Hitachi High-Tech

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- The product description given herein applies as of April, 2008. Note that there is a possibility of sales of the product being discontinued without prior notice, or of partial change in the appearance or specifications of the product for improvement.
- Individual catalogs are prepared for each of the instruments described, so please inquire with our sales representative if you would like to obtain them.

Table of Contents

Chapter 1. What is the SEM?

1.1 What can we do with a SEM? 1

1.2 Principle and Structure of the SEM 4

1.3 Procedure for SEM Observation 7

Chapter 2. Sampling

2.1 Tools Used 9

2.2 Sampling 9

2.3 Metal Coating 11

2.4 Ion Milling 12

Chapter 3. Let's Observe the Following with a SEM!

3.1 Machined Products, Materials 13

3.2 Semiconductor Wafer, Liquid Crystal Display 17

3.3 Biological Samples 19

3.4 Foodstuffs 21

Chapter 4. What causes these images? 22

Chapter 5. SEM Instruments Available 27

Chapter 6. Q&A for persons wanting to know more about the SEM! 29

6.1 Concerning formation of the electron beam 31

6.2 Concerning Evacuation 37

6.3 Generation, detection and usage of SEM signals 39

6.4 Viewing conditions for acquiring good SEM images 49

6.5 Principle and applications of low vacuum SEM 60

6.6 Generation/detection of x-rays and elemental analysis method ... 63

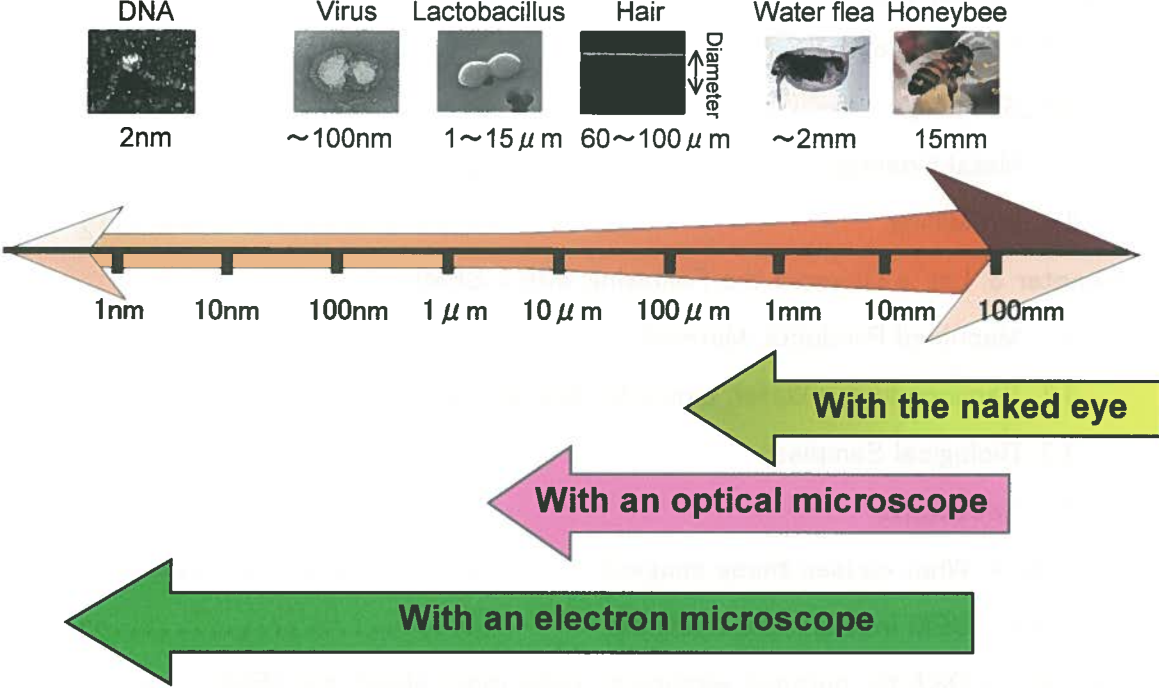
6.7 How to improve accuracy in x-ray analysis 70

6.8 Principle and application of STEM 75

Chapter 1. What is the SEM?

1.1 What can we do with a SEM?

The resolving power of the human eye is around 0.1 mm. To observe objects smaller than this, an optical microscope or electron microscope must be used.



Electron microscopes are available in a scanning electron version (SEM)[*1] and a transmission electron version (TEM)[*2]. The SEM is used particularly for observing the fine structure of a specimen surface at high magnification, while the TEM is used mainly for observing the inner structure of a specimen at high magnification. We will introduce here the features of the SEM.

*1 SEM: Scanning Electron Microscope
*2 TEM: Transmission Electron Microscope

Features of the SEM

1. All solid surfaces can be observed in a range from low to high magnifications.
2. Focal depth is greater than that of an optical microscope, allowing us to acquire a stereoscopic image.
3. Combination with an x-ray analyzer permits compositional analysis of a microscopic area.

Now let's compare the images obtained with an optical microscope and the SEM.

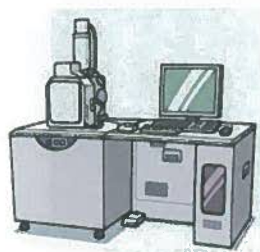
Sample used is a fiber employed in parasols for blocking ultraviolet rays.

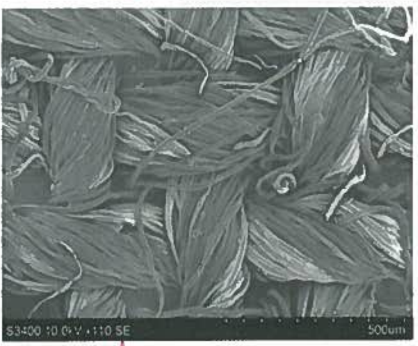
Observation with optical microscope

Fiber observed with an optical microscope. Although the OM provides color information, it has a shallow depth of focus, and when focusing on the circled part in the image at the left, the portion having a slightly different height comes out as an unclear image.

×110 magnification

Observation with SEM

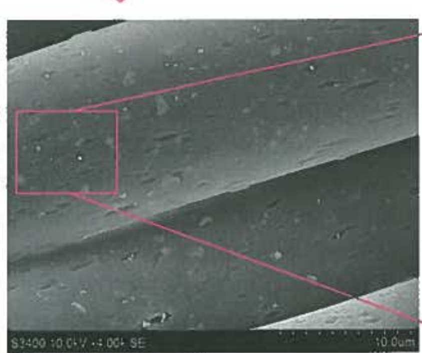




The sample was observed with an SEM at the same magnification as used with the OM. Although the SEM image is black & white (lacks color information), it has a greater depth of focus and provides stereoscopic information.

×110 magnification

When further enlarged!

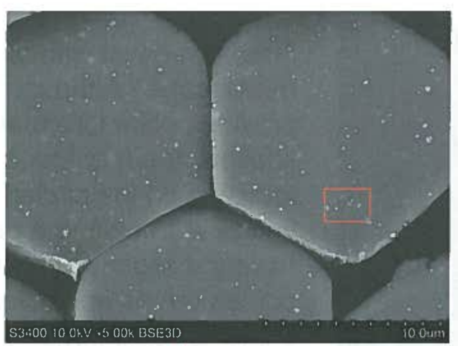


×4,000 mag.

×15,000 mag.

When observing an SEM image with the magnification increased to ×4,000, inorganic matter (white particles) that are used to block UV rays can be seen dispersed in the fiber. Note that SEM images include secondary electron and backscattered electron information (explained later), and the above are secondary electron images. By further increasing magnification to ×15,000, it can be seen that the inorganic matter becomes particles of 100 to 500 nm in size. A cross-sectional observation can be made next to see how the inorganic particles are dispersed in the fiber.

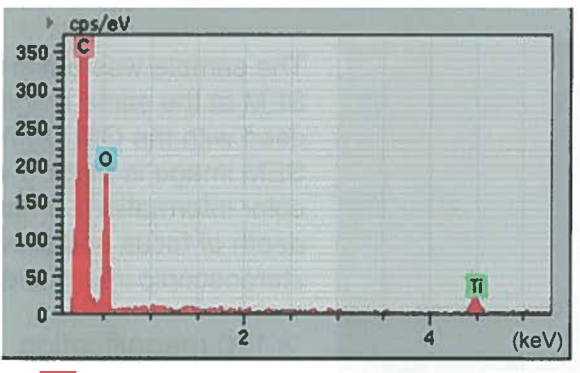
Observation and Compositional Analysis of Fiber Cross-section



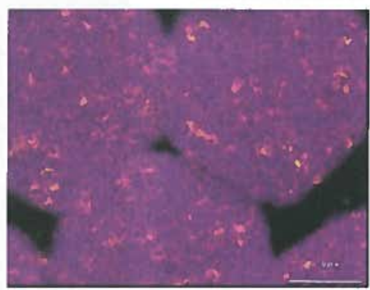
Backscattered electron image (× 5,000 magnification)

The cross-sectional structure of the fiber was observed with a backscattered electron detector. This permits conformation of how the white particles are dispersed in the fiber.

Let's put this to (compositional) analysis.



X-ray spectrum of area enclosed in rectangle above



C Ti X-ray mapping image of C and Ti

First of all, let's cut the fiber observed before and observe its cross-sectional structure via a backscattered electron detector. Since a BSE image permits detecting a difference in average atomic number as a difference in contrast, a location having different compositional elements can be clearly seen. The white glistening particles are inorganic matter, and one can see how they are dispersed in the fiber. Also, when an electron beam is applied to the sample, characteristic x-rays will be produced as well. By attaching to the electron microscope an x-ray analyzer that captures these x-rays, it can be seen what elements exist and where they are located.

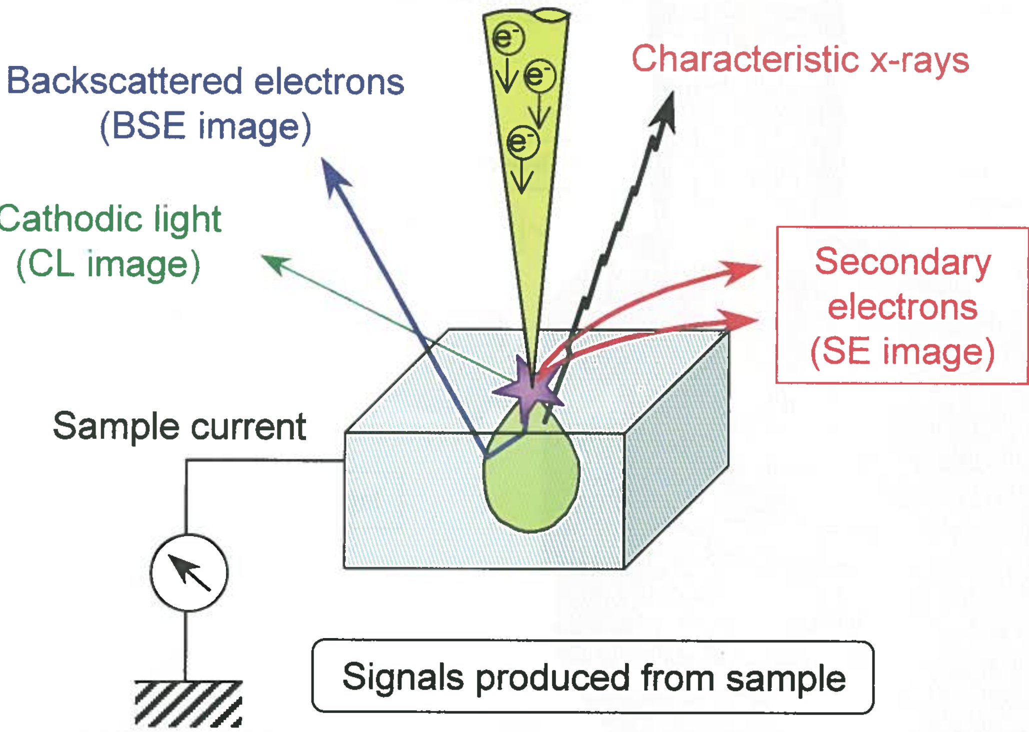
By conducting x-ray analysis on the part enclosed in the rectangle on the BSE image, it is seen from the spectrum that titanium (Ti) exists in the sample. Now, by conducting x-ray mapping on carbon (C) and Ti in the same visual field as on the BSE image, it will be clear that the fiber contains organic matter (C mainly) and that particles of Ti (actually TiO_2) which serve to block UV rays are dispersed in it.

1.2 Principle and Structure of the SEM

- Let's learn the principle and structure of the SEM.-

What is the SEM?

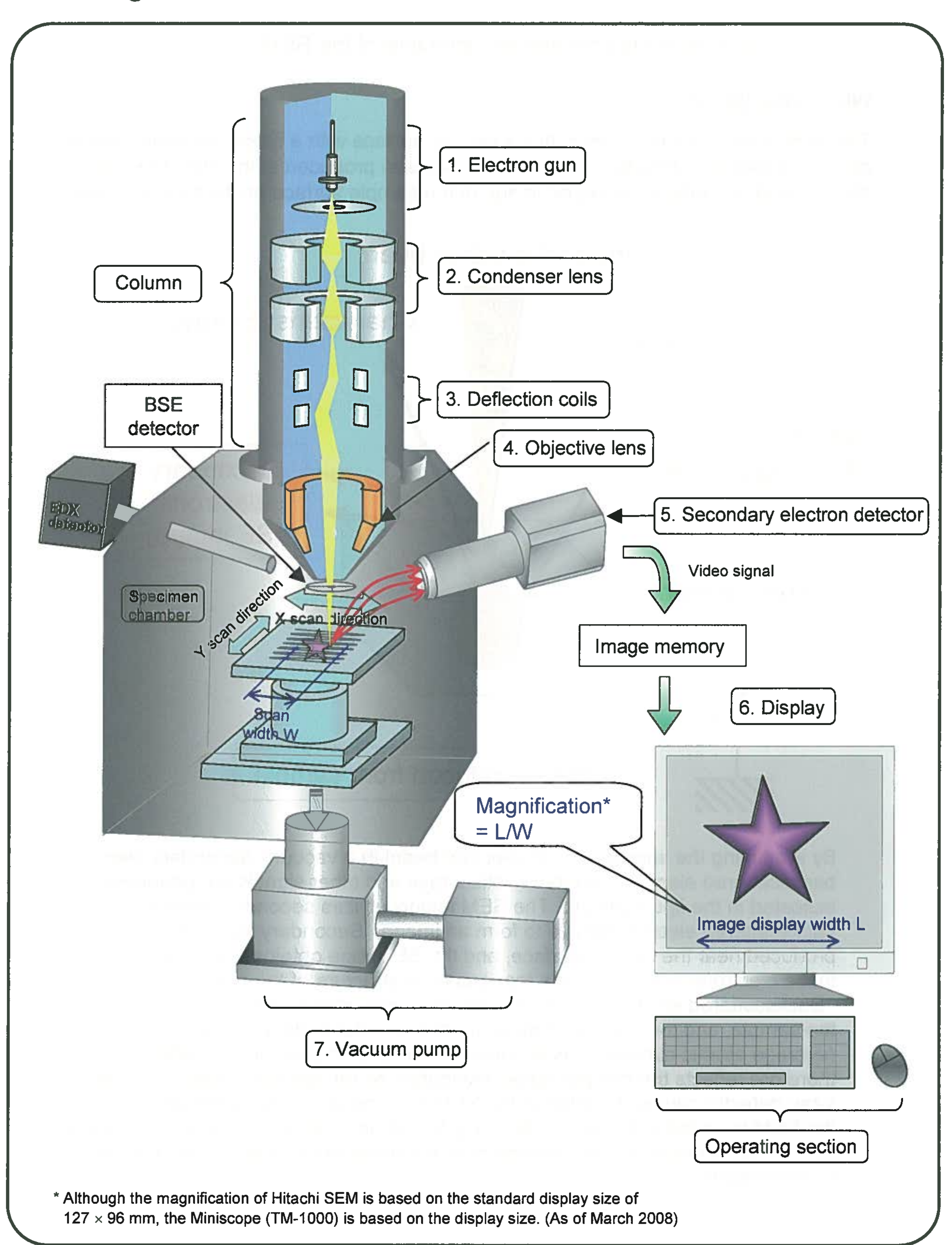
The SEM is an instrument that scans a sample surface with a finely converged electron beam in a vacuum, detects the information (signals) produced at that time from the sample, and presents an enlarged image of the sample surface on the monitor screen.



By irradiating the sample with an electron beam in a vacuum, secondary electrons, backscattered electrons, characteristic x-rays and other signals are generated as indicated in the figure above. The SEM mainly utilizes secondary electron or backscattered electron signals to form an image. Secondary electrons are produced near the sample surface, and the SE image obtained upon detecting these electrons reflects the fine topographical structure of the sample.

Backscattered electrons are those reflected upon striking the atoms composing the sample, and the number of these electrons is dependent on the composition (average atomic number, crystal orientation, etc.) of the sample. A BSE image therefore reflects the compositional distribution on the sample surface. Besides, an x-ray detector can be mounted to the SEM for conducting elemental analysis. So the SEM is usable not only for observing the sample structure, it is also applicable as an x-ray analyzer for determining what elements are included in the sample and to what degree.

Configuration of SEM



* Although the magnification of Hitachi SEM is based on the standard display size of 127 × 96 mm, the Miniscope (TM-1000) is based on the display size. (As of March 2008)

As shown in the figure at the left, the SEM consists mainly of column, specimen chamber, display and operating section. The interior of the column is kept in a high vacuum, and the electron beam produced by the electron gun (prior to striking the sample) is converged into a fine beam via the electromagnetic lenses (condenser and objective lenses). And by applying a scan signal to the deflection coils, the electron beam is scanned along the sample surface. The specimen chamber is equipped with a specimen stage having a specimen goniometer, a secondary electron detector for detecting signals produced from the sample, and depending on the instrument, a backscattered electron detector and/or an x-ray detector. Connected below the specimen chamber is a vacuum pump for keeping the interior of the column and specimen chamber in a high vacuum.

1. Electron gun

A mechanism that emits electrons from a metal and accelerates them in a strong electric field. There are three types of electron gun according to the method of emission --- field emission type (FE) electron gun, Schottky electron gun and thermal electron gun. For details refer to Chapter 6.

2. Condenser lens

An electromagnetic lens (coil) used to converge the electron beam emitted from the electron gun into a fine beam.

3. Deflection coil

A mechanism used to scan the electron beam in X and Y directions and change the area (magnification) to be scanned. The SEM magnification, as indicated in the figure, is determined by the ratio of width of image display area (L) to width of electron beam scan on the sample (W).

4. Objective lens

Used to converge the electron beam into a fine beam and focus it onto the sample surface. There are three types of lens, in-lens, semi in-lens and out-lens, according to the object of observation or resolution. Refer to Chapter 6 for details.

5. Secondary electron detector

Efficiently captures, converts to electric signal and amplifies the secondary electrons produced from the sample. Refer to Chapter 6 for the method of detection.

6. Display

Converts the detected and amplified secondary electron signal to brightness and provides an enlarged image.

7. Vacuum pump

Evacuates the column and specimen chamber to a high vacuum level (10^{-4} to 10^{-6} Pa). Refer to Chapter 6 for details on evacuation.

1.3 Procedure for SEM Observation

We introduce here the procedure for SEM observation using the S-3400 SEM as an example.

(1) Startup of instrument

(1) Turn ON power to the instrument.

(2) Log in to PC and then start up the SEM software.

(2) Preparation of sample to be observed

(3) Attach conductive tape to specimen stub and then mount the sample on this.

(Refer to Chapter 2 for detailed specimen preparation method.)

(4) Set specimen stub on exclusive holder and adjust height with "height gauge".
(Refer to instruction manual of applicable instrument.)

(3) Setting of sample to microscope

(5) Press AIR on front panel of column and introduce air into specimen chamber.
(6) Pull out specimen stage gently and set specimen holder on center of stage.
(7) Press EVAC on front panel of column and evacuate specimen chamber.
(8) When evacuation of specimen chamber is finished, start observation of sample.

(4) Observation of sample

Actual SEM operation is confined to this. Very simple, don't you agree ?

(9) Click the accelerating voltage 'ON' icon.

(10) Automatically set brightness and focus at low magnification.

ABCC (auto brightness adjustment) →AFC (auto focus)

(11) Search for a field of interest and set a desired magnification.

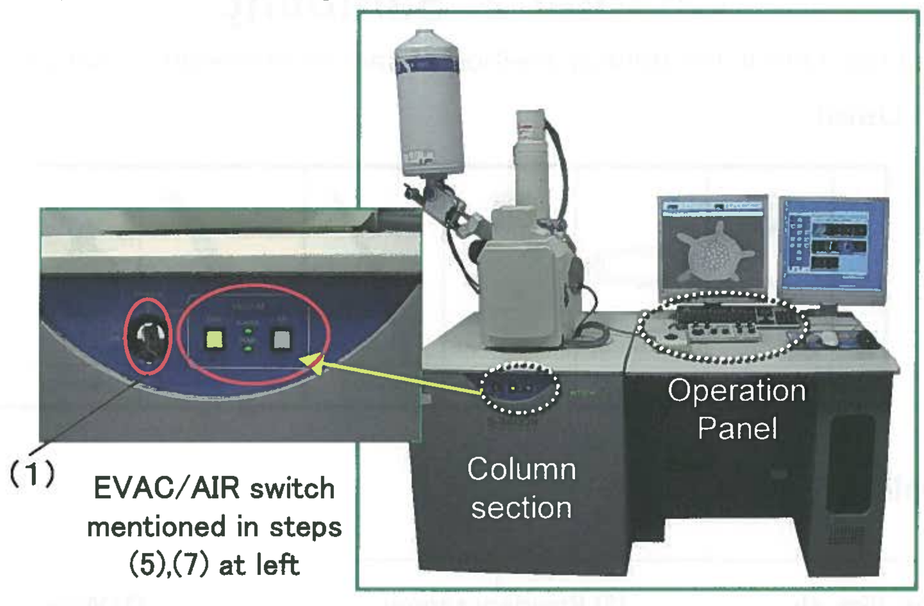
(5) Acquisition and saving of image

(12) Click icon of the software level and acquire a digital image.

(13) Select a capture box image and click the "Save" icon.

(14) Select a folder, input a file name and click the "Save" icon again.

<Example of operating screen>



(10)

(12)

(9)

ON

5.00kV

x30

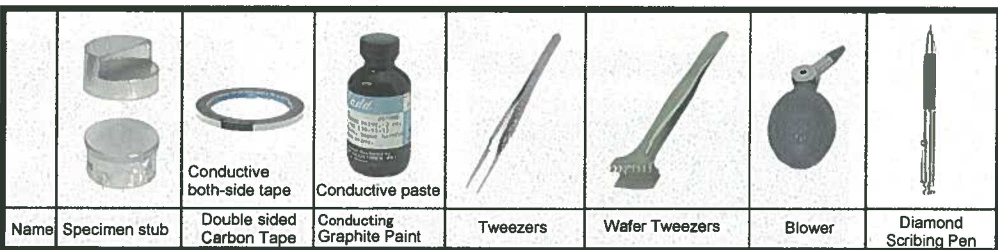
ABCC

AFC

Chapter 2. Sampling

Introduced here is the general method of sample preparation (sampling).

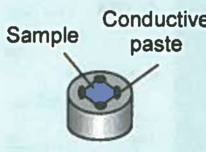
2.1 Tools Used



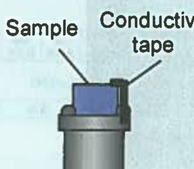
		Conductive both-side tape	Conductive paste				
Name	Specimen stub	Double sided Carbon Tape	Conducting Graphite Paint	Tweezers	Wafer Tweezers	Blower	Diamond Scribing Pen

2.2 Sampling

(1) Bulk sample, film, etc.



Sample is fixed in place by attaching it to conductive double sided tape or conductive paste, then mounted on specimen stub.

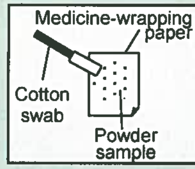


With a large sample, after fixing it on specimen stub, conductive tape is applied from top surface of sample to specimen stub to ensure conductivity between sample and stub.

(2) Powdery sample

Sprinkling method

Apply water-soluble carbon paste thinly to specimen stub.

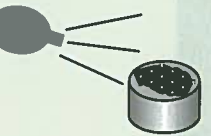


Before the paste dries, sprinkle the powdery sample attached to cotton swab onto specimen stub.

Too fast

OK

Too slow



Finally blow off excess powder using a blower.

Suspension method

(used especially when particles must be dispersed well)

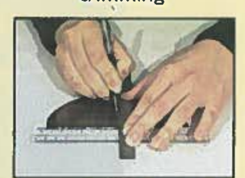
Take a small amount of powder particles into a test tube, add 5 to 10 mL of a solution that doesn't react chemically with the powder, then either shake the solution or disperse it using an ultrasonic cleaner.

Apply drops of the suspension onto aluminum foil, and spread it out using a blower or the like.

When it has dried, cut off part of the aluminum foil containing the powder and fix it to specimen stub using conductive paste.

(3) Wafer or glass sample

Observation of surface, trimming



1) Place a rather thick ruler before the wafer.
2) Place another ruler onto the section to be marked off, then mark off the sample while holding it firmly in place.
3) Remove waste or unwanted matter with a blower.

4) Place sample onto the ruler, align the edges of the ruler and marked-off part exactly then place medicine-wrapping paper onto it.
5) While holding the top of ruler (sample) from above the medicine-wrapping paper, slightly raise the sample.
6) Hold near the edge and divide the sample.
7) Remove waste or unwanted matter with a blower.

Repeat steps 1) to 7) above until you obtain the size you want for trimming. A standard trimming size is 30 mm square in the case of a wafer and 80 mm square in the case of glass.

Specimen stub	A nonmagnetic material such as aluminum is generally used for the specimen stub on which a sample is placed. Specimen stubs come in various sizes and shapes, and are selected according to the purpose of observation (cross-sectional observation, etc.). With Hitachi's SEM, the rear of the stub has M4 threading.
Double sided Conductive Tape	Both carbon and copper are available for Double Sided conductive tape, though carbon tape is mainly used. This tape is used for fixing sample to specimen stub or for grounding the sample and stub.
Conductive paste	Conductive paste is used for firmly fixing a sample in place and is suitable for high-magnification observation at ×50,000 or higher. A water-soluble paste should be used for a sample susceptible to organic solvent. It is convenient to use a toothpick or the like for applying paste to specimen stub.
Tweezer	Used when sampling so as not to touch the sample surface.
Blower	Used for removing excess sample or waste from sample surface or cross-section after sampling of a powdery substance.
Diamond Scribing pen	Used for marking off a sample surface in preparing a cross section of wafer or glass sample.

Cross-section preparation

This is a method of preparing a cross section of a trimmed sample.

1) While holding the sample, mark four corners of the cross section to be observed for identification purposes (unmarked surface is the cross section for observation).

2) Prepare a ruler that is slightly thicker than the sample.
3) Place the ruler onto the section to be marked off, then mark off about 5 mm near the edge of sample.
4) Remove waste or unwanted matter with a blower.

5) Place sample onto the ruler, and align the edges of the ruler and marked-off part exactly.
6) While holding the sample from above the ruler, slightly raise the sample.
7) Hold near the edge and divide the sample.

Cross section preparation is successful when surface to be observed is like a mirror.

Carry out sampling with carbon paste applied to cross-section specimen stub.

(4) Biological sample

Such samples are usually observed in dried condition since the interior of the electron microscope is evacuated.

Perfusion fixation
(Substitution of fixative solution with blood and formalin, glutaraldehyde, etc.)
↓
Fine sectioning of sample
↓
Immersion fixation
(Formalin, glutaraldehyde, etc.)
↓
Post-fixation
(Osmium tetroxide)
↓
Conductive staining
(Tannic acid – osmium tetroxide, etc.)
↓
Dehydration
(Ethanol ascending system, etc.)
↓
Drying
(Critical point drying, freeze-drying etc.)
↓
Observation with SEM

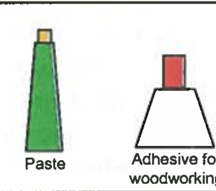
The above is the general procedure for taking a biological sample in nearly the natural state and subjecting it to drying.

Reference document:
Scanning electron microscope technology for medical and biological applications (medical publications center)

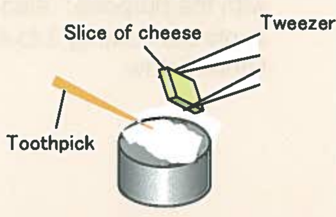
* For observation with a low-vacuum SEM, the pretreatment can be simplified for the observation.

(5) Foodstuffs, samples containing oil

* These are observed with a low-vacuum SEM. In some cases, a cool stage may be used for cooling the sample for observation.



Prepare paste or adhesive.



Example of sampling

Leaf

Slice of cheese

Apply a little paste or adhesive to the specimen stub and fix the sample (cut to a small size) onto this.

2.3 Metal Coating

(1) Purposes of coating

- To make the sample surface conductive (prevention of charge-up)
- To increase the production rate of secondary electrons (increase image information)
- To prevent damage to sample

(2) Film thickness of coating

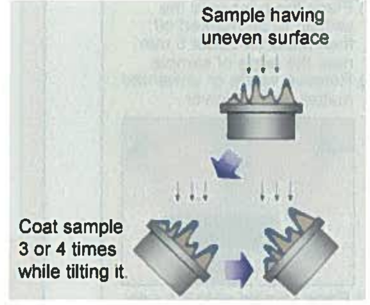
Although a thickness of several nanometers is standard for the film coating, it varies with the magnification used for the observation. A somewhat thinner coating is suitable for high-magnification observation. And for observation at low magnification, a slightly thicker coating will provide a clear image having a good S/N ratio.

(3) Kinds of metal targets

<table>
<tr><td>Au</td><td>Gold</td><td rowspan="2">For general-use SEM</td><td rowspan="4">Coating particles
↓
Fine</td><td rowspan="4">Evaporation film suitable for SEM observation:
1. Film of uniform quality having adequately fine particles
2. Secondary electron discharge rate is good.
3. Stable against oxidation</td></tr>
<tr><td>Au-Pd</td><td>Gold-palladium</td></tr>
<tr><td>Pt—Pd</td><td>Platinum-palladium</td><td rowspan="2">For FE-SEM</td></tr>
<tr><td>Pt</td><td>Platinum</td></tr>
</table>

The particles of the coating film become finer in the order indicated in the above table. On a sample coated with gold, the particles can be observed at a magnification of ×50,000 or ×60,000 and higher. For this reason a gold-palladium coating is often used with a general-use SEM. In the case of a SEM having a high resolution such as the FE-SEM, a fine coating film with which the particles cannot be seen is required. Therefore, since the particle status of the coating film varies with the coating material, the target must be selected in accordance with the purpose. Also, for coating a sample having an uneven surface, it is recommended to apply the coating 3 to 4 times while tilting the sample in different directions as shown in the figure below.

Magnetron sputtering device



Coating of sample having uneven surface

(4) C (carbon) coating

A vacuum evaporator is usually employed for carbon coating. Since the particles of the evaporation film are fine and of low density, this type of coating is suitable for elemental analysis or observation of BSE images.

2.4 Ion Milling

(1) What is ion milling?

Ion beam milling is a technique that utilizes a broad ion beam (BIB) of approximately 1 mm in diameter discharged from the gun to sputter atoms from the sample surface; it is used for eliminating machining flaws from the sample surface or in preparing cross-sectional samples of multiple film layers.

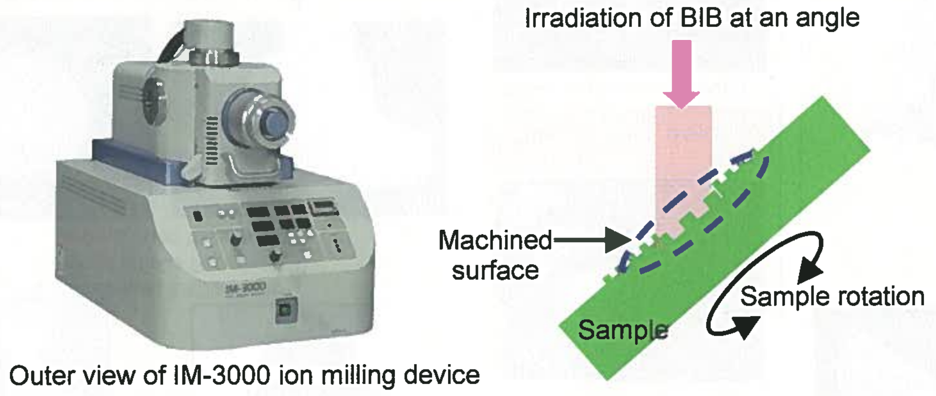
(2) What is BIB ion milling?

2-1 Surface processing (flat milling method)

The sample surface is uniformly etched by irradiating the ion beam at an angle while rotating the sample.

Main applications:

- Elimination of oxide film or contamination from a wide area of about 5 mm diameter on sample surface
- Elimination of machining flaws from machined sample surface
- Observation of crystal grains or layers formed by relief milling



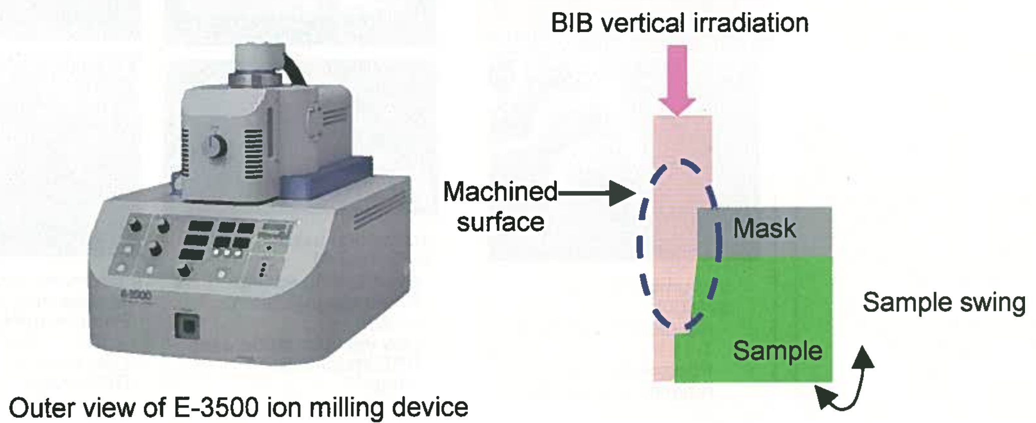
Outer view of IM-3000 ion milling device

2-2 Cross-section processing

An even cross section covering a wide area (about 1 × 1 mm) of a sample can be prepared by placing a shielding plate on the sample and blocking part of the ion beam irradiated onto the sample, then etching around the edge of the shielding plate.

Main applications:

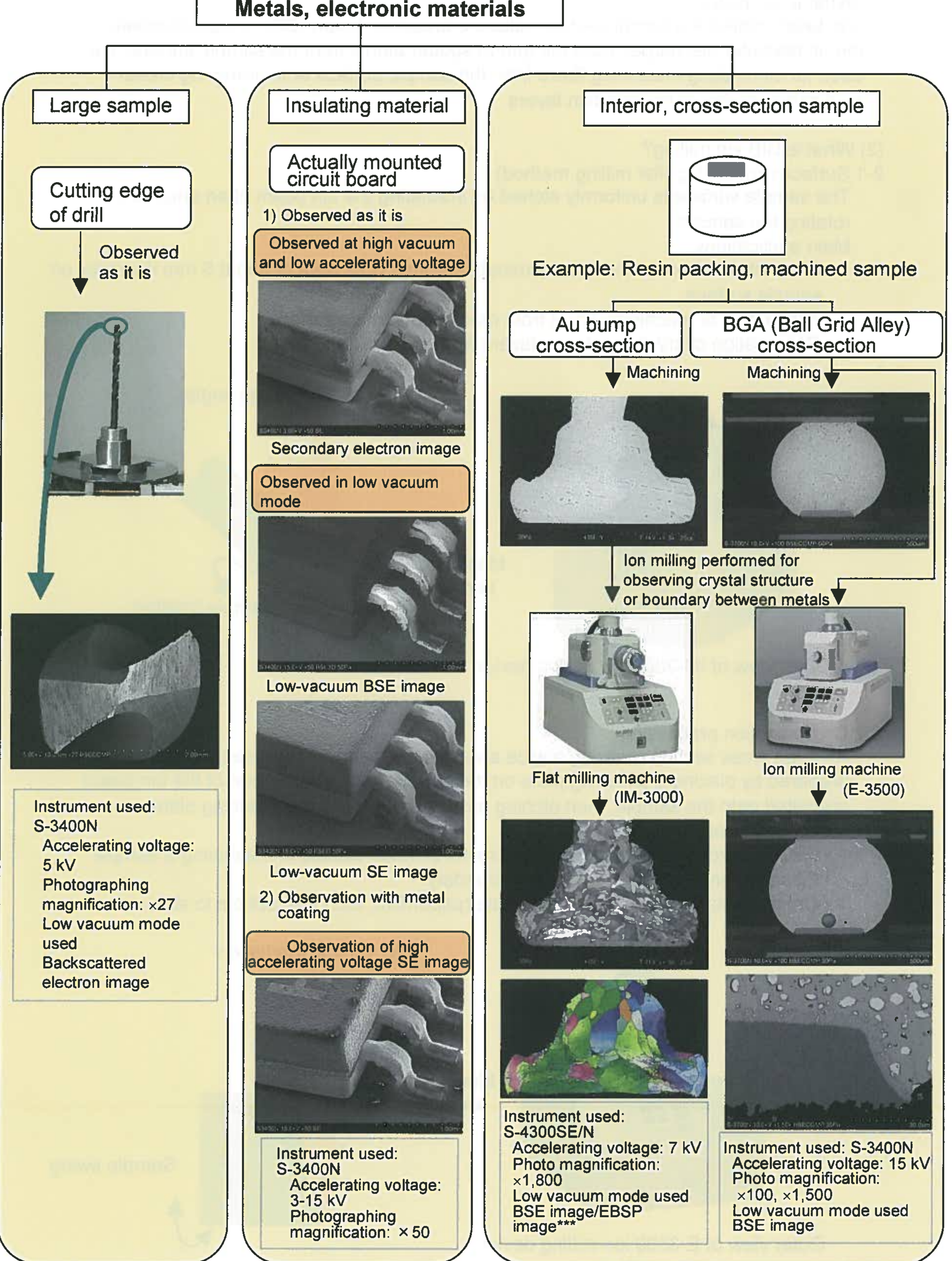
- Evaluation/observation of complex cracks or voids caused in machining a sample
- Observation/analysis of laminated boundary
- Cross-sectional observation of sample (paper/film, etc.) susceptible to stress

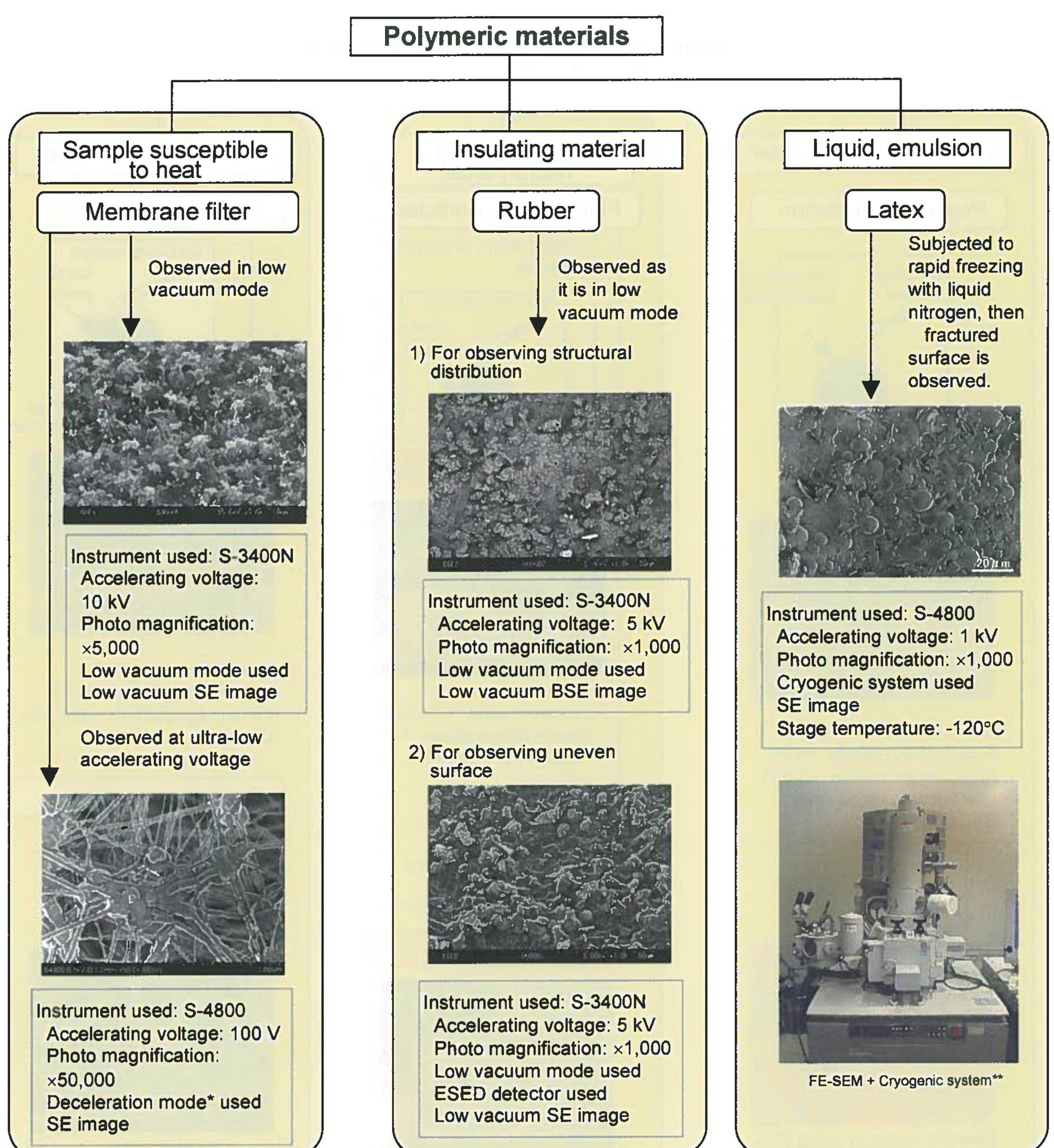


Outer view of E-3500 ion milling device

Chapter 3. Let's Observe the Following with a SEM!

3-1 Machined Products, Materials (1)



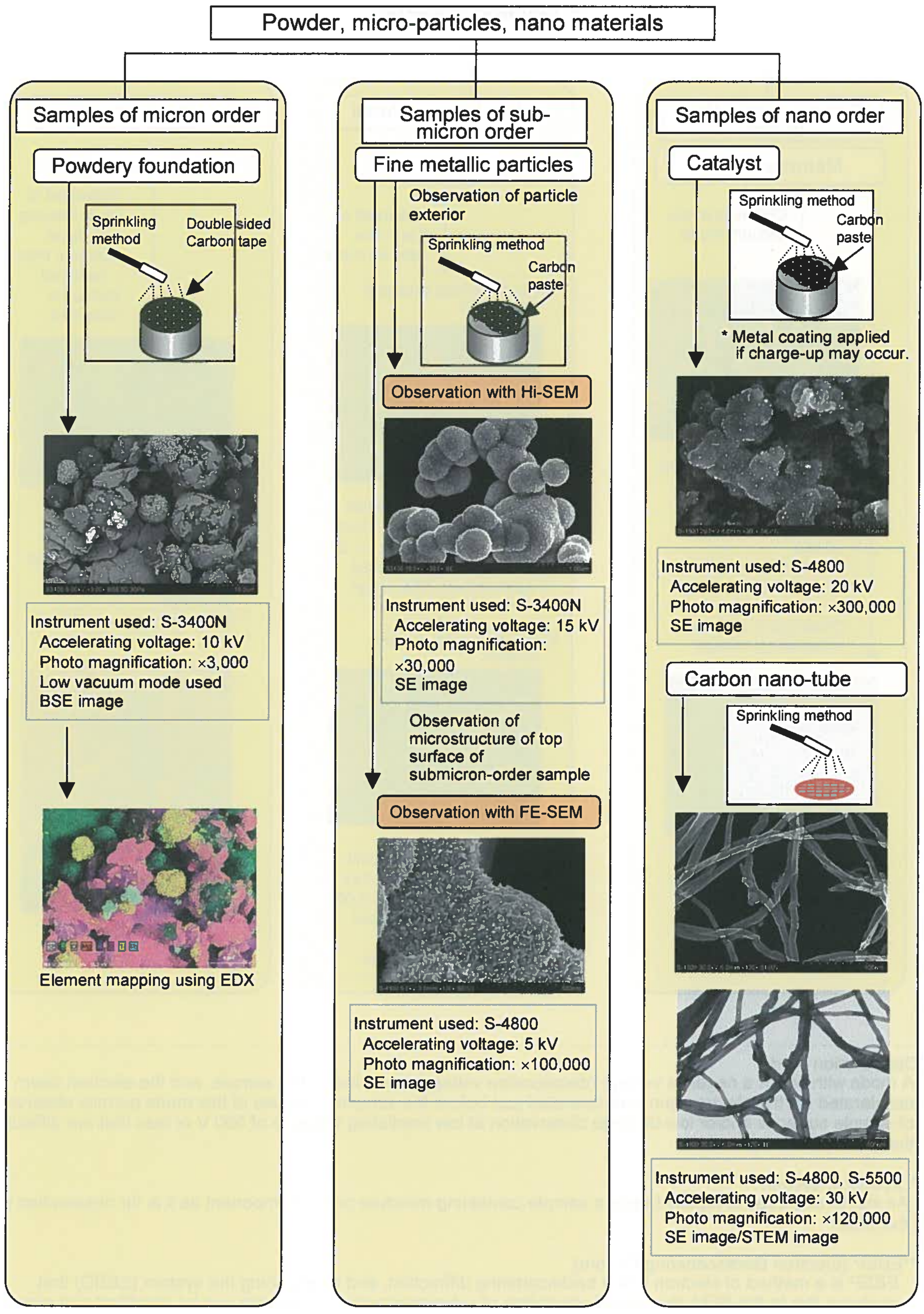


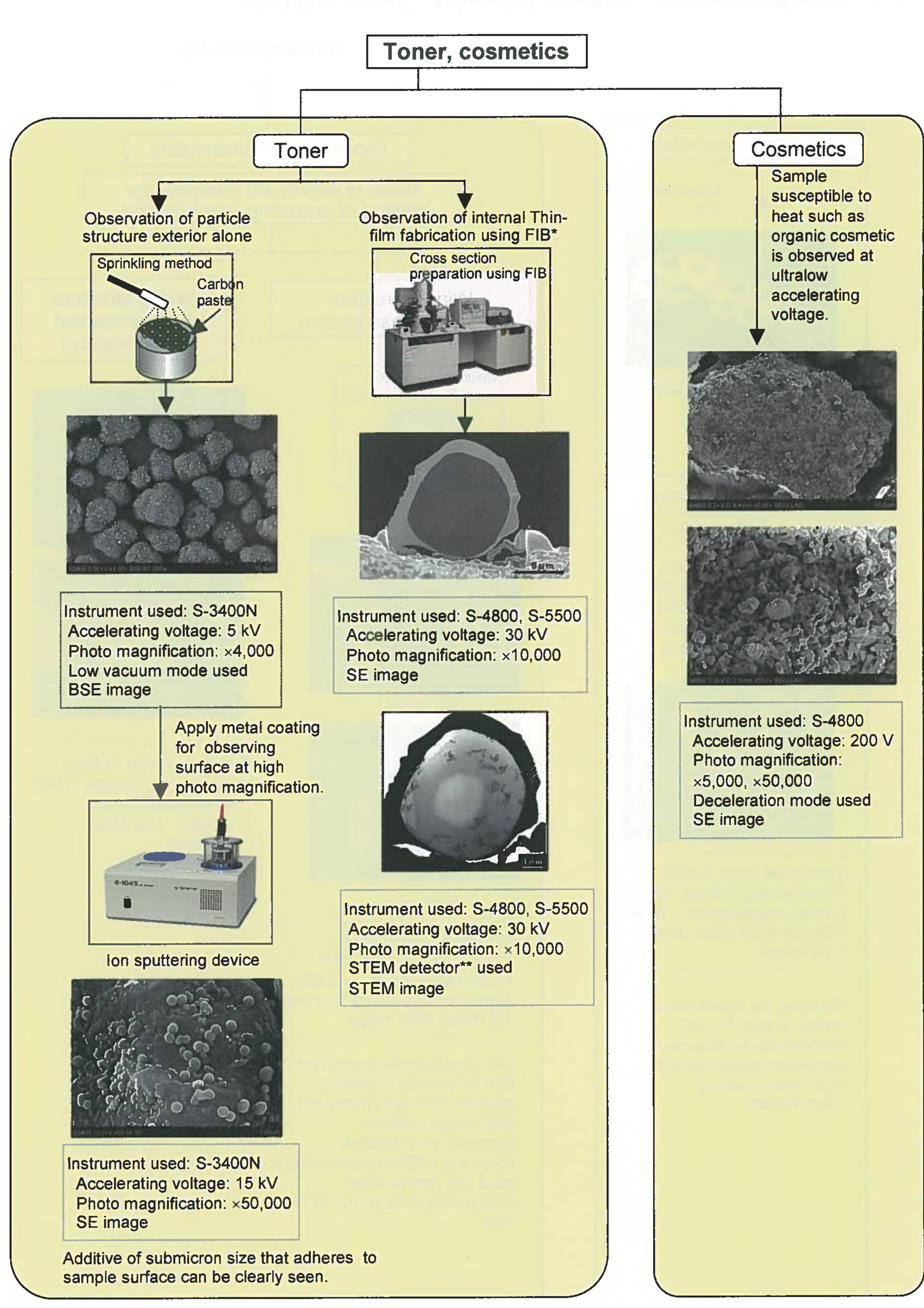
*Deceleration mode
A mode with which a negative voltage (deceleration voltage) is applied to the sample, and the electron beam accelerated via the electron gun is decelerated just before the sample. The use of this mode permits observation of sample surfaces and/or low-damage observation at low irradiating voltages of 500 V or less that are difficult with the usual SEM.

**Cryogenic system
An instrument used to rapidly freeze a sample containing moisture or oily component as it is for observation with the SEM.

***EBSP (Electron Backscattering Pattern)
EBSP is a method of electron beam backscattering diffraction, and by attaching the system (EBSD) that analyzes this to the SEM, the crystal orientation of submicron order of a sample can be identified and mapping images of the crystal orientation can be acquired.

3-1 Machined Products, Materials (2)

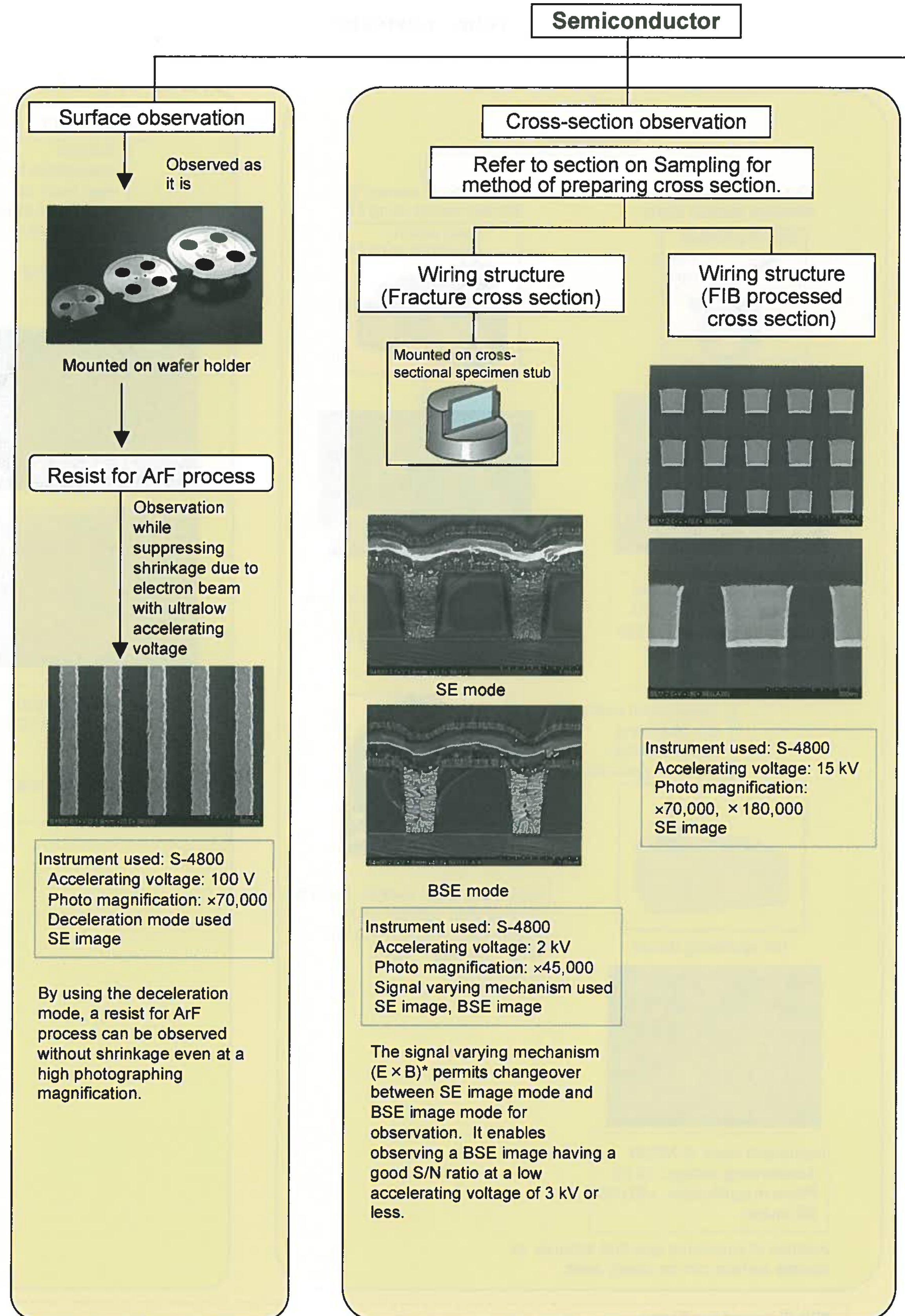




*FIB (Focused Ion Beam)
FIB is an abbreviation of Focused Ion Beam Fabrication/Observation System. It permits not only observation with a scanning ion microscope (SIM) image, but also microstructural fabrication of a sample by means of a finely converged ion beam.

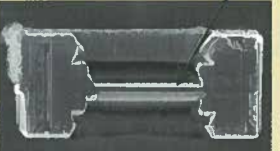
**Refer to 6.8 in Chapter 6 for the STEM detector.

3-2 Semiconductor Wafer, Liquid Crystal Display



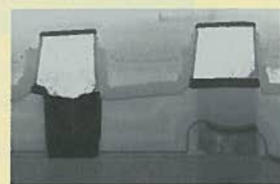
* E × B is an energy filter system

FIB processing system is used for preparing cross section of microscopic area or for thin-film processing free of stress.



Thin film processing via FIB

LSI



Instrument used: S-5500
Accelerating voltage: 30 kV
Photo magnification: ×50,000, ×500,000
STEM detector used
BF-STEM image

Observation of transparent electrode surface

Mounted on cross-sectional pecimen stub with carbon paste

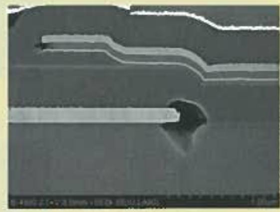
Instrument used: S-4800
Accelerating voltage: 5 kV
Photo magnification: ×400,000
SE image

Observation of TFT (thin- film transistor) cross section

Although Fracture cross section can be observed, use of FIB processed cross section is recommended for a clearer view of layer structure.

FIB processing system is used for preparing cross section of microscopic area or for thin- film processing free of stress.

SE mode

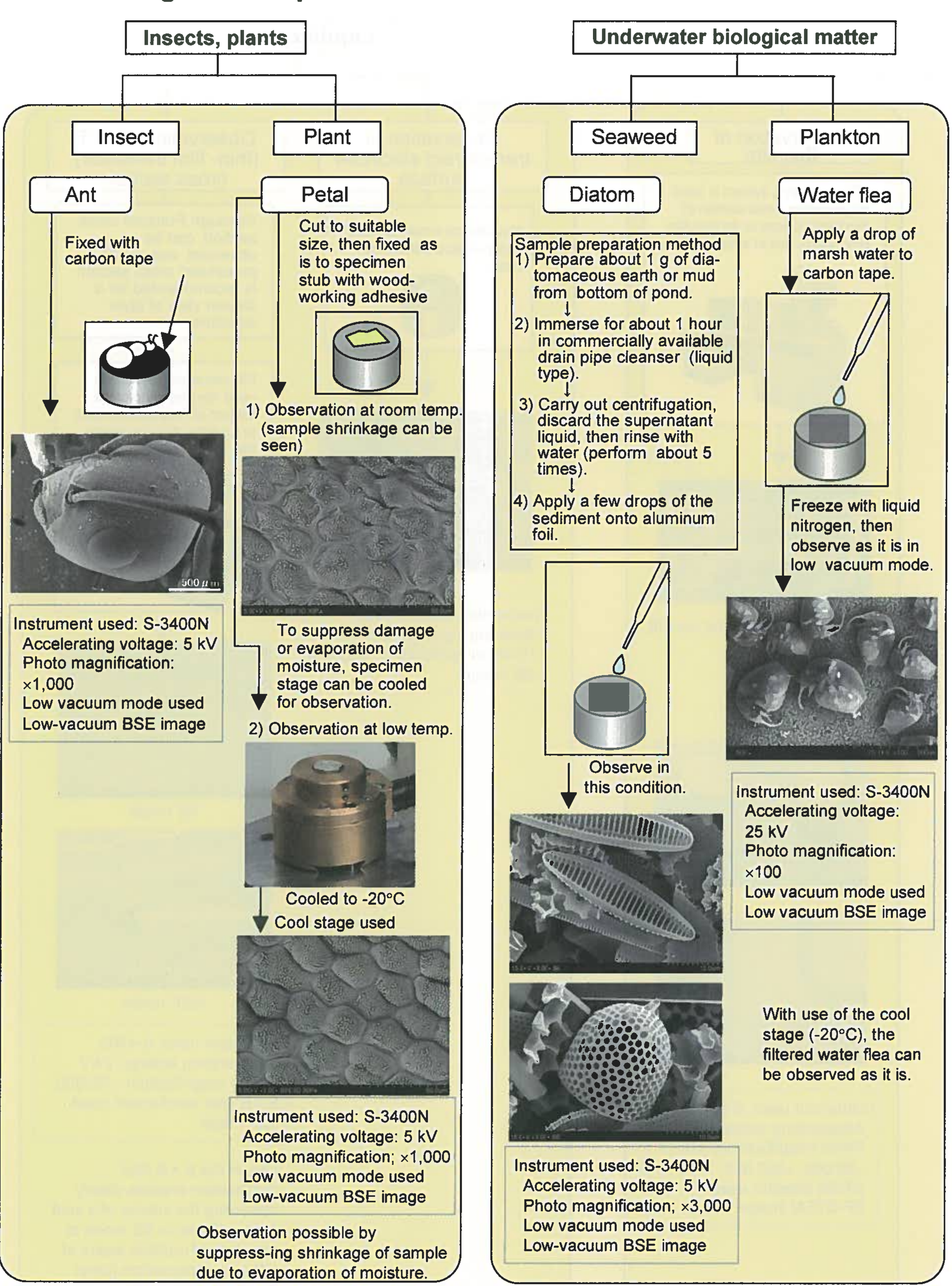


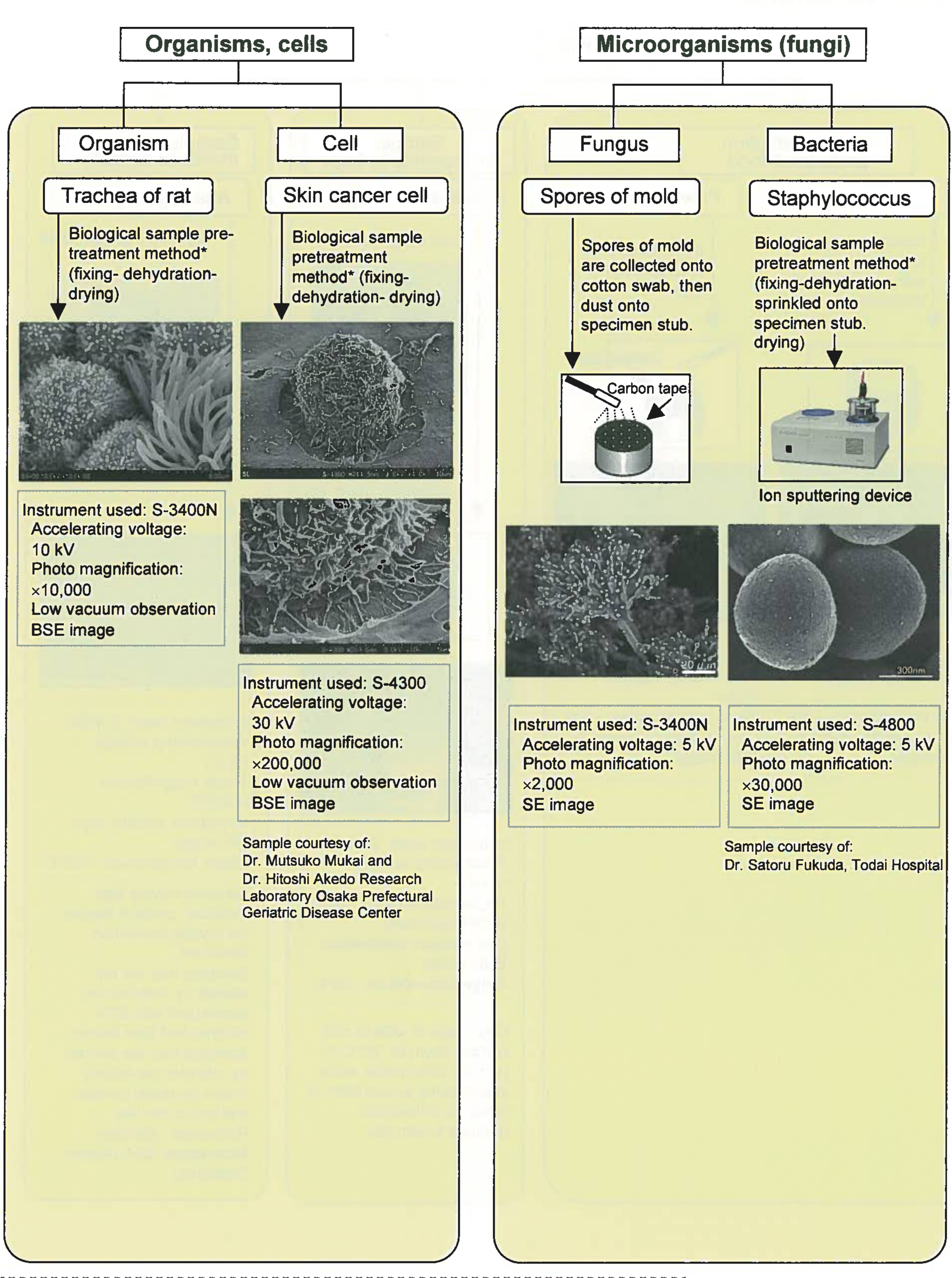
BSE mode

Instrument used: S-4800
Accelerating voltage: 2 kV
Photo magnification: ×50,000
E × B filter mechanism used
SE image

Use of the E × B filter mechanism enables clearly observing the interior of a void (upper figure) in SE mode or structure of multiple layers of differing composition (lower figure) in BSE mode.

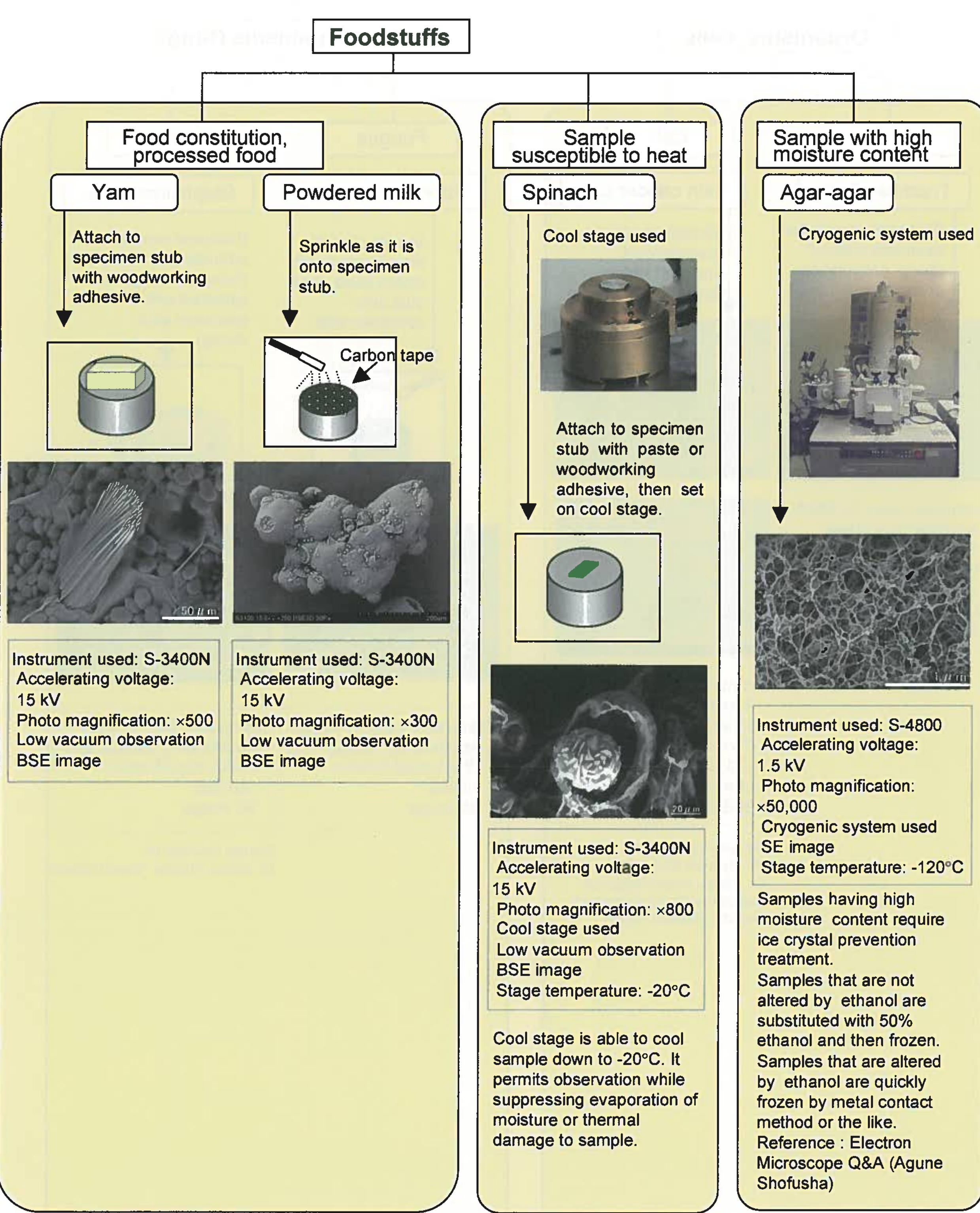
3-3 Biological Samples





Refer to preceding page 10 for biological sample pretreatment method.

3-4. Foodstuffs



Cool stage is able to cool sample down to -20°C. It permits observation while suppressing evaporation of moisture or thermal damage to sample.

Samples having high moisture content require ice crystal prevention treatment.
Samples that are not altered by ethanol are substituted with 50% ethanol and then frozen.
Samples that are altered by ethanol are quickly frozen by metal contact method or the like.
Reference : Electron Microscope Q&A (Agune Shofusha)

Chapter 4. What causes these images?

Phenomenon

- Image moves
  A C D
- Sample structure cannot be discerned
  A B C
- Image is distorted
  A C D E
- Image fluctuates
  A E
- Brightness is unstable
  A B E
- Cannot be focused
  A B C E

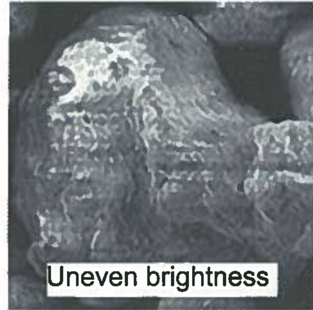
Probable cause

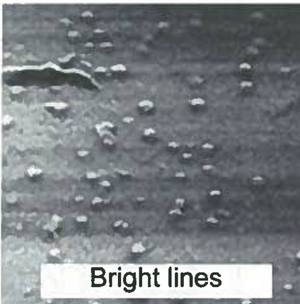
- A Charge-up
- B Contamination
- C Beam damage
- D Effect of external disturbance
- E Other

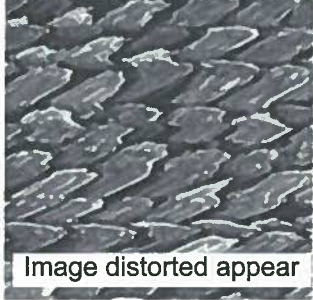
*Causes and countermeasures are described on the following pages.

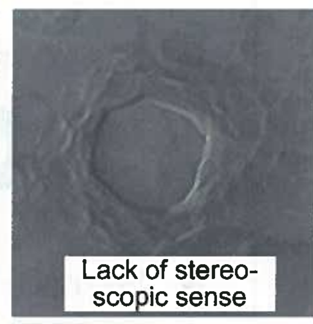
A What is the charge-up phenomenon?

Charge-up occurs during observation of non-conductive samples, and may be conspicuous especially when scan speed or magnification is changed. Below are examples of the charge-up phenomenon.

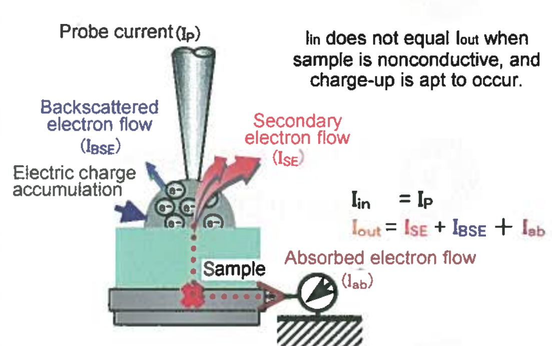


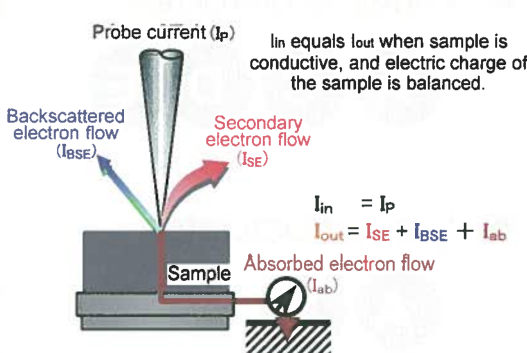






Examples of Charge-up Phenomenon





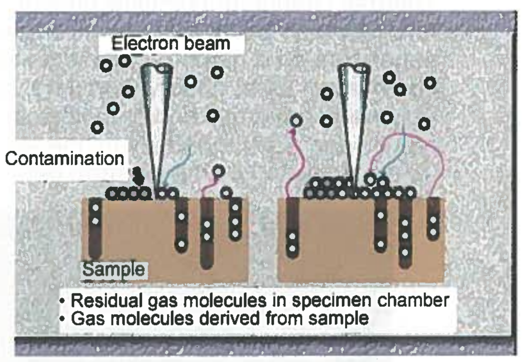
In the electron beam irradiation area on the sample surface, there are an incoming electron flow Ip and an outgoing electron flow Io (Io = I_{SE} + I_{BSE} + I_{ab}) as indicated in the above figures. When the sample surface is conductive, Io is equal to Ip. But when the charge becomes unbalanced, Ip does not equal Io, charge-up will appear and the surface potential will vary.

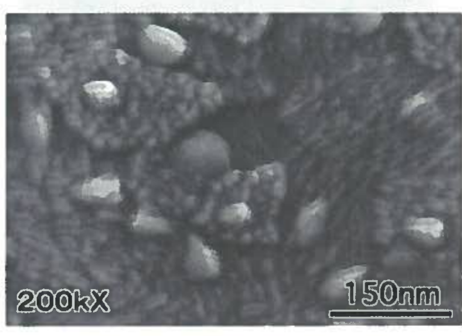
Following are countermeasures for the charge-up phenomenon.

1. Reduce the accelerating voltage.
2. Reduce the sample irradiating current.
3. Apply a metal coating.
4. Integrate the image (form an image by superimposing images obtained at rapid scan).
5. Observe images in low vacuum mode.
6. Utilize a low-acceleration BSE signal (eliminate SE signal by means of signal varying mechanism). Refer to 4-8 through 4-10 in Chapter 6 for details.

B What is contamination?

The phenomenon by which gas molecules of hydrocarbons existing around the sample collect on the sample due to electron beam irradiation, then bond together and adhere to the sample surface is referred to as contamination (see figure at the right). With the electron beam irradiating the sample, the clarity of the image at that area decreases as shown in the figure below left and it becomes darker. The reason for the darkness is thought to be that the matter accumulated on the sample surface suppresses the discharge of secondary electrons from the sample.





Example of Sample Contamination

The following steps are required in order to reduce the contamination:

- Reduction of residual gas molecules in specimen chamber (improvement of vacuum level)
- Reduction of gas molecules derived from sample

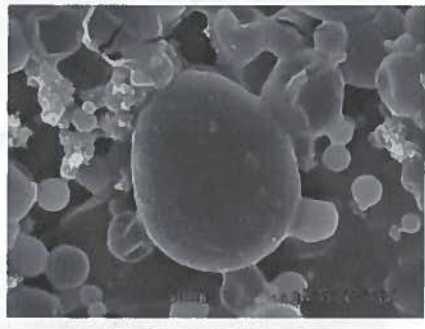
Concrete measures to achieve the above reductions are as follows.

1. Use a minimum amount of conductive paste or tape when mounting the sample in the instrument.
2. Thoroughly dry the conductive paste with a dryer or the like prior to inserting sample into the instrument for observation.
3. Heat and degas the sample in a vacuum device.
4. Carry out focusing as quickly as possible and avoid observing the same location for a long time especially at high magnification.
5. Observe samples while cooling the sample surroundings with a cold trap.

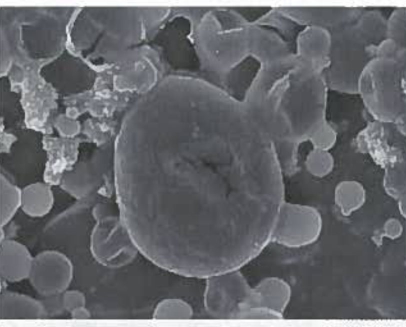
The reason why contamination is conspicuous on the left side of the electron beam scanning area is that time is provided in electron beam control for the electrons to remain there. To prevent a stray magnetic field from affecting the image, a method of scanning that is synchronous with the power frequency (50 or 60 Hz) is utilized (power supply synchronous scan), and it requires a waiting time before the start of scan. A recent method of preventing this contamination is to equip the SEM with a beam blanking function with which the electron beam cannot irradiate the sample during the waiting time before the start of scan.

What is beam damage?

Thermal change or chemical change occurring on a sample due to electron beam irradiation is referred to as beam damage. Temperature rise of the sample due to the electron beam is dependent on a number of factors including accelerating voltage and intensity of the beam, observation area, observation time, specific heat and heat conductivity of the sample, and others. Polymeric materials and/or biological samples are generally susceptible to heat, and may be readily damaged thermally by the electron beam. An example of thermal damage to polymeric material caused by the electron beam is given below.



No beam damage



Damage due to beam irradiation

Following are countermeasures available for beam damage.

1. Reduce the sample irradiating current.
2. Lower the accelerating voltage.
3. Apply metal coating to the sample (to improve heat conductivity).
4. Observe the sample while cooling it.

D Effect of disturbance

Fringes or distortion appearing on a SEM image profile may be caused by vibration or a stray magnetic field. Countermeasures for image disturbance due to vibration are given below.

1. Keep the instrument well away from vibration sources such as air-conditioner or pumps.
2. Do not let high-voltage cables from the column come in contact with the wall or other installation items.
3. Don't let the draft from an air-conditioner outlet contact the column directly.

Countermeasures for image disturbance due to a stray magnetic field are as follows.

1. Keep the instrument well away from magnetic field sources such as transformer or large capacity power cables.
2. Shorten the working distance (see 1-7 or 4-5 in Chapter 6) and apply strong excitation to the condenser lens to counter the effect of a magnetic field.
3. Use a magnetic field canceller.

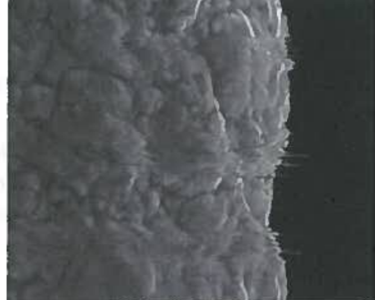


Image affected by vibration

Image affected by magnetic field

E Other causes of image abnormalities

Symptom	Possible causes
Sample moves	• Sample is not fixed in place adequately when sampling. • Screw of specimen holder is not tightened adequately. • Sample is inserted incompletely onto specimen stage. • Compressor operated while the stage is locked.
Image fluctuates	• Irradiating current is low (change the excitation of condenser lens). • Lower image is being observed at short WD in the case of semi in-lens SEM. • WD is long in low vacuum SEM observation mode.
Focus cannot be obtained	• Inadequate optical axis alignment • Objective aperture contaminated • Recheck the instrument parameters.

Chapter 5. SEM Instruments Available

FE-SEM Lineup

As of March 2008

Ultrahigh-Resolution Field-Emission SEMs

S-5500

In-lens type FE-SEM (high vacuum)

Due to a strongly excited objective lens, a world's highest resolution of 0.4 nm (at 30 kV) has been realized, and operability is improved by means of a newly developed display unit and GUI.

S-4800

Semi in-lens type FE-SEM (high vacuum)

This instrument permits observing the surface of large samples (150 mm diameter) plus elemental analysis. It also allows observing the topmost surface of a sample with minimum damage at a low irradiating voltage of 500 V or less by applying the deceleration function.

Ultrahigh-Resolution Analytical SEM

SU-70

Semi in-lens type Schottky SEM (high vacuum)

A Schottky type FE-SEM that permits various analyses of specific locations and ultrahigh resolution. In addition to elemental analysis, it allows analysis of crystal orientation and crystal defects plus evaluation of impurities.

High-Resolution SEM

SU6600

Out-of-lens type Schottky SEM (low/high vacuum)

This instrument permits observation/evaluation at a high resolution simultaneously with material analysis. The vacuum level of the specimen chamber can be freely changed between high and low vacuum (10 Pa to 300 Pa).

Hi-SEM Lineup

Scanning Electron Microscopes

S-3400N

Out-of-lens type SEM with thermionic gun (low/high vacuum)

Equipped with new type electron optics plus the same GUI system as used in the S-4800/S-5500 SEM. It is a low vacuum SEM featuring high-level functions and easy operation.

S-3700N

Out-of-lens type SEM with thermionic gun (low/high vacuum)

Incorporates the functions of the highly evaluated electron optics and control system used in the S-3400N. And a newly designed large specimen chamber accommodates samples up to 300 mm in diameter. Wide-range observation over a maximum of 203 mm diameter is feasible.

Scanning Electron Microscope

SU-1500

Out-of-lens type SEM with thermionic gun (low/high vacuum)

A compact, high-performance low-vacuum SEM providing an image resolution of 3.0 nm despite its small size (main unit width of only 55 cm).

Tabletop Microscope

TM-1000

Tabletop microscope (low vacuum)

Features Compact design desk-top size, simple operation comparable to a digital camera. This instrument enables observation of uncoated samples of insulating material plus observation of three-dimensional shapes at a large focal depth.

Chapter 6. Q&A for persons wanting to know more about the SEM!

1. Concerning formation of the electron beam ························· 31

1) How is a fine electron beam formed?
2) How is a finer electron beam obtained?
3) What kinds of electron guns are used in the SEM?
4) What are the differences between the kinds of electron guns?
5) What is the operating principle of the electron lens?
6) What is lens aberration?
7) What kinds of objective lenses are used in the SEM?

2. Concerning evacuation ·· 37

1) Why must the electron beam path be placed in a vacuum?
2) How is the evacuation performed?
3) What merits and/or demerits do the vacuum pumps have?
4) What kind of maintenance do vacuum pumps require?

3. Generation, detection and usage of SEM signals ····················· 39

1) What happens when electrons strike the sample?
2) How are backscattered electrons produced?
3) What kind of characteristics do backscattered electrons possess?
4) How are backscattered electrons detected?
5) What is the signal varying mechanism?
6) What are backscattered electrons used for?
7) How are secondary electrons produced?
8) What kind of characteristics do secondary electrons possess?
9) How are secondary electrons detected?
10) What are secondary electrons used for?

4. Viewing conditions for acquiring good SEM images ················· 49

1) What is a good SEM image?
2) What effect does accelerating voltage or condenser lens current have on image quality?
3) How must accelerating voltage be selected according to the sample?
4) What is the function of the objective lens aperture?
5) How does working distance (WD) affect image quality?
6) What is astigmatism and how is it corrected?
7) What is focal depth?
8) What is the charge-up phenomenon?
9) How is sample surface potential changed by charge-up?
10) How is the image disturbed by charge-up?
11) What are the advantages and disadvantages of sample coating?
12) What sample coating methods are available and how do they differ?
13) What is the principle of the magnetron sputtering method and plasma film formation method?

5. Principle and applications of low vacuum SEM ······················60
1) What kind of instrument is the low vacuum SEM?
2) Why is it that an insulating material can be observed without the charge-up phenomenon by means of the low vacuum SEM?
3) How is SEM signal detection performed in the low vacuum SEM?
4) What are considered to be the demerits of the low vacuum SEM?
5) Please give a concrete example of application of the low vacuum SEM.

6. Generation/detection of x-rays and elemental analysis method ········63
1) How are x-rays produced?
2) What kinds of characteristic x-rays are there?
3) Are there any x-rays other than the characteristic ones produced by electron beam irradiation?
4) What is the extent of the area (depth, spread) in which characteristic x-rays are produced?
5) What kind of x-ray detector is used in the SEM?
6) The principle and instrument configuration for EDX.
7) What is the multichannel pulse height analyzer?
8) The Principle and instrument configuration for WDX.
9) What is the tandem type proportional counter used in WDX?
10) Are there any particular cautions to be observed in analysis using WDX?
11) What are the differences between EDX and WDX?
12) How is quantitative analysis performed?

7. How to improve accuracy in x-ray analysis ····························70
1) What should be observed in order to improve the accuracy in x-ray analysis?
2) How can characteristic x-rays be efficiently produced?
3) What should be done in order to avoid detecting x-rays produced from other than the sample (location of interest)?
4) Is there a method for reducing the x-ray generation area?
5) How does the shape of the sample affect the analytical accuracy?
6) What kind of pseudo peak appears in EDX?
7) What should be done to achieve high-accuracy EDX analysis with the low vacuum SEM?

8. Principle and applications of STEM ··································75
1) What kind of instrument is the STEM?
2) What are the characteristics of the STEM signal?
3) What features are obtained by combining EDX analysis with the STEM?
4) What is the method of preparing samples for STEM observation/analysis?
5) What are the applications of STEM (EDX)?

1. Concerning formation of the electron beam

1) How is a fine electron beam formed?

In the usual SEM instrument, the electron source (diameter d_o) formed by the electron gun (See1-3 in chapter 6) is reduced and made into a fine electron beam (diameter d_k) by the condenser lens and objective lens as indicated in Fig. 1. The condenser and objective lenses of the SEM are both electromagnetic lenses (1-5). The magnification M_1, M_2 (or reduction rate in SEM) of these lenses is expressed by $M_1 = b_1/a_1$, $M_2 = b_2/a_2$, and the final beam diameter is $d_k = d_o \cdot M_1 \cdot M_2$.

Although it would seem that d_k could be made infinitely smaller by reducing M_1 and M_2, it is not quite that simple. First of all, if b_1 is shortened and M_1 reduced excessively, the electrons incident on the objective lens will be reduced and a probe current (irradiating current) required for formation of an image will not be obtained (see Fig. 1). Actually the condenser lens current is adjusted so that M_1 will be minimized in the range where a sufficient probe current can be obtained (4-2). Also, since image distortion caused by lens aberration will be added (1-6), there is a limit to the reduction of d_k. Note that α here represents opening angle of the electron beam (1-5).

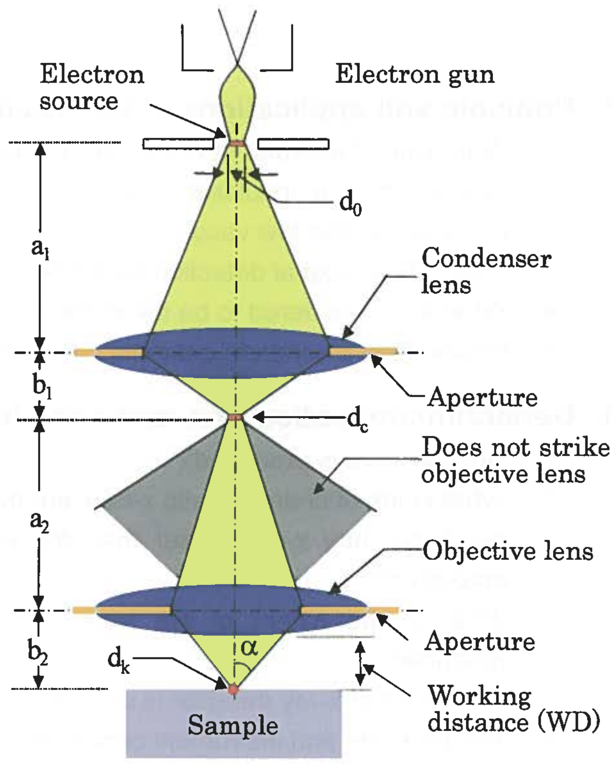


Fig. 1 Scheme for Obtaining a Fine Electron Beam

2) How can we obtain an even finer electron beam?

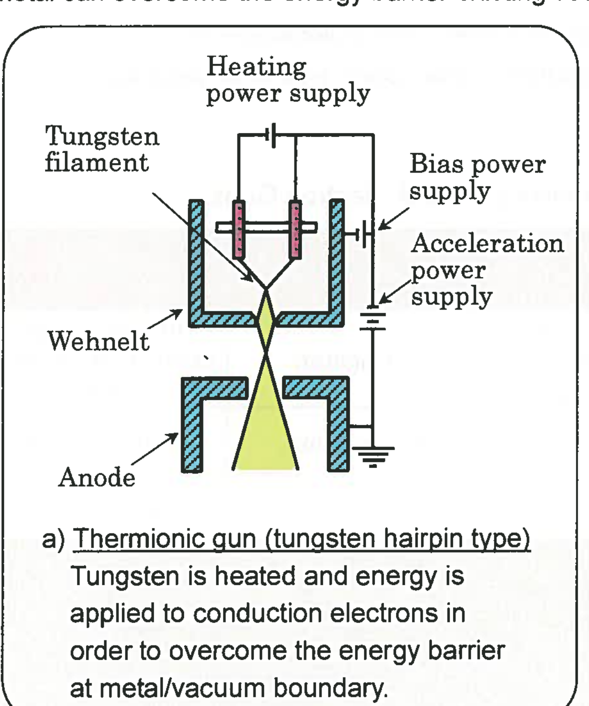
In order to obtain a fine electron beam in the SEM, the instrument utilizes an electron gun featuring a small electron source, a large current density (current (in A/cm^2) per unit area), and a small energy width (energy variation (in eV)) of discharged electrons, plus an objective lens having very little aberration. See (1-4) and (1-7) for more details on the characteristics of the electron gun and objective lens.

Following are points to keep in mind when setting the viewing conditions.

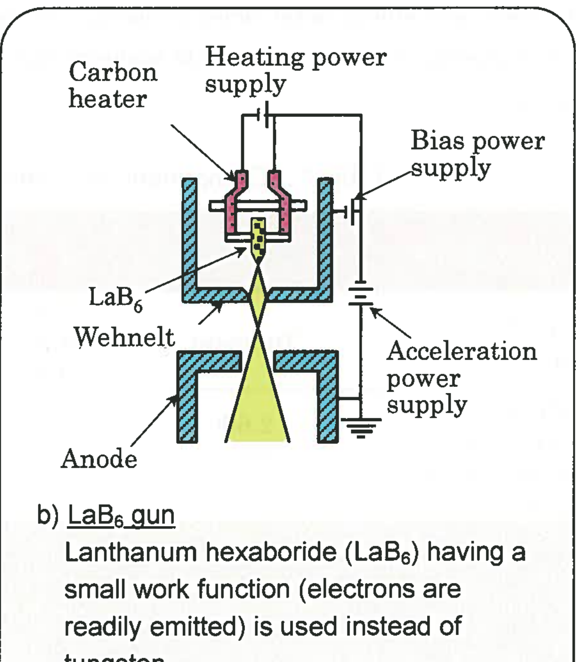
1) Adjust the condenser lens current appropriately as mentioned on (1-1).
2) Use the objective lens at the shortest possible working distance (WD)(1-7, 4-5).
3) Set an appropriate bore diameter for the objective lens aperture in order to minimize lens aberration (1-6).
4) Since the emission current decreases with a lower accelerating voltage in a SEM using a thermionic source (1-3), carry out filament height adjustment and bias adjustment to obtain the desired current.
5) Be sure to carry out astigmatism correction (4-6).

3) What kinds of electron guns are used in the SEM?

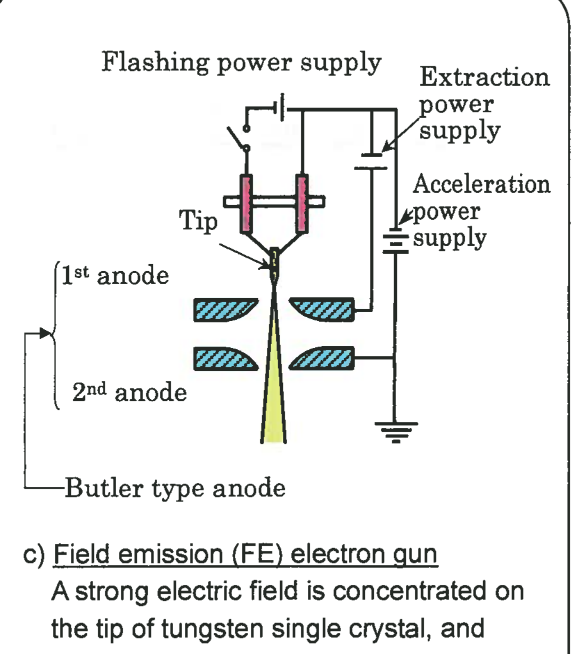
Thermionic type gun (tungsten hairpin type, lanthanum hexaboride (LaB_6) type), field emission (FE) gun and Schottky type gun are generally used in the SEM. Figure 2 illustrates the configuration and principle of electron emission for each type of gun. Electron emission is achieved in respective cases by applying thermal excitation (applying energy to electrons while heating), by using a tunnel effect (forcing electrons out of an energy barrier via a strong electric field), and by using the Schottky effect (lowering the energy barrier by means of a strong electric field), with which conduction electrons in metal can overcome the energy barrier existing at the boundary of metal and vacuum.



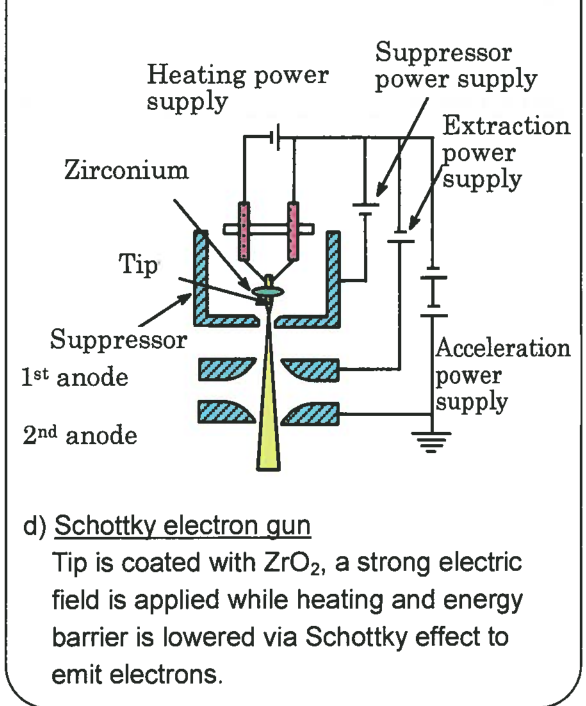
a) Thermionic gun (tungsten hairpin type)
Tungsten is heated and energy is applied to conduction electrons in order to overcome the energy barrier at metal/vacuum boundary.



b) LaB_6 gun
Lanthanum hexaboride (LaB_6) having a small work function (electrons are readily emitted) is used instead of tungsten.



c) Field emission (FE) electron gun
A strong electric field is concentrated on the tip of tungsten single crystal, and energy barrier is narrowed and electrons forced out via the tunnel effect.



d) Schottky electron gun
Tip is coated with ZrO_2, a strong electric field is applied while heating and energy barrier is lowered via Schottky effect to emit electrons.

Fig. 2 Configuration and Principle of Electron Emission with Various SEM Electron Guns

4) What are the differences between the kinds of electron guns?

Table 1 compares the characteristics of the tungsten hairpin type thermionic gun, LaB_6 thermionic gun, and FE and Schottky electron guns. Important factors in the formation of a fine electron beam are electron source diameter, brightness and energy width, and the FE gun excels in all of these characteristics, thus finding wide use in high-resolution SEMs. As a universal type of electron gun, the tungsten hairpin type thermionic gun operates with a relatively simple evacuation system and provides a large probe current, and is applicable to a wide range of uses from low vacuum SEM (5-1) up to EPMA (6-4). The Schottky gun is slightly inferior to the FE gun in electron source diameter, brightness and energy width while providing a large probe current, and is applicable to a multi-functional high-resolution SEM featuring high-resolution observation plus x-ray analysis function (6-6, 6-8).

Table 1 Comparison of Characteristics of SEM Electron Guns

Kind of gun	Tungsten hairpin	LaB_6	Field emission	Schottky
Cathode material	Tungsten	Lanthanum hexaboride single (multi) crystal	Tungsten	Tungsten single crystal/zircon-ium oxide
Cathode temperature (K)	2,600	1,800	Room temp.	1,700 to 1,800
Cathode work function (eV)	4.4	2.6 to 2.7	4.1	2.7 to 2.9
Electron source diam.	30 μm	10 μm	5 nm	20 nm
Brightness ($A/cm^2 \cdot sr$)	10^6	10^7	10^9	10^8
Energy width (eV)	2.0	1.5	0.2	0.3 to 1.0
Max. probe current (A)	10^{-7}	10^{-7}	10^{-9}	10^{-7}
Working pressure (Pa)	10^{-4}	10^{-5}	10^{-8}	10^{-7}
Cathode lifetime	~50 hr	~1000 hr	1 year or more	~9 months

5) What is the configuration and/or operating principle of the electron lens?

Electron lenses include magnetic field lens and electrostatic lens, and the description here covers the magnetic field lens that is generally employed in the SEM. The magnetic field lens serves to focus the electron beam formed by the electron gun (1-3) into a fine probe and irradiate it onto the sample surface, and the SEM utilizes one or two condenser lenses and an objective lens for this purpose. The configuration and operating principle of the condenser and objective lenses in the SEM are shown in Fig. 3.

The electron beam incident on these lenses is affected by the magnetic field produced through the gap (pole-piece) in the magnetic path, is converged while rotating around the optical axis, and forms a reduced image of the beam on the image plane. Note that the objective lens is designed so as not to interfere with sample movement or secondary electron, x-ray signal detection (3-7, 6-5).

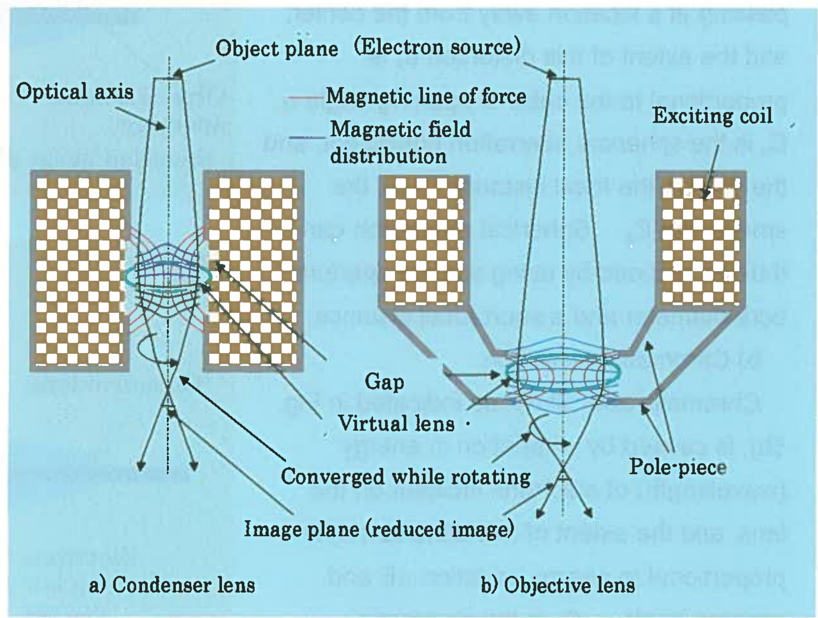


Fig. 3 Configuration and Function of Electron Lens

This action is explained in further detail in Fig. 4. The electrons emitted from each point of the electron beam are focused onto the corresponding image points by the electron lens and form a reduced image of the beam on the image plane. The reduction rate M at this time is expressed by $M = b/a$ (1-1).

The reduced image from the condenser lens becomes the beam for the objective lens, and the reduced image from the objective lens is irradiated onto the sample.

Note that the reduced image includes some distortion caused by lens aberration (1-6). The aperture angle α is determined geometrically from aperture diameter of the lens (bore diameter of aperture) and focal distance, and has a large influence on lens characteristics such as lens aberration and focal depth which will be explained later (4-4).

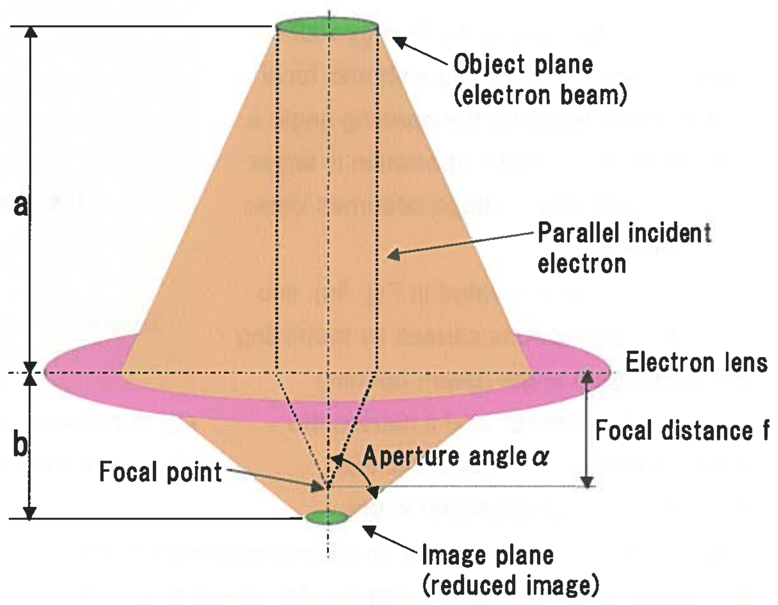


Fig. 4 Reduction of Electron Beam by Magnetic Field Lens

6) What is lens aberration?

Lens aberrations mainly include spherical aberration, chromatic aberration, diffraction and astigmatism (4-6), among which it is difficult to completely exclude astigmatism. It is therefore recommended to carry out observation under conditions in which these aberrations are minimized in order to obtain good SEM images.

a) Spherical aberration

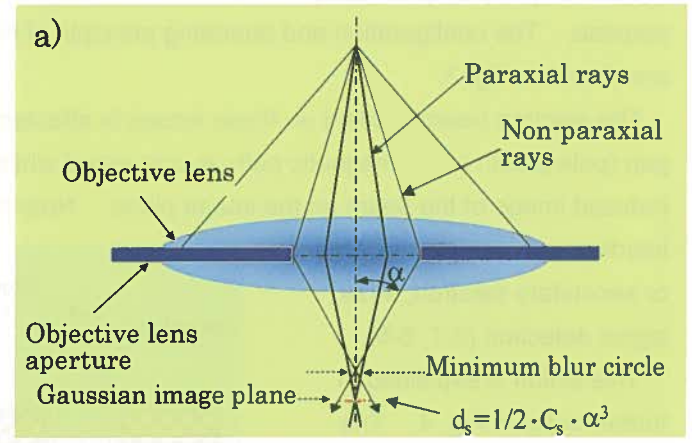
Spherical aberration, as shown in Fig. 5a), is a distortion caused by a difference in convergent positions between electrons passing near the lens center and electrons passing at a location away from the center, and the extent of this distortion d_s is proportional to the cube of opening angle α. C_s is the spherical aberration coefficient, and the shorter the focal distance (1-5), the smaller the C_s. Spherical aberration can thus be reduced by using a small aperture bore diameter and a short focal distance.

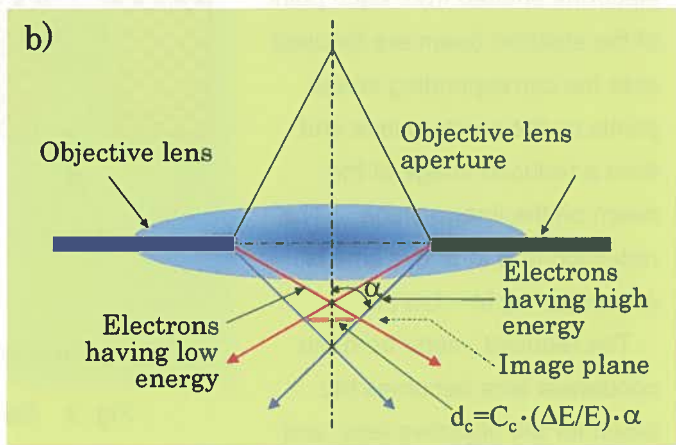
b) Chromatic aberration

Chromatic aberration, as indicated in Fig. 5b), is caused by a variation in energy (wavelength) of electrons incident on the lens, and the extent of this distortion d_c is proportional to energy variation ΔE and opening angle α. C_c is the chromatic aberration coefficient, and becomes smaller as the focal distance becomes shorter. Chromatic aberration can therefore be reduced by decreasing the energy width of electron beam (1-4), using a shorter focal distance and reducing the opening angle α. The effect of chromatic aberration is larger as the accelerating voltage becomes lower.

c) Diffraction

Diffraction, as illustrated in Fig. 5c), is a diffraction of electrons caused by restricting the convergent angle (beam opening angle α) of electrons, and it causes the beam to widen. The extent of this aberration d_d is proportional to the wavelength λ of electrons and in inverse proportion to opening angle α. Although it can be reduced by increasing the aperture diameter, the spherical and chromatic aberrations will increase, so the aperture diameter must not be enlarged too much.





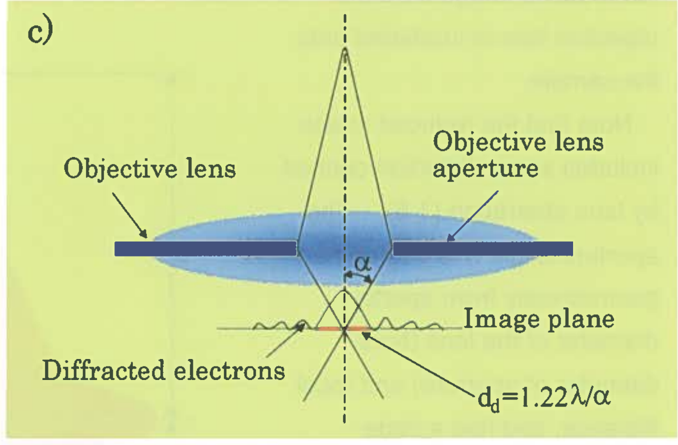


Fig. 5 Generation of Lens Aberrations and Their Extent
a) Spherical aberration, b) Chromatic aberration,c) Diffraction

7) What kinds of objective lenses are used in the SEM?

Spherical and chromatic aberrations of the objective lens become smaller by shortening the focal distance f. So in addition to the out-of-lens type of objective lens generally used with the SEM, an in-lens type having a short f for high-resolution SEMs and a snorkel type (or semi in-lens type) have been developed.

a) Out-of-lens type objective lens

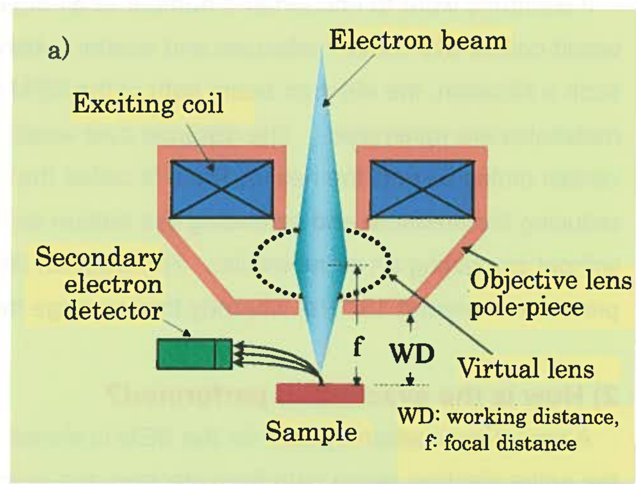
With this lens, as indicated in Fig. 6a), a virtual lens is formed at a position above the sample, so there is a limit on how much f can be shortened. Although this makes it rather unsuitable for a high-resolution SEM, it is convenient for observation of large samples since a large working distance (WD; distance from lower face of lens pole-piece to surface of sample) can be provided. This lens is applicable to a multifunctional SEM since it permits observation/analysis of magnetic material with which leakage magnetic field around the sample is small.

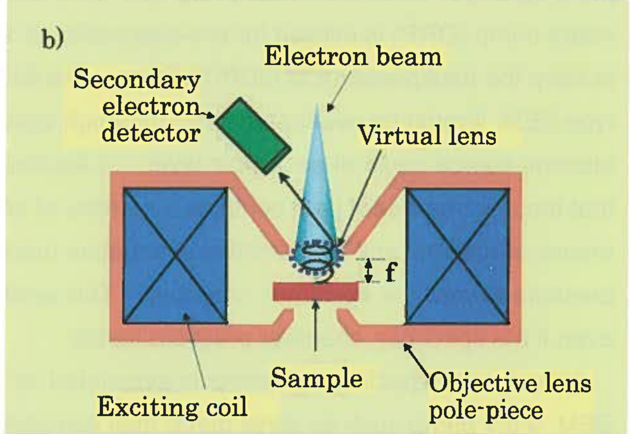
b) In-lens type objective lens

The feature of this lens, as indicated in Fig. 6b), is that f can be extremely shortened because a sample is placed in the gap between upper and lower pole-pieces. It is therefore applicable to ultrahigh-resolution SEMs, but there is a limit on sample size and it is unsuitable for observing strongly magnetic material. Secondary electrons are spun upward by the strong magnetic field of the lens and detected by an SE detector mounted above the exciting coil and lens.

c) Snorkel (semi in-lens) type objective lens

With the snorkel type lens, as indicated in Fig. 6c), a pole-piece gap is formed and by bringing the virtual lens close to the sample, large samples can be observed at a short f (≒ WD). Secondary electrons are detected by the upper SE detector when f is short, or by the lower SE detector when f is long.





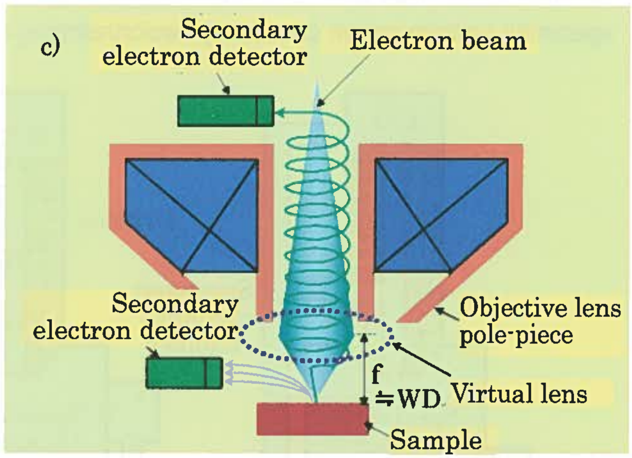


Fig. 6 Kinds of Objective Lenses Used in SEM
a) Out-of-lens type, b) In-lens type,
c) Snorkel (semi in-lens) type

2. Concerning Evacuation

1) Why must the electron beam path be placed in a vacuum?

If electrons were to encounter a number of air molecules on their way toward the sample, they would collide with those molecules and scatter, whereby they would not reach the sample. To avoid such a situation, the electron beam path of the SEM must be kept in a vacuum with which air molecules are minimized. The distance over which an electron advances after once colliding with a certain molecule until the next collision is called the "average free motion of the electron", and by reducing the pressure and extending this motion as far as possible, the electron can reach the sample without contacting an air molecule. The electron beam path of the SEM is generally kept at a pressure of around 10^{-3} Pa, whereby the "average free motion of the electron" is about 40 m.

2) How is the evacuation performed?

A typical evacuation system for the SEM is shown in Fig. 1. With a SEM having a thermionic gun, the entire electron beam path from electron gun to specimen chamber is evacuated by oil diffusion pump (ODP) or turbo molecular pump (TMP), and maintained at a pressure of 10^{-3} to 10^{-4} Pa. An oil rotary pump (ORP) is utilized for pre-evacuation at specimen exchange or for post-evacuation in order to keep the backpressure of ODP/TMP low. As for the electron gun chamber of the field emission type SEM, it must be evacuated to an ultrahigh vacuum of around 10^{-8} Pa to keep the surface of the electron source clean at an atomic level. A sputter ion pump (SIP) is used for this purpose. Note that the electron beam path contains a number of small partitioned compartments connected by means of orifices, and a differential evacuation mechanism is utilized for gradually increasing the pressure toward the specimen chamber. This enables keeping the electron gun pressure constant even if the specimen chamber pressure varies.

Although the specimen chamber is evacuated by ODP or TMP the same as with the thermionic type SEM, a dry pump such as scroll pump may sometimes be used in place of ORP for evacuation of the specimen exchange chamber or post-evacuation. The reason for this is to minimize the effect of specimen contamination caused by backstreaming of oil vapor from ORP.

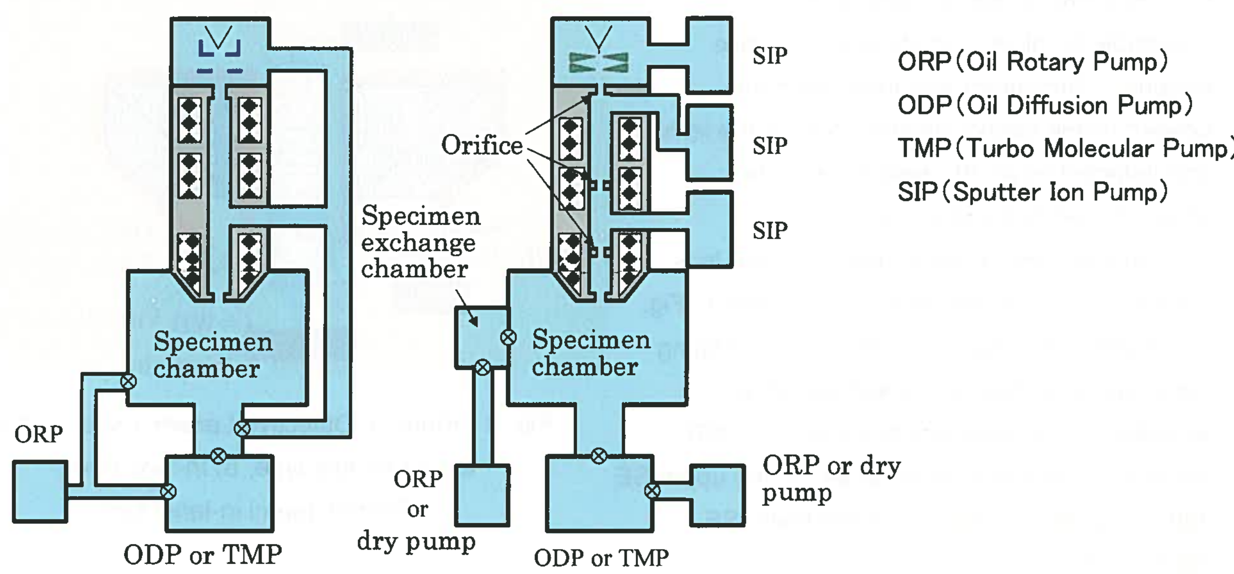


Fig. 1 Example of SEM Evacuation System

3) What are the merits and demerits of each type of vacuum pump?

Table 1 gives the operating pressure and merits or demerits of the main vacuum pumps.

Table 1 Operating Pressure and Merits/Demerits of Vacuum Pumps

Kind of pump	Operating pressure (Pa)	Merits	Demerits
ORP	Atmospheric pressure to 10^{-1}	• Usable for pre-evacuation (atmos-pheric pressure) • Simple structure and low-priced	• Unsuitable for oil-free application • Floor vibration is rather large.
Dry pump (scroll pump etc.)	Atmospheric pressure to 10^{-1}	• Usable for pre-evacuation (atmos-pheric pressure) • Oil-free evacuation	• Structure is rather complex and high-priced.
ODP	10^{-2} to 10^{-7}	• Rapid evacuation • No noise or vibration caused	• Some dispersion of oil vapor • Requires pre- and post-evacuation • Requires wafer chiller
TMP	10^{-1} to 10^{-8}	• Oil-free evacuation • Wide range of operating pressure	• Complex structure and high-priced • Requires pre- and post-evacuation
SIP	10^{-3} to 10^{-8}	• No mechanical vibration • Low power consumption	• Evacuation rate is low. • Requires pre-evacuation

4) What kind of maintenance do vacuum pumps require?

The ORP pump requires oil exchange once every six months to a year, to be carried out by the user. Exchange should be performed using the oil and method prescribed in the instruction manual for the pump. Although other types of pumps have almost no maintenance to be carried out by the user, follow the pump instruction manual as to frequency of periodical inspection specified by the manufacturer.

3. Generation, detection and usage of SEM signals

1) What happens when electrons strike the sample?

An electron is very small in comparison with the space between the atoms which compose a specimen. Therefore, when an electron penetrates the specimen, it is scattered through interactions with the atoms. In this process, some of the incident electrons fly back into vacuum as backscattered electrons (see 2) Fig 3), but others lose energy while emitting secondary electrons Fig 3), x-rays Fig 3), light (cathode luminescence), etc. and finally remain inside the specimen as shown in Fig. 1. In case of a conductive specimen, the primary electrons remaining inside the specimen are detected as specimen (absorbed) current.

The electron dispersion inside a specimen can be visualized by the technique of Monte Carlo simulation. An example of this visualization is shown in Fig. 2. This example reveals that the incident electrons are dispersed over a wider area when their energy is higher and the specimen density is smaller.

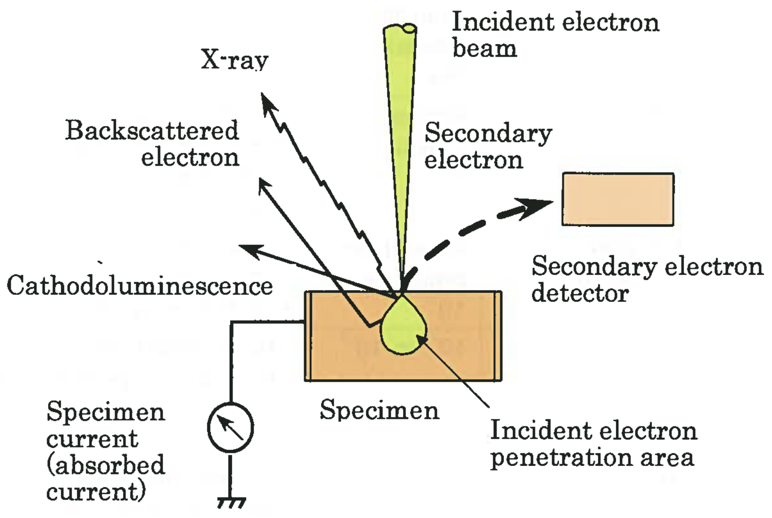


Fig. 1 Interactions between Incident Electrons and Specimen

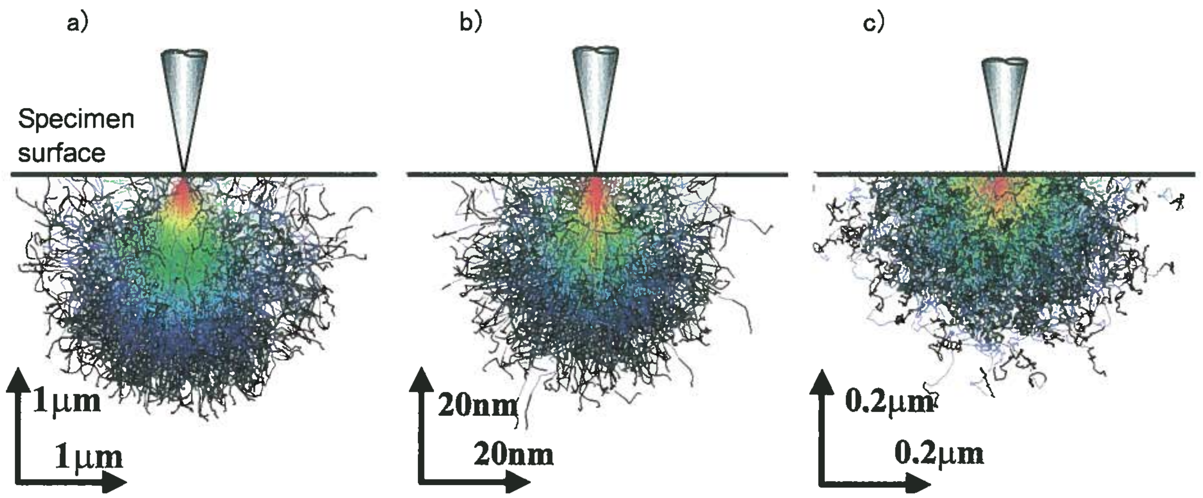


Fig. 2 Electron Scattering inside Specimen as Visualized by Monte Carlo Simulation

a) Upon incidence of 15 keV on carbon (C) specimen

b) Upon incidence of 1 keV on carbon (C) specimen

c) Upon incidence of 15 keV on gold (Au) specimen

2) How are backscattered electrons produced?

Backscattered electrons (also called reflected electrons) are given birth in the following process. The electrons incident on a specimen cause interactions with the constituent atoms of the specimen and are scattered backward into the vacuum, or in the direction opposite to the beam as shown in Fig. 3. Electron scattering in a specimen are classified into 2 types; one is elastic scattering in which incident electrons are scattered at a large angle with almost no loss in energy, and the other is inelastic scattering in which incident electrons loose energy but are scattered at a small angle. As a typical inelastic scattering, we know the emission process of secondary electron and characteristic x-ray.

The elastically scattered electrons having approximately the same energy as the incident electrons are scattered from the vicinity of specimen surface into the vacuum, while the inelastically scattered electrons which have lost energy substantially are scattered from a comparatively deep location in the specimen into the vacuum. These electrons are collectively called backscattered electrons (reflected electrons).

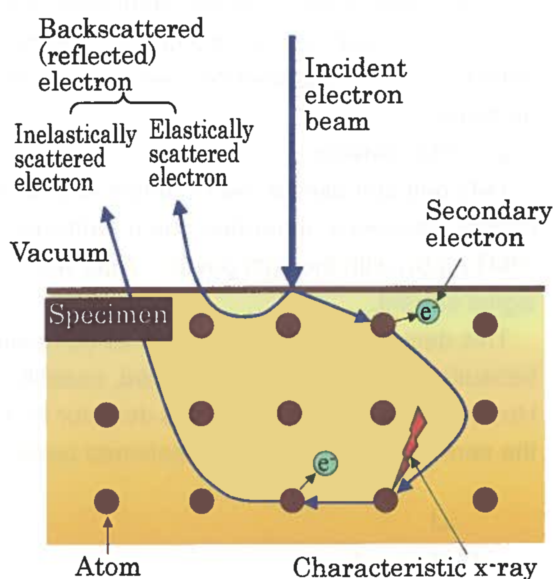
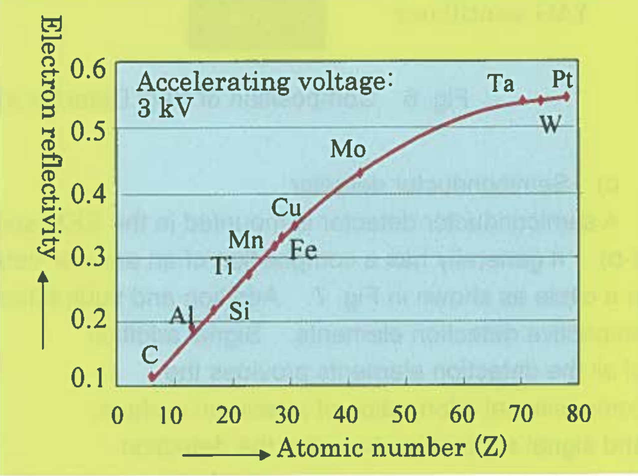


Fig. 3 Scattering of Incident Electrons inside Specimen

3) What kind of characteristics do backscattered electrons possess?

The count ratio of backscattered electrons to electrons incident on specimen surface is called "electron reflectivity." A change in this reflectivity renders a contrast to backscattered electron image. Figure 4 shows differences in electron reflectivity among some specimens. It is obvious that this reflectivity value becomes larger as the mean atomic number (or density) of specimen increases. Thus, backscattered electron signal covers the contrast which reflects the specimen composition, so it is considered that this signal provides information about the compositional distribution of specimen surface.

Also, it is known that the electron reflectivity changes depending on the incident angle of electron beam and it becomes larger for a rise in specimen tilting angle as exemplified in Fig. 5. This signifies that the topographic information of specimen surface is contained in the backscattered electron signal.



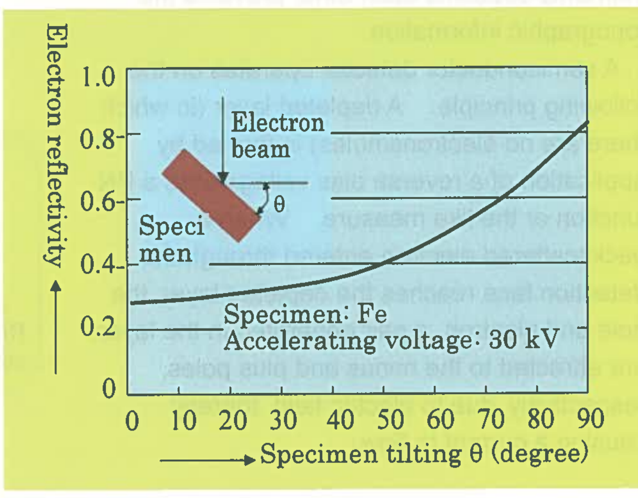


Figure at upper right
Fig. 4 Electron Reflectivity's Dependency on Atomic Number

Figure at right
Fig. 5 Electron Reflectivity's Dependency on Specimen Tilting

4) How are backscattered electrons detected?

Backscattered electrons are detectable with a backscattered electron detector which comes in various types such as semiconductor detector, YAG detector and signal varying mechanism-applied detector (3-5)). Summarized here is each structure and characteristic of the YAG and semiconductor detectors.

a) YAG detector

YAG detector uses a YAG (Yttrium Aluminum Garnet) crystal for the scintillator, with which phosphorescence generated due to entrance of backscattered electrons is led to the photomultiplier (PMT)(3-9)) with the light guide. After re-conversion into electrons, amplification is made to obtain a signal current.

This detector has features such as compatibility with image observation at a high scan speed because of its high response speed, usability over a broad range of accelerating voltages, etc. However, it is difficult to use this detector for 4-split signal detection and arithmetic processing unlike the semiconductor detector mentioned below.

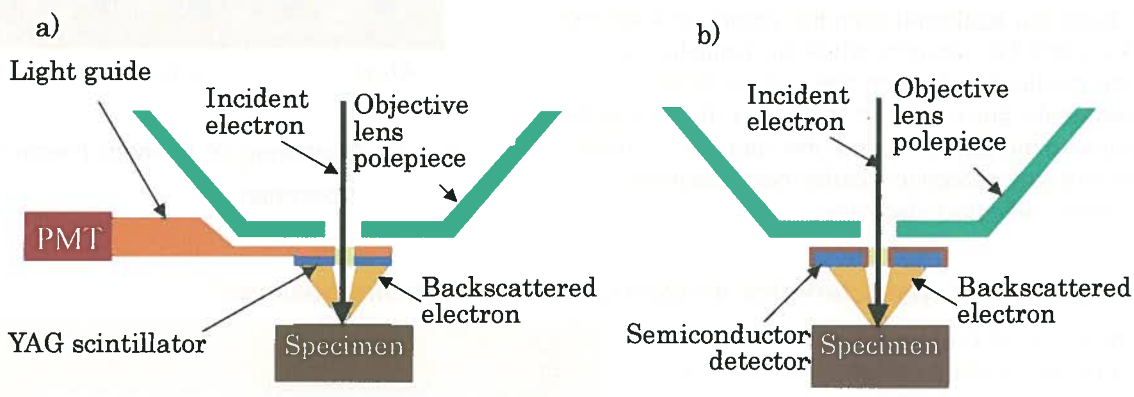


Fig. 6 Composition of YAG Detector a) and Semiconductor Detector b)

b) Semiconductor detector

A semiconductor detector is mounted in the SEM specimen chamber in a geometry shown in Fig. 6-b). It generally has a composition of an annular detector in which 2 or 4 detection elements are laid in a circle as shown in Fig. 7. Addition and subtraction are allowed between the signals of the respective detection elements. Signal addition of all the detection elements provides the compositional information of specimen surface, and signal subtraction between the detection elements opposing each other provides the topographic information.

A semiconductor detector operates on the following principle. A depleted layer (in which there are no electrons/holes) is formed by application of a reverse bias voltage onto a PN junction or the like measure. When a backscattered electron entered through the detection face reaches the depleted layer, the hole and electron, a pair generated in the layer, are attracted to the minus and plus poles, respectively, due to electric field, thereby causing a current to flow.

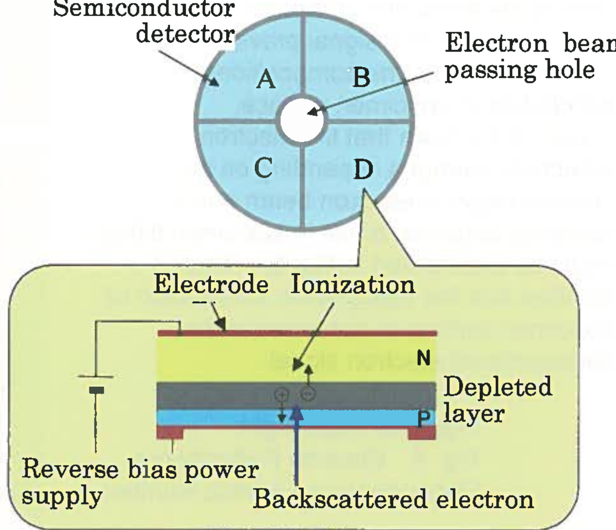


Fig. 7 Principle and Composition of Semiconductor Detector

5) What is the signal varying mechanism?(E × B filter)

In a SEM equipped with a semi-in-lens type (snorkel type) or in-lens type objective lens (1-7)), the secondary electrons (3-7)) spiral upwards due to the strong lens magnetic field, but the backscattered electrons (3-2)) take different trajectories from those of the secondary electrons due to a difference in the energy possessed. When a control electrode is set up on the trajectories of the backscattered electrons, these electrons collide with the electrode, thereby emitting the secondary electrons as shown in Fig. 8. Let us assume that the control electrode is set at a positive potential as shown in Fig. 8a). The secondary electrons emitted from the electrode are pulled back by the positive field and cannot arrive at the detector. On the other hand, the secondary electrons emitted from the specimen surface move upward while making a rotary motion and are attracted to the electric field of the secondary electron detector followed by detection. In short, only the secondary electron information will be detected.

Contrarily, let us assume that the control electrode is set at a negative potential as shown in Fig. 8b). The secondary electrons emitted from the specimen surface cannot move upward blocked by the negative field, but the those emitted from the electrode move upward and are attracted by the electric field of the secondary electron detector and detected with the detector. The detected secondary electrons also have the backscattered electron information, so a backscattered electron image is available.

This mechanism allows observation of backscattered electron images in a low accelerating voltage range from a few hundred V to thousand kV where detection is impossible with a general backscattered electron detector, so the compositional distribution on the uppermost specimen surface can be observed at a high resolution. Note that the ExB filter shown in Fig. 8 has a mechanism for automatically correcting the misalignment of the electron beam axis due to the electric field of secondary electron detector at all the accelerating voltages selectable. Figure 9 introduces imaging examples of ceramic (Al_2O_3/Ni composite material)(at accelerating voltage 2 kV). In the image a) formed at a control voltage of +200 V, the surface topography is visible due to the secondary electron information, and in the image b) formed at -150 V, the compositional distribution of Ni particles is visible due to the backscattered electron information.

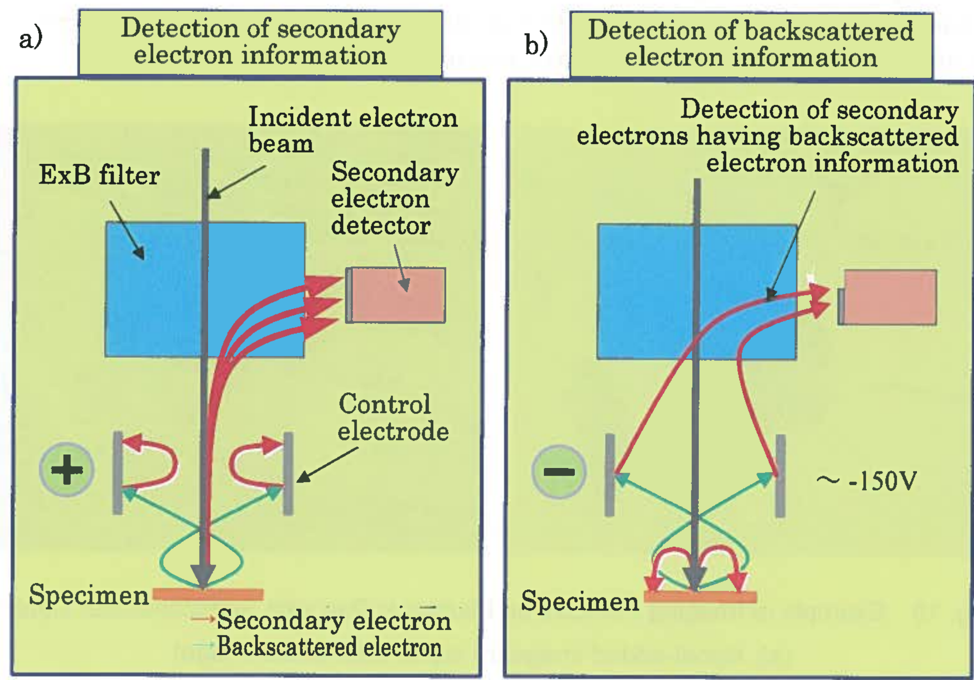


Fig. 8 Operating Principle of Signal Varying Mechanism

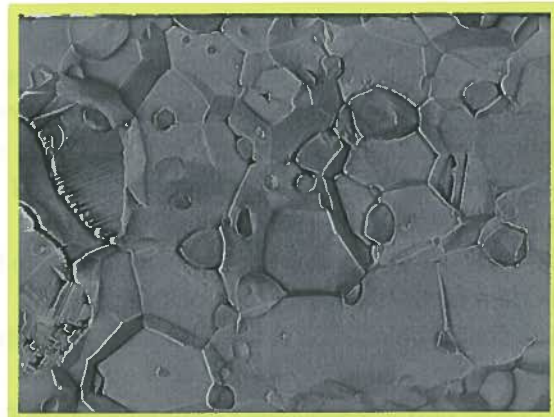
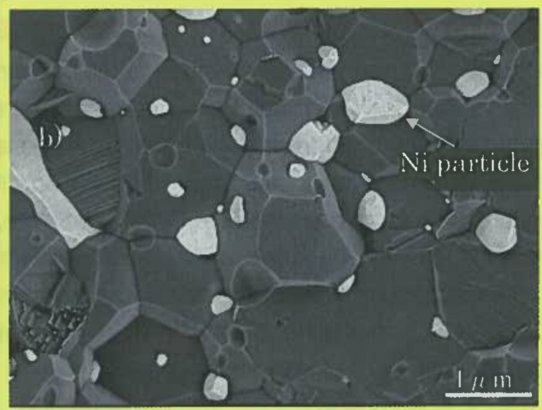



Fig. 9 Imaging Examples of Ceramic (Al_2O_3/Ni Composite Material) with Signal Varying Mechanism
a) Secondary electron image (control voltage: +200 V),
b) Backscattered electron image (control voltage: -150 V)
Accelerating voltage: 2 kV, photographing magnification: 20,000x
Specimen provided by: Prof. Tohru Sekiya, Institute of Multidisciplinary Research for Advanced Materials, Tohoku University

6) What are backscattered electrons used for?

Because backscattered electrons have the characteristics described on Page 40), they are used for visualization of the compositional distribution and topography of specimen surface. Figure 10 shows imaging examples of particles on an electronic part with an annular semiconductor detector (Fig 7)). These images were formed by adding and subtracting the signals from the A/C and B/D detection elements of the detector. The compositional distribution and topography of specimen surface are clearly observable in the signal-added image a) and signal-subtracted images b), respectively.

Also, Fig. 11 shows an image of the cross-section of a Giant Magnetic Resistor (GMR) device at an accelerating voltage of 15 kV using a YAG detector. In particular, the GMR film of about 30 nm thick could be imaged sharply. Since a YAG detector has such a high sensitivity as shown here, it is adopted for imaging the compositional distribution in a micro-area.

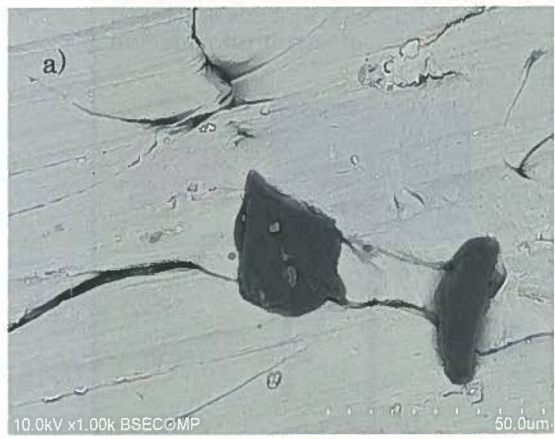
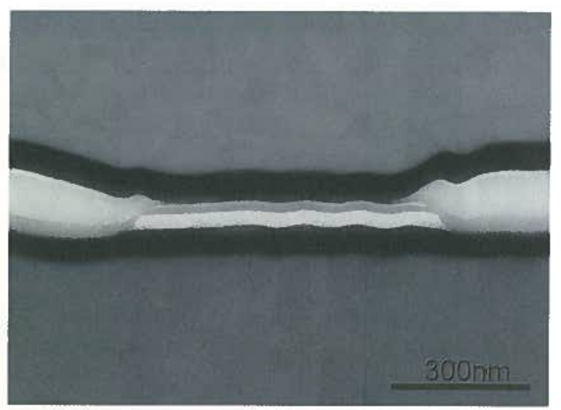


Fig. 10 Example of Imaging Particles on Electronic Part with Semiconductor Detector
(a): signal-added image, b) signal-subtracted image)

Fig. 11 Observation of GMR Film (magnetic head) with YAG Detector
GMR: Giant Magnetic Resistor
Accelerating voltage: 15 kV



7) How are secondary electrons produced?

Emission of secondary electrons occurs in the process where incident electrons penetrate a specimen and then inelastically scattered. A free electron in the specimen may receive an electrostatic force (Coulomb force) from the incident electron or scattered electron and thereby part of the latter's energy, and then jump out into the vacuum as shown in Fig. 12. This electron is called a "secondary electron." However, the energy received by a free electron is as small as a few ten eV max. Therefore, it has been thought that only the secondary electrons produced within a depth range of about 10 nm below the specimen surface can escape into the vacuum.

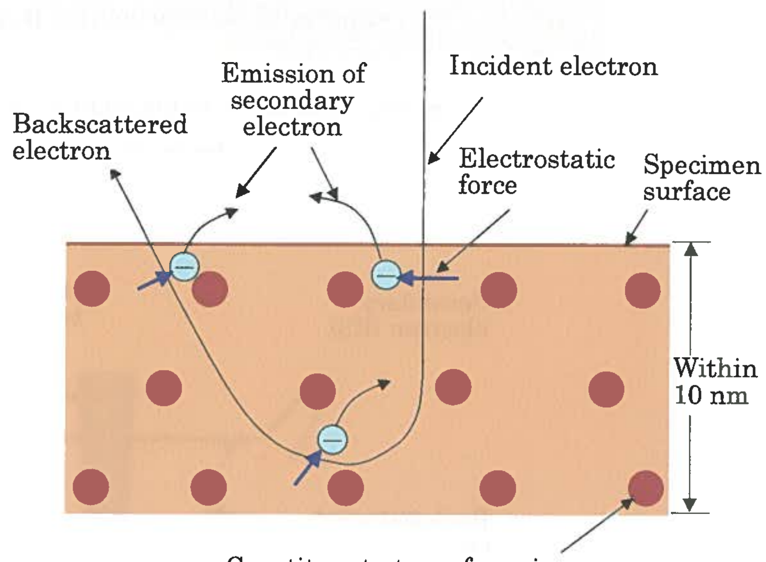


Fig. 12 Emission of Secondary Electrons from Specimen

8) What kind of characteristics do secondary electrons possess?

Figure 13 shows the energy distribution of the secondary and backscattered electrons emitted when a specimen is irradiated with the incident electron beam. As is evident from this figure, the secondary electrons have a very small energy of a few ten eV or less from the initial stage of emission in comparison with the backscattered electrons. This signifies that the secondary electron is emitted from an extremely narrow area with the information of specimen surface alone as shown in Fig. 12. Therefore, a high resolution image is expectable from the secondary electron signals.

In actuality, however, the secondary electrons are also emitted from a specimen when the backscattered electrons spring out of the specimen. So, an expected resolution may not be obtainable depending on specimen. For measures to evade this, refer to 4-3) and 4-11). The secondary electrons emitted from the electron beam irradiation point, those emitted along with the springing-out process of the backscattered electrons and those emitted due to the backscattered electrons' collision with parts other than specimen are called "SE1," "SE2" and "SE3" for their mutual distinction, of which SE2 and SE3 carry information about the backscattered electrons.

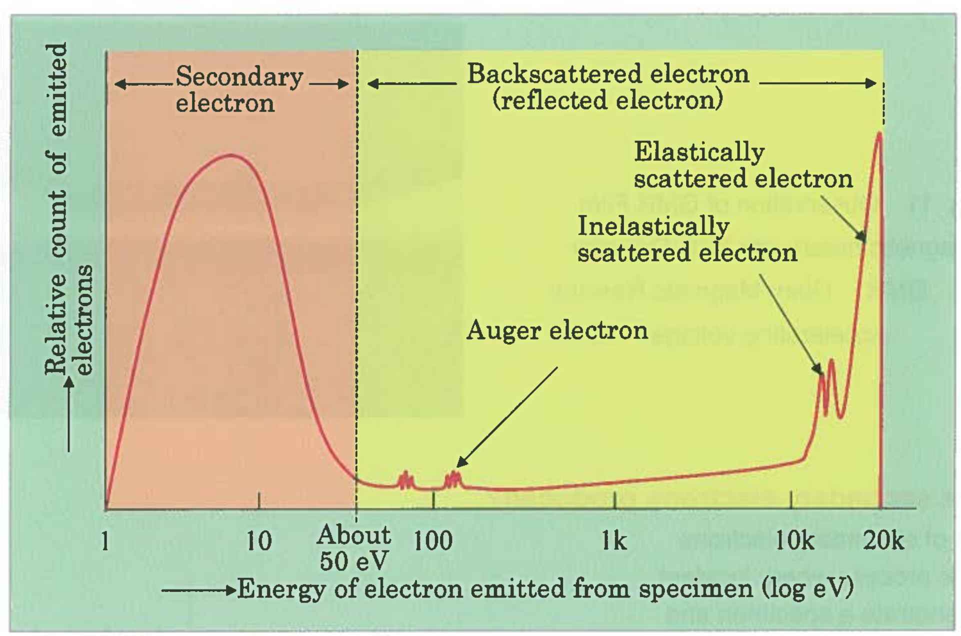


Fig. 13 Energy Distribution of Electrons Emitted from Specimen due to Beam Irradiation

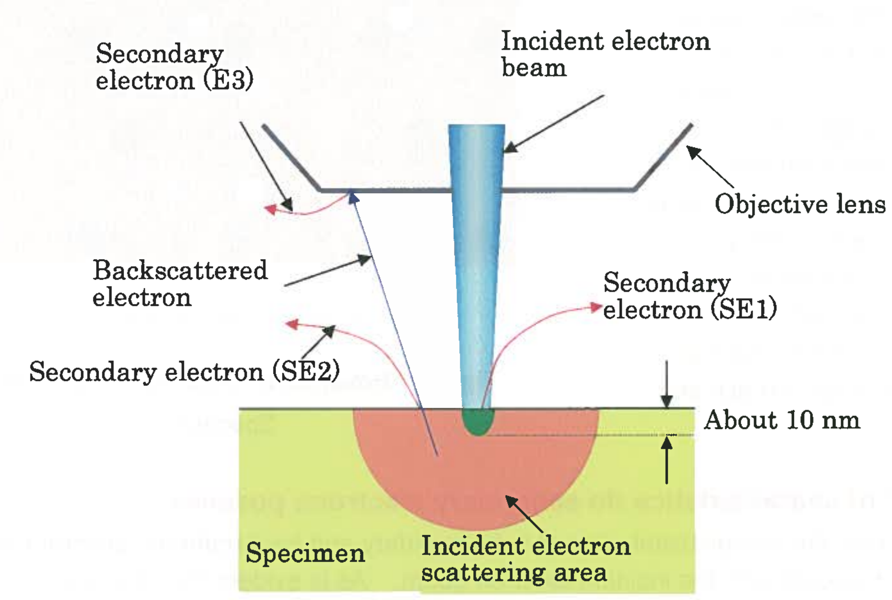


Fig. 14 Area of Secondary Electron Emission from Specimen Surface

The ratio of the number of emitted secondary electrons (N_{SE}) to the number of incident electrons (N_{PE}) is called secondary electron emission yield δ and expressed by the following equation.

$$\delta = N_{SE}/N_{PE}$$

Generally, δ varies substantially with the material and structure of a specimen, incident electron energy (accelerating voltage) and the incident angle of incident electrons. Figure 15 traces the change of δ at different incident electron energy levels for some materials. Although the profile of δ is not so different among the specimens, it is understandable that the maximum value δ_m and the

corresponding energy E_{pm} are substantially dependent on specimen. The δ_m and E_{pm} values of the main specimens are indicated in this figure.

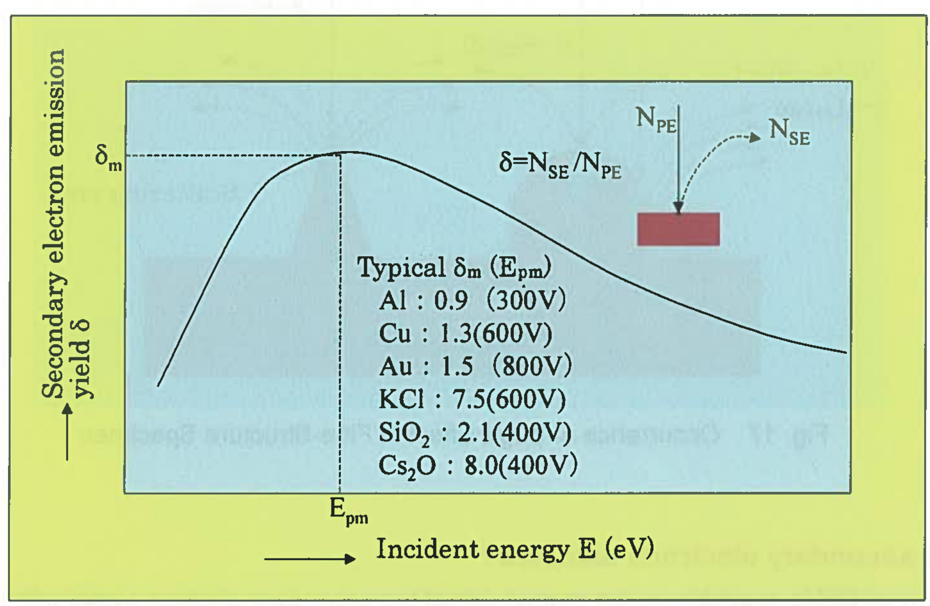


Fig. 15 Dependency of Secondary Electron Emission Yield on Specimen (excerpt from Dr. Yoshiyuki Takeishi's Experimental Physics Course)

It is also known that δ varies with the incident angle θ of electron beam as shown in Fig. 16. Thanks to this characteristic, the 3-dimensional information that reflects the fine topography of a specimen is obtainable with the secondary electron image. Also, there is a characteristic that δ becomes large at the edge of a specimen. On a secondary electron image, there are cases where white shining of the edge called "edge effect" is observable. It is thought that this phenomenon is attributable to a relative rise in emission yield at the edge which is caused by widening of the secondary electron emission area as shown in Fig. 17.

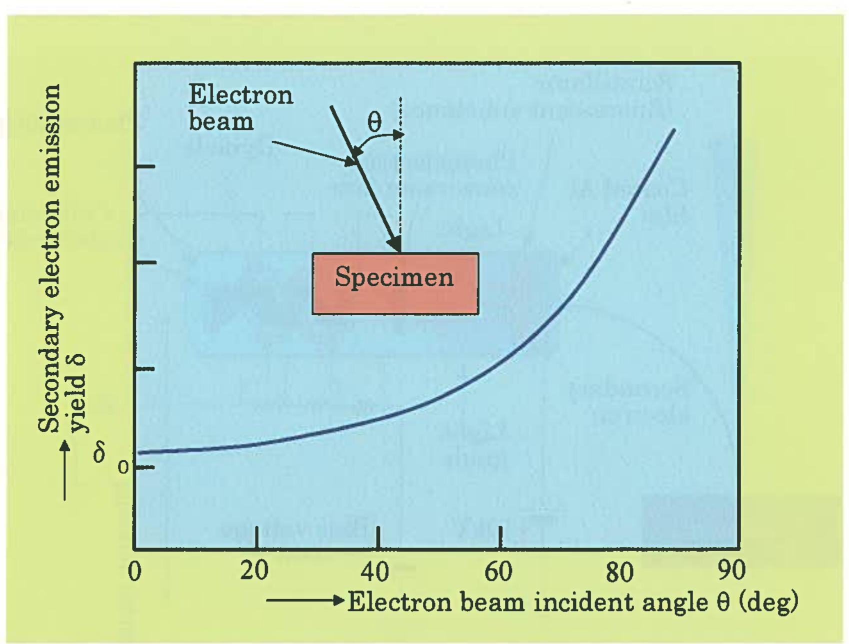


Fig. 16 Change in Secondary Electron Emission Yield at Different Electron Beam Incident Angles

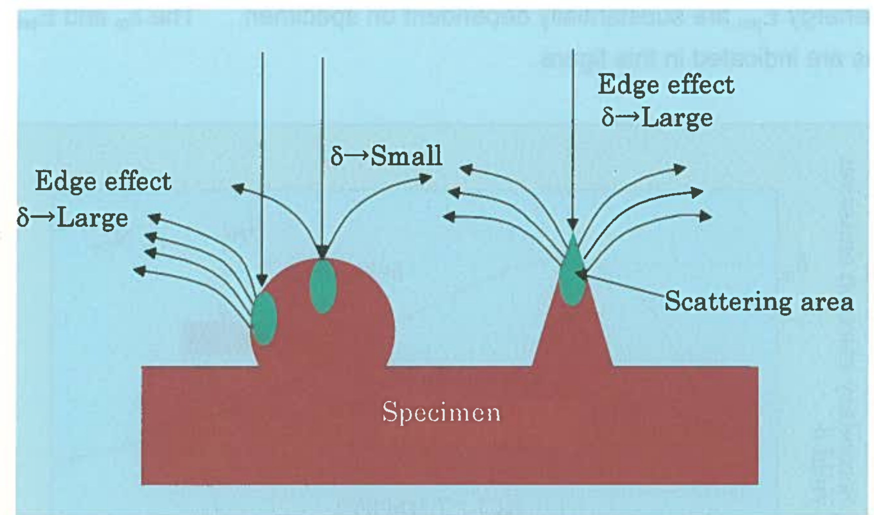


Fig. 17 Occurrence of Edge Effect in Fine-Structure Specimen

9) How are secondary electrons detected?

The majority of SEMs available on the market detect the secondary electron signal with the ET detector invented by Everhart and Thornley in 1960. With this detector, the secondary electrons are accelerated towards the 10 kV electric field and hit against the scintillator for conversion into the optical signals, which are led to the photomultiplier through the light guide and reconverted into electrons on the photoelectric conversion face as shown in Fig. 18. These electrons are accelerated with the electric field and hit against the first dynode (coated with a substance having a high secondary electron emission yield δ (3-8)) larger than 5 to produce a number of secondary electrons. These electrons are then led to the next dynode to produce a larger number of secondary electrons. Thus, the number of secondary electrons is multiplied sequentially and finally they are taken out as a signal current. This ET detector has features such as a long service life, high S/N ratio and fast response speed.

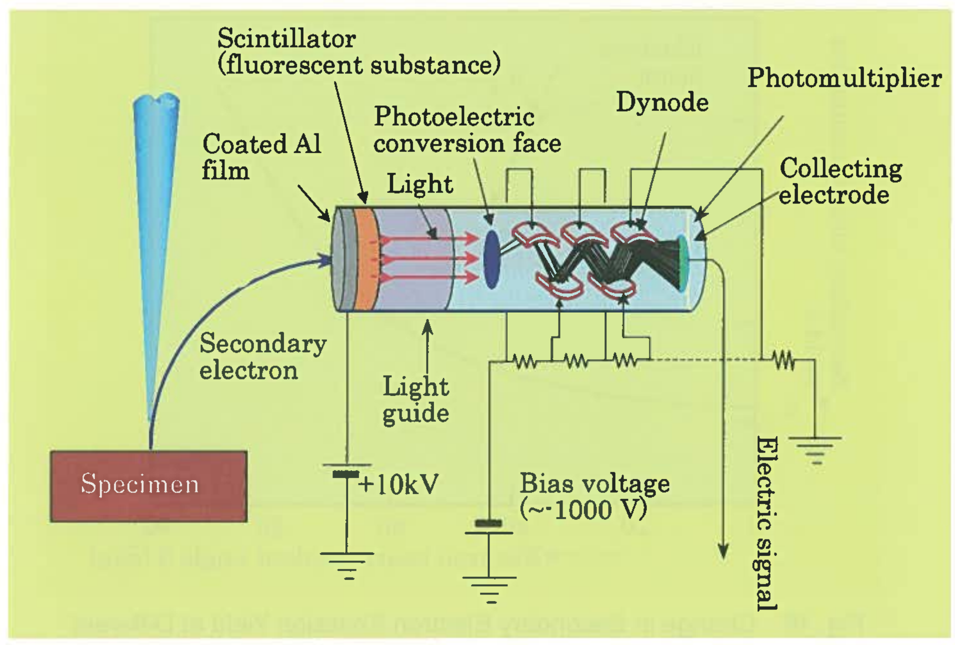


Fig. 18 Structure and Operation of ET Secondary Electron Detector

10) What are secondary electrons used for?

Because a secondary electron image gives the information which reflects minor topographic features on specimen surface, it is applied in extensive field. A SEM image generally refers to a secondary electron image. This image is indispensable particularly for high resolution observation, based on the information source of secondary electron is shallow and small.

Figure 19 shows a secondary electron image of catalytic specimen in which Pt particles of a few nm or less on the C carrier is clearly visualized. Heavy metal particles, etc. on a substrate of a comparatively low density are less affected by SE2 and SE3, high resolution observation is allowed using a comparatively high accelerating voltage like this example.

Fig. 20 shows an example of toner specimen surface. A specimen consisting of only the substances having such a low density as this specimen is generally observed using a low accelerating voltage (4-3)) or after metal coating on the specimen surface (4-9), 4-10)) in order to reduce electron scattering inside the specimen. However, metal coating involves a risk that nanometer-order microscopic information may be lost. Therefore, a coated film needs to be as thin as possible (smaller than a few nm). Although an imaging example with a low accelerating voltage of 100 V is posted here, it has been reported that the distribution of the dispersed substances of 10 to 30 nm on the toner surface could be imaged satisfactorily at accelerating voltage 500 V.

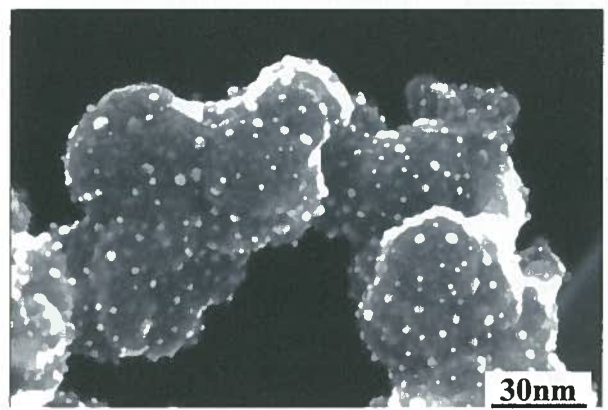


Fig. 19 Imaging Example of Catalyst (Pt/C)
Accelerating voltage: 30 kV

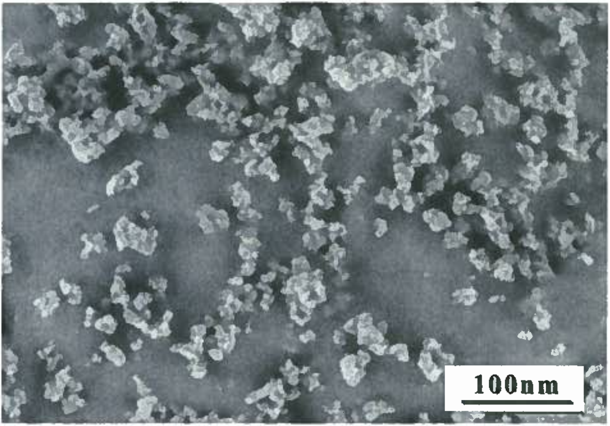


Fig. 20 Example of Toner Surface Imaging with Low Accelerating Voltage
Accelerating voltage: 100 V

4. Viewing conditions for acquiring good SEM images

1) What is a good SEM image?

As requirements for a "good SEM image," the following can be pointed out in general.

<1> Necessary information as given below is clearly expressed.

Topographic information, compositional information, crystal information, etc.

<2> Wrong information as given below is not contained.

Various artifacts, damage, external disturbance, etc.

<3> Gradation and brightness level are appropriate.

2) What effect does accelerating voltage or condenser lens current have on image quality?

Changing the accelerating voltage alters the incident electron energy to a specimen. This entails changes of lens aberration (1-6)), area of electron scattering in the specimen (3-1)), secondary electron emission yield (3-8)), etc. In consequence, various image quality changes as shown in Fig. 1 are brought about. Therefore, accelerating voltage is one of the important operating parameters for obtaining a good SEM image. The image quality changes listed in Fig. 1 depend largely on the substance and shape of a specimen. So you should see this figure for reference only.

Change to a different condenser excitation current alters the focal length (1-5)) of this lens. This entails changes of probe current and probe diameter (electron beam diameter). In consequence, the current conditions of image resolution, S/N ratio, charge-up (4-7)) and specimen damage will change. In the case of an FE-SEM (1-3)), the probe diameter can be said to be less affected by change of the condenser lens current than a thermionic emission SEM (1-3)), because the electron source diameter of FE-SEM is smaller.

Relationship between Accelerating Voltage and Image Quality

Items related to image quality	Accelerating voltage: Low	→ High
Image information (depends largely on specimen)	Surface information	Internal information
Image resolution (depends largely on specimen)	Low	High
Specimen damage (depends largely on specimen)	Slight	Serious
Contamination (depends largely on specimen)	Easy to see, not easy to stick	Not easy to see, apt to occur
Charge-up (depends largely on specimen)	Little	Much
Contrast	High	Low
Emission of characteristic x-ray	Two or threefold level of x-ray excitation energy	

Fig. 1 Relationship between Accelerating Voltage Change and Image Quality in SEM

Change of Image Quality when Varying Condenser Lens Current

Affected element and influence over image	Condenser lens current: Small	→ Large
Probe current	Large	Small
Electron beam diameter (FE-SEM less affected)	Large	Small
Image S/N	Better	Worse
Charge-up (depends largely on specimen)	Much	Little
Specimen damage (depends largely on specimen)	Serious	Slight

Fig. 2 Change of Image Quality due to Condenser Current Adjustment

3) How must accelerating voltage be selected according to the sample?

Contrast of a secondary electron image is dependent significantly on the shape, density, etc. of a specimen. The structures of bulk specimens for SEM are roughly classifiable into 6 categories shown in Fig. 3a) to f). For each category, an accelerating voltage which can be thought to be appropriate in general is described. However, attention should be paid because an appropriate accelerating voltage differs depending on the object of observation (desired kind of information) even among the specimens of the same structure. Note that the low accelerating voltage, medium accelerating voltage and high accelerating voltage described here stand for 2 kV or lower, 3 to 15 kV and 16 kV or higher, respectively. In Fig. 3, δ and ρ denote secondary electron emission yield and specimen density, respectively. However, if charge-up (4-8) to 4-10)) occurs because of an electrically non-conductive specimen, viewing at a low accelerating voltage of 1 kV or less or a preliminary coating process (4-11) to 4-13)) is required regardless of specimen structure.

Fig. 3a) For viewing a high-density particle, etc. on low-density substrate

SEM is most-suited for this kind of specimen. Because the quantity of SE1 is larger than SE2 and SE3, high-resolution imaging is possible.

Particles or the like can be observed at the high accelerating voltage, though the fine structure on the surface of a substrate can
be better imaged at the low accelerating voltage.

Fig. 3b) For viewing a low-density particle, etc. on high-density substrate

SE1 and SE2 produced from the substrate including SE are more than those due to particles, etc. Therefore, the particles, etc. cause black contrast in general. The low accelerating voltage is suitable for viewing the particles, etc., but the surface structure of the substrate itself can be imaged at a wide range of accelerating voltages.

Fig. 3c) For viewing high-density particles, etc. just below the surface of low-density substrate

SE1 and SE2 emission is not so expectable. Depending on the information brought by SE3 at the medium/high accelerating voltage, the particles, etc. in the specimen can be viewed. However, for viewing the surface structure of the substrate itself, the low accelerating voltage is better.

Fig. 3d) For viewing a low-density particle, etc. just below the surface of high-density substrate

It is difficult to view the particles, etc. directly from the surface. Cross-sectional observation is required.

However, the surface topography of the substrate itself can be viewed at a wide range of accelerating voltages.

Fig. 3e) For viewing fine topography of low-density substrate surface directly without coating

Because the incident electrons are scattered widely inside a specimen, viewing at the low accelerating voltage is unavoidable.

Fig. 3f) For viewing the coated fine topographic features on the surface of low-density substrate

Since the information is mainly sent from a high-density coated film, viewing is allowed at a wide range of accelerating voltages.

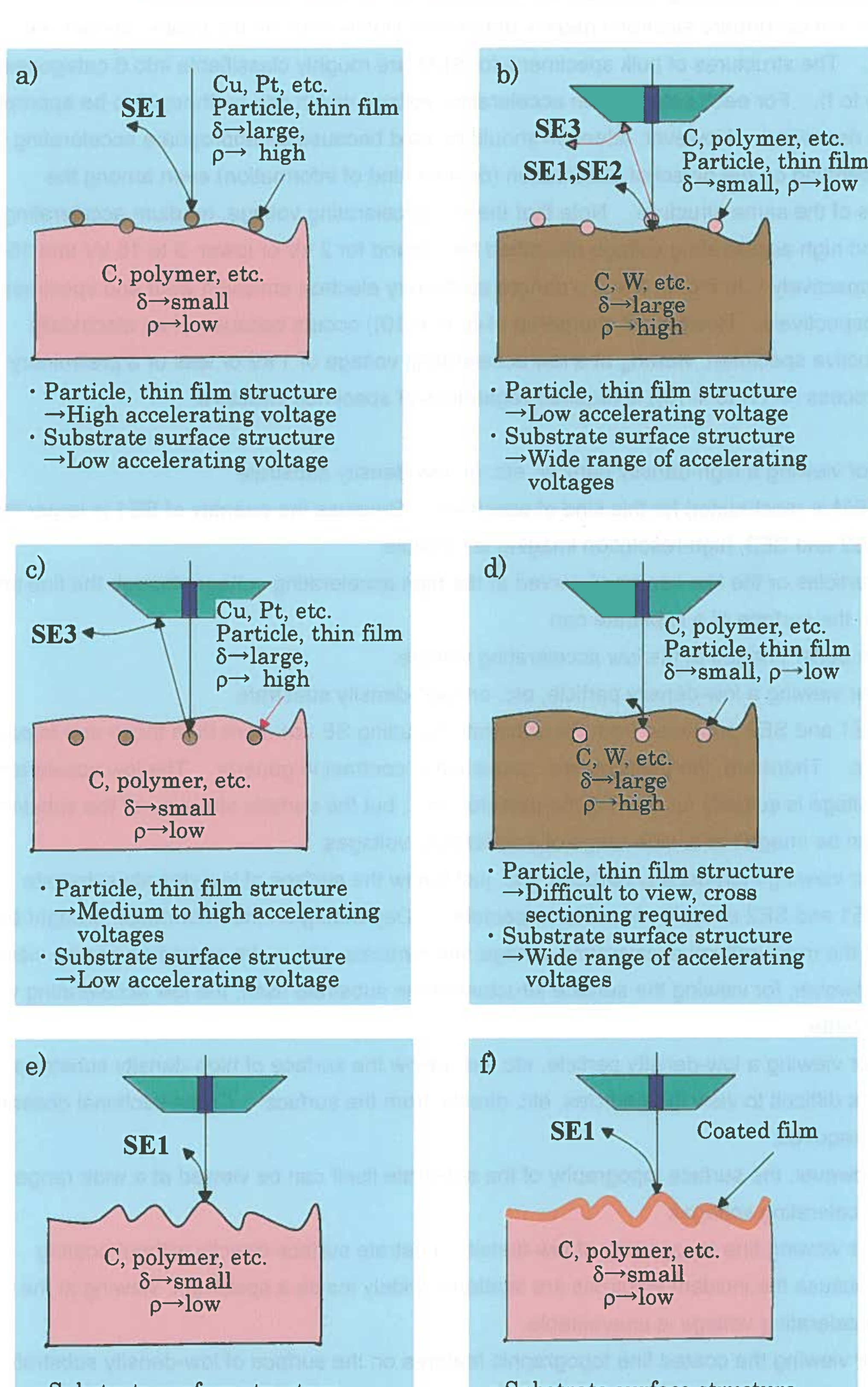


Fig. 3 Various Specimen Structures and Accelerating Voltage Setting Suitable for their Observation

4) What is the function of the objective lens aperture?

The objective lens aperture controls the divergence angle (1-5)) of electron beam and the number of electrons (probe current) passing through it, and affects the electron beam diameter (resolution), focal depth (4-7)), image S/N ratio, etc. as shown in Fig. 4. For obtaining the minimum electron beam diameter, optimum aperture size needs to be selected in general. The optimum size varies with SEM. So reference should be made to the instruction manual of each SEM. For improving image S/N ratio by increasing the probe current, a larger aperture is required, and a smaller aperture is required for increasing the focal depth.

Objective Lens Aperture Bore Size's Influence over Image Quality

Variable factor and its influence over image	Aperture bore size: Small	⇨ Large
Beam diameter	Optimum value existing	
Probe current	Small	Large
Focal depth	Deep	Shallow
Image S/N ratio	Low	High

Fig. 4 Functions of Objective Lens and its Influence over Image Quality

5) How does working distance (WD) affect image quality?

Changing the working distance (WD, 1-7)) brings about changes in the focal length of objective lens and the divergence angle (1-5)) of electron beam. Extending the WD enlarges the diameter of electron beam, but increases the focal depth (4-7)). Also, when the WD is extended, the scanning area of SEM widens and magnification decreases. However, this does not cause any inconvenience in practice because a modern SEM automatically corrects magnification by detecting the objective lens current. On the other hand, the influence by floating magnetic field (AC 50/60 Hz, DC magnetic field, etc.)(see Chapter 4) becomes larger as the WD is extended and image troubles may arise. Therefore, you should be careful about WD extension in high-magnification observation.

Working Distance's Influence over Image Quality

Variable factor and its influence on image	WD: Short	⇨ Long
Focal length of objective lens (when image focused)	Short	Long
Excitation strength of objective lens	Strong	Weak
Electron beam diameter	Small	Larger
Magnification	Increased	Reduced (Actually auto correction works)
Focal depth	Shallow	Deep
Influence over floating magnetic field	Small	Large

Fig. 5 WD Change's Influence over Image Quality

6) What is astigmatism and how is it corrected?

Astigmatism refers to the blurring of an image which occurs when the focal point fx of an electron passing through the X axis of a lens is different from the focal point fy of an electron passing through the Y axis. This is caused by an asymmetrical magnetic field due to misaligned electron optics, charge-up phenomenon due to contamination of the electron beam path, etc. as shown in Fig. 6. Therefore, when astigmatism has occurred, the cross-sectional shape of electron beam at fx is an ellipse long in the vertical direction because focus is attained in the X direction alone, and that at fy is an ellipse long in the horizontal direction. At the correct focal point fc (also called "least circle of confusion," 1-5)) between fx and fy, the cross section has a shape of a complete circle, but a fine electron beam is unobtainable because focus is attained at this point in neither X nor Y direction actually. Hence, astigmatism correction is required. For this purpose, each current of the X and Y astigmatism correction coils is adjusted so that fx and fy match fc. In actuality, astigmatism does not always match the X or Y axis, i.e., it occurs in a random direction, but astigmatism in a random direction is correctable by adjusting the vector quantity of the X/Y magnetic field in each correction coil.

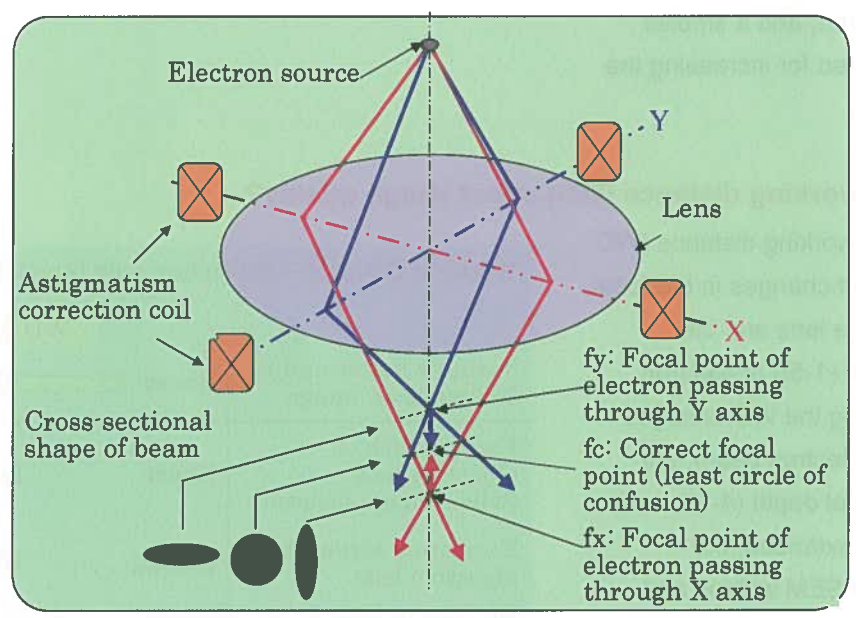


Fig. 6 Astigmatism and its Correction Method

Figures 7a) and b) show images at fx and fy under presence of astigmatism. Because the electron beam is elliptical, image stretching has occurred. Stretching directions at fx and fy are 90° different from each other. Photo c) shows an image at fc. Although image stretching did not appear, the entire image is blurred. So, the current of each astigmatism correction coil was adjusted so as to obtain the sharpest image possible. The result is shown in d). Now it appears that astigmatism correction has been completed. But, when magnification was increased, blurring becomes conspicuous again as shown in e). So, adjustment was repeated so as to make the image sharpest at fc, and the image in photo f) was obtained. By confirming the astigmatism correction at a magnification two or three times as high as the photographing magnification in the above-mentioned way, complete correction can be made.

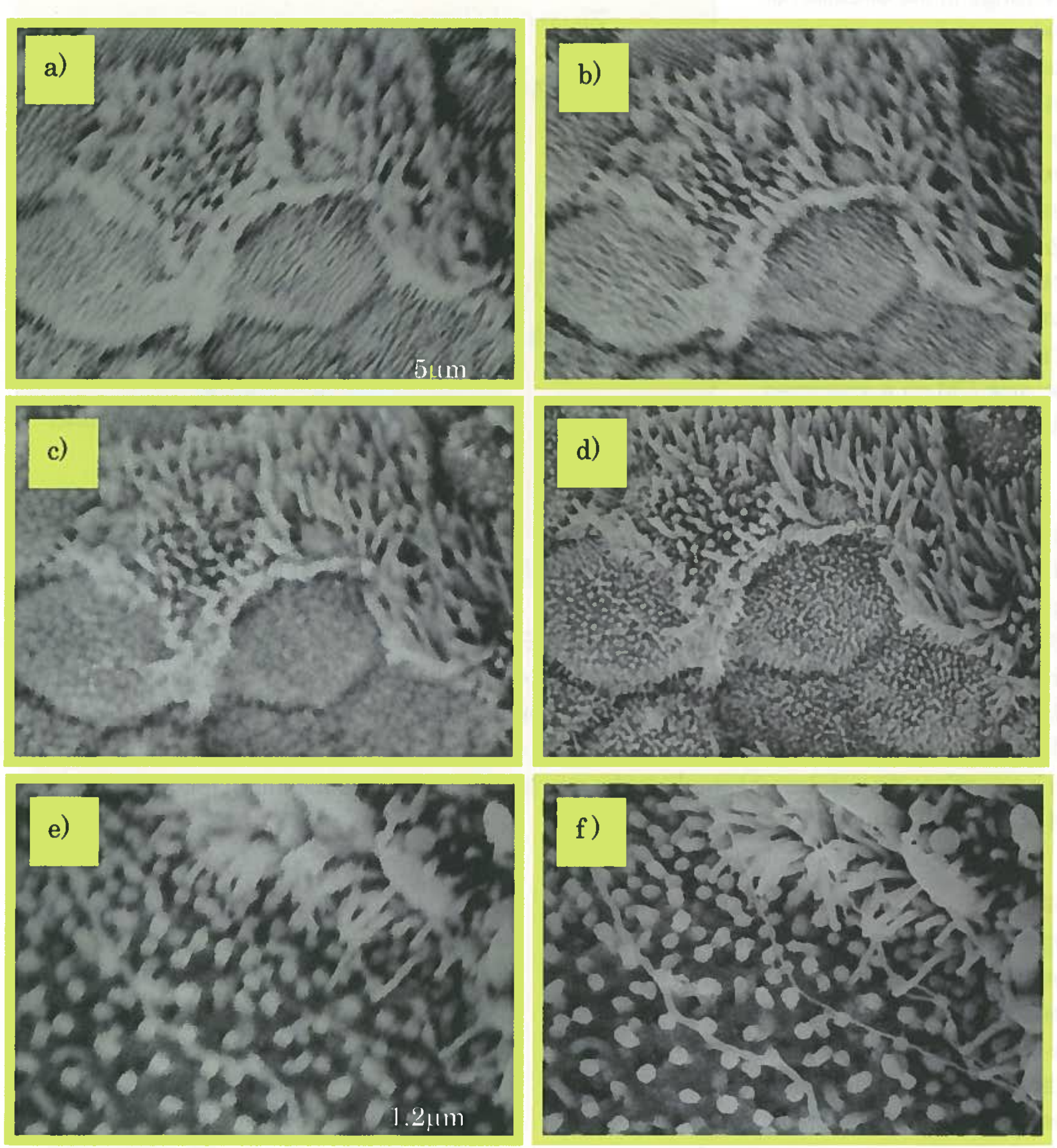


Fig. 7 Astigmatism Correcting Procedure (specimen: inner wall of rat's trachea)

a), b): Under presence of astigmatism, image stretching is observable at fx and fy, and its direction is 90° different between the two points.

c): At fc between fx and fy, the image is blurred, but stretching is not observable.

d): At fc, the astigmatism correction knob is manipulated so that the image becomes the sharpest. As a result, this image can be obtained.

e): When magnification is increased, blurring becomes visible again. This signifies that astigmatism correction is not completed.

f) Repeat astigmatism correction, and the sharp image will be formed.

7) What is focal depth?

Focal depth D refers to a distance range in the direction of height where an image appears to be focused even when the focal point is deviated due to the surface unevenness of the image as shown in Fig. 8. This depth is determined by the divergence angle 2α of electron beam and the minimum size recognizable on an image. In case of SEM, the minimum size corresponds to the pixel size δ/M on a specimen. Therefore, the following equation holds.

$$D = 2\delta/\alpha M$$

Where, δ and M stand for the pixel size on display (or photograph) and a magnification, respectively. This signifies that the focal depth of SEM is longer when the size of the objective lens aperture (4-4)) is smaller, the working distance (WD)(4-5)) is longer and magnification is lower. As magnification increases, the pixel size δ/M on a specimen becomes smaller. So, beyond a certain magnification (usually, a few ten thousand times), the electron beam spot overlaps neighboring pixels. In such a condition, the electron beam diameter can be thought as the minimum recognizable size instead of the pixel size δ/M.

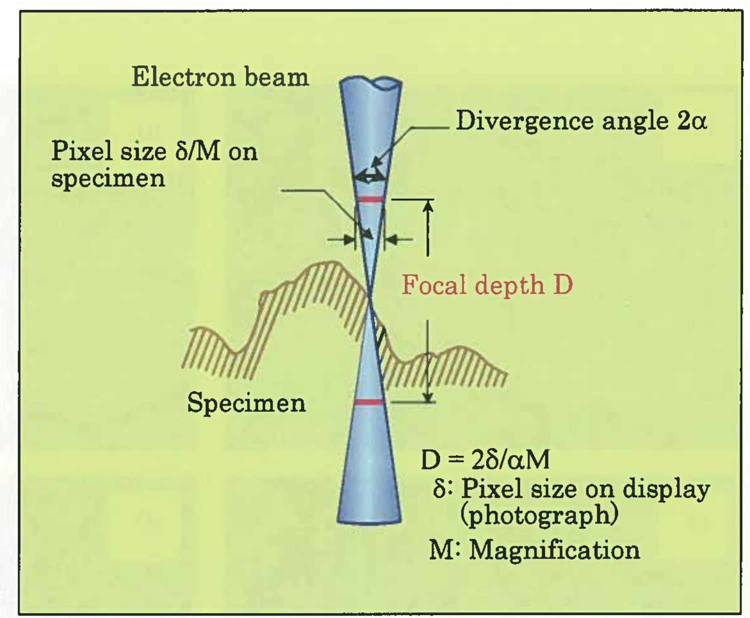


Fig. 8 Focal Depth in SEM

8) What is the charge-up phenomenon?

Charge-up is a phenomenon where the negative charge of the incident electron beam accumulates (charged up) on the surface of a non-conductive specimen and the potential in the beam-incident area changes to cause various image troubles. The charge-up phenomenon occurs when the total number of electrons emitted from a specimen, or backscattered electrons (I_{BSE}), absorbed electrons (I_{ab}) and secondary electrons (I_{SE}) is not equal to the number of incident electrons (I_p), as shown in Fig. 9.

The potential on specimen surface becomes negative when $I_p > I_{BSE} + I_{ab} + I_{SE}$.

And, it becomes positive when $I_p < I_{BSE} + I_{ab} + I_{SE}$.

In case of a non-conductive specimen, I_{ab} is almost zero and I_{BSE} is determined by the specimen substance. Therefore, the quantity of I_{SE}, namely secondary electron emission yield (3-8)) determines the charge-up situation.

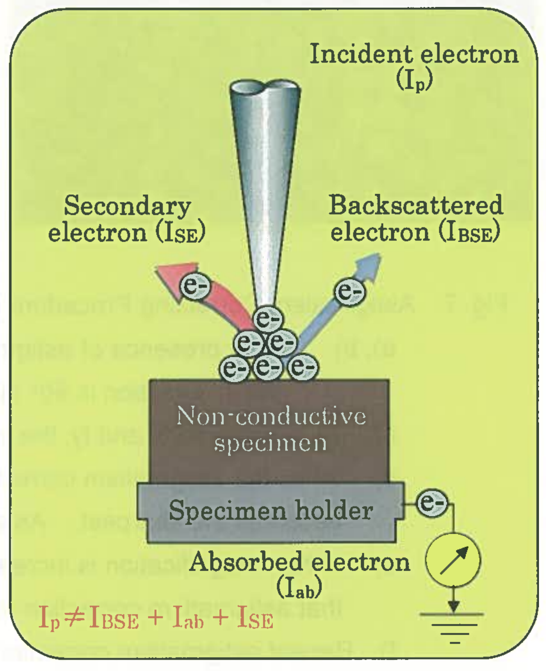


Fig. 9 Charge-up Phenomenon on Surface of Non-conductive Specimen

9) How is sample surface potential changed by charge-up?

If charge-up occurs, the potential on the specimen surface changes toward the positive or negative side until the secondary electron emission yield δ becomes 1 as shown in Fig. 10. Therefore, the level of specimen surface potential may be approximated by the difference between the actual incident electron energy and the incident electron energy where δ equals 1 on the observed specimen. However, the incident electron energy where δ equals 1 varies with specimen and imaging conditions. In the example shown in Fig. 10, it is probable that the potential changes about 900 V toward the positive side when the incident energy is 100 eV and close to 9 kV toward the negative side when the energy is 10 keV. However, the surface potential is not always constant in actual cases, because this potential may be significantly different locally due to a complicated specimen profile or charging/discharging may be repeated at certain intervals.

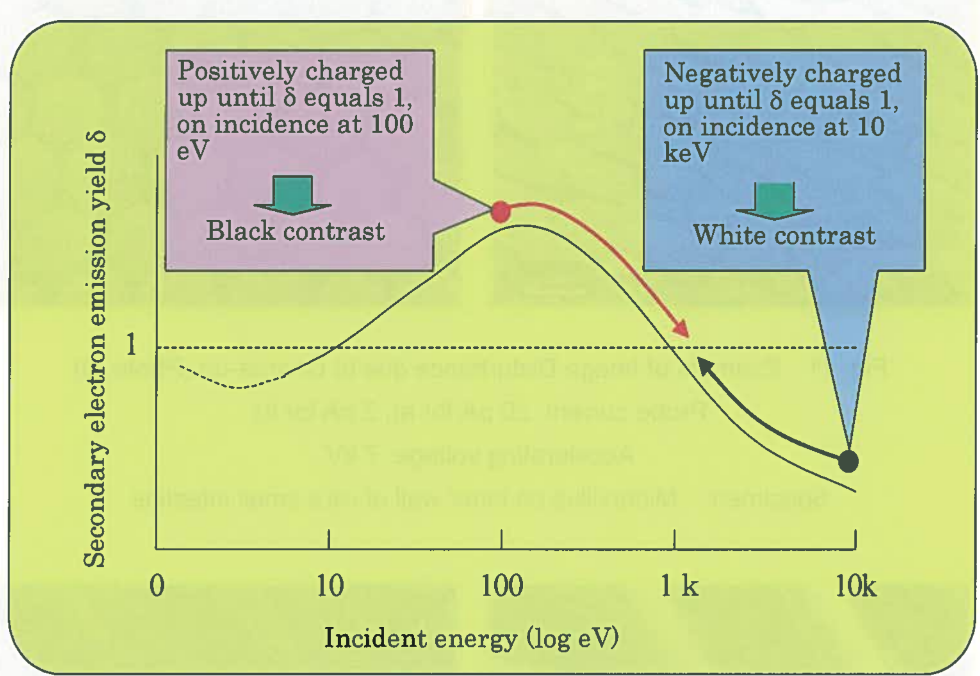


Fig. 10 Change in Specimen Surface Potential at Occurrence of Charge-up

10) How is the image disturbed by charge-up? And, how can the image disturbance be prevented?

Image troubles due to charge-up phenomenon can be sorted into two types; <1> image disturbance and <2> abnormal contrast. There are cases where both types <1> and <2> concur. Type <1> occurs as shown in Fig. 11a) because the irradiation position of incident electron beam changes, affected by an electric field caused by charge-up. This disturbance arises when the specimen surface is charged up at a comparatively high potential. For preventing this disturbance, reduction of the probe current as shown in Fig. 11b), lowering of the accelerating voltage, etc. are effective. Type <2> occurs as shown in Fig. 12a) because secondary electron emission is affected by the charge-up electric field. In case of a positive potential, the emitted secondary electrons are pulled back to the specimen side and cannot reach the detector, so a black zone appears. In case of a negative potential, a white zone appears. Preventive measure is basically the same as in <1>. In Fig. 12b),

abnormal contrast has almost been suppressed by reducing the accelerating voltage to 0.7 kV. Although some trial and error process is necessary because the secondary electron emission yield (3-8)) varies with specimen substance and imaging conditions, it is generally thought that the charge-up phenomenon can be suppressed substantially within an accelerating voltage range from 0.5 to 1 kV. However, if the charge-up phenomenon cannot be prevented by changing the probe current and accelerating voltage, then it is required to vacuum-coat a specimen (4-11)) or view it in a low vacuum (5-2)).

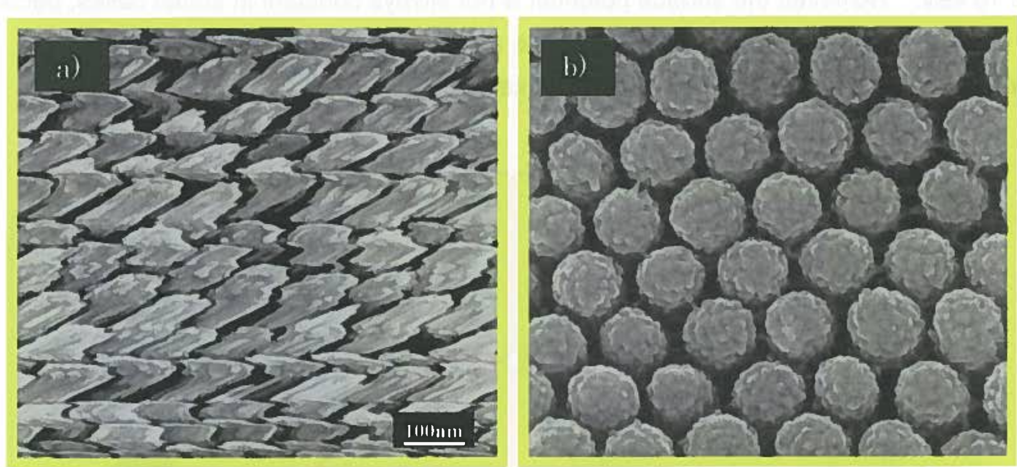


Fig. 11 Example of Image Disturbance due to Charge-up (Photo a))
Probe current: 20 pA for a), 2 pA for b)
Accelerating voltage: 7 kV
Specimen: Microvillus on inner wall of rat's small intestine

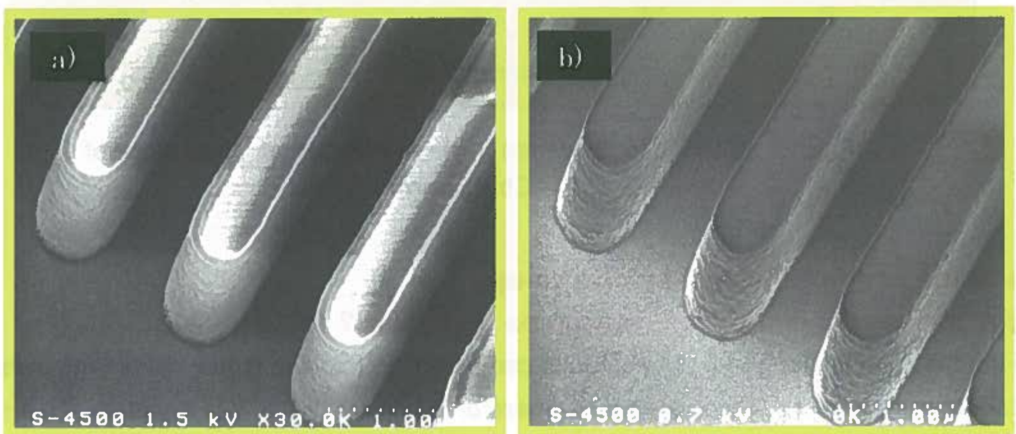


Fig. 12 Example of Abnormal Contrast due to Charge-up (Photo b))
Accelerating voltage: 1.5 kV for a), 0.7 kV for b)
Probe current: About 1 pA
Specimen: Resist pattern

11) What are the advantages and disadvantages of sample coating?

Sample coating is intended to prevent the charge-up phenomenon by allowing the charge on the specimen surface go to ground through the coated conductive film as shown in Fig. 13. Besides, specimen coating has advantages, for example, damage of the specimen under observation can be reduced because thermal conductivity is improved, and the quantity of secondary electron signal increases.

On the other hand, specimen coating has disadvantages, for example, the shape and size of a nanometer-order fine structure may change, and specimen information about elemental composition and surface potential may be lost. Therefore, it is required to select an appropriate coating method according to the object and purpose of SEM imaging or avoid coating in undesirable cases.

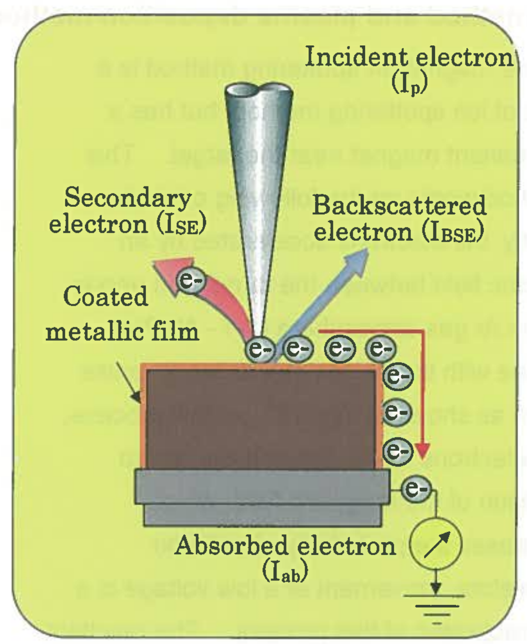


Fig. 13 Prevention of Charge-up by Specimen Coating

12) What sample coating methods are available and how do they differ?

The generally employed coating methods are listed in Table 1. Among the methods, those frequently used for high-resolution SE image observation are magnetron sputtering and plasma deposition methods. However, any method suffices for observation of low-magnification images alone, and the resistance heating method is used in carbon coating for x-ray analysis.

Table 1 SEM Specimen Coating Methods and their Characteristics

Method	Coating material	Graininess	Ease of operation	Spread	Damage	Supplement
Resistance heating	C, Pt-C, etc.	◎	×	×	×	Slightly poor in film thickness reproducibility (vacuum evaporation)
Ion beam	Cr, W, etc.	◎	△	×	△	High-energy (a few keV) ion beam used
Electron beam	C, Pt-C, etc.	◎	×	×	○	Less thermal damage, ultrahigh vacuum/extremely low temperature method applicable
Ion sputtering	Au-Pd, etc.	△	◎	○	×	Risk of thermal damage by glow discharge
Magnetron sputtering	Pt, Pt-Pd, etc.	○	◎	○	◎	Less specimen damage because of low discharge voltage
Plasma deposition	Os	○	○	○	○	OsO_4 gas ionized by plasma discharge

13) What is the principle of the magnetron sputtering method and plasma deposition method?

The magnetron sputtering method is a kind of ion sputtering method, but has a permanent magnet near the target. This method works on the following principle. Firstly, the electrons accelerated by an electric field between the target and anode in the Ar gas atmosphere (1-1 - 10 Pa) collide with the Ar gas molecules to ionize them as shown in Fig. 14. In this process, the electrons move dynamically due to addition of the magnetic field, which increases the probability of collision. Therefore, movement at a low voltage is a characteristic of this process. The resultant Ar ions are accelerated by the electric field and hit against the target (platinum Pt), and the Pt particles are ejected out of the target in a sputtering phenomenon. The particles are accumulated on the specimen surface to form a coating film. Since the magnetron sputtering method works at a low voltage, reduction of specimen damage and enhancement of film thickness controllability can be expected.

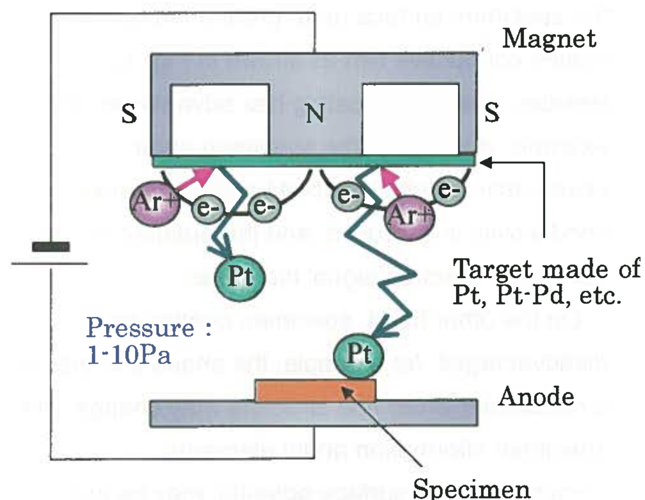


Fig. 14 Principle of Magnetron Sputtering Method

The osmium plasma deposition method is based on the following principle. Plasma discharge is started in the osmium oxide (OsO_4) gas atmosphere as shown in Fig. 15 to cause the chemical reactions given below, thereby sticking the metal osmium (Os) to the specimen surface.

<1> $OsO_4 \rightarrow Os^{4+} + 2O_2 + 4e^-$

<2> $OS^{4+} + 4e^- \rightarrow Os$

By this method, an amorphous and dense Os film is obtainable. Also, the electrode structure has been improved for reducing specimen damage, so discharge at a voltage as low as 500 V has been realized.

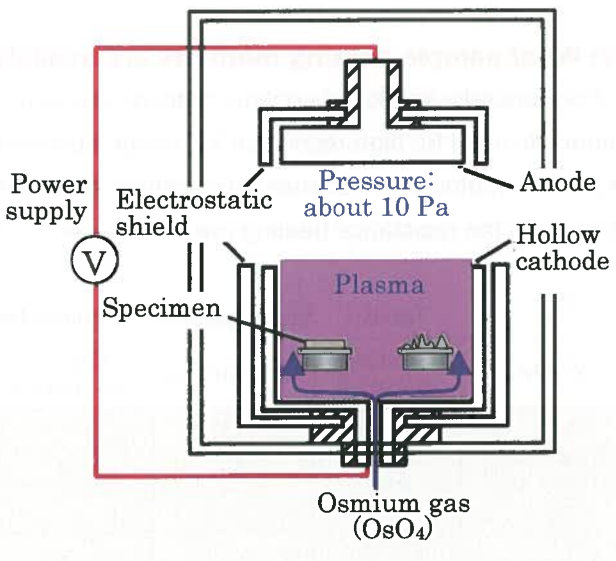


Fig. 15 Principle of Osmium Plasma Deposition Method

5. Principle and applications of low vacuum SEM

1) What kind of instrument is the low vacuum SEM?

Low vacuum SEM is a SEM which can be operated at a higher specimen chamber pressure (lower vacuum condition) than general SEMs. The specimen chamber pressure in a general SEM is kept within 10^{-3} to 10^{-4} Pa, while the pressure in a low vacuum SEM is kept at a much higher pressure, or a few to several hundred Pa. Operation at a high specimen chamber pressure enables electron microscopists to view a non-conductive specimen quickly in a nondestructive way omitting a step of specimen preparation and also suppress vaporization of moisture and oil in a specimen during observation. Note that almost all the commercial low vacuum SEMs are also usable as general high vacuum SEMs through changeover of the evacuation system.

2) Why is it that an insulating material can be observed without the charge-up phenomenon by means of the low vacuum SEM?

When a non-conductive specimen is viewed with a general SEM (high vacuum SEM), image trouble may occur due to charge-up phenomenon (4-8)) to disable normal viewing. For circumventing this, the specimen surface is usually coated with a thin film of metal (4-12), 4-13)). However, coating involves impairing of the original specimen characteristics, which may lead to a kind of destructive observation.

For such vulnerable specimens, a low vacuum SEM is effective. At a high specimen chamber pressure, the mean free path of an electron (2-1)) is short (see Table 1), and the primary electron, secondary electron and backscattered electron collide with the residual gas to ionize them as shown in Fig. 1. The positive ions resulting from the ionization move, attracted by the negative charge accumulated on the specimen surface and neutralize the charge. In this mechanism, a low vacuum SEM suppresses the charge-up phenomenon to allow direct observation of non-conductive specimens.

Table 1 Mean Free Path of Electron (in N_2 gas)

Specimen chamber pressure	Mean free path
10^{-3}Pa	About 40m
13Pa	About 3mm
270Pa	About 0.1mm

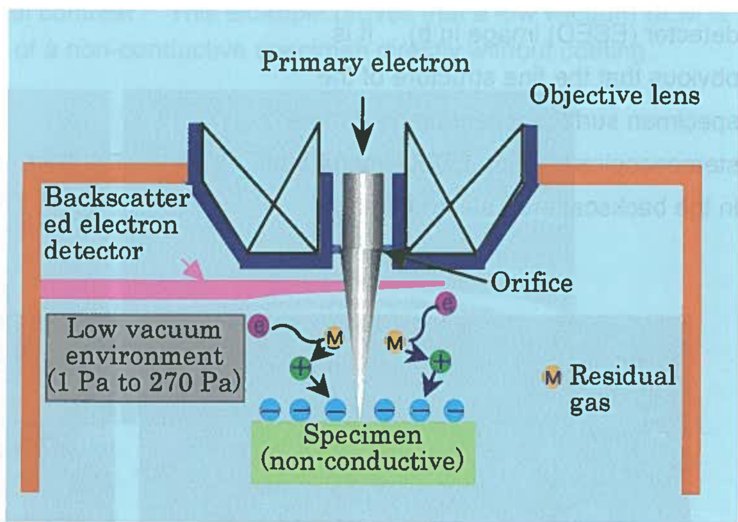


Fig. 1 Suppression of Charge-up Phenomenon in Low Vacuum Environment

6. Generation/detection of x-rays and elemental analysis method

1) How are x-rays produced?

When an incident electron ejects an orbital electron in a specimen composing atom in the process of its inelastic scattering (3-2)), the atom assumes an excited state (it is ionized). Then, an electron is brought from an outer electron orbit into the vacant shell immediately to assume the steady state again as shown in Fig. 1. This phenomenon is called a "transition." An excess energy produced in this process of electron transition is emitted as an x-ray. Because the difference in energy between two orbits takes on a value unique to the element, the energy of the emitted x-ray is also unique to the element. Hence, this x-ray is called a "characteristic x-ray," which is used for elemental analysis. For generating a specific characteristic x-ray, an electron having a larger energy than that of the characteristic x-ray needs to be projected. Note that the relationship between energy E (keV) and wavelength λ (nm) can be expressed by the following equation; $E = 123.96/\lambda$.

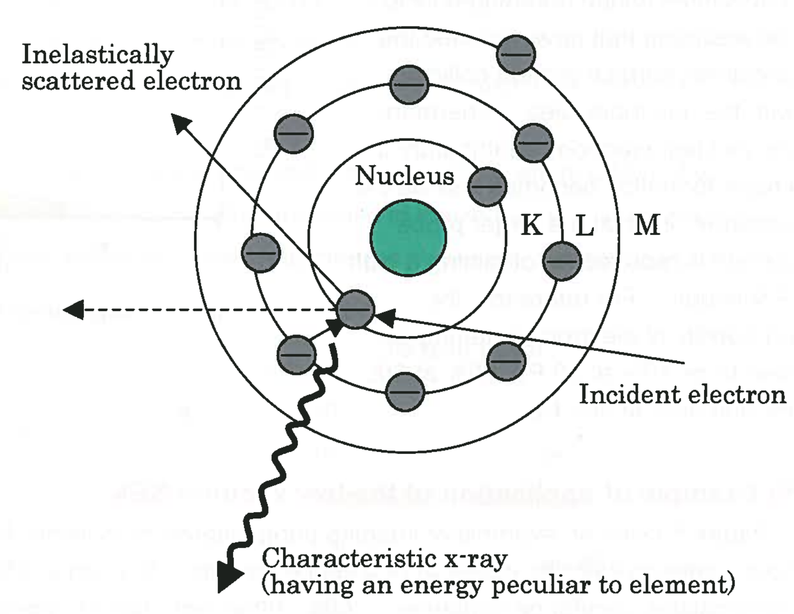


Fig. 1 Generation of Characteristic X-ray in Sodium (Na) Atom Model

2) What kinds of characteristic x-rays are there?

There are many characteristic x-rays due to a difference in the energy between the orbits where an electron transition (6-1)) occurs. Typical ones are shown in Fig. 2. For example, the characteristic x-rays generated upon transition from the L_{II} orbit to the K orbit and from M_{III} to K are called $K\alpha_1$ line and $K\beta_1$ line. And, a pair of $K\alpha_1$ and α_2 lines and that of $K\beta_1$ and β_2 lines may be collectively called "$K\alpha$ lines and $K\beta$ lines," respectively or a pair of α and β lines may be called "K, L or M lines." Among the characteristic x-rays of each element, the K lines have the largest energy followed by the L lines, and M lines in this order.

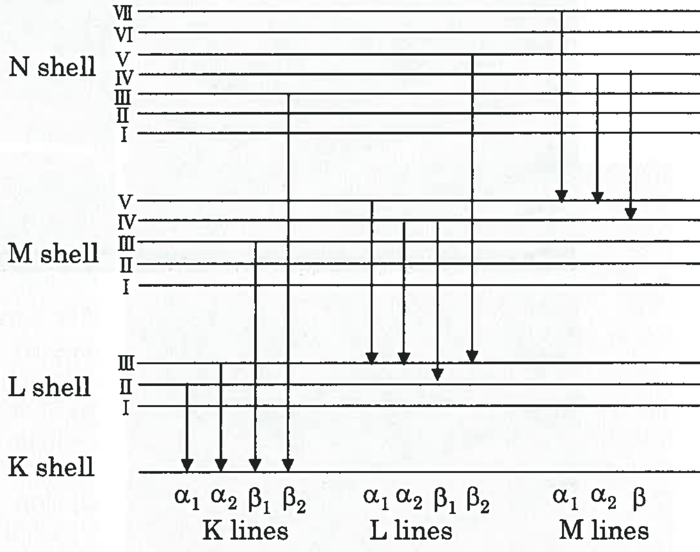


Fig. 2 Typical Characteristic X-rays and their Names

3) Are there any x-rays other than the characteristic ones produced by electron beam irradiation?

Figure 3 shows the characteristic x-ray spectrum of a stainless steel specimen which was measured with an energy-dispersive x-ray spectrometer (6-5)). Characteristic x-rays are displayed as small-width peaks, and when the central energy of each peak can be determined, the element contained in the specimen can be known. On the other hand, this spectrum contains the background just resembling the back of a whale, which is called a "continuous x-ray or white x-ray." The continuous x-ray is the energy emitted as an x-ray when the electron incident on a specimen is bent on its trajectory and decelerated by the electrostatic field of a nucleus. This x-ray does not have a value unique to an element. Therefore, the x-ray intensity after excluding this background is employed for quantitative analysis (6-12)).

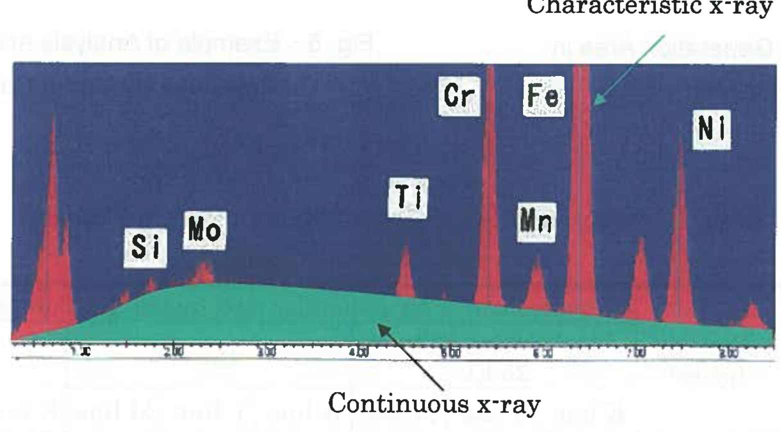


Fig. 3 X-ray Spectrum of Stainless Steel Specimen with Energy-dispersive X-ray Spectrometer

4) What is the extent of the area (spread, depth) in which characteristic x-rays are produced?

X-ray analysis with a SEM is a micro-area analysis for collecting the information in a microscopic x-ray generation area, so it is very important to know the area (spread, depth). It can be thought that the x-ray generation area lies down to a level at which the incident electron energy reaches the critical excitation energy capable of exciting the characteristic x-ray (primarily excited x-ray generation area) as shown in Fig. 4. Therefore, the generation area of K line (L line) is smaller than that of L line (M line). This relationship can be modeled comparatively easily by Monte Carlo simulation. An example of this simulation is shown in Fig. 5. However, this example is based on an infinitesimal electron beam diameter. Since a spread due to the electron beam diameter is superimposed actually, the x-ray generation area may be larger than in Fig. 5. Furthermore, when two or more elements coexist, the fluorescence excitation area, where a higher-energy characteristic x-ray excites the characteristic x-rays of other element, will be superimposed. For convenience, Table 1 lists the x-ray generation areas of representative elements at accelerating voltages of 25, 15 and 5 kV. If the generation area (magnification) is changed by scanning the electron beam, its X/Y plane is freely settable, but its depth is as shown in Fig. 4.

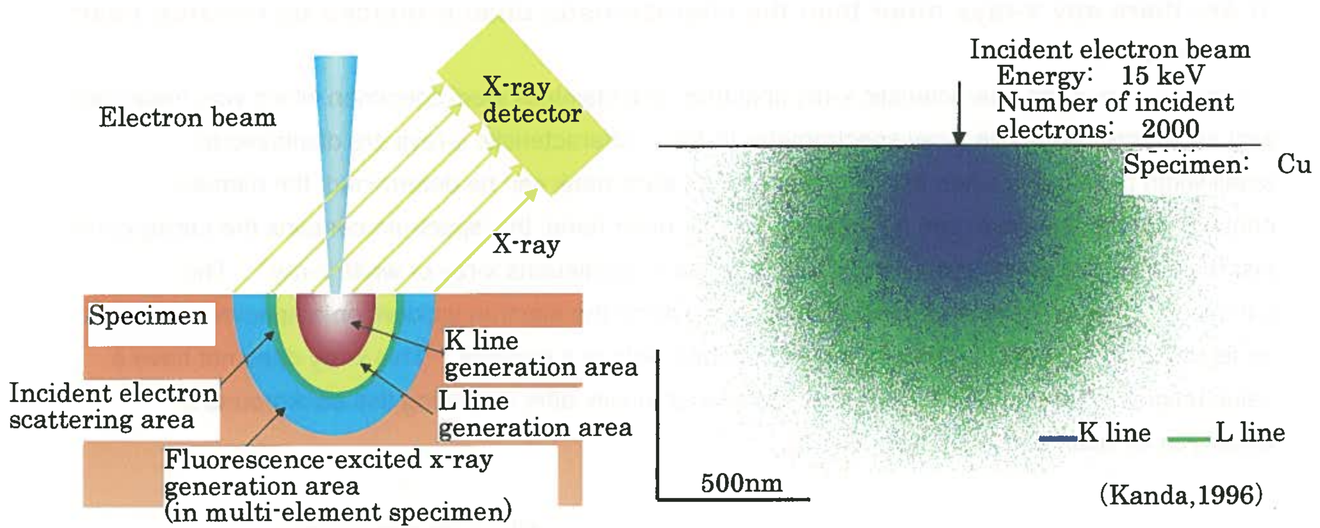


Fig. 4 X-ray Generation Area in Specimen

Fig. 5 Example of Analysis Area Presumption for Cu Specimen by Monte Carlo Simulation

Table 1 Approximate X-ray Generation Area in Main Elements

Element	Atomic number	Density (g/cm³)	Approximate x-ray generation area (radius μm, but nm in shaded cells) Accelerating voltage 25 kV			15kV			5kV		
			K line	L line	M line	K line	L line	M line	K line	L line	M line
C(graphite)	6	2.22	5.99			2.52			423		
Mg	12	1.74	7.62	7.62		3.29	3.29		475	533	
Al	13	2.70	4.90	4.90		2.11	2.11		287	288	
Si	14	2.33	5.63	5.63		2.31	2.43		352	458	
S	16	2.07	6.73	6.89		2.57	2.93		326	453	
Ca	20	1.55	8.01	8.33		3.23	3.45		205	545	
Ti	22	4.54	2.52	2.73		1.07	1.16		23.0	180	
Cr	24	7.19	1.56	1.71		635	717			110	
Fe	26	7.87	1.41	1.51		532	638			98.2	
Cu	29	8.96	1.18	1.26		406	547			77.9	
Ge	32	5.36	2.00	2.11		575	883			133	
Mo	42	10.2	548	884	1.02		413	433		56.6	70.2
Pd	46	12.0	138	748	854		182	364		45.6	56.7
Sn	50	7.30		1.19	1.41		540	580		46.2	93.3
Ta	73	16.6		458	529		182	236			36.5
W	74	19.3		443	529		160	197			32.7
Pt	78	21.4		352	454		122	175			27.6

5) What kind of x-ray detector is used in the SEM?

For analyzing the elemental composition through detection of the characteristic x-rays (6-1)) generated by irradiating the specimen surface with an electron beam, an Energy Dispersive X-ray Spectrometer (EDX or EDS) and a Wavelength Dispersive X-ray Spectrometer (WDX or WDS) are available. The former is configured by a combination of Solid State Detector (SSD)(6-6)) and multi-channel pulse height analyzer (6-7)), and the latter by a combination of wavelength dispersion mechanism using a curved crystal and proportional counter tube (6-8), 6-9)). Each type of spectrometer has advantages/drawbacks and are selectively used according to analytical purpose and object. A general purpose SEM equipped with EDX/WDX or EDX/WDX system equipped with an exclusive electron optics are generically termed EPMA (Electron Probe Micro Analyzer) or XMA (X-Ray Micro Analyzer).

6) Principle and instrument configuration for EDX.

An SSD generally used for EDX is a Si(Li) detector in which Li+ ions are diffused in a p-type single crystal Si to neutralize the acceptor (p type impurity, B^-, etc.), thereby forming an i layer (intrinsic layer) without an electric charge as illustrated in Fig. 6. When an x-ray is incident on the i layer, electron-hole pairs are produced in proportion to the x-ray energy and they are moved to the respective electrodes by the reverse bias field, so a signal current flows. The energy for obtaining a pair of electron and holes in a Si(Li) detection device (which is called "ionization energy") is about 3.9 eV. Therefore, electron-hole pairs are produced as given by dividing the incident x-ray energy with 3.9, and a pulse current having a height corresponding to the number of those pairs is taken out. Also, since the i layer is formed in a thickness of several millimeters, characteristic x-rays having a wide energy range from a few hundred eV to a few ten keV can be detected. The signal current is amplified with the preamplifier, its pulse waveform is shaped with the proportional amplifier and sent to the multi-channel pulse height analyzer (6-7)). In usual operating status, the Si(Li) detector and preamplifier are cooled with liquid nitrogen.

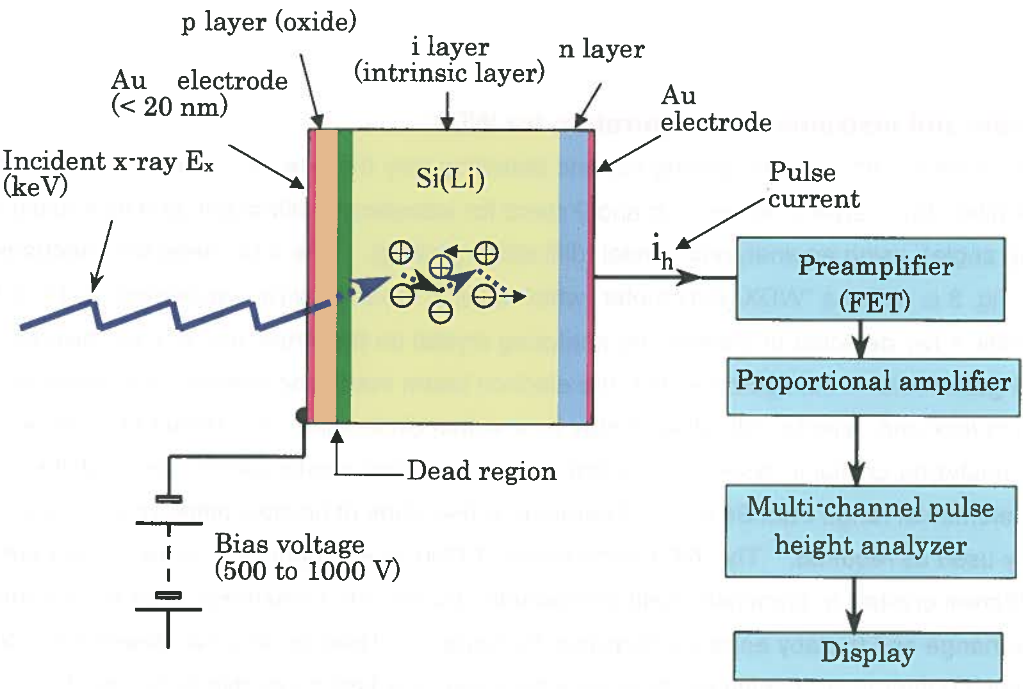


Fig. 6 Operating Principle of Si(Li) Detector for EDX

7) What is the multichannel pulse height analyzer?

A multichannel pulse height analyzer is used for detecting the pulse heights of a signal pulse current with the multi-channel window and sort out the characteristic x-rays using the pulse heights as diagrammed in Fig. 7. The pulse height of the signal current is proportional to x-ray energy. Therefore, when one plots x-ray energy on the abscissa, and the number of x-rays (intensity) corresponding to the energy on the ordinate, a characteristic x-ray spectrum can be drawn. Elements are qualitatively analyzable by measuring the energy at the center of each peak in this spectrum.

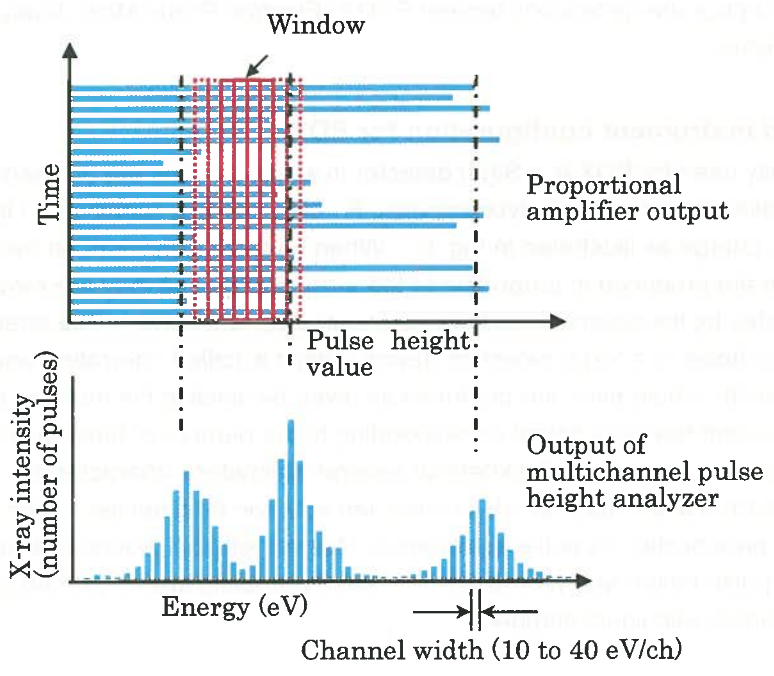


Fig. 7 Principle of Multichannel Pulse Height Analyzer

8) Principle and instrument configuration for WDX.

WDX is an instrument used for sorting out and detecting only the x-ray of a wavelength that satisfies Bragg condition ($n\lambda = 2d\sin\theta$, where λ, d and θ stand for wavelength, diffraction grating's ruling interval and Bragg angle), using an analyzing crystal (diffraction grating). The x-ray detection mechanism shown in Fig. 8 is called a "WDX goniometer" which selects the energy (or wavelength, 6-1)) of the characteristic x-ray detected by moving the analyzing crystal on the virtual line at x-ray take-off angle ϕ. The WDX goniometer is configured so that the electron beam irradiating position on a specimen, analyzing crystal and detector (slit) always stay on a virtual circle called the "Rowland circle" even when the analyzing crystal is moved. In practice, one analyzing crystal cannot detect all the x-rays within an elemental range from Be to U. Therefore, a few kinds of crystals different in "d" are selectively used as required. The WDX exclusive for EPMA is equipped with plural goniometers having different crystals to comprise multiple channels (usually 2 to 5 channels) in order to minimize crystal exchange and thereby enhance analytical throughput. Used as an x-ray detector for WDX is a tandem type counter (6-9)) configured by combining a gas flow type proportional counter for low-energy x-ray detection and a gas-filled type proportional counter for high-energy x-ray detection.

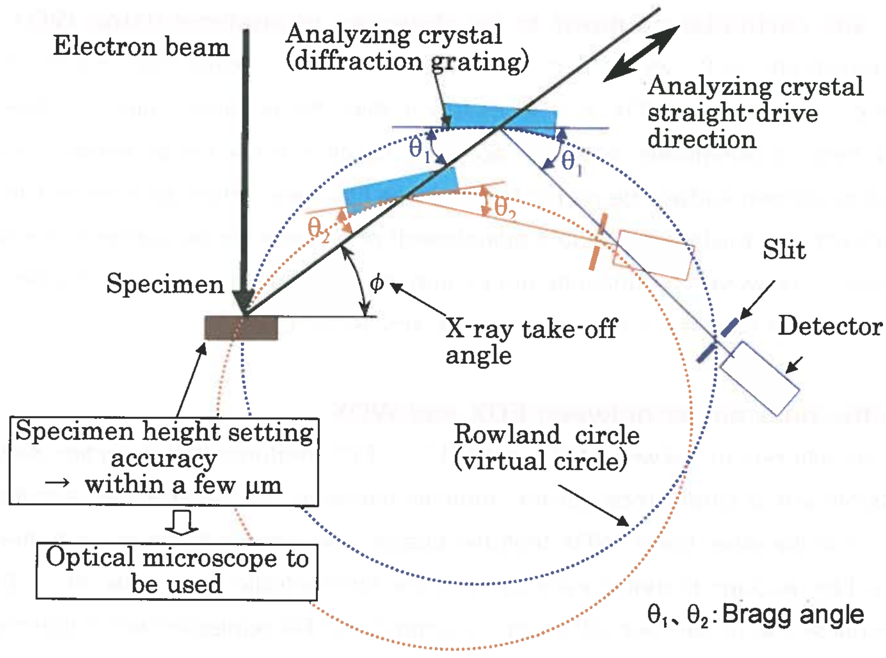


Fig. 8 Principle of WDX Goniometer

9) What is the tandem type proportional counter used in WDX?

The detector used for WDX is a tandem type proportional counter configured by adding a gas-filled type proportional counter for high energy after a gas flow type proportional counter for low-energy x-ray detection as illustrated in Fig. 9 so that x-rays in a wide range of energy can be detected. A low-energy x-ray easily passes through the thin polymer film (used for separation from specimen chamber vacuum) of the former counter and ionizes the Ar in the Pr gas (Proportional counter gas, mixed gas of Ar 90% + CH_4) always flowed as ionization gas, and the electrons produced in the process of ionization are collected with the electrode to obtain a signal current. On the other hand, a high-energy x-ray passes through the Be film at the inner location and ionizes the Xe molecules filled in the gas-filled type proportional counter, thereby obtaining a signal current in the same way as the gas flow type.

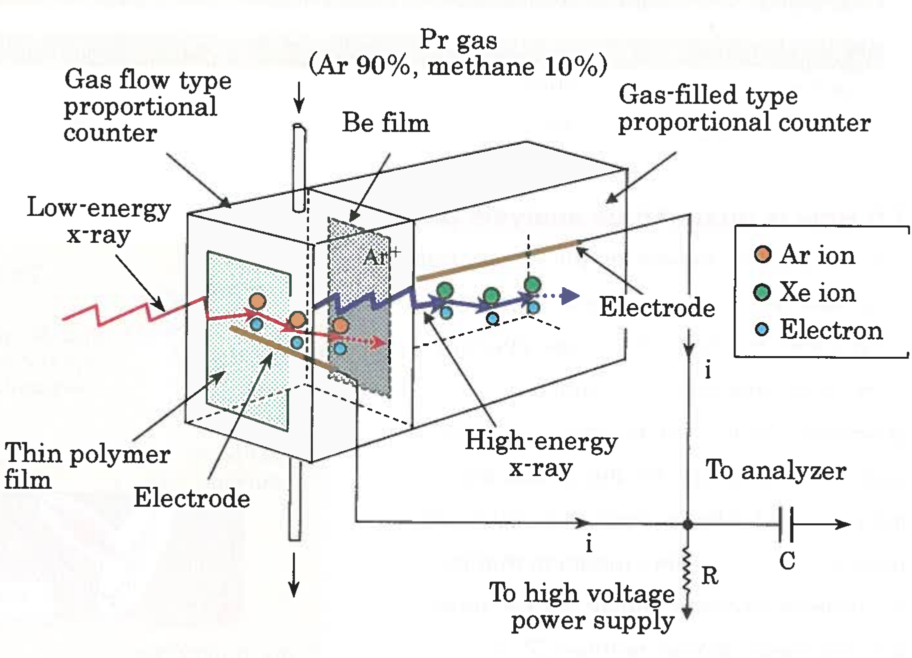


Fig. 9 Structure of Tandem Type Proportional Counter Employed for WDX

10) Are there any particular cautions to be observed in analysis using WDX?

WDX has a goniometer (6-8)) vertically mounted (vertical type) or horizontally mounted (horizontal type). For using a vertical type WDX, it is necessary to align the position (height) of specimen surface with the focus of goniometer within an accuracy range of a few μm as shown in Fig. 8. This requires that the specimen surface be mirror-finished to a flat plane within an error of a few μm (7-5)) and that the Z-directional positioning (height adjustment) of a specimen be carried out strictly with the optical microscope. However, a horizontal goniometer does not need such a strict specimen positioning and it can be handled in almost the same way as an EDX.

11) What are the differences between EDX and WDX?

Table 2 lists the differences between EDX and WDX. EDX features that detection sensitivity is high, analysis is possible with a small probe current, multiple elements can be analyzed simultaneously in a short time, etc. On the other hand, WDX features energy resolution a factor of lox better than EDX, detection limit is high enough to detect trace amount/low concentration elements, etc. EDX is mounted to almost all the SEMs, and WDX are mounted to SEMs equipped with a thermionic-emission electron gun (1-3)) and Schottky electron gun (1-3)).

Table 2 Differences in Characteristics between EDX and WDX

Item	EDX method	WDX method
Analysis system	Simultaneous analysis of multiple elements	Sequential analysis system (multi-channel simultaneous analysis possible)
Analyzable elements	$(^{5}B)^{6}C$ to ^{92}U	^{4}Be to ^{92}U
Energy resolution	125 to 150 eV	10 to 20 eV
Analysis time	Fast (30 to 400 sec)	Slow (20 minutes or longer)
Probe current	10^{-9} to 10^{-11} A	10^{-7} to 10^{-9} A
Detection sensitivity	High (about 100 times the WDX's)	Low
Detection limit	Low (down to 0.3%)	High (down to 0.01%)
Specimen damage	Slight	Heavy
Mounting space	Small	Large

12) How is quantitative analysis performed?

It is difficult to determine the concentration C_A of element A in an unknown specimen simply from the ratio of its x-ray intensity I_{AU} to the x-ray intensity I_{AS} of standard specimen (100% concentration), namely I_{AU}/I_{AS}. The reason for this is that the following ZAF effects need to be taken into account; <1> "Z" effect meaning that the occurrence of characteristic x-rays varies with the mean atomic number (Z) of a specimen, <2> "A" effect, i.e., a

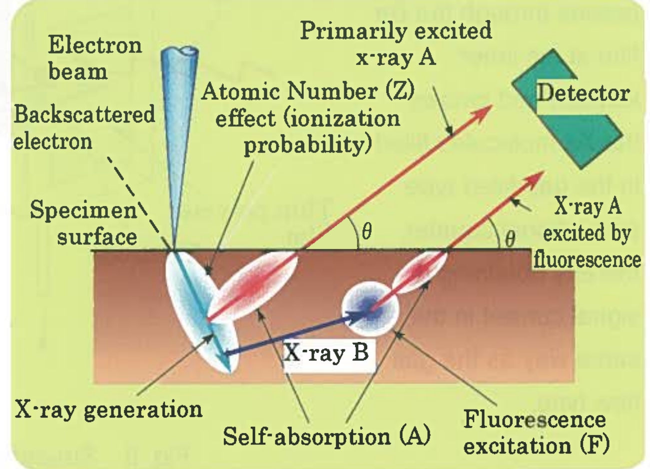


Fig. 10 ZAF Effects in X-ray Emission from Specimen

characteristic x-ray generated in the specimen receives a self-absorption by the specimen until it escapes into vacuum and <3> "F" effect, i.e., a certain characteristic x-ray is subjected to fluorescence excitation by other x-ray (see Fig. 10).

Therefore, the concentration C_A in element A in an unknown specimen is obtainable by the following equation, taking each correction of the Z, A and F effects into account.

$$C_A = I_{AU}/I_{AS} \cdot Z \cdot A \cdot F$$

An actual ZAF correcting calculation would become significantly complicated for a multiple-element specimen. So, a computer is generally used for this calculation. A program for this calculation is incorporated in all EDX and WDX systems. Therefore, analysts do not perform the ZAF correcting calculation directly by themselves. Rather, it can be thought that C_A accuracy is dependent substantially on the measurement accuracy of I_{AU}/I_{AS}. In the following section "7," key points for improving this measurement accuracy are covered. Also, the standardless quantitative analysis, in which a quantitative analysis is carried out by using a database on the x-ray intensities of standard specimens instead of using a standard specimen, has been put into practical use in the majority of instruments in this analytical category and effectively utilized for determining approximate concentrations.

7. How to improve accuracy in x-ray analysis

1) What should be observed in order to improve the accuracy in x-ray analysis?

For carrying out x-ray analysis with a high accuracy, the following requirements need to be met; <1> efficient emission of the desired characteristic x-ray, <2> exclusion of the x-rays generated in other than specimen (or target location) from detection, <3> restriction of x-ray generation area (its spread, depth) within the desired analysis area (spread/depth), <4> minimization of x-ray generation area, <5> flattening of specimen surface as far as possible, <6> absence of pseudo peaks in EDX, etc. Some of these requirements do not agree with each other, but appropriate conditions should be employed in response to each analytical purpose.

2) How can characteristic x-rays be efficiently produced?

The ratio of characteristic x-ray intensity I_{cha} to background (continuous x-ray 6-3)) intensity I_{con} has a relationship with the ratio of incident electron energy E_p to critical excitation energy (6-4)) E_{ex} as shown in Fig. 1. As is evident from this figure, it is effective to select an E_p/E_{ex} value within 2 to 3 for the most efficient generation of a characteristic x-ray. Although satisfying this condition simultaneously for multiple characteristic x-rays is difficult in actuality, it is important to select an accelerating voltage at which all the characteristic x-rays to be detected can be excited and the characteristic x-ray of particular interest can be excited most efficiently among all the x-rays.

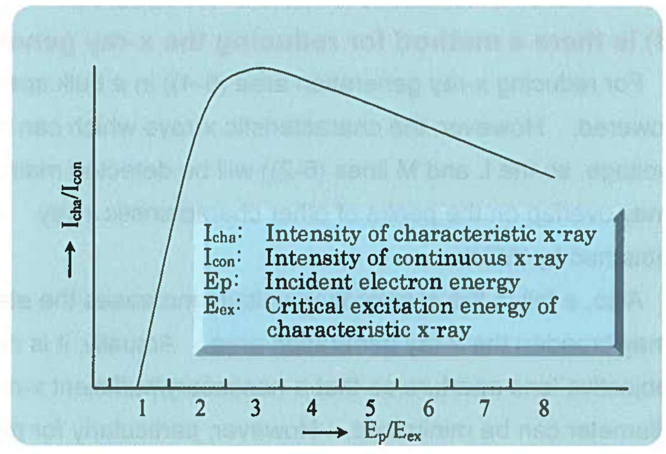
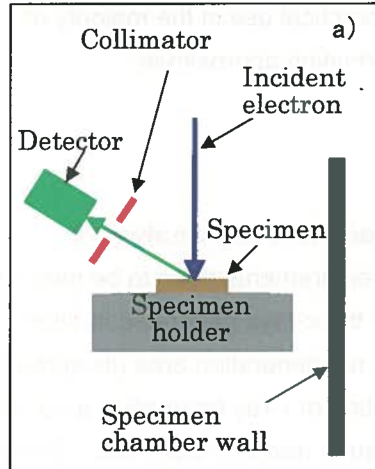
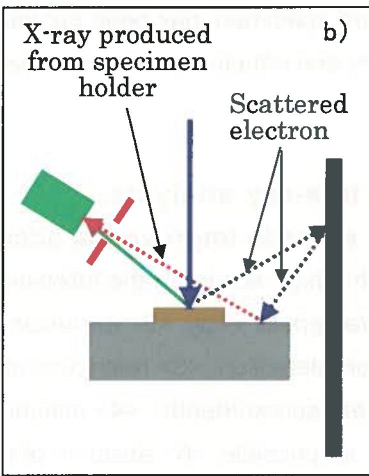


Fig. 1 Relationship between Characteristic X-ray Intensity and Incident Electron Energy (Accelerating Voltage)

3) What should be done in order to avoid detecting x-rays produced from other than the sample (location of interest)?

When the detector and collimator look right into the electron beam irradiation point on the specimen surface as shown in 2a), the x-rays produced from other than the specimen are not detected usually. However, when the detector and collimator look at part of the specimen holder because the specimen position is deviated as shown in 2b), the x-ray excited by a scattered electron on the surface of the specimen holder will be detected. And, when the specimen position is further deviated and the detector/collimator looks at the specimen chamber wall as shown in Fig. 2c), the x-rays produced from the specimen surface will hardly be detected and the x-rays excited on the specimen chamber wall will mostly be detected. Therefore, analysis is always required in the status shown in 2a), but the condition in Fig. 2-b) or c) may be arranged unconsciously, because a SEM specimen has a complicated shape. So attention should be paid.





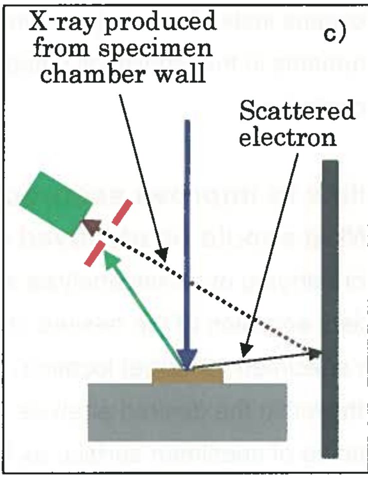


Fig. 2 Undesirable Detection of X-rays Produced from Other Than Specimen (location of interest)

a) Correct specimen position where detector/collimator looks right into specimen

b) Detector/collimator looking at specimen holder due to deviation of specimen position

c) Detector/collimator looking at specimen chamber wall due to further deviation of specimen position

4) Is there a method for reducing the x-ray generation area?

For reducing x-ray generation area (6-4)) in a bulk specimen, the accelerating voltage needs to be lowered. However, the characteristic x-rays which can be excited are limited at a low accelerating voltage, so the L and M lines (6-2)) will be detected mainly. In some cases, the L and M line peaks may overlap on the peaks of other characteristic x-ray. In such cases, high accuracy analysis can be obtained by WDX.

Also, a fall in the accelerating voltage increases the electron beam diameter of SEM (4-2)), which may broaden the x-ray generation area. Actually, it is necessary to optimize the condenser lens and objective lens aperture so that a necessary/sufficient x-ray signal is obtainable and the electron beam diameter can be minimized. However, particularly for micro-area analysis, it is effective to use a SEM equipped with a Schottky electron gun (1-3), 1-4)) by which a comparatively small electron beam diameter is obtainable even at a large probe current.

5) How does the shape of the sample affect the analytical accuracy?

When the specimen surface is extremely uneven, analytical accuracy would be degraded because <1> advance of x-rays to the detector is blocked, <2> a calculation error occurs due to change in the ZAF effects (6-12)) and other. Therefore, it is deemed desirable to flatten the specimen surface as far as possible by mechanical polishing after resin embedding, etc. as shown in Fig. 3. If flattening process is unusable for some reason, specimen tilting and rotation need to be adjusted so that advance of x-rays toward the detector will not be blocked and the x-ray take-off angle will not change.

Also, influence by the internal structure of a specimen cannot be neglected as illustrated in Fig. 4. Figure 4a) shows that the elemental area of interest is distributed in the form of a thin film inside a specimen. In this case, a fall in the characteristic x-ray intensity of the target element cannot be denied. Figure b) is an example showing that the distribution of the target element is narrow on the surface, but wide and deep at the inside. In this case, the characteristic x-rays are produced from a far wider area than that of a secondary electron image. Figure c) illustrates a case where a target ion which is not contained in the matrix is detected. Thus, a wrong analytical result may be obtained depending on the shape of a specimen, but it is effective for ensuring accuracy to measure as many analysis points as possible and obtain an average value. Since EDX and WDX analyses are basically micro-area analyses, a mean value among a number of measurement results should be obtained for determining the macroscopic compositional information of a specimen.

Fig. 3 Example of X-ray Analysis Specimen Mechanically Polished after Resin Embedding

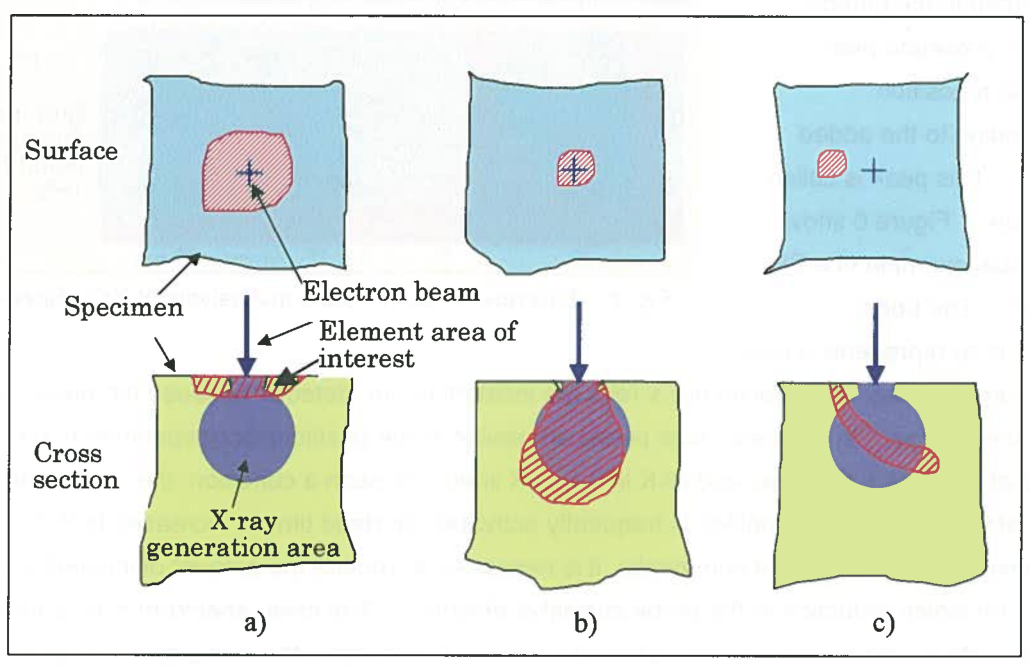


Fig. 4 Relationship between Different Internal Specimen Structures and X-ray Generation Area

6) What kind of pseudo (Artifact) peaks appear in EDX?

Main pseudo (artifact) peaks in EDX analysis are described below.

<1> Escape Peak

Upon incidence of a characteristic x-ray on a Si solid state detector (6-6)), the Si-K line is excited. As a result, some energy of the incident characteristic x-ray is lost, which gives rise to a pseudo peak (called an escape peak). Therefore, the escape peak appears at an energy position given by subtracting the energy (1.74 keV) of Si-K line from that of the characteristic x-ray. Actually, the majority of EDX systems are provided with the escape peak removal function, so these peaks can be readily removed.

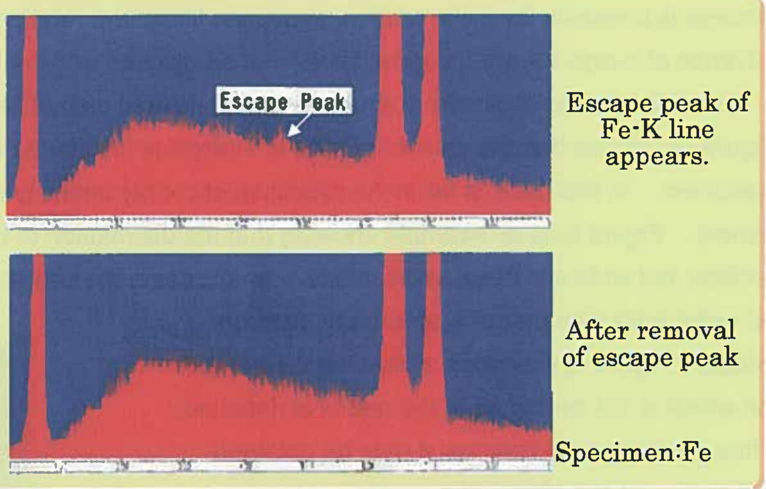


Fig. 5 Example of Escape Peak in Analysis of Fe Specimen

<2> Sum Peak

When two characteristic x-rays are incident on the detector in shorter than its resolving time, the charges due to the respective x-rays cannot be separated in the detector. Therefore, a pseudo peak appears at a position corresponding to the added energies. This peak is called a sum peak. Figure 6 shows an analytical example of a SiO_2 specimen. The upper spectrum in a) represents a case where a large number of characteristic x-rays are incident on the detector because the probe current is excessively large. In this case, sum peaks are visible at the positions corresponding to the energies of O-K line + Si-K line, and Si-K line + Si-K lines. In such a condition, the pileup rejection function of the proportional amplifier is frequently activated, so dead time is increased to 35%.

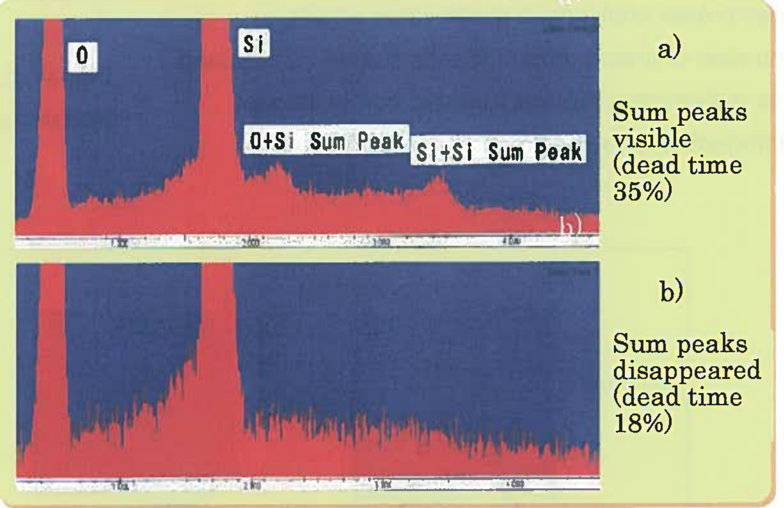


Fig. 6 Example of Sum Peaks in Analysis of SiO_2 Specimen

For preventing occurrence of sum peaks, it is necessary to reduce the amount of incident x-ray per unit time, for which reduction of the probe current is effective. The lower spectrum in b) is the one measured with the probe current reduced to about 1/5. In this case, the sum peaks disappeared and the dead time was shortened to 18%.

<3> System Peaks

During x-ray analysis, backscattered electrons and x-rays are flitting about near a SEM specimen, and unnecessary x-rays from other than the element contained in the sample are generated everywhere in the specimen chamber. If the detector looks at an unnecessary x-ray generating location directly, the x-ray from it will be detected as a system peak only to downgrade the analytical accuracy. Before start of your analysis, therefore, it is necessary to check for system peaks by using a carbon (C) specimen which does not contain impurities. As a preventive measure, it is essential that the detector looks straight into the electron beam irradiating position on a specimen as explained in 7-3). If the system peaks still appear, the specimen shape, structural materials near the specimen, collimator position of the detector, etc. should be reviewed.

7) What should be done to achieve high-accuracy EDX analysis with the low vacuum SEM?

For EDX analysis of a nonconductive specimen, it is common to apply a thin coating film to the specimen surface. In this regard, imaging in a low vacuum of a few ten Pa or more is advantageous because EDX analysis can be carried out without specimen coating. However, characteristic x-rays might be generated in a substantially wide area because the incident and backscattered electrons are scattered due to collision with residual gas molecules in a low vacuum environment (5-4)).

As is well known, electron scattering in a low vacuum environment increases suddenly when pressure exceeds 100 Pa and due to this phenomenon, the x-ray generation area widens suddenly and the intensity of low-energy x-rays falls due to absorption by the residual gas molecules.

Hence, a pressure range from about 30 Pa where a charge-up phenomenon can be reduced, to 80 Pa where electron scattering and x-ray absorption are comparatively small, is considered an appropriate EDX analysis condition with a low vacuum SEM. Figure 7 gives an analytical example of the surface of a heat resistant sheet for heat sinks at accelerating voltage 15 kV and pressure 30 Pa. Detection of unnecessary element peaks due to scattering was at a negligible level, and low-energy elements were detected with a high S/N ratio.

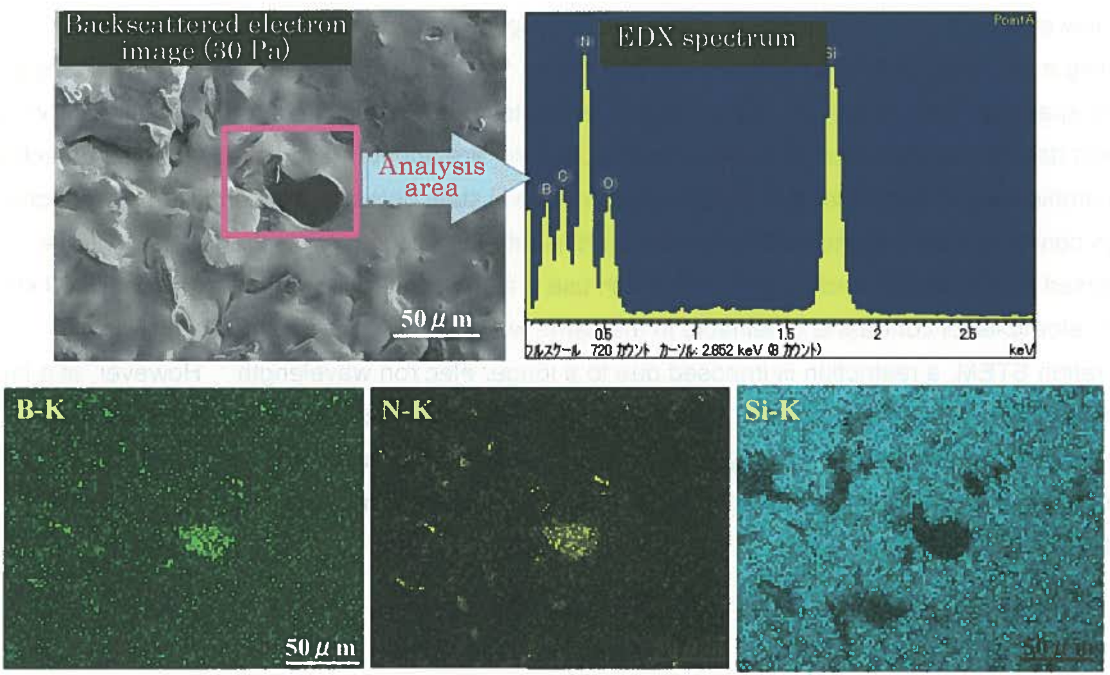


Fig. 7 Analytical Example of the Surface of Heat Resistant Sheet
Accelerating voltage 15 kV, specimen chamber pressure 30 Pa

8. Principle and application of STEM

1) What kind of instrument is the STEM?

A STEM (Scanning Transmission Electron Microscope) is an instrument designed to obtain a magnified image through detection of the transmitted and scattered electrons (8-2)) by irradiating a thin-film specimen (of about 100 nm thick) with a convergent electron beam while scanning it two-dimensionally on X-Y plane as schematically diagrammed in Fig. 1. The transmitted electrons carry information about the density and crystalline state of a specimen, while the scattered electrons provide information about compositional distribution.

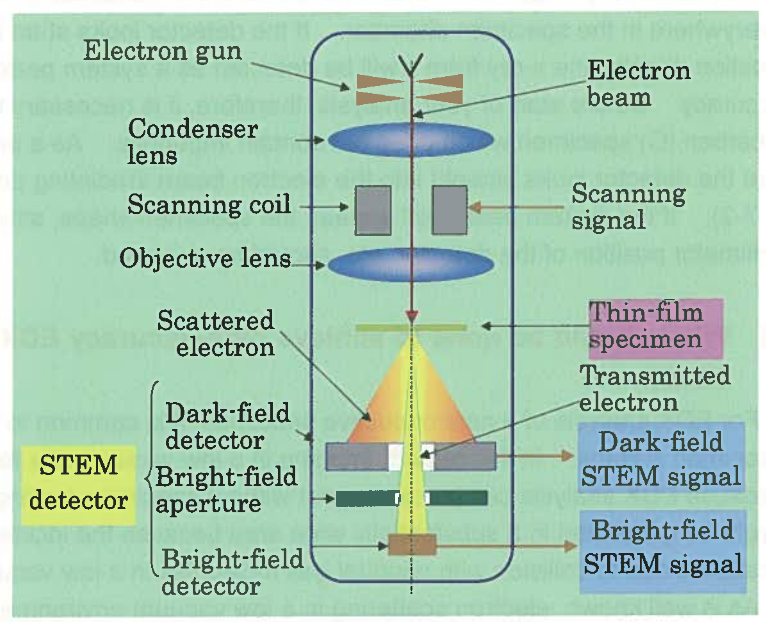


Fig. 1 Instrumental Configuration of STEM

STEMs are roughly classifiable into 3 types; <1> a TEM to which electron beam scanning function and detector are added, <2> an exclusive STEM instrument equipped with an electron gun for 200 kV or higher and <3> a low acceleration STEM (up to 30 kV) with a STEM detector mounted to the SEM column. Explained here is the characteristics and operation method of the low acceleration STEM based on SEM.

The low acceleration STEM uses the same electron-optics as SEM's for its mechanism for irradiating a specimen with a convergent electron beam, to which a specimen holder for mounting a thin-film specimen like the one for TEM, a bright-field detector for detection of transmitted electrons, a dark-field detector for detection of scattered electrons, etc. are added. Employed as these detectors are a combination of scintillator and photomultiplier, a solid state detector, a mechanism for detection through converting the transmitted/scattered electrons into the secondary electrons, etc. With a TEM-based STEM and an exclusive STEM which use a high accelerating voltage (generally 200 kV or higher), atomic-level contrast is obtainable in the same way as TEM. By contrast, with a low acceleration STEM, a restriction is imposed due to a longer electron wavelength. However, at a low accelerating voltage, sharp contrast is obtainable because electron scattering is large (scattering power is high) even from a low-density specimen. Therefore, application of a low acceleration STEM is expected in an extensive field covering polymer materials, semiconductor materials, nanotechnological materials, etc.

2) What are the characteristics of the STEM signal?

When the electron beam is projected onto a thin-film specimen, the electrons subjected to elastic and inelastic scatterings (3-2)) and those transmitted through the specimen without being scattered both come out in the incident beam advancing direction as shown in Fig. 2. Now, let's detect only the transmitted electrons by shutting off the scattered electrons with the bright-field aperture. Now, the specimen area having a high density is observable as a dark area, and that having a low density as a bright area so far as specimen thickness is even. Such an image is called a "bright-field STEM image." Contrast of this image becomes higher as the accelerating voltage is increased further. Also, in case of a crystalline specimen, the contrast which reflects the condition of electron diffraction is superimposed, so the area satisfying this condition is displayed in dark contrast.

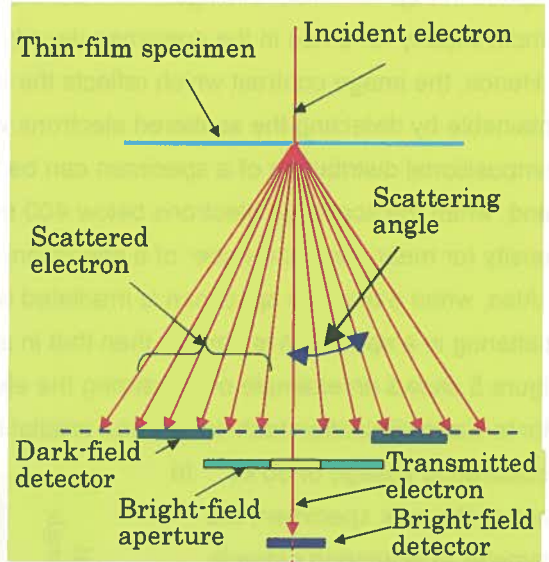


Fig. 2 Electron Transmission and Scattering when Electron Beam Passes through Thin-film Specimen

On the other hand, an image formed through detection of scattered electrons with the dark-field detector is called a "dark-field STEM image." Because the scattering angle of scattered electron differs largely depending on specimen thickness, density and mean atomic number, the dark-field detector needs to be mounted at the optimum position for each specimen.

Usually, the shape and mounting position of this detector are optimized so that the detector is applicable to specimens in the widest range possible. Some models of STEMs are equipped with a mechanism for changing the detector position from outside vacuum.

At present, the escape angle of scattered electrons from a specimen can be estimated by simulation. This feature makes the use of low-acceleration/dark-field STEM images easier. Figure 3 shows an example with a specimen thickness of 100 nm. From this figure, it is understandable that in the distribution of scattered electron angles, the maximum value is reached at a higher angle as the specimen density rises. A correlation is noticeable between the specimen density and the number of scattered electrons

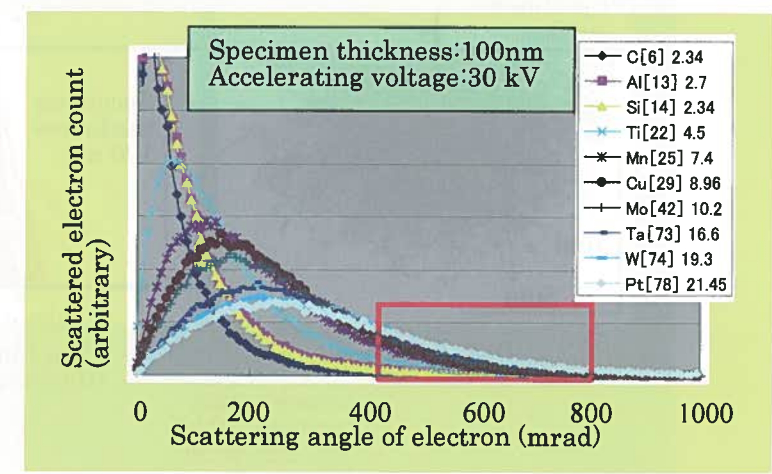


Fig. 3 Example of Monte Carlo Simulation to Determine Angle Distribution of Scattered Electrons with 10 Kinds of Thin-film Specimens (100 nm thick)

within an angle range from 400 to 800 mrad enclosed by red box in Fig. 3. This correlation is graphed in Fig. 4. From this figure, it is confirmable that the number of scattered electrons increases almost linearly for a rise in the specimen density.

Hence, the image contrast which reflects the specimen density (or mean atomic number) is obtainable by detecting the scattered electrons within a range from 400 to 800 mrad, so the compositional distribution of a specimen can be known by a dark-field STEM image. On the other hand, when the scattered electrons below 400 mrad are detected, the contrast which reflects the density (or mean atomic number of a specimen) cannot always be obtainable as is evident from Fig. 3.

Also, when a thin-film specimen is irradiated with an electron beam, the diameter of electron scattering in a specimen is smaller than that in a bulk specimen, so a high resolution can be expected. Figure 5 shows an example of visualizing the electron scattering in aluminum (Al) specimens by the Monte Carlo simulation technique when irradiating each specimen with electron beam at an accelerating voltage of 30 kV. In case of the bulk specimen, the diameter of scattering extends about a few μm, while scattering stays in a narrow area of about 20 nm in case of the thin-film specimen. Thus, use of STEM signals enables the electron microscopist to visualize the fine structure and compositional distribution in a specimen with a nanometer-order high resolution.

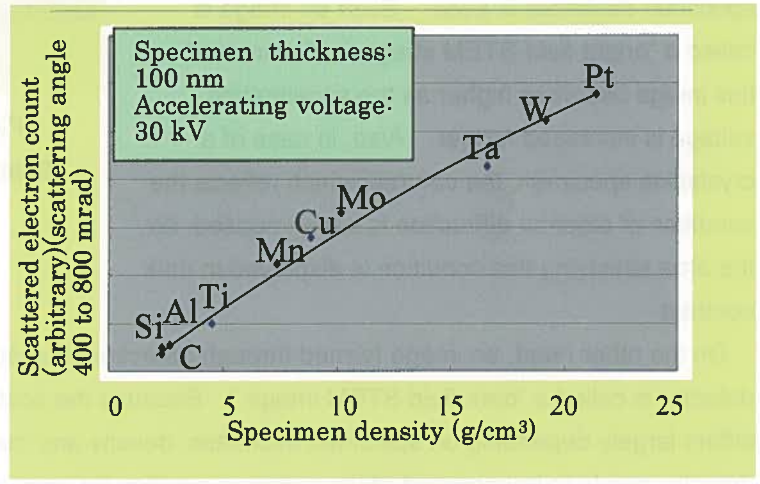


Fig. 4 Change in Scattered Electron Count versus Specimen Density

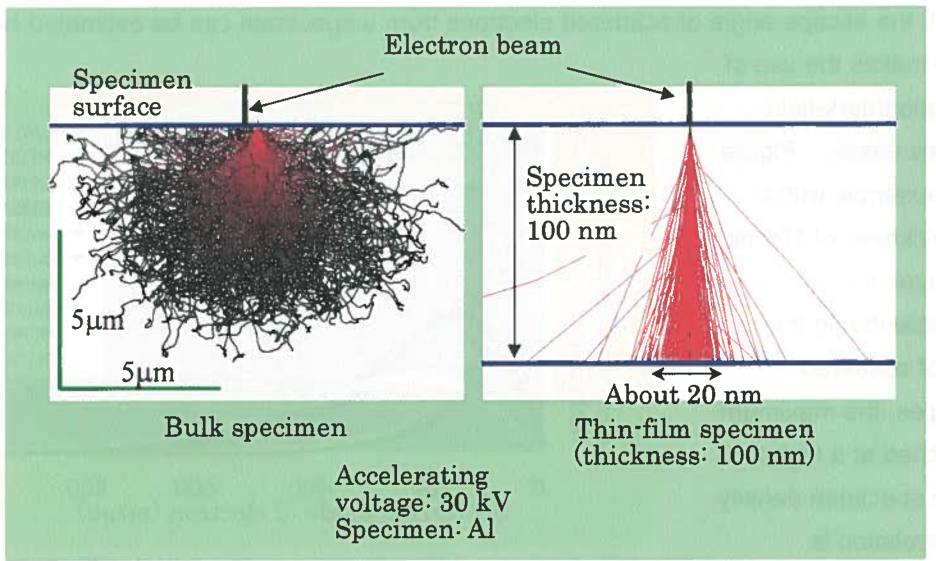


Fig. 5 Example of Monte Carlo Simulation of Electron Scattering in Al Specimens (Accelerating voltage: 30 kV, thin-film specimen thickness: 100 nm)

3) What features are obtained by EDX analysis using thin-film samples?

EDX analysis of thin-film specimens has features such as; <1> capability of analyzing a very narrow area thanks to a small electron scattering diameter in specimens (8-2)), <2> Z correction alone being sufficient, i.e., no need for the ZAF correcting calculation (6-13)) unlike analysis of bulk specimens and <3> high S/N ratio because less affected by continuous x-ray (6-3)). Since the thin-film quantitative analysis software is incorporated in the majority of EDX analysis systems, quantitative values are readily available. However, one should be careful about this analysis because system peaks (7-6)) may be caused by the characteristic x-rays from a specimen supporting grid, etc.

Figure 6 compares the x-ray mapping images of the cross-sectional structure of a multi-layer specimen including Al and Cr layers which was formed into a bulk or thin film. With the bulk specimen, its layer structure can hardly be confirmed, but the Al layer (about 150 nm thick) and Cr layer (about 100 nm thick) are clearly visible with the thin-film specimen. Thus, thin-filming of a specimen can enhance the resolution of element mapping images remarkably.

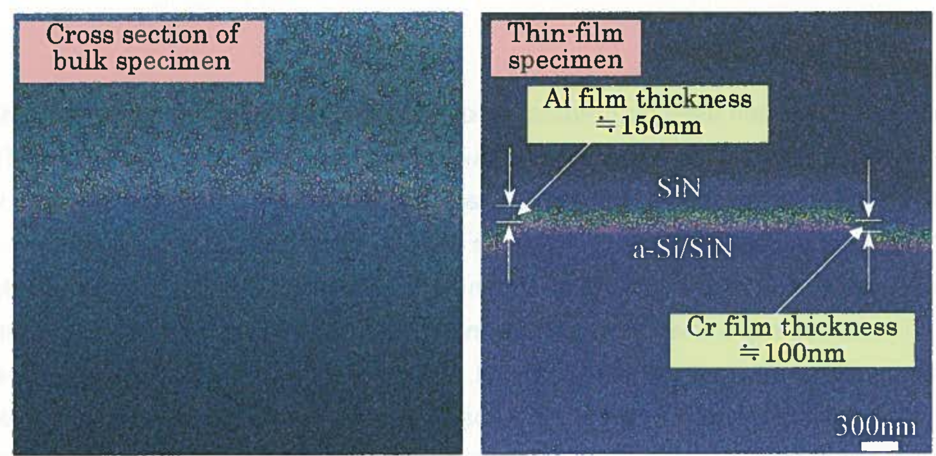


Fig. 6 Comparison of SiN/Al/Cr/SiN Multi-layer Cross Section Imaging between Bulk and Thin-Film Specimens
Specimen preparation: Cross-sectioning and thin-filming with FIB,
Accelerating voltage: 15 kV, Instrument used: Model S-4700 FE-SEM

4) What is the method of preparing samples for STEM observation/analysis?

The methods for STEM specimen preparation are almost the same as for TEM. Dispersion method, microtomy, FIB milling, etc. are mainly adopted. They are selectively used in response to the object and purpose of observation/analysis.

1) Dispersion Method

Such fine powdery specimens (various nanotechnological specimens, etc.) through which an electron beam can pass easily are usually dispersed on a grid after placing a carbon supporting film on it. In concrete procedure, such a powdery specimen is dispersed in a dispersion medium to prepare a suspension, which is dripped on the carbon supporting film followed by drying as illustrated in Fig. 7. As a dispersion medium, water, methanol, n-butanol, xylene or the like is used. Because a medium's reactivity and dispersion effect differ from one specimen to another, some trial and error will be required. It is also employed to pour the specimen directly on the carbon supporting film without using a dispersion agent and then give vibrations or lightly spray air in order to blow off excess specimen. A grid with carbon supporting film is commercially available.

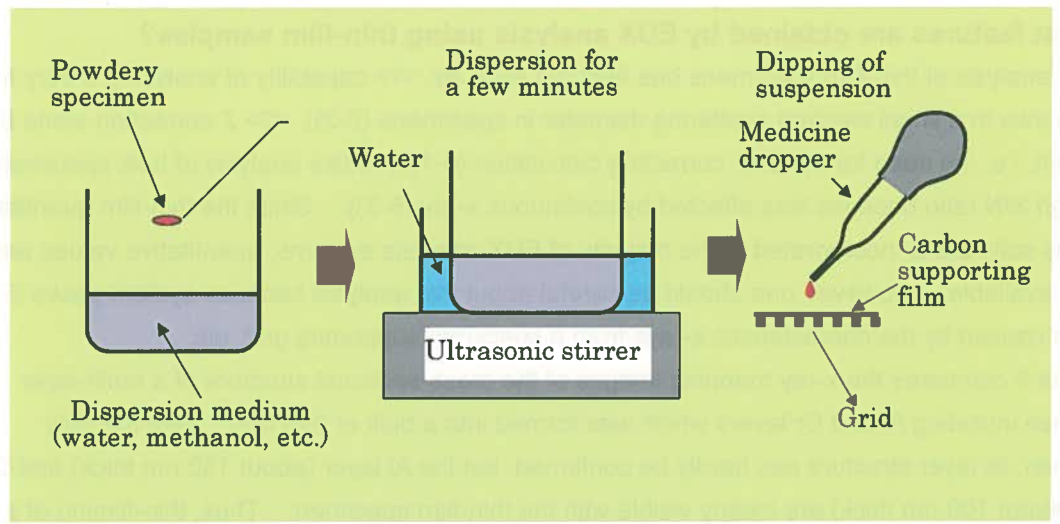


Fig. 7 Preparation of Specimen for STEM Observation/Analysis by Dispersion Method

2) Microtomy

This method has long been used for preparation of biological and polymer specimens, and recently applied also to thin-filming of metals and semiconductor materials with a diamond knife. The cutting principle of a microtome is as shown in Fig. 8, i.e., a specimen is cut by moving it vertically with respect to the edge of a diamond knife. Because the vertical motions of a specimen and specimen feeding motions are all automated, a number of thin-film specimens can be prepared quickly in succession. In case of a specimen consisting of materials different in physical characteristics (hardness, etc.) such as the specimen A/B in Fig. 8, there is a drawback that peeling is apt to occur at the boundary of materials A and B. But this drawback can be improved, for example, by resin embedding after sandwiching such a specimen with dummy materials. For details of the improving methods, refer to the reference document at the end of this text.

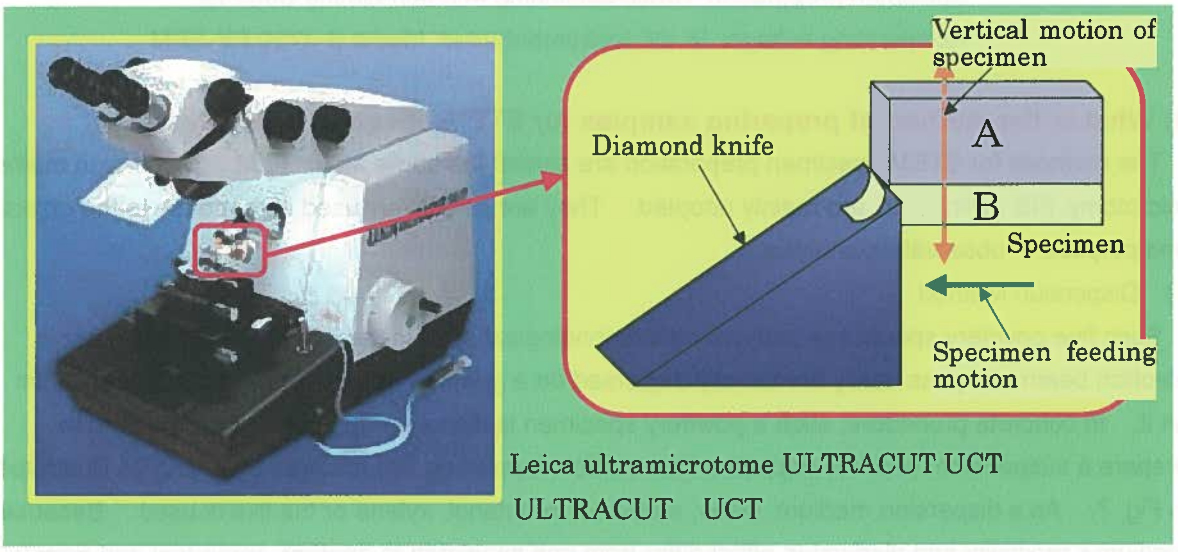


Fig. 8 Appearance of Microtome and its Cutting Principle

3) FIB Method

The FIB (Focused Ion Beam) method is a thin-film preparation method under rapid dissemination and features that a specific area alone in a specimen can be formed into a thin film using a focused gallium (Ga) ion beam. The basic 3 functions of FIB are shown in Fig. 9. A thin-film specimen is prepared by using all the basic functions. Firstly, a SIM (Scanning Ion Microscope) image is formed through detection of the secondary electrons ejected upon irradiation of the ion beam, and while observing this image, a specimen processing position is determined. Next, a desired part of the specimen is cut out by the sputtering function, and then the cut-out specimen part is deposited on the specimen holder by the deposition function. The whole process of this specimen preparation is carried out with a micro-sampling unit mounted to the FIB system. An example of viewing the details of this process with SIM images is introduced in Fig. 10.

Let's begin with determination of the processing position with a SIM image and deposit a tungsten (W) film for protection as shown in Fig. 10-a). Then, restrict the specimen area of interest to a small piece of about 15 μm wide by means of ion beam so that the small piece is supported at only the location (micro-bridge) indicated by the arrow in Fig. 10-b). Tilt the specimen and cut off its bottom by the ion beam (see c)), and then bond a probe to the small piece by W deposition (see d)). Next, cut off the micro-bridge as shown in Fig. e) and take out the small specimen piece completely from the original specimen. Subsequently, stick the small specimen piece to the specimen holder by W deposition as shown in Fig. 11-a) and process the location of microscopic/analytical interest into a thickness of about 100 nm as shown in 11-b). In this way, a STEM specimen can be extracted from a specific part of a bulk specimen with the FIB system equipped with a micro-sampling unit.

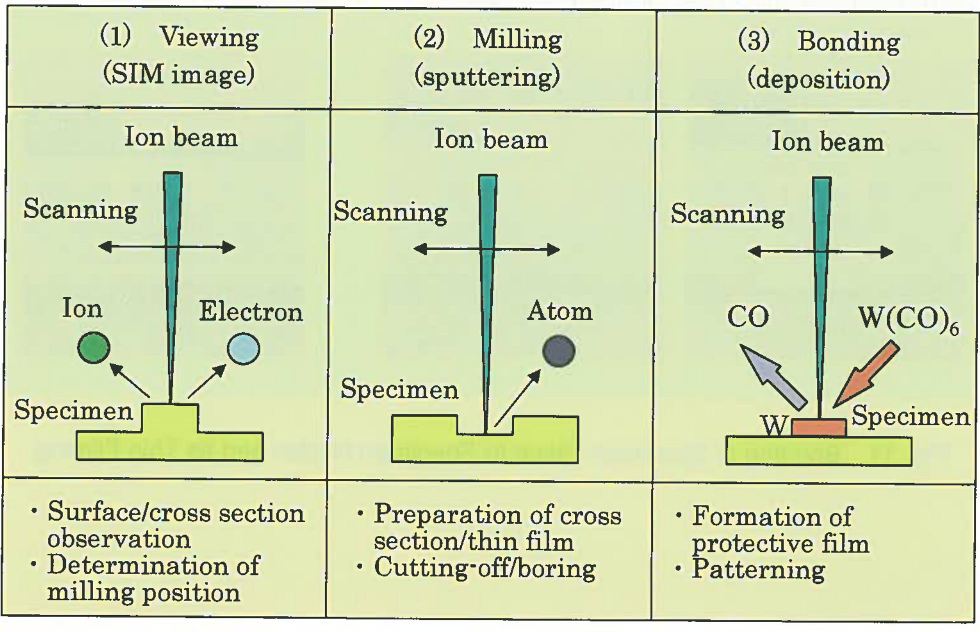


Fig. 9 Basic Functions of FIB

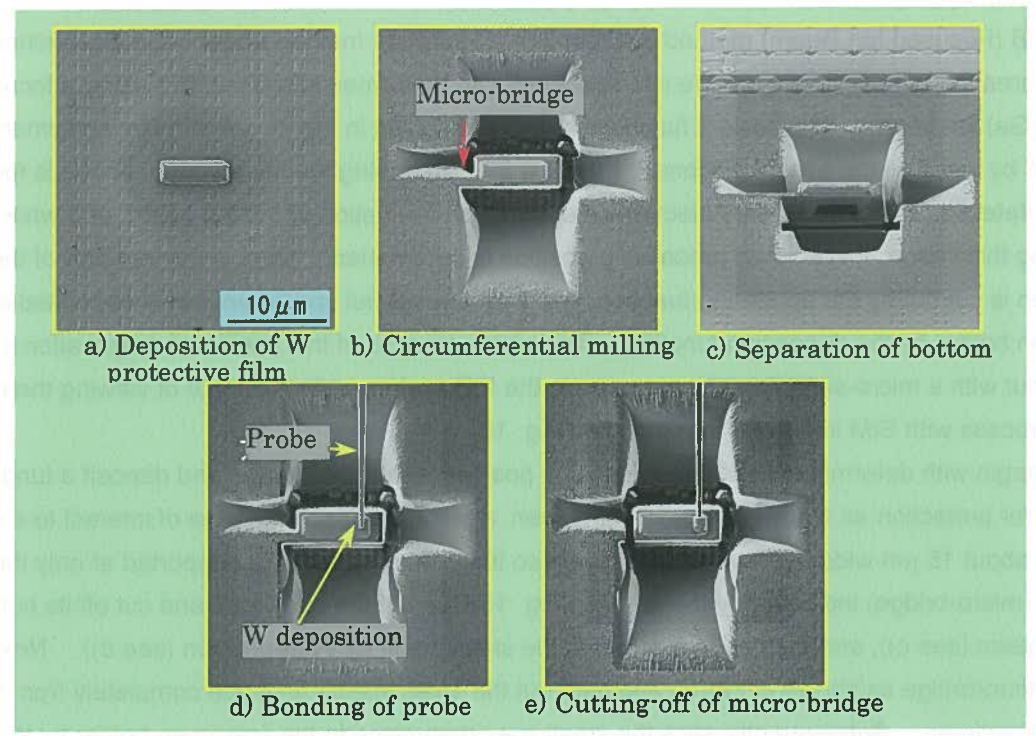


Fig. 10　Small Specimen Piece Extraction Process with Micro-sampling Unit

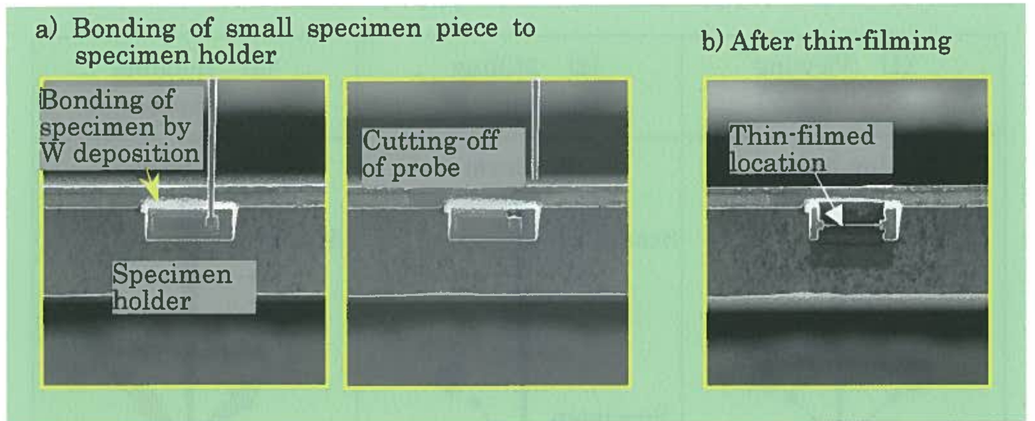


Fig. 11　Bonding of Specimen Piece to Specimen Holder and its Thin Filming

5) What are the applications of STEM (EDX)?

Since a low acceleration STEM is capable of imaging the internal structure of a specimen with high resolution and contrast, its application is rapidly expanding as a tool for viewing of polymer materials, composite materials and nanotechnological materials, and for evaluation/failure analysis of semiconductor devices and various recording devices. Particularly in low acceleration STEM image observation with a SEM equipped with an FE or Schottky electron gun (1-3)) and an in-lens or semi-in-lens (snorkel type) objective lens (1-7)), uncoated specimen observation (heavy-metal staining unnecessary) can be achieved with nanometer-order resolution.

Figure 12 compares the bright-field images of the cross-sectional structure of a toner particle for copying machine which were formed at accelerating voltage 15 kV and 200 kV, respectively. At accelerating voltage 15 kV, each structure of wax and dispersing material are imaged with higher contrast than at 200 kV. As is evident from this figure, the low acceleration STEM imaging features high contrast for specimens having a comparatively low density. This thin-film specimen was prepared using the FIB system equipped with a micro-sampling unit. For evading specimen damage, a tungsten (W) protective film was coated on the particle surface by the deposition function beforehand.

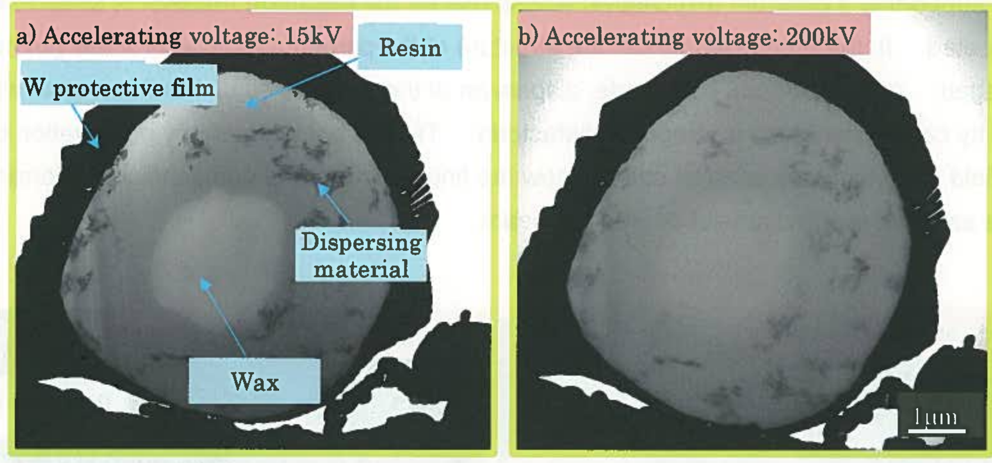
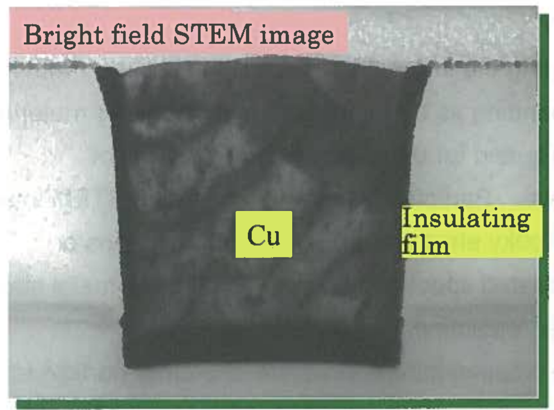
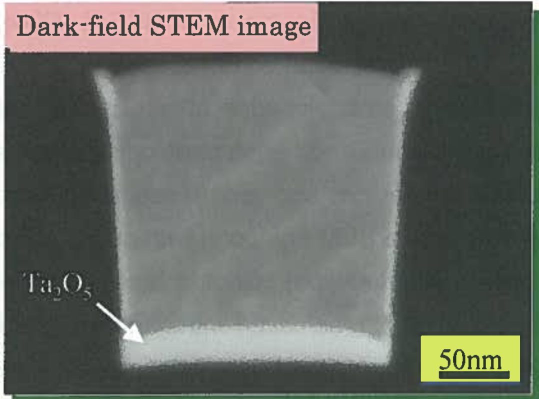


Fig. 12 Bright-field Images of Cross-sectional Structure of Toner Particle (accelerating voltage: 15 kV, 200 kV)(thin film formed by FIB system equipped with micro-sampling unit, film thickness: about 100 nm)

Shown in Fig. 13 are bright and dark-field STEM images of the cross-sectional structure of the copper (Cu) in LSI at an accelerating voltage of 30 kV. In the bright-field image, contrast dependent on electron transmissivity and crystalline information (such as grain boundary and defect) can be seen. And in the dark-field image, contrast due to specimen density and mean atomic number could be obtained. In particular, the barrier metal layer (Ta_2O_5) formed between Cu and insulating film is clearly observable. The scattered electron detection angle in this dark-field imaging was within a range from 400 to 700 mrad, and the specimen was prepared using the FIB system equipped with a micro-sampling unit.

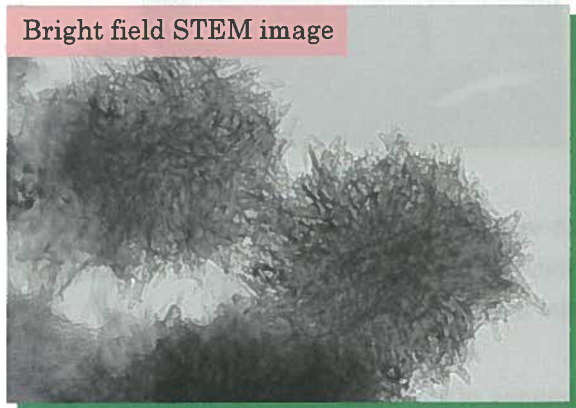




Detection angle: 400 to 700 mrad

Fig. 13 Example Viewing of LSI Cu Pattern Cross Section with Bright and Dark-field Images
Accelerating voltage: 30 kV, Specimen thickness: About 100 nm

Figure 14 shows the bright and dark-field STEM images of the nanotechnological material (carbon nanotube) supporting a platinum (Pt) catalyst whose use for the electrode material of small fuel cell, etc. is expected. In the bright-field mode, the structure of the carbon nanotube of a few nm dia. is clearly imaged. And in the dark field mode, dispersion of the Pt particles of 1 to 2 nm each which are supported by carbon nanotube is imaged satisfactorily. Thus, a complementary observation of bright and dark-field STEM images enables one to know the fine structure and compositional information in a micro-area as small as the diameter of electron beam.



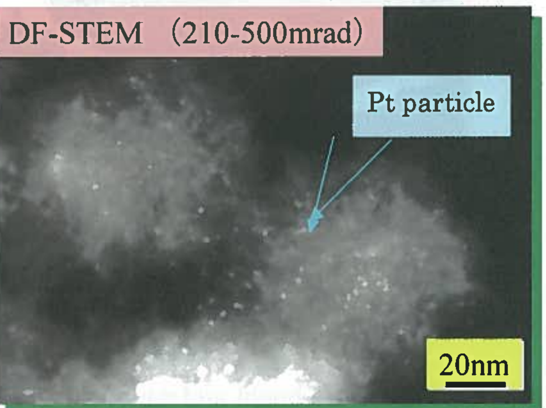


Fig. 14 Bright and Dark-field STEM Images of Catalyst (Pt/carbon nanohorn)
Accelerating voltage: 30 kV, Specimen Preparation: Direct observation by pouring on grid
Specimen by courtesy of: Japan Science and Technology Agency/Fundamental and environmental research laboratories

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