NIMS NOW International

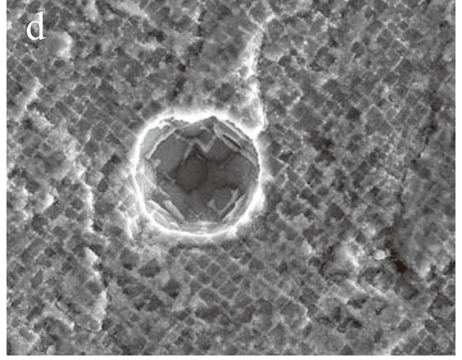
2010. June

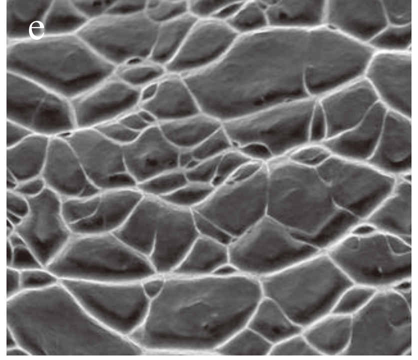
Increasing the Reliability of Materials

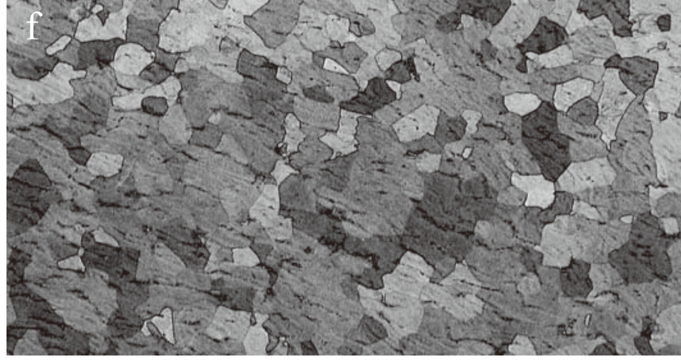

Field Emission-Scanning Electron Microscope (FE-SEM)

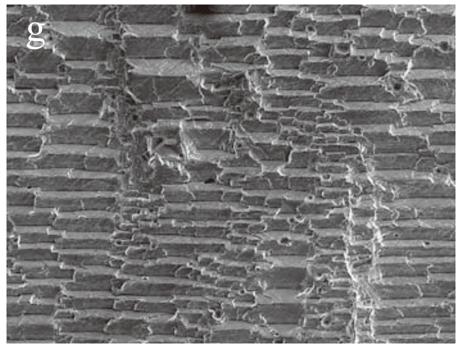
Increasing the Reliability of Materials

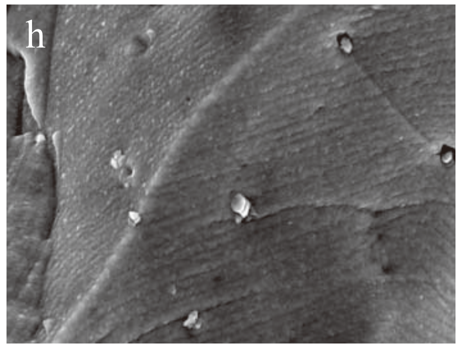
Every materials on earth are constantly exposed to weather, or may be damaged in severe environments. The same is also true for rocket engines during short trips into space. Investigating this kind of damage to materials is the job of the NIMS Materials Reliability Center. We collect and provide information on the mechanisms of corrosion and fatigue in the materials used in society, and we also conduct on-the-spot investigations of accidents where fatigue is the suspected cause. This issue of NIMS NOW introduces the activities of this team of experts as they explore the reliability and safety of material objects.

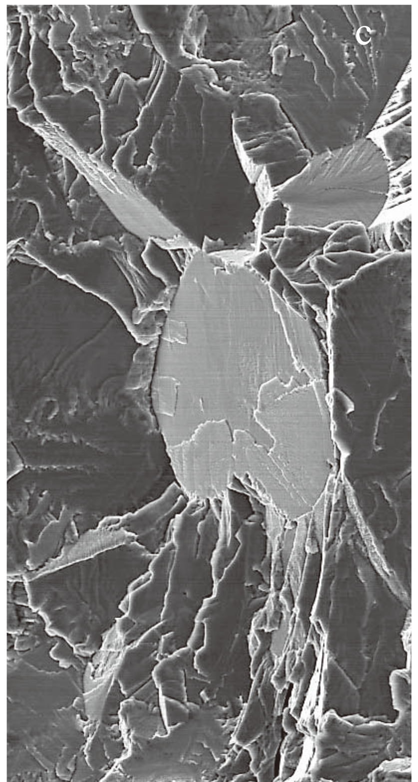












Activities of the Materials Reliability Center

Toshio Ogata, Managing Director, Materials Reliability Center

The Materials Reliability Center is working to enhance the safety and reliability of the equipment and structures that are widely used in society. The Center researches and evaluates the composition of materials, their use environment, and other factors that affect the strength properties and life of structural materials, and widely provides material property data which are also used in the design of equipment and structures.

A team of experts providing valuable feedback to society.

The Materials Reliability Center has an organization consisting of 6 groups, the Creep Resistant Materials Group, Fatigue Group, Corrosion Group, Special Environment Group, Micro-Nano Materials Engineering Group, and Materials Creation and Reliability Group. The researchers in the first 4 groups are concurrently assigned as staff of the Data Sheet Station, where they are engaged in the business of publishing Data Sheets in compliance with an ISO 9001 Quality Management System.* Data Sheets reflect the needs of society, which is our ultimate client. Because these Data Sheets are also referenced in the design of structural materials and in related standards, this is important work with a social deep relationship and contribution.

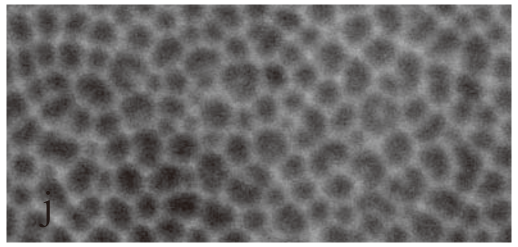
Because NIMS is a public research institution in the field of materials, the Materials Reliability Center accepts requests for accident investigations every year. These investigations require at least 2-3 months before a report can be prepared.

Unlike the publication of scientific papers, the preparation of Data Sheets and accident investigations are jobs which are difficult to evaluate in terms of research results and individual performance, and from the viewpoint of working time, they must be given higher priority than research activities. Nevertheless, the Materials Reliability Center carries out these works with awareness as a team of professionals that contributes to society by providing everyday research results as valuable feedback.

A high evaluation of the Materials Reliability Center, both in NIMS and in the broader society.

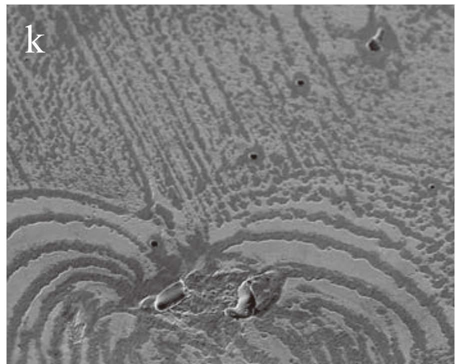
The Materials Reliability Center has received commendations recently, from both inside and outside NIMS. In March 2009, the Special Environment Group and Fatigue Group received a Letter of Appreciation from the President of the Japan Aerospace Exploration Agency (JAXA) for their work in connection with space use materials, and in April 2010, the Corrosion Group and Fatigue Group received the NIMS President's Commendation for their achievements in accident investigations.

*ISO 9001 (JIS Q 9001): This is an international standard for quality management systems, rather than for product quality as such.



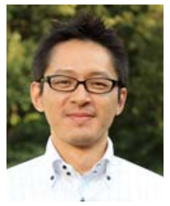
(a) The Japanese HII-A launch vehicle.
(b) Members of the Materials Reliability Center introduced in this issue.
(c) Features observed at fatigue fracture surface of titanium alloy (SEM image).
(d) Microstructure and small pores observed at fatigue fracture surface of Ni-base single crystal (SEM image).
(e) Dimple pattern observed at ductile fracture surface of titanium alloy (SEM image).
(f) Microstructure of titanium alloy (OM image).
(g) Features observed at fatigue fracture surface of Ni-base single crystal (SEM image).
(h) Features observed at fatigue fracture surface in weld of Ni-base alloy (SEM image).
(i) NIMS staff providing guidance for atmospheric exposure test in the Philippines.
(j) Microstructure observed at electrolytic polished surface of aluminum alloy (SEM image).
(k) Features observed at origin of fatigue fracture of Ni-base single crystal (SEM image).
(l) Metallic microstructure of Ni-base alloy (SEM image).

(Fractography images : Hideshi Sumiyoshi)
SEM: Scanning Electron Microscope, OM: Optical Microscope



Reliability Assessments in Extremely Severe Environments

Contributing to the Japanese Space Rocket H-IIA



Toshio Ogata
Special Environment Group,
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Special Environment Group,
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From space rockets to linear motor cars.

The Special Environment Group of the Materials Reliability Center collects and accumulates data on the mechanical properties of materials in environments at cryogenic temperatures (-200°C and lower) and high pressure hydrogen environments for structural materials that are actually applied in space rocket engines, which are used at the temperature of liquid hydrogen (-253°C and higher), and equipment for superconducting applications such as nuclear fusion reactors and magnetic levitation motor cars (linear motor cars), which use liquid helium (-269°C), and in research aimed at increasing their reliability.

The activities of the Special Environment Group include investigation of the test conditions for tensile tests, fatigue tests, impact tests, and other tests performed at cryogenic temperatures and the development and establishment of related test methods, accumulation of material test data with high value at the world level using the developed test devices and test methods, and elucidation of the unique properties of materials in cryogenic environments, including deformation behavior, microstructural changes, etc.

Contribution to the H-IIA rocket.

Following the accident involving the No. 8 H-II rocket in 1999, which was a Japan's self developed launch vehicle, data obtained using these testing techniques provided useful reference for the elucidation of the cause of the accident in the accident investigation committee. The NIMS Special Environment Group also made an important contribution to the elucidation of the cause of the failure, by conducting material tests at cryogenic temperatures at the request of the committee, and also to the analysis of the fracture surface of the failed rocket engine, which was pulled up from the ocean bottom at a depth of 3000m.

Following this, NIMS concluded a joint research agreement with the Japan Aerospace Exploration Agency (JAXA; then the National Space Development Agency of Japan, NSDA) in connection with "Coordination of Data on the Strength of Materials for Use in Domestically Developed Rockets." Under this agreement, the NIMS Special Environment Group constructed a database and publishes annual data sheets (NIMS Space Use Materials Strength Data Sheet) on materials which are practically used in liquid fuel rocket engines, not limited to the cryogenic temperature of the liquid hydrogen environment, but also extending to the high temperature engine combustion chamber.

Fracture characteristics were different at cryogenic temperatures.

In research on the fracture mechanisms of these materials, in titanium alloys researchers found a fatigue fracture accompanying twin deformation (**Fig. 1**) occurred at cryogenic temperature and are investigating the relationship between the occurrence of this fatigue fracture and the lower fatigue strength at the temperature. In Ni-base super alloy the group clarified the correspondence between the pattern (striations) on the stripes that are visible at the fracture surface where a fatigue crack has propagated, and the results of fracture analysis based on the stress intensity factor range ⊿K (**Fig. 2**).

The material properties collected and assessed in research led by this NIMS group were reflected in changes in the engine design standard for the H-IIA rocket, and in improvement in operating conditions. This made an important contribution to improvement of the reliability of the H-IIA rocket and to continuing successes in rocket launches.

In the future, these test technologies and the evaluation of the properties will be necessary and indispensable for the development of Japan's next-generation domestic rocket. However, the work of the Special Environment Group is not limited to evaluation of material properties. The group is also participating in studies aimed at achieving high reliability and reducing costs in the H-IIA rocket, and is contributing to overall inspections and investigation of defects in parts of onboard equipment of space rockets.

In March 2009, the Special Environment Group received a Certificate of Appreciation from the President of JAXA for its many important contributions to space transportation systems, beginning with the domestic launch vehicles H-IIA and H-IIB[1)].

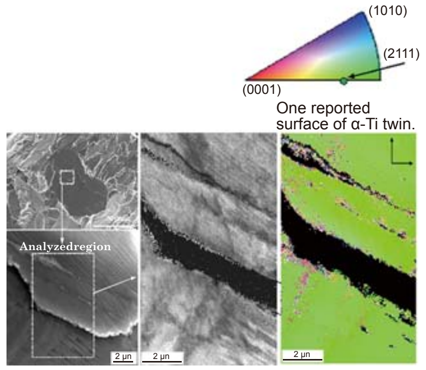

Fig. 1 Results of an analysis of the cryogenic fatigue fracture surface of Ti-5Al-2.5Sn ELI by the SEM-EBSD method (SEM-EBSD: Scanning Electron Microscope-Electron Back Scatter Diffraction).

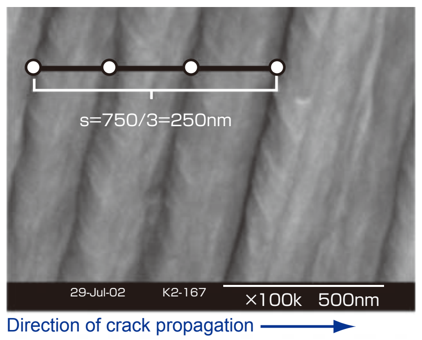

Fig. 2 Striations observed in the vicinity of ⊿K =34MPa・$\sqrt{m}$ in Ni-base alloy 718.

1) http://www.nims.go.jp/news/archive/2009/03/vk3rak0000004gl3.html

Aiming at the Construction of an Asian Atmospheric Corrosion Network

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Exposure to the atmosphere causes corrosion in bridges, automobiles, and railroads.

Corrosion occurs in various forms in various environments. The forms of corrosion include delayed fracture, crevice corrosion, stress corrosion cracking, corrosion fatigue, atmospheric corrosion, and others. The Corrosion Group of the NIMS Materials Reliability Center was launched simultaneously with the establishment of the NIMS Materials Information Technology Station (MiTS) in 2001, and is engaged in research on the topics of atmospheric corrosion and local corrosion of stainless steel (crevice corrosion, stress corrosion cracking).

Because many structures such as bridges, steel towers, automobiles, and railways are exposed in the air (in the atmospheric environment), atmospheric corrosion is a critical issue for maintaining those structures in a sound condition.

The Corrosion Group took over the exposure tests of low alloyed steels which were begun in 2000 at 3 locations in Japan (Tsukuba, Choshi, Miyakojima), and evaluates their atmospheric corrosion properties and compiles and publishes Data Sheets on the materials. In order to simulate the environmental conditions on the inside of bridge girders and under the eaves of structures, where the washing effect of rain that removes deposits of sea salt, etc. is not seen, the group is promoting standardization of a "sheltered exposure test." To complement its Data Sheets, the group also carries out evaluations of environmental corrosiveness using atmospheric corrosion sensors.

Atmospheric corrosion is a serious problem in Asia due to the hot, humid climate.

Because many parts of Asia have hot, humid climates, the problem of deterioration of metals due to corrosion is extremely serious in this region. Therefore, we are aiming at the construction of an atmospheric corrosion network in the Asian region in cooperation with universities and research institutes in the various Asian countries (**Fig. 1**). We are collaborating with researchers in 9 countries and regions of Asia, and also hold annual workshops, to which we invite researchers from these countries and regions to exchange information on atmospheric corrosion in their respective areas. Joint exposure tests are already being carried out in 4 countries. We visit counterpart countries to give advice on test methods, data analysis, and related issues, and also share information.

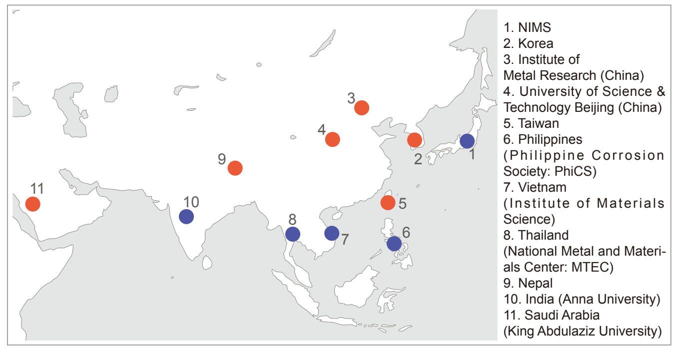

Fig.1 Collaboration with the Asian region (red: collaboration with country, region, or institution; blue, joint exposure test).

Stainless steel is a representative corrosion-resistant metal material. However, in aqueous solutions which contain chlorides such as NaCl, stainless steel is also subject to the forms of local corrosion called pitting and crevice corrosion, and these become the initiation site for stress corrosion cracking (SCC). In particular, because crevice corrosion occurs in environments with low chloride concentrations, it is a major problem in actual equipment. Therefore, the Corrosion Group is investigating the chemical, electrochemical, and mechanical properties of crevice corrosion and SCC, and is studying the environmental conditions under which these types of corrosion damage occur.

Corrosion research expanding to the world.

At present, we are shifting our focus to the study of "when, where, and how" these types of corrosion damage occur and grow, including elucidation of the mechanisms. Because this kind of systematic research is unprecedented anywhere in the world, we are receiving researchers and students from other countries, including Sweden and Vietnam, among others.

Based on our knowledge in connection with corrosion, including these research results, the Corrosion Group is carrying out analyses and investigations of cases of corrosion damage.

Fig.2 Providing guidance on an exposure test in the Philippines.

In recent years, much Japanese technology has been exported to other countries. As part of this, there have been requests for investigations of cases of corrosion and studies of the corrosion environment affecting super express trains in foreign countries, and we have performed site surveys and tests reproducing corrosion phenomena, etc. As the Asian countries construct social infrastructure, countermeasures for deterioration due to corrosion has become an urgent matter, and an increasing number of laboratories performing case analyses are being established in universities and research institutes. The NIMS Corrosion Group is strengthening its collaboration with these analysis laboratories, not only by receiving researchers, but also by visiting these countries to provide advice on experimental techniques and other needs.

Failure Analysis by Fractography

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Objects of investigation include the Monju reactor and SPring-8 synchrotron radiation facility.

The Fatigue Group of the NIMS Materials Reliability Center has been engaged in fatigue research on domestically-manufactured practical materials for more than 40 years, since it was part of the former National Research Institute for Metals (NRIM, a predecessor of NIMS). The group has also elucidated the causes of numerous accidents utilizing fatigue research to date, actively responding to failure analyses requested by public organizations. This article introduces some of these failure analyses.

Table 1 shows examples of the accidents which the Fatigue Group has investigated in recent years. The objects of investigation include “Failure of aircraft part,” “Damage to the SPring-8 synchrotron radiation facility,” and “Failure of deep-sea exploration vessel part,” among others. The results of these investigations revealed that many of these accidents were caused by fatigue. Although the list of accidents in **Table 1** is limited to the years since 2000, other important accidents which were investigating by the NIMS Fatigue Group, leading to an elucidation of the cause of the accident, have included the “JAL jumbo jet crash accident” in 1985, the “Na leak accident at the Monju fast breeder reactor” in 1995, and the “No. 8 H-II rocket accident” in 1999.

Year	Accident
2000	Failure of aircraft engine part (fatigue)
2000	Failure of aircraft main landing gear (fatigue)
2003	Failure of aircraft engine part (fatigue)
2003	Failure of aircraft main landing gear (fatigue)
2004	Damage of SPring-8 roof (fatigue)
2004	Failure of aircraft main landing gear (fatigue)
2005	Failure of aircraft main landing gear (ductile facture)
2005	Failure of aircraft main landing gear (ductile facture)
2007	Failure of aircraft launching winch (fatigue)
2007	Failure of deep-sea exploration vessel hydraulic system (fatigue)
2008	Failure of aircraft tail rotor part (fatigue)
2009	Failure of deep-sea exploration vessel screw part (fatigue)
2009	Failure of power plant system piping (fatigue)

Table 1 Main examples of accidents investigated by the Fatigue Group since 2000.

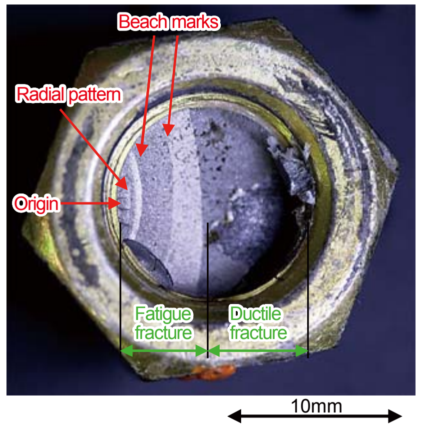

Fig.1 Macroscopic observation of part which suffered fatigue fracture.

Fractography produces convincing evidence.

Fractography (fracture surface analysis) is a powerful technique in this type of failure analysis. By observing the break (fracture surface) which occurred in the accident with an electron microscope, it is possible to educe a great deal of information related to the cause of the accident from the morphology of the fracture surface. For example, **Fig. 1** shows an example of macroscopic observation of the fracture surface of an engine part (bolt) which failed as a result of fatigue. The following information can be obtained from **Fig. 1**:

(1) The “radial pattern” is a pattern which advances from the point of origin in a radiating shape in the direction of crack propagation. Thus, both the point of origin of the fracture and the direction of crack propagation can be understood from this radial pattern.

(2) The “beach mark” is a pattern which oriented at right angles to the direction of crack propagation when a variable load acts on a part, and also forms with a concentric shape. Beach marks are crucial evidence indicating that the cause of failure was fatigue.

Investigation requires knowledge and experience.

On the other hand, when a part which has suffered fatigue fracture, like that in **Fig. 1**, is observed at high magnification under an electron microscope, a stripe pattern called “striation” is sometimes observed. Striations form in each cycle of repeated loading, and thus are typical traces of fatigue. To a certain extent, it is also possible to determine the load acting on the part and the number of cycles by investigating the spacing of the striations (see **Fig.2**).

However, in actuality, striations are rarely observed. In many cases, a featureless fracture surface called a “microstructure-dependent fracture surface” is observed. There are also cases in which inexperienced investigators mistake the striped pattern formed by beach marks or similar features for striations. This means that ample experience and knowledge are necessary in order to identify the actual morphology of the fracture surface.

Failure analysis contributes to the reliability of materials.

Conversely, fatigue fracture accidents are also “teachers” in fatigue design and fatigue research. For example, the lessons of the No. 8 H-II rocket accident led to a renewed recognition of the inadequacy of fatigue strength analysis of rocket materials, and this developed into joint research by NIMS and the Japan Aerospace Exploration Agency (JAXA). It is generally thought that fatigue is the cause of the majority of accidents involving the failure of mechanical structures. From this viewpoint, we will continue to perform failure analyses in the future, and at the same time, we will also work to advance fatigue research so that it will be possible to respond to a larger number of accidents.

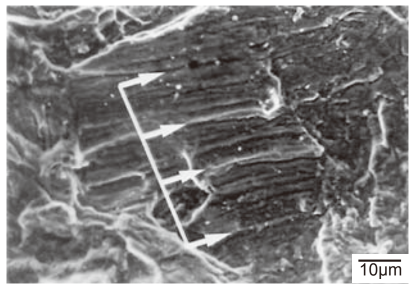

Fig.2 Striations obtained by microscopic observation of the fracture surface of a part which suffered fatigue fracture (No. 8 H-II rocket accident).

Atom Probe Tomography of Insulating Ceramics Using Ultraviolet Pulsed Laser

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The three dimensional atom probe (3DAP) is the only tomography technique that can resolve individual atoms. However, until recently, application areas of this technique had been limited to electric conductive materials. Recent successful implementations of pulse lasers to assist field evaporation have expanded the application areas of the atom probe technique to a wide variety of materials including semiconductors and thin film devices. However, even with this new technique, applications have been limited to electric conductive materials with a few exceptions on thin film oxides.

In this work, we have demonstrated that even insulating bulk ceramics can be quantitatively analyzed by the 3DAP technique using short wavelength pulsed laser. First, we fabricated a sharp needle like specimen called "tip" with a radius of approximately 50 nm from sintered ceramics using the focused ion beam (FIB) method. The microfabricated tip was supported on top of a tungsten needle and a high voltage ranging from 5-15 kV was applied to the tungsten wire to generate a ultrahigh electric field on the tip surface. Next, a femtosecond laser with the wavelength of 343 or 257 nm was irradiated on the tip apex (**Fig.1**). This induced field evaporation of the atoms on the tip surface. The field evaporated ions are accelerated in the normal direction to the equipotential surface and projected to the detector, on which atom-resolved position and time-of-flight measurements were performed with a projection magnification of a few millions.

Finally, two dimensional elemental maps obtained by this method was reconstructed to 3D atom tomography as shown in **Fig. 2**. The atom probe tomography reconstructs the positions of atoms in a zirconia (ZrO_2)-spinel ($AlMg_2O_4$) nanocomposite. The usage of the short wavelength laser for assisting the field evaporation has dramatically reduced the chance of specimen rupture, which had been a big drawback of the conventional atom probes. Furthermore, the UV laser assisted field evaporation led to a substantial improvement in the mass resolution of atom probe mass spectroscopy. Furthermore, the advances in the specimen preparation technique using the FIB nanofabrication in a high resolution scanning electron microscope (SEM) enabled the preparation of tips from any specific sites in materials and devices. With these merits, the UV laser assisted atom probe will be a versatile technique to obtain atom tomography from all kinds of inorganic materials and devices.

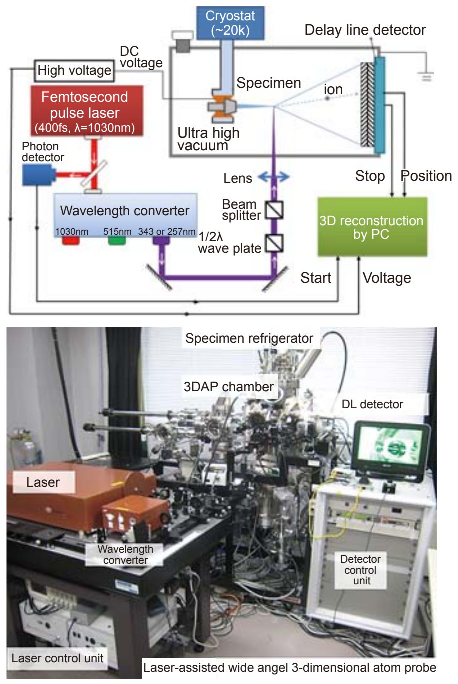


Fig.1 Schematic illustration of the UV laser assisted atom probe.

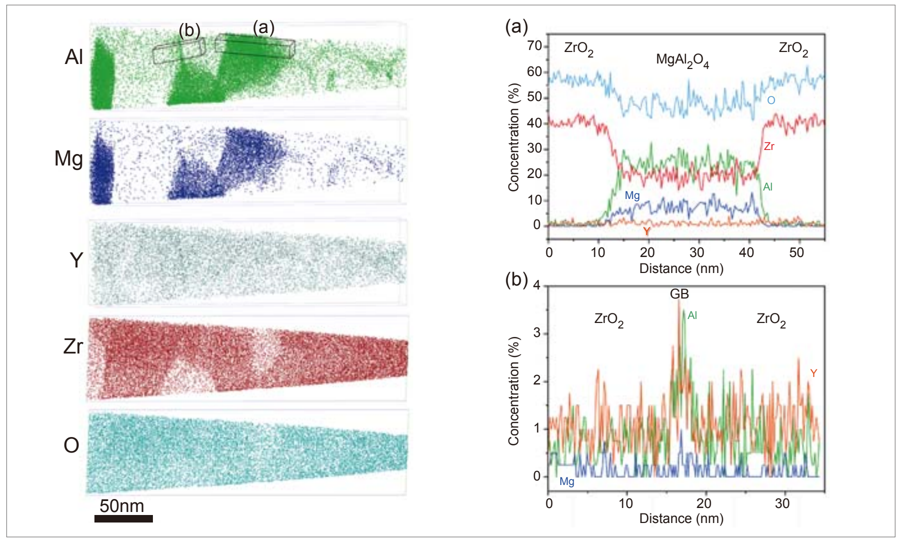
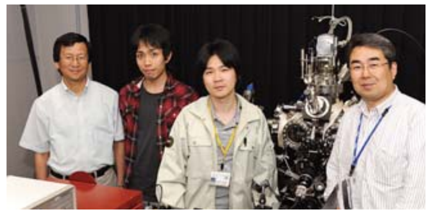


Fig.2 3D atom tomography taken from a zirconia-spinel nanocomposite material and the composition profiles calculated from the inset volumes.

Profile



Tadakatsu Ohkubo (right) Doctor of Engineering. Joined NEC in 1989, served as research associate at Nagaoka University of Technology in 1992, research associate at Osaka University in 1994, joined NIMS as Senior Researcher in 2002, and current position from 2006. Visiting Professor of Faculty of Industrial Science and Technology, Tokyo University of Science.
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Masaya Kodzuka (second left) Graduate student at Doctoral Program in Materials Science and Engineering, Graduate School of Pure and Applied Sciences, University of Tsukuba. Junior Researcher at NIMS.
Kazuhiro Hono (left) Ph.D. at Penn State in 1988, Post Doc at Carnegie Mellon Univ. (1988), Research Associate at Institute for Materials Research, Tohoku Univ. (1990), joined NRIM as Senior Researcher in 1995. Current position from 2004 and Professor of Materials Science and Engineering, Graduate School of Pure and Applied Sciences, Univ. Tsukuba.

Eco-friendly Semiconductor: Potential as a Light-Emitter

A Controlled Interface of "Monolayer-Semiconductor" Nanostructure Leads to the Unique Luminescence Property

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Nano Ceramics Center

Yoshio Sakka
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Fine Particle Processing Group

We live surrounded by various "lights" and "colors." In fact, we enjoy increasingly useful and more diverse benefits from optical and optoelectronic applications including optical communication tools, sensors, medical devices by the precise control of light-source in direction, linewidth, color, and intensity, and the appropriate use of laser light sources.

The phosphors are one of important items to produce a light-emitter, and include various types, such as organic dyes, rare earths, and compound semiconductor quantum dots. Bulk silicon (Si), a platform for large-scale integrated circuits, has a significantly poorer optical performance due to its indirect bandgap structure[1)]. Modification of crystalline Si such that its three-dimensional physical size does not exceed the bulk exciton Bohr radius[2)] for the crystal leads to the blueshifted photoluminescence (PL)[3)] property. Unlike bulk Si, it has been known that such nanostructures exhibit the size-dependent PL features in the visible region.[1)]

In this research, we discovered that the quantum confinement effect in Si allowed further control of the wide emission wavelength, that is, the UV–visible region, by precise size control and surface organic passivation.

Fig. 1 is optical absorbance and PL spectra of the unoxidized Si nanocrystals produced by a laser chemical synthesis process.[2)] The high molecular coverage of organic monolayers on the nanocrystal surface allowed the synthesis of the unoxidized Si colloidal dots. Due to the wide size-distribution, we see a broad PL spectrum from the colloidal nanocrystals.

The interesting point is the PL component which appears in the UV region. A few papers have reported similar PL properties, but the PL origin at UV wavelengths is not entirely clear. To understand the UV-PL origin, we next challenged to synthesize highly monodispersed Si nanocrystals with average physical size of less than 2.5 nm via the inverse micelle method. As shown in **Fig. 2**., we succeeded for the first time in observing size-dependent PL features in the UV-blue region.[3)] The relative quantum yield[4)] of the PL was high, at approximately 20%, which is comparable to those of some quantum dots of II-VI and III-V compound semiconductors (e.g., CdSe, etc.). And the PL lifetime was estimated at 0.9-4.5ns.

Importantly, these results suggest us the opportunity to develop Si-based light emitters working in the wide wavelengths in UV-visible regions by the precise control of colloidal Si nanocrystals.

Si is a nontoxic material to the environment, and shows a high chemical affinity for C, O, and N for covalent linkages, thereby producing a variety of organic derivatives hybridized at the molecular level. Thus, the increase of options in Si emitters offers a breakthrough for further development of optical and optoelectronic applications.

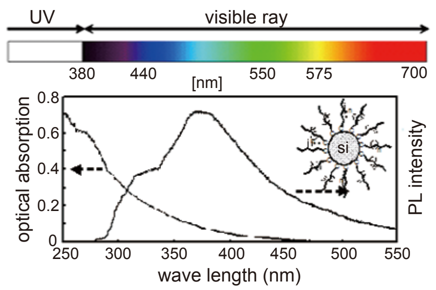

Fig.1 Optical absorption and emission spectra of colloidal Si nanocrystals terminated with organic monolayers produced by laser chemical synthesis.

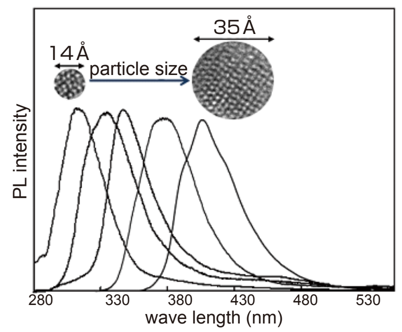

Fig.2 The size-dependent PL spectra in UV-blue region. The spectra were measured at room temperature.

Acknowledgements
These results were obtained through collaborative research with Dr. Tohru Tsuruoka and Dr. Tsuyoshi Hasegawa (Atomic Electronics Group, MANA), Dr. Seiichi Furumi (Wave Optics Group, Optronic Materials Center, NIMS), and Dr. M. Linford and Dr. M. Asplund (Brigham Young University). This work was supported in part by the JST PRESTO program.

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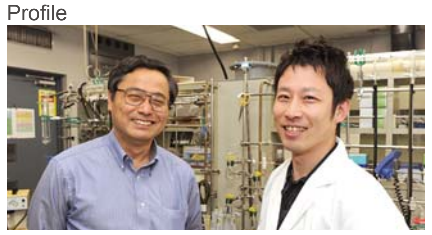
2) N. Shirahata, M. Linford, S. Furumi, L. Pei, Y. Sakka, R. Gates, M. Asplund, Chem. Commun. (2009) 4684.
3) N. Shirahata, T. Tsuruoka, T. Hasegawa, Y. Sakka, Small 6 (2010) 915.

1) Indirect bandgap structure : In the indirect bandgap structure, since an electron cannot shift from the lowest-energy state in the conduction band to the highest-energy state in the valence band without a change in momentum, phonon assistance is needed for the successful transition.
2) Bulk exciton Bohr radius : The quantum confinement effect is seen when the crystal size of the nanoparticle is of the same magnitude as the wavelength of the electron wave function. Specifically, the effect is described as a result of electrons and electron holes being squeezed into a dimension that approaches a critical quantum measurement, called a bulk exciton Bohr radius. In such a specific environment, the electrons and electron holes confined in the small nanocrystal could have certain discrete energies, determined by the potential barrier and boundary conditions.
3) Photoluminescence : An excitation to a higher energy state results in a return to a lower energy state accompanied by the emission of a photon.
4) Relative quantum yield : A quantum yield is defined as a ratio of the number of photons emitted as light to the number of photons absorbed. The relative quantum yield can be estimated by the comparison it with that of a standard phosphor.

Profile


Naoto Shirahata (right) PhD. JSPS-PD in April 2001, served as NIMS ICYS Researcher, Leading Researcher, and current position from April 2010. JST PRESTRO Researcher.
Yoshio Sakka (left) PhD. Joined NRIM (predecessor of NIMS) in 1983, and current position from 2006. Professor at University of Tsukuba. MANA Senior Researcher.

Development of Confocal Scanning Transmission Electron Microscopy

From 2-Dimensional Images to 3-Dimensional Images

Masaki Takeguchi
Advanced Electron Microscopy Group,
Advanced Nano Characterization Center

Ayako Hashimoto
Advanced Electron Microscopy Group,
Advanced Nano Characterization Center

Kazutaka Mitsuishi
Nano Growth Group,
Quantum Dot Research Center

Transmission electron microscopy (TEM) and scanning TEM (STEM) are imaging techniques, which use electrons that have passed through a specimen, and therefore basically produce projection images. Although two-dimensional analyses of shape, structure and composition of a specimen are possible with atomic level resolution, it is difficult to obtain three-dimensional (3D) information.

However, in the fields of research on inorganic materials, biomaterials, and device materials, as the objects have reached the nanometer size, 3D analysis and observation of their shapes with TEM/STEM level resolution is now desired.

One approach is an electron beam version of X-ray computer tomography (CT), which is already used in practical situations in medical and other fields. This is a method in which images are acquired from multiple orientations by rotating a specimen inside an electron microscope, and reconstructing 3D structure on a computer from those images. 3D analysis with resolution on the order of 1 nm in three directions (XYZ) is now possible with this technique.

Another approach is a method in which slice images of an arbitrary depth are acquired by reducing the size of a convergent electron beam in z direction in order to reduce the focal depth. 3D image can be obtained by scanning a specimen in the XYZ directions.

This method has attracted attention recently because it is possible to obtain z-sliced images directly, without 3D reconstruction processing, while maintaining the atomic level resolution of TEM/STEM in lateral direction. We developed a confocal STEM, which is a further extension of this technique, and succeeded in 3D observation of the internal structures of nanostructures.

Fig. 1 shows the electron optical system of the confocal STEM. The illumination lens is used to converge the incident electron beam on the specimen, and the imaging lens transmits only the electron beam from the designated depth through the pinhole above the detector, enabling confocal imaging.

A high-resolution 3D specimen scanning system was developed for observation of 3D images maintaining this confocal optical system. This makes it possible to scan specimens with an accuracy of less than 0.1 nm in the XYZ directions. In order to obtain images at an arbitrary depth more clearly, an annular dark field (ADF) confocal optical system was conceived. By applying these two new technologies, it is now possible to obtain depth resolution of 100 nm with a conventional 200keV electron microscope, and depth resolution on the order of 20 nm with an aberration corrected 200keV electron microscope.

Fig. 2 shows an example of a carbon nanocoil with a spiral structure. This demonstrated that the 3D shape of the nanocoil can be obtained by acquiring 27 sequence slice images and performing 3D reconstruction on those images. **Fig. 3** shows an example of a cross-sectional image of the internal structure of hollow porous silica particles encapsulating iron oxide. This observation revealed that a silica core containing iron oxide exists in the interior, and this core (indicated by the arrow) is also hollow.

As these examples suggest, high expectations are placed on the application of this confocal STEM to observation of various nanomaterials and environmental materials, as a 3D observation technique which makes it possible to designate the position of particles, precipitates, etc. in 3D and observe cross-sectional images of internal structures. We are also engaged in research with the aim of further improving the depth resolution of this technique.

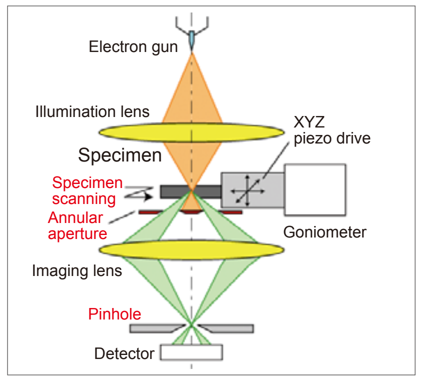


Fig.1 Optical system of the confocal STEM.

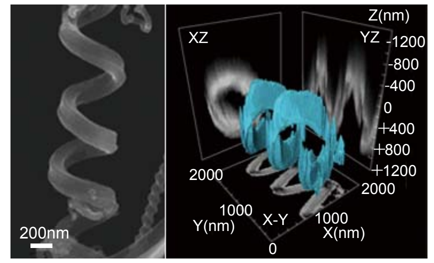


Fig.2 SEM image of a carbon nanocoil (left), and result of 3-dimensional observation by confocal STEM (right). Specimen courtesy of Yoshikazu Nakayama and Kaori Hirahara, Osaka University.

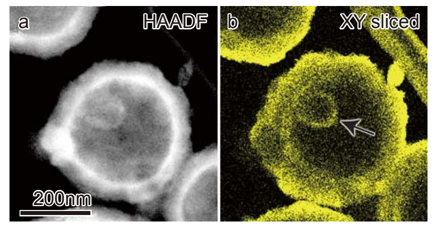
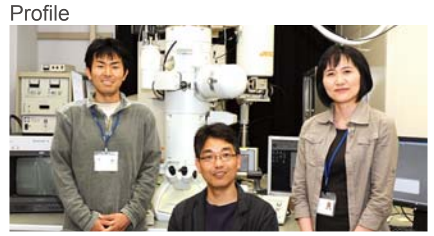


Fig.3 ADF-STEM image of hollow porous silica particles (left), and XY cross-sectional image at a selected depth by confocal STEM. Specimen courtesy of Yufang Zhu, ICYS, NIMS.

Profile


Masaki Takeguchi (center) Doctor of Engineering. Completed Osaka University Graduate School. Joined JEOL in 1993, NRIM in 1998, and current position from 2006. Guest professor at Saitama Institute of Technology.
Ayako Hashimoto (right) Doctor of Engineering. Completed Keio University Graduate School. PD and JSPS-PD at National Institute of Advanced Industrial Science and Technology from 2002, joined Kanagawa Academy of Science and Technology in 2005, PD and ICYS-researcher at NIMS from 2007, and current position from 2010.
Kazutaka Mitsuishi (left) Doctor of Science. Completed Tokyo University of Science Graduate School. Joined NRIM in 1997, current position from 2006. Guest professor at Saitama Institute of Technology.

The Increasing Importance of Materials Science

Dr. Junichi Sone Vice President, NIMS

Dr. Junichi Sone is the NIMS Vice President from a private-sector research center. While remaining mindful of the differences in the sense of values of private companies and Independent Administrative Institutions (IAI) like NIMS, Dr. Sone gives NIMS high marks for its outstanding quality as a materials research center and proposes a vision of the future for NIMS. In this interview, Dr. Sone discusses his aspirations and expectations for NIMS.

—What were your impressions on coming to NIMS from a company in the private sector?

I was at NEC for 35 years, so I am still learning about NIMS. However, the first difference I might mention between NIMS and the private sector is the extremely good research environment at NIMS. Company researchers can only be jealous. At one time, there was also a boom in basic research in the private sector, but today, perhaps because of the division of roles, private sector labs have considerably downsized. The research infrastructure at the IAIs is overwhelmingly superior.

The sense of values is also different. In companies, profitability is the first priority, and economic principles are the core value. In contrast, the IAIs are always conscious of Japan, that is, they are thinking about what we should be doing for a country. Before coming to NIMS, I spent a year at the Center for Research and Development Strategy (CRDS) of the Japan Science and Technology Agency (JST) under a concurrent appointment, and I became strongly aware of this orientation through the discussions while I was there.

—The difference in values is large, isn't it?

Today, we can no longer expect the kind of constantly-rising economic growth that we enjoyed in the past. I strongly feel that it is very important that we think about what Japan, as a nation, should do for the future. For example, where information technology is concerned, how Japan should compete with global internet companies like Google and Amazon is an extremely difficult problem. Businesses of materials and devices based on materials science are in a different environment. In this field, Japan has a strong base, and the values which materials provide are universal and common to all nations. For this reason, this is an important field industrially for Japan, and also a very challenging subject. In that sense, I think that I'm very fortunate that, as it happens, I can be involved in this kind of work at NIMS.

—You were originally a researcher, I believe?

That's right. My background is in physics. While I was affiliated with the Fundamental Research Laboratories at NEC, my work concerned nanotechnology and the

environment and energy. We also did joint research with NIMS, so I had a chance to see NIMS from the outside. However, at the time, the closest relationship with an IAI was with AIST (National Institute of Advanced Industrial Science and Technology).

—What differences do you feel between looking at NIMS from the outside and now, being part of NIMS?

I now realize that NIMS has far greater potential than I felt at the time. If this potential can be developed, I think that there is an ample possibility that NIMS can become a research center that leads materials research in the world. At present, the environment and energy are major global problems, but I think that a large part of the solution will depend on materials. There are points where we must unavoidably place our hopes on innovation in materials. The United States has also invested heavily in the environment and energy technologies since Mr. Obama became President. All of this suggests that materials science will become increasingly important.

—So, what kind of research institute do you hope that NIMS will become?

Of course, I hope that NIMS will demonstrate its presence as a representative Japanese materials research institute. In particular, I hope that NIMS will become a preeminent institute in the field of the environment and energy, as these are extremely urgent issues.

It is also important that the technologies which are created here contribute to industry, and thereby strengthen Japan. However, it is essential that this relationship be broad-ranging, and not be limited simply to individual projects. I would like to pursue such a collaboration scheme with industry.

—What are your interests outside of work?

At present, I'm in a full study mode, and I don't have time for other things. My hobby is iGo. When I was young, I was completely absorbed in the game, and I was a member of NEC's representative team. Three of us formed a team that won the Kawasaki Mayor's Cup. In sports, I really like to move my body. I also play golf, but I'm not very good at it. When I retire, I'd like to spend my time playing golf and iGo, i.e. G and G.

—Do you have any orders for younger people?

Adopt an attitude of always challenging the new, and continue your research in a way that you won't have any lingering regrets. It's also important to constantly make new relationships with people. Even if you assemble a group of 10 outstanding researchers, if you simply continue in that form, you will eventually lose your creativity and become a group without vitality.

In addition, NIMS is also making progress in this respect, but it's important to do research with a global perspective. Work that's only recognized in Japan is absolutely meaningless. I say this once again because the value which materials provide is common to every country.

—What do you recommend as a method of inspiring vitality?

Perhaps continuing to have the broad-mindedness to accept various kinds of people. Also, establishing a system by which being in NIMS becomes a career for researchers. In that respect, I think the ICYS has been an outstanding experiment.

Profile
Doctor of Science. Completed Master course in Department of Physics, Graduate School of Science, the University of Tokyo. Appointed as Vice President of NIMS, after served as General Manager at Fundamental Research Laboratories and Vice President of Central Research Laboratories at NEC.

Making NIMS a Global Hub of Materials Research

Dr. Eiji Muromachi Vice President, NIMS

Dr. Muromachi, who was in the vanguard of research at NIMS as a NIMS Fellow, was recently appointed Vice President. His first job in that position was to respond to the new government's budget screening process. ["Project screening": Public hearings to identify and eliminate wasteful government-funded projects.] Here, Dr. Muromachi discusses his efforts in this dealing with this difficult reality, as well as his dreams for the future.

—What changed when you suddenly moved from the research field to become Vice President?

My very first job was the government's budget screening process. Because I put all of my energy into that, I had the real feeling that it was completely different from research. The pressure was so intense that sometimes I couldn't even eat.

This is an important turning point for NIMS, so I intend to do my best. However, the real problem is what we do going forward. The experience in the budget screening process made me feel anew just how difficult it is to explain our work to the Japanese people in a way that's easy to understand.

—It's a question of explanatory ability, isn't it?

That ability is really put to the test in a situation like that. In that sense, doing good public relations work every day builds up that ability. The role of public relations is really important.

—Do you think that individual researchers also need to have that kind of explanatory ability?

It's a little hard to tell every researcher that he or she has to master that ability. Rather, I think it's better to provide an effective system for translating the researcher's message into an easily-understood form. Because we can't demand that every individual have explanatory ability, it's important to respond as an organization.

—As the person with overall responsibility for general affairs, what is your thinking on what must be done going forward?

The first issue, after all, is reform in the administrative division. The staff of the administrative division is decreasing year by year, and we're also making efforts to reduce personnel. In fact, we've reduced general administrative costs by 15%, but we're still being pressed from outside to make further cuts. The only way to meet those demands is to use computers for data processing and implement energy savings. One possible proposal for promoting energy savings may be to assign each staff member a number for centralization of information management. We'll also rely on computers as much as possible.

—Incidentally, could you give us your thoughts on the ideal form of science and technology as a whole in Japan?

One thing that I feel strongly is that the key words for the next era will be the environment and energy. Both are important, and they exist in an inseparable relationship.

To cite just one example, I think we can expect that the price of crude oil will increase in the future. When it's two to three times higher than it is now, it might become

necessary for Japan to change its social structure. What we can do when that time comes is a key issue. Of course, greater use of solar, wind, and similar energy sources will be indispensible. The importance of fuel cells and secondary cells also goes without saying. However, in addition, we must not forget nuclear power. NIMS has established a Cluster for Nuclear Power Plant Materials and is currently studying how we can contribute to nuclear power. I think that our role need not be limited to structural materials. There is also a great deal that NIMS can do in the area of materials for processing radioactive wastes. I hope that NIMS will further heighten its presence in this field.

—What hobbies do you have outside of your work?

Usually I sleep when I have a day off. Recently, I've been taking walks. I used to walk for about 2 hours when I was still doing research, but now it's about 30 minutes after lunch.

After that, I play iGo. I look for a partner at an internet site, and we play.

I come from a family of farmers, so we have a rice field. When we have long weekends, I spend one day working in the field. I'm a farmer about 3 days a year, but that way, I don't feel guilty when I eat the rice that's harvested (Laughs). Sometimes my wife picks vegetables, and I'm relieved when I come home.

—Do you think you'll return to research?

I want to do research again. Whether or not it's a good thing to be completely out of research, I feel that it's important to think about administrative matters from the viewpoint of a researcher. What I'd like to do in the future is research on new superconductors. I've been dreaming about room temperature superconductors for more than 20 years.

—Finally, do you have any words for young researchers?

Devote yourself single-mindedly to research while you're young, because you won't be able to when you grow older. I would like to give young people time for research. I want to create that kind of environment. Today's young people are truly outstanding, so much so that I probably couldn't become a researcher if I were starting out today.

My goal is to create the world's top materials research institute, centering on that kind of young people, and to fulfill the functions expected in a core research institute. This is one dream that I really want to make come true.

Profile
Doctor of Science. Completed Master course in Department of Applied Chemistry, Graduate School of Engineering, Tohoku University. Appointed as Vice President of NIMS after served as Senior Researcher at National Institute for Research in Inorganic Materials (NIRIM), Director of Superconducting Materials Center at NIMS, and Fellow at NIMS.

The 1st Singapore-Japan Workshop on Progress in Nanomaterials

(April 19-20, 2010) NIMS recently held the "1st Singapore-Japan Workshop on Progress in Nanomaterials – Applications to Electronics, Energy, and Health" on April 19-20. The event was held jointly with Institute of Materials Research and Engineering (IMRE) in Singapore and the Nanoscience & Nanotechnology Initiative NanoCore (NUS-NNI-NanoCore) of the National University of Singapore. The workshop was rich in content, with respective sessions on spintronics, carbon electronics, oxide electronics, micro-characterization techniques, sustainable energy materials, and the social impact of nanomaterials.

The purpose of this workshop was to promote research exchanges and collaboration among the three organizations, NIMS, IMRE, and NUS-NNI-NanoCore. In the future, workshops will be held alternately in Japan and Singapore as an annual event.

On April 20, the NIMS participants visited IMRE and NUS-NNI-NanoCore for substantive discussions on future research cooperation and collaboration and for tours of the advanced facilities at those organizations.

Announcement of NIMS Conference 2010 in July

NIMS will hold the NIMS Conference 2010 at the Tsukuba International Congress Center (Epochal Tsukuba) from July 12 to July 14. The main theme of this year's conference is "Challenges of Nanomaterials Science – Towards the Solution of Environment and Energy Problems." The NIMS Innovative Center of Nanomaterials Science for Environment and Energy (ICNSEE) will play a central role in the conference.

During the conference, organized symposium with lectures by distinguished researchers from Japan and other countries will be also held. Symposium will cover 8 sub-themes: "Theoretical and Computational Studies for the Materials Development", "Rechargeable Batteries", "Photovoltaics", "Design of High Quality Fuel Cell Materials and Devices", "Nano Photocatalysis: Possibilities and Challenges", "Nano Interface Characterization for E&E Materials", "Practical Social Needs for Thermoelectric Power Devices", and "Environmental Catalysts and Materials".

Another featured part of the conference will be the presentation of this year's NIMS Award. The NIMS Award is a prestigious prize which is given to individuals or groups which have achieved remarkable breakthroughs in materials science and technology and related fields in the past 10 years.

For advance registration (no charge) and more detailed information on the NIMS Conference 2010, please visit the following website:
http://www.nims.go.jp/nimsconf/2010/

Hello from NIMS

After working at The University of Tokyo from 2003 and Chiba University, I joined NIMS in April of 2007 because of the special atmosphere of NIMS for foreign researchers, in particular, ICYS – International Center for Young Scientists, which allows me to do what I have interest in. I live in Tokyo since 2003 and become inert to move to other places, despite the high cost of living there. It is very convenient to go anywhere from Tokyo. Travel to the most cities of China, such as Beijing, Shanghai, Guangzhou, and Hong Kong, is within 5 hour by plane. When my son was about 2 years old, we visited Izu and Hakone, but experience in Hakone remains his and my best impression even though we visited other interesting places such as Disneyland. Now, my son always wants to go to Ueno Park and Kasai Seaside Park. We routinely sense the special and various taste of Japanese Gardens. My family really gets used to the life of Japan, experiencing distinct seasonal festivals like Hanami (flower-viewing) in spring, Hanabi (firework) in summer, Koyo (red-leaf) in autumn, and Oshogashu (New Year's holiday) in winter. We enjoy the green, safe, and sometimes flat life in Japan.

[Kasai Seaside Park in May of 2010.]

Mingsheng Xu
(China)
ICYS-Sengen researcher
(April 2007- present)

[Minakami Kogen after ICYS 2010 Workshop (Jan. 2010)]

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