The Role of the High Voltage Electron Microscopy Station

- Electron Microscopy for Nanotechnology and the 21st Century -

Nanotechnology has attracted attention as a technology for manipulating atoms and molecules. In order to manipulate atoms and molecules more precisely, it is necessary to observe and analyze their positions and states with high accuracy and speed. For this reason, the transmission electron microscope (TEM) has assumed increasing importance. In particular, not only is it possible to understand the arrangement of atoms and molecules directly from high resolution observation images, but a total analysis of the nanostructure is also possible by combining this with spectroscopic data obtained at the same time.

The High Voltage Electron Microscopy Station (HVEMS) was created in May 2004 with the aim of realizing "electron microscopy for nanotechnology and the 21st century." As its main mission, HVEMS is conducting research activities which include advanced technical development for various kinds of TEM which would be difficult to introduce in general research institutes, pioneering materials research using these devices, and joint-use technology for making this equipment available to other nanotechnology researchers as user facilities (see figure).

Activities in advanced technical development include (1) technical development of dynamic in-situ observation techniques under various environmental conditions using electron beam and ion beam irradiation, (2) technical development to obtain high resolution and high strength/high quality electron beams by correcting aberrations in the electron lens, (3) techniques for visualization of electronic states using the energy filter electron microscope, and (4) development of a system for remote operation of electron microscopes using internet technology and effective use by a large number of researchers and students in joint experiments and educational activities. < Continued on p.2

Mission of the High Voltage Electron Microscopy Station

Advanced Technical Development
1) Technical development of dynamic in-situ observation techniques under various environmental conditions
2) Improvement of resolution and electron beam strength by aberration-corrected electron lens
3) Visualization techniques for electronic states using energy filter electron microscope
4) Development of internet electron microscopy

Pioneering Materials Research
1) Micro-fabrication on 1 nm order utilizing focused electron beam
2) Fabrication of nano-branched metallic structures using surface charge phenomenon
3) Analysis of crystallographic structures of oxide superconductors and strongly correlated electron system materials
4) Observation of magnetic structures of ferromagnets by Lorentz electron microscopy

Joint Use
External use of NVEMS’s group of distinctive electron microscopes as part of MEXT “User Support Program for Nanotechnology with In-situ, High Resolution and Analytical Electron Microscopy”

Fig. Mission of the High Voltage Electron Microscopy Station.
Research Achievements in Nanotechnology Support

In nanotechnology, simultaneously with techniques for manipulating atoms and molecules, "technology for high accuracy/high speed observation and analysis of atoms and molecules" is also indispensable. It is safe to say that the success or failure of the latter will be a determining factor in the development of nanotechnology. Because the transmission electron microscope achieves resolution in the 0.1 nm region, enabling easy observation of atoms and molecules, it is increasingly important in the field of nanotechnology. Among the particular advantages of this technique, the arrangement of atoms can be determined intuitively from atomic-level data obtained from high resolution observation images, and it is also possible to analyze the states of atoms by a comprehensive interpretation which includes data from energy dispersive X-ray spectrometry (EDS) and electron energy loss spectrometry (EELS) measured at the same time. However, these techniques are not the exclusive province of electron microscope-related researchers. Ideally, they should be used widely by nanotechnology researchers as a whole and actively employed to achieve progress in research.

Among transmission electron microscopes, the ultra-high voltage electron microscope is a particularly large-scale device, and introduction by general research institutes is difficult. Therefore, in fiscal year 2002, Japan’s Ministry of Education, Culture, Sports, Science and Technology (MEXT) began a "User Support Program for Nanotechnology with In-situ, High Resolution and Analytical Electron Microscopy" for the purpose of promoting joint use of this type of electron microscope. In this program, NIMS is the organizing institute for support with electron microscopes, and is providing opportunities for outside researchers to use its three ultra-high voltage electron microscopes, which have distinctive features suitable for nanotechnology.

FY2004 was the third year of this Support Program, which is now well-established in both operation and use and is beginning to produce significant research achievements. The number of registered users is currently 300. NIMS received requests for 58 research projects in FY2004, of which 45 were implemented. The status of publication of the results is shown in Table and Figure. Other results included two patent applications.

The fact that this large number of research results was obtained, together with the wide recognition of this analysis support program among nanotechnology researchers, has made it possible to propose research topics in a diverse range of fields. We also feel that it is necessary to develop new technologies and methods, not simply advanced devices, and introduce these into this program. In the future, while listening to a wide range of opinions from users and nanotechnology-related researchers, we will make every effort to ensure that important results are achieved in the field of nanotechnology by allowing an larger number of researchers to use electron microscope technology.

For more details: http://www.nims.go.jp/hvems/

Table Status of publication of research results.

<table>
<thead>
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<th>Publication as original paper</th>
<th>Journal publication other than the left</th>
<th>Conference presentations (oral/poster)</th>
<th>Total</th>
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<td>5 (1)</td>
<td>83 (4)</td>
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* FY2004. Figures in parentheses are numbers for publication scheduled/submission stage and not included in the left figures.

The Role of the High Voltage Electron Microscopy Station

Pioneering materials research focuses on (1) micro-fabrication of arbitrary metallic and semiconductor nanostructures on the 1 nm order utilizing a high strength focused electron beam technique, and evaluation of the properties of the resulting nanostructures, (2) fabrication of nano-branched metallic structures using the surface charge phenomenon and application to surface effect materials, (3) analysis of the crystallographic structures and local defects of oxide superconductors and strongly correlated electron system materials, and (4) observation of the magnetic structures of ferromagnets by Lorentz electron microscopy.

In the area of joint use, NIMS is the organizing institute in the "User Support Program for Nanotechnology with In-situ, High Resolution and Analytical Electron Microscopy" supported by the Japanese government’s Ministry of Education, Culture, Sports, Science and Technology (MEXT). As part of this program, HVEMS is providing opportunities for general nanotechnology researchers to use its group of distinctive transmission electron microscopes, beginning with two ultra-high voltage electron microscopes, by making these facilities available to outside users. HVEMS also provides comprehensive technical support in all stages of experiments, for example, in the preparation of electron microscope specimens, which is necessary for observation, and image analysis of the observation results.

The 21st century will be a century of progress as science and technology continue to develop a harmonious relationship with society. In this respect, we believe that electron microscope technology will not simply contribute to science and technology as a tool, but will also make a broad contribution to society as a whole through use in educational situations and diffusion of scientific and technical achievements.

For more details: http://www.nims.go.jp/hvems/
The State of the Art in Advanced Electron Microscopy

- Aberration-Correction Technology and Internet Microscopy Technology -

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Electron microscopes are increasingly important in nanomaterials research as a tool for structural analysis and composition evaluation with atomic resolution. Our group is engaged in research which includes technical development of ultimate nano analytical techniques by improving the performance of electron microscopes and technical development to provide wide opportunities for many researchers to use these high performance electron microscopes.

In principle, aberration in the axially-symmetrical magnetic field lenses of electron microscopes is necessarily positive and cannot have negative aberration like that in optical lenses. Therefore, it had been considered impossible to eliminate aberration by using multiple lenses. Recently, however, with progress in studies of aberration correction technique by using an asymmetrical magnetic field, an aberration correction device has been used practically for high resolution TEM observation. This technique is also applied in forming a finely focused electron beam. Recently, the technique of imaging and analyzing by scanning a finely focused electron beam has attracted attention, as it is possible to observe scanning images and analyze with atomic resolution if an electron beam can be focused at less than atomic size. We have developed an aberration correction device composed of 12 poles for a illumination lens system which enables correction of high order aberration (see Fig. 1), and have now confirmed that this device makes it possible to obtain an electron beam more than one order stronger than that with the conventional technology while maintaining focusing at the atomic size.

As the second area of our work, because advanced electron microscopes are not easy to operate and maintain, we developed a remote operation system utilizing the internet in order to make these advanced electron microscopes widely available to a large number of researchers. As shown in Fig. 2, this system enables free operation from a web browser by accessing the electron microscopes at NIMS via the internet. As one application, operation terminals for these electron microscopes have been introduced at Super Science High Schools designated by the Ministry of Education, Culture, Sports, Science and Technology (MEXT) beginning in 2003 and are now being used in practical situations such as classroom work and club activities. Declining interest in the sciences has become a problem in recent years, and from this viewpoint as well, more interactive experiential learning is gaining popularity as an alternative to passive education. At present, high school students are sending NIMS samples obtained in their own experiments and outside practical training, and are themselves observing and analyzing these samples using this internet electron microscope system. Joint research with universities and other research institutes employing this remote operation technique has also begun.

For more details: http://www.nims.go.jp/hvems/nano_char/ENG/E-researchtopics.html

NIMS Holds Workshop with Polish Institute

(October 12-14, Poland) -- The 2nd Japan/Poland materials workshop was held at Poland’s Institute of Nuclear Chemistry and Technology (INCT). The topic of the event was “Materials for Sustainable Development in the 21st Century.” The workshop was planned on the occasion of the signing last year of a memorandum of understanding (MOU) between the NIMS Ecomaterials Center (EMC) and INCT. Characterized by lively discussions, the event featured 11 lectures from the Japanese side, including 9 by NIMS members, and 14 lectures from the Polish side, by researchers from the Warsaw University of Technology and Jagiellonian University as well as the INCT. In closing the workshop, the two sides considered the possibility of future cooperative research relationships and ongoing workshops for the creation of a recycling-based society.
Materials Research using Cryogenic-Temperature Lorentz Electron Microscope

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The interior of a magnet is an aggregate of small regions called magnetic domains. Individual domains are in fact small magnets, which are arranged in the magnet so as to form the most stable state. The magnetic domain structure is particularly important in fields where magnets are applied. For example, the magnetic domain itself corresponds to one data bit in magnetic recording media. This means that reducing the size of magnetic domains as far as possible is one key to high density recording. Moreover, high resolution observation of these fine magnetic domain structures has also become important.

We are therefore carrying out research in which we are investigating the magnetic domain structures of advanced magnetic materials using the Lorentz electron microscope. The Lorentz electron microscope is one type of transmission electron microscope, and is a device which is capable of observing the magnetic domain structure in the interior of specimens of ferromagnetic materials by taking advantage of the fact that the incident electrons are deflected by the Lorentz force corresponding to the magnetization in the specimen. Distinctive features of Lorentz electron microscopy include high spatial resolution on the nano order and the possibility of dynamic observation in real time.

**Figure 1** is a Lorentz electron microscope image at a temperature of 17 K.

![Fig. 1 Lorentz electron microscope image of (La, Sr)₃Mn₂O₇ at temperature of 17 K.](image)

**Figure 2** shows the results of elemental mapping of (La, Sr)₃Mn₂O₇. As in Figure 1, the counterparallel magnetic domains have formed alternating layers, but here, their direction is perpendicular to the c axis and their width has become extremely narrow, at ≈50 nm.

We think that the formation of this magnetic domain structure having a nano-order width is related to the crystal structure of the substance and the orbital states of the electrons.

Note: This work was carried out as joint research with Dr. Tsuyoshi Kimura of the Los Alamos National Laboratory and Prof. Yoshinori Tokura of the University of Tokyo.

For more details: [http://www.nims.go.jp/aperiodic/hrtem/](http://www.nims.go.jp/aperiodic/hrtem/)

Advanced Materials Analysis using TEM-EELS

There are many examples in which the macroscopic properties of materials and devices depend on the distribution of elements and chemical bonding states in extremely small regions. For example, in the dielectric thin films used in semiconductor devices, the interface with the substrate determines the properties of the device. High voltage electron microscopy already has spatial resolution which enables direct observation of crystallographic structures, but its discrimination in analyzing elements and chemical bonds is still inadequate. Therefore, we are analyzing elements and chemical bonding states at the sub-nanometer level using electron energy loss spectrometry in the transmission electron microscope (TEM-EELS).

As a typical example of elemental analysis, **Fig. 1** shows the results of elemental mapping of a silicon nitride-based sintered compact (Si₃N₄) with added oxides. Although TEM images are essentially monochromatic, we obtained an RGB color image by energy-filtered observation of electrons after inelastic scattering by the respective elements Si, N, and O. This revealed that the added oxides exist at the grain boundaries. Spatial resolution in elemental mapping is currently on the order of 0.5 nm. This
Precise Analysis of Structural Order and Disorder in Co-Based Layered Cuprates

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Direct observation of the atomic arrangement using the transmission electron microscope is an extremely important experimental technique which makes it possible to evaluate the internal structures of materials at the atomic level. This method, which is called high resolution electron microscopy, also demonstrates great effectiveness in the analysis of substances with unknown structures and analysis of complex fine structures and modulated structures. Using an ultra-high resolution ultra-high voltage electron microscope which has extremely high resolution of approximately 0.1 nm, we are engaged in the analysis of complex superstructures and fine structures of strongly correlated electron system materials such as Cu oxide (cuprate) high temperature superconductors and colossal magnetoresistance (CMR) manganese oxides, which have attracted great attention as candidate materials for nanoelectronics. As one example of work, here, we will describe the analysis of a Co-based layered cuprate.

Fig. 1 is a high resolution electron microscope image of the Co-12s2 phase (s = 1-3) of a series of Co-based layered cuprates, which are expressed by the compositional formula CoSr$_7$(Y,Ce)$_s$Cu$_2$O$_{5+2s}$. The image was obtained at an acceleration voltage of 820 kV. The black dots correspond to the component atoms. The layers with the brightest contrast are charge storage blocks which include Co atoms. The fact that their spacing becomes larger as the value of s increases can be seen clearly in the image. In all of the phases, two CoO$_4$ tetrahedral chains, which have a symmetrical mirror-image relationship, are arranged alternately within the layers, as shown in Fig. 2a, and in the 1212 phase (s = 1), regular ordering was also confirmed in the interlayer direction, as shown in Fig. 2b. Furthermore, as shown in Fig. 2c, it was also found that the ordering in the interlayer direction becomes disordered in the 1222 phase (s = 2) and the 1232 phase (s = 3), where the distance between the charge storage blocks becomes longer. The existence of CoO$_4$ tetrahedral chains in Co-based layered cuprates had been suggested based on neutron diffraction experiments. However, this complex form of ordering was revealed for the first time by applying the high resolution electron microscopy.

These results show the relationship between the basic structure and layering pattern of charge storage blocks in layered cuprates, and are therefore considered useful for the design and development of new superconductors. We hope to contribute to the progress of nanotechnology by developing more advanced analytical techniques using the high resolution electron microscopy.

For more details: http://www.nims.go.jp/aperiodic/hrtem/

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is determined by electro-optical problems due to chromatic aberration in the objective lens and the principle of indeterminacy in quantum mechanics, in that the inelastic scattering process does not exist locally.

In order to investigate chemical bonding states, high energy resolution sufficient to enable analysis of the electronic structure is required. Using a field emission electron gun and original measurement and analysis software developed by the High Resolution Characterization Group, we realized the highest energy resolution (0.23 eV) with a conventional-type TEM. We also made measurements at the sub-nanometer level using an original method called spatially resolved EELS. Figure 2 shows an example of analysis of a dielectric thin film for use in semiconductor devices. Here, the bonding state of Al and Si was determined at intervals of 0.28 nm. Applying this technique in combination with first-principles calculation, we found that the coordination number of Al atoms changes depending on their depth, the Si substrate is oxidized, etc. We also developed a material characterization method using TEM-EELS and are engaged in research on application to various materials.

Note: Portions of this research were carried out under the NEDO/MIRAI project and the Nanotechnology Support Project.

For more details: http://www.nims.go.jp/aperiodic/hrtem/recent/eels-e.html

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Fig. 2 Example of analysis of dielectric thin film for use in semiconductor devices.
Electron beam induced deposition (EBID) is a useful method of fabricating nanostructures which takes advantage of the fact that metals and their compounds can be deposited by vapor deposition when an organometallic precursor gas is decomposed by electron beam irradiation and the target nanostructure can be fabricated by controlling the size and position of the beam. In general, nanostructures are fabricated using an electric conductor as the substrate, but in this case, the size of the structure depends on the size of the electron beam. Thus, in order to fabricate a nanostructure, the beam must be smaller than the size of the nanostructure.

In contrast to this conventional technique, we succeeded for the first time in the world in fabricating a nanosized branched structure with branches less than 3 nm in size and nanowires with diameters of less than 3 nm by using an insulator substrate. This fabrication method has various advantages. For example, the distinctive size of the structure (size of branch, diameter of wire, etc.) is virtually independent of the size of the irradiated electron beam because this technique utilizes the fact that surface of the insulator substrate is charged by electron beam irradiation, fabrication of different distinctive structures is possible by controlling the intensity of the electron beam, etc.

As an example of the fabrication of these nanostructures, using a transmission electron microscope equipped with a 200 kV field emission electron gun, an electron beam was irradiated on an insulator substrate while controlling the irradiation position with nanometer precision. Use of a precursor gas supply system made it possible to introduce gas containing the target element on the surface of the substrate while maintaining high vacuum in the electron microscope specimen chamber and fabricate nano-branched and nanowire structures on the substrate. Figure 1 shows a schematic diagram of the fabrication process.

Figure 2 shows an example of the results. Here, W(CO)6, which is a species of organometallic gas, was introduced in the vicinity of the surface of an Al2O3 insulator substrate, and a nanowire structure grew when the electron beam was irradiated on the substrate. The wire-like substance which can be observed in the figure has a diameter of less than 3 nm. By controlling the irradiation conditions, we also succeeded in fabricating nanodendrite and nano-branched structures.

An analysis of the fabricated nanostructures revealed that these structures consist mainly of the target element, have a high content of nanosized crystal grains, display electric conductivity, and have an extremely large surface area. This technique enables easy control of the size of the structure as a whole and the location where the structure is formed. Furthermore, numerous combinations of substrates and gas sources can also be used. Based on these features, application of the results of this research in a wide range of fields is expected, including research and development on the fabrication/arrangement of surface effect devices, sensors, nanosized catalysts, and catalyst carrier structures, and in the chemical, biotechnology, and pharmaceutical industries, among others.

For more details: http://www.nims.go.jp/hvems/nano_char/ENG/E-researchtopics.html

MOU with Germany's University of Hannover

(October 19, Hannover) -- The Ecomaterials Center (EMC) signed a memorandum of understanding (MOU) on research cooperation with the Institute of Technical Chemistry (TCI), University of Hannover, Germany. The two institutes plan to promote exchanges of researchers, exchanges of research information, and joint research in fields related to basic and applied research on photocatalyst materials. TCI has carried out numerous deeply-interesting research studies linked to photocatalysts and nanotechnology. High expectations are placed on joint research achievements.
**Nanofabrication at the 1 nm Level using Focused Electron Beam**

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Recently, focused ion beam (FIB) devices have been widely used in the preparation of electron microscope specimens. For example, decomposition of gases such as tungsten hexacarbonyl (W(CO)$_6$) and phenanthrene (C$_{14}$H$_{10}$) using an FIB beam and deposition of these substances to form a protective layer for regions to be observed with the electron microscope has become a routine procedure. This is one example of a practical use of the technique called beam induced deposition. Using this method, it is possible to fabricate nanostructures by a local vapor deposition process in which a precursor gas such as an organometallic gas is introduced on the surface of a material and the gas is then decomposed by irradiation with a tightly focused ion beam or electron beam.

Based on the fact that an electron beam can be focused more tightly than an ion beam, research is being carried out on electron beam induced deposition (EBID) as a method of fabricating smaller structures. We are currently engaged in groundbreaking research on the fabrication of 1 nm level nanostructures, which had been considered impossible, using an electron beam obtained with the high acceleration voltage of the transmission electron microscope (TEM).

**Figure 1** is a photo showing the appearance of the ultra-high vacuum TEM (JEM-2000VF) being used in this research. In addition to adapting a conventional TEM column for use under an ultra-high vacuum, a large number of ports were provided in the specimen chamber, making this device suitable for a variety of experiments. **Fig. 2a** and **Fig. 2b** show the results of high resolution electron microscope observation of nanodots which were obtained by passing W(CO)$_6$ in the vicinity of an Si substrate and irradiating an electron beam under positional control. **Fig. 2c** shows the results of observation by high-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM). Using an irradiation time of 5 sec, nanodots were fabricated at the positions where the white lines in the figure intersect. Observation with the conventional high resolution electron microscope would be difficult at this size. However, because HAADF-STEM makes it possible to obtain stronger contrast based on differences in atomic number, the existence of heavy elements can be clearly confirmed by this method.

This technique has a number of potential applications, such as control of the arrangement of catalyst particles at the 1 nm level. Moreover, because a high level of electron microscope technology is necessary in evaluating these microscopic structures, a synergistic effect can also be expected from development in both directions.

For more details: [http://www.nims.go.jp/hvems/nano_char/ENG/E-researchtopics.html](http://www.nims.go.jp/hvems/nano_char/ENG/E-researchtopics.html)

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**NIMS Winter Events 2005**

**Crystal Festival (Nov. 24, 2005)**

- Prayer for workplace safety.
- Comedy show by executives.

**Xmas & New Year Illumination Display**

*(Dec. 16, 2005 - Jan. 8, 2006)*

Illuminations designed by local students and made by NIMS volunteers.
Hello from NIMS

I was very much both pleased and surprised by this invitation to write a short essay for "Hello from NIMS." On the one hand, after I have spent more than 10 years in Tsukuba, it would probably be strange to begin with the usual words: "Hello! My name is Igor Solovyev. I came from Ekaterinburg (Russia), and I love Japan!" I do have a great sense of respect for Japan and the Japanese people, but after 10 years it becomes more than words, because if you think in a different way, what holds you here? It is also impossible to squeeze all of the last 10 years of my life here into a couple of sentences in this essay. I anticipate many "frequently asked questions" and will try to respond to some of them. (1) Ekaterinburg is a big city located in the Ural area near the border between Europe and Asia. (Geographically, it is in Asia, and in this sense, there is no difference from Japan!) We have a long winter and plenty of snow, which I miss here... (2) The most difficult question for me is "Are you fluent in Japanese?" To tell the truth, I am not, and typically this "lack of education" is counterbalanced by the efforts of my family: my wife and two very much "Japanized" children.

My research field is first-principles electronic structure calculations, strong correlations and magnetism in transition-metal oxides. I have had a chance to work in several research centers in Japan. Ironically, all that time I was living near NIMS and could see this huge Sengen building almost every day, but I had never been inside this Institute before I finally joined it in 2005. I was impressed by the "guidance tour" organized for all newcomers (and I had to regret again that I had not learned Japanese before in order to understand it!). I am looking forward to many collaborations, and I hope that I will be able to learn and contribute to research projects with titles containing the two magic words "nano" and "bio."

Hello there! I am Yan Ma, a Ph.D. student in the Light Materials Group of the Materials Engineering Laboratory. This April I left my hometown, Beijing, where I had worked for nearly 10 years in the field of nuclear reactor materials. With a great desire to improve my scientific research ability, I came to NIMS and started my Ph.D. program in a new research field, Ni3Al intermetallic compounds. Ni3Al has been known as an excellent high-temperature structural material, but recently my group has found that it displays catalytic activity for hydrogen generation from methanol. So I am studying the catalytic properties of Ni3Al for steam reforming of methane, considering a new application in fuel cells.

Together with high-level research, NIMS has a beautiful natural environment, artistic courtyard and clean workshop. It gives me an undisturbed life to study here. Furthermore, the people in my group are very kind and warmhearted to me, especially my supervisor. When I have some trouble in my study, I always think of his words: "just try your best." Every year at the NIMS Matsuri (festival), more than 20 Chinese colleagues gather to make our traditional food, a kind of dumpling called gyouza. Moreover, people from different countries with different cultures and backgrounds come together, which gives me an open window from which I can look out on the world far beyond my research field.

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