from Tsukuba, Japan to the world

NIMS NOW International

National Institute for Materials Science

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Special Features

Introduction of 20 New Projects and Recent Achievements
- Advanced Nano Characterization Center -

Aiming at Advanced Nanocharacterization Technology for Materials Research

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Managing Director
Advanced Nano Characterization Center

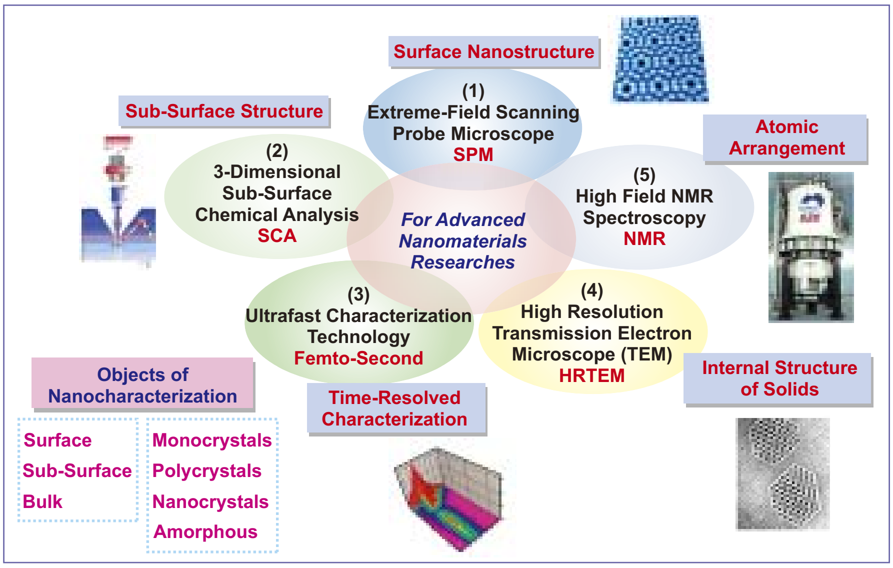

Fig. Development of the state-of-the art technologies for in-depth characterization of nanomaterials.

The Advanced Nano Characterization Center (ANCC) was launched as a research center in NIMS' Key Nanotechnologies field. The following five research groups have been established in the ANCC to conduct research and development on advanced characterization infrastructure technologies for multi-dimensional evaluation of the structures, physical properties, and functions of a diverse range of substances and materials in which structures are controlled at the nano scale.

(1) Advanced Scanning Probe Microscopy Group
(2) Advanced Surface Chemical Analysis Group
(3) Ultrafast Spectroscopy Group
(4) Advanced Electron Microscopy Group
(5) High Field NMR Group (Nuclear Magnetic Resonance Spectroscopy)

The ANCC is responsible for the project "Development of Advanced Nanocharacterization Infrastructure Technologies for Nanomaterials Research" during the period of NIMS' 2nd Mid-Term Program (FY2006-2010). By realizing an even higher level of core competence technologies for material characterization and evaluation at NIMS, we aim to establish high-level characterization and evaluation technologies for advanced substances and materials, ranging from surface atomic structures to the nanostructures of the sub-surface layer/internal structure of solids.

< Continued on p.2

in this issue

NIMS News

ICYS

ICYS-ICMR Summer School 2006 on Nanomaterials

Group Photo.

< Continued on p.3

Aiming at Nanoprobe Technology for Creation of Advanced Materials and Search for Functions

Daisuke Fujita, Akiko Itakura
Keiko Onishi, Keisuke Sagisaka
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Advanced Nano Characterization Center

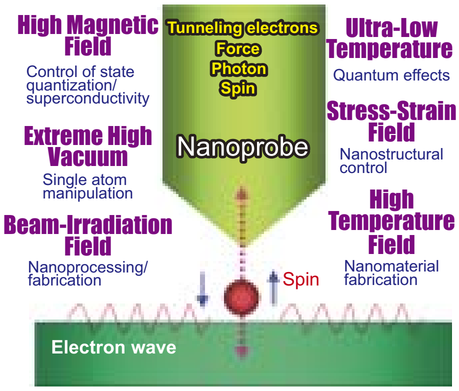

Fig. 1 Advanced nanoprobe technologies integrated with diverse physical field manipulations for creation of nanomaterials and search for new functions.

One key task for NIMS is research and development of advanced substances and materials. In order to create novel materials with nanoscale structures and search for new functions originating in physical properties in the nano region, the establishment of nanocharacterization technologies different from the conventional concepts is demanded. From this viewpoint, we are promoting the development of nanoprobe characterization technologies integrated with manipulation of diverse physical fields. Concretely, we are developing and applying "active nanocharacterization technologies" such as nanoanalysis integrated with nanofabrication technologies, nanostructural control by external field response, the search for new functions in extreme environments where quantum effects appear, etc.

As shown in **Fig. 1**, our aim is to develop technologies which realize multi-dimensional nanocharacterization with atomic resolution while realizing ultra-low temperature fields, high magnetic fields, ultra-high vacuum fields, stress fields, and similar conditions as manipulations of extreme fields in a nanocharacterization space. For example, in an ultra-low temperature field, manifestation of quantum effects such as electron wave interference and the single electron effect becomes remarkable, but when this field is combined with an ultra-high vacuum field, high accuracy characterization of energy states and manipulation of single atoms become possible. With further addition of a high magnetic field, quantization of electronic states and control of superconducting states also become possible. At present, we are involved in the development of high resolution nanoprobes combining 3 extreme-field manipulations. Using this kind of advanced nanoprobe technique, we recently realized confinement of 1-dimensional electron waves at the atomic scale by transferring atoms to arbitrary positions on a silicon surface, and succeeded in imaging the quantized state density at each energy level (**Fig. 2**). It can be understood that the number of maximum peaks increases one at a time, corresponding to the quantization state.

On the other hand, stress fields, variable temperature fields, and the like are closely related to the fabrication of nanomaterials, and integration with nanocharacterization is useful in elucidating this mechanism. For example, by applying a stress-strain field, it is possible to control the surface nanostructures and other phenomena (**Fig. 3**). In a variable temperature field, nanofabrication utilizing surface precipitation becomes possible and, for example, elucidation of the mechanism by which low-dimensional nanocarbon is created can be expected.

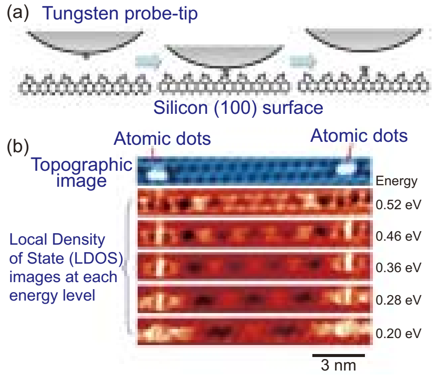

Fig. 2 (a) Schematic diagram of point contact transfer of probe-tip atom (tungsten) to silicon surface, and (b) imaging of the 1-dimensional state of electron waves confined between atomic dots transferred onto a single dimer row on the Si (100) surface.

Based on these achievements, we hope to accelerate the development of characterization technologies which contribute to the creation of advanced nanomaterials by developing an original nanoprobe system, and promote the standardization and popularization of these technologies.

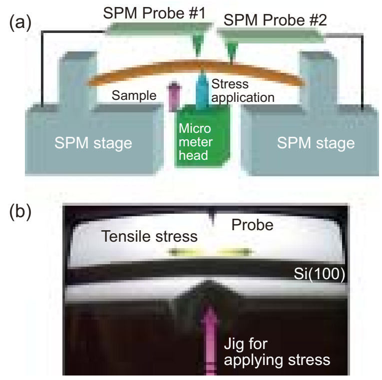

Fig. 3 (a) Schematic diagram of stress-field nanoprobe with dual probe-tips, and (b) photo while tensile stress is applied to Si(100) specimen.

For more details: http://www.nims.go.jp/nanophys6/eindex.htm

< Continued from p.1

Aiming at Advanced Nanocharacterization Technology for Materials Research

In particular, in order to clarify the nanoscale structure and novel functions possessed by new substances/new materials, these respective groups are promoting the development of nanocharacterization technologies including (1) atomic resolution scanning probe microscopy characterization technologies in extreme-field environments, (2) 3-dimensional nanostructure analysis technologies for wide-area surface structures, (3) ultrafast femtosecond-level time-resolved spectroscopy characterizationtechnologies, (4) ultra-high resolution transmission electron microscopy (TEM) characterization technologies, and (5) high resolution solid-state nuclear magnetic resonance (NMR) spectroscopy characterization technologies using NIMS magnets, which boast the world's highest magnetic field.

In addition, the ANCC aims to provide characterization technologies and data which contribute to materials research based on these advanced technologies. This Special Feature introduces the missions and research activities of each of the ANCC's five groups.

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

Development of a High Speed Electron Simulator for Wide-Area-3-Dimensional Analysis

Shigeo Tanuma, Sei Fukushima
Takashi Kimura
Advanced Surface Chemical Analysis Group
Advanced Nano Characterization Center

On the development of electronic materials and composite materials, 3-dimensional analysis technique with high speed and wide area, which enables measurement/analysis of large surface areas in both the planar and depth directions with high resolution of several 10s of nanometers or less, is strongly required especially for the failure analysis in the practical application of materials. For this purpose, the Advanced Surface Chemical Analysis Group is developing a variety of advanced analytical methods, including an Auger electron spectroscopy (AES) device, X-ray photoelectron spectroscopy (XPS), and electron probe micro analyzer (EPMA) etc. However, because there are limits to achieving objectives with individual instrument, the development of novel measurement and analysis methods which combine these techniques is indispensable. For this reason, the modeling of the electron transport process in the inelastic and elastic scattering which occur when electrons move in solids and an accurate knowledge of the physical quantities related to the scattering are extremely important. Our Group is, therefore, attempting to achieve higher precision in the fundamental physical quantities of the inelastic mean free paths (IMFPs), stopping power (SPs), etc. of electrons and develop a 3-dimensional wide-area electron simulator to enable accurate measurement/analysis of electrons having energies on the order of 100 eV to 10 keV, which is the most useful range in the practical surface analysis.

IMFP, which describes the inelastic scattering of electrons in solids, is the most basic quantity expressing surface sensitivity and the matrix effect in surface electron spectroscopy techniques such as AES and XPS. In practical analysis under ISO standards, a general formula called TPP-2M (Tanuma-Powell-Penn formula) based on IMFP values calculated from the individual dielectric functions of substances is widely used, but experimental verification of the accuracy of this formula is inadequate. Therefore, we investigated the energy and material dependency of experimentally determined IMFPs with 13 elemental solids using elastic peak electron spectroscopy (EPES), in which specimens are bombarded with electrons and the elastic peaks of electrons that are reflected without energy loss are measured. Partial results of this research are shown in **Fig. 1**. The ratio of electron energy and IMFP shows a good linear relationship with electron energy expressed on a log scale. Using this plot, which is called Fano plot, it is possible to determine the energy dependency of IMFP. **Figure 2** shows a comparison of the experimental values, theoretical calculated values, and results obtained with the TPP-2M formula for the IMFP of silver. These results show that the difference between the value calculated with the TPP-2M formula and the experimental values is on the order of 10 %, verifying the accuracy of the calculation method. Thus, we are proceeding with the development of a 3-dimensional wide-area surface layer electron simulator based on the development of a precise database for the fundamental physical quantities describing the inelastic scattering of electrons. With this, it is expected to be possible to perform composite analyses of data from AES, XPS, EPMA, EPES, etc., enabling high speed, high accuracy 3-dimensional analysis of the surface layer of specimens. The research on EPES is being carried out jointly with Prof. Keisuke Goto of Nagoya University of Technology.

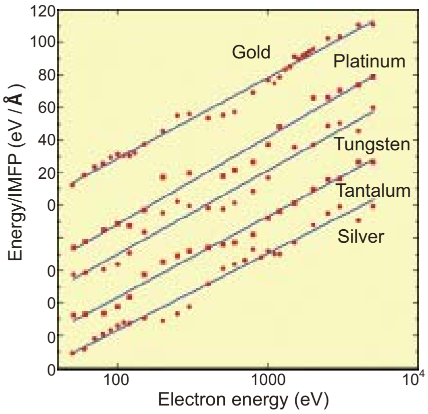

Fig. 1 Energy dependency of electron inelastic mean free paths (IMFPs).

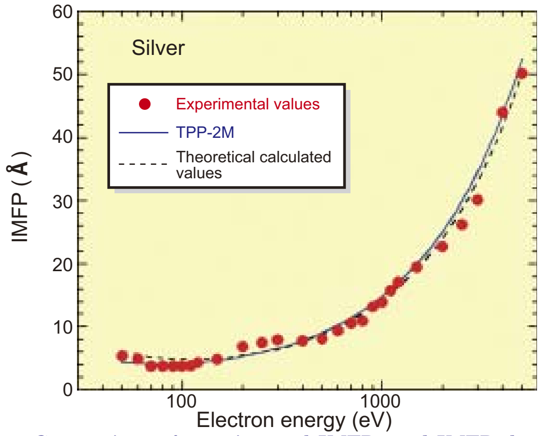

Fig. 2 Comparison of experimental IMFPs and IMFPs by general formula TPP-2M for silver.

NIMS News

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ICYS-ICMR Summer School 2006 on Nanomaterials

Poster Session.

(July 22-28, NIMS) -- The first ICYS-ICMR Summer School on Nanomaterials was successfully held with 17 internationally prominent scientists (invited lecturers), 59 Ph.D. students from 12 countries (Japan: 25, USA: 19, other foreign countries: 15) and 39 observers from NIMS. The objective of this school was to provide the opportunity to get the interdisciplinary concepts in the nanomaterial research fields for participants. This school was quite instrumental for participants in broadening the prospective of their own researches through the lectures as well as in stimulating each other through oral presentations and poster sessions. Co-organizers also offered some Japanese cultural programs making participants enjoyable and plentiful with not only scientific but also cultural experiences. The establishment of international network among participants was also another important mission of this school, and the school was closed with fruitful achievements.

ICYS has signed a Memorandum of Understanding (MOU) with the International Center for Materials Research (ICMR) at University of California at Santa Barbara in 2005.

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

Time-Resolved Observation of Extremely High-Frequency Phonons in Semiconductors

Masahiro Kitajima, Kunie Ishioka
Ultrafast Spectroscopy Group
Advanced Nano Characterization Center

Photo-response of solid materials contributes greatly to our daily life, in wide range of phenomena from photosynthesis in plants to the retina in our eyes catching light to solar cells generating electric power. In every photo-response process, light is quickly absorbed by electrons in the material, whose energy is then transferred into the atomic motion and breaking chemical bonds between the atoms. The photo-absorption occurs in femtosecond (fs: 10^{-15} second) timescale; the energy transfer in picosecond to nanosecond, still too fast for human eyes to catch up. This is why we are developing a novel technique to monitor the ultrafast photo-response of solid materials, which is especially important for evaluating metallic and semiconductor materials for opt-electronic devices.

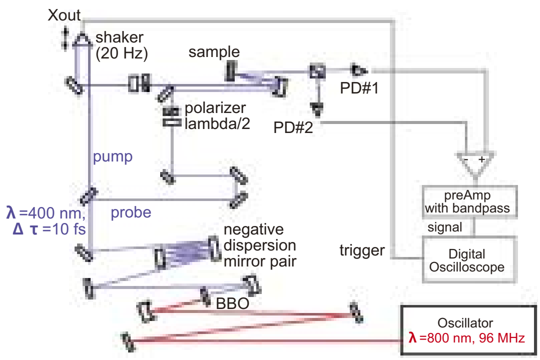

Fig. 1 Schematic drawing of experimental setup for pump-probe reflectivity measurements using sub-10 fs pulsed laser as a light source.

We investigate ultrafast electronic and phononic dynamics in semiconductors and metals using a simple pump-probe setup with a fs pulsed laser (**Fig. 1**). Photo-response in semiconductors and metals is dominated by the cooling processes of photo-generated electrons, in which the hot electrons give their excess energy away to other electrons and the collective motion of the atoms in the solid (phonons). We have observed, for example, impulsively excited C-C stretching of graphite (**Fig. 2**) in time domain, whose frequency is as high as 47 THz. The transient modulation in the vibrational frequency gives an evidence of strong coupling with non-thermal electrons and the in-

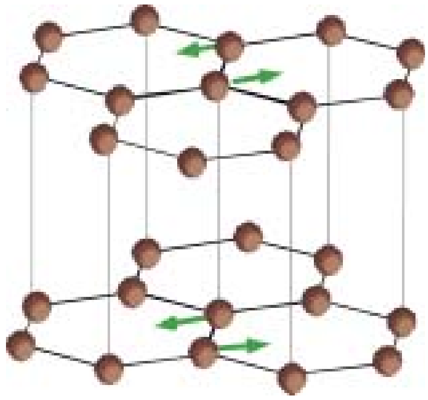

Fig. 2 Schematic drawing of the in-plane C-C stretching phonon of graphite (E_{2g2} mode), whose frequency is 47 THz.

plane shear phonon. Our experiment requires ultrashort laser pulses for time-resolution, as well as techniques to detect a very small change in the optical properties of the material. We are planning to apply our experimental method to characterize the ultrafast opt-electronic properties of doped diamonds, carbon nanotubes and graphene (a single layer of graphite). This research is a collaboration with Prof. Hrvoje Petek of the University of Pittsburgh.

For more details: http://www.nims.go.jp/ldynamics/Femto.html

Development of High Field Solid-State NMR and Application to Nanomaterials Research

Tadashi Shimizu, Masataka Tansho
Atsushi Goto, Kenjiro Hashi
High Field NMR Group
Advanced Nano Characterization Center
(Concurrently with High Magnetic Field Station - User Facility)

The objectives of this project are the development of the world's highest performance NMR system by combining the one of the world's outstanding groups of magnets, which has already been developed by NIMS and includes a 920/930 MHz magnet, 40 T class hybrid magnet, and others, and NIMS' spectrometer development technology, and to apply this system in order to overcome key problems in nanomaterials research.

The advantages of NMR (nuclear magnetic resonance) include the fact the parts of materials which manifest functions can be measured selectively at the nano level even, for example, in amorphous substances and mixtures, and the 3-dimensional chemical structure and its dynamic properties can be elucidated with pinpoint accuracy. A representative example of a conventional application of NMR is the identification of molecular structures in pharmaceutical product development. When new pharmaceuticals are registered, the law requires that the NMR data be attached.

With the essential NMR technology, it is possible to observe approximately 90 % of the elements in the Periodic Table of the Elements (elements other than those shown in white in the **table**). However, use of the conventional technology had been limited to only hydrogen and carbon, which were particularly advantageous from the viewpoints of sensitivity and resolution. To realize high sensitivity and high resolution with other elements, a high magnetic field of 20 T or more is essential. In particular, because the elements shown in red in the Periodic Table have quadrupolar spins (i.e., their nuclear spin quantum number I is larger than 1), a high field is the only method of improving resolution. As shown in the **table**, the elements having quadrupolar spin include numerous elements which are important for

$I = 1/2$ $I > 1$ $I = 1$ Unstable or $I = 0$

1 H																	3 He
7 Li	9 Be											11 B	13 C	14 N	17 O	19 F	21 Ne
23 Na	25 Mg											27 Al	29 Si	31 P	33 S	35 Cl	Ar
39 K	43 Ca	45 Sc	47 Ti	51 V	53 Cr	55 Mn	57 Fe	59 Co	61 Ni	63 Cu	67 Zn	69 Ga	73 Ge	75 As	77 Se	79 Br	83 Kr
85 Rb	87 Sr	89 Y	91 Zr	93 Nb	95 Mo	Tc	101 Ru	103 Rh	105 Pd	107 Ag	111 Cd	113 In	119 Sn	121 Sb	125 Te	127 I	129 Xe
133 Cs	137 Ba	La	177 Hf	181 Ta	183 W	187 Re	187 Os	193 Ir	195 Pt	197 Au	199 Hg	203 Tl	207 Pb	209 Bi	Po	At	Rn
Fr	Ra	Ac	Ku	Ha													

139 La	Ce	141 Pr	143 Nd	Pm	147 Sm	153 Eu	157 Gd	159 Tb	163 Dy	165 Ho	167 Er	169 Tm	171 Yb	175 Lu
Ac	Th	231 Pa	235 U	237 Np	239 Pu	241 Am	247 Cm	247 Bk	Cf	Es	Fm	Md	No	Lr

Table. Periodic Table of the Elements.

Development of High Performance Electron Microscopes and Application to Advanced Materials Research

Yoshio Matsui, Masaki Takeguchi
Koji Kimoto, Akira Hasegawa
Nobuhiro Ishikawa, Toru Hara
Kazuo Furuya
Advanced Electron Microscopy Group
Advanced Nano Characterization Center

Fig. 1 Appearance of ultra-high vacuum, aberration-corrected STEM.

1. Introduction

The Advanced Electron Microscopy Group, Advanced Nano Characterization Center is a group of front-line specialists in electron microscopy. The Group has developed various advanced electron microscopes, beginning with two ultra-high voltage electron microscopes, and has made numerous important achievements, including dynamic in-situ observation of advanced nanomaterials, ultra-high resolution structural analysis, etc. This article introduces the "High Resolution STEM" and a "Nano Magnetic Structural Analysis System" as two examples of recent development work.

2. Development and Application of High Resolution STEM

The Group has devoted great effort to the development of scanning transmission electron microscopes (STEM), which have attracted attention as a new method of observing atomic images, and developed two advanced devices of this type. One is an ultra-high vacuum, aberration-corrected STEM. With the conventional magnetic field-type lens, correction of aberration had been considered impossible because concave lenses could not be used, but this Group developed an aberration-corrected STEM (**Fig. 1**) which was the first to incorporate this advanced technology, thereby achieving 1 Å resolution. The second new device is a high brightness, high energy-resolution STEM which is equipped with a cold-field-emission electron gun (Cold-FEG). An atomic image (annular dark-field: ADF) of silicon nitride (Si_3N_4) taken with this device is shown in **Fig. 2**. The individual silicon atoms can be clearly observed as white spots.

3. Development and Application of Nano Magnetic Structural Analysis System

In substances with strongly-correlated electron systems, as represented by superconducting oxides, nano-level observation of the micro magnetic structure (magnetic domains) at ultra-low temperatures is an extremely important problem, but this was difficult with conventional electron microscopes. Therefore, this Group developed two devices, a holographic electron microscope and a Lorenz electron microscope, which were specially designed for use in observation of magnetic structures at the nano level. The former is effective in quantitative analysis of the distribution of magnetization, while the latter is effective for dynamic observation of magnetic domains and magnetic domain walls at the nano level. **Figure 3** (offered Dr. Toru Asaka, JSPS Postdoctoral Fellow) shows an example of observation of the magnetic structure of manganese oxide, which is a promising next-generation magnetic material, at ultra-low temperature with the Lorenz electron microscope. The domain walls can be clearly identified as alternative black and white lines.

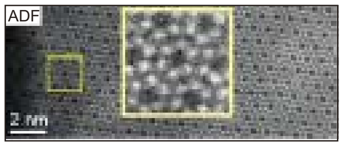

Fig. 2 Example of observation of atomic image (ADF) of silicon nitride (Si_3N_4) with high brightness, high energy-resolution STEM. The square in the center is an enlarged image.

4. Conclusion

The Advanced Electron Microscopy Group is engaged in research with the aim of realizing electron microscopes which demonstrate the world's high performance in identification of atoms by introducing more advanced aberration correction technology and cutting-edge technologies such as new X-ray spectroscopy methods, etc.

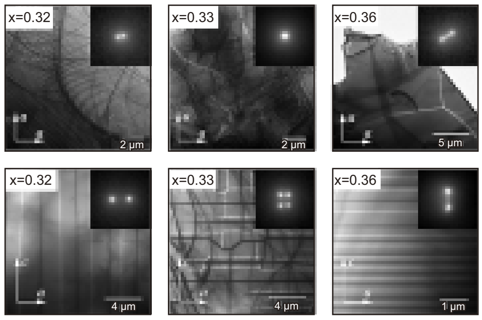

Fig. 3 Ultra-low temperature Lorenz electron microscope image (temperature: 17 K) showing dramatic change in the ferromagnetic domain structure of lamellar manganese oxide $La_{2-2X}Sr_{1+2X}Mn_2O_7$ as a result of slight change in the amount of hole-doping X. The upper part of the photo shows an example of observation along the c-axis; the lower part shows observation along the b-axis is perpendicular to the c-axis.

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

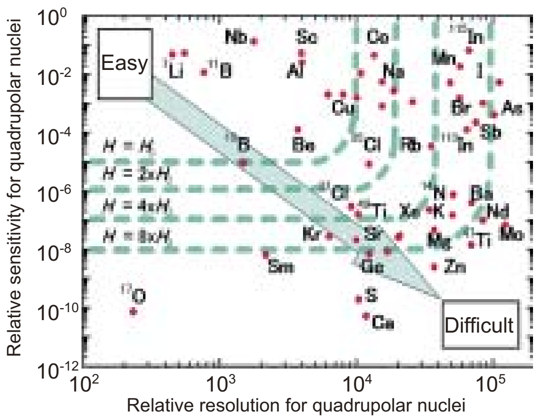

Fig. Sensitivity and resolution with quadrupolar nuclei. Higher magnetic fields (contour line) are advantageous.

practical applications. The condition in which observation of elements that had been difficult to observe with conventional NMR becomes possible with a high field is shown by the contour line in the **figure**. Because the absolute value of the magnetic field is not necessarily meaningful, this **figure** shows the results for high magnetic fields using relative values, with a magnetic field of H_0 (arbitrary intensity) as a standard.

Development of NMR using hybrid magnets, which is currently the only technology capable of producing a steady field of 30 T or higher, is underway in the United States, EU, China, and Japan. Only the US has already completed development of an NMR specification magnet and advanced to collection of NMR data, but the EU is now constructing a magnet targeting the NMR specification. The existing 40 T class hybrid magnet at NIMS boasts the world's 2nd highest field, following only one facility in the US. At present, NIMS is involved in a series of development activities which will upgrade this magnet so that it also satisfies the NMR specification.

Doctoral Program in Materials Science and Engineering
Graduate School of Pure and Applied Sciences, University of Tsukuba
Additional Entrance Exam for April or August 2007 Admission

The Doctoral Program in Materials Science and Engineering will hold an Additional Entrance Exam in February 2007 for enrollment in April or August 2007. The program is managed by NIMS and the University of Tsukuba, and selected scientists from NIMS have joined the graduate school faculty and supervise students' thesis research.

We are also now beginning an "Early one-year completion program," which allows adult students who already have a Master's degree to complete their course within one year at minimum. Please feel free to contact us for details at anytime.

SCHEDULE (Tentative)

Distribution of Application Documents	Late November 2006
Application Period	January 9-11, 2007
Oral and Written Examination	February 5-6, 2007
Announcement of Test Results	February 20, 2007
Enrollment	April 1, or August 1, 2007 (At student's option)


Research Training.

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For more details: http://www.nims.go.jp/graduate/english/

Hello from NIMS

Cai Kai (China)
Research Fellow (April 2005 - Present)
Particle Simulation & Thermodynamics Group
Computational Materials Science Center


[Having a good time near the Imperial Palace]

Hello! My name is Kai Cai. I am a postdoctoral researcher working in the Coating Materials Group at NIMS. After receiving my Ph.D. degree at Tsinghua University in China in 2003, I came to Japan with my husband and continued my research career at the Tokyo Institute of Technology. I joined NIMS in April 2005.

For a researcher working in the multidisciplinary field of materials science, NIMS is a rather ideal institute where you can communicate and cooperate with people of different backgrounds in a relaxed atmosphere. I am happy to cooperate with Dr. Murakami and Dr. Ode. The difference in our previous research topics compensates for our defects in knowledge and produces a synergistic effect, I believe, and as a result, we can try new methods in our work.


[Having fun at Kashiwa-no-ha Park, my daughter RunRun]

It is really exciting news for me that my husband joined NIMS this April. Now we can work together and live just walking distance from NIMS, with our lovely 2-year-old daughter. Every day while my husband and I are at the office, my daughter plays with her new friends in the nursery school.

Compared to the crowded Tokyo area, Tsukuba is a quiet and rural place, which is more favorable for us. Every day we can see the beautiful scene of Mt. Tsukuba surrounded by clouds and mist from our balcony. Tsukuba is a small town in terms of population, but it is a big city from the viewpoint of research. I have heard a saying that one has never been to Japan if he has never been to Kyoto. In my opinion, this also applies to this unique town: Tsukuba is a place one must visit if he/she is a researcher who has once worked in Japan.

NIMS
National Institute for Materials Science

PUBLISHER
Dr. Hisao Kanda

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http://www.nims.go.jp/

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