# Invitation to the SEM World

For people who are using the SEM for the first time





1m



Human



1/1000 m 1m(millimeter)



Ant(2mm)



Resolution of SEM →

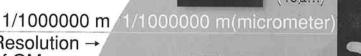
Resolution of -naked 'eye



Lilly pollen  $(40 \mu m)$ 



Thickness of human hair



 $(\sim 70 \mu m)$ 



Resolution → of OM



irus ~100nm)



Bacteria (a few  $\mu$ m)



Resolution of SPM → Resolution of TEM →





Carbon nanotube (~1nm)



Atom ₩ (~0.1nm)



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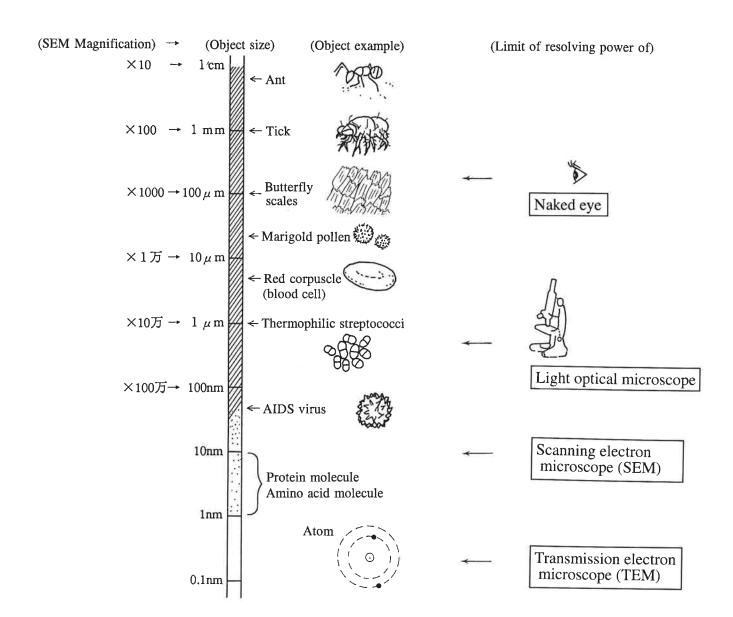
# 1 What is the SEM?

# 1.1 Observation of a Tiny Object in Magnified Scale

Scanning Electron Microscope → SEM

"A tool to observe an invisible tiny object in a stereographic image with a magnified scale"

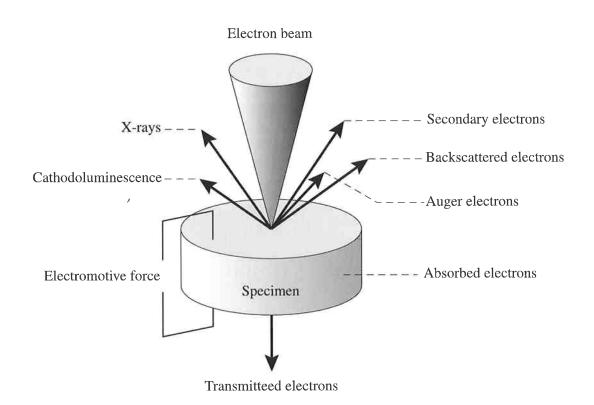
Some typical objects that can be viewed with microscopes are shown below at the corresponding position of the scale below. There are many tools that can be used to view microscopic objects. Among them, the Scanning Electron Microscope (SEM) covers a wide range of magnification, about  $\times$  10 to  $\times$  1,000,000. The figure below, "(SEM Magnification)" indicates an appropriate SEM magnification for observing each object. Some of the SEM's most important features are easy magnification changing over, large depth of field (depth of focus), and stereographic image display.

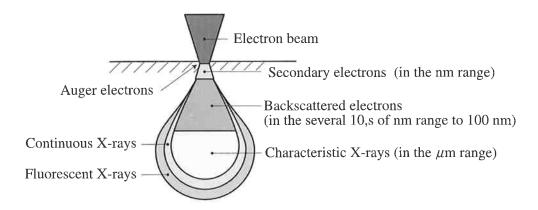


#### 1.2 Principles Underlying the SEM

#### (1)Information obtained by electron beam irradiation

When a specimen is irradiated with an electron beam, interactions between the incident electron beam and the constituent atoms in the specimen produce various signals, as shown in the following two figures. The upper figure illustrates the kind of signals obtained, whereas the lower figure indicates the depth within the specimen from which these signals are generated.

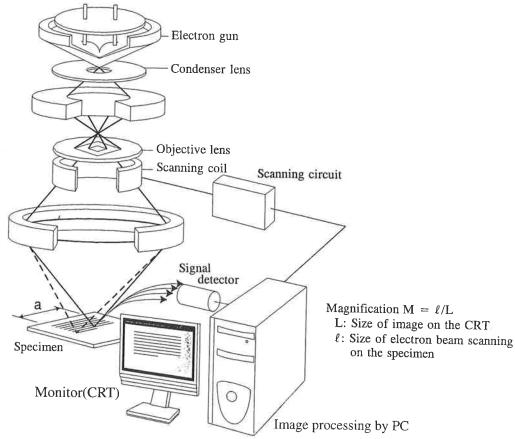




Depth of generation of various quanta and their spatial resolutions (by Goldstein)

#### (2) Principle of magnification

When scanning a specimen surface with a finely focused electron beam (one of only several nano meters), information will be emitted from each point of the scanning (refer to the above). The emitted information is converted into an electric signal, amplified, then fed into an observation CRT. On the CRT, the information is used to control the brightness of the corresponding spot. The spot on the CRT is shown in real time with the electron beam scanning on the specimen surface. Thus, the information emitted from the specimen surface is displayed on the CRT as an image. In this example, the magnification of the displayed image is defined as the ratio of the size of the image on the CRT to the size of the electron bean scanning on the specimen surface. The type of information obtained can be changed by switching the signal. In this way, a specific desired characteristics of the specimen surface can be seen on the CRT in a magnified scale.



#### 1.3 What is the SEM used for ?

As described in the previous section, various kinds of signals can be obtained by an SEM. These signals carry different types of information, and are used for different purposes, as listed below.

(Signal)	(Mode	of Operation)		(Purpose of SEM)
				(Information carried)
Secondary electrons		(SEI)	$\rightarrow$	Topographical observation of surface
Backscattered electro	ons	(BEI)	$\rightarrow$	Compositional observation of surface
X-rays		(X-ray)	$\rightarrow$	Elemental analysis of specimen
Transmitted electron	IS	(TEI)	$\rightarrow$	Internal structure observation
Cathodoluminescenc	e	(CL)	$\rightarrow$	Internal characteristics observation
Electromotive force		(EBIC)	$\rightarrow$	Internal characteristics observation
Secondary electrons	or	(ECP)	$\rightarrow$	Crystalline structure
Backscattered electro	ons	(MDI)	<b>→&gt;</b>	Magnetic domain observation

Some typical examples for each are shown in the following pages.

# 2. Observation Examples

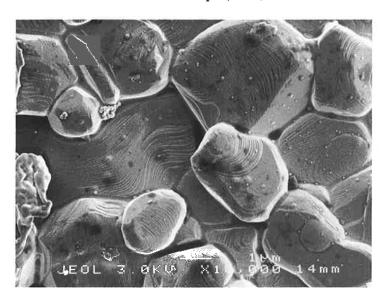
#### 2.1 Topographical Observation (Secondary Electron Image)

The number of secondary electrons emitted from a specimen surface depends greatly on the incident angle of the electron beam to the specimen surface. In other words, the secondary electron signal depends on the surface undulation of the specimen. Moreover, since the energy of secondary electrons is very low, they are only emitted from a thin layer on the specimen surface. Thus, secondary electron signals are considered to be the most suitable signal for observing a specimen's surface topography.

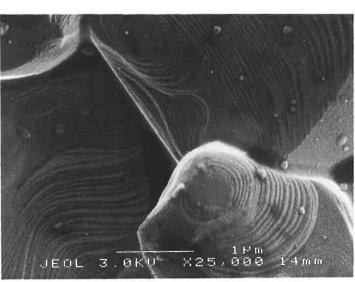
The following lists some examples of how this type of SEM observation is used in various application fields:

#### (1) Material Science

SEMs are used in the most advanced material research and development fields, such as superconducting materials, and architecture materials. The photographs below show a fractured surface of a ceramic material. You may see crystal steps which were formed during sinter. Since surface structure can be observed on a bulk specimen, specimen preparation is very much easier than with transmission electron microscope (TEM) work.



Specimen: Fractured surface of ceramic

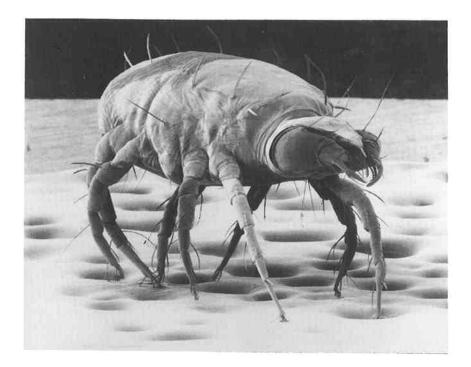


Magnification: × 10,000

Magnification:  $\times$  25,000

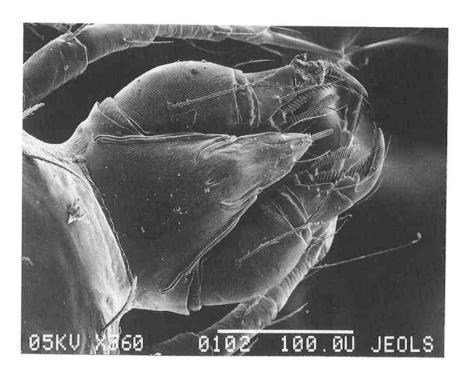
#### (2) Biology

Biology, especially bio-technology, is one of the most trendy area of research today. In this field the SEM is used to observe large objects such as insects and animal tissues, as well as small objects such as bacteria and viruses. The photographs below are a side view and a top view of a tick. This shows how the SEM can be a powerful tool in entomological taxonomy, which is just one example of the many applications of this tool in this field.



Specimen: Tick

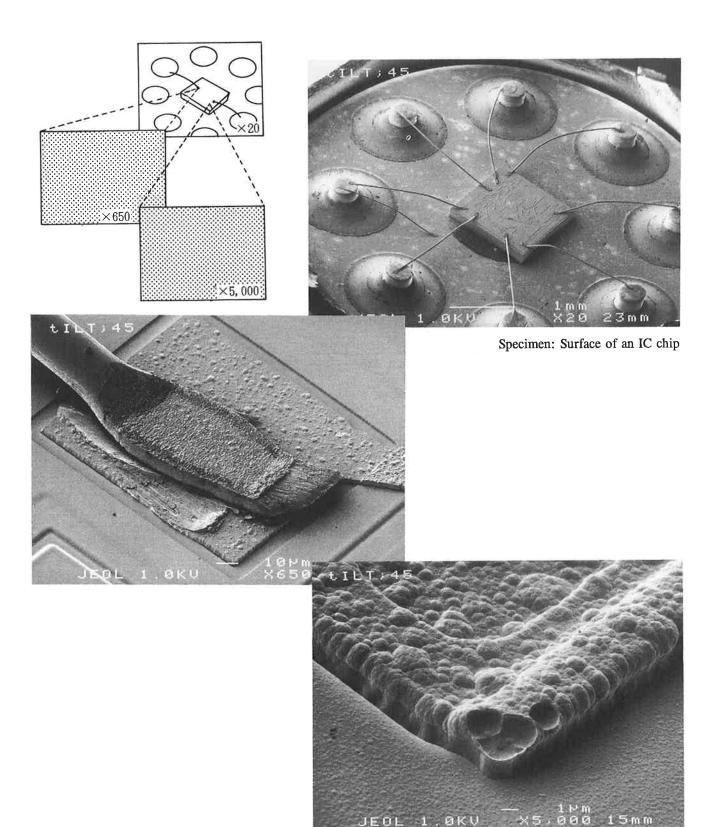
Magnification: × 100



Magnification: × 360

#### (3) Electronics

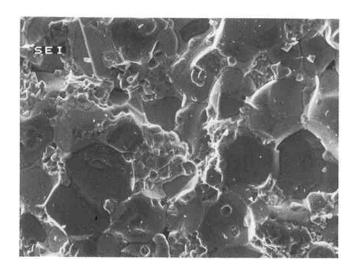
The SEM also shows its power in quality control and failure analysis in the semiconductor and electronics fields, as well as in research and development. The examples shown below are observations of welds of bonding wire and the surface structure of an IC chip. A large depth of focus, one of the major features of an SEM, presents us with fine and detailed structural images in both the large and tiny parts of the specimen.



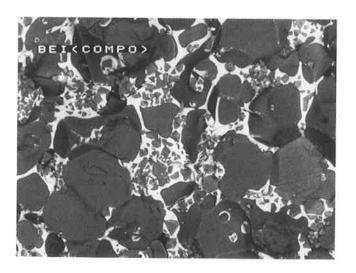
# 2.2 Compositional Observation (Backscattered Electron Image)

Since the energy of backscattered electrons is very much higher than that of secondary electrons, it carries information from deeper layers of the specimen surface. The number and scattering direction of the backscattered electrons are determined by the average atomic number of the substances composing the specimen surface and the incident angle of the electron beam to the specimen surface.

As shown in the examples below, a specimen's surface topography can be seen with the secondary electron image (SEI, top), and composition difference (grain and grain boundary) can be clearly distinguished with the backscattered electron image.



Secondary electron image



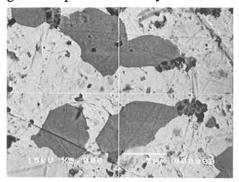
Backscattered electron image (Composition image)

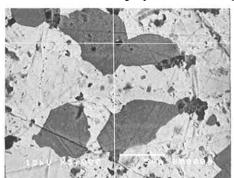
Specimen: Varistor Accelerating voltage: 15 kV Magnification: × 2,000

#### 2.3 Elemental Analysis (X-ray Image)

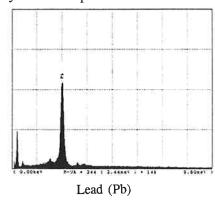
Characteristic X-rays are emitted from a specimen when an electron beam irradiates it. By detecting and analyzing the characteristic X-rays, you can identify the elements contained in the specimen (qualitative analysis). Also, you can determine the weight concentration of the contained elements (quantitative analysis). Electron beams are very finely focused. Therefore, by using the spot mode you can perform an elemental analysis of very small area on a specimen surface. Also, by scanning the electron beam over a specified area, you can obtain an averaged element concentration. Moreover, line analysis and areal analysis and X-ray image observation are possible.

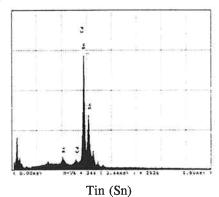
- a) First, observe the specimen surface with a secondary electron image and/or a backscattered electron image.
- b) Designate the point to be analyzed with the cross point of the cursor lines displayed on the image.



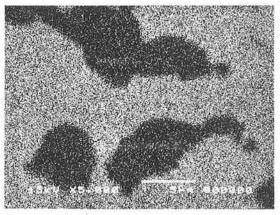


c) Position the electron beam at the designated point on the specimen, then perform a qualitative analysis. The spectra below were obtained by EDS analyses.

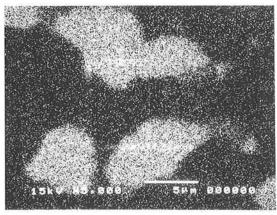




d) Observe the distribution of an element by selecting a specified characteristic X-ray.



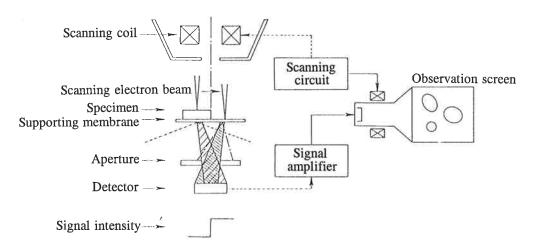
X-ray image of lead (Pb)



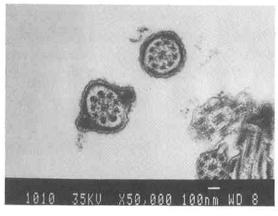
X-ray image of tin (Sn)

# 2.4 Inner Structure Observation (Transmitted Electron Image)

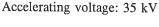
If a specimen is thin enough for the irradiated electron beam to penetrate it, electrons will be scattered during penetration of the specimen. The degree of scattering depends on the product of the density of the specimen substance  $(\rho)$  and the specimen thickness (t). Namely, electrons penetrating through a not dense area (small " $\rho \times t$ " value) will not be as largely scattered as than electrons penetrating through a dense area (large " $\rho \times t$ " value). Thus, a larger signal is detected through the not dense area. When a thin section is cut from a bulk specimen and observed with transmitted electrons, an information about the inner structure of the thin section can be obtained.

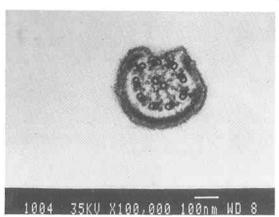


Principle underlying the SEM transmitted electron image observation



Specimen: Tail of a mouse sperm Instrument: General use FE-SEM/TED





 $\times$  50,000

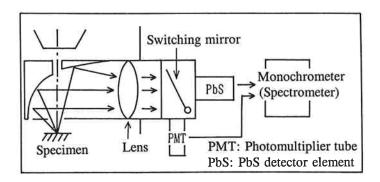
 $\times$  100,000

#### 2.5 Light Signal Observation (Cathodoluminescence Image)

Certain kinds of specimens (sulfide, oxide, mineral, semiconductor, etc.) emit visible light (0.3 to 2  $\mu$ m wavelength) when the hole created in the valence band by electron beam irradiation recombines with an electron. This emitted light is called cathodoluminescence (CL), and is used for detection of impurities or structural defects in a specimen, and for research using antibodies in biology. Cathodoluminescence detection using an SEM is especially useful compared with other methods. This is because, the character of a cathodoluminescence image can be studied in a micro-area and compared with a secondary electron image, backscattered electron images and other information.

An example of detector construction (spectrometer type):

A specially designed mirror effectively collects the cathodoluminescence emitted from a specimen, and directs it to outside of the specimen chamber. There, the light is analyzed by a monochrometer. Two detectors are provided for different wavelength ranges.

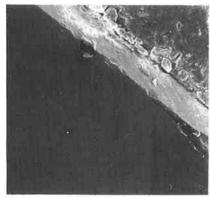


#### Specimen:

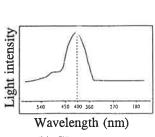
Artificial diamond

A secondary electron image of an artificial diamond is shown in (a). A uniform plane surface is observed.

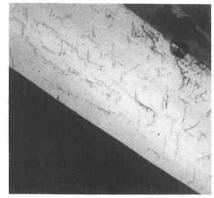
The cathodoluminescence spectrum in (b) was obtained from a point on the specimen. The peak intensity is seen at the 400 nm wavelength. (c) is the cathodoluminescence (CL) image of the same area as in (a) at the 400 nm wavelength.



(a) Secondary electron image



(b) CL spectrum

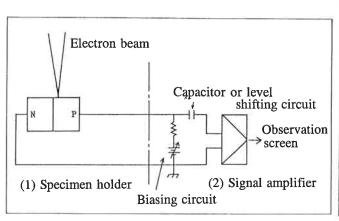


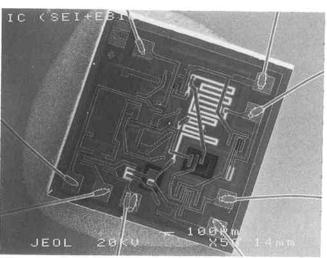
(c) Cathodoluminescence (CL) image

Secondary electron image and CL image of an artificial diamond

# 2.6 Electron Beam Induced Current (EBIC) Image (or Electromotive Force Image) and Potential Contrast (SEI) Image

(1) Electron Beam Induced Current (EBIC) Image or Electromotive Force Image
Irradiated electrons lose energy in interactions with the specimen's atoms during penetration of
the specimen. If a P-N junction exists in a semiconductor material, and electron-hole pairs are
generated in the depletion layer in the vicinity of the junction, these electron-hole pairs will be
detected as a current or a voltage. This is called Electron Beam Induced Current (EBIC) or
Electromotive Force, and is used for identification of defects or failure points in an IC chip. The
principle underlying this, and the obtained EBIC image are shown in (a) and (b), respectively.
The image shown in (b) is an SEI - EBIC combined image and shows the EBIC generated area
in dark and bright contrasts.





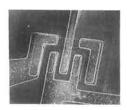
(a) EBIC circuit diagram (with a bias voltage)

(b) SEI - EBIC combined image

#### (2) Potential Contrast

If potential differences exist on a specimen surface, trajectories of the secondary electrons are affected, resulting in different image contrasts. This is called Potential Contrast, and is used for failure analysis of semiconductor devices.

The images below show the potential contrast observed in a simple transistor element by applying a bias voltage of 0 to 2 V between the collector and base.



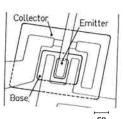
Vcd = 0 V



Vcd=0.5 V



Vcd = 2 V



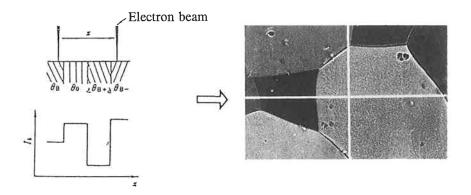
50 μ m

#### 2.7 Crystal Structure Analysis

#### (1) Electron Channeling Contrast (ECC)

When an electron beam irradiates a single crystalline specimen, the numbers of backscattered electrons, forward-scattered electrons, and absorbed electrons depend on the incident angle of the electron beam to the crystal orientation. In other words, the contrast in the secondary electron image and backscattered electron image depends on the crystal orientation. This is called Electron Channeling Contrast (ECC).

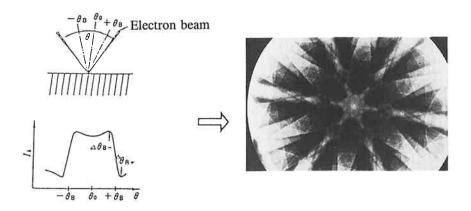
When an electron beam scans on a polycrystalline specimen, the ECC signal will vary as shown below.



#### (2) Electron Channeling Pattern (ECP)

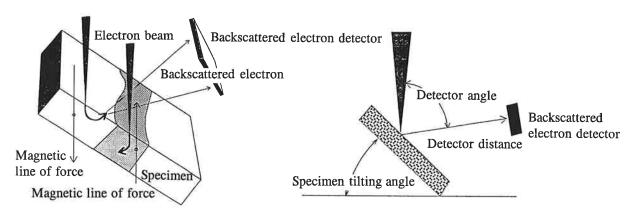
Usually, an electron beam scans a specimen surface in the X and Y directions. If the electron beam is positioned at a specified point on a crystalline specimen, and is rocked around the optical axis, i.e. the electron beam incident angle is varied across all the angles of the azimuth, the detected signal (usually backscattered electrons) varies with the angle between the electron beam and crystal lattice plane. In this case, a pattern showing the crystal structure and orientation of the specimen is obtained. This is the so called Electron Channeling Pattern (ECP), and is used for analyses of crystal structure and orientation.

An electron beam is scanned (rocked) in the  $\theta$  direction. The detected signal varies as shown in the figure, and an ECP is obtained on the CRT.



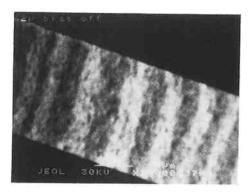
#### 2.8 Magnetic Domain Observation

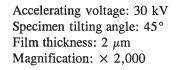
A small ferromagnetic segment in a specimen is called a Magnetic Domain. When an electron beam is irradiated on a specimen that includes magnetic domains, the trajectory of the generated secondary electrons and backscattered electrons may be affected by the small magnetic fields that are induced by the magnetic domain. This causes some variations in the detected signal, resulting in a magnetic domain contrast. The magnetic domain observation is used primarily for research on iron cores and magnetic films.

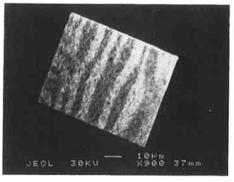


Principle of Type II Magnetic Domain contrast

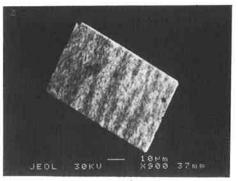
Specimen: Sendust film







Film thickness: 2  $\mu$ m Magnification:  $\times$  900

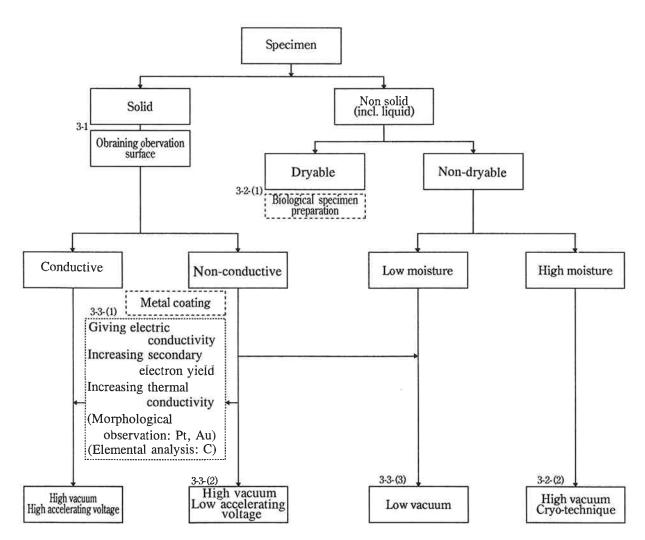


Film thickness: 5  $\mu$ m Magnification:  $\times$  900

# 3 Specimen Preparation and Observation Technique

Because of the wide variety of SEM application field, there are many kinds of specimen that are observed. Therefore, many different specimen preparation techniques have been developed depending on the object and the purpose of the research. In every case though, there are three major points that must be taken into consideration during specimen preparation. They are summarized as follows:

- 1. The specimen surface must be clean.
  - A SEM observes the surface layer of a specimen. Therefore, it is essential that the specimen surface be clean. In order to observe an inner structure, fracturing, polishing and cutting are commonly performed. In some cases, ion etching or chemical etching are done to remove an unwanted film coated on the specimen surface.
- 2. The original morphologic construction must be maintained. If a specimen containing water or gas is brought into a vacuum, the specimen may shrink or deform. Therefore, for biological specimens, fixation, dehydration and drying are done prior to observation. Cryo-techniques where the specimen is frozen to fix the morphological structure are often used for emulsion specimens.
- When a specimen is irradiated with an electron beam, some electrons are emitted from the specimen as secondary electrons and backscattered electrons. The rest of the irradiated electrons may be absorbed in the specimen. However, if the specimen has no electric conductivity, the absorbed electrons can charge the specimen. This charge causes many errors in observations. Metal coating, observations with low accelerating voltage, or observations under low vacuum (higher vacuum pressure) are done to prevent a specimen from acquiring an electrostatic charge.



#### 3.1 Obtaining a Clean Specimen Surface

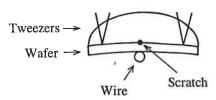
In all SEM works, the goal is to observe the specimen surface as it actually exists. Because of this, if a specimen surface has been contaminated, or covered by unwanted materials, the contaminants must be removed. Or, if the object to be observed or analyzed exists inside a specimen, the desired object must be disclosed on the specimen surface.

Also, although elemental analysis work by a SEM can be done with a roughly surfaced (natural surface) specimen, more accurate analysis results are obtained with a flat surfaced specimen. Several methods to disclose an object on the specimen surface and to obtain a flat specimen surface are introduce below.

#### (1) Fracturing

Measurement of the thickness of a coated film on a wafer is an example of an observation of the inside of a specimen. In such cases, fracturing can be used. The surface obtained by fracturing is flat enough for elemental analysis. (This technique cannot be applied to ductile materials such as gold or copper.)

(Specimen) Cross section of a wafer

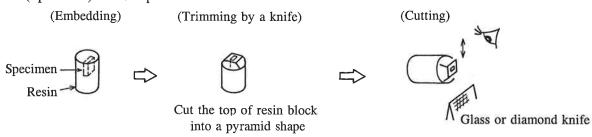


- (1) Make a small scratch at the point to be fractured.
- (2) Put a piece of thin wire at the opposite face of the scratch.
- (3) Put a pair of tweezers symmetrically about the scratch, and squeeze them. The wafer will fracture at the scratched point.

#### (2) Embedding and cutting

To observe an inner structure of biological or organic specimen, it is common to use the specimen preparation method of embedding and cutting. The specimen is first embeded in a resin block, then the resin block is cut by a microtome or similar tool (e.g., JEOL's JSD-700 Surfacer) until the object structure is disclosed on the cutting surface.

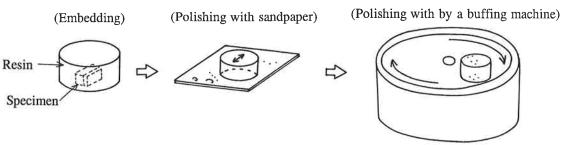
#### (Specimen) Rubber plate



#### (3) Embedding and polishing

To observe an inner structure of a metal or mineral specimen, it is common to use the specimen preparation method of embedding and polishing. The specimen is first embedded in a resin block, and then polished with sandpaper and then with a buffing machine.

(Specimen) Cross section of a plated metal



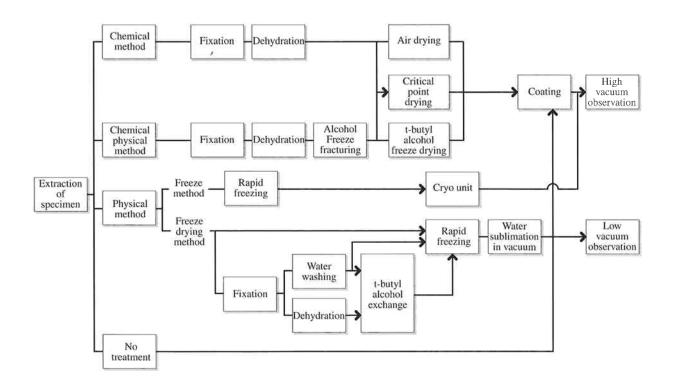
#### 3.2 Maintaining the Morphologic Construction

In order to observe a specimen surface as it actually exists, special care must be taken. This is especially true for hydrous specimens.

## (1) Specimen preparation techniques for biological specimen

Since biological tissues include a lot of water, it is impossible to use them directly for SEM observation as the SEM is a vacuum instrument. In order to observe such specimens with a SEM, you must fix the composed protein and lipid substances in their living morphological shape by chemical or physical methods. Then, the specimen must be dehydrated.

These specimen preparation procedures are basically as shown below. For the details of each step and the proper treatment for different type of specimens, refer to the appropriate technical books.



Fixation : This is done to fix the composed protein and lipid substances in their living

morphological shape, so as to prevent the specimen from deformation when in a

vacuum.

Dehydration: In this step, alcohol or acetone is substituted for water so as to remove water from the

specimen.

Drying: This removes liquid substances from the specimen.

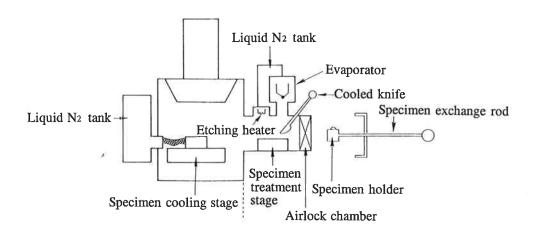
Coating : This deposits a metal film on the specimen surface so that it is electrically conductive.

#### (2) Cryo-observation

Substances such as foodstuffs, cosmetics, paints and emulsive specimens include much water or oil, but can not be treated with chemical treatments and drying processes. In such cases, so called cryo methods can be used. Namely, these liquid specimen are frozen by liquid nitrogen to fix the structure.

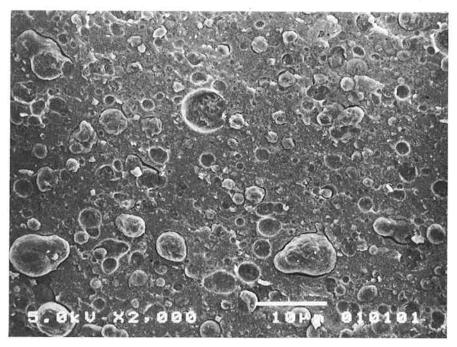
The basic procedures are as follows:

- a) Place and adhere a small amount of specimen on the specimen holder.
- b) Set the specimen holder into the specimen exchange rod, and immerse the specimen holder into liquid nitrogen to freeze it.
- c) Insert the specimen holder into the specimen treatment stage, and perform the appropriate specimen treatment.
- d) Transfer the specimen holder to the specimen cooling stage, and then observe it.



Specimen: Processed cheese

Specimen: Processed cheese Magnification: × 2,000

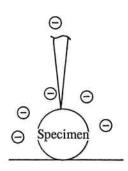


Note: For specimens which do not include so much water and oil as emulsive foodstuffs and cosmetics, a low vacuum SEM (LV SEM) may be suitable for observations.

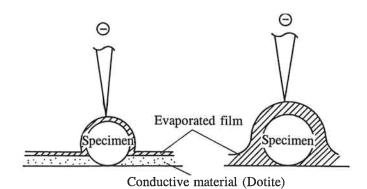
#### 3.3 To Prevent a Specimen from Acquiring Electrostatic Charge

#### (1) Metal Coating

For an electrically conductive specimen, observation of the specimen without metal coating is the best method. For a non-conductive specimen, however, metal coating is usually applied to give the specimen electrical conductivity. This decreases the specimen's capacity to acquire an electrostatic charge, and increases the yield of secondary electrons. The important thing to remember when applying a metal coating is that the coat of metal film must be as thin as possible.



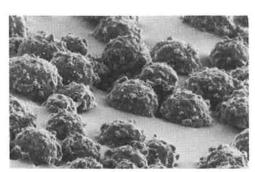
 Non-coating: A low accelerating voltage is used to prevent the specimen from acquiring an electrostatic charge.



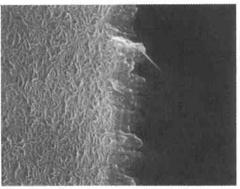
o) Appropriate coating: Details of the specimen surface morphology should remain. A higher accelerating volt-

age can be used.

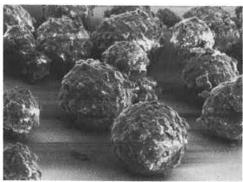
c) Heavy coating: A higher accelerating voltage can be used, but details of the specimen surface morphology are covered by the thickly coated film.



(a) Tilting angle 45°



(a) Appropriate coating

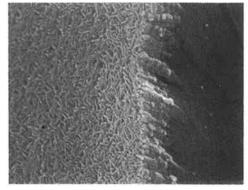


(b) Tilting angel 45°

 $(5kV, \times 2,000)$ 

Specimen: Toner

Fig. (a) was obtained by spreading and pressing toner particles on adhesive tape. No electrostatic charge is observed even under a high accelerating voltage. In Fig. (b), the toner particles were spread on a nail acrylic surface, and electrostatic charge is observed.



(b) Heavy coating

 $(15 \text{ kV}, \times 10,000)$ 

Specimen: Cross Section of a glass plate coated with a metal

Since glass is a non-conductive material, metal coating is necessary to observe it with an SEM. However, if the coated metal film is very thick, fine structures on the cross section are covered by the coated metal.

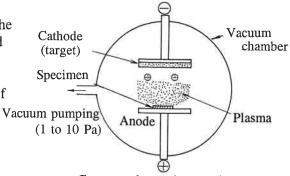
#### **Coating Devices**

There are several methods for coating metal on a specimen surface.

1. Ion Spattering Device (Low vacuum - several Pa order)
The metal used to coat the specimen is placed on the
cathode plate. Ions generated by glow discharge hit the
plate and eject the metal atoms from it. These ejected
metal atoms deposit on the surface of the specimen,
which has been placed on the anode plate.

Thickness of the coated film depends on the amount of

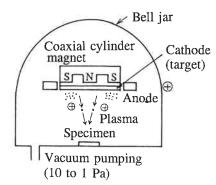
ion current and sputtering time.



Counter electrode type ion sputtering device

2. Magnetron sputtering device (Low vacuum - several Pa order)

This is another kind of ion sputtering device. In a magnetron sputtering device, the cathode and anode are coaxially arranged, and a coaxial cylinder magnet is placed behind the cathode. The advantage of this construction is that specimen damage due to electron or ion bombardment is reduced.

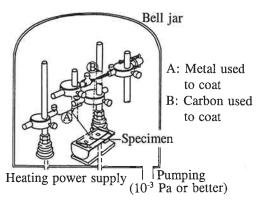


Magnetron sputtering device

3. Vacuum evaporation device (High vacuum - about 10<sup>-3</sup> Pa)

In this kind of coating device, a piece of the metal used to coat the specimen is wound around a tungsten wire, or put into a tungsten basket. When the tungsten is heated by an electric current, the metal evaporates and deposits on the specimen surface.

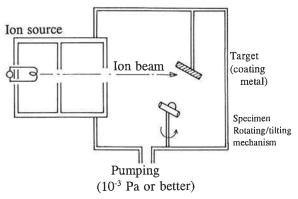
Generally, the specimen is rotated and tilted during the evaporation to obtain an uniformly coated film. Carbon coating and aperture cleaning can also be done with this device.



Vacuum evaporation device

4. Ion beam sputtering device (High vacuum - 10<sup>-3</sup> Pa)
In an ion beam sputtering device, an inert gas ion beam is irradiated on the metal used to coat the specimen.
The sputtered metal is deposited on the specimen. Very finely coated films can be obtained with this device.

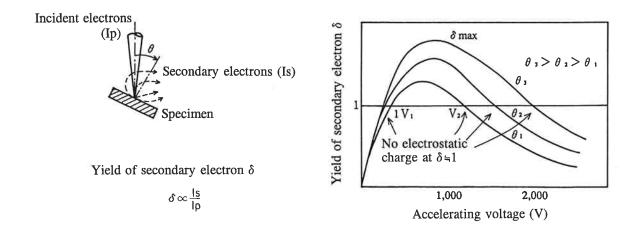
Specimen damage is also very low, because the ions do not hit the specimen surface.



Ion beam sputtering device

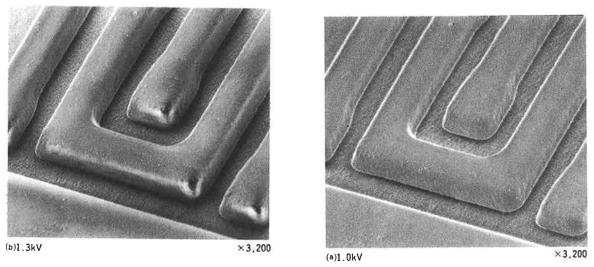
#### (2) Low accelerating voltage observation

In order to observe a non-conductive specimen that has not been coated with metal, it is necessary to balance the amount of electrons incident into and emitted from the specimen.



As shown in the figure, no electrostatic charge is expected when Is/Ip≒1.

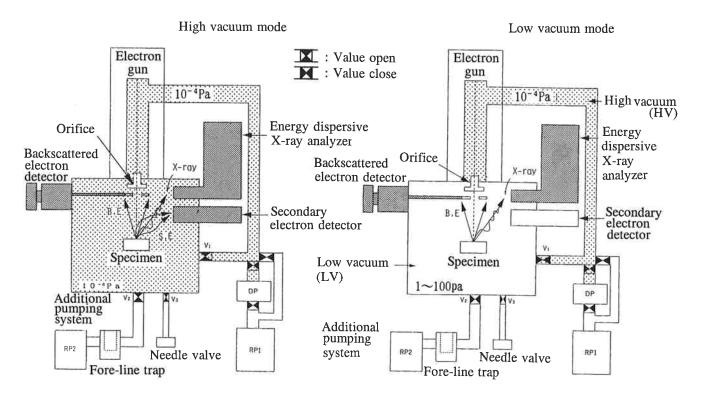
Shown below is an example of a photoresist on an IC.



Specimen: Photoresist on an IC pattern Electrostatic charge is eliminated at an appropriate accelerating voltage.

#### (3) Low vacuum observation

Generally, an electron optical column and a specimen chamber of a SEM are evacuated in a high vacuum. The Low Vacuum SEM (LV SEM) is a SEM on which an additional pumping system has been added so that the specimen chamber can be maintained in a low vacuum state while the column is in a high vacuum state. In such a condition, gas molecules surrounding the specimen are ionized by the incident electron beam and the electrons emitted from the specimen. This results in neutralization of the electrostatic charge on the specimen surface. Thus, a non-conductive specimen can be observed or elemental analysis can be carried out without metal coating. A hydrous specimen or specimen containing oils can also be observed with an LV SEM.



Comparison of detected signals

	High vacuum	Low vacuum
Secondary electron (SEI)	0	×
Backscattered electron (BEI)	0	0
Elemental analysis (EDS)	0	0
(WDS)	0	×
Cathodoluminescence	0	0

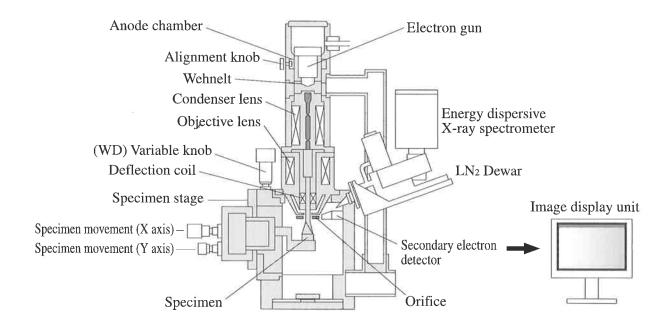
Comparison of the applications of high and low vacuum

High vacuum	Low vacuum
<ul> <li>High resolution observation by SEI</li> <li>Trace element analysis and deconvolution of superimposed spectra by WDS</li> <li>Micro-area analysis</li> </ul>	<ul> <li>Observation and analysis of non-conductive specimens without metal coating</li> <li>Observation and analysis of hydrous or oily specimens</li> <li>Observation and analysis of outgassing specimens</li> <li>Insitu observation during heating or tensile test of non-conductive specimens</li> </ul>

# 4. Functions of SEM's Individual Components

#### 4.1 Here is How SEM Operates

The electron beam produced by the electron gun is finely focused and irradiates the specimen surface. The specimen surface is scanned point-by-point with this small (diameter) electron probe in both X and Y directions. Secondary and backscattered electron signals generated by the electron beam irradiation are collected by the detector. The detected signals are amplified and used to modulate the brightness on the CRT brightness. Then, secondary and backscattered electron images are produced and displayed on the image display unit. Forming a small electron probe and detecting the signals efficiently make it possible to obtain high resolution.

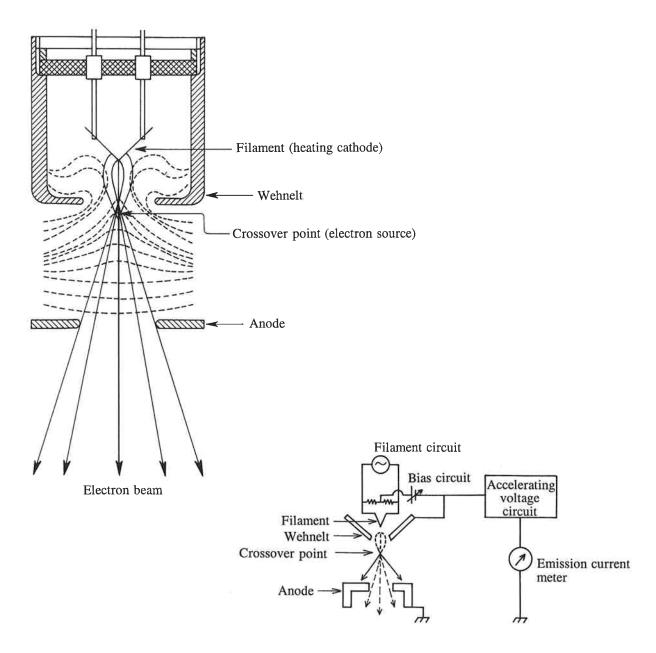


Standard Configuration of SEM

#### 4.2 Generation of Electron Beam by Electron Gun

The electron beam of the electron microscope is generated by the electron gun. The electron gun is classified roughly into two types: the thermionic emission gun and the field emission gun.

The thermionic emission gun consists of three electrodes: a filament, Wehnelt, and an anode. Firstly, thermoelectrons are emitted from the filament, then an electron beam is produced by giving an energy to the thermoelectrons, by applying an accelerating voltage between the filament and anode. During this process, the thermoelectrons are collected at one point by the bias voltage applied between the filament and Wehnelt. This point is called "crossover point". The filament is available in two types: the tungsten filament shaped like a hair-pin and the  $LaB_6$  cathode filament. The  $LaB_6$  filament, having a small crossover point, provides high brightness of source. The principle of the thermionic gun is shown in the figure below:



A SEM fitted with a field emission electron gun can provide higher brightness and higher resolution.

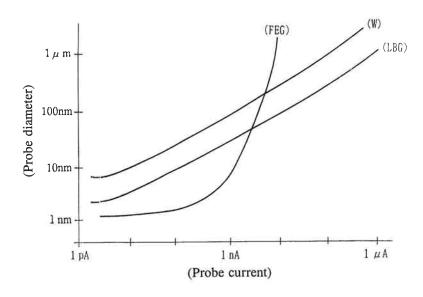
# (1) Comparison of the features of individual electron guns

For the SEM, the three types of electron guns shown in the table below are used generally.

	Hairpin filament (W)	LaB <sub>6</sub> filament (LBG)	Field emission (Cold cathode FEG)
Shape	V⊛	LeB.	Y®
Brightness (A/cm² sr) (Acc. volt. 20 kV)	5 × 10 <sup>4</sup>	2 ~ 3 × 10 <sup>5</sup>	~ 10 <sup>7</sup>
Size of electron source	20 μm	10 μm	5 ~ 10 nm
Current stability (Short-term) (Long-term)	0.5% or less	1% or less 2 to 3% or less/hr	5% (10 <sup>-8</sup> Pa) 5% or more/15 min
Service life	50 to 100 hr	300 to 500 hr	One year or more
Operating vacuum degree (Pa)	10 <sup>-4</sup>	10 <sup>-2</sup>	10 <sup>-8</sup>
Operating temperature (K)	2800	1800	Room temperature
Main features	Stability Large current	High resolution at large current	High resolution

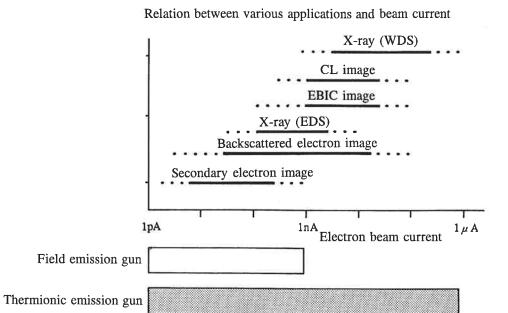
#### (2) Probe current and probe diameter

The probe diameter changes with the probe current approximately in the following relation.

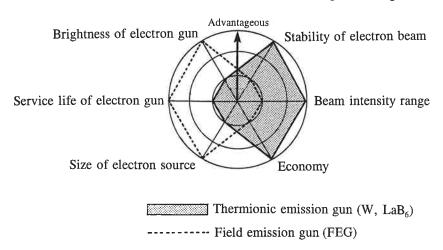


The SEM is used for diverse applications, depending on the purpose and the information you wish to gain. Its accelerating voltages range from several hundreds of volts to several tens of kV, allowing you to select the optimum accelerating voltage suited for your observation purpose. The probe current is also widely variable from  $10^{-6}$  to  $10^{-12}$  A.

Generally, currents of  $10^{-11}$  to  $10^{-12}$  A are used for observation of secondary electron images, whereas  $10^{-7}$  to  $10^{-8}$  A are needed for elemental analysis with WDS, observation of cathodolumine-scence and EBIC images.

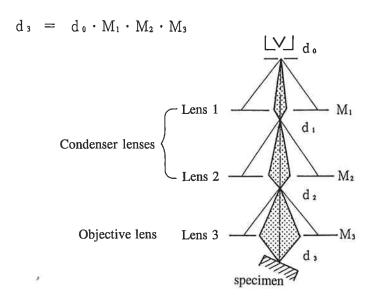


The figure below compares the main features of the SEM fitted with a thermionic emission gun and the SEM fitted with an FEG (field emission gun). As seen from the figure, it may safely be said that the FE SEM is suited to high magnification morphological observation, whereas the SEM fitted with a thermionic emission gun is suited to applications that require a large current.



#### 4.3 Focusing the Electron Beam by Lenses

The electron beam diameter (probe diameter = d3) on the specimen surface is given by the following formula including the diameter (cross over = d0) of the electron beam generated from the electron gun:

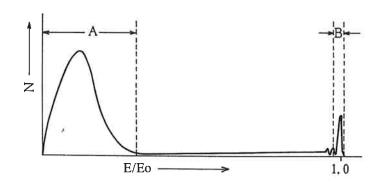


where M1, M2 and M3 are the demagnification rates of the individual lenses.

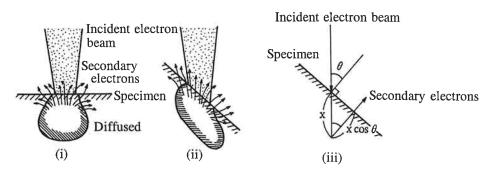
#### 4.4 Detection of Secondary Electrons

The signal that is used the most frequently in the SEM is called the secondary electron signal. (a) shows the energy distribution of backward scattering electrons that are generated when the specimen is illuminated with an electron beam. On the ordinate is plotted the number (N) of electrons generated, and on the abscissa is plotted the ratio of the energy of the generated electrons (E) to the energy of incident electrons (Eo). Region A is a low-energy region ranging from 0 to several tens of eV, and the electrons in this region are generally called secondary electrons. Region B is a high-energy region, and the electrons in this region are called backscattered electrons.

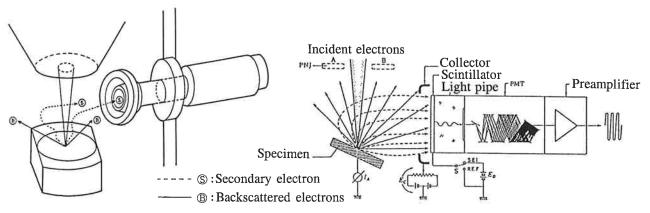
- (b) shows the dependence of generated secondary electrons on the specimen tilt angle. Assuming the amount of secondary electrons as Is and the proportional coefficient as K, we obtain  $I_s = K \cdot 1/\cos\theta$ . It is seen that increasing the tilt angle increases the amount of secondary electrons.
- (c) shows the principle diagram of the secondary electron detector. In order to collect low-energy electrons drawn in dotted lines, a voltage called a "post acceleration voltage" (approx. 10 kV) is applied to the scintillator. Since secondary electrons emitted in all directions are collected, an image such as by shadowless illumination is obtained.



(a) Energy distribution of backscattered electrons.



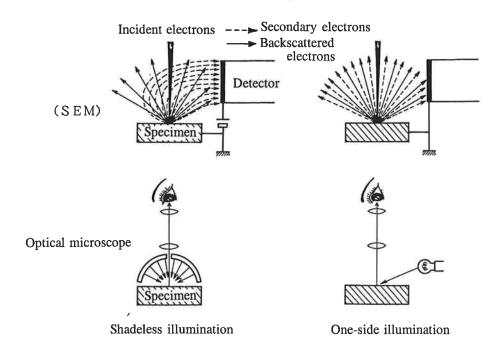
(b) The amount of secondary electrons generated depends on the specimen tilt angle.



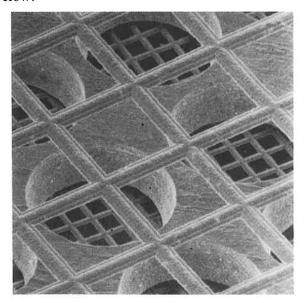
(c) (i) Secondary electron detection.

(ii) Secondary electron detector

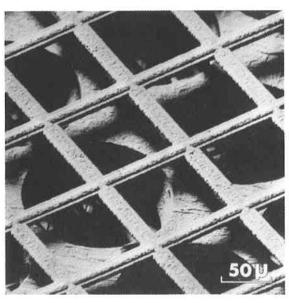
The figures below compare an image obtained with secondary electrons, which have low energy, and an image obtained with backscattered electrons, which have high energy as will be mentioned later. Since the secondary electron image is formed by secondary electrons emitted in all directions, a shadowless illumination image such as with an optical microscope is obtained. Since the backscattered electron image is formed only by those electrons emitted toward the detector, a one-side illumination image such as with an optical microscope is obtained.



A secondary electron image and a backscattered electron image of superposed grids are compared below:



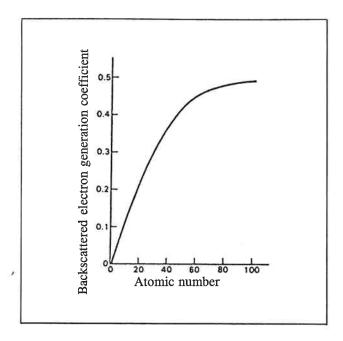




Backscattered electron image

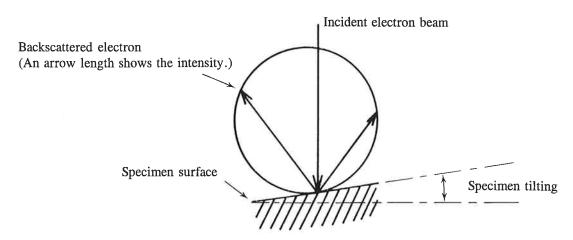
#### 4.5 Detecting Backscattered Electrons to "Aid in Analysis"

The secondary electron image shows a contrast that depends mostly on the surface morphology, whereas the backscattered electron image depends largely on the specimen composition, showing a contrast depending on the mean atomic numbers of the specimen constituent elements. If the specimen surface is even, the backscattering coefficient of incident electrons is larger for larger atomic numbers as shown in (a), thus resulting in a brighter image in the composition image mode.



(a) Backscattered electron generation coefficient.

With a sample having a uniform composition and an uneven surface, backscattered electron intensity distribution is as shown in (b).

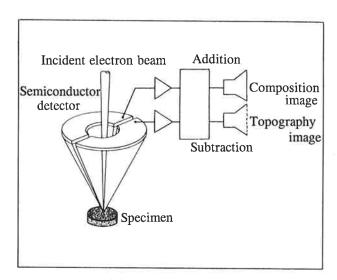


(b) The intensity of the backscattered electron signal depends on the specimen tilt angle.

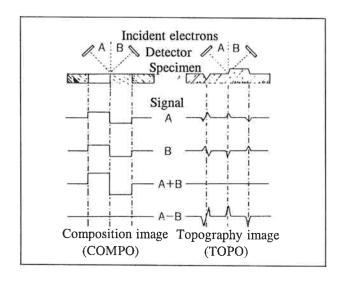
The backscattered electron detector installed on each of JEOL's SEMs is an annular type semiconductor detector with two or four divided detection elements. The specimen composition, topography, and stereoscopic feel can be emphad by operational amplifiers that process the signals detected by the individual elements.

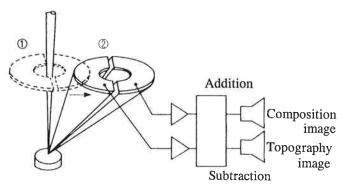
Three types of images can be obtained from backscattered electrons

The backscattered electron detector can be used for both high and low vacuum (refer to 3.3 (3)).



Specimen and backscattered electron detector.

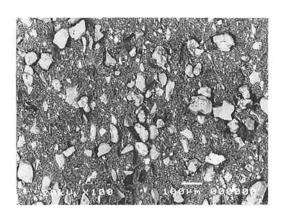




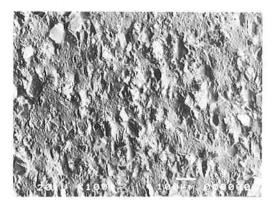
A stereoscopic (SHADOW) image is obtained by shifting the detector to position (2).

Backscattered electron signals and three types of images.

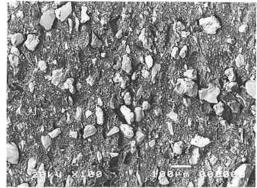
Composition image (COMPO) Represents the composition differences (Z) in the specimen.



Topography image (TOPO)
Represents the topography of the specimen surface.



Stereoscopic image (SHADOW)
Emphasizes stereoscopic appearance by synthesizing composition and topography signals.

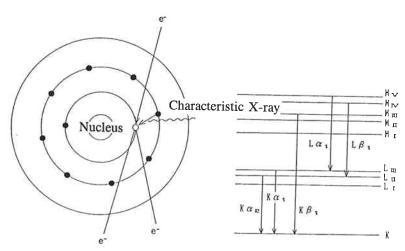


Specimen: Sand-containing rubber eraser Magnification: ×100

# 4.6 Detection of X-rays

#### (1) Generation principle of X-rays

By analyzing the characteristic X-rays generated when the specimen is irradiated with an electron beam, we can identify the elements that constitute the specimen (qualitative analysis) or make quantitative calculation of their weight concentrations (quantitative analysis).



#### · Characteristic X-rays

When a specimen is irradiated with an electron beam, inner shell electrons of specimen atoms are ejected, with the result that vacancies are formed at their original positions. To fill the vacancies, outer shell electrons transfer to the vacancies and their excessive energy is emitted in the form of electromagnetic wave. Since the energies of individual shell electrons are determined by the type of elements, the X-rays generated by electron transfer are characteristic of the elements generated by electron transfer, thus the name of characteristic X-rays.

There are two methods of analyzing characteristic X-rays: one by the wave-length dispersive X-ray spectrometer (WDS) and the other by the energy dispersive X-ray spectrometer (EDS). WDS has been put to general use since it was developed by R. Castaing et al. of France around 1960, in a form fitted to the electron probe microanalyzer. On the other hand, EDS has come into wide use since it was developed by J.C. Russ, et al. At present, EDS is used more widely than WDS because of its operational ease and because it allows overall qualitative analysis to be carried out in a short period of time. Both have merits and demerits, making it necessary to selectively use them depending on the purpose.

Their principal features are as shown in the table below:

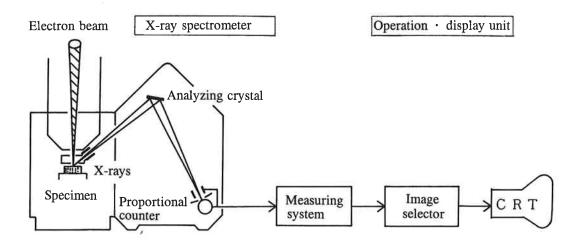
Comparison items	WDS	EDS	
Measurable element range	<sub>4</sub> Be*1) ~ <sub>92</sub> U	<sub>5</sub> B*2) ~ <sub>92</sub> U	
Measurement method	Wavelength dispersion system by analyzing crystals	Energy dispersion system by Si(Li) semiconductor detector	
Resolution	$\lambda = 0.7 \times 10^{-3} \text{nm}$ $(E = 20 \text{eV})$	$E = 150 \text{eV}$ $(\lambda = 6 \times 10^{-3} \text{nm})$	
Measuring speed	Δ	0	
Multi-element simultaneous measurement	Δ	0	
Specimen damage - contamination	Δ	0	
Detection limit	50 ~ 100 PPM	1,500 ~ 2,000 PPM	
X-ray detection rate per unit current ratio	Low	High	

<sup>\*1)</sup> For Be, an optional crystal is needed.

<sup>\*2)</sup> A detector for light elements is needed.

#### (2) Wavelength dispersive X-ray spectrometer (WDS)

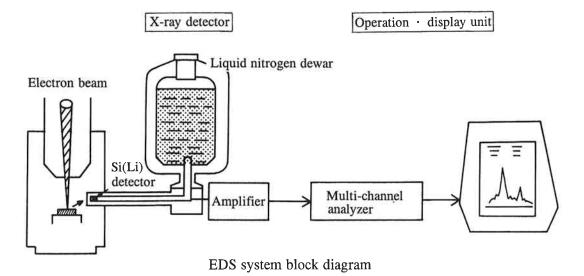
X-rays generated from a specimen have different wavelengths depending on elements. X-rays emitted from the specimen are dispersed in their wavelength by the analyzing crystal and the X-rays having the specified wavelength are detected by a proportional counter. After they are processed by an electric circuit such as an amplifier, X-ray intensities are counted and indicated. Different wavelengths are detected by using analyzing crystals differing in position and lattice spacing.



WDS system block diagram

#### (3) Energy dispersive X-ray spectrometer (EDS)

Characteristic X-rays emitted from the specimen have energies characteristic of individual elements. With the EDS, X-rays are detected by an Si(Li) semiconductor detector placed at the tip of the detector. The height of current pulses generated by X-ray illumination is proportional to the energy of incident X-rays. By calibrating the multi-channel analyzer in advance using a standard specimen, characteristic X-rays from an unknown specimen can be measured for element identification.



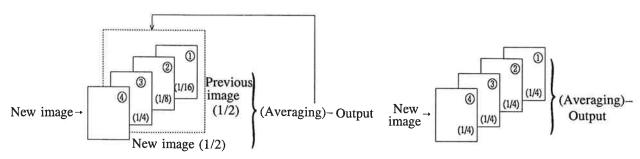
#### 5. New Functions of SEM

#### 5.1 Image Enhancing

SEM signals are originally analog signals. However, converting them into digital signals facilitates observation in an unshaded room. The SEM can be used more easily by means such as image storage into memory, image accumulation, and noise reduction by averaging.

(Recurcive filter)

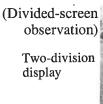
(Accumulation mode)

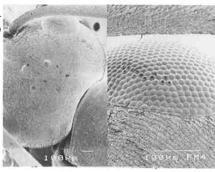


- 1. (1) to (4) show the order of input.
- 2. ( ) indicates the weight when carrying out averaging; the weights of older images reduce with the input of a new image.
- 3. This function continues automatically.
- 1. 1 to 4 show the order of input
- 2 The specified number of input images is accumulated with uniform weight.
- 3. For acquiring new images, it is necessary to restart the function.

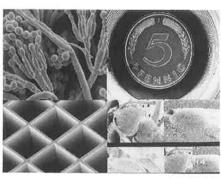
#### 5.2 Image Processing

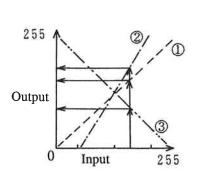
It is possible to divide a image screen into two or four parts for image comparison and to make pixel calculation.

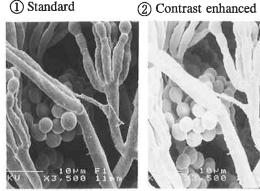




Four-division display

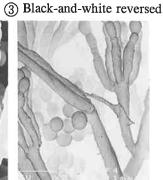






Standard





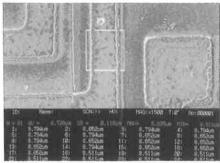
## 5.3 Image Analysis

More quantitative observation and judgement can be done by analyzing SEM images.

## (1) Image analysis

Synthesis and smoothing of images and measurement of image areas and perimeters are possible.

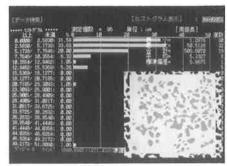
#### Line width automatic measurement



Specimen: IC × 1,500

Six different line width measuring functions using a cursor or frame are provided. Measured values include absolute values, mean values, standard deviation values, maximum values and minimum values. All of them can be directly read out in microns.

#### Particle size measurement



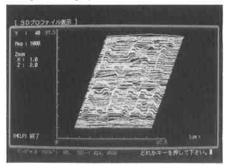
Specimen: Solder

More than 50 measurement items such as the area, perimeter, and shape factor are provided and continuous measurement is possible automatically.

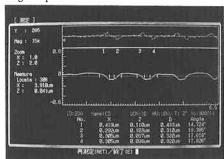
## (2) 3D height measurement

The surface heights and depths on a specimen are measured. Bird's eye view display and contour map display are possible.

#### 3-D profile display



Hight/depth measurement

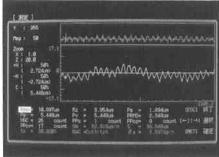


Specimen: Compact disk × 15,000

## (3) Surface roughness measurement

The surface roughness of a specimen is measured on the basis of roughness signals obtained with a 4-division backscattered electron detector. 34 items conforming to JIS/ISO standards can be measured.

## Surface roughness measurement



Specimen: Polished metal surface × 50

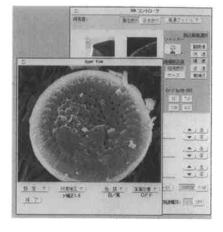
A total of 34 items are measured in the nanometer order, including eight types of surface roughness measurement such as 10 point average roughness (Rz) based on JIS/ISO standards, center line average roughness (Ra) and average peak-to-peak distance (Sm).

## 5.4 New Image Filing System

So far, images obtained from the specimen have been stored as photos. However, with the increase of photos, their storage and search has become difficult. The image filing system allows a large number of SEM images to be filed into and called up from an magnetooptical disk easily.

## (1) Observation

SEM images can be observed also on the computer screen by the latest video technique. Besides, it is possible to change the size of the observed image or display the image in colors.



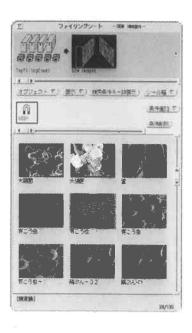
## (2) Filing

Top priority is given to the ease of filing. Almost all operations for filing can be easily carried out by mouse operation using the most advanced graphical user interface.



## (3) Image search

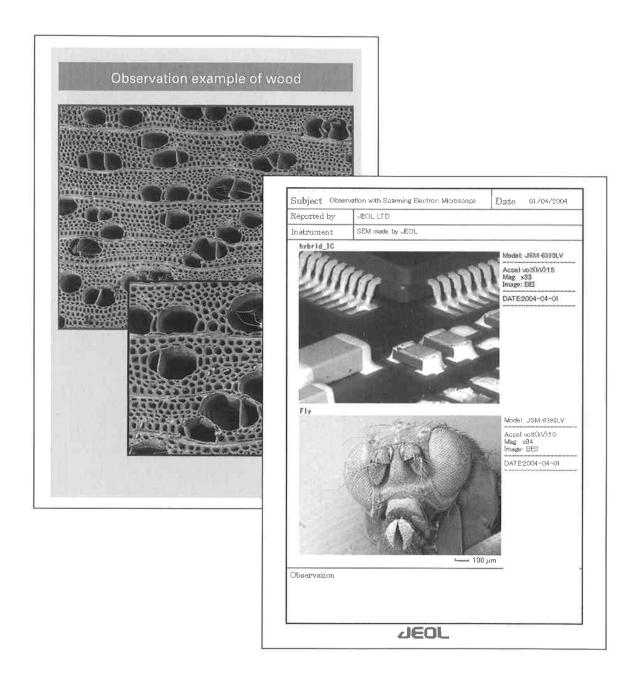
Image search can be carried out easily using the most advanced database and graphical user interface. By setting keywords, desired images can be searched rapidly and called up on a sheet.



## (4) DTP (Desk Top Publishing) report making

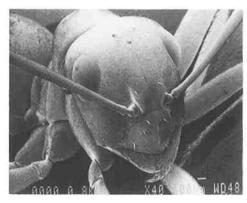
This function allows report making jobs such as image layout, illustration/drawing making, and editing to be performed freely.

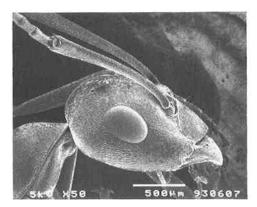
(Example of report prepared)



## 5.5 Observation from Various Angles

The SEM's stage is constructed so as to allow the specimen to be held stably and observed from various angles. Namely, it can be rotated and tilted, as well as moved in the X, Y, and Z directions.

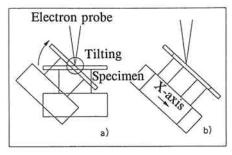




(The specimen is observed from different angles by rotation and tilting.)

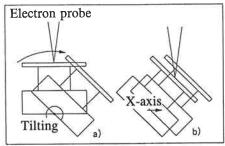
## **Eucentric and Noneucentric Stages**

A specimen stage that is designed so that the specimen tilt axis agrees with the observation point, is called the eucentric stage. It's feature is that the field of view is not shifted or the focus is not largely changed by specimen tilting.



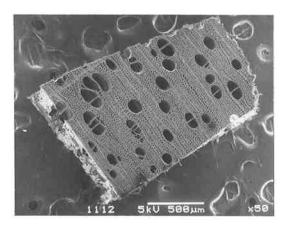
With eucentric stage

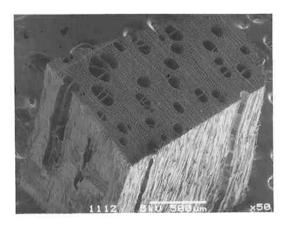
- a) The electron probe position (observation point) on the specimen is not shifted by specimen tilting.
- b) The focus is not changed when moving the specimen in the X-direction in a tilted state.



With noneucentric stage

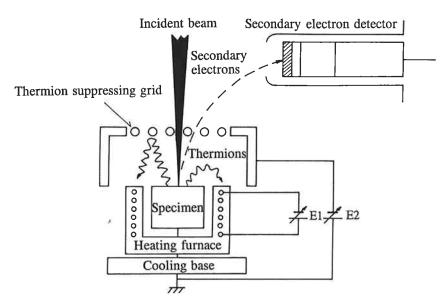
- a) The electron probe position on the specimen is shifted with specimen tilting.
- b) The focus is changed when moving the specimen in the X-direction in a tilted state.





## 5.6 Specimen Observation While Heating It

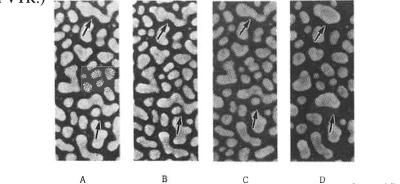
Specimen observation with the SEM is generally made in a static state kept at room temperature. But it is also possible to heat the specimen during observation, for recording its state changes dynamically. Besides, by heating the specimen, contaminants can be prevented from sticking to its surface. When morphological changes of the specimen are slow, photography can be done at a standard scanning speed, but when the changes are rapid, image recording can be carried out using a TV-rate scanning speed, by connecting video signals to a VTR, etc. As an example, changes of Au particles sputter coated on a carbon plate are shown below.



Changes of Au particles by heating:

Shown below are changes in a secondary electron image of sputtered Au particles on a polished carbon plate, which were caused by heating the particles in a SEM. The same area was measured at four varying temperatures (A, B, C, D). It is seen that the Au particles increased in roundness with temperature, with their surface areas decreasing.

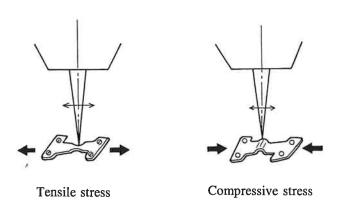
(Since Au particles change rapidly, observation is made using a TV rate scanning speed and recording is done on VTR.)



Temperature (350K) (390K) Magnification:  $\times$  100,000

## 5.7 Observation of Changes of Specimen under Stress

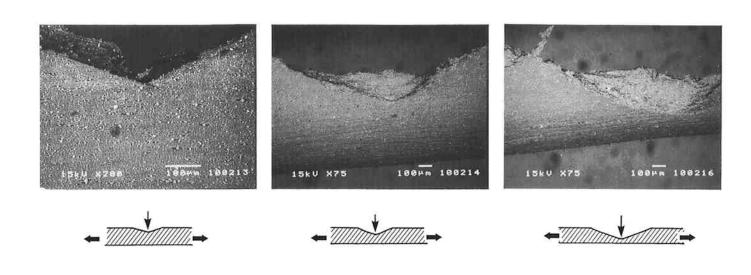
SEM observation is ordinarily made with the specimen kept in a still state. But, depending on purposes, observation is made on the growth of cracks or specimen distortion while applying a tensile or compressive stress to the specimen. In such cases, specimen changes are so rapid that standard photographing speed cannot be used. Therefore, recording is done on a VTR while observing at a TV rate scanning speed. Here, a high-vacuum SEM is used for observation of conductive specimens, but a low-vacuum SEM is suited for observation of coated nonconductive specimens, because the nonconductive portions get exposed when the specimen is destroyed by the application of a stress.



Tensile test of coating on lead wire (with low-vacuum SEM):

Tensile stress: 3.8 kg Vacuum pressure 15 Pa

Photographing magnification: × 75



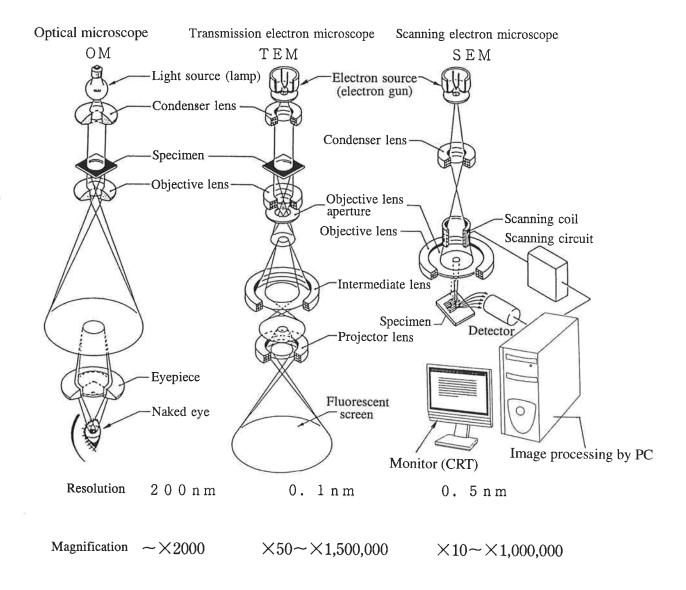
# 6. Comparison of Scanning Electron Microscope with Optical Microscope and Transmission Electron Microscope

## 6.1 Comparison by Principle Diagrams

Below are compared three types of microscopes that are used generally.

The optical microscope (OM) taken up here is for observing transmitted images. With this OM, the light passing through a sufficiently thin specimen is magnified through glass lenses. When observing a specimen with a transmission electron microscope (TEM), a specimen that is made thin enough to transmit an electron beam is prepared, and a beam which has passed the specimen and scattered is observed after being magnified by electron lenses.

In scanning electron microscope (SEM) observation, an electron beam that is finely focused by electron lenses is scanned over the specimen and the brightness on the Monitor (CRT) is modulated by the signals obtained.



Standard Configuration of SEM

## 6.2 Comparison of SEM Image with OM and TEM Images

We compared photos of glomerulus obtained with an OM, SEM and TEM. With a bulk-state specimen, a SEM image has a large focal depth as compared with an OM image.



OM image:

 $\times$  200

Specimen: Rat's glomerulus (free cell)

O<sub>s</sub>O<sub>4</sub> stained

OM image:

 $\times 200$ 

Specimen: Rat's glomerulus (section)

HE stained

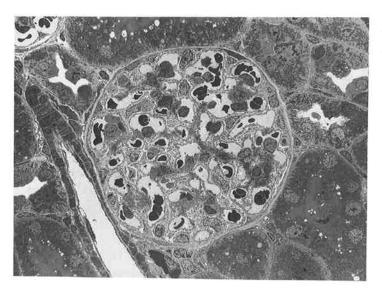


SEM image: × 750 (secondary electron image)

Specimen: Rat's glomerulus

Surface morphology of

the bulk-state specimen is observed.



TEM image: × 750

Specimen: Rat's glomerulus

The internal structure of

a thin section specimen is observed.

## 6.3 Differences between SEM and OM

The biggest difference between the OM and SEM is that the former uses light for image magnification, whereas the latter uses a scanning electron beam. Both depend on wavelength for resolution, and the SEM uses an electron beam of a short wavelength to raise resolution. Since an electron beam can be stably obtained only in vacuum, a SEM specimen is observed in vacuum. Namely, although the OM allows both solid and liquid specimens to be observed in the atmosphere, the SEM requires solid specimens that are not deformed in vacuum.

		Optical microscope (OM)	Scanning electron microscope (SEM)
Specimen	State	In the atmosphere Solid Liquid	In vacuum Solid
	Conductivity	Not needed	Needed (high-voltage observation) Not needed (low-vacuum observation)
Features	Focal depth	Small	Large

## 6.4 Differences between SEM and TEM

Both are electron microscopes

Generally, the SEM is used to observe a bulk specimen, and the image obtained shows the morphology of its surface. With a rugged-surfaced specimens, therefore, its morphology may change depending on the direction from which it is viewed.

With the TEM, it is necessary to make the specimen thin enough to transmit the electron beam, so thin sections cut from a bulk specimen are observed. This makes it possible to obtain inside information on the specimen. Obtainable information differs with sections obtained and with the direction in which they are cut out.

	Scanning electron microscope	Transmission electron microscope
Ultimate resolution	0.5 nm (5Å)	0.1 nm (1 Å)
Accelerating voltage	0.2 ~ 50 kV	20 ~ 1250 kV
Specimen shape	Bulk	Thin film
Obtainable image	Surface topography	Specimen inside
	Example: Cone with a ball in it	Example: Cone with a ball in it
	The image appears different with the direction from which it is seen. Image interpretation is simple.  (a) ((•) From top (b) From below (c) From side	© <u></u>
Features	Provides images appearing stereoscopic	Provides images having high resolution

## 7. Description of Terms

#### Abbe's theory

Even if the perfect lens of aperture angle  $2\alpha$  were used, the image of a point source would not be a point but a blurred circle. The diameter of the circle is given by  $d=0.61\lambda/n \cdot \sin\alpha$ , where  $\lambda$  is the wavelength of the light, n is the refractive index of the medium, and  $\alpha$  is a half of the aperture angle of the lens, respectively. This equation is called "Abbe's equation", and gives the theoretical limit of resolution.

#### Absorbed electrons/Absorbed electron image

Absorbed electrons are electrons that irradiate a specimen, then lose their energy and are absorbed by the specimen. The image obtained from these absorbed electrons is called an Absorbed Electron Image. This signal is influenced by the average atomic number and surface roughness of the specimen, and is complementary to the backscattered electron image.

#### Accelerating voltage

This is the voltage used to accelerate the electrons emitted from a cathode in order to irradiate them on a specimen.

#### Air lock mechanism

This mechanism is used to exchange a specimen without any vacuum deterioration in the specimen chamber and electron optical column. There are two methods of exchanging a specimen. One is a drawout method in which the entire stage is withdrawn and air is introduced to the entire electron optical column, causing the vacuum to be broken. And the other is an air-lock method in which the sample is exchanged through a small chamber (pre-evacuation chamber) which is separated from the overall electron optical column by means of an isolation valve. In this latter method, air is introduced only to the pre-evacuation chamber when exchanging the specimen, hence the inside of the electron optical column is not exposed to the atmosphere. This method is thus superior from the viewpoint of maintaining the inside of the optical column in a clean vacuum.

#### **Analytical SEM**

The abbreviation of Analytical Scanning Electron Microscope. A SEM is originally used to observe the surface morphology of an object. Although it cannot perform elemental analysis with a pure SEM function only, if X-ray detectors which detect the characteristic X-rays emitted from the specimen are installed in the SEM, it becomes possible to identify and quantitatively analyze the chemical composition in micro-areas on the specimen while observing the SEM image. A scanning microscope which is provided with this function is called Analytical SEM.

## Angstrom (Å)

Unit of length (1 Å = 0.1 nm =  $10^{-10}$  m)

#### Anode

The anode is an accelerating electrode which gives the specified energy to the thermoelectrons that are emitted from a filament (cathode).

#### **Astigmatism**

If the bore of pole piece of an electromagnetic lens is not a perfect circle or if the material of the pole piece is magnetically non-uniform, the resulting magnetic field will not be axially symmetrical, and also the refractive power will be different in the two mutually orthogonal planes passing through the axis. As a result, the electron beam which should normally be focused at a point will appear as an ellipse. This phenomenon is called astigmatism. If there is an astigmatism in the objective lens (cross section of the electron beam is ellipse), a sharp image cannot be obtained even at the in-focus condition, and the image blurs in one direction at the out-of-focus condition.

#### Auger effect/Auger electron

When the atoms of a specimen that are excited by electron beam irradiation and return to the normal state, the released energy is used mainly to generate X-rays or light. But, a portion of the released energy is used to release electrons in the atoms of the specimen. This phenomenon is called the Auger effect and the electrons emitted are called Auger electrons.

## Backscattered electron detector

This is a highly sensitive annular type semiconductor element (P-N junction) which is located above the specimen. It detects electrons that are scattered backward from the specimen surface. The detector is divided into a pair of P-N junctions, each of which produces a signal. These two (or in some cases, four) signals are subjected to addition and subtraction processing in an operational amplifier, thus enabling the data pertaining to the effect of the surface topography of the specimen and also the data pertaining to the effect of the atomic number of the sample to be skillfully separated from each other and displayed. The signal resulting from addition is called a COMPO (composition) image, and the signal resulting from subtraction is called a TOPO (topographic) image. These images are utilized as the most effective means of performing EPMA analysis.

#### **BEI**

The abbreviation of Backscattered Electron Image. This refers to an image obtained with a signal by backscattered electrons from the specimen surface. A BEI image depends upon the surface topography of the specimen and also the average atomic number of

the elements contained in the specimen.

#### **Brightness control (of image)**

A brightness control adjusts the brightness of the image by changing the video signal level.

#### Brightness (of electron gun)

This is one of the values used to express the performance of an electron beam source. It is defined as the current generated per unit solid angle or unit area.

#### Cathodoluminescence (CL)

When a specimen is irradiated with an electron beam, the electrons in the valence electron bands of the atoms comprising the specimen are excited. Light emitted during the process in which the hole created in the valence electron bands recombine with the electrons is called cathodoluminescence.

#### Characteristic X-rays

When X-rays or an electron beam is irradiated on a substance, one of the shell electrons of the atom is released. Then, an electron in a higher energy shell drops into the vacancy. The energy difference, in this case, is released as an X-ray. This is called characteristic X-ray. The wavelength (or energy) of the characteristic X-rays is determined by the energy difference between these two shells, and the measurement of wavelength (or energy) identifies the type of the element. Each characteristic X-ray is named with the atomic symbol following K, L or M,  $\alpha$ ,  $\beta$  or  $\gamma$  and 1, 2 or 3, etc. These symbols are defined by the wavelength and energy level.

#### Charging Up

When an electron beam irradiates an electrically non-conductive specimen, electrostatic charges build up on the specimen during SEM observation. This phenomenon is called "charging up" (or aquiring electrostatic charge, or building up electrostatic charge). If the sample acquires the electrostatic charge during observation of a SEM image, the contrast of the image will become abnormal and unstable. Also, it will be impossible to obtain a sharp image. In order to prevent this, the sample is provided with a metal coating, or the image is observed under a low accelerating voltage.

#### **Composition image (COMPO)**

This image is obtained by adding the signals obtained from two or four backscattered electron detectors located symmetrically about the electron beam. The signal represents the composition (mean atomic umber) of the specimen, and is called a composition (COMPO) image.

#### Condenser lens

This is an electromagnetic lens located just below the electron gun in order to make the electron beam

emitted from the electron gun into a fine beam. It plays an important role in controlling the electron beam intensity and also the diameter of the electron probe.

#### Contrast

Contrast is the difference between the black and white (dark and bright parts) of an image. The contrast of a secondary electron image corresponds to the number of secondary electrons emitted from the specimen surface and captured by the secondary electron detector. The number of emitted secondary electrons depends greatly on the following factors:

- \* Incident angle of the electron beam to the specimen
- \* Edge effect
- \* Atomic number effect
- \* Accelerating voltage effect
- \* Effect of the magnetic field or electric field existing on the specimen surface

#### Critical point drying method

The greatest problem attendant to drying a specimen is the surface tension of the liquid. This causes shrinking and wrinkles. A method of drying a specimen by adjusting the pressure and temperature so that the distinction between liquid and gas disappears and also the surface tension disappears is called a critical point drying method. Generally, carbon dioxide, which is non-toxic, inexpensive and can be obtained readily, is used in this method.

#### **CRT**

This is an abbreviation of Cathode Ray Tube.

#### Cryo unit (CRU)

This is a device for freezing a biological specimen which contains water, or a specimen such as an emulsion. It maintains the specimen holder inside the microscope at a low temperature (between -150 and 160 °C) to enable the sample to be directly observed. The CRU has a cold stage in the specimen pre-evacuation chamber, in addition to the cold stage in the specimen chamber, enabling the specimen to be fractured or coated with gold without destroying the vacuum.

## Depth of field/Depth of focus

The depth of field is the distance between two object planes where the in-focus images can be obtained. The depth of field L is given by the equation  $L=(r/M-d)/2\alpha$ . Here, r is the minimum distance between two dots (lines) that can be resolved with the naked eye, M is the magnification, and d is the probe diameter. From this theoretical equation, it can be seen that if a small objective aperture and a long WD is selected, the  $\alpha$  becomes small. As a result, the large depth of field is obtained.

#### DFU

The abbreviation of Dynamic Focus Unit.

If a relatively flat sample is observed at a relatively low magnification with the largely tilted specimen stage, an obtained image is out-of-focus at the upper and lower edges (or the left and right edges, depending upon the equipment) of the CRT screen. The DFU corrects this out-of-focus image by changing the objective lend focal length with scanning positions.

#### **EBIC**

The abbreviation of Electron Beam Induced Current. If a semiconductor device specimen has a P-N junction in it, a large number of carriers are generated in the vicinity of the junction when the electron beam irradiates the junction. This causes an electromotive force, and results in a flow of electron current across the junction. This current is called Electron Beam Induced Current, and the image obtained when the electron beam is scanned on the specimen is called the EBIC image. EBIC image observation is an important method for evaluating the integrity of a P-N junction or observing the crystal defect in a semiconductor device.

#### **ECC**

The abbreviation of Electron Channeling Contrast. The number of secondary electrons or backscattered electrons emitted from the specimen surface differs with to the crystalline structure of the specimen surface. ECC is an image contrast representing this phenomenon in the SEM image.

#### **ECP**

The abbreviation of Electron Channeling Pattern. The number of backscattered electrons or secondary electrons emitted from the specimen surface differs with to the crystal structure and crystal orientation of the specimen surface. ECP is a pattern representing the crystal structure and orientation obtained when rocking the electron beam around the optical axis.

## **EDS**

The abbreviation of Energy Dispersive Spectrometer. This is an X-ray spectrometer which uses a semiconductor detector to detect the energies of X-rays emitted from the specimen surface and convert them into electric pulse heights.

#### **Electromagnetic lens**

This is an electron lens which uses a magnetic field to focus an electron beam.

#### Electromotive force image

This is an image which is obtained by detecting a voltage or current signal generated in a sample when irradiated by an electron beam. (see [EBIC])

#### Electron beam

An electron beam is a fine flow of electrons in one

direction at a roughly constant velocity. Normally, it is generated by accelerating thermoelectrons in a vacuum.

#### Electron gun

An electron gun is a device that generates an electron beam which is equivalent to the light source of an optical microscope. It consists of a hot cathode (filament) made of a 0.12-mm-diameter hair pin shaped tungsten wire, a control electrode (Wehnelt cylinder, or grid), and an anode. The tungsten hairpin type electron gun is generally used in an electron microscope.

#### **Electron optics**

This is a science on the motion of free electrons under the influence of electrostatic and electromagnetic fields. It utilizes the fact that an electrostatic and electromagnetic fields act on the path of an electron in a similar way to the action of a glass lens in conventional light optics. This science is indispensable for designing electron microscopes, CRT, vacuum tubes, photomultiplier tubes (PMT), and electron accelerators.

#### Electron probe

This is an electron beam that is focused finely on the surface of a specimen.

#### Element distribution image (X-ray image)

This is the 2-dimensional representation of the distribution of specific elements that exist on the specimen surface, obtained by elemental analysis.

#### **EPMA**

The abbreviation of Electron Probe Microanalyzer. This is an instrument which can analyze the composition elements in a very small area on the solid specimen surface, without destroying the specimen (non-destructive analyzer). An accelerated electron beam is used as a probe. It is also called an XMA.

#### **Evacuation time**

This is the time required to evacuate a vacuum system from the atmospheric pressure to the specified pressure.

#### Fluorescence

A substance that emits lights when it is exposed to light or an electron beam, or placed in an electric field, is called a fluorescent substance, and the phenomenon of light emission is called fluorescence. Luminescence that is produced by the stimulus of an electron beam is called cathodoluminescence, and luminescence resulting from X-ray, infrared or ultraviolet excitation is called photoluminescence.

#### Freeze-drying method

This is a method of drying a specimen containing water by freezing it using, for example, liquid

nitrogen, then sublimating the water in a vacuum. It is used for biological specimen preparations; however, it is necessary to be careful of water crystals that grow when the specimen is frozen.

#### **In-line SEM**

This SEM is used in a semiconductor production line. It has a clean vacuum and a large specimen stage, and uses a low accelerating voltage to avoid the specimen damage by electron beam irradiation. It requires high-level automated functions such as auto focus and auto specimen loading. A small foot print is required, too.

#### Ion sputtering device

The ion sputtering device is used to prepare SEM specimens. It creates a glow discharge in a low vacuum of about 10 to 1 Pa, and ionizes the residual gas or introduced argon gas. These gas ions strike a negative potential cathode target at high velocity, sputter the metal of the cathode, and deposit the sputtered metal on the surface of the specimen in the vicinity. Thus the specimen is metal coated. Alternatively, it can be used to shoot ions at the surface of a specimen instead of the cathode target, so as to remove (etch) an unwanted film on the specimen surface.

## LaB<sub>6</sub> cathode (or tip, or filament)

This cathode is made by sharpening a lanthanumhexaboride crystal into a conical shape. It is used as a thermoelectron source in an electron gun.

## Line analysis

This is a type of analysis which is used to investigate the 1-dimensional (along with a line) concentration distribution of elements over a certain range of the specimen.

## Low vacuum (LV) observation

If the vacuum system of the specimen chamber is separated from other parts of the SEM, and the pressure raised to several Pa, gas molecules remaining in the specimen chamber will be ionized by electrons generated from the incident electrons and the sample, neutralizing the electrostatic charge on the specimen. This is one method for observing an electrically non-conductive specimen without metal coating.

#### MAC

The abbreviation of Magnification Automatic Control. In a SEM, if the working distance (WD) changes, the scanning range at the specimen surface changes, resulting in a magnification change. MAC controls the amplitude of the scanning current (magnification) according to the objective lens current (working distance) so as to maintain the amplitude constant and keep the magnification unchanged.

## Magnetic domain

This is a small unit area (domain) of a ferromagnetic material, such as iron, nickel or cobalt, in which the magnetic field is uniform and saturated.

#### Metal coating

When observing or analyzing an electrically non-conductive specimen with a SEM or EPMA, the surface of the specimen is coated with a thin film of electrically conductive material in order to prevent a buildup of electrostatic charge on the specimen. For observation of a SEM image, the specimen is coated with a heavy metal such as gold, gold-palladium alloy or platinum, while for X-ray analysis, the specimen is coated with a light substance such as carbon or aluminum, using a vacuum deposition device or ion sputtering device.

#### nm

The abbreviation of nanometer. This is a unit of length.  $1 \text{ nm} = 10^{-9} \text{ m}$ .

## Objective lens

The objective lens is the lens that is nearest the specimen. In SEM, it is used to focus the electron beam on the specimen surface (focusing).

#### **OM**

The abbreviation of Optical Microscope.

## Optical microscope

This is an instrument that is used to obtain an enlarged image of a small object, using visible light. Generally, it consists of a light source, condenser lens, objective lens, and an eyepiece lens. The object is placed immediately outside the focal point of the objective lens (convex lens), resulting in a large real image. This real image is brought to the inside of the focus point of another convex lens (eyepiece). The observer sees the virtual image of this real image, which is a further enlarged version of the object.

#### Pa

The abbreviation of pascal. This symbol represents a unit of pressure. It is used mainly in vacuum engineering.

1 Pa = 10  $\mu$  bar, 133 Pa = 1 Torr = 1 mmHg

## Penning vacuum gauge

This is a cold cathode ionization vacuum gauge. When a voltage of several thousand volts is applied between a ring-shaped anode and the left and right disk-shaped cathodes, and also a magnetic field of several hundreds to one thousand gauss is applied in the direction of the axis of symmetry, the electrons emitted from the cold cathode electrode ionize the gas molecules. The degree of vacuum (pressure) is measured utilizing the fact that the discharge current is roughly proportional to the pressure of the residual

gases. The presence of the magnetic field increases the chance of gas ionization, resulting in a high sensitivity.

#### **PMT**

The abbreviation of Photo Multiplier Tube. This is an electron tube to convert light into electricity. It utilizes the phenomenon of the photoelectron emission, and carries out the secondary electron multiplication several times in order to increase the number of electrons, then collects them in the anode.

#### **Polepiece**

The magnetic field generated at the gap of an electromagnetic lens must be strong and highly axially symmetrical. In order to realize this, it is necessary to select the material of the polepieces very carefully and to machine the polepieces with a high degree of precision. They are made from high-permeability material such as Armco iron.

#### **Potential contrast**

This is a kind of contrast of a SEM image. The number of secondary electrons, which are emitted from the specimen and detected by the secondary electron detector, varies with the electrostatic potential of the specimen surface. The contrast caused by this phenomenon is called the potential contrast. This potential contrast is often utilized together with the EBIC in the evaluation and research of semiconductor devices.

#### Pre-evacuation chamber

This mechanism is used to exchange specimens without any vacuum deterioration in the specimen chamber and electron optical column. There are two methods of exchanging a specimen. One is a drawout method in which the entire stage is withdrawn and air is introduced to the entire electron optical column, causing the vacuum to be broken. And the other is an air-lock method in which specimens are exchanged through a small chamber (pre-evacuation chamber) which is separated from the overall electron optical column by means of an isolation valve. In the latter method, air is introduced only to the preevacuation chamber when exchanging specimens; hence, the inside of the electron optical column is not exposed to the atmosphere. This method is thus superior from the viewpoint of maintaining the inside of the optical column in a clean vacuum.

#### Qualitative analysis

This type of analysis is used to determine what elements are contained in a certain part of a specimen.

#### Quantitative analysis

This type of analysis is used to determine the weight concentration of each composite element at a certain part of a specimen.

#### Resolution

This is the ability to discern the fine structure of an object, or the ability to identify two points or two lines at a very short distance from each other.

## Secondary electron detector

The most widely used type of secondary electron detector consists of a combination of a scintillator and a photomultiplier tube. A positive potential of 10 kV is applied to the scintillator, accelerating low-energy secondary electrons and causing them to collide. As a result, light is emitted. This light passes through a light pipe to the photomultiplier tube, and is converted into an electrical signal which becomes an image signal.

#### SEI

The abbreviation of Secondary Electron Image. This refers to an image formed with a signal by secondary electrons emitted from the specimen surface by a SEM or EPMA. The depth from the specimen surface at which secondary electrons release is only about 10 nm; hence, there is little scattering of the incident electrons at the specimen surface, and the best image resolution is obtained among other images obtained by a SEM or EPMA. The contrast of a secondary electron image depends upon the surface roughness and profile of the sample, as well as upon differences in the composition of the sample.

## Solid image (SHADOW)

This image is obtained by processing (combination of adding and subtracting) the signals that are obtained from two or four backscattered electron detectors located symmetrically about the electron beam. Namely, by mixing TOPO and COMPO signals, an enhanced 3-dimensional effect is obtained, and is called a solid (SHADOW) image.

## Spherical aberration coefficient (Cs)

Spherical aberration is an aberration in which the image formation point (focus point) of the light passed near the center of a lens differs from that passed through the periphery of the lens. The coefficient that depends upon the spherical aberration of each lens is called the spherical aberration coefficient.

#### Stigmator

The stigmator is a device which makes correction for astigmatism. As astigmatism is caused by an axially asymmetrical magnetic field in the lens polepiece, it is possible to correct it by placing another asymmetrical magnetic field in the lens and adjusting the strength and direction of it. This correction lens is called a stigmator. Generally, it consists of a pair of quadrupole electromagnetic coils.

#### TEM

This is the abbreviation of Transmission Electron Microscope.

#### Tesla (T)

The Tesla is a unit of magnetic flux density in the international system of units.

1 T = 10 4 G [Gauss]

#### Thermoelectrons

These are electrons that are emitted from the surface of a metal when it is heated to a high temperature.

#### Topographical image (TOPO)

This image is obtained by subtracting the signals obtained from two or four backscattered electron detectors located symmetrically about the electron beam. The signal represents the topography of the specimen surface, and is called a topographical image.

## Transmission electrons, transmission electron image

If the specimen is sufficiently thin, the electrons that irradiate the specimen at high energy can pass through the specimen with almost no energy loss. Such electrons are called transmission electrons. Transmission electrons can be detected by a detector located beneath the specimen. The image formed by the detected signal is called a transmission electron image.

## Tungsten filament

The tungsten filament consists of a tungsten wire of about 0.12 mm diameter which is bent into the shape of a hairpin, and heated to about 2800°C by an electric current directly through it. Tungsten has a high melting point and a small evaporation rate, does not react with residual gas at high temperature, has high mechanical strength, and can be handled easily. For these reasons, it is used as the cathode material of the majority of electron guns.

## Vacuum

This is a state of a space which is filled with gas at a pressure of less than a normal atmospheric pressure.

#### Vacuum deposition device

This device, a glass bell jar that has about 250 mm inner diameter and about 200 mm height in which a high vacuum of  $10^{-3}$  to  $10^{-4}$  Pa is obtained. It melts and evaporates carbon or a metal at high temperature and deposits this vapor as a uniform film on the surface of a specimen.

#### Vacuum pressure (vacuum degree)

This is a term which indicates the pressure of the vacuum measured in the unit of pascal (Pa).

#### Vacuum pump

An electron microscope requires a highly efficient evacuation system. There are two categories of the vacuum pumps. One operates from atmospheric pressure and the other does not operate until the pressure is reduced to a certain extent. The evacuation systems of most SEMs employ a combination of an oil rotary pump for the former type and an oil diffusion pump for the latter type. An oil rotary pump can reduce the pressure from atmospheric pressure to  $10^{-1}$  Pa. An oil diffusion pump can reduce the pressure from 1 to  $10^{-5}$  Pa.

#### **WDS**

The abbreviation of Wavelength Dispersive Spectrometer. This is an X-ray spectrometer which employs the Bragg reflection by a crystal lattice to measure the wavelengths of characteristic X-rays emitted from the specimen surface. To enable all elements (except gas elements) from boron (B) to uranium (U) to be analyzed using a wavelength dispersive X-ray spectrometer (WDS), four analyzing crystals, MYR (STE), TAP, PET, and LiF, are necessary. The FCS is a spectrometer in which all four of these crystals are installed, enabling all elements to be analyzed.

#### Wehnelt cylinder

A Wehnelt cylinder is an electron beam control electrode which is made of cylindrically shaped stainless steel with 1 to 2 mm diameter hole for an electron beam passing through it. It functions to control and stabilize the electron beam emitted from the cathode when a bias voltage is applied to it. It is sometimes called a grid.

#### Working distance (WD)

The working distance (WD) is the distance from the lower face of the lower pole piece of the objective lens to the surface of the specimen. In order to observe a specimen whose surface is very irregular, necessitating an increase in the depth of field, the WD is increased. In this case, greater effectiveness can be obtained by selecting the optimum aperture diameter of the objective lens.

#### X-rays

Generally, the term X-rays refers to electromagnetic waves which were discovered by Roentgen in 1895. These rays are diffracted by a crystal lattice. The wavelengths of X-rays extend over a range of 10 nm to 0.001 nm, which lies between normal ultraviolet rays and  $\gamma$ -rays. In a SEM, a specimen is irradiated with an electron beam, resulting in the generation of characteristic X-rays that have a wavelength peculiar to the composition of the specimen. These X-rays are detected, and used to identify the elements in the specimen and also to perform quantitative analysis.

#### **ZAF** correction method

Quantitative analysis by an EPMA involves obtaining the relative intensity (Ki = Ii unk /Ii STD) from the X-ray intensity (CPS) of a standard specimen and that of an unknown sample under the same conditions. This relative intensity (Ki) is an approximation of the true weight concentration (Ci). The true concentration is obtained by applying correction for matrix effects (ZAF effects), including the atomic number (Z) effect, absorption (A) effect and fluorescent excitation (F) effect, to this relative intensity. This correction method is called the ZAF method.



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