State of the Art and the Future Technologies of Electron Microscope

Sumitomo Chemical Co., Ltd.
Tsukuba Research Laboratory
Yoshiaki HONDA

Development of nanotechnology necessitates the structural, physical and chemical characterization of constituent advanced materials at the nanometer level. For example, compound semiconductors used to optical and electronic devices are composed of many thin layers in nanometer level. Electron microscopy is one of the most effective methods for these kinds of analysis, which gives direct information of the structure and the elements distribution with high spatial resolution. Recently, new imaging techniques in electron microscope have been developed. This review describes state of the art and future technologies of electron microscope.

This paper is translated from R&D Report, "SUMITOMO KAGAKU", vol. 2004-Ⅱ.

Introduction

Recently, materials with differing characteristics and phases are being combined on the nanometer (nm) level, and advanced materials are being developed using composites of the functions for each. Sumitomo Chemical is developing advanced materials such as compound semiconductor (GaAs, etc.) electronic and optical devices that exhibit high-level performance by controlling thin films and compositions on the nm level and polymer materials that give high performance by controlling the microphase separation structure.

Observation of shapes using transmission electron microscopes (TEM) with a spatial resolution on the sub-nm level (atomic level) is necessary as a means for analyzing the structures of these advanced materials, and this importance is steadily increasing. Furthermore, elemental analysis that makes use of TEM because of the recent technical advances is moving toward having spatial resolutions on the order of nm, and structural analysis from both the aspect of shape and that of composition has become possible. This paper describes the recent advances in electron microscope technology and the principles, applications and future outlook focusing on TEM.

Main Technologies for Electron Microscopes

1. TEM, SEM, EPMA, Augier and CL

If the electron beam in the vacuum in an electron microscope strikes a thin sample, it passes through the sample after interacting in various ways with the elements included in the sample. Secondary electrons, reflected electrons, characteristic X-rays Augier electrons and fluorescence are emitted from the sample as a result of these interactions. On the other hand, the electrons that pass through the sample include scattered electrons, elastic scattered electrons that have been without an accompanying

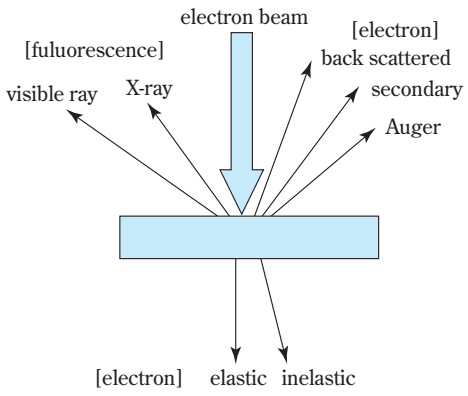


Fig. 1 Interactions of the electron beam and substance

loss in energy, and inelastic scattered electrons that have lost some of their energy because of scattering (**Fig. 1**).

Methods for observing the shape of samples using electron beams can roughly be divided into scanning electron microscopes (SEM), which detect secondary electrons and reflected electrons, and TEMs, which form images of the electrons passing through the sample.

With a SEM, a narrowly focused electron beam scans the surface of the sample, and the image is obtained by displaying the intensity of the secondary electrons and reflected electrons produced at this time in synchrony with the scanning rate on a monitor (**Fig. 2**). The image contrast is obtained because the efficiency with which secondary electrons are generated varies according to the composition of the material and the detection efficiency for the secondary electrons varies according of the angle of the surface of the sample to the detector; in addition, the contrast due to the surface shape can be emphasized or the contrast due to changes in composition can be emphasized using the acceleration voltage for the electron beam incident to the sample. SEMs with field emission electron guns have achieved a spatial resolution of 1nm, but there are cases where the image is blurred by the scattering of electrons within the sample, and the spatial resolution for the image that can actually be obtained partially depends on the form and other aspects of the sample.

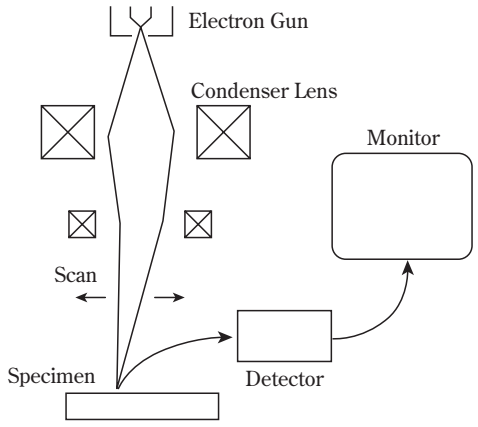


Fig. 2 Schematic of a SEM

With TEM, the sample is irradiated with parallel electron beams, and the image of the electrons passing through the sample is formed on a fluorescent screen using an electromagnetic lens (**Fig. 3**). Recording of images is generally done through the photosensitivity of negative film to the electron beam, but recently images are also being formed using imaging plates and CCD cameras, as will be discussed later. The image contrast is diffraction contrast (also called absorption contrast), which makes use of the fact that the scattering angle for electrons varies according to the density of the material and the crystal orientation, and phase contrast obtained from the interference of electron beams with altered phases due to the potential within the sample. The spatial resolution for TEM is mainly determined by the performance of the object lens (spherical aberration) and the width of the energy of the incident electron beam (chromatic aberration), and they have a spatial resolution making observations of 0.x nm or single atom separation possible (**Fig. 4**).

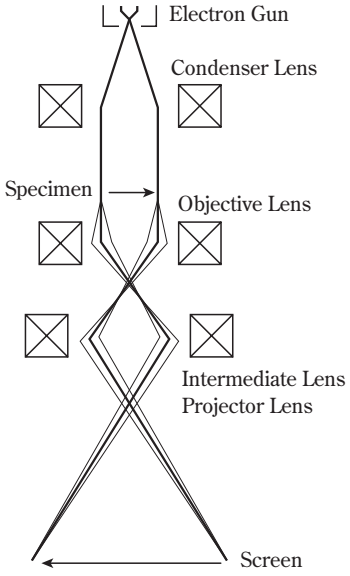


Fig. 3 Schematic of a TEM

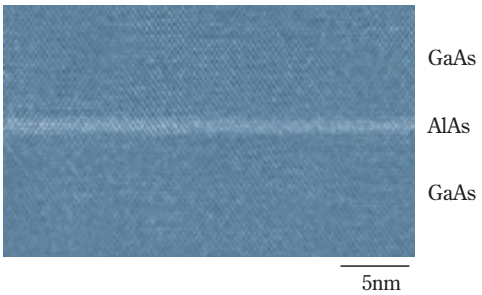


Fig. 4 Example of TEM image. It is seen the arrangement of atoms.

Other than this, there are scanning transmission electron microscopes (STEM) that scan the surface of the sample with a narrowly focused electron bean and detect the electrons passing through. There are dedicated STEM units, but most are handled as functions found in TEM or SEM. The spatial resolution of STEMs is determined by the diameter of the electron probe, and in the most recent devices, there is spatial resolution of 1nm or less.

With TEM and STEM, it is necessary to make the sample thin enough so that electrons can pass through for observation, and furthermore, the alignments during observations require skill. With SEM, observations can normally be made just by carrying out a vapor deposition process to give the sample conductivity, and since the operations during observations are comparatively easy, there is high throughput. Therefore, observations that can be made with SEM are generally done with SEM, and when the spatial resolution of SEM is insufficient, TEM is used. Similar images are obtained with TEM and STEM, and the divisions in use are not so clear, but in analyses that use diffraction phenomena TEM is used, and when we wish to emphasize the contrast due to differences in composition, STEM is used.

The elemental analysis using electron microscopes that is widely used is energy dispersive X-ray spectroscopy (EDS) where the characteristic X-rays excited by the electron beam are detected. These devices can be attached to any SEM, TEM or STEM device, and generally, a pulse voltage proportional to the X-ray energy in a semiconductor detector that uses Li doped Si crystals is generated, and the spectrum is obtained by counting the number of pulses (X-ray intensity) for the voltage (X-ray energy). Since the characteristic X-ray energies differ with the element, the elements present in the range of the electron beam exposure can be identified from the peak energy values appearing in the spectrum. In addition, since measurements are made while the incident electron beam scans the sample with SEM and STEM, it is possible to obtain an elemental mapping image that shows the positions of the elements. With bulk materials, the spatial resolution is on the order of several hundred nm because of the scattering of the electron beam within the material, but with thin film materials, a spatial resolution of several nm, just about the same as the diameter of the electron beam probe, can be obtained.

Similarly, among the methods for detecting characteristic X-rays, there is wavelength dispersive X-ray spectroscopy (WDS). With WDS, detection is divided according to the X-ray energy (wavelength) generated using X-ray diffraction. Since the energy resolution is higher than EDS, the capacity for identifying elements is greater with WDS, but measurement time is longer than for EDS, and since vibration arises when the analyzing crystal is moved mechanically, this is almost never used in combination with TEM, where high-level spatial resolution analysis is carried out. When used in combination with SEM, it is handled as a dedicated device (EPMA) designed with importance placed on stable exposure with a high current electron beam probe, which is effective for elemental analysis, rather than for spatial resolution in image observations.

Augier electron spectroscopy carries out elemental analysis by detecting Augier electrons instead of characteristic X-rays. Since the Augier electron energy used in the analysis is comparatively low at 30–3000 eV, there is a strong interaction with solids, the Augier electrons generated within the sample that can escape into a vacuum without losing energy are limited to the electrons generated in a shallow range of several nm from the surface of the sample. Therefore, Augier electron spectroscopy is commonly handled as a surface analysis method having a spatial resolution on the order of 10nm rather than as electron microscopy.

Cathode luminescence (CL) detects fluorescence centered on visible light excited by the electron beam, and even though it cannot be used for elemental analysis, it is a method that can be used for analyzing energy levels and concentration of impurities and defects, distortion in crystals and the like. Measurements are generally made by attaching a detector to a SEM, but it may be attached to a TEM. In addition to the diameter of the electron beam probe, the spatial resolution depends on the sample because the diffusion length of the carrier generated, and at the highest, a spatial resolution of several tens of nm is obtained.

Elemental analysis can be carried out using electron energy loss spectroscopy (EELS), which obtains a spectrum by detecting electron beams according to energy in a method that makes use of

electrons passing through the sample. The details of the principles will be discussed later in the section on energy filtered TEM.

As in the above, it is possible not only to observe the form of samples by examining the interactions arising when they are irradiated with an electron beam using electron microscopes, but also to obtain structural information centered on the elemental composition.

2. FIB Processing

As was mentioned above, samples must be made thin, on the order of the thickness of films through which the electron beam can pass to carry out structural analysis with TEM, and this thickness is normally 100nm or less, with better results obtained the thinner the samples are made. If there are fine grains that an electron beam will pass through from the start, observations are possible just by scattering on a support film for TEM observation, but in the case of normal bulk materials, forming a thin film from the material is necessary for TEM observations. Therefore, from the beginning of TEM development, electro-polishing was developed for metallic materials and microtome preparation for biological materials and polymer materials; furthermore, ion thinning and other thin film formation technologies have been developed and used according to the properties of the material, with an emphasis on semiconductor materials. However, the development of materials in recent years has come to combining materials with different properties as was discussed at the beginning of this article, and composite materials, such as polymers and ceramics, for example, cannot be made into thin films using conventional technology. In addition, along with the miniaturization of defects and other targets of analysis as exemplified by the increasingly small size of semiconductor devices, a necessity for forming thin films of specific minute parts has arisen.

In these circumstances, focused ion beam (FIB) processing, the development of which has progressed for use in semiconductor processes, has been developed as a technology used in sample preparation apparatus for TEM. Normally, Ga is used as a liquid metal ion source for FIB, and a Ga ion beam accelerated to a voltage of around 10 – 40 kV is brought to convergence on the submicron level scanned across the sample to expose it; the area of the sample to be scanned is cut out and processed by means of sputtering (**Fig. 5**). The surface shape can be observed using the secondary electron image obtained by detecting the secondary electrons excited by the ion beam, so processing can progress while the shape of the part to be processed is checked (**Fig. 6**). In addition, materials with different properties can be processed to a uniform thickness simultaneously, and this can be applied to composite materials that could not be made into thin films by conventional methods. Since the processing rate is fast, and normally a sample with a 10μm square surface area can be formed into a thin film with a thickness of around 100nm in several hours, the effect is an overall

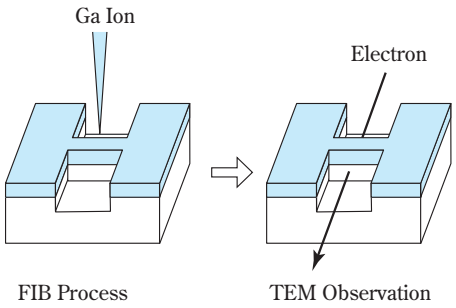


Fig. 5 Schematic of an FIB processing.

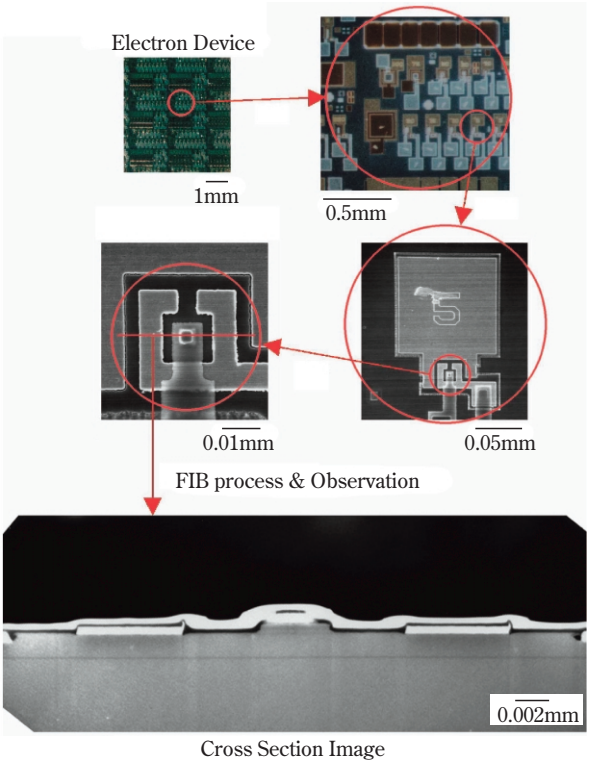


Fig. 6 Example of Observation used the FIB processing

improvement in the TEM structural analysis speed, since the sampling rate has been the rate controlling factor.

The negative side of FIB processing is that damage to the processed surface is greater than with conventional sampling methods, and the quality of TEM images is lower than it has been conventionally. Besides carrying out the processing with a low acceleration voltage in the final stages of FIB processing to obtain good quality TEM images, there are also techniques such as removing the damaged layer on the processed surface by methods such as ion thinning or chemical polishing after the completion of the FIB processing.[2)]

3. Positioning of Other Local Analytical Methods

It can be said that there is no practical method for elemental analysis with a spatial resolution on the order of nm other than TEM. Scanning tunneling microscopes (STM) and atomic force microscopes (AFM) also give images of atoms for observations of shape, and even though extremely high-level data has been reported for some, there is the restriction of the targets of observation being limited to surfaces that are kept clean to the order of atoms, while elemental analysis is naturally impossible. If we limit ourselves to a one-dimensional analysis, resolution in the direction of depth of a nm or less has been obtained with analysis in the direction of depth using Augier electron spectroscopy, but this is limited to the comparatively narrow range of a depth a little less than 1μm from the surface of a sample with a flat surface.

While each of the analytical methods has a field in which it is good, TEM, which can perform elemental analysis on the order of nm while checking the form of the material basically regardless of the characteristics of the material, is currently the most effective method for structural analysis on the nm level.

Recent Topics in the Various Electron Microscope Technologies

The spatial resolution of SEM rapidly improved to its maximum of 1nm or less with the field emission (FE) electron gun becoming practical in the second half of the 1980s, but since the first devices had a structure where the sample was inserted within the object lens, the size of samples that could be observed was limited to several mm; furthermore, the spatial resolution was reduced remarkably when the acceleration voltage was lower to obtain information on the very surface of the sample. After this, device performance was improved through a reassessment of the objective lens design and the like, and at present, spatial resolutions of 1nm have been achieved with SEMs that can make observations on large samples of several cm, without alteration; furthermore, since they can handle low acceleration voltage observations, functions for reducing the speed of the electron beam, with its high final acceleration voltage, just before irradiation using a negative voltage has been developed, and a spatial resolution of 1.4nm has been obtained even if the acceleration voltage is lowered to 1KV using this function. In addition, detection methods where the proportion of secondary electrons that include much information on shape and reflected electrons that contain much information on composition can be adjusted to any value have been developed. It has become possible to do everything from controlling these devices to capturing images on personal computers, and operability and throughput have improved.

The brightness and interference in the electron beam during high magnification observations have also been improved in TEM through the use of FE electron guns, and there have been rapid improvements in spatial resolution with additional improvements to objective lenses. Along with this, it is possible to observe atomic images with ideal samples even with STEM, and use as a method where the contrast can be interpreted easier than in TEM images in some analyses of atomic positions has begun. In addition, with STEM, there is the characteristic of being able to obtain contrast corresponding to element numbers by using high angle annular dark field (HAADF) observations through the detection of the electrons scattered at wide angles. In imaging systems, imaging plates and CCD cameras, which have higher sensitivity and a greater dynamic range than conventional negative film have become practical, and there is progress in increasing performance. By this means it is possible to expose high resolution images and dark field images, where there are problems with image blurring due to sample drift during exposure, for short periods of time, and further, it is possible to carry

out quantitative analysis on electron beam diffraction images that include convergent beam electron diffraction (CBED). Furthermore, rapid observations in bright rooms are possible using computer control of devices and observations made using CCD cameras, and there have been great improvements in the overall speed of analytical operations along with the progress in imaging systems.

There have also been improvements in EDS analytical functions, and multivariate analysis and other methods have become practical for EDS spectra with functions for automatically correcting the sample drift during observations and large amounts of peak overlap. On the other hand, Schottky emission (SE) electron guns, which are a form of FE electron gun, have been installed for EPMA electron guns. While the electron beam probe has a slightly larger diameter for SE electron guns than for the conventional FE electron guns, highly stable, high-density electron beams can be obtained over long periods of time even at low acceleration voltages, so a large improvement in the spatial resolution in elemental mapping has been realized.

Among these developments, energy dispersive x-ray spectroscopy (EDS) has currently become the most popular as a method for compositional analysis of minute parts using electron microscopes, but the development of energy filtering TEM (EFTEM) based on the electron energy loss spectroscopy (EELS) that will be discussed later is actively being pursued in search of even higher performance.

In addition, electron staining is necessary to obtain contrast in TEM observations of polymer materials, but the staining conditions vary according to the type of polymer, and because it requires a great deal of time to find the appropriate staining conditions for the new composite polymer systems, it is difficult to carry out staining on the order of nm without irregularities. Therefore, there is a need for technologies where polymers can be observed without staining. This is also being solved using EF-TEM.

Furthermore, conventional TEM observations have been limited to two-dimensional shape observations because of the imaging of samples that have been cut into thin fragments, but a need for structural analysis of three-dimensional solids, which is an impossibly high level with present technology alone, has arisen.

Methods for the observation of solid shapes using tomography have become practical through the recent advances TEM devices themselves and computers to deal with these needs. [3)]

Under these circumstances, Sumitomo Chemical has been carrying out technical investigations into EF-TEM by participating in the Ministry of Education, Culture, Sports, Science and Technology Comprehensive Nanotechnology Support Project and the like, and it has been developing analytical technology and conducting investigations into the applications. In the next section, we will show the results for EF-TEM measurement principles and those obtained through participation in the project mentioned above along with introducing the possibilities for applications in tomography as a development of these.

Energy Filtering TEM (EF-TEM)

1. Principles

EF-TEM is a method that selects electrons with specific energies among the electrons that pass through the sample, and it can improve the quality of nm order elemental analyses and TEM images and emphasize the contrast, etc. The electron beam incident to the sample causes the interactions shown schematically in **Fig. 7** with the atoms in the sample, and as a result, the electrons passing through the sample can be generally classified into the following five types.

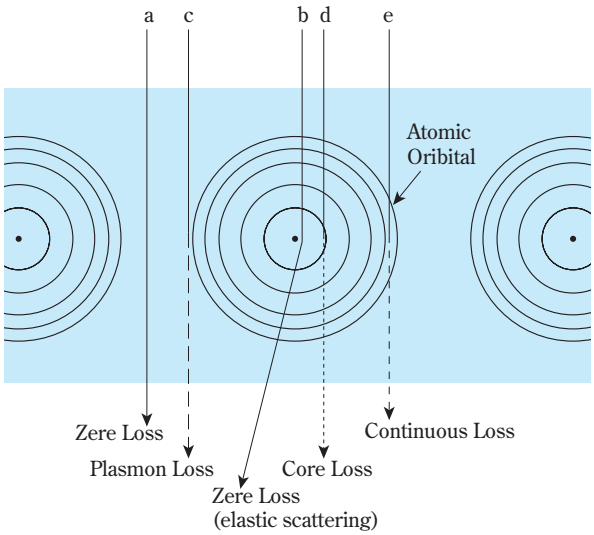


Fig. 7 Classification of the electron that transmitted a specimen

(a) Electrons that are scattered by the atoms and pass through

(b) Electrons that have undergone elastic scattering by the atoms

Electrons a and b are electrons that have not been caused to lose energy within the sample, and they are called zero loss electrons. These electrons do not contain chemical information (composition and the like) about the sample.

(c) Electrons that have lost energy when exciting plasmons (collective excitation of electrons) in the electron clouds within the sample. These are called plasmon loss electrons.

(d) Electrons that have lost energy when exciting the inner valence shell electrons in the atoms. These are called core loss electrons, and the energy lost varies with the element.

(e) Electrons that undergo a continuous energy loss due to excitation of outer valence shell electrons in the atoms. These electrons do not contain any information about the structure of the sample.

Fig. 8 shows the excitation process for electrons corresponding to each of these in an actual example of an EELS spectrum where the horizontal axis is the amount of energy lost and the vertical axis electron beam intensity. The zero loss electron peak, plasmon loss electron peak and the core loss electron peaks above the large background due to electrons that have undergone continuous energy loss all appear.

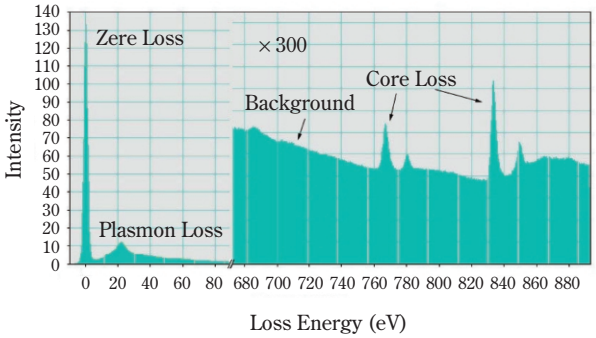


Fig. 8 Example of EELS spectra

Fig. 9 shows the EF-TEM optical system for separating electrons with different amounts of energy loss. The general classifications for energy filtering systems include in-column and post-column types. The post-column type is attached under the electron microscope, and the electron beam is bent 90° and is one where a normal EELS detector of the type where the electron beam is bent 90° is extended with an imaging lens and CCD camera. In-column types mainly use omega (Ω) filters. A filter comes between the intermediate lens and projector lens, and the electron beam that has been bent returns to the original optical axis again. It is possible to use a normal fluorescent screen or camera chamber for imaging. There is no great difference in the basic principles or performance with the in-column type and post-column type, but each has its characteristics in terms of operability. Since the electron beam returns to the optical axis with the in-column type, the blurring (non-point) of the image arising in the filter disappears; furthermore, since the entire fluorescent screen can be used, switching with normal TEM images is easy in addition to the large surface area imaging and diffraction image imaging being advantageous, with operation that feels close to a normal TEM. On the other hand, with the post-column type, the electron beam brought to the detector is limited to a range of approximately 1cm, and the astigmatism arising in the filter remains unchanged, etc., making adjustments in the optical axis in the detector and separate from the main TEM unit necessary. Since operation is more difficult that the in-column type, there has been work recently on improving the automatic axial adjustment functions.

With Ω filters, the electrons that pass through

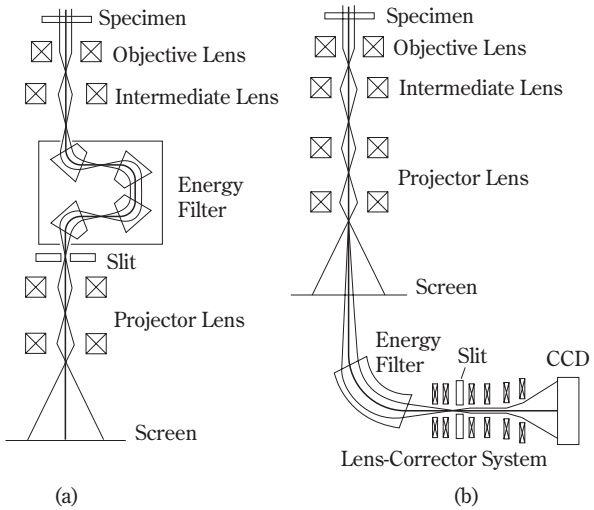


Fig. 9 Schematic of an in-column type EF-TEM (a) and a post-column type EF-TEM (b)

the sample describe an Ω shaped path because of the magnetic field, but energy dispersion that makes use of the fact that the curvature of this path varies according to the electron energy is brought about (**Fig. 10**). By introducing a slit that selects the energy into the energy dispersion plane obtained, only electrons with a specific energy can be selected and guided to the TEM imaging system. The images obtained are typically digitized directly by a CCD camera and stored in a computer rather than on negative film, and it is possible to easily perform accurate alignment among images when calculations are performed after imaging has taken place.

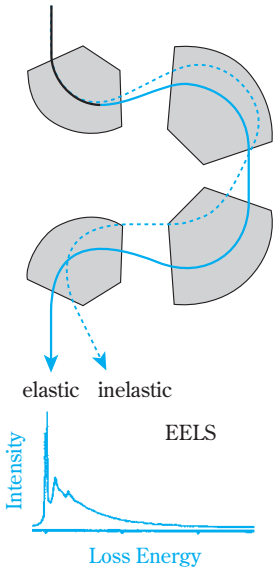


Fig. 10 Ray diagram of an Ω filter

Specific elemental mapping images are obtained by matching the energy selection slit to the core loss electrons for a specific element and correcting for the background. The original TEM spatial resolution is reflected in the spatial resolution for the mapping image, and compositional analysis on the order of nm is possible.[4] In addition, the energy for the electrons used in the TEM image can be arranged by matching the energy selection silt to zero loss electrons, so it is possible to avoid image blurring due to chromatic aberrations, and images (zero loss images) that are clearer than normal TEM images can be obtained. Furthermore, the intensity of the electron beam that has lost energy is very sensitive to the composition of the sample, so if imaging is done with the introduction of an energy selection slit in a suitable energy position, images (energy loss images) with contrast stronger than normal TEM can be obtained.

2. Characteristics.

While the EDS method, which is the most popular method for analyzing minute parts, has made technical advances in the last few years as was mentioned earlier, there is an insufficient S/N ratio and since scattered electrons and secondary X-rays arising in locations other than those being analyzed because of the primary X-rays generated, it is difficult to analyze compositional changes in areas of several nm. The capacity of EDS has just about reached its limits, and fundamental improvements are necessary to greatly improve performance over what it currently is, but no new outlook for new technology that can be thought of as possibly becoming practical in the next few years has been established.

In contrast to this, there have been reports of examples of compositional analysis the single atom level with EF-TEM, and it has a high spatial resolution compared with EDS. In addition, it is characterized by having a higher spectral energy resolution than EDS (EDS: 130 eV, EELS: 0.x eV), and not only is the overlapping of the peaks for different elements avoided, but also analysis up to the energy shift is possible according the chemical form.

Comparative results on the performance of current EDS and EELS for actual measurements using the same microscope are shown in **Fig. 11**. Fig. 11 (a) gives comparative results for the spectra of carbon, nitrogen and oxygen included in many polymers, and since the peaks for the various elements are all overlapping with EDS, the data is difficult to analyze, while with EELS, each of the elements is clearly separated and detection is carried out with a good S/N ratio. By forming an image using some of the electrons in the spectrum that have been stained, it is possible to obtain a mapping image showing the distribution of the various elements. Fig. 11 (b) is an example of measurements of barium titanate using a dielectric, but with EDS the peaks for titanium and barium are almost completely overlapping, making analysis of composition difficult, while with EELS, there is clear a separation detected, and we can see that detailed analysis is possible.

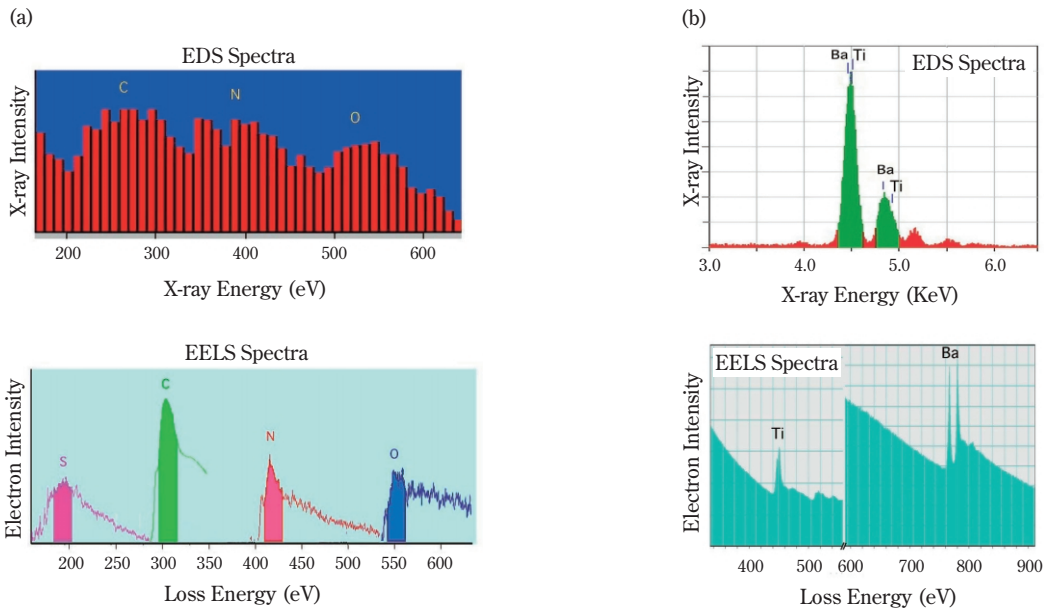
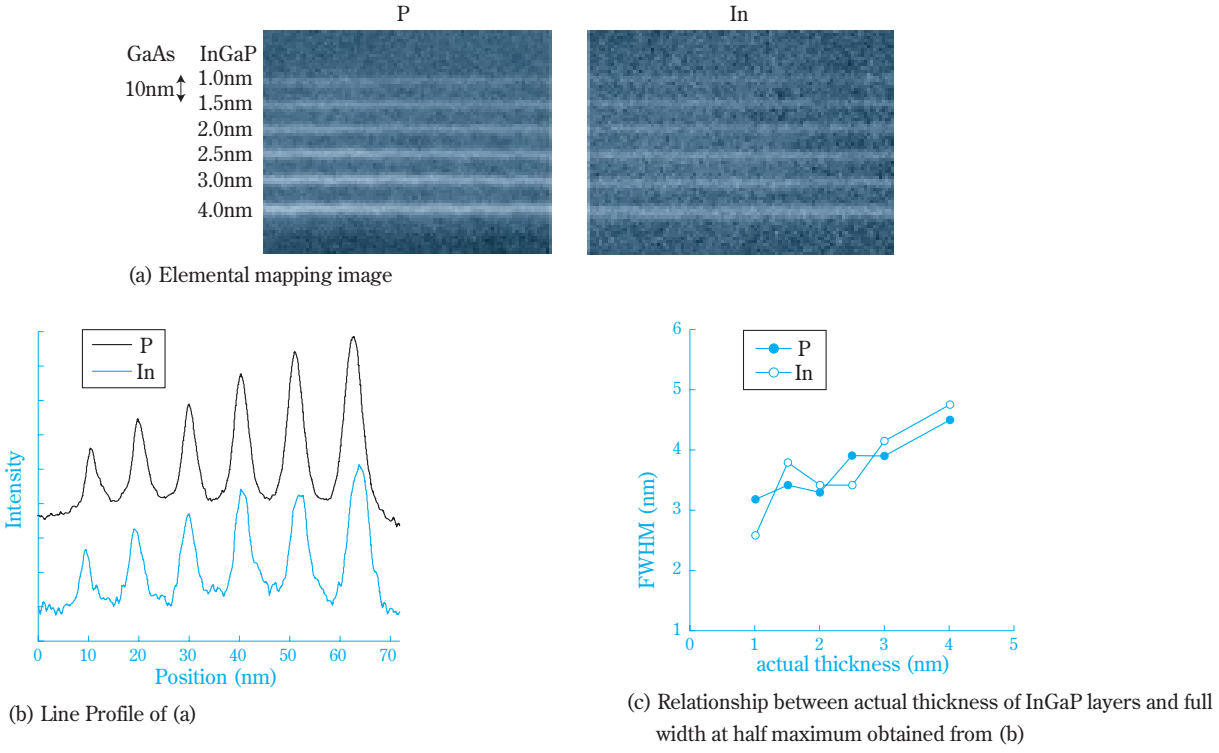


Fig. 11 Comparison of the EELS spectrum and EDX spectrum (a) Polymers, (b) Bariumtitanate

3. Examples of Applications

(1) Semiconductor Compounds [5, 6]

Fig. 12 (a) is an example of the imaging in an elemental mapping image for a semiconductor compound. This is a InGaP / GaAs multilayer film that includes InGaP layers that vary in thickness from 1nm to 4nm, and a thin sample with a thickness of approximately 100nm in the direction (orientation perpendicular to the plane of the paper) the electron beam passes through was prepared using FIB processing of the cross section of the laminated film. By optimizing the imaging conditions such as the electron beam energy used for imaging, it was possible to clearly detect the distribution of the P and In present in the InGaP layers. Plotting the peak width at half height for the peaks found from

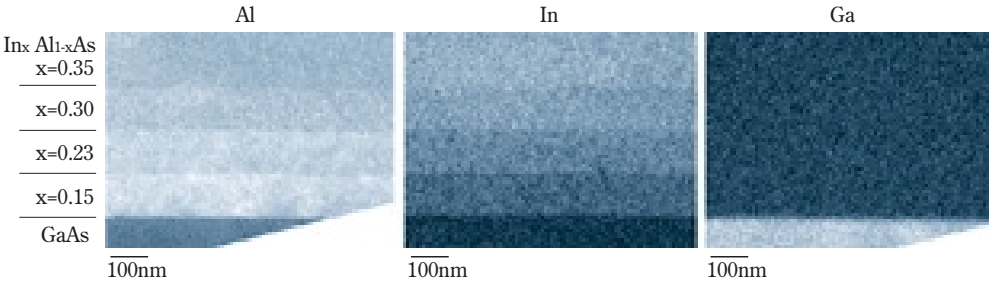


(a) Elemental mapping image

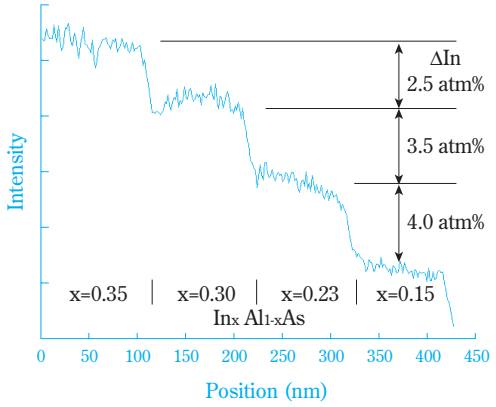
(b) Line Profile of (a)

(c) Relationship between actual thickness of InGaP layers and full width at half maximum obtained from (b)

Fig. 12 Example of elemental mapping data by EF-TEM (InGaP/GaAs multi layer)



(a) Elemental mapping image



(b) Line profile of the In mapping image of (a)

Fig. 13 Example of elemental mapping data by EF-TEM (InAlAs multi layer grown on GaAs substrate)

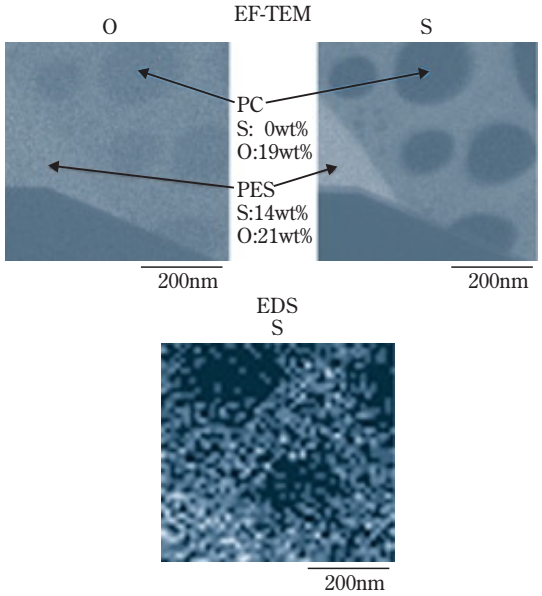


Fig. 14 Elemental mapping images of a polymer blend (PES/PC) by EF-TEM and EDS

the line profile for the mapping image versus the actual film thickness in the InGaP layers, we estimated that the spatial resolution was 2nm by extrapolating the zero point for film thickness. It can be expected that the spatial resolution will be improved by making the sample film thinner in the direction of electron beam passage and, further, carrying out processing to remove the damage arising during FIB processing after it is done.

Fig. 13 is the line profile for the elemental mapping image for the cross section of InAlAs layers grown on a GaAs substrate where the composition is changed in four gradations. The image contrast changes even for a variation in the composition of about 2 atm%, and this shows that EFT-TEM has well tuned sensitivity for small changes in composition.

(2) Polymer Materials [7)]

Fig. 14 shows an example of imaging for an elemental mapping image for a polyether sulfone (PES) and polycarbonate (PC) blade. A thin fragment of the sample with a film thickness of 40–90nm in the direction of electron beam passage formed using microtome preparation was used. Three types of contrast were obtained in the phase soluble part for the two types of polymer having only a 2wt% difference in oxygen composition, and this confirms that the EF-TEM has a high detection sensitivity for small changes in the composition of polymers. In addition, blurring of the contrast at the phase interfaces in the mapping image is 10nm or less, and we found that it is possible to analyze the elemental distribution in organic compounds with a spatial resolution of several nm. With the same sample and measurements using EDS, there is a great deal of noise and the mapping image cannot be obtained, making analysis of the details of the composition difficult.

Three-Dimensional TEM Using Tomography

1. Principles

The basic principles of tomography are shown in

Fig. 15. The TEM image contrast can be thought of as imaging of the density distribution of the sample being observed in the direction of the passage of the electron beam. The one-dimensional Fourier transform of the image formed in the direction perpendicular to the straight line that images the axis of rotation of the sample is the same as the part perpendicular to the imaging orientation for the two-dimensional Fourier transform of the sample density distribution within the plane perpendicular to the axis of rotation. Because of this, imaging is carried out from many directions, and if the function carrying out the Fourier transform for each is rearranged in a circle, the function for the two-dimensional Fourier transform of the density distribution for the sample can be found. We can obtain the three-dimensional density distribution for the sample by performing an inverse Fourier transform on this function.

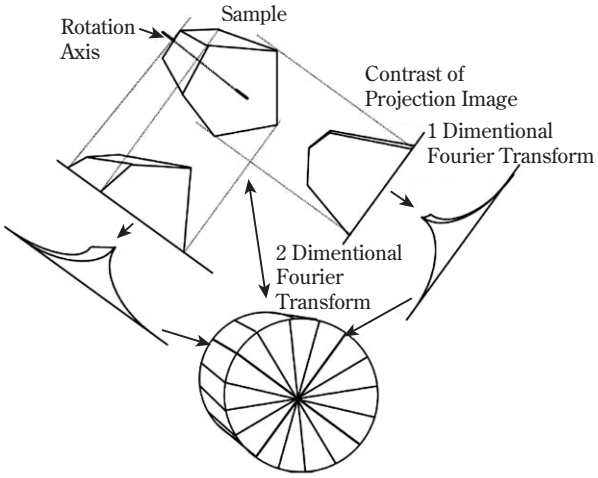


Fig. 15 Principle of tomography method

2. Characteristics

To carry out one tomographic observation, around 120 TEM images must be taken with the conditions arranged while tilting the sample, but when imaging is actually done, the sample moves out of the field of vision with the tilting. In addition, the height of the sample changes and the focal position for the lens slips, so scrupulous caution and patience are necessary. In addition, since each of the positions for the image obtained is irregular, tomography calculations cannot be made without alteration, and positioning of the images must be carried out accurately before tomography calculations. Because of this type of technical obstacle, tomographic observations with electron microscopes have not disseminated in general even though research has been going on since around 1970, and it has finally moved toward practicality because of improvements in the precision of sample stages and increased performance in CCD cameras and personal computers.

Since samples must be made into thin fragments with a thickness that an electron beam can pass through in TEM observations, there is the limitation that the thickness of the three-dimensional shape from which the tomography results are obtained be the thickness of the sample or less. The thickness for the samples that are normally used is several tens of nm, but the thicker it becomes, the greater the blurring of the image due to inelastic scattered electrons, so it becomes impossible to obtain an image that truly reflects the density distribution of the sample. The use of zero loss images that cut out the electrons that lose energy using EF-TEM is being investigated as a means for avoiding this.

3. Example of Application [8)]

Fig. 16 shows an example of tomography being applied to the analysis of the lamella structure of a styrene-isoprene copolymer. The TEM image is acquired, and even though the lamella structure being observed in the grain boundaries can be seen as being complicated because the internal structure is overlapping in the direction of observation, the structure in the direction of depth can be separated using tomography. The continuity of the lamella phase was clearly discerned.[8)] We can expect that

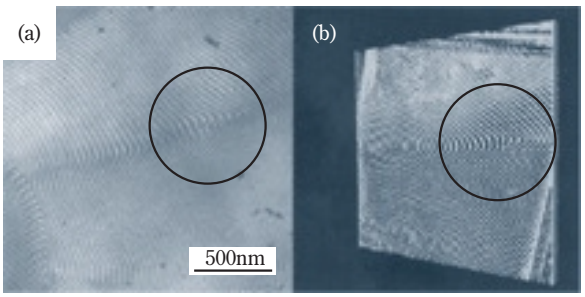


Fig. 16 (a)TEM image of the lamella structure in styrene-isoprene block copolymer
(b)Three dimension reconstruction image that were obtained by tomography method (ref.8)

progress will be made in clarifying the mechanisms for generating the properties of the materials using this kind of detailed analysis of structures.

Conclusion

A half century has passed since the development of TEM, but with the increasing necessity for structural analysis using TEM in nanotechnology, technical progress is currently still being made. In areas related to the technology discussed in this paper, there have started to be reports on examples of three-dimensional analysis of elemental distributions combining EF-TEM elemental mapping and tomography.

Besides this, important movements underway at present include the active development of technology for uniformly providing the electron beam energy incident to the sample using a monochromator and technology for increasing the perfection of lenses using correction of spherical aberration (Cs collimator) that are going on centered around Europe and the United States. These technologies make it possible to improve the probe diameter in STEM, the EELS energy resolution and, further, the spatial resolution of TEM images, and some of this is already becoming practical.

Fundamental improvements for EDS analysis are being investigated, and even though it is thought that it will be another several years before there is anything practical, there have been reports of spectra having an energy resolution equal to WDS with methods that use microcalorimetry that makes use of superconductive transitions, and on the other hand, there have been attempts at equipping TEMs with multichannel detectors in WDS.

Developments in electron microscopes are continuing in this manner, and it can be assumed that the importance in the development of advanced materials will increase even more in the future.

References

1) T.Ishitani, et.al. : J.Electron Microscopy, **43**, 332 (1994)
2) T.Matsutani, K.Iwamoto, T.Nagatomi, Y.Kimura and Y.Takai : Jpn.J.Appl.Phys., **40**, 481 (2001)
3) H.Jinnai, T.Kajihara, H.Watashiba, Y.Nishikawa and R.J.Spontak : Phys.Rev.E Rapid Communications, 64, 010803 (R) (2001)
4) Y.bando et.al. : Jpn.J.Appl.Phys., **40**, L1193 (2001)
5) M.Mitome, Y.Bando, D.Golberg, K.Kurashima, Y.Okura, T.Kaneyama, M.Naruse and Y.Honda : Microscopy Resarch and Technique, **63**, 140 (2004)
6) Y.Honda, Nanotechnology Support Project 2002 annual report, 115 (2003)
7) Y.Honda, Nanotechnology Support Project 2003 annual report, 304 (2004)
8) H.Jinnai, T.Nishi, ELECTRON MICROSCOPY, 39(1), 31 (2004)

PROFILE

Yoshiaki Honda

Sumitomo Chemical Co., Ltd.
Tsukuba Research Laboratory
Research Associate