet irradiation, the sample preparation and final surface cleaning.

Microbeam analysis — Scanning electron microscopy — Qualification of the scanning electron microscope for quantitative measurements

1 Scope

This document describes methods to qualify the scanning electron microscope with the digital imaging system for quantitative and qualitative SEM measurements by evaluating essential scanning electron microscope performance parameters to maintain the performance after installation of the instruments. The items and evaluating methods of the performance parameters are selected by users for their own purposes.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 16700:2016, *Microbeam analysis — Scanning electron microscopy — Guidelines for calibrating image magnification*

ISO/IEC 17025:2017, *General requirements for the competence of testing and calibration laboratories*

ISO 22493, *Microbeam analysis — Scanning electron microscopy — Vocabulary*

ISO/TS 24597:2011, *Microbeam analysis — Scanning electron microscopy — Methods of evaluating image sharpness*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 22493 and the following 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
scanning electron microscope
SEM
instrument that produces magnified images of a specimen by scanning its surface with an electron beam

[SOURCE: ISO 16700, 3.1]

3.2
image
two-dimensional representation of the specimen surface generated by SEM

[SOURCE: ISO 16700, 3.2]

3.3
image magnification
ratio of the linear dimension of the scan display to the corresponding linear dimension of the specimen scan field

[SOURCE: ISO 16700, 3.3]

3.4
scale marker
line/generated line (intervals) on the *image* (3.2) representing a designated actual length in the specimen

[SOURCE: ISO 16700, 3.4]

3.5
reference material
RM
material, sufficiently homogeneous and stable with respect to one or more specified properties, which has been established to be fit for its intended use in a measurement process

[SOURCE: ISO Guide 30:2015, 2.1.1, modified — Note 1 to entry to Note 4 to entry are omitted.]

3.6
certified reference material
CRM
reference material (RM) (3.5) characterized by a metrologically valid procedure for one or more specified properties, accompanied by an RM certificate that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability

[SOURCE: ISO Guide 30:2015, 2.1.2, modified — Note 1 to entry to Note 4 to entry are omitted.]

3.7
calibration
set of operations which establish, under specified conditions, the relationship between the magnification indicated by the SEM and the corresponding magnification determined by examination of an *RM* (3.5) or a CRM

[SOURCE: ISO 16700, 3.7]

3.8
accelerating voltage
absolute acceleration potential V_a [V] of the final anode to the electron emitter

Note 1 to entry: For the electron charge q_e [C], the accelerated electron will obtain the energy $|q_e|V_a$ [J] = V_a [eV] ≡ E_L and enter the sample with this energy provided that the initial energy E_I from the emitter is negligible. The "landing energy" to the specimen means this energy E_L, typically expressed in the unit eV or keV.

Refer to Clause 4 concerning eV.

4 Symbols and abbreviated terms

$A_{ACF,1}$, $A_{CCF,n}$ — areas of the binarized pictures $F_{BAC}(I_1)$ and $F_{BCC}(I_1, I_n)$ respectively, typically expressed in pixel

ACF — auto-correlation function

CCF — cross-correlation function

D_{Hn}, D_{Vn} — drift quantities of the n-th image I_n $(n=1, 2, \cdots)$ for the horizontal (H) and vertical (V) directions respectively, typically expressed in pixel or in nm

D_X, D_Y — displacements for X and Y directions respectively from the origin, typically expressed in nm

max $|D_X|$, max $|D_Y|$ mean the largest absolute values of displacements from the origin in X and Y directions respectively

D_O — distance $D_O = \left(D_X{}^2 + D_X{}^2\right)^{1/2}$ from the initial position (X_O, Y_O) which is regarded as the origin, typically expressed in nm

max D_O - the largest value of the distance D_O

d — pitch length of the RM or the CRM, typically expressed in nm

d_S — measured mean pitch length, typically expressed in nm

d_{SA} — averaged value of measured d_S, typically expressed in nm

CG — contrast to gradient method for evaluating image sharpness

DR — derivative method for evaluating image sharpness

FT — Fourier transform method for evaluating image sharpness

eV — electronvolt, a unit of energy equal to approximately 1.6×10^{-19} joules (J). By definition, it is the amount of energy gained (or lost) by the charge of a single electron moving across an electric potential difference of 1 volt

$F_{AC}(I_1)$ — auto-correlation function of the image I_1

$F_{CC}(I_1, I_n)$ — cross-correlation function for the initial image I_1 and n-th image I_n $(n=2, 3, \cdots)$ in the measurements

$F_{BAC}(I_1)$ — binarized picture of the auto-correlation function $F_{AC}(I_1)$ of the initial image I_1 by using a thresholding level T_B

$F_{BCC}(I_1, I_n)$ — binarized picture of the cross-correlation function $F_{CC}(I_1, I_n)$ for the initial image I_1 and the n-th image I_n $(n=2, 3, \cdots)$ by using a thresholding level T_B

HFW — horizontal field width

H_{P1}, V_{P1} — horizontal (H) and vertical (V) peak positions of the auto-correlation function $F_{AC}(I_1)$ respectively

H_{Pn}, V_{Pn} — horizontal (H) and vertical (V) peak positions of the cross-correlation function $F_{CC}(I_1, I_n)$ respectively

I_{BG}	background noise image
I_{FB}	flat image whose signal intensity of the element (i, j) is the mean value S_{MEAN} of the background noise image I_{BG}
I_{PB}	processed images obtained from the background noise images I_{BG}, for example, by a method of contrast enhancement
I_{OS}	original secondary electron (SE) or backscattered electron (BE) scanning image
$I(i, j)$	signal intensity of the element (i, j) of the image I, where i and j mean horizontal and vertical numbers of the element respectively measured from the initial element (1, 1)
$I_{ref}(i, j)$	signal intensity of the element (i, j) of the reference image I_{ref} which is set to the flat image I_{FB} when calculating the peak signal-to-noise ratio S_{PSNR} $\max\left[I_{ref}(i, j)\right]$ means the maximum value of the given n_{IM}-bit imaging mode. If n_{IM} = 8-bit imaging mode, then $\max\left[I_{ref}(i, j)\right]=255$
$I_{test}(i, j)$	signal intensity of the element (i, j) of the test image I_{test} which is set to the background noise image I_{BG} when calculating the peak signal-to-noise ratio S_{PSNR}
$I_{refS}(i, j)$	signal intensity of the element (i, j) of the reference image I_{refS} which is be obtained from the test image $I_{testS}(i, j)$ by applying a suitable image filter to reduce the image noise
$I_{testS}(i, j)$	signal intensity of the element (i, j) of the test image I_{testS} which is usually not the background noise image I_{BG} but actual SE or BE scanning image
I_p	primary electron beam current (probe current)
kV	kilovolt
$k_{A,n}$	ratio of the area $A_{CCF,n}$ to the area $A_{ACF,1}$ $(k_{A,n}=A_{CCF,n}/A_{ACF,1})$
L	image size (total pixels of the image area), typically expressed in pixel such as $L_H \times L_V$
L_H, L_V	horizontal (H) and vertical (V) image sizes (lengths) respectively, typically expressed in pixel
L_p	pixel size, typically expressed in nm
L_{pHA}	horizontal (H) line profile which is obtained vertically averaged for specified band areas from the background noise image
L_{pVA}	vertical (V) line profile which is obtained horizontally averaged for specified band areas from the background noise image
l_S	the total measured length by a scaler on the screen or the photograph ($l_S = n_P \cdot d_S$)
M	image magnification for setting
N_M	number of measurements for beam current
N_{MD}	number of measurements for image drift
N_{MM}	number of measurements for image magnification

N_{MS}	number of measurements for image sharpness
n_P	total number of pitches for measurement
R_L	image sharpness, typically expressed in nm
R_{PX}	image sharpness, typically expressed in pixel
S_{MAX}	maximum value of intensities of pixels in an image
S_{MEAN}	mean value of intensities of pixels in an image
S_{MIN}	minimum value of intensities of pixels in an image
S_{PSNR}	peak signal-to-noise ratio
S_{STD}	standard deviation of intensities of pixels in an image
V_a	accelerating voltage
WD	working distance

5 General principles

The best performance of any SEM is at some optimized set of instrument settings; therefore, throughout this document for the various assessments those imaging parameters and instrument settings should be used if those are specified by the SEM's manufacturer for achieving the best performance. These basic principles are useful for users' own purpose in many cases.

5.1 Condition setting

Some SEMs have only one accelerating voltage in these specifications; others may have more (e.g. 15 kV and 1 kV). All the assessments should be performed at all specified accelerating voltages and magnifications if those parameters and settings are applicable to user's purposes and evaluations. If optimization of SEM-based measurements requires different parameters (accelerating voltage, beam current, etc.) for users' own purpose, then for these all the assessments should be performed and the results recorded in the report.

Beyond setting the magnification, accelerating voltage, beam current, all pertinent parameters to the values specified by the instrument manufacturer for proving the best resolution performance, it is important to set the focus (astigmatism), contrast, and brightness to their SEM-specific, best settings for taking images.

5.2 Contrast/brightness setting

Only images with properly set contrast and brightness should be used for the various measurements and quantitative SEM assessments. The contrast and the brightness must be set so that all pixels have grey-scale levels that never reach the lowest (dark, under-saturation) or the highest (bright, over-saturation) level. This is important to make sure that no information is lost by setting contrast and brightness values incorrectly.

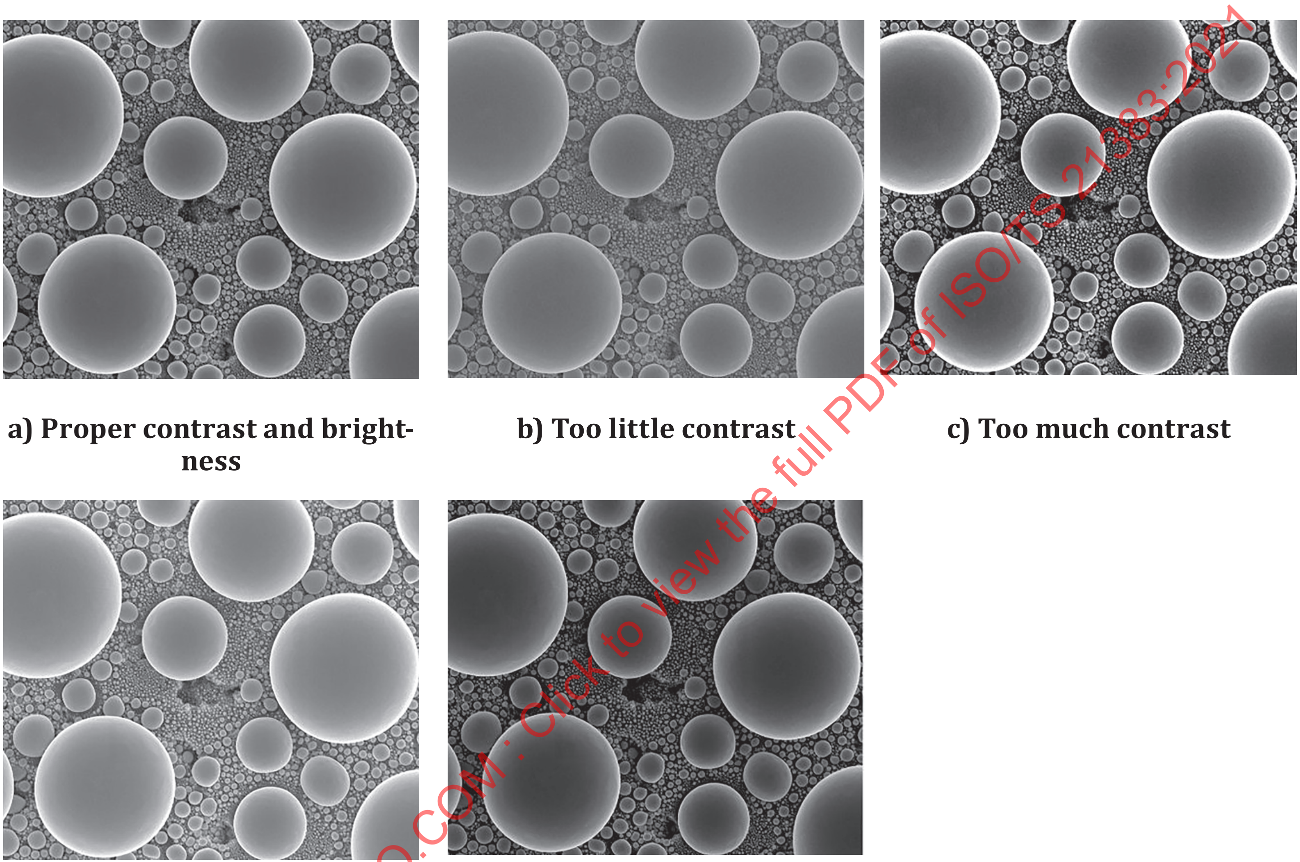
If the system has the signal monitor or can generate the histogram for the relative signal range [0,1] in the acquisition of SEM image, verify that the signals are within the range [0.2, 0.8] approximately.

In 8-bit imaging mode, in properly set images the intensities of the image pixels vary in between 0 and 255 grey levels. In 16-bit imaging mode the intensities of the image pixels vary in between 0 and 65535 grey levels.

Figure 1 shows examples of secondary electron (SE) images taken at accelerating voltage 5 keV, 19,5 μm HFW. Contrast and brightness are properly set a) and wrong b), c), d) and e). Figure 2 shows their corresponding histograms.

5.3 Sample preparation

Concerning the samples, there will not be the perfect or the almighty sample which are applicable to many evaluation items. In general, the best samples are different for the measurements of image sharpness, drift and drift-related distortions, electron beam induced contamination, image magnification and linearity. Select the ideal sample which is suitable for the required evaluation accordingly.



a) Proper contrast and brightness

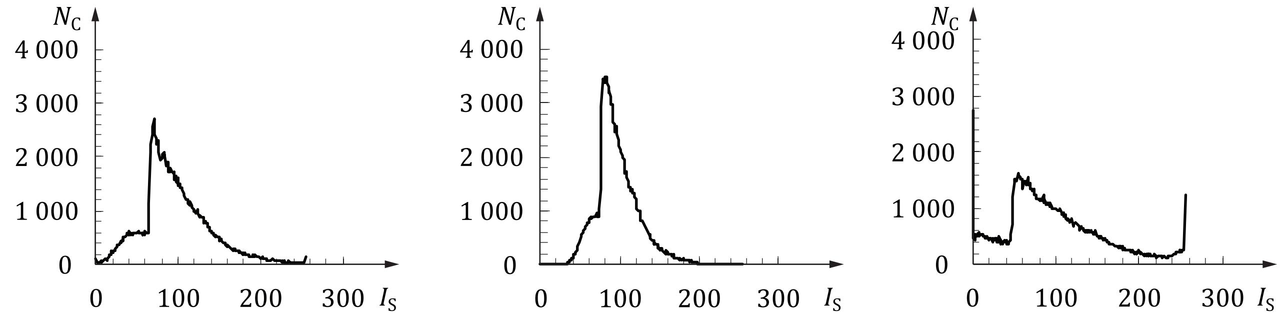
b) Too little contrast

c) Too much contrast

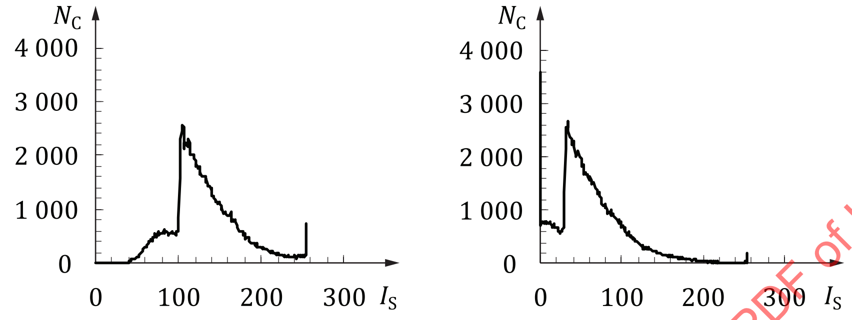
d) Too much brightness

e) Too little brightness

Figure 1 — Examples of SE images for various contrast and brightness setting



a) Proper contrast and brightness **b) Too little contrast** **c) Too much contrast**



d) Too much brightness **e) Too little brightness**

Key

I_S signal intensity for 8-bit imaging mode

N_C number of counts

Figure 2 — Examples of histograms corresponding to the images in Figure 1

6 Measurement of image sharpness

The definition and the explanation of the term “image sharpness” are described in ISO TS 24597 for the SEM. Image sharpness is strongly related to the focusing ability of the SEM in forming the primary electron beam. It is one of most widely used, but certainly not sufficient performance parameter. However, it is very useful to know the image sharpness because the algorithms do not depend on the human sense and give quantitative results by using the procedures described below.

On the other hand, the term “Lateral resolution” or “Spatial resolution” are not defined strictly in SEM even though these terms are popular in the surface chemical analysis.

NOTE Refer to ISO 18115-1:2013 for terms used in spectroscopy[1].

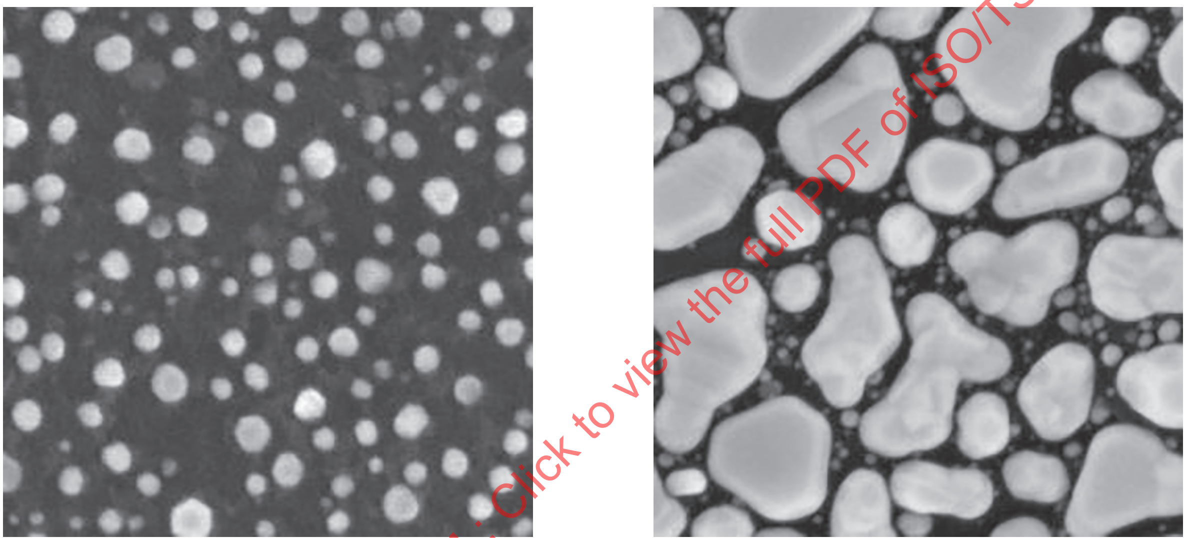
Even if various SEM manufacturers use these terms, the notion of resolution is not established scientifically, it is sample-and method-dependent, and there is no accurate way of measuring it today. The focusing ability is related to the size and shape of the primary electron beam at the surface of the sample. Its measurement is also very difficult, especially for sub-nanometre focuses, i.e., beam sizes. Furthermore, these values are not related to beam focusing only, but to interaction volume, stability of probe scanning and external disturbances as well.

To acquire the images for the evaluation of the image sharpness, refer to the Clause 4 “Steps for acquisition of an SEM image” of ISO TS 24597. “4.1 General”, “4.2 Specimen”, “4.4 Selection of the field of view” and “4.7 Contrast-to-noise ratio of the image” will be useful information. The structure of the sample should not be “periodic mesh” or “line and space” because some specific features or frequencies are emphasized in the signal analysis. The samples as shown in Figure 3 a) is typical and appropriate because the particles with different diameters are randomly distributed, and their surface are flat and edges are sharp.

Set the focus and astigmatism to their best, the magnification, accelerating voltage, beam current, working distance to the values specified by the instrument manufacturer for proving the best image sharpness performance, and take several images.

To evaluate the SEM image sharpness performance, follow the procedure in ISO/TS 24597:2011. Select the evaluation method from the 3-methods DR (Derivative) method, FT (Fourier transform) method and CG (Contrast-to-gradient) method. For one image, plural methods can be used if necessary. Valuate that the obtained results are allowable or not for the quantitative measurements. Report evaluated results with the evaluation methods and the valuation.

Figure 3 shows the examples of the selected SEM images with the image size L=512×512 for the evaluation of the image sharpness R_{PX} [pixel] or R_L [nm]. The obtained values of image sharpness R_L are 1,9 nm for Figure 3 a) and 3,3 – 4,2 nm for Figure 3 b). The example of the evaluation process for these images is shown in Table A.1.



a) Accelerating Voltage V_a = 15 kV, beam current I_P = 43 pA, 265 nm HFW.

b) Accelerating Voltage V_a = 1 kV, beam current I_P = 43 pA, 657 nm HFW.

Figure 3 — Selected SEM images for the evaluation of image sharpness. Sample: Evaporated gold on carbon.

See Clause 12 and Annex A for further pertinent information.

7 Measurement of drift and drift-related distortions (imaging repeatability)

Unintended motions make the primary electron beam land on wrong, unintended locations on the sample, which results in poor repeatability and/or distorted, blurry images, especially at high magnifications. These typically arise from mechanical and acoustical impacts, temperature variation of the room and the cooling water, hysteresis, adverse external electromagnetic fields, and from the noise in various circuits of the SEM. In the SEM with high working rate for many years, charging of contaminated apertures and inner walls of the liner tube for electron beam could be possible reason for unintended disturbances.

So, it is very useful to know the drifts and the drift related distortions quantitively and qualitatively by applying the procedures described below because these properties tell us the upper limit of the significant measurements.

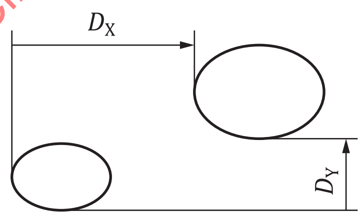
Modern SEMs can have close to 1 nanometre image sharpness, so even very small unintended motions can interfere or easily ruin the imaging and measurement performance of the SEM. Both the sample stage with the sample and the primary electron beam make unintended motions and they result in a combined error in the actual location where the electron beam-sample interaction takes place.

It is important to make sure that the geometry distortions of the SEM are sufficiently small for acquiring images and for carrying out measurements within requirements. These, depending on the task at hand, may be diverse, e.g. for high-quality, fine image sharpness imaging are a lot more stringent than for simple microanalysis. Depending on the intended use, the drift-related performance should be valuated for one-minute (typical secondary electron image acquisition time), ten-minute (analytical acquisition), and for one and many hours-long time periods.

7.1 Measurement of image drifts within specified time intervals.

To determine the drift performance, set the focus to its best, magnification, accelerating voltage, beam current, to the values specified by the instrument manufacturer for proving the best image sharpness performance, and without intentionally changing the sample location, perform at least the 1- and 10-minute assessments, and if it is relevant for the task at hand, add further, longer ones. For longer time drift measurements, the magnification shall be lowered to keep the reference pattern always in the central 80 % of the image area (refer to ISO 16700). The guiding principle is to measure the drift for as long periods of time as the time of the measurement or procedure carried out by the SEM. Valuate that the measured drifts are allowable or not in the quantitative SEM measurements. Report all results as image drift performance values and the valuation.

When drift performance is measured by watching the displacements (movements) D_X and D_Y of a marked object (or a particle) in the recorded image, use the determined left end or the right end of the object for the x – direction, and use the determined bottom end or the top end of the object for the y-direction as shown in Figure 4. This is the reason why the definition of the displacement become unclear under the drift-related distortions. For the drift measurement, the initial position (X_0, Y_0) of the object should be near the centre of the field (the display). The initial position (X_0, Y_0) is regarded as the origin, and the distance D_0 from the origin is defined as $D_0 = (D_X{}^2 + D_X{}^2)^{1/2}$.



Key

D_X displacement for X direction, expressed in nm

D_Y displacement for Y direction, expressed in nm

Figure 4 — Measurement method of the displacement

After the installation of the SEM, the drifts may be enlarged owing to the increased movements of the floor or the increased fluctuation of the external electromagnetic fields. Then, the obtained results do not reflect original SEM performances but the condition of the environments, or the ability of the isolation and the shielding. Measure the movements of the floor or the fluctuation of the external field by the methods SEM manufacturer have performed if possible. Consider the source of the change, and report the estimated results. Improper sample preparation, such as the sample surface with non-conductor and the insufficiently grounded samples, will also cause the severe drifts.

The acquired images in the drift measurement are also utilized for measurement of drift-related distortions (imaging repeatability). Refer to 7.2 and 7.3.

7.1.1 One-minute drift measurement

Take 5 consecutive images at every 15 second or so, play them in sequence to visualize the nature and the extent of the drift, find the largest displacement max $|D_X|$ and max $|D_Y|$ in X and Y directions respectively among them. Report them as 1-minute drift performance value with the largest X and Y direction motion, both the largest values max $|D_X|$ and max $|D_Y|$ and the largest value max D_0 of the distance D_0 from the origin.

A graphical example of the measurement is shown in Figure 5 a), and the numerical example for the report is shown in Table B.1.

Image acquisition time t_a and image storing time t_s will be selected as users like within the total 15 seconds. The point is to accomplish the image sequence acquisition within one minute.

If these fast images prove to be too noisy for reliable measurements, and sufficiently higher beam currents are used, then this condition should be added to the report because the drift performance may depend on the beam current.

7.1.2 Ten-minute drift measurement

Take 11 images, one at start and then at every minute or so, play them in sequence to visualize the nature and the extent of the drift, find the largest displacement max $|D_X|$ and max $|D_Y|$ in X and Y directions respectively among them. Report them as 10-minute drift performance value, both the largest values max $|D_X|$ and max $|D_Y|$ and the largest value max D_0 of the distance D_0 from the origin.

A graphical example of the measurement is shown in Figure 5 b), and the numerical example for the report is shown in Table B.2.

The time t_a and the time t_s will be chosen as users like within the total one minute.

7.1.3 One-hour drift measurement

Take 21 images, one at start and then every 3 min or so, play them in sequence to visualize the nature and the extent of the drift, find the largest displacement max $|D_X|$ and max $|D_Y|$ in X and Y directions respectively among them. Report them as 1-hour drift performance value, both the largest values max $|D_X|$ and max $|D_Y|$ and the largest value max D_0 of the distance D_0 from the origin.

A graphical example of the measurement is shown in Figure 5 c), and the numerical example for the report is shown in Table B.3.

7.1.4 Long-term larger than one-hour drift measurement

Obtain the standard interval t_{ID} [min] of the recording (measurement) using Formula 1:

$$t_{ID} = 0.25\, t_{DM}{}^{x} \quad (1)$$

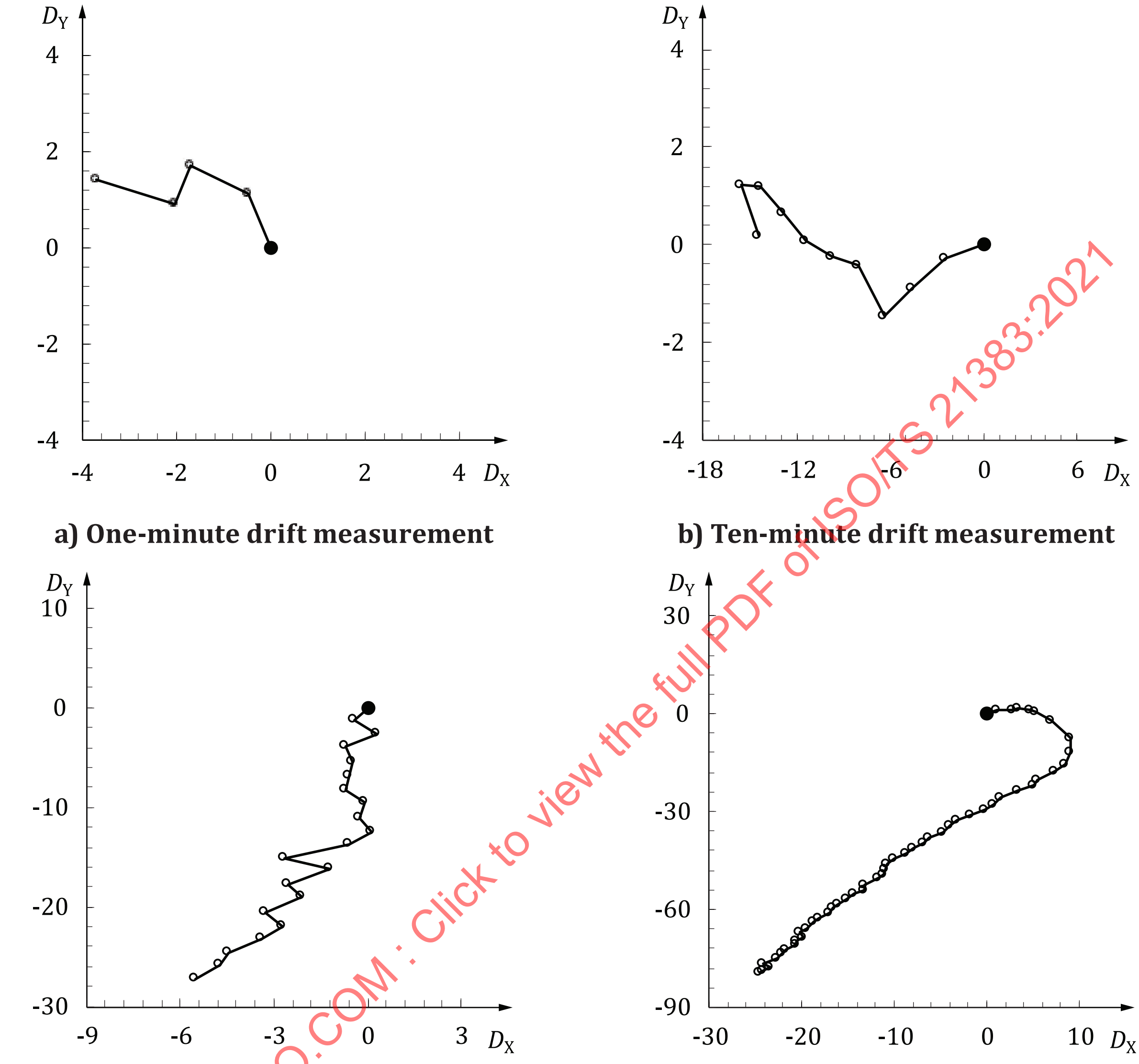
where

t_{DM} [min] is the total time of drift measurements;

$x = 0.605$ is the approximate constant.

Take images at every t_{ID} [min] or so, play them in sequence to visualize the nature and the extent of the drift, find the largest displacement max $|D_X|$ and max $|D_Y|$ in X and Y directions respectively among them. Report them as long-term drift performance value, both the largest values max $|D_X|$ and max $|D_Y|$ and the largest value max D_0 of the distance D_0 from the origin.

A graphical example of the long term (ten-hour) measurement is shown in Figure 5 d), and the numerical example for the report is shown in Table B.4.



a) One-minute drift measurement

b) Ten-minute drift measurement

c) One-hour drift measurement

d) Long term (ten-hour) drift measurement

Key

D_X displacement for X direction, expressed in nm

D_Y displacement for Y direction, expressed in nm

Figure 5 — Examples of the measurement of the drift

Some automatic software-based measurements are used for a drift-compensated imaging[6],[7] if the SEM user requests these functions to decrease the effects of the drifts.

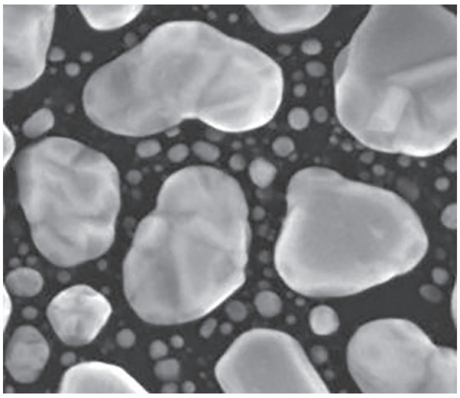
See Clause 12, Annex B and "B.2 Measurement of image drifts within specified time intervals" and "B.5 Measurement of the drifts and drift-compensated imaging".

7.2 Evaluation of the drift and the drift-related distortions by using image overlay

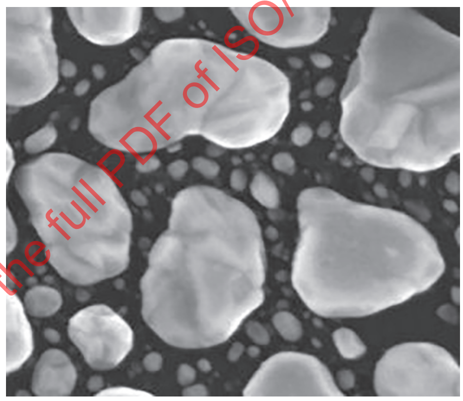
To assess the slow scan imaging repeatability performance, set the focus and astigmatism to their best to prove the best image sharpness for the accelerating voltage, electron beam current and working distance which are selected by the users for their own purposes. Then take 4 consecutive images as shown in Figure 6[7] at suitable location over the resolution reference sample without changing anything or moving the sample. Select the areas from the original images so that their largest common sections are nearly centred (drift adjusted) by using suitable image processing software[4]. Add the 4 original images I_{OSn} (n=1, 2, 3, 4) to the report, and add their selected (drift adjusted) images for the overlay, and the overlay image I_{DAO} that is generated by overlaying them so that their largest common sections are nearly centred as shown in Figure 7 b). Valuate that the overlay image I_{DAO} is allowable or not for the quantitative measurements and add the valuations to the report.

If the simple overlay image I_{SO} is required to observe the magnitude of the drift, select the centre areas from the original images. Add the 4 original images I_{OSn} to the report, and add their selected images in which drift are not adjusted for the simple overlay, and the overlay image I_{SO} that is generated by overlaying them as shown in Figure 7 a).

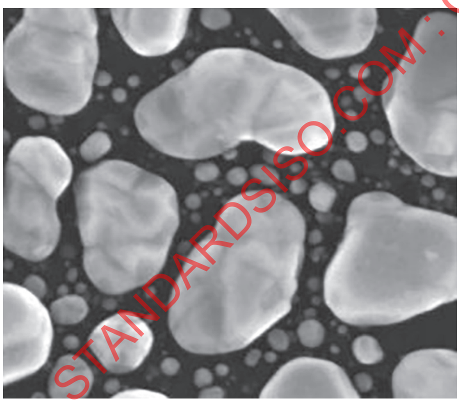
NOTE If the size of an original image is 1280x960 pixels, then the size of the selected image is typically 512x512 pixels.



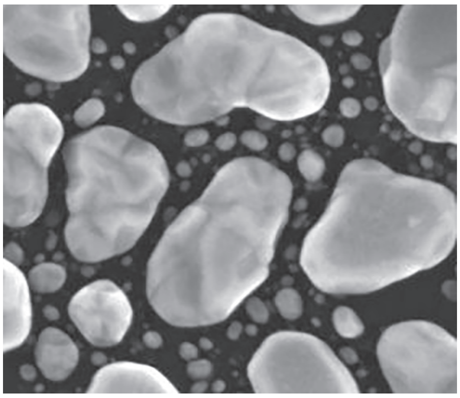
a) The first image I_{OS1}



b) The 2nd image I_{OS2}

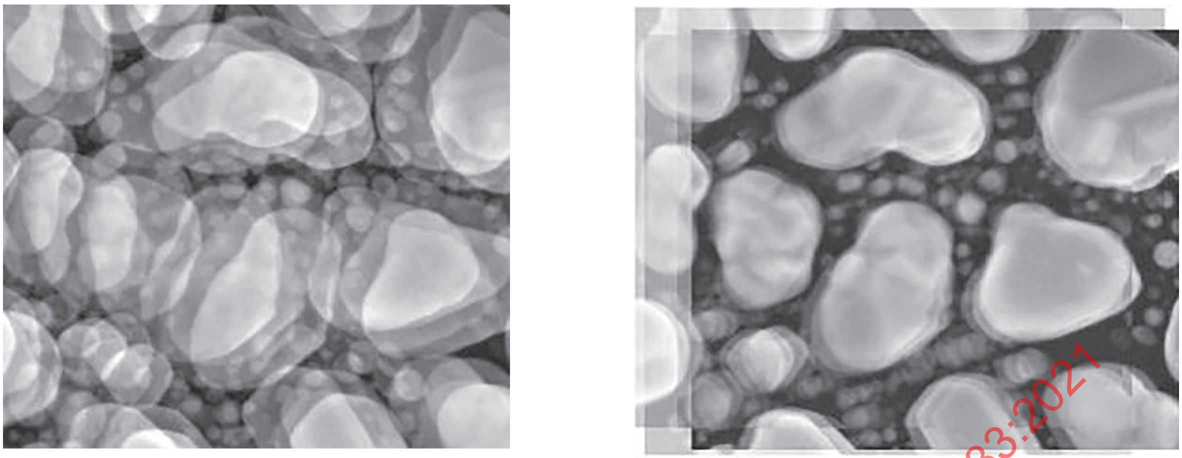


c) The 3rd image I_{OS3}



d) The 4th image I_{OS4}

Figure 6 — Four consecutive, slow-scan images for qualitative assessment of drift-related distortions. 256 nm HFW. Refer to the bibliography [7]



a) Simple overlay image I_{SO} b) Drift adjusted overlay image I_{DAO}

Figure 7 — Overlaid images by using 4 simple consecutive images and 4 drift adjusted images. Refer to the bibliography [7]

To assess the fast scan imaging repeatability performance, set the focus and astigmatism to their best to prove the best image sharpness for the accelerating voltage, electron beam current and working distance which are selected by the users for their own purposes. Then take many consecutive images at a suitable location over the resolution reference sample without changing anything or moving the sample. Select the areas from the 4 typical images (original images) $I_{\mathrm{OS}n}$ $(n=1,\ 2,\ \cdots)$ so that their largest common sections are nearly centred (drift adjusted) as the assessment of the slow scan imaging repeatability. Add the 4 original images $I_{\mathrm{OS}n}$ to the report, and add their selected (drift adjusted) images for the overlay, and the overlay image I_{DAO}. Valuate that the overlay image I_{DAO} is allowable or not for the quantitative measurements, and add the valuations to the report.

Concerning the software-based technics, refer to the annexes "B.4 Measurement of the distortions caused by high-frequency motions or stage vibration" and "B.5 Measurement of the drifts and drift-compensated imaging".

See Clause 12, Annex B and "B.3 Measurement of image drift and the drift-related distortions by using image overlay" and "B.5 Measurement of the drifts and drift-compensated imaging".

7.3 Evaluation of the drift and the drift-related distortions by using cross-correlation function (CCF)

This method can be used to evaluate the quantities of the drifts and distortions with some numerical indexes at the same time.

7.3.1 Measurement of the drifts by using the CCF

For the acquired images $I_1, I_2, \cdots, I_n, \cdots$, as shown in Figure 8, calculate the cross-correlation function $F_{\mathrm{CC}}(I_1, I_n)$, where I_1 is the image at the start of the measurement, and I_n $(n=1,2,\cdots)$ is the n-th image in the measurements. The function $F_{\mathrm{CC}}(I_1, I_1)$ is same as the auto-correlation function $F_{\mathrm{AC}}(I_1)$ of the image I_1. Let (H_{P1}, V_{P1}) and (H_{Pn}, V_{Pn}) be the peak positions of the two-dimensional distribution of the $F_{\mathrm{AC}}(I_1)$ and the $F_{\mathrm{CC}}(I_1, I_n)$ respectively, and let $2 \leq n$, then Formula 2 and Formula 3

$$D_{\mathrm{H}n} = H_{\mathrm{P}n} - H_{\mathrm{P}1} \qquad (2)$$

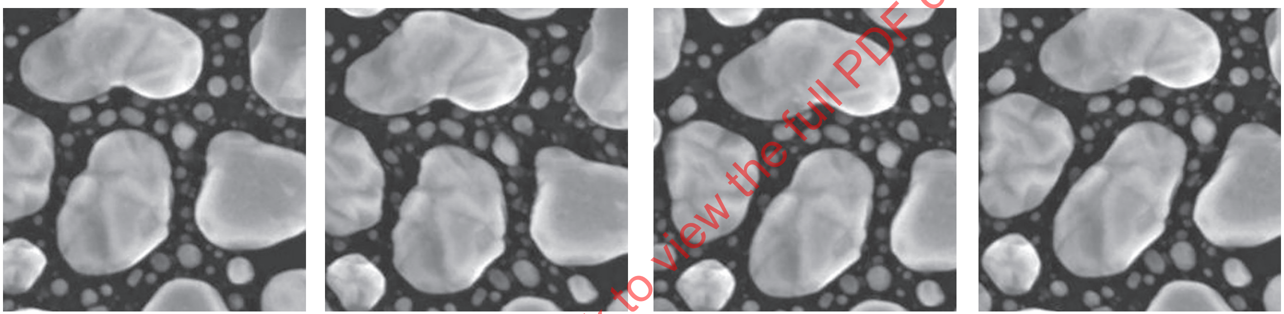
$$D_{Vn} = V_{Pn} - V_{P1} \quad (3)$$

mean the drift quantities for horizontal (H) and vertical (V) directions. If $D_{Hn} < 0$, then the area of the scan is shifted to the left and the obtained scanning image is shifted to the right. In a similar manner, if $D_{Vn} < 0$, then obtained scanning image is shifted to the bottom. Calculate $F_{CC}(I_1, I_n)$ and obtain the (H_{Pn}, V_{Pn}) for I_n $(n=2,3, \cdots)$ then the drift (D_{Hn}, D_{Vn}) for each image I_n can be obtained.

In the case of SEM imaging, the electron beam is scanned from left to right in the horizontal (H) coordinate, and scanned from top to bottom in the vertical (V) coordinate. Therefore, the larger value of the vertical coordinate means lower position in the image. This coordinate system (H, V) is different from the usual right-handed system (X, Y). Apart from the vertical direction, similar drifts data can be obtained as shown in the tables from Table B.1 to Table B.4, and Figure 5 from subfigure a) to d). Valuate that the drifts are allowable or not in the quantitative measurements for the specified (selected) images I_n.

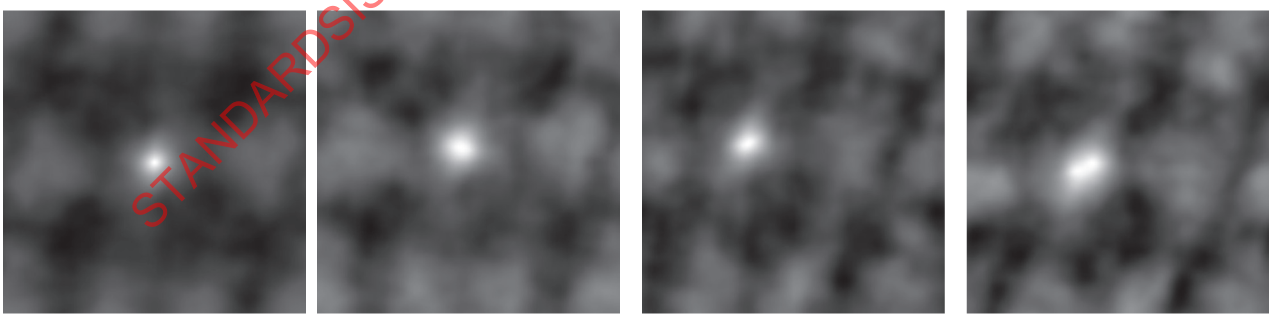
Report the original specified images I_n $(n=1,2,\cdots)$ as shown in Figure 8, auto-correlation function $F_{AC}(I_1)$ and cross-correlation functions $F_{CC}(I_1, I_n)$ as shown in Figure 9, the peak positions (H_{P1}, V_{P1}) and (H_{Pn}, V_{Pn}) and the drifts (D_{Hn}, D_{Vn}) as listed in Table B.5 and the valuation.

See Clause 12, Annex B.6 and "B.6.1 Measurement of the drifts by using the CCF".



a) The first image I_1 b) The 2nd image I_2 c) The 3rd image I_3 d) The 4th image I_4

Figure 8 — Four consecutive, slow-scan images I_n $(n=1,2,3,4)$ selected from Figure 6, 180 nm HFW



a) $F_{AC}(I_1)$ b) $F_{CC}(I_1, I_2)$ c) $F_{CC}(I_1, I_3)$ d) $F_{CC}(I_1, I_4)$

Figure 9 — Examples for the auto-correlation function $F_{AC}(I_1)$ and the cross-correlation functions $F_{CC}(I_1, I_n)$ for $n=2,3,4$

7.3.2 Measurement of the distortions by using the CCF

Let $F_{BAC}(I_1)$ and $F_{BCC}(I_1, I_n)$ be the binarized pictures of $F_{AC}(I_1)$ and $F_{CC}(I_1, I_n)$ respectively by using the thresholding level T_B which is the half of the maximum signal. If the signal range of the function $F_{CC}(I_1)$ and $F_{CC}(I_1, I_n)$ is normalized to [0, 255], then T_B = 255/2. Each binarized picture shows the cross section of the peak distribution at the signal intensity T_B = 255/2 as shown in Figure 10.

a) $F_{BAC}(I_1)$ **b)** $F_{BCC}(I_1, I_2)$ **c)** $F_{BCC}(I_1, I_3)$ **d)** $F_{BCC}(I_1, I_4)$

Figure 10 — Examples for the binarized pictures $F_{BAC}(I_1)$ and $F_{BCC}(I_1, I_n)$ for $(n=2,3,4)$

Let $A_{ACF,1}$ and $A_{CCF,n}$ be the areas of the binarized pictures $F_{BAC}(I_1)$ and $F_{BCC}(I_1, I_n)$ respectively. If the ratio

$$k_{A,n} = A_{CCF,n} / A_{ACF,1} \tag{4}$$

becomes larger than 1, then the drifts and the distortion will be increased. Note that the distribution of the particles in the image at the specified measurement number n should be nearly similar to that at the start of the measurement to reduce the artefact owing to the change of the mean diameter of the particles. Valuate that the ratio $k_{A,n}$ is allowable or not in the quantitative measurements for the specified (selected) images I_n $(n=2,3,\cdots)$.

Report the binarized pictures $F_{BAC}(I_1)$ and $F_{BCC}(I_1, I_n)$, the areas $A_{ACF,1}$ and $A_{CCF,n}$ and ratios $k_{A,n}$ as listed in Table B.6, and the valuation.

See Clause 12, Annex B.6 and "B.6.2 Measurement of the distortions by using the CCF".

8 Measurement of electron-beam-induced contamination

Lack of electron-beam-induced contamination is key to high-quality SEM imaging and measurements in some applications. It is possible to achieve essentially clean SEM operation for some types of samples, meaning that there is no formation of visible electron-beam-induced deposition of carbonaceous layer. With clean instrument and a clean sample the operator can indefinitely image or measure the sample, instead of covering it with a carbonaceous layer, i.e., contaminating it. Electron-beam-induced contamination can arise from an unclean sample or instrument or both. Both instrument and sample can be cleaned so that essentially contamination-free operation is as far as possible. See Annex C for further pertinent information.

However, there are some cases where polluted samples were inserted into the sample chamber for their own purpose. After these works, there is the case which the effect of a cleaning process applied is insufficient. Then refer to the following procedure for the cleaning methods and the assessment of cleanliness. It is very helpful of the procedure to maintain the cleanliness of the instrument in some degrees for users.

8.1 Cleaning of the sample surface

Some original samples have unexpected contaminants on the surface. These are usual because the objects to be observed may be gathered from natural environment or may be selected from the industrial unprocessed products. These contaminants should be eliminated to perform excellent SEM observation as far as the structures of the samples are not damaged by several cleaning process. Several proper cleaning methods are described below. However, the effect of the cleaning will depend on many factors such as the types of samples, quantities of contaminants on the surface, cleaning methods selected and applied, operating conditions of some cleaning devices etc. The best methods and conditions should be taken for user's application and requirements accordingly:

1) Baking the sample in the vacuum or in the atmospheric pressures under the permitted temperature[8].

2) Plasma cleaning of the samples in the vacuum pressures[9],[10],[11],[12].

3) Ultraviolet (UV) irradiation and the exposure to Ozon in the vacuum or in the atmospheric pressures[13].

4) Electron beam shower[14],[27].

5) Immersion in cleaning or etching solutions[10],[12].

Read the instruction manuals precisely and carefully when some commercial devices or solutions are applied for the cleaning. The cleaning methods using optional devices 2) and 3) are usually effective and interact with surface area only. This feature is superior compared with other methods in general. However, the effect of the cleaning is lower than some chemical methods 5) if the thickness of contamination layer is large[15].

In the case of some semiconductor samples such as Si, so called piranha solution (piranha etch) may be applied[10],[12],[16] to clean the sample surfaces as the elimination procedure of the photoresist residues from the wafers under the strictly regulated conditions in factories. The ability of the solution to dissolve the contaminants is very high[16].

On the other hand, any SEM users working in installation rooms or in laboratories should take maximum precautions when using the piranha solutions because the solutions are very dangerous. The users shall be familiar with the Safety Data Sheets (SDS)[17] provided by suppliers, and with the safety guidelines or the standard operating procedure (SOP) which are published by official institute[18].

WARNING — The SDS will be made according to the standard format of the Globally Harmonized System of Classification and Labelling of Chemicals (GHS)[19]. Read each section (1 to 16) precisely for user's and co-worker's safety. Especially, Hazards identification (SECTION 2), First aid measures (S. 4), Firefighting measures (S. 5), Accidental release measure (S. 6), Handling and storage (S. 7), Exposure controls/personal protection (S. 8), Stability and reactivity (S. 10), Toxicological information (S. 11) etc.

The use of the piranha solution is prohibited in some laboratories owing to the hazard to the human beings. To prevent some avoidable accidents, typical examples should be referred[20].

If the alternatives can be used for the described purposes, those are applied instead of the piranha solutions[21].

8.2 Cleaning of the inner surfaces of the sample chamber

There are some cases that inner surfaces of the chamber are not clean ideally. These are caused by the several reasons as follows:

a) Insertion of the dirty or polluted samples into the chamber.

b) Use of the unclean sample holders.

c) Use of some types of lubricant oil for mechanical movements.

d) Unclean components of the standard and the electron-optional devices in the chamber, the sample stage and the electron-optical column etc.

These factors depend on the history of the operations, mechanical and electron-optical structures of the SEM. The reduction of some of contaminants by using following method are applied although the cleaning is not ideal or perfect. Some of the cleaning methods described in 8.1 can also be applied for this purpose.

1) Baking the sample chamber in the vacuum or in the atmospheric pressures under the permitted temperature.

2) Plasma cleaning of the components or the inner surface of sample chamber in the vacuum pressures.

3) Ultraviolet (UV) irradiation and the exposure to Ozon in the vacuum or in the atmospheric pressures.

8.3 Measurement method of the contamination

There are several methods to valuate the quantity of the contamination. Some of the proper or specialized methods are listed below. If these valuations are required for the quantitative SEM measurements, chose the methods which will be applicable to the present SEM and will be suitable for the purpose of the measurements.

8.3.1 Measurement of the height of the contamination growth

The assessment method described below is based on the measurement of the contamination growth in the operating conditions used for required quantitative SEM measurements.

1) Set the same operating conditions (accelerating voltage, beam current, working distance, etc.) which will be used in the quantitative SEM measurement including the samples except for the image magnification.

2) Set the best resolution imaging parameters which will be specified by the manufacturer for proving the SEM performance concerning resolution if possible.

3) To assess the contamination being in sight, set the image magnification M larger than about M_0 = 300 000 x or so. Smaller scan area or fine beam diameter will increase the rate of contamination growth. If higher image magnification cannot be applied in the SEM, then stop the scanning and apply the spot beam to the sample[22].

4) Keep the beam irradiation time t_{irr} from about 5 to 10 min.

5) Set the image magnification lower to observe the contaminated position. Tilt the sample stage by the angle θ_{SS} from about π/6 to π/3 rad (30 to 60 degree) without losing the contaminated irradiated position.

6) Measure the length or L_C of the corn or the outer ring made by the contamination growth. The height h_C [nm] of the corrected by the tilt angle θ_{SS} [rad] is given by Formula 5:

$$h_C = L_C / \sin(\theta_{SS}) \quad (5)$$

where

L_C is the length of the corn or the outer ring, typically expressed in nm;

θ_{SS} is the tilt angle of the sample stage, typically expressed in rad.

Valuate the measured height is allowable or not in the quantitative SEM measurements. Report the measurement conditions, results and the valuation.

Regarding to the relations between the rate of contamination growth and the beam conditions, some historical studies will be helpful if the conditions are different from the above[22]-[27]. The beam size and the beam current density will affect the rate.

8.3.2 Measurement of relative carbon concentration of the contamination by the X-ray analysis

The assessment method described below is based on the measurement of the surface concentration of the carbon compared with the C (graphite) standard sample by applying X-ray micro beam analysis.

1) Set the same operating conditions (accelerating voltage, beam current, working distance, etc.) which will be used in the quantitative SEM measurement including the samples except for the image magnification.

2) To assess the contamination being in sight, set the image magnification M from about M_0 = 10 000 x to 50 000 x or so. In this case, scan areas are from several microns to 10 microns order. Alternatively, the spot beam with the similar beam diameter can be applied without the scanning.

3) Measure the C-Kα intensities $I_C(t)$ [cps] at the start $t = t_S$ [min], measure $I_C(t)$ for every several minutes, and measure $I_C(t)$ at the end of measurement t_E = about 30 [min].

4) Measure the C-Kα intensity I_{CS} [cps] for the C (graphite) standard sample by using the specified beam current in 1). Then calculate the rate R_C [%/min] of the contamination growth per minute.

$$R_C = 100 \cdot \frac{I_C(t_E) - I_C(t_S)}{I_{CS} \cdot (t_E - t_S)} \quad (6)$$

where

t_S, t_E	are the times at the start and end of measurements respectively, expressed in minutes [min];
$I_C(t_S)$, $I_C(t_E)$	are the C-Kα intensities at the start and end of the measurements respectively, expressed in count per second [cps];
I_{CS}	is the C-Kα intensity for the C (graphite) standard sample, expressed in count per second [cps].

5) Valuate that the rate R_C is allowable or not in the quantitative SEM measurements. Report the measurement conditions, results and the valuation.

8.3.3 Measurement of the surface contamination by the change of SEM signal intensities

The assessment method described below is based on the measurement of the change of the signal intensities of the secondary electron (SE).

1) Set the best resolution imaging parameters, including the magnification specified by the manufacturer for proving that the SEM meets its resolution specification. Take one SEM image and save the image. As a typical sample with the clean surface, the amorphous silicon patterns (for example, [2]) can be used.

2) Go up to twice as high magnification and continuously image the sample for 10 min.

   Go back to the original magnification, and take another image.

3) If there is any visible darkening as shown in Figure C.1 b), whitening such as Figure C.5 b) or the image introduced in the reference[28], swelled frame as indicated in Figure C.1 a), any carbonaceous

structure in the middle of the second image, there is the possibility that the instrument or the sample or both fail to meet these requirements in the laboratory.

To compare the changes of the SE signals of the acquired images quantitatively (numerically), obtain the line profiles by setting the averaging areas near the centre of the images. In this case, the SE signals I_{SE} shall be normalized for a given sample by the adjustments of contrast and the brightness. For an example, set $I_{SE} = 0$ when beam is turned off and $I_{SE} = 1$ when the beam is irradiated at bright and flat position. Valuate that the level of contamination is allowable or not in the quantitative SEM measurements.

4) If the specification for the image sharpness was not met owing to the obvious contamination growth, first clean the sample by using piranha solution for semiconductor surfaces or alternative substitutions for metallic surfaces as described in 5) of 8.1. after the valuation of the above 3) of this subclause.

WARNING — Some of the samples for the calibration of image magnification will be applied for this purpose. For examples, refer to the ISO 16700, Annex A, "Reference materials for magnification". However, their surfaces are sometimes coated with metallic films which will be corrosive to piranha solutions and will react with them violently.

5) If the instrument -with the clean sample- fails the test again, it is the source of contamination due to the inner surfaces of the sample chamber needs to get cleaned with low-energy plasma or other cleaning process such as 3) listed in 8.2, until no visible contamination develops.

6) Repeat these procedures 1) to 5) at required primary electron-beam accelerating voltage until the SEM has specifications for image sharpness, and for other values such as drift and drift-related distortions. Generally, electron-beam-induced contamination build-up intensifies at low electron-beam accelerating voltages.

7) Upon successful accomplishment of this assessment, record the final image pairs (before and after the ten minutes continuous imaging at the specified magnification) to prove contamination-free operation, and record beam current as well. Report all results as electron-beam-induced contamination performance.

See Clause 12, Annex C, "C.2 Contamination growths for the unclean sample and the unclean instrument", "C.3 The effect of the plasma cleaning and the electron beam shower" and "C.4 The quantitative treatments of the image signal" for further pertinent information.

9 Measurement of the image magnification and linearity

Setting the image magnification, in other words, the calibration of magnification is essential for measuring size and shape based on SEM images. It is not unusual that the actual image magnification is different from what is stated by the SEM either as a certain length of a fiducial mark, the horizontal field width (HFW) or as the magnification (these may not agree with each other either). It is also not unusual that the accuracy of the image magnification changes in ranges, so it is indispensable to measure it for accurate measurements. For these, it is recommended to place an image magnification calibration by using a reference material (RM) or a certified reference material (CRM) at the same height as the sample under measurement, as the magnification changes with working distance and focus setting. The *X* and *Y* image magnifications should be equal, otherwise, the image is distorted, for example, a circular object appears oval.

There are many cases where the observing and the operating conditions of the SEM are much different from the standard conditions. It is very useful for users to understand the real or the calibrated magnification by using the procedures described below accordingly. The procedures are applicable to not only users specialized purposes but also general aims.

9.1 Measurement of the image magnification

To measure the accuracy of image magnification in *X* and *Y* direction follow the procedure in ISO 16700. According to the ISO 16700, 6.5 basic conditions are as follows:

— The measured distance l_S should be approximately ten times larger than the pitch d.

— Repeat the measurement at least three times at separate locations at least 3 mm apart on the recorded (displayed) image.

To decrease the effect of the hysteresis of the magnetic objective lens, use lens clear button if the system has this function. Or adjust the focus intensity from weaker side (for longer working distance WD) to stronger side (shorter WD). And verify that the change of the WD is minimized in the focus adjustment.

The measured width of the patterns depends on the signal types (SE or BSE) and the detector types of the signal etc. Specify these types even though the measured pitch does not depend on these types. The image magnification is measured by using the several number (e.g. 10) of pitch patterns.

Set the focus and astigmatism to their best to prove the best image sharpness for the accelerating voltage, electron beam current and working distance which are selected by the users for their own purposes at suitable locations over the reference sample.

For the image magnification setting M, select the appropriate pitch d from the RM or the CRM. To satisfy the ideal condition $10 \cdot d \leq l_S$, determined the total number n_P of pitches. Let l_S be the total measured length by the scaler (on the screen or photograph), then measured mean pitch length d_S is given by Formula 7:

$$d_S = l_S / n_P \quad (7)$$

Let N_R be the repeated number of measurements, say $3 \leq N_R$, then obtain the averaged value d_{SA} of d_S. The calibrated magnification M_C is given by Formula 8:

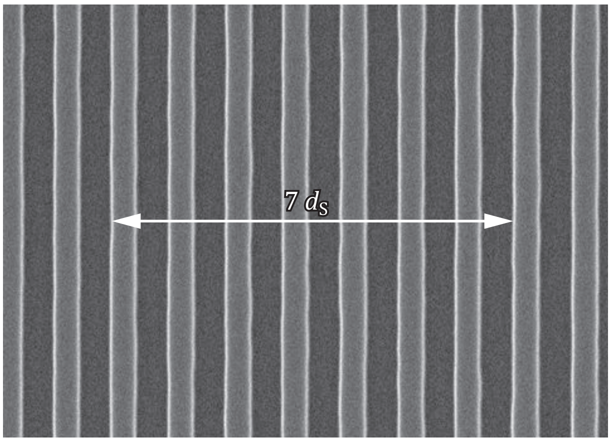
$$M_C = M \cdot (d_{SA} / d) = (M \cdot d_{SA}) / d \quad (8)$$

which is equivalent to Formula (1) of ISO 16700.

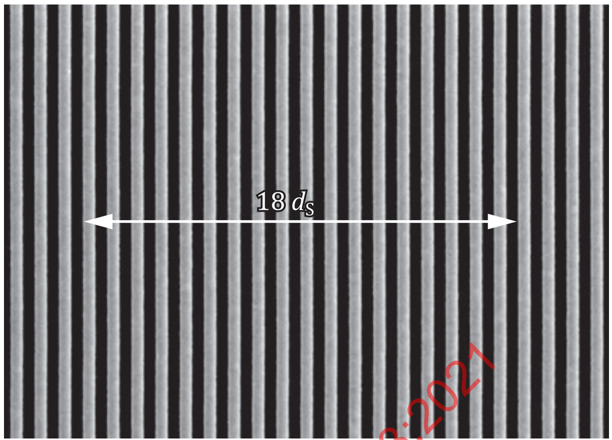
The number of magnifications should be determined based on the number of magnification ranges of the SEM, which is known by the manufacturer's service personnel. Service personnel can correct the instrument's image magnification setting to achieve satisfactory accuracy. Additionally, carry out these calibration measurements at the magnifications used for quantitative measurements if required by the user.

Figure 11 shows typical examples of the measurement of the image magnifications by using the various pitches and the lengths $l_S = n_P \cdot d_S$.

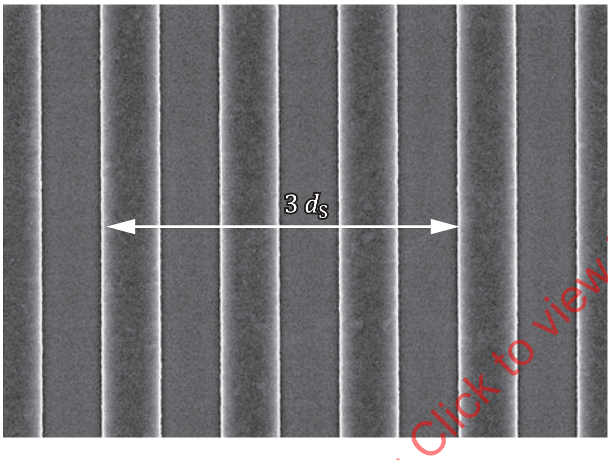
Valuate that the accuracy A_m of the image magnification and that A_s of the scale marker are allowable for the quantitative measurements. Report the calibration results in *X* and *Y* directions and the valuations as shown in Table D.1 to Table D.3. Add representative images that show where on the reference material (RM) the image magnification calibration measurements took place.



a) Pitch of the RM: d = 100 nm.
Total number of pitches: n_P = 7
Image Magnification setting: M = 150 000.
Accelerating voltage: V_a = 15 kV



b) Pitch of the RM: d = 500 nm.
Total number of pitches: n_P = 18
Image Magnification setting: M = 12 000.
Accelerating voltage: V_a = 1 kV



c) Pitch of the RM: d = 1 000 nm.
Total number of pitches: n_P = 3
Image Magnification setting: M = 30 000.
Accelerating voltage: V_a = 15 kV

Figure 11 — Measurement of the image magnification using various pitches

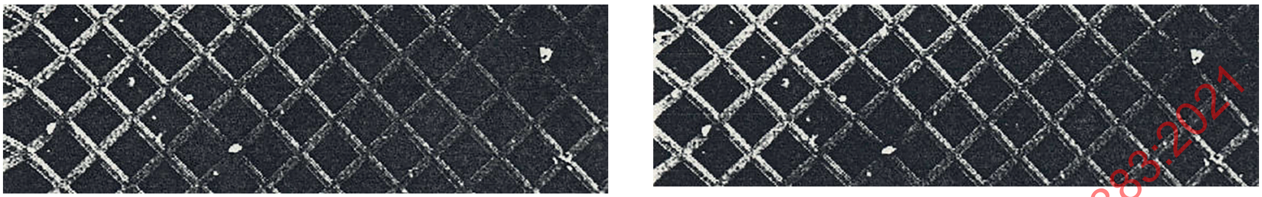
See Clause 12, Annex D and "D.2 Calibration of the image magnification" for further pertinent information.

9.2 Measurement of the image linearity

Lack of linearity in the primary electron beam scanning makes a given size object appear different in size and shape, depending on what part of the field of view (SEM image) it is. This kind of distortion is not uncommon, especially with SEMs that use coil-based beam scanning and can cause significant errors in quantitative measurements. The distortions are usually caused by the delay or the drop down of the scanning signal owing to the failure of the electric circuits. These distortions due to the failures may be observed at the fast scanning speed or at the lower magnifications in particular. With some SEMs, it is advantageous to not use that portion of the SEM image where the lack of linearity is too high.

To assess the linearity, use a sample with the suitable mesh structures (square grids). Rotate the mesh 45 degrees from orthogonal horizontal and vertical position to observe the distortion easy. This reason is that the distorted scanning signals are not simple but complicated. In this case, pay attention to the left edge of the image and take an image. Valuate that the distortions are allowable or not for the quantitative measurements. It is recommended to omit the distorted area from quantitative measurements.

Figure 12 shows examples of the distorted images caused by the scanning system at first scan speed. The sample is the mesh (square grid) with d = 100 µm pitch and is rotated 45 degrees by the specimen stage. Severe distortions are observed in Figure 12 a) for the left edge approximately 5 % of the image. On the other hand, the distortions are reduced in Figure 12 b).



a) Severe distortions for the left edge approximately 5 % of the image. Sample: mesh with *d* = 100 µm pitch

b) Reduced distortions for the same observing area

Figure 12 — Example for edge distortions at first scan speed

10 Measurement of background noise

In SEMs, the primary electron beam in a complex interaction with the sample generates a number of signals, which can be detected by various types of detectors. The intensity of the signals is proportional to the primary electron beam current. Ideally, with primary electron beam turned off or blocked, there should be no signal detected. In practice, SEMs do have some remaining “signal”, the so-called background noise. The frequency and intensity distribution of the background noise depend on the SEM, the type of detector and signal chain used. It may also change with time, as a detector may become noisy or as adverse signals may arise from some source. It is important to assess this noise, because it is present in all images and measurements. If too high amount and/or non-random noise indicate a problem, i.e. the SEM needs repair, or if no improvement is feasible, then the inevitable noise must be known. The noise depends on scanning speed and the setting of noise filters or image processing functions which can be installed in some types of SEM.

It is important to know the noise properties under the given observing conditions because the properties are used to improve the image quality by adjusting both the signal gain for a detector and the electron beam current.

If the assessment of a selected detector is required, follow the procedures described in 10.1 and 10.2.

10.1 Evaluation methods by using noise profiles and processed images

To assess the background noise performance of the selected detector, use a suitable high-contrast (e.g. gold-on carbon) sample. Set the magnification, accelerating voltage, beam current, all pertinent parameters to the values which are selected by the users for their own purposes. Set the scanning speed, and noise filters if the system has, the focus, contrast, and brightness to their appropriate settings, and take one original image I_{OS}. Turn off the electron beam and take another image I_{BG} (background noise image). In some SEMs this can only be done by blocking the primary electron beam. Save both images taken by the selected detector for processing.

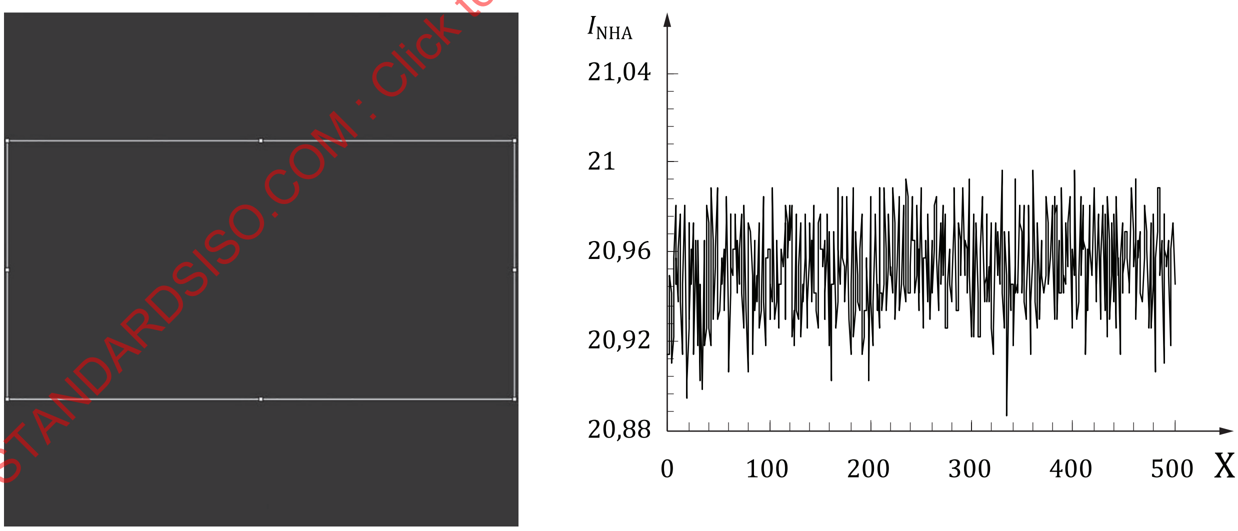
Report the minimum S_{MIN}, the maximum S_{MAX}, the mean S_{MEAN} and the standard deviation S_{STD} using the signal intensities of both the original images I_{OS} and the background noise image I_{BG} [4]. These values indicate the measure of the relative gain of the detector. It is important to use only actual image content portions of the images by the signals from the detector, omitting fiducial marks, letters, and other information.

To valuate the noise components quantitatively, obtain the horizontal and vertical line profiles L_{pHA} and L_{pVA} which are obtained vertically and horizontally averaged respectively for specified band areas from the background noise image I_{BG}. From these profiles, valuate that the noise intensities and the noise variations are allowable or not in the quantitative measurement. Report both profiles L_{pHA} and L_{pVA}, and the valuation. From Figure 13 to Figure 16 show typical examples of these background noise images I_{BG} and line profiles L_{pHA} and L_{pVA}.

Figure 13 a) shows the background noise image I_{BG1} obtained by turning off the beam irradiation, and shows the selected area (averaging area) to indicate the horizontal line profile L_{pHA} of the signal distribution for horizontal direction (e.g. 1-512) by averaging vertical direction (e.g. 128 – 384). The obtained horizontal line profile L_{pHA} is shown in Figure 13 b). The minimum difference can be expressed less than 0.005 in the figure even though the minimum value is 1 in the 8-bit signal system. This reason is the results of averaging of many elements (e.g. 256).

In a similar manner, Figure 14 a) shows the selected area (averaging area) to indicate vertical line profile L_{pVA} of the signal distribution for vertical direction (e.g. 1-512) by averaging horizontal direction (e.g. 128 – 384) for the same background noise image I_{BG1}. The obtained vertical line profile L_{pVA} is shown in Figure 14 b).

As a result of the comparison of Figure 13 and Figure 14, it is obvious that the profile L_{pVA} is almost similar to L_{pHA} and the noise distributions are natural for the both directions. Furthermore, the noise quantities are relatively small. As the valuation of the measurements for the background noise image I_{BG1}, the noise properties will be good under these operating conditions.



a) Averaging area for the vertical direction on the background noise image I_{BG1}

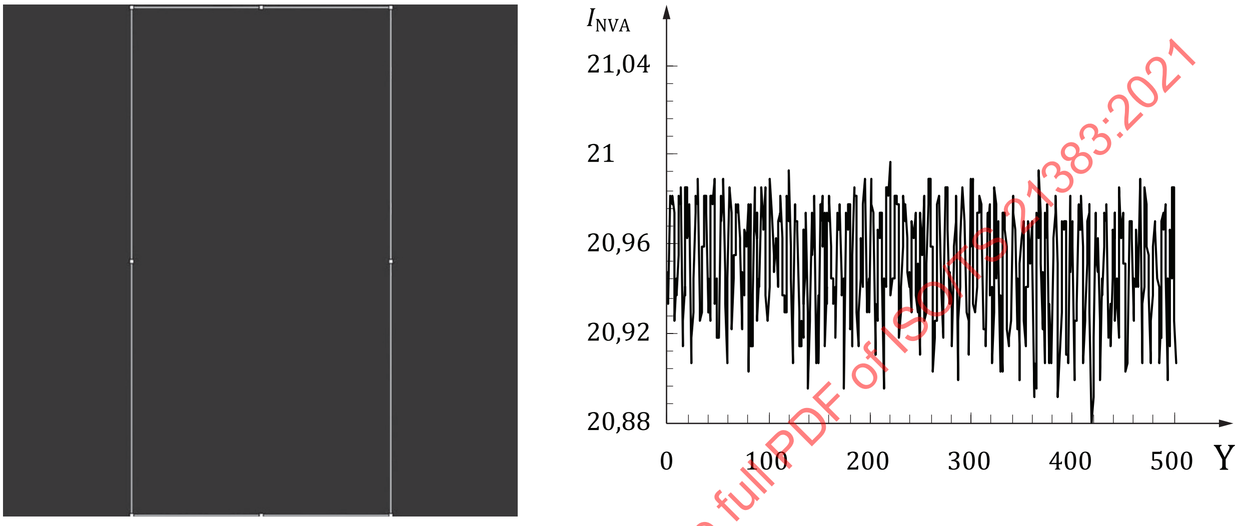
b) Horizontal line profile L_{pHA} of the vertically averaged noise

Key

X relative distance of the horizontal direction in pixel unit

I_{NHA} averaged noise for the horizontal line profile

Figure 13 — Averaging area on the background noise image I_{BG1} and the horizontal line profile L_{pHA}



a) Averaging area for the horizontal direction on the background noise image I_{BG1}

b) Vertical line profile L_{pVA} of the horizontally averaged noise

Key

Y relative distance of the vertical direction in pixel unit

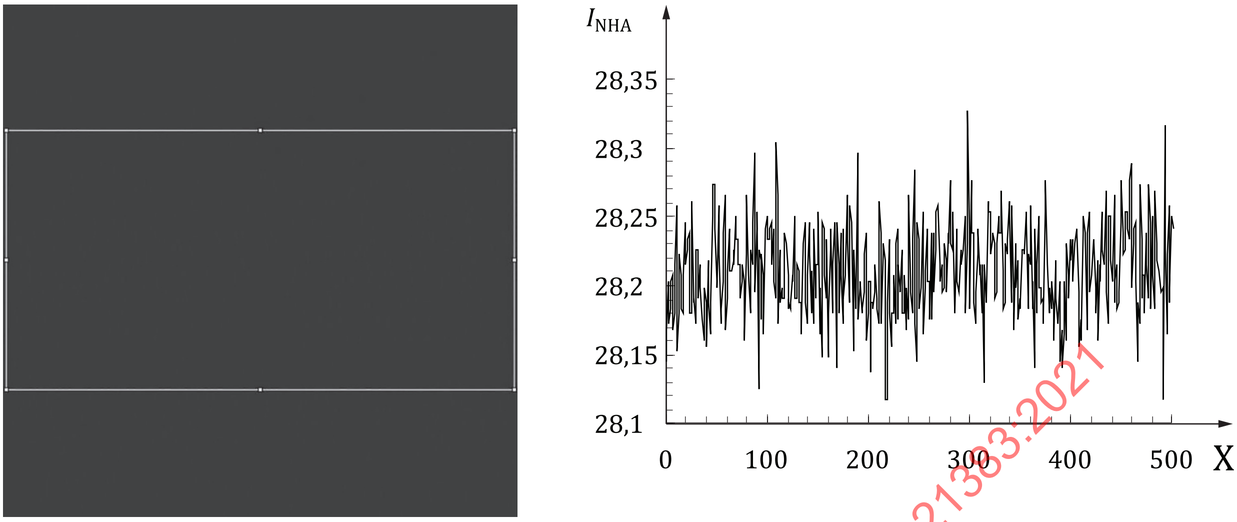
I_{NVA} averaged noise for the vertical line profile

Figure 14 — Averaging area on the background noise image I_{BG1} and the vertical line profile L_{pVA}

Figure 15 and Figure 16 show another example of the background noise image I_{BG2}. While the horizontal line profile L_{pHA} by averaging vertical is natural, vertical line profile L_{pVA} by averaging horizontal direction shows irregular fluctuations. Furthermore, the magnitude of the fluctuation for L_{pVA} is about several times larger than that for the L_{pHA}.

To reveal the various noise components on the background noise image I_{BG}, adjust the contrast and brightness for the enhancement of the contrast by using image processing function. Let I_{PB1} and I_{PB2} be the processed images obtained from the background noise images I_{BG1} and I_{BG2} respectively by a method of contrast enhancement. Figure 17 and Figure 18 show examples of these images I_{PB1} and I_{PB2} respectively by using so called "histogram equalization". By these adjustments, the difference of the background noise images I_{BG1} and I_{BG2} can be shown more clearly.

NOTE The S_{MIN}, the S_{MAX}, the S_{MEAN}, the S_{STD}, the profiles L_{pHA} and L_{pVA} are easily obtained by using analysing tools equipped in many free image processing software[4]. The histogram is sometimes used to adjust the contrast and brightness of the image to enhance the contrast of an image. For the automatic enhancement of the contrast, the functions so called "normalization" or "histogram equalization" are used[4].



a) Averaging area for the vertical direction on the background noise image I_{BG2}

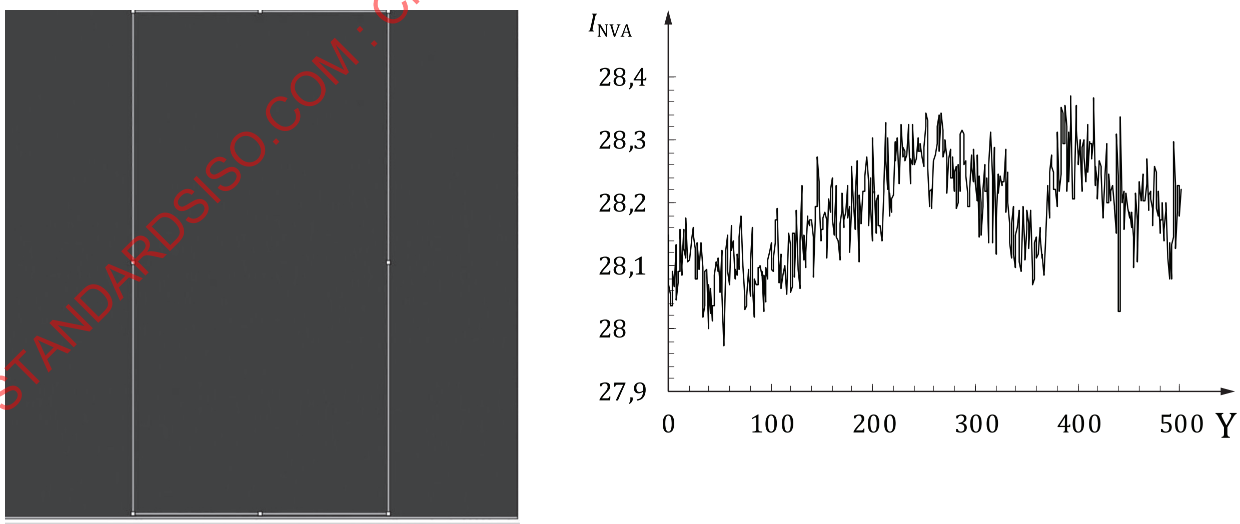
b) Horizontal line profile L_{pHA} of the vertically averaged noise

Key

X	relative distance of the horizontal direction in pixel unit
I_{NHA}	averaged noise for the horizontal line profile

Figure 15 — Averaging area on the background noise image I_{BG2} and the horizontal line profile L_{pHA}

As the valuation of these measurements for the background noise image I_{BG2}, the noise properties will be poor under these operating conditions.



a) Averaging area for the horizontal direction on the background noise image I_{BG2}

b) Vertical line profile L_{pVA} of the horizontally averaged noise

Key

Y relative distance of the vertical direction in pixel unit

I_{NVA} averaged noise for the vertical line profile

Figure 16 — Averaging area on the background noise image I_{BG2} and the vertical line profile L_{pVA}

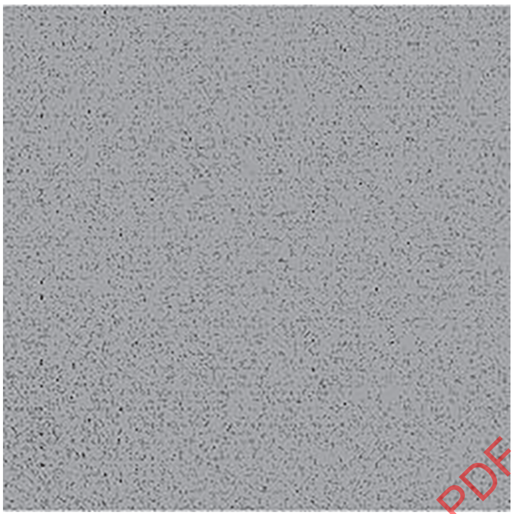


Figure 17 — Contrast enhanced image I_{PB1} from the background noise image I_{BG1}

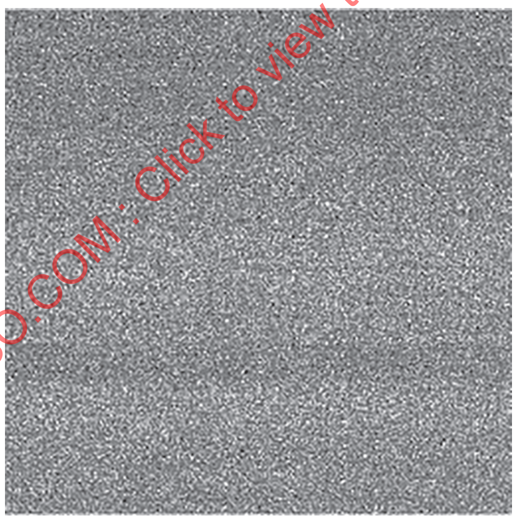
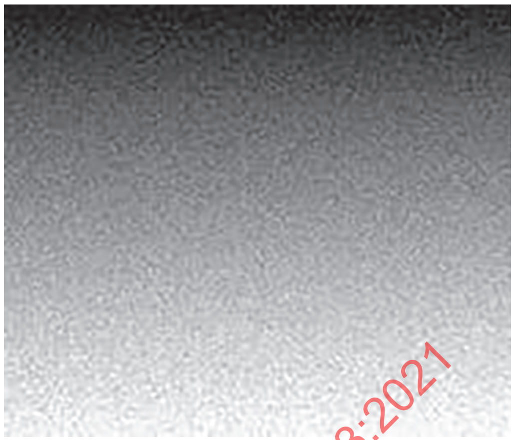


Figure 18 — Contrast enhanced image I_{PB2} from the background noise image I_{BG2}

Figure 19 shows a remarkable case in which the ramp of the brightness may be observable for the original image before image processing. Figure 19 a) shows the original background noise image I_{BG3}, and Figure 19 b) shows the processed image I_{PB3} by the histogram equalization. The ramp of the brightness is severe for the vertical direction.

a) Original background noise image I_{BG3}

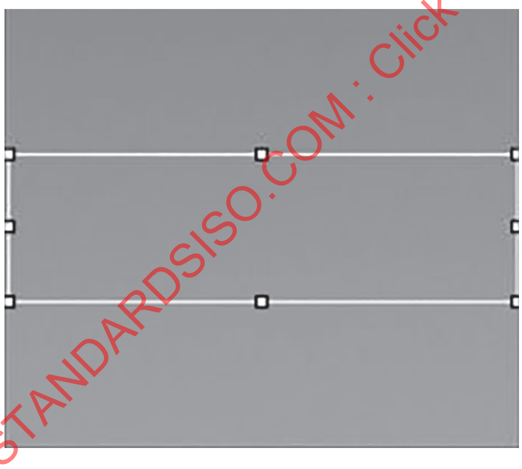


b) Contrast enhanced image I_{PB3} from the background noise image I_{BG3}

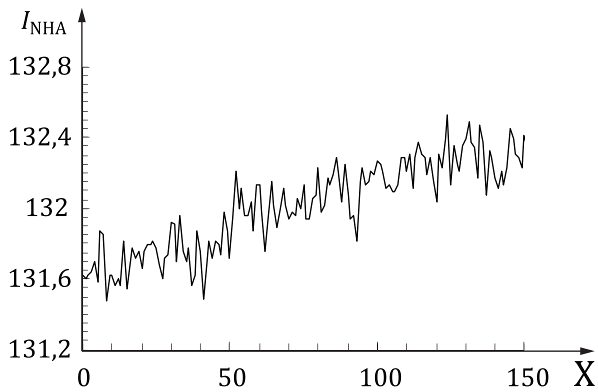
Figure 19 — Background noise image I_{BG3} and the processed image I_{PB3}

Figure 20 shows the selected area of the noise image I_{BG3} to indicate the horizontal line profile L_{pHA} of the noise distribution for horizontal direction (e.g. 1-192) by averaging vertical direction (e.g. 64 – 128). The obtained line profile L_{pHA} is shown in Figure 20 b).

In a similar manner, Figure 21 a) shows the selected area to indicate the vertical line profile L_{pVA} of the noise distribution for vertical direction (e.g. 1-192) by averaging horizontal direction (e.g. 64 – 128) for the same background noise image I_{BG3} as Figure 20. The obtained line profile L_{pVA} is shown in Figure 21 b). The change of the grey level about 25/255 = 0.1 (10 %) are observed.



a) Averaging area for the vertical direction on the background noise image I_{BG3}



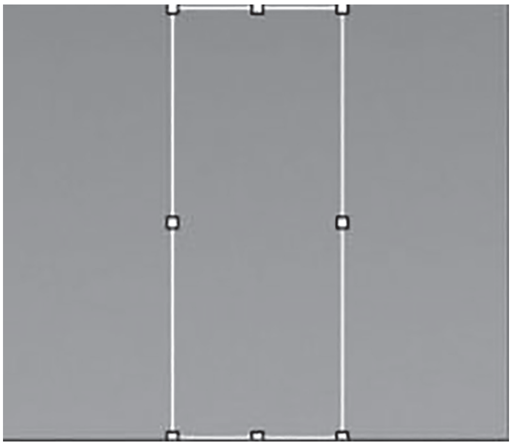
b) Horizontal line profile L_{pHA} of the vertically averaged noise

Key

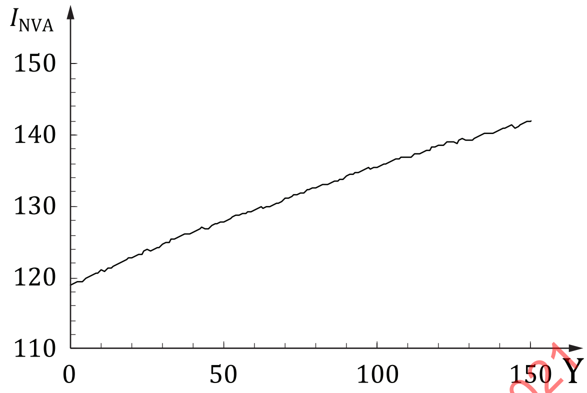
X relative distance of the horizontal direction

I_{NHA} averaged noise for the horizontal line profile

Figure 20 — Averaging area on the background noise image I_{BG3} and the horizontal line profile L_{pHA}



a) Averaging area for the horizontal direction on the background noise image I_{BG3}



b) Vertical line profile L_{pVA} of the horizontally averaged noise

Key

Y relative distance of the vertical direction

I_{NVA} averaged noise for the vertical line profile

Figure 21 — Averaging area on the background noise image I_{BG3} and the vertical line profile L_{pVA}

Observe the processed image I_{PB} compared with the profiles L_{pHA} and L_{pVA} and valuate that the non-random components, or slowly varying ramps are allowable or not in a quantitative measurement. Report the processed image I_{PB} with the line profiles L_{pHA} and L_{pVA} and the valuated results.

As the valuation of these measurements for the background noise image I_{BG3}, the noise properties will be very poor under these operating conditions.

Relatively simple quantitative noise measurement methods have been developed for noise calculations that work either in the analogue[29] or Fourier domain. These methods -due to their limitations and the design of the signal chain of the SEM- may not be useful for direct comparisons of the performance of different instruments, rather to compare the performance of a particular SEM over time using the same sample and SEM settings.

10.2 Evaluation methods by calculating numerical image properties

Generate the flat image I_{FB} in which the signal intensities of the elements (i, j) are the mean value S_{MEAN} of the background noise image I_{BG}. Let the background noise image I_{BG} and the flat image I_{FB} be the test image I_{test} and the reference image I_{ref} respectively. The flat image is applied as the reference image because the flat image will not depend on the type of filtering or image processing. And the denominators will not be excessively small values. Then calculate the peak signal-to-noise ratio S_{PSNR} and valuate that the value is allowable or not in the quantitative measurement. Add the value S_{PSNR} and the valuation to the report. The typical formulae to obtain S_{PSNR} are described as follows:

Let $I(i,j)$ be the signal intensity of the element (i,j) of the image I with the horizontal and vertical image size L_H [pixel] and L_V [pixel] respectively. The mean value S_{MEAN} of the image I is calculated by Formula 9:

$$S_{MEAN} = \frac{1}{L_H L_V} \sum_{i=1}^{L_H} \sum_{j=1}^{L_V} I(i,j) \tag{9}$$

Then, standard deviation S_{STD} of the image is defined by Formula 10 provided that the image data $I(i,j)$ are based on the population:

$$S_{STD} = \sqrt{\frac{1}{L_H L_V} \sum_{i=1}^{L_H} \sum_{j=1}^{L_V} \left(I(i,j) - S_{MEAN}\right)^2} \tag{10}$$

Let the image $I_{test}(i,j)$ $(=I_{BG})$ and $I_{ref}(i,j)$ $(=I_{FB})$ be the test image and the reference image (or filtered image) respectively. Then calculate the peak signal-to-noise ratio S_{PSNR} in decibel [dB] unit defined by Formula 11:

$$S_{PSNR} = 10 \cdot \log_{10} \frac{\{\max[I_{ref}(i,j)]\}^2}{\frac{1}{L_H L_V} \sum_{i=1}^{L_H} \sum_{j=1}^{L_V} \left[I_{ref}(i,j) - I_{test}(i,j)\right]^2} \tag{11}$$

Here, $\max\left[I_{ref}(i,j)\right]$ means the maximum value of the given n_{IM}-bit imaging mode. If n_{IM} = 8-bit imaging mode is applied, then $\max\left[I_{ref}(i,j)\right] = 255$. In the S_{PSNR} calculation, the reference image $I_{ref}(i,j)$ is set to the flat (plane) image I_{FB} defined by Formula 12:

$$I_{ref}(i,j) = S_{MEAN} \text{ for all } (i,j) \tag{12}$$

For an actual SEM image $I_{testS}(i,j)$ which is usually not the background noise image I_{BG}, the signal to noise ratio S_{SNR} in decibel [dB] unit of the test image $I_{testS}(i,j)$ can be calculated by the Formula 13:

$$S_{SNR} = 10 \cdot \log_{10} \frac{\sum_{i=1}^{L_H} \sum_{j=1}^{L_V} [I_{refS}(i,j)]^2}{\sum_{i=1}^{L_H} \sum_{j=1}^{L_V} \left[I_{refS}(i,j) - I_{testS}(i,j)\right]^2} \tag{13}$$

by using a suitable reference image $I_{refS}(i,j)$ which will be obtained from the test image $I_{testS}(i,j)$ applying image filter to reduce the image noise.

The following numerical values are obtained by applying the background noise image I_{BG1} cited in Figure 13 and Figure 14 using Formulae (9) to (12).

Mean value: S_{MEAN} = 21.9

Standard deviation: S_{STD} = 14.6

Peak signal-to-noise ratio in [dB]: S_{PSNR} = 24.8

If the assessments of the other detectors are required, follow the procedures described in 10.1 and 10.2.

See Clause 12.

11 Measurement of the primary electron beam current

Allowable stability of the primary electron beam current I_P is different for the purpose of quantitative measurement by the applied SEM. And the beam current stability depends on the type of electron source[30].

It is very useful for users to find the magnitude of the I_P and the stability of the I_P by applying the procedures described below. If the measured currents I_P s are much different from the nominal values, then the electron-optical column might have some irregular conditions for an example. If the stabilities of the I_P are not enough as compared with the values of a conventional measurement, then the electron source might have some troubles.

Using a Faraday cup which is located at the sample stage and a suitable picoampere meter[5], set the beam current larger than about 50 pA. The beam current range less than 10 pA is not recommended because the fluctuation of several hundred femto-ampere can be observed owing to a measurement system unrelated to actual values.

Measure the dark current several times at every minute, calculate the mean and standard deviation values, and report the dark current result. Verify that the values are negligible compared with the values of the beam current. Turn on the primary electron beam, aim it at the centre of the Faraday cup, and move up to high magnification, to make sure that all electrons fly into the hole.

WARNING — The measurement circuit from the Faraday cup must be closed (or grounded) electrically through the measurement device (picoampere meter). And the contact of the human body with the circuit must be avoided strictly not to get electric shock and not to be electrocuted. The electric charges will stay in the circuit corresponding to the capacities and the resistances of the circuit.

11.1 Ten-minute primary electron beam current measurement

Take 11 readings (at start and then at every minute), calculate the mean and the standard deviation values from the readings, report them as 10-minute beam current measurement result and valuate that the result is are allowable or not in a quantitative measurement. Figure 22 a) and Table 1 show the examples of the ten-minute primary electron beam current measurement. Report the valuated results and all individual readings as well.

11.2 Long-term primary electron beam current measurement

Take readings at start and then at every specified interval, calculate the mean and the standard deviation values from the readings, report them as long-term primary electron beam current performance metric, indicate the beginning and the end of the measurements, and valuate that the result is allowable or not in a quantitative measurement. Figure 22 b) and Table 2 show the examples of the long-term primary electron beam current measurement. Report the valuation and all readings as well.

Let t_{CM} [min] be the total time of beam current measurements. Then obtain the standard interval t_{IC} [min] of the recording (measurement) by the Formula 14:

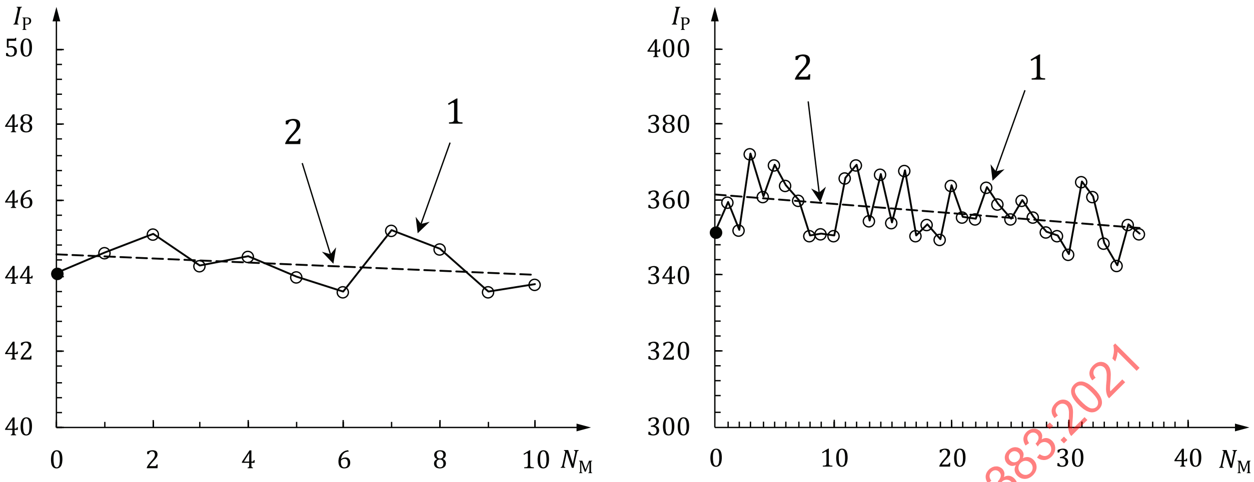
$$t_{IC} = 0{,}25 \cdot t_{CM}{}^{x} \qquad (14)$$

where

t_{CM} is the total time of drift measurements, expressed in minute;

$x = 0.605$ is the approximate constant.

The length of this assessment depends on the task at hand. An example is hours-long x-ray microanalysis, especially when compositional maps are produced.



Key

1 data series of the measured beam currents, shown in solid line

2 linear trendline to the data series, shown in dashed line

N_M number of the beam current measurements after the start

I_P beam current, expressed in pA

Figure 22 — Measurement of the primary electron beam current

See Clause 12, Annex E, and "E.2 Electron beam current stabilities for various landing energy" and "E.3 The handcraft of the Faraday cup and the usage".

Table 1 — Example of the ten-minute beam currents

Number of measurements N_M	Beam current I_P pA
0	44,1
1	44,6
2	45,1
3	44,3
4	44,5
5	44,0
6	43,6
7	45,2
8	44,7
9	43,6
10	43,8
Average	44,3
Standard deviations	0,56

Table 2 — Example of the three-hour beam currents

Number of measurements	Beam current
N_M	I_P
	pA
0	351,7
1	359,6
2	352,2
3	372,4
4	360,8
… [a]	… [a]
32	360,9
33	348,4
34	342,7
35	353,6
36	350,9
Average	357,0
Standard deviations	7,29

[a] The descriptions for the number N_M = 5 – 31 are abbreviated.

12 Reporting Form

Concerning the report of the results, refer to the typical requirements described in ISO/IEC 17025:2017, 7.8 "Reporting of results", especially 7.8.2 "Common requirements for reports (test, calibration or sampling)".

NOTE As an example, typical reporting style is shown as follows:

a) Title: Qualification of the SEM for Quantitative Measurements.

b) The name of Laboratory:

Address (Location):

c) The number of the calibration report:

d) The name and address of the customer, and relevant information:

e) The identification of the method used (e.g. ISO/TS 21383, ISO 16700).

f) Evaluated instrument: SEM:

1) The manufacturer's name, model name and the serial number of the SEM.

g) Instruments for the measurements (e.g. Picoampere Meter).

2) The manufacturer's name, model name and the serial number of the instrument.

h) The name and identification of the reference materials (RM) used.

i) The measurement conditions and results:

3) The specific operating values of accelerating voltage, beam current, image magnification, working distance, scanning speed and the type of the image (SEI or BEI) etc.

j) The valuation of the results:

k) The name of the person conducting the measurements.

l) The date and time when the investigation has been carried out.

Other similar reporting forms will be used, but the results specified above will be practical and useful.

Annex A
(informative)

Measurement of image sharpness

Various SEM manufacturers use some kind of spatial resolution calculation methods for their instruments, even if the “resolution” is not defined in scientifically sound ways. However, the precise algorithms are not open and are not approved officially. Therefore, their methods and some examples of the evaluated results will not be introduced here even though those methods have been used in some cases to determine the specification of the SEM supplied by the manufactures. Regular assessment of image sharpness has been found particularly beneficial in making the SEM operate at or even better its manufacturer-specified resolution performance.

ISO/TS 24597:2011 describes a procedure for obtaining good-quality, repeatable image sharpness measurement values.

The following examples listed in Table A.1 are obtained by using the programs based on the TS 24597 and the selected images shown in Figure 3.

Table A.1 — Examples of the evaluations of the image sharpness

Image file	V_a	I_p	L	HFW	L_p	N_{MS}	R_{PX}		R_L	
							DR	FT	DR	FT
	kV	pA	pixel	nm	nm/pixel		pixel	pixel	nm	nm
Figure 3 a)	15	43	512x512	265	0,517	1	3,59	3,64	1,86	1,88
	15	43	512x512	265	0,517	2				
	15	43	512x512	265	0,517	3				
---						...				
						Average	3,6	3,6	1,9	1,9
Figure 3 b)	1	43	512x512	657	1,283	1	3,34	2,63	4,3	3,4
	1	43	512x512	657	1,283	2				
	1	43	512x512	657	1,283	3				
						...				
						Average	3,3	2,6	4,2	3,3

Key

V_a accelerating voltage

I_p electron beam current (Probe current)

L image size, typically expressed in pixel

HFW horizontal field width

L_p pixel size, typically expressed in nm per pixel

N_{MS} number of measurements for image sharpness

R_{PX} image sharpness, typically expressed in pixel

R_L image sharpness, typically expressed in nm:

Average: Averaged value for the number of measurements N_{MS}

Valuation: As the results of the measurement of the image sharpness, the instrument meets requirements, good for our quantitative measurements.

Annex B
(informative)

Measurement of image drift and distortions caused by unintended motions

B.1 Overview

This annex gives some examples of following measurements and reporting forms:

— Image drifts within specified time intervals.

— Image drift and the drift-related distortions by using image overlay.

— The distortions caused by high-frequency motions or stage vibration.

— The drifts and drift-compensated imaging.

— The image drifts and distortions by using the cross-correlation method.

B.2 Measurement of image drifts within specified time intervals

The examples of the measurement methods and the test reports are shown as follows. The obtained graphs from the tables are shown in Figure 5 a) to d). These graphs will be included in the report as the components of the test results.

B.2.1 One-minute drift measurement

The graph for the displacement (D_X, D_Y) is shown in Figure 5 a).

Table B.1 — Example of the one-minute drift measurement

Number of measurements	Elapsed time	Displacement		Distance from origin
N_{MD}		D_X	D_Y	D_0
	s	nm	nm	nm
1	0	0	0	0,0
2	15	-0,49	1,1	1,2
3	30	-1,69	1,68	2,4
4	45	-2,04	0,91	2,2
5	60	-3,72	1,39	4,0

Key

N_{MD} number of measurements for image drift

D_X displacements for X direction, expressed in nm

D_Y displacements for Y direction, expressed in nm

D_0 distance $D_0 = \left(D_X{}^2 + D_X{}^2\right)^{1/2}$ from the origin, typically expressed in nm

max $|D_X|$, max $|D_Y|$ the largest absolute values of displacements from the origin in X and Y directions respectively

max D_0 the largest value of the distance D_0

Table B.1 *(continued)*

Number of measurements	Elapsed time	Displacement		Distance from origin
N_{MD}		D_X	D_Y	D_O
	s	nm	nm	nm
Largest displacement	max|D_X| or max|D_Y|	3,72	1,68	
Largest distance from origin	max D_O			4,0

Key

N_{MD} number of measurements for image drift

D_X displacements for X direction, expressed in nm

D_Y displacements for Y direction, expressed in nm

D_O distance $D_O = \left(D_X{}^2 + D_X{}^2\right)^{1/2}$ from the origin, typically expressed in nm

max $|D_X|$, max $|D_Y|$ the largest absolute values of displacements from the origin in X and Y directions respectively

max D_O the largest value of the distance D_O

Valuation: As the results of one-minute drift measurement, the instrument meets requirements, good for our quantitative measurement.

B.2.2 Ten-minute drift measurement

The graph for the displacement (D_X, D_Y) is shown in Figure 5 b)

Table B.2 — Example of the ten-minute drift measurement

Number of measurements	Elapsed time	Displacement		Distance from origin
N_{MD}		D_X	D_Y	D_O
	min	nm	nm	nm
1	0	0	0	0,0
2	1	-2,41	-0,26	2,4
3	2	-4,64	-0,9	4,7
4	3	-6,35	-1,46	6,5
5	4	-8,12	-0,43	8,1
6	5	-9,77	-0,24	9,8
7	6	-11,44	0,07	11,4

Key

N_{MD} number of measurements for image drift

D_X displacements for X direction, expressed in nm

D_Y displacements for Y direction, expressed in nm

D_O distance $D_O = \left(D_X{}^2 + D_X{}^2\right)^{1/2}$ from the origin, typically expressed in nm

max $|D_X|$, max $|D_Y|$ the largest absolute values of displacements from the origin in X and Y directions respectively

max D_O the largest value of the distance D_O

Table B.2 *(continued)*

Number of measurements	Elapsed time	Displacement		Distance from origin
N_{MD}		D_X	D_Y	D_O
	min	nm	nm	nm
8	7	-12,88	0,64	12,9
9	8	-14,37	1,19	14,4
10	9	-15,61	1,24	15,7
11	10	-14,44	0,2	14,4
Largest displacement	max|D_X| or max|D_Y|	15,61	1,46	
Largest distance from origin	max D_O			15,7

Key

N_{MD} number of measurements for image drift

D_X displacements for X direction, expressed in nm

D_Y displacements for Y direction, expressed in nm

D_O distance $D_O = \left(D_X{}^2 + D_X{}^2\right)^{1/2}$ from the origin, typically expressed in nm

max $|D_X|$, max $|D_Y|$ the largest absolute values of displacements from the origin in X and Y directions respectively

max D_O the largest value of the distance D_O

Valuation: As the results of ten-minute drift measurement, the instrument meets requirements, fair for our quantitative measurements.

B.2.3 One-hour drift measurement

The graph for the displacement (D_X, D_Y) is shown in Figure 5 c)

Table B.3 — Example of the one-hour drift measurement

Number of measurements	Elapsed time	Displacement		Distance from origin
N_{MD}		D_X	D_Y	D_O
	min	nm	nm	nm
1	0	0	0	0,0
2	3	-0,43	-1,34	1,4
3	6	0,32	-2,7	2,7

Key

N_{MD} number of measurements for image drift

D_X displacements for X direction, expressed in nm

D_Y displacements for Y direction, expressed in nm

D_O distance $D_O = \left(D_X{}^2 + D_X{}^2\right)^{1/2}$ from the origin, typically expressed in nm

max $|D_X|$, max $|D_Y|$ the largest absolute values of displacements from the origin in X and Y directions respectively

max D_O the largest value of the distance D_O

[a] The descriptions for the number N_{MD} = 6 – 17 are abbreviated.

Table B.3 *(continued)*

Number of measurements	Elapsed time	Displacement		Distance from origin
N_{MD}		D_X	D_Y	D_O
	min	nm	nm	nm
4	9	-0,69	-3,94	4,0
5	12	-0,48	-5,43	5,5
… [a]	… [a]	… [a]	… [a]	… [a]
17	48	-2,69	-21,96	22,1
18	51	-3,39	-23,24	23,5
19	54	-4,45	-24,6	25,0
20	57	-4,73	-25,84	26,3
21	60	-5,5	-27,25	27,8
Largest displacement	max$\|D_X\|$ or max$\|D_Y\|$	5,5	27,25	
Largest distance from origin	max D_O			27,8

Key

N_{MD} number of measurements for image drift

D_X displacements for X direction, expressed in nm

D_Y displacements for Y direction, expressed in nm

D_O distance $D_O = \left(D_X{}^2 + D_X{}^2\right)^{1/2}$ from the origin, typically expressed in nm

max $|D_X|$, max $|D_Y|$ the largest absolute values of displacements from the origin in X and Y directions respectively

max D_O the largest value of the distance D_O

[a] The descriptions for the number N_{MD} = 6 – 17 are abbreviated.

Valuation: As the results of one-hour drift measurement, the instrument meets requirements, fair for our quantitative measurements.

B.2.4 Long term (ten-hour) drift measurement

The graph for the displacement (D_X, D_Y) is shown in Figure 5 d)

Table B.4 — Example of the long term (ten-hour) drift measurement

Number of measurements	Elapsed time	Displacement		Distance from origin
N_{MD}		D_X	D_Y	D_O
	min	nm	nm	nm
1	0	0	0	0,0
2	12	1,2	0,91	1,5
3	24	2,8	1,22	3,1
4	36	3,33	1,61	3,7
5	48	4,71	1,32	4,9
... [a]	... [a]	... [a]	... [a]	... [a]
47	552	-22,59	-74,99	78,3
48	564	-24,16	-76,5	80,2
49	576	-23,37	-77,63	81,1
50	588	-24,21	-78,83	82,5
51	600	-24,63	-79,21	83,0
Largest displacement	max$\|D_X\|$ or max$\|D_Y\|$	24,63	79,21	
Largest distance from origin	max D_O			83,0

Key

N_{MD} number of measurements for image drift

D_X displacements for X direction, expressed in nm

D_Y displacements for Y direction, expressed in nm

D_O distance $D_O = \left(D_X{}^2 + D_X{}^2\right)^{1/2}$ from the origin, typically expressed in nm

max $|D_X|$, max $|D_Y|$ the largest absolute values of displacements from the origin in X and Y directions respectively

max D_O the largest value of the distance D_O

[a] The descriptions for the number N_{MD} = 6 – 46 are abbreviated.

Valuation: As the results of ten-hour drift measurement, the instrument does not meet requirements, poor for our quantitative measurements.

B.3 Measurement of image drift and the drift-related distortions by using image overlay

Geometry distortions impact imaging and measurement quality. For general imaging with the intended use other than measurements, qualitative assessment can be sufficient. For measurement purposes quantitative valuation is required, these unintended motions directly contribute to measurement uncertainty.

Simple repeated imaging, at a site of a sample that has a number of easily discernible details, can reveal a lot about the geometry distortions of the SEM. These distortions arise from unintended motions of the sample stage and the primary electron beam and can easily cause severe repeatability problems. Depending on the field of view (magnification) of the image and its acquisition speed, the amplitude and frequency of these unintended motions show up in various ways in the SEM image. Generally, as the field of view gets smaller, the geometry distortions of the SEM image become higher. At very low magnifications the dominant problems typically arise from imperfections of the scanning of the electron beam, at high magnifications both sample stage- and primary electron beam-related reasons are common. High-frequency mechanical vibrations and adverse electromagnetic fields manifest themselves either as image blur or as jagged edges of various sample features or both. In many instances, these cause only a couple or a few nanometres worth of geometry distortions, but that might be unacceptably high.

Traditionally slow image acquisition is used to acquire sufficiently noise-free images, but at high magnifications these are deformed by sometimes prohibitively high distortions. Slow image acquisition at low magnifications might help to avoid some of the problems, but for high-magnification imaging where modern SEMs excel, usually they perform at their best with high-speed image acquisition, especially when it is combined with methods that line up the inherently noisy fast images before they are added together to generate the final, sufficiently noise-free image. Some SEMs have built-in function for this; others can take advantage of free software available for this purpose.

The amplitude of unintended motions may be very similar for a wide range of fields of view (magnifications), but their contribution to image grows at smaller and smaller fields of view (at higher magnifications). It is the operator's task to find the best, optimized imaging conditions for the SEM. Imaging parameters, such as field of view, acquisition speed, frame time, the number of image pixels and dwell time, etc. may also influence these effects of distortions. There are qualitative and quantitative assessments available for the evaluation of unintended motions. Qualitative assessment is recommended to justify that the unintended motions do not cause noteworthy quality problems in imaging or significant errors in measurements. Quantitative assessment is necessary if this is not the case, i.e., the errors are unacceptably high, and or compensatory measures are necessary to achieve the required imaging and measurement quality.

Figure 6 shows an example for qualitative assessment for drift-related distortions using a sample of evaporated gold particles on polished carbon substrate. The 256 nm horizontal field of width images show both low-frequency- (drift-) and high-frequency- (vibration-) related problems.

These may not be obvious at a glance, but more thorough observation reveals the lack of good repeatability, which can be seen especially well, if one overlays the four images. If the SEM worked perfectly, all these four images would show exactly the same sample details, the four image frames would line up completely, and there would be no blurred, distorted or missing regions. As shown in Figure 7 a), a simple frame overlay exposes that there is indeed a drift-related problem. Whether the extent of this is negligible or not, depends on the intended use of the image. For example, for a biological cell sample, the actual shape of the various organelles may not be very important, as they naturally vary a great deal. On the other hand, for nanometre-scale particle measurements this amount of distortion might be completely unacceptable.

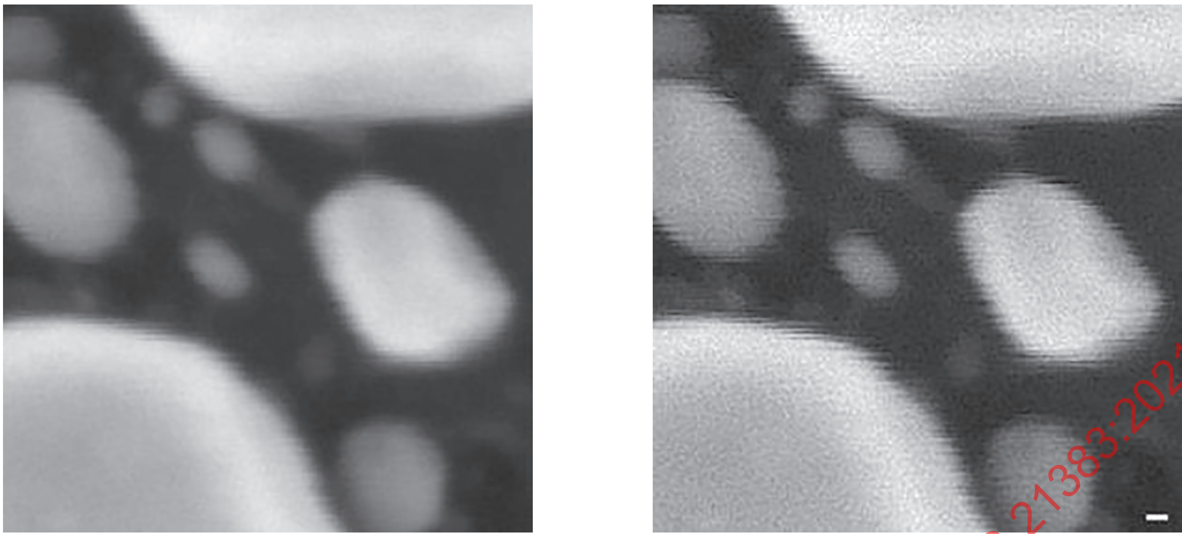
Regarding to the results of measured data as shown in "7.2 Evaluation of the drift and the drift-related distortions by using image overlay", the following valuation is conducted.

Valuation: As the results of the measurement of the drift-related distortions, the instrument meets requirements, fair for our quantitative measurements.

B.4 Measurement of the distortions caused by high-frequency motions or stage vibration

On Figure B.1 a), a digitally magnified portion of one of those images as shown in Figure 6 allows for qualitative assessment of distortions caused by unintended high-frequency motions, which can arise from mechanical vibrations, adverse electromagnetic fields, etc.

Figure B.1 b) illustrates that the actual repeatability problem is somewhat less severe, as one may find shared regions of the four repeated images that are less distorted and where better repeatability can be achieved at the expense of losing valuable regions of the images. Clearly, it is better to opt for superior imaging methods with less distortion, if possible.



a) Original distortions caused by unintended high-frequency motions

b) A processed (sharpened) version that shows these distortions clearer. 50 nm HFW, the small white fiducial marker is 2 nm wide and 0,5 nm tall

Figure B.1 — Digitally magnified portion of the images that reveals distortions

The small horizontal displacements (small spikes) of the edges of the gold particles are longer with larger amplitude high-frequency unintended motions. These distortions are more expressly visible on Figure B.1 b), on the processed (sharpened) version of the sample area on Figure B.1 a). In this case these are mostly a couple of nanometre in size. Similar motions are present in all three dimensions, but due to the scanning type image acquisition, only the horizontal component is easily observable.

The best way of qualitatively assessing these motions depends on the SEM, image acquisition speed, and the amplitude and frequency of the unintended motions, and therefore some experimentation is necessary. 50 or 60 Hz distortion is typical, but there might be other frequency strong contributions to this problem, which may demand more complex and quantitative assessment. The alleviation of these problems is not trivial, but in many cases, one can achieve significant improvement based on the knowledge gained by this evaluation. For measurement purposes quantitative evaluation is required, because these unintended motions directly contribute to measurement uncertainty.

A quantitative assessment method starts with the sequential acquisition of a large number of images while everything else is invariant, i.e., the focus is set close to its best, the field of view is the same, so is the sample location. One then can play these images as a short movie to see what kind of changes occurred. Human perception is very sensitive to changing scenes, and so one can easily visualize the results of unintended motions. If the SEM worked perfectly, only randomly varying noise would be observable, otherwise movements, changing shapes, etc. are seen.

B.5 Measurement of the drifts and drift-compensated imaging

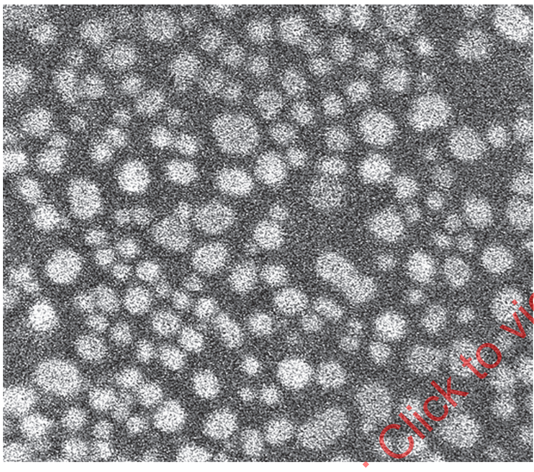
Figure B.2 show typical drift of the images and the effect of drift compensation. The images with 298 nm HFW are taken for the sample of gold on carbon. The frame acquisition time is 1 second and the time among the frames is 9 second for recording, saving and waiting. The total number N_{MD} of measurements for image drift is 50. Total time from the start to the end is $(1+9)\times50=500$ s. The quantity of the drift is estimated from the first image and the last image as shown in Figure B.2 a) and b) respectively. When comparing Figure B.2 c) with Figure B.2 a), the drift-compensated image indicates the drift-free property and the improved image quality by the accumulation. On the other hand, simply integrated image without the compensation indicates the severe drifts as shown in Figure B.2 d). One can organize these images into a series and view them as a short moving picture.

Quantitative assessments allow for tracking the unintended motions in the acquired images and for calculating their amplitudes and directions (approximated motion segments). Figure B.3 shows the trajectories of the images acquired consecutively without the drift-compensation in Figure B.2. The direction of the unintended motions changes in time, and their maximum extent has reached approximately 42 nm in *X* and 0, 8 nm in *Y* directions over a span of 500 s.

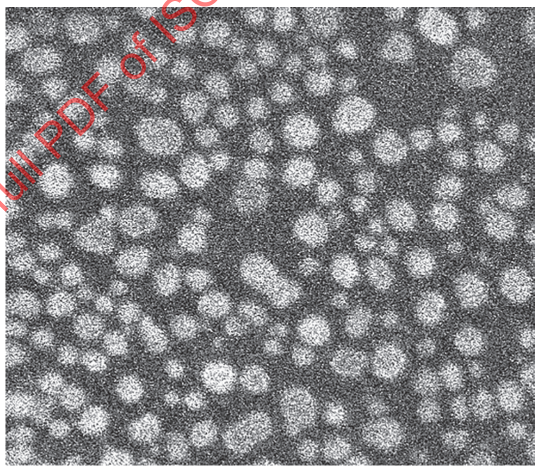
These quantitative methods can also be used to compensate for a significant part of the undesired motions. As an example, a method based on free software[6],[7] is shown.

This method uses two-dimensional Fourier transform-based compensatory technique that finds the centres of each image, lines them up by these and finally generates a better final image that is significantly closer to reality than a traditional slow-scan image, in which the drifts may cause potentially huge distortions, or simple averaging of fast images that may result in very blurry images. Figure B.4 a) shows the typical results of traditional fast-scan, and Figure B.4 b) shows two-dimensional Fourier transform-based, drift-compensated imaging for small field-of-view images.

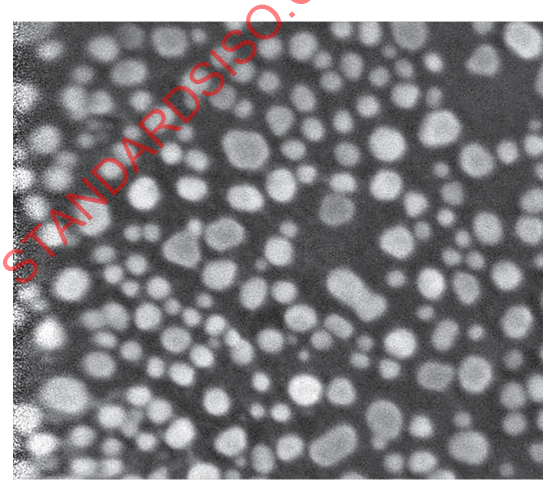
As the results of this type of fast imaging can be superior to other methods, some manufacturers SEMs have this capability built in. For other SEMs, the drift-compensation software or other similar software can help in obtaining improved imaging and measurement results.



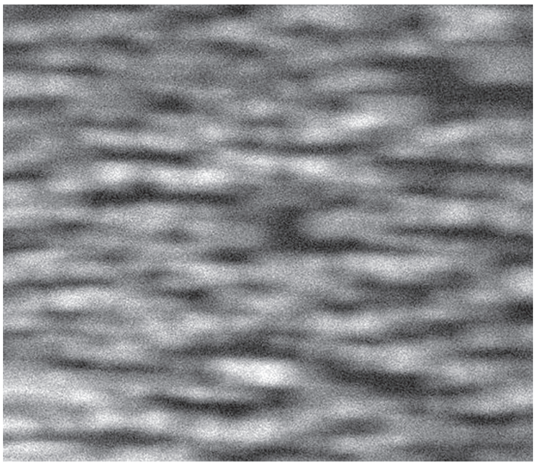
a) The first image at the start of the drift measurement



b) The last image at the end of the drift measurement

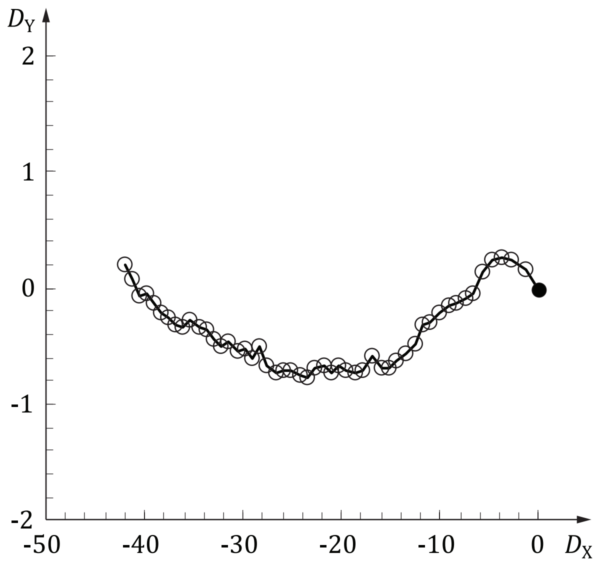


c) Drift-compensated final image by using 2D Fourier transform-based method



d) Traditional integrated final image

Figure B.2 — Drift of the 50 images for 500 s and the effect of the drift-compensation



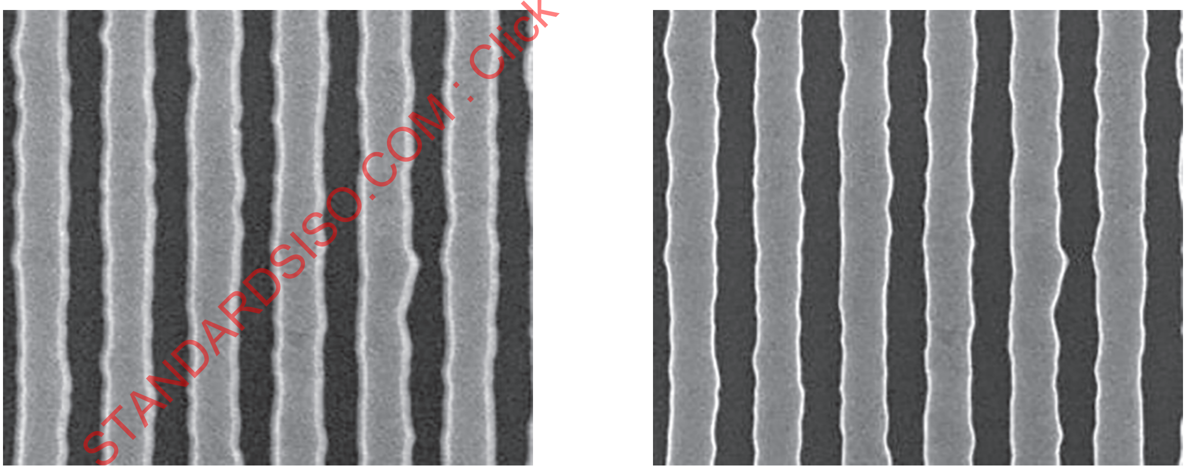
Key

D_X displacement for X direction, expressed in nm

D_Y displacement for Y direction, expressed in nm

Figure B.3 — Trajectories of the images acquired consecutively without the drift-compensation

Valuation: As the results of the measurement of the drift-compensation, the instrument meets requirements, excellent for our quantitative measurements.



a) Traditional fast-scan imaging

b) Fourier transform-based, drift-compensated imaging

Figure B.4 — The effect of drift-compensated imaging. 1 µm HFW

Valuation: As the results of the measurement of the drift-compensation, the instrument meets requirements, excellent for our quantitative measurements.

B.6 Measurement of image drifts and distortions by the cross-correlation method

B.6.1 Measurement of the drifts by using the CCF

Let (H_{Pn}, V_{Pn}) be the horizontal and vertical peak positions respectively for the auto-correlation function $F_{AC}(I_1)$, $(n=1)$ or the cross-correlation function $F_{CC}(I_1, I_n)$, $(n=2, 3, \cdots)$. Then as numerical examples, the peak positions (H_{Pn}, V_{Pn}) and drifts (D_{Hn}, D_{Vn}) are summarized in Table B.5 for the images I_n $(n=1,2,\cdots)$ which are shown in Figure 8 with size 512x512. The pixel size is estimated to be about 0,35 nm.

Table B.5 — Measurement of the drifts from the cross-correlation function

Image number	Image	Correlation function	Peak position		Drifts in pixel		Drifts in nm	
n	I_n	$F_{AC}(I_1)$ or $F_{CC}(I_1, I_n)$	H_{Pn}	V_{Pn}	D_{Hn}	D_{Vn}	D_{Hn}	D_{Vn}
			pixel	pixel	pixel	pixel	nm	nm
1	I_1	$F_{AC}(I_1)$	257	257	0	0	0,0	0,0
2	I_2	$F_{CC}(I_1, I_2)$	244	232	-13	-25	-4,7	-9,1
3	I_3	$F_{CC}(I_1, I_3)$	180	225	-77	-32	-28,1	-11,7
4	I_4	$F_{CC}(I_1, I_4)$	213	258	-44	1	-16,0	0,4

Key

n the number of acquired image from the start of the measurement, $n=1, 2,\cdots$

I_n the n-th acquired image in the measurements, $n=1,2,\cdots$

$F_{AC}(I_1)$ auto-correlation function of the initial image I_1

$F_{CC}(I_1, I_n)$ cross-correlation function for the initial image I_1 and n-th image I_n $(n=2, 3, \cdots)$

H_{Pn}, V_{Pn} horizontal and Vertical peak positions respectively for the $F_{AC}(I_1)$, $(n=1)$ or the $F_{CC}(I_1, I_n)$, $(n=2, 3, \cdots)$

D_{Hn}, D_{Vn} drift quantities of the n-th image I_n $(n=1, 2, \cdots)$ for the horizontal (H) and vertical (V) directions respectively, typically expressed in pixel or in nm

Valuation: As the results of the measurement of the drift-related distortions, the instrument meets requirements, fair for our quantitative measurements.

B.6.2 Measurement of the distortions by using the CCF

Let $F_{BAC}(I_1)$ and $F_{BCC}(I_1, I_n)$ be the binarized pictures of the auto-correlation function $F_{AC}(I_1)$ and the cross-correlation function $F_{CC}(I_1, I_n)$ respectively as shown in Figure 10. Then numerical examples of the areas $A_{CCF,n}$ and $A_{ACF,1}$ and the ratio $k_{A,n}$ are summarized in Table B.6.

Table B.6 — Measurement of the drift-related distortions

Image number	Image	Binarized picture	Area (Number of pixel)	Ratio of the areas
n	I_n	$F_{BAC}(I_1)$ or $F_{BCC}(I_1, I_n)$	$A_{ACF,1}$ or $A_{CCF,n}$	$k_{A,n}$
			pixel	
1	I_1	$F_{BAC}(I_1)$	2549	1,00
2	I_2	$F_{BCC}(I_1, I_2)$	4440	1,74
3	I_3	$F_{BCC}(I_1, I_3)$	3489	1,37
4	I_4	$F_{BCC}(I_1, I_4)$	6372	2,50

Key

n the number of acquired image from the start of the measurement, n=1, 2,···

I_n the n-th acquired image in the measurements, n=1,2,···

$F_{BAC}(I_1)$ binarized picture of the auto-correlation function $F_{AC}(I_1)$ of the initial image I_1 by using a thresholding level T_B

$F_{BCC}(I_1, I_n)$ binarized picture of the cross-correlation function $F_{CC}(I_1, I_n)$ for the initial image I_1 and n-th image I_n (n=2, 3, ···) by using a thresholding level T_B

$A_{ACF,1}$, $A_{CCF,n}$ areas of the binarized pictures $F_{BAC}(I_1)$ and $F_{BCC}(I_1, I_n)$ respectively

$k_{A,n}$ ratio of the area $A_{CCF,n}$ to the area $A_{ACF,1}$ ($k_{A,n} = A_{CCF,n} / A_{ACF,1}$)

Valuation: As the results of the measurement of the drift-related distortions, the instrument meets requirements, fair for our quantitative measurements.

