Overview

Hitachi's Measurement and Analysis Technologies for Future Science and Social Innovation

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MEASUREMENT AND ANALYSIS TECHNOLOGIES THAT UNDERPIN SOCIAL INNOVATION

Measurement and analysis, meaning the ability to observe, measure, and analyze, are essential to the progress of science and society. Many new scientific discoveries were brought about by advances in measurement and analysis technologies, and they are collectively described as the "Mother of Science." In industry, a wide range of measurement and analysis technologies are used in everything from research and development to actual production where they underpin things like the development of new technologies and improvements in product quality. Measurement and analysis serve as a platform for innovation and the creation of innovative solutions, contributing to the progress of society by advancing a variety of fields such as the environment, renewable energy, new materials, life sciences, and electronics.

Hitachi is working on developing measurement and analysis technologies and on enhancing products and solutions, particularly electron microscopes and analyzers. Moreover, Hitachi is seeking to make advances in measurement and analysis technologies and to contribute through the latest such technologies to the progress of science and society by working not only in-house, but also through collaborative creation with universities, research institutions, and other companies in Japan and elsewhere.

Hitachi is currently promoting its Social Innovation Business, which seeks to create a safe and secure way of life by dealing with the problems of customers and society through solutions that combine advanced information technology (IT) with social infrastructure. Measurement and analysis technology underpins the achievement of social innovation by serving as a foundation for the progress of both IT and social infrastructure.

REQUIREMENTS OF SOCIETY AND DEVELOPMENT OF TECHNOLOGIES AND SOLUTIONS FOR MEASUREMENT AND ANALYSIS

Hitachi entered the measurement and analysis systems business in the 1960s, with involvement in precision measurement instruments such as flow meters, spectrophotometers[(a)], mass spectrometers[(b)], and various types of electron microscopes, including transmission electron microscopes [TEMs[(c)]], scanning electron microscopes [SEMs[(d)]], and field-emission scanning electron microscopes [FE-SEMs[(e)]]. Hitachi has a history of supplying leading-edge measurement and analysis technology by developing new measurement techniques such as non-destructive testing that use methods such as X-ray or ultrasound,

(a) Spectrophotometer
An instrument for determining the proportion of light absorbed by a sample at different wavelengths by directing light of particular wavelengths at the sample and measuring how much of the light is transmitted. Spectrophotometers are widely used to measure and view things like solution concentrations for liquid samples and physical properties or molecular structure for solid samples.

(b) Mass spectrometer
A mass spectrometer is used to identify or quantify the content of a sample by ionizing it (by a variety of means), breaking it apart electromagnetically, and detecting the constituent atoms and molecules. Many different types of mass spectrometers with different ionization or detection techniques are available to suit different end uses.

(c) TEM
Transmission electron microscope. A TEM is a type of electron microscope that passes a beam of accelerated electrons through a thinly sliced sample and directs the electrons scattered or refracted by the atoms in the sample toward a fluorescent screen or charge-coupled device (CCD) camera to form an electron beam diffraction pattern or TEM image. TEMs can perform high-resolution imaging of the internal structure of materials.

scanning probes, synchrotron radiation applications, non-invasive optical biometrics, and by improving their performance and making other enhancements.

In response to market requirements, Hitachi has also developed products and solutions that provide general-purpose measurement and analysis technologies in the form of special-purpose instruments. Examples include measurement SEMs(f) that are based on an FE-SEM and are used for measuring the dimensions of semiconductor devices, and automated clinical analyzers that are based on spectroscopic analysis technologies. Because these special-purpose products incorporate automation and operational improvements that make it easy to perform high-quality measurement and analysis, they have made a major contribution to industrial progress and to a safe and secure way of life through widespread use in areas such as manufacturing and healthcare.

Amid a shift from focusing on performance to focusing on objectives, there has been an increase recently of field-specific demands. For the development of new materials, there is demand for "operand measurement," meaning the measurement and analysis of catalysts or devices in operation, while in medicine and life sciences there is a demand for the integration and interoperation of electron microscopes that can obtain detailed morphological information from live tissue using optical measurement techniques for obtaining color information. In response to these demands, Hitachi is working on the development of new measurement and analysis technologies and new functions and solutions.

As the requirements, the objects to be measured, and the reasons for doing so become more diverse, it is important to have a wide range of measurement and analysis methods and to be able to put together an appropriate mix of equipment and techniques to suit specific objectives or requirements. In 2013, Hitachi High-Technologies Corporation consolidated the analytical instruments business within Hitachi by acquiring full ownership of SII NanoTechnology Inc. from Seiko Instruments Inc. and forming Hitachi High-Tech Science Corporation. In addition to consolidating a high level of analysis technology built up in fields such as scanning probe microscopes [SPMs(g)], optical measurement, ion optics, mass spectrometry, and thermal analysis, Hitachi High-Tech Science also intends to satisfy diverse requirements using linkage systems that integrate different measurement and analysis instruments, including electron microscopes. It has established a demonstration room equipped with various instruments that provides what is needed to run demonstrations of these instruments working together, and that it is hoped will serve as a venue for collaborative creation with customers.

DEVELOPMENT OF LEADING-EDGE PRODUCTS AND SOLUTIONS THROUGH COLLABORATION

Development of Atomic-resolution Holography Electron Microscope

Electron microscopes can observe, measure, and analyze with high resolution. The instrument that has taken this high resolution to the greatest extreme is the atomic-resolution holography electron microscope(h). With funding from the Funding Program for World-Leading Innovative R&D on Science and Technology (FIRST Program), Hitachi launched a project to develop a holography electron microscope with

(d) SEM
Scanning electron microscope. An SEM is a type of electron microscope that obtains a magnified image by scanning a tightly focused beam of primary electrons from an electron source (a device that emits a beam of electrons) across a sample and using a detector to detect the resulting electrons, which may be secondary electrons emitted by the sample or reflected electrons that are emitted when the incident electron beam changes direction inside the sample. SEMs can view the three-dimensional structure of material surfaces with high resolution.

(e) FE-SEM
Field-emission scanning electron microscope. Field emission (FE) means the high-density emission of electrons that occurs when a high voltage is applied to the tip of a sharply pointed negative electrode (electric field emission probe) at ultra-high vacuum. FE-SEMs are SEMs that use this phenomenon for their electron source, and are characterized by the high resolution that results from the very high intensity of the FE electron source.

(f) Measurement SEM
Also known as a critical dimension scanning electron microscope (CD-SEM). Measurement SEMs are mainly used for quality management on production lines for semiconductor and other electronic devices. A measurement SEM incorporates an SEM (originally intended as an observation instrument) that has been designed specifically for the measurement of microcircuit pattern dimensions by equipping it with the repeatability and calibration functions required for use as a measurement instrument.

(g) SPM
Scanning probe microscope. The general term for microscopes that performs surface profile observations and physical property analysis by scanning a small needle-sharp probe over a sample near its surface to determine physical quantities (including tunnel current, interatomic forces, friction, and magnetic force) that act between the probe and sample. Common forms of SPM include scanning tunneling microscopes (STMs) and atomic force microscopes (AFMs).

(h) Atomic-resolution Holography Electron Microscope
Electron beam holography forms an image of the interference pattern created by an electron beam behaving as a wave. Similarly, a holography electron microscope performs electron beam holography in an electron microscope. It can observe and measure the three-dimensional shape of the material and the microscopic electric and magnetic fields inside the material or in empty space by generating an electron hologram from the interference between the electron beam that passes through regions where the sample is present and the electron beam that passes through regions where the sample is absent. Because the holography electron microscope developed by Hitachi has sufficient resolution to resolve individual atoms, it is called the atomic-resolution holography electron microscope.

an accelerating voltage of 1.2 MV in March 2010. After overcoming many difficulties, this succeeded in achieving world-leading resolution of 43 pm in December 2014[(1)] (see Fig. 1).

The atomic-resolution holography electron microscope not only features high resolution, it can also measure electromagnetic fields with atomic resolution. This means it has potential for use in advancing the development of new materials that can help overcome energy and environmental problems, such as the development of high-performance magnets for hybrid or other electric vehicles, and contribute to the progress of basic science through the development of groundbreaking new materials. By using the atomic-resolution holography electron microscope as an advanced measurement and analysis platform and sharing it with others from outside of Hitachi, Hitachi intends to proceed with open innovation in collaboration with leading research institutions in Japan and elsewhere[*].

Fig. 1—Atomic-resolution Holography Electron Microscope. Hitachi achieved world-leading resolution of 43 pm in December 2014 by incorporating a spherical aberration corrector onto an ultra-high voltage electron microscope for the first time ever, overcoming numerous technical challenges relating to the performance and stability of the electron microscope itself. The microscope can measure the electromagnetic fields inside a material at the atomic level. It is anticipated that the microscope will assist in the development of new functional materials by uncovering the mechanisms that determine the performance of materials such as those used in magnets, battery electrodes, and superconductors.

Global Collaboration with CEOS of Germany and CEMES of France

Hitachi is engaged in collaboration with partner businesses and research institutions on the development of solutions and equipment. One example is a project to incorporate a spherical accelerating corrector into a large TEM with high acceleration voltage in partnership with Corrected Electron Optical Systems GmbH (CEOS) of Germany.

Spherical aberrations in the electron lenses used in electron microscopes are an obstacle to improving resolution. CEOS successfully developed a spherical aberration corrector in the mid-1990s. However, to get the best performance from the corrector, the electron microscope in which it is installed must have a high level of stability. Hitachi, Ltd. and Hitachi High-Technologies have been working with CEOS on a joint project to incorporate this spherical aberration corrector into TEMs since 2003. To date, the project has succeeded in installing the corrector in a 200-kV scanning transmission electron microscope [STEM[(i)]] and a 300-kV TEM. In the case of the atomic-resolution holography electron microscope, the installation of the spherical aberration corrector in the large, ultra-high-voltage (1.2-MV) TEM helped achieve world-leading resolution. Engaging in collaborative creation not only means working together to take on the challenges of technical innovation, it also results in close contact between the people involved. This issue of *Hitachi Review* includes an article contributed by Prof. Dr. Max Haider, who led the development of the spherical aberration corrector, that gives an overview of the joint development by CEOS and Hitachi.

In collaboration with the Center for Materials Elaboration and Structural Studies (CEMES), an institute of the French National Center for Scientific Research, Hitachi also developed and delivered an electron microscope with world-class resolution by installing an aberration corrector in a 300-kV TEM. CEMES uses TEMs for the research and development of magnetic materials for use in permanent magnets for hybrid vehicles or hard disk drive (HDD) heads. While electron microscopes use a magnetic field to

* The development process, instrument characteristics, future applications, and other details about the atomic-resolution holography electron microscope are described in reference 1) (*Hitachi Review* 64, Nov. 2015).

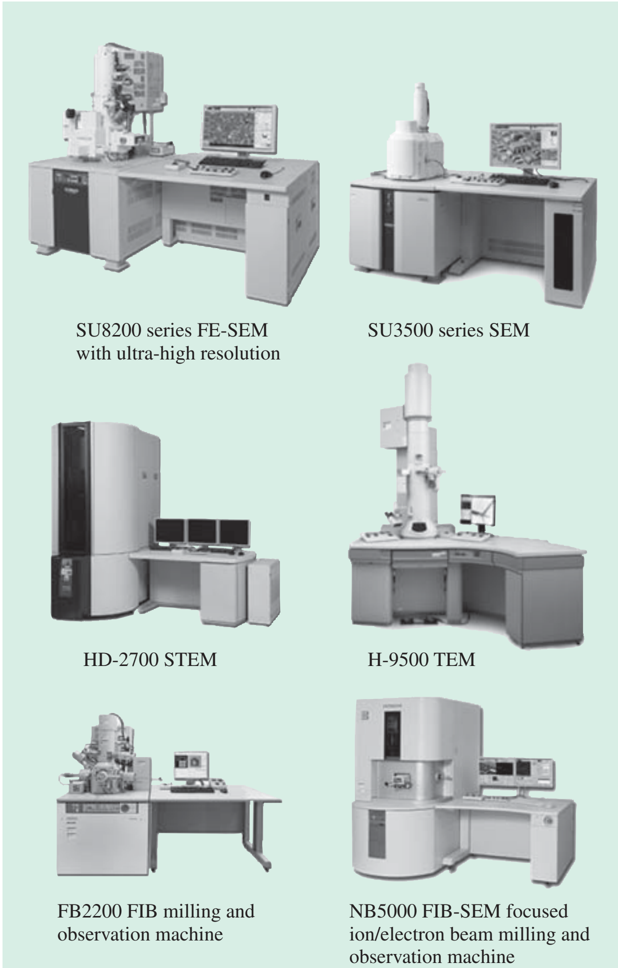
(i) STEM
Scanning transmission electron microscope. A type of TEM, an STEM scans a tightly focused electron beam over the sample (like an SEM) and uses a detector located under the sample to detect the transmitted electron beam and form an image. STEMs are used in applications such as observing grain boundaries in materials made up of crystals of different types.

CEMES: Center for Materials Elaboration and Structural Studies
FE-TEM: field-emission transmission electron microscope

Fig. 2—FE-TEM with Spherical Aberration Corrector.
The microscope was developed and supplied in collaboration with CEMES, an institute of the French National Center for Scientific Research. CEMES is recognized as a leading European laboratory for materials science and is well known for the high level of its technology for electron microscopes. I2TEM can perform both interference fringe observation and atomic-level kinetic observation (in situ observation), and so it was named I2TEM in reference to the two initial i's of interference and in situ. An inscription reading '愛^{2}TEM' (愛 is pronounced 'I' and means 'love') was added to the main body to commemorate the passion of the project participants.

control the electrons, there was a strong need for high-resolution observation of magnetic materials in an environment free of magnetic fields (where the sample is not influenced by the magnetic field used for electron beam control). A high-resolution field-emission transmission electron microscope (FE-TEM) fitted with a field emission (FE) electron gun able to achieve high resolution was used as the base instrument, and the CEOS spherical aberration corrector was installed to further improve the resolution (see Fig. 2). As a result, the instrument achieved a spatial resolution of 0.5 nm in the targeted region that was free of magnetic

(j) FIB
Focused ion beam. FIB systems are used to perform milling on the surface of a sample by scanning a very tightly focused ion beam over it, or to acquire microscope images of the exterior material by detecting the secondary electrons that are emitted as a result. They have the same design and functions as an SEM except that whereas an SEM uses an electron beam, an FIB system works by directing a beam of gallium or noble gas ions at the sample. Because these ions are much more massive than electrons, they cause spattering by expelling atoms from the sample. This effect can also be used for etching the sample to perform milling to expose a cross section for observation, and for the preparation of TEM samples by extracting a thin fragment from a particular location on the sample.



FE: field emission SEM: scanning electron microscope
TEM: transmission electron microscope FIB: focused ion beam
STEM: scanning transmission electron microscope

Fig. 3—Electron Microscope and FIB Milling and Observation Machine.
Hitachi High-Technologies has a full product range that extends from models with high resolution and performance to general-purpose models. FE microscopes achieve high resolution by using an FE electron source, a technology that Hitachi High-Technologies was the first in the world to commercialize.

fields. It is seen as having potential future uses that include improving the performance of permanent magnets for hybrid vehicles or achieving higher densities and read speeds on HDDs. Potential uses are also being sought in cancer treatment and other medical applications.

Satisfying Leading-edge Requirements for Electron Microscopes and FIBs

Hitachi High-Technologies has an extensive range of electron microscopes and focused ion beam (FIB[(j)]) products, including TEMs, SEMs, FE-SEMs, STEMs, and FIBs (see Fig. 3). The development and supply of functions and other solutions to meet leading-edge requirements for these instruments is ongoing, including improvements to resolution and other aspects of performance.

One of these leading-edge requirements is for in situ observation ("operand measurement"), meaning the observation of materials in actual use. There is strong demand for studying things like catalyst reaction mechanisms and what is happening inside lithium-ion batteries or fuel cells. In response, Hitachi High-Technologies has developed an environmental control mechanism. This uses an atmospherically isolated sample holder to prevent the sample from coming into contact with the air and is equipped with functions such as heating or gas injection. It is used for in situ observation using a variety of measurement and analysis techniques, and is supplied as a system that can be used in conjunction with an SEM, TEM, or SPM, or with sample preparation systems such as for FIB milling (see Fig. 4).

Another leading-edge requirement is for three-dimensional analysis, meaning the three-dimensional imaging and analysis of things like material composition and the internal structure of devices. To satisfy this demand, Hitachi High-Technologies has developed a realtime three-dimensional analytical combined FIB and SEM instrument (see Fig. 5). This provides an easy way to build up a three-dimensional image of the internal structure of a sample by progressively milling small quantities of material and performing automatic imaging. It achieves high resolution by using an SEM to perform the imaging during FIB milling. The three-dimensional analysis of things like composition and crystal orientation that are essential to the study of materials is also made possible by fitting additional instruments such as an X-ray analyzer or electron backscattering diffraction analyzer.

COLLABORATION WITH KEY OPINION LEADERS

To satisfy these leading-edge requirements, Hitachi is actively pursuing collaboration with key opinion leaders at universities and other research institutions in Japan and elsewhere to create new technologies and other solutions.

This issue of *Hitachi Review* carries articles contributed by leaders in the deployment of the leading-edge measurement and analysis techniques to research and development covering the latest research findings and example applications, and describing work on the development of new solutions being undertaken in partnership with Hitachi.

Application to Fuel Cell Research Using Controlled-environment SEM/STEM In Situ Observation

In situ observation of catalysts and other functional materials and three-dimensional analysis of their microstructure are being used in the development of new materials for environmental and new energy fields.

Professor Akari Hayashi of Kyushu University is researching fuel cell catalysts for the use of hydrogen energy. She had a strong need for a way to perform realtime observations of surface reactions on fuel

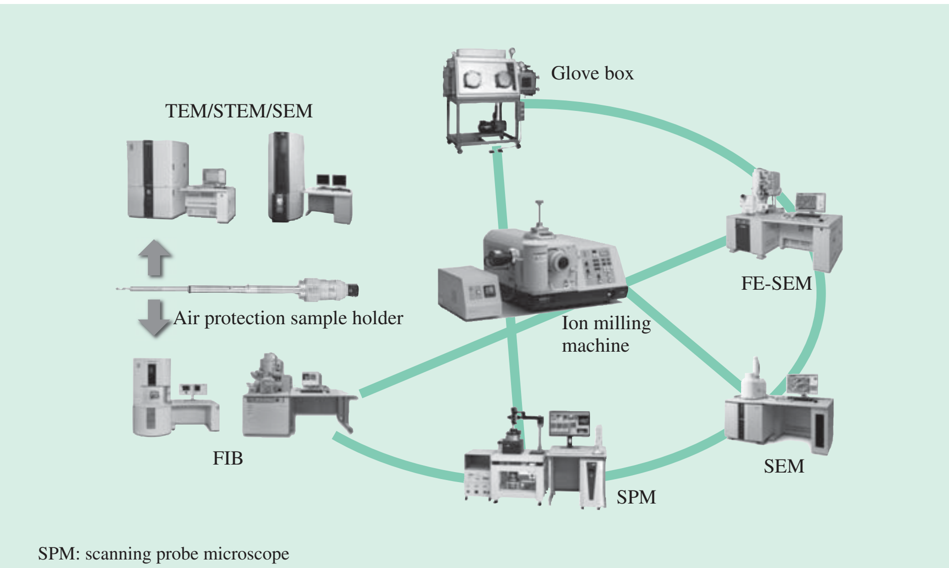


Fig. 4—Linkage System with Air Protection Sample Holder.
The sample holder is designed to prevent exposure of the sample to the atmosphere and is also equipped with functions for things like heating or gas injection. The same holder can be used in electron microscopes, SPMs, and sample preparation machines. The system combines the different types of measurements, and performs in situ measurement of samples under the conditions in which they are used in practice ("operand measurement").

Fig. 5—NX9000 Realtime Three-dimensional Analytical FIB-SEM.
The NX9000 can analyze the three-dimensional structure of microscopic regions by building up a series of consecutive cross section images through an automatic process of repeated FIB section milling and SEM observation. The three-dimensional analysis of things like material composition and crystal orientation is also possible by fitting additional instruments such as an X-ray analyzer or electron backscattering diffraction analyzer.

cell electrode catalysts under the conditions (such as atmosphere and temperature) in which the catalysts are used. The environmental control mechanism described above (see Fig. 4) was a good match for these requirements. She used the environmental control mechanism to set up an experimental laboratory for catalyst surface reactions inside an SEM or STEM and undertook in situ observations. In addition to nanoscale observations of degradation mechanisms and what changes occur under the conditions in which catalysts are used, the technique was also used to assess catalyst life.

Three-dimensional Microstructure Analysis of Materials Using Orthogonally-arranged FIB-SEM

There is growing demand in the development of new materials for three-dimensional analysis to obtain more detailed information on the internal structures of samples. It was against this background that Hitachi worked with Dr. Toru Hara, Group Leader, the National Institute for Materials Science (NIMS), to develop a realtime three-dimensional analytical combined FIB and SEM instrument (see Fig. 5) and observation techniques for using the new instrument. The instrument is used to perform detailed observation of metals, ceramics, and other materials through its ability to build up and analyze three-dimensional images by automating FIB milling and SEM observation and analysis.

Use of High-resolution SEM/STEM for Structural Analysis of Materials with Regular Porous Structure

Assistant Professor Toshiyuki Yokoi of the Tokyo Institute of Technology focuses his research in the field of resource and catalytic chemistry on zeolites, a class of high-performance catalysts that are environmentally conscious. Zeolites are microporous crystalline minerals that contain voids of uniform size at the molecular scale (0.3 to 1 nm). One of the problems with the structural analysis of zeolites under a high-resolution SEM or STEM is that the electron beam causes changes in the shape of the sample. However, by achieving higher resolution at a low acceleration voltage, the technology is contributing to the development of zeolite catalysts by making it possible to image the shape of the zeolite surface on the order of a few nanometers.

Applications in Biology and Medicine, and iPS Cell Research

Electron microscopes have been used in biology and medicine for such tasks as making structural observations of microscopic viruses, cells, and biological samples since they were first developed.

Professor Akira Sawaguchi of the University of Miyazaki has put a lot of effort into promoting the use of electron microscopes in biomedical applications, instructing young medical researchers on the importance of observing microscopic features. Meanwhile, making practical use of induced pluripotent stem (iPS) cells requires techniques for verifying their quality, and Professor Akira Sawaguchi has sought to use electron microscopy for this purpose. The professor is contributing to the study of iPS cells by enabling realtime observation with a link between a laboratory in Kyoto and the University of Miyazaki to monitor the day-by-day growth of iPS cells.

Realtime Stereo SEM for Three-dimensional Imaging of Structure of Biological Samples

Many of the biological functions of organisms have yet to be elucidated and there is a high level of demand for ways of making detailed observations of biological samples. Practice to date has been to perform dissections and observe them under an optical microscope. However, because of the limits on the observation of microscopic structures, Professor Tatsuo Ushiki of Niigata University wished to build a system that would allow dissections to be performed while viewing three-dimensional images under an SEM.

Accordingly, Hitachi High-Technologies, Niigata University, Shizuoka University, and Eizo Nanao Corporation (now EIZO Corporation) jointly participated in a Japan Science and Technology Agency (JST) program for the development of advanced measurement and analytical techniques and equipment and succeeded in implementing an SEM capable of realtime three-dimensional imaging and a high-resolution monitor for viewing the images with the naked eye. The system has been incorporated into a commercially available SEM and is used for structural observations in a variety of fields, including materials development as well as biology.

In addition to the initiatives described here, Hitachi is also participating in open innovation and collaborative creation with universities and research institutions in Japan and elsewhere involving a large number of different measurement and analysis techniques, not only electron microscopy. In the future, Hitachi intends to continue to work on the development of measurement and analysis techniques that satisfy leading-edge requirements by drawing on a global network of advanced research.

CREATING INNOVATIONS AND BREAKTHROUGH TECHNOLOGIES, DEVELOPING NEW MARKETS

In addition to creating revolutionary innovations and breakthrough technologies for measurement and analysis through the development of proprietary technologies, Hitachi is also working to expand the scope of measurement and analysis techniques.

Tabletop Electron Microscope for Observation under Atmospheric Pressure

Normally, observation using electron microscopes is done in a vacuum to prevent scattering of the electron beam due to collisions with molecules in the atmosphere. However, because water-containing samples such as biological materials are subject to evaporation in a vacuum, it has been difficult to view them in their raw state. Moreover, it was believed that obtaining SEM images at atmospheric pressure was impractical because electron beam scattering has a large effect under these conditions. For the newly developed AE1500 atmospheric-pressure tabletop electron microscope (see Fig. 6), Hitachi High-Technologies went back to the principles behind electron beam scattering to consider how to build the instrument and succeeded in obtaining crisp SEM images under atmospheric pressure by developing a correction technique that removes the influence of the scattered electron beam from the SEM image. In the future, the technology is expected to find uses in applications such as food, cosmetics, pharmaceuticals, and medicine where SEMs have not been widely used in the past.

Fig. 6—AE1500 Atmospheric-pressure Tabletop Electron Microscope.
Hitachi High-Technologies built an atmospheric-pressure SEM featuring simple sample setting by challenging the common-sense view that obtaining SEM images at atmospheric pressure was impractical due to scattering of the electron beam. Water-containing samples such as biological material can be viewed in their raw state without the water evaporating. The technology is expected to find new uses in applications such as food, cosmetics, pharmaceuticals, and medicine.

Development of New Markets Using Tabletop Electron Microscope

The TM series were the first tabletop electron microscopes to go on the market (see Fig. 7). Past electron microscopes have been expensive devices used by skilled operators. The TM series of tabletop microscopes are designed to be as easy to use as an optical microscope while still providing the resolution of an electron microscope, combining tabletop installation with simple and easy operation. They have been adopted at industrial workplaces where electron microscopes have not been used in the past. As part of its corporate social responsibility activities, Hitachi High-Technologies also uses the TM series to support science education throughout the world.

FIB-based Photomask Repair System

While FIB machines have diverse uses, Hitachi has developed a technique for repairing defects on photomasks and implemented it in the form of an FIB system specifically for use in semiconductor device manufacturing (see Fig. 8). In place of a liquid metal ion source of the type used to generate

Fig. 7—TM3030 Tabletop Microscope.
The TM3030 is a tabletop electron microscope that is easy to use, with minimal restrictions on where it can be installed. With its ability to be fitted with a cooling stage for samples and an (optional) X-ray analyzer for composition analysis, the microscope is opening up new markets such as manufacturing plants. Users include natural history museums and science museums as well as elementary schools, junior high schools, and other educational institutions.

ion beams for the past 30 years or more, Hitachi has made a breakthrough in miniaturization technology by developing a novel gas field ion source. Hitachi has achieved the minimum process dimension, optical, and other characteristics required for photomask repair by improving the resolution of scanning ion microscope images[k], and has demonstrated its use for repairing defects in photomasks or extreme ultraviolet (EUV) masks for the latest microelectronic devices.

PROVIDING SOLUTIONS IN DIVERSE FIELDS FROM ANALYSIS AND OBSERVATION TO MEASUREMENT

To perform accurate analyses efficiently in response to diverse needs, it is useful to combine a variety of different analysis techniques. Hitachi High-Technologies has been consolidating its analyzer business in Hitachi High-Tech Science. Hitachi High-Technologies intends to supply solutions to a wide variety of fields by further developing its technologies for SPM, optical measurement, ion optics, mass spectrometry, thermal analysis, and X-ray and other techniques for non-destructive testing, and by integrating measurement and analysis equipment.

(k) Scanning ion microscope image
An image generated by detecting the secondary electrons discharged when a sample is scanned with an focused ion beam (FIB). This provides better composition and crystal orientation contrast than an SEM image. The instrument that obtains the images is called a scanning ion microscope (SIM).

Fig. 8—Photomask Defect Repair System Using FIB.
The system is used to view defects on semiconductor photomasks and repair them using micro-fabrication technologies such as etching or deposition. The same system can be used for imaging, material removal and deposition using an FIB.

Fig. 9—AFM5500M SPM.
The AFM5500M simultaneously images the three-dimensional profile of a sample and maps its physical properties at the nano level by scanning its surface with a needle-sharp probe. Applications range from nano-scale research and development to industrial measurement applications such as quality management.

SPMs

SPMs can measure the surface profile of a sample or its mechanical, electrical, and other properties with high (sub-nanometer) resolution. They are suitable for making precise measurements over microscopic regions of several hundred micrometers or less. With excellent resolution in both the horizontal and vertical directions, they can perform height and other shape measurements with nanometer precision. In response to demand from industrial measurement applications, the latest SPMs (see Fig. 9) are capable of making high-quality measurements, with simple

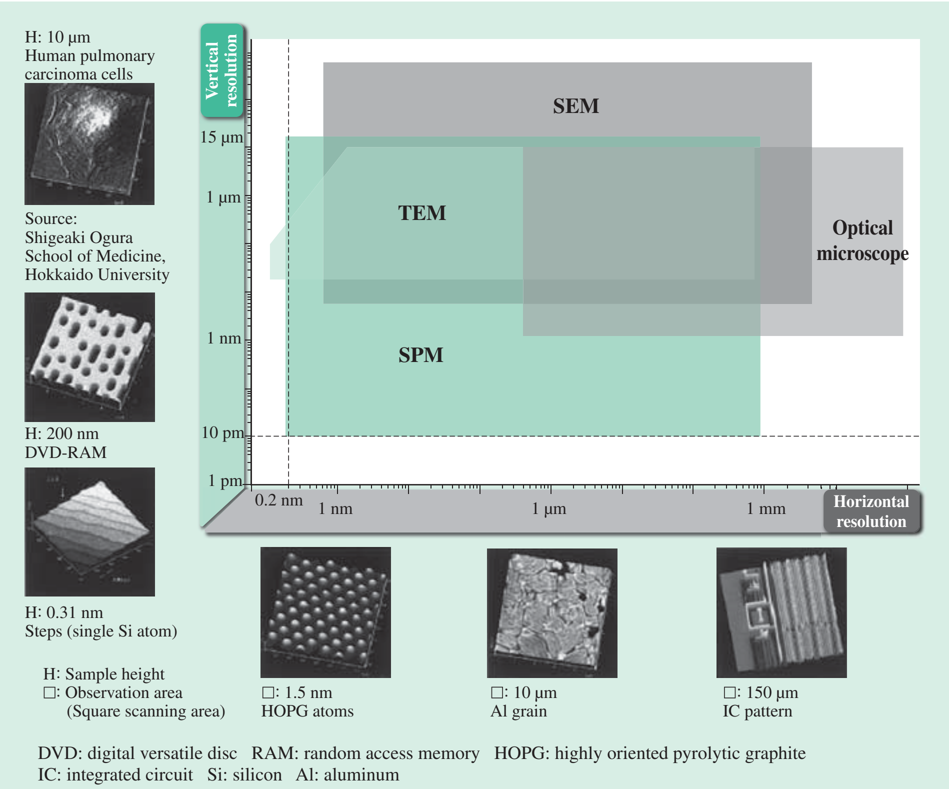


Fig. 10—Comparison of Resolutions of Different Types of Microscopes.
SPMs have excellent vertical resolution and can also measure physical properties. SPMs can be used to complement SEMs and TEMs.

operations that do not depend on the skills of the operator, thanks to improved three-dimensional shape measurement precision and improved ease-of-use through measurement automation.

Hitachi High-Tech Science has also developed a controlled-environment SPM unit that can be used to perform analyses under a variety of conditions, including in vacuum or in solution. This unit can be used to perform electrical measurements under ultra-high vacuum in a way that minimizes the influence of water adsorbed by the sample surface, and to map the properties of the sample under heating or cooling conditions. By taking advantage of these capabilities, applications for SPMs are expanding from nano-scale research and development to industrial measurement and quality control.

Microscope Linkage System

As noted above, SPMs can perform height and other shape measurements, three-dimensional length measurements, and physical properties measurements with nanometer precision over microscopic regions of several hundred micrometers or less. SEMs, by contrast, can obtain surface profile images over a wide area more quickly than SPMs. This means that SPMs and SEMs complement each other (see Fig. 10). Accordingly, the two different types of microscopes can be used together to perform complementary observations and measurements in ways that take advantage of their respective strengths.

Hitachi High-Technologies Group has developed a technique for observing the same location using a number of different microscopes by providing a shared alignment sample holder and a coordinate linkage function for SEMs, SPMs, and the coherence scanning interferometers (CSIs) described below (see Fig. 11). It is possible, for example, to measure the same location using an SEM and SPM and combine the information from the two microscopes for evaluation. This leads to the creation of new observation solutions, with one example application being a study of a lithium-ion battery that combines the contrast of an SEM with SPM surface potential observations (see Fig. 12) to display both its internal structure and the electromagnetic properties at the same location. Hitachi High-Tech Group intends to supply comprehensive solutions that use this linkage system to make it easy to integrate observation, analysis, and measurement by a number of different methods.

Coherence Scanning Interferometer (CSI)

CSIs use optical interference to perform rapid measurements of surface profile over a wide area or things like the thickness of multi-layer transparent

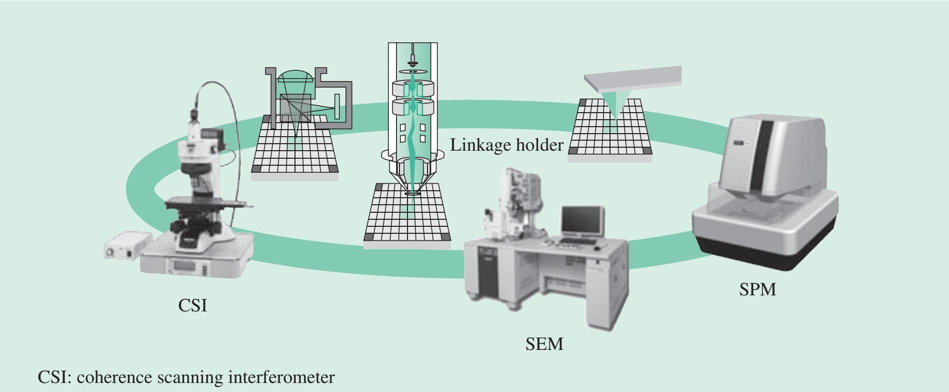


Fig. 11—Microscope Linkage System.
This system provides a simple way to observe the same sample location on a number of different microscopes (SPM, SEM, and CSI) by using a shared alignment sample holder and a coordinate linkage function.

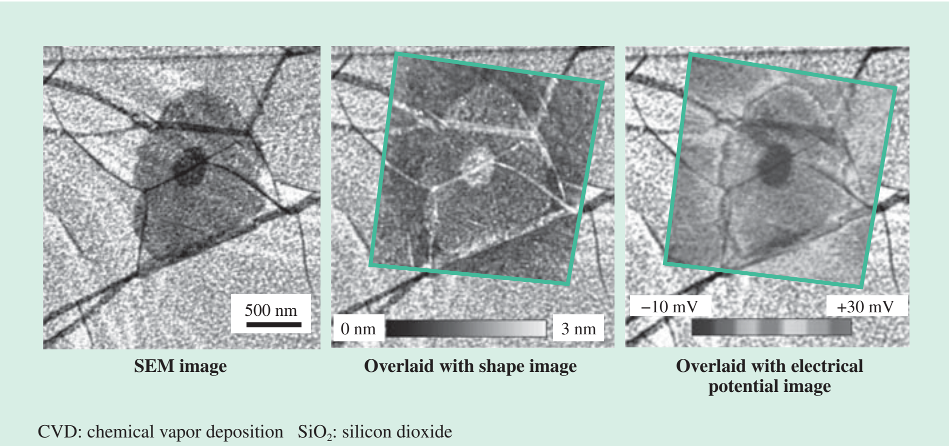


Fig. 12—Example of SEM-SPM Linkage Application.
The images show graphene grown by CVD on SiO_2. They are obtained by overlaying shape and electrical potential images acquired for the same location by an SEM and an SPM. The contrast in the SEM image comes from the SPM images of shape and surface potential.

film (see Fig. 13). They take only a few seconds to make surface profile measurements with high vertical resolution of 0.01 nm over a wide area (several millimeters). They also provide a non-contact and non-destructive way to measure the film thickness or layer cross section of multi-layer transparent film, or to detect the presence of contaminants, peeling, or other defects. A coordinate linkage function is also available for integration with an SEM or SPM. It is anticipated that the scope of applications will expand in the future to include things like control of surface roughness over wide areas or in inspection devices in the film production process.

Food Analysis Technology Driven by Fluorescence Fingerprints

The food industry has a close relationship to people's daily lives and there is growing demand for things like the identification of origin and type or composition analysis for reasons of food safety. Hitachi High-Tech Science has developed an analytical technique based on spectroscopy that uses the fluorescence fingerprints of food samples (the pattern of wavelengths, intensities, and other properties of fluorescent light) (see Fig. 14). This provides a simple and low-cost way to analyze the identity of samples and so on by efficiently acquiring large quantities of fluorescence fingerprint data using a high-speed scanning fluorescence spectrophotometer and subjecting it to statistical analysis to obtain a small number of distinctive indicators. This has been made possible by advances in information technology

Fig. 13—CSI VS1000 Series.
An optical interference technique is used to achieve both a wide field of view and high vertical resolution (two attributes that in the past were believed difficult to achieve at the same time). This takes advantage of the properties of light to combine non-contact surface profile measurement and layer cross-section measurement.

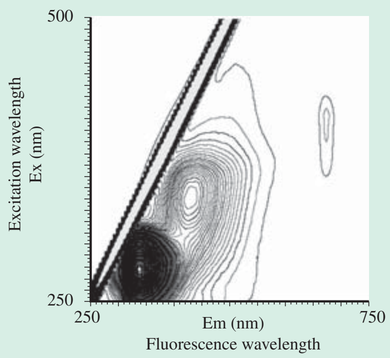


Fig. 14—Fluorescence Fingerprint Example (Pineapple Juice).
Fluorescence fingerprinting treats the fluorescence pattern obtained from factors such as the wavelength and intensity of fluorescent light like a human fingerprint. This meets demand for things like the identification of origin and type or composition analysis due to rising concern about food safety and security.

(IT) and information processing. There is interest in using the technology in the food industry, and Hitachi High-Tech Science intends to work on application development with research institutions and other partners.

Compact Mass Detector for HPLC

In the drug development and manufacturing sectors, there is growing demand for uses such as research into chemical synthesis or production management where structural and quantitative analyses are performed and composition analysis using liquid chromatography (LC)/mass spectrometry (MS) systems that combine a mass spectrometer with high performance liquid chromatograph [HPLC[(l)]]. By making the system significantly smaller and more compact with easier operation and maintenance through the development of the Chromaster 5610 compact mass detector (see Fig. 15), Hitachi High-Tech Science has overcome concerns about the installation requirements for previous large mass spectrometers and about their operation and maintenance and made it possible for HPLC users to enjoy the high level of qualitative analysis performance that is characteristic of a mass spectrometer. This issue of *Hitachi Review* describes three applications from the drug development sector [the analysis of the intermediate products of synthesis by direct infusion, the screening of microorganism culture fluid by LC/MS, and the analysis of a mixture by thin-layer chromatography-mass spectrometry (TLC-MS)]. In the future, Hitachi High-Tech Science intends to proceed with the development of applications that will expand uses for mass spectrometers by taking advantage of its simplicity.

Fluorescent X-ray Technique for Coating Thickness Measurement and Particle Contaminant Analysis

As it seeks to achieve smaller size and higher performance, the control of things like defects or particle contaminants that have an influence on quality is an important issue for the electronics sector, including semiconductors and other electronic components or lithium-ion rechargeable batteries. The sector uses non-destructive techniques such as X-ray fluorescence (XRF).

XRF analysis uses the secondary (fluorescent) X-rays emitted by a sample when it is exposed to incident X-rays to perform quantitative analysis and to identify the elements present in a sample. The technique is characterized by its speed and by its being non-destructive.

Coatings and platings are used on the electronic components, circuit boards, connectors, and other

HPLC: high performance liquid chromatograph

Fig. 15—Compact Mass Detector for HPLC.
Hitachi High-Tech Science supplies systems that are easy for a large number of users to operate in fields such as drug development by incorporating a compact Chromaster 5610 mass detector (right) into a Chromaster HPLC system (left).

(l) HPLC
High-performance liquid chromatograph. A chromatograph is a device for separating a mixture into its component parts and recovering (isolation or refinement) or quantifying them. A liquid chromatograph is used to perform measurements on liquid samples, or on solids that have been dissolved in a solvent.


XRF: X-ray fluorescence

Fig. 16—FT150 Series High-performance Fluorescent X-ray Coating Thickness Gauge.
The FT150 series gauge performs fast non-contact measurement of the thickness of the coatings (platings) used on electronic components for smartphones and other devices. It satisfies quality control requirements at leading-edge production facilities by providing a specific-purpose gauge based on XRF analysis.

items used in devices such as smartphones, and there is growing demand for ways to measure coating (plating) thickness. Hitachi High-Tech Science has utilized the characteristics of XRF analysis to commercialize techniques for exposing small areas to X-rays and the highly sensitive detection of X-rays in the form of a coating thickness measurement system (see Fig. 16). On production lines for electronic components used in smartphones and other devices, the system is helping ensure the durability of electrical connections involving the pins and other connectors on electronic components by being able to deal with plating thicknesses at the nanometer level over areas several tens of micrometers in size.

Meanwhile, the use of lithium-ion rechargeable batteries and fuel cells is growing in a variety of fields as a way to reduce the load on the environment. Management of contamination by metal particles in the production process is important for ensuring the safety and quality of these cells. Hitachi High-Tech Science has developed an X-ray particle contaminant analyzer that combines particle detection by transmitted X-ray imaging with element identification by XRF analysis in the same system (see Fig. 17). The system helps improve cell yield and operating life by dealing with small metal particles of around 20 μm in manufacturing process for products such as lithium-ion rechargeable batteries and fuel cells.

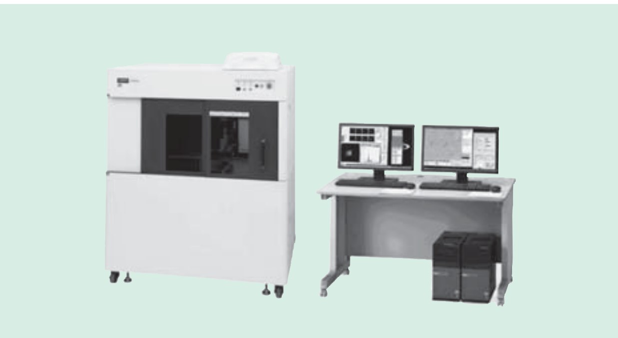


Fig. 17—X-Ray Particle Contaminant Analyzer EA8000.
The EA8000 is used to ensure the safety and quality of lithium-ion rechargeable batteries and fuel cells by dealing with contamination by metal particles in the production process. It operates quickly and efficiently, with functions that extend from the detection of metal particles to the analysis of their composition.

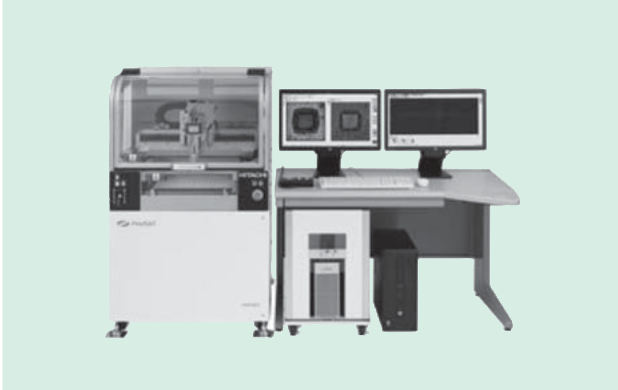


Fig. 18—Scanning Acoustic Tomograph (SAT).
The SAT machine uses ultrasound to perform non-destructive detection of cracks, delamination, and voids in electronic components. In response to demand for higher resolution and greater miniaturization, the instrument utilizes a focused ultrasound beam and image restoration to image small defects of between 1 and 1.6 μm, which are difficult to detect using conventional technology.

Non-destructive Defect Inspection Using Ultrasonic Imaging

Along with X-rays, ultrasonic imaging is another widely used technique for the fast and non-destructive detection of defects and similar in materials, with marked improvements having been made in things like resolution and detection speed. On the latest scanning acoustic tomograph (SAT) machine commercialized by Hitachi Power Solutions Co., Ltd. (see Fig. 18), the ability to detect microscopic internal defects has been improved to between 1 and 1.6 μm, more than twice the performance of previous models, by using the resolution-enhancing technology and image restoration technology to enable use with smaller semiconductors and other electronic devices. This is helping make semiconductors and electronic devices more reliable through the imaging of microscopic defects that were difficult to detect on past machines.

DEVELOPMENT OF INNOVATIVE TECHNOLOGY AND STRENGTHENING OF TECHNOLOGY PLATFORMS

Research & Development Group, Hitachi Ltd. is responsible for the development of innovative technology for measurement and analysis and the strengthening of technology platforms. The group works in parallel with product development at Hitachi, systematizing technology platforms and different fields of technology so as to strengthen and collate specific technologies that support fundamental product characteristics such as performance and reliability[(2)].

In addition to contributing to quality improvement and reliability in manufacturing at Hitachi, the purpose of the inspection and measurement technology platform for manufacturing is to provide an impetus to the inspection and measurement equipment business. Four core technologies have been selected: (1) visible inspection and measurement, (2) non-destructive inspection, (3) chemical measurement and probe imaging, and (4) optical 3D shape measurement. Hitachi is working on the development of leading-edge technologies that include techniques for the precise detection of external and internal defects and the imaging or identification of conditions or situations that are not yet evident.

In the case of the charged particle control platform for the electronics sector, Hitachi is developing measurement and processing techniques that use charged particles such as electrons and plasma neutrons or photons such as X-rays, synchrotron radiation, or laser light. In the application of electron beams to measurement, Hitachi is developing instruments such as measurement SEMs for the critical dimension measurement of semiconductor devices using an electron gun and electron optics with excellent stability and resolution. In the case of plasma control, Hitachi is using the creation and control of plasmas and the analysis of surface reactions to develop nanometer-level microfabrication techniques for semiconductors. In the case of measurements that use high-energy quantum beams such as X-rays or synchrotron radiation, Hitachi is utilizing high-intensity beam lines to develop measurement techniques for functional materials such as the computed tomography (CT) imaging of composites or chemical bonds, or in the case of the non-invasive optical measurement of biological samples using infra-red and other low-energy radiation, is utilizing things like Raman scattering of infra-red laser light to develop cell measurement techniques that do not require staining for use in regenerative medicine or drug development.

CONTRIBUTING TO SCIENCE AND SOCIETY THROUGH WORK ON ADVANCES IN MEASUREMENT AND ANALYSIS TECHNOLOGIES

Measurement and analysis technologies are used everywhere from research and development to quality management, supporting progress in science and the safety and security of society. Hitachi intends to make an ongoing contribution to the development of new technologies and solutions that bring about social innovations by continually striving to make further progress in measurement and analysis technologies and supplying the best solutions for the measurement and analysis requirements of different fields.

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