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B-C-N Nanotubes

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As a substitute for ordinary carbon nanotubes, B-C-N ceramic-based nanotubes are an object of intense interest. B-C-N materials display semiconducting properties and are expected to be used in a variety of applications including photoluminescence materials (light sources), high temperature nanotransistors, lightweight electrical conductors, high temperature lubricants, etc. These applications depend not only on the material composition, but also on the arrangement of B, C, and N atoms. Here, we will report on the creation of a hollow BC_xN (1 × 5) nanotube with a homogeneous distribution of B, C, and N atoms. These were fabricated by reacting bundles of CN_x (x < 0.1) nanotubes with B₂O₃ and CuO under an

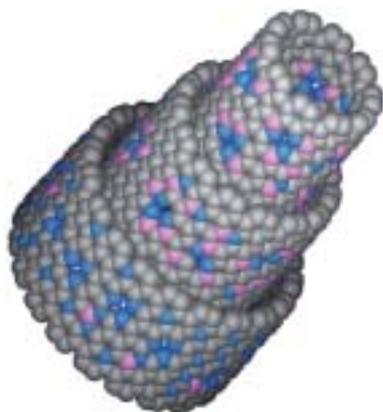


Fig. 2: Structural model of 3-layer B-C-N nanotube (B: blue, C: gray, N: pink).

N₂ atmosphere at approximately 1800 °C. Current-voltage measurements confirmed that the individual BC_xN nanotubes behave as semiconductors.

Fig. 1 shows a bright-field image of a BC_xN nanotube bundle (Fig. 1a) and B, C, and N elemental maps obtained using energy filtering (Fig. 1b, c, d). From these images, it is clear that B, C, and N atoms coexist within the same tube, suggesting that these elements are uniformly distributed in the bundles. We believe that it was possible to obtain this new result by use of CN_x nanotubes filled with nitrogen gas as the starting material, together with a careful selection of the reaction temperature. < Continued on p.2

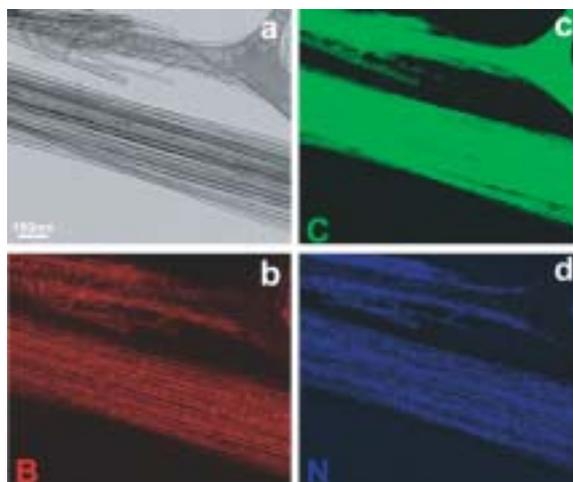


Fig. 1: Electron microscope image of B-C-N nanotube bundle and atomic mapping of B, C, and N (a: Bright-field image, b: B atom, c: C atom, d: N atom).

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Nobel Laureate in Physics, Dr. Heinrich Rohrer, visited NIMS



Ms. Rohrer (left), Dr. Rohrer (Center front), and NIMS executives.

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SPECIAL FEATURES

International Cooperation

-Joint Research with MOU Institutes-

NIMS NEWS

Development of Titanium Surface Treatment Technology in Aqueous Solution

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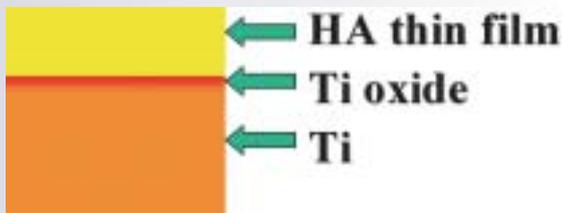


Fig. 1: Formation of HA thin film on Ti surface. A TiO_2 film exists originally.

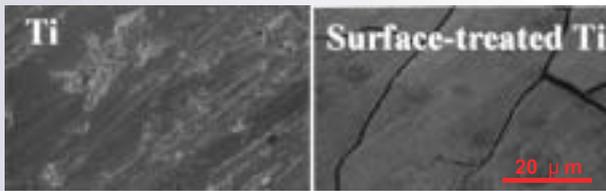


Fig. 2: Formation of HA thin film on surface-treated Ti.

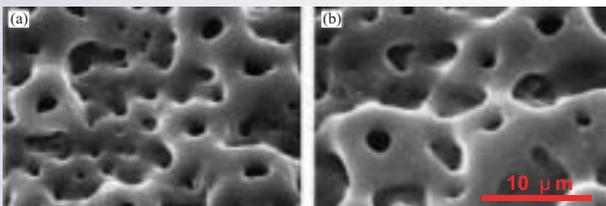


Fig. 3: Porous TiO_2 formed on Ti surface.

cial biofluid, HA forms on the surface rapidly in comparison with untreated material (Fig. 2). Thus, this work clarified the fact that immersion in this artificial biofluid can simulate relatively well the speed of bone formation in the actual body.

In (2), it was found that porous TiO_2 can be obtained on the surface by applying a high voltage, high electric current to Ti in a solution containing Ca^{2+} and PO_4^{3-} (Fig. 3). Formation of bone on this surface is accelerated by activation of the osteoblasts which are responsible for bone formation. This has also been confirmed in animal experiments (Fig. 4).

Kyungpook National University is an institution with a history of actively promoting research on biomaterials. In the future, NIMS hopes to cooperate with this institute to enable mutually complementary research. Based on the research outlined here, we plan to apply these treatment technologies in the development of scaffolding materials for regenerative medicine.

Surface treatments are applied to metal-based biomaterials for a variety of purposes, including improvement of corrosion resistance, wear resistance, biocompatibility with hard tissue (property by which bone forms quickly on material surface), and the antimicrobial property in dental implant. Because metal-based materials have also been applied in circulatory organ-related devices in recent years, surface treatment technique for imparting an antithrombosis property have become an object of research. Among these trends, when the aim is to improve biocompatibility with hard tissue, the mainstream practice is coating of hydroxyapatite (HA) on a Ti surface by plasma spraying. However, problems with this method include the low strength of the HA film itself and poor bonding strength of the Ti interface with HA. For this reason, surface treatment methods in an aqueous solution, such as electrochemical methods and hydrothermal treatments, have become necessary (Fig. 1). Moreover, the bonding strength of the material interface with the bone is affected by the morphology of the material surface. It is known that bonding of bone on a material surface is fastest and most effective when the HA film thickness is 50-400 μm .

In this research, we conducted a study of electrochemical methods of (1) forming HA on the surface (NIMS) and (2) forming a porous material on the surface (Kyungpook National University).

In (1), it was possible to form an extremely thin HA film on the surface by keeping Ti at a potential in the passive region or repeatedly applying potential. When this surface-treated Ti is immersed in a artificial

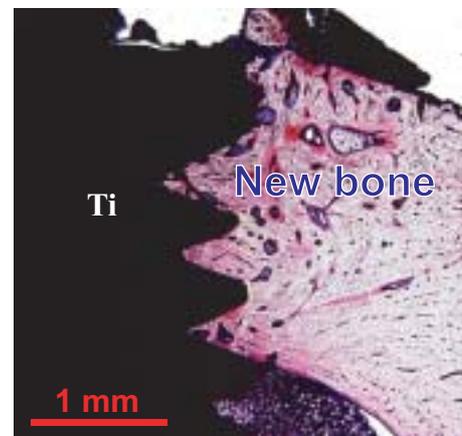


Fig. 4: Formation of bone on Ti surface embedded in rabbit tibia.

For further information, please visit: http://www.nims.go.jp/bmc/bmc02/rsm_e.html

< Continued from p.1

B-C-N Nanotubes

A structural model of a 3-layer nanotube consisting of B, C, and N is shown in Fig. 2. The B, C, and N atoms are shown in blue, gray, and pink, respectively. In each layer, we have hypothesized that numerous BN domains are embedded in an island-like form in a graphite sheet of C. The crucial point involving the current-voltage measurement is that the semi-conducting performance (i.e., band gap) of this structure can be tuned through variation of the band gap from ~ 5 eV (pure BN) to ~ 1 eV (C doped with BN) by changing the number of the BN units in the C nanotube.

The new B-C-N nanotube with a homogeneous structure which we have created for the first time is expected to give birth to a wide range of new applications.

For further information, please visit: <http://www.nims.go.jp/abg/eng/index.html>

http://www.ipicyt.edu.mx/ingeipicyt/dmatm/i_IntroduccionDMATM.htm

Influence of Nanodomain on Properties of CeO₂-based Solid Electrolyte for Fuel Cells Applications

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John Drennan
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Active research and development on small-scale, high-performance fuel cells for use in households and small businesses is underway with the aim of realizing energy savings and reducing CO₂ emissions. In the development of small-scale fuel cells, R&D on solid electrolytes which display high performance in the "low temperature region" of 500 °C and less is important. Although the nanostructure of the electrolyte is not a problem with conventional fuel cells, research on this low temperature region has revealed that the influence of the nanostructure is crucial in low temperature electrolytes.

Ceria-based material is considered a promising material for practical application in low temperature solid electrolytes. Therefore, we are jointly studying the influence of the nanostructure on electric conduction properties in ceria-based solid electrolytes

with Prof. John Drennan. We have already observed the microstructure in several kinds of ceria-based sinters with the transmission electron microscope. As a result, we found for the first time that a nanosize domain (nanodomain) with a different crystallographic structure from ceria coexists in ceria, and this is a cause of reduced conduction.

Fig. 1 shows the grain size dependence of the conductivity and activation energy of samples prepared by pressure-less sintering and pulse current pressure sintering (PS) using Dy_{0.2}Ce_{0.8}O_{1.9} nanospherical powders. In sinters with small grain sizes, conductivity decreases temporarily, but then rises rapidly at grain sizes under 250 nm. A further increase in conductivity was also possible by applying PS. Among the microstructure observation results, in the

electron diffraction pattern observed from sample b in **Fig. 1**, in addition to spots (bold arrows) originating from a fluorite structure, small strength spots (thin arrows) originating from a distorted pyrochlore structure and diffuse scattering originating from nanodomains (pyrochlore structure) were observed, and the size of the domain surrounded by the dotted line in the lattice image also appears to extend to 10 nm (part indicated by red arrow in **Fig. 2**). On the other hand, in sample a, the domain size was small, being on the order of 1 nm, indicating that suppression of domain growth is critical for property improvement. In the future, we plan to conduct a more detailed structural analysis of domains and propose a solid electrolyte with higher properties by innovation in the solid electrolyte production process.

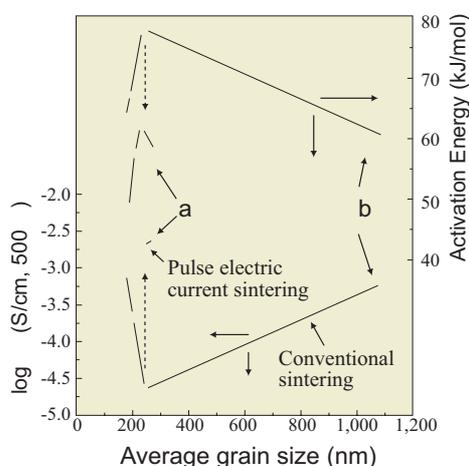


Fig. 1: Influence of grain size on electrolytic properties of Dy_{0.2}Ce_{0.8}O_{1.9} sinters.

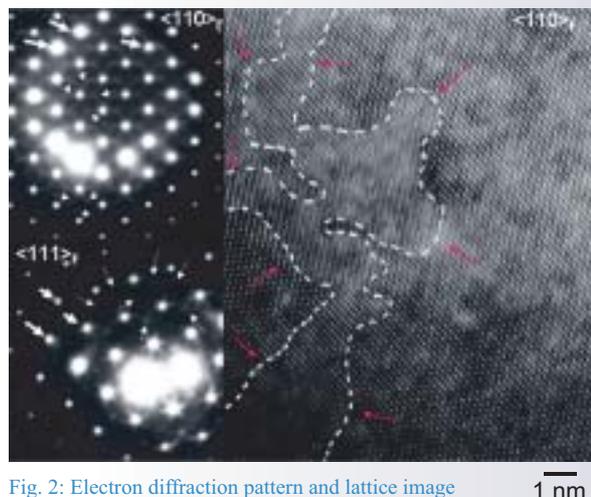


Fig. 2: Electron diffraction pattern and lattice image observed from sample b.

For further information, please visit: <http://www.nims.go.jp/ecoenergy/mori-files/contents1.htm>
http://www.uq.edu.au/nanoworld/about_cmm.html

Successful Development of Red Phosphor for White Light LEDs — New-generation Lighting —

A new red phosphor for use in white light-emitting diodes (LEDs) was successfully synthesized in joint work by Naoto Hiroasaki of the Non-Oxide Ceramics Group, Advanced Materials Laboratory (AML), and a group under Prof. Hajime Yamamoto and Assistant Prof. Kyoto Uheda of Tokyo University of Technology. This material was produced by mixing silicon nitride, aluminum nitride, calcium nitride, and europium nitride powders in a moisture/air sealed glove box, placing the mixture in a boron nitride crucible, and reacting the material in nitrogen under 10 atm. at 1800 °C. The resulting substance displays extremely intense light emission and excellent properties, with virtually no deterioration in the temperature range from -240 °C to 100 °C. Use of the developed red phosphor will enable production of more natural lighting by adding a reddish tint to white LEDs. Use in flat panel displays and other products is also expected, taking advantage of its properties. Evaluation of the material in cooperation with manufacturers is planned, aiming at practical application in two years.

For further information, please visit: <http://www.nims.go.jp/sialon/index.html>

Development of Multi-functional Photocatalysts and Their Potential Application to Air Purification

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Electroceramics Group
Advanced Materials Laboratory (AML)

Nitin K. Labhsetwar
Environmental Materials Unit
NEERI, India

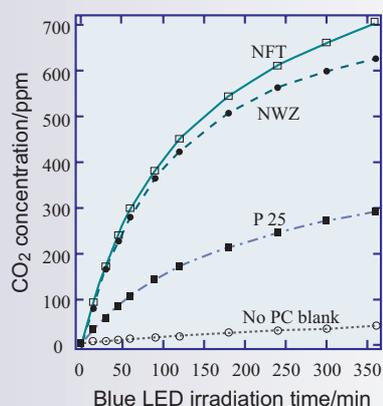


Fig. 1: CO₂ evolution during acetaldehyde decomposition on the NWZ and NFT photocatalysts.

Visible-light-active TiO₂-based photocatalysts: N-F-codoped TiO₂ photocatalyst (NFT) was developed successfully. Fig. 1 indicates that the NFT powder is consisted with porous particles, the structure of the particles is composed of nanosized crystallites like the case of NWZ. The NFT powder can absorb part of the visible light spectrum due to N-doping. Moreover, the surface of NFT particles demonstrates strongly acidic due to F-doping. Both highly porous surface and strongly acidic surface will be beneficial for the adsorption for reactants. As shown in Fig. 2, the NFT powder shows a high visible light activity in the acetaldehyde decomposition, being much higher than that of P 25.

Multi-functional materials by composite of activated carbon aerogel (ACA) and photocatalyst: ACA is a novel adsorbent with large surface area and high pore volume. It can condense low-concentration environmental pollutants (e.g., trichloroethylene) and temporarily stores them in its porous structure. Pollutants adsorbed around the pore openings are first degraded by the photocatalyst, and accompanying this, pollutants deeper in the pore are gradually diffused to the pore opening until finally all of the pollutants are decomposed.

A great deal of potential for application to air purification can be expected for our multi-functional materials.

Thermocatalytic and photocatalytic technologies have been attracting much interest in the remediation of environmental pollutants. In the area of thermocatalysts, we are engaged in the researches on Ru-based oxide catalysts for the remove of NO_x, particulate matter, and dioxins in exhaust gas, and a good result was achieved by model experiments. On the other hand, photocatalyst has an outstanding advantage, i.e., it has strong capacity in the mineralization of environmental pollutants with a low concentration. However, a photocatalyst generally has a low adsorptivity for the pollutant molecules. Therefore, the development of multi-functional catalysts (visible-light response, high activity, and high adsorptivity) will be important for practical applications. Based on these considerations, we focus our attention on the development of the following novel materials.

Visible-light-active ZnO-based photocatalysts: N-doped WO₃-ZnO composite photocatalyst (NWZ) was prepared by spray pyrolysis. The SEM images of the NWT powder are shown in Fig. 1. They are hollow spherical particles; the shell of the particle is composed of nanosized crystallites. As shown in Fig. 2, NWZ powder demonstrates high photocatalytic activity in acetaldehyde decomposition. The visible light photocatalysis of ZnO was greatly improved by simultaneous introductions of nitrogen and WO₃.

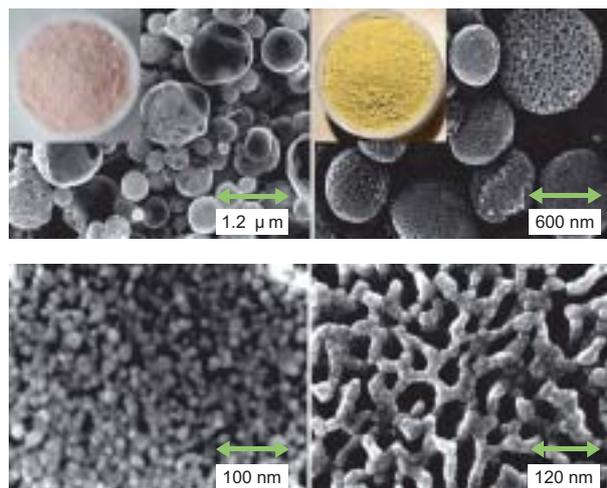


Fig. 2: Sprayed nanosized NWZ powder (left line); sprayed porous NFT powder (right line).

For further information, please visit: http://www.nims.go.jp/denzai/index_e.html
<http://www.neeri.nic.in/EMU.html>

MOU with Institute of Nuclear Chemistry and Technology, Poland



Director Yamada (EMC), Director Walis (INCT), and Prof. Michalik (INCT).

On October 25, the NIMS Ecomaterials Center (EMC) and the Institute of Nuclear Chemistry and Technology (INCT, Poland) signed a memorandum of understanding (MOU) on "Materials and Nanotechnology for Environmental Purification." The two institutes agreed to form a cooperative research relationship for creation of materials which will contribute to realizing a recycling-oriented society and plan to promote joint research programs and conduct mutual visits by researchers, exchanges of human resources, and exchanges of research information over a five-year period.

For further information, please visit: http://www.nims.go.jp/emc/emc_eng/index_e.html
<http://www.ichtj.waw.pl/>

Nobel Laureate in Physics, Dr. Heinrich Rohrer, visited NIMS

On November 25-26, NIMS received a visit from Dr. Heinrich Rohrer, the 1986 Nobel Prize winner in Physics for his design of the Scanning Tunneling Microscope (STM) and a former Fellow at the IBM Zurich Research Laboratory.

On 25th, Dr. Rohrer gave a lecture entitled "Birth and Adolescence of STM," which was the same title as his Nobel lecture, at an International Center for Young Scientists (ICYS) Special Seminar. He later had one-on-one discussions with six ICYS Research Fellows on their research work. In the evening, NIMS researchers and their families were honored to welcome Dr. Rohrer to the annual Crystal Festival, which featured an Okinawa dance performance and historical drama (see back cover). On the following day, he participated in a "Nanotechnology Mini-workshop" hosted by Nanomaterials Laboratory (NML), ICYS and Institute for Solid State Physics of University of Tokyo, and awarded prizes to participants who made excellent presentations.

For further information, please visit: <http://www.nims.go.jp/icys/Rohrer.htm>



Director Skorokhod (IPMS), Vice-Director Ragulya (IPMS), and Dr. Sakka (MEL).

MOU with Institute for Problems of Materials Science, Ukraine

On October 11, the NIMS Materials Engineering Laboratory (MEL) signed an MOU with the Institute for Problems of Materials Science (IPMS, located in Kiev) of the Ukraine Academy of Science. The IPMS is known as a key institute in the fields of powder metallurgy and ceramics in the former Soviet sphere, as shown by the fact that it edits the international journal *Powder Metallurgy and Metal Ceramics*. The two sides agreed to a further expansion of exchanges, which have centered on post-doctorates to date, and will promote mutual visits, exchanges of human resources, and international exchanges of research results in a wide range of fields, including nanotechnology based on powder processes.

For further information, please visit: http://www.nims.go.jp/MEL/index_E.htm

Introduction of New Fellows

In fiscal year 2003, NIMS inaugurated a system under which outstanding researchers from outside the organization are invited as NIMS Fellows. In order to create an environment in which the excellent researchers within NIMS can also devote themselves to their research activities in a settled atmosphere, NIMS expanded its Fellow program to include researchers in NIMS itself. As part of this effort, the following three persons were newly approved as NIMS Fellows. The appointments were made by the President Kishi based on a review by the Executive Committee, applying the standard that Fellows "have a record of remarkable achievements in materials-related fields, and can also be expected to make important contributions to research activities at NIMS in the future." NIMS Fellows will also be reemployed after reaching retirement age so that they can continue to provide leadership through research activities.

<http://www.nims.go.jp/icys/au/au-03.html>

Dr. Yoshio Bando

Doctor of Science (1975). Completed doctoral course in Dept. of Science, Graduate School, Osaka University. Researcher at National Institute for Research in Inorganic Materials (NIRIM; 1975), where he also served as Senior Researcher and then Supervising Researcher. Named Director, Advanced Beam Analysis Group, Advanced Materials Laboratory (AML), with concurrent appointments as General Manager of the Integrated Strategy Office (2001) and Director-General of the International Center for Young Scientists (ICYS; 2003), NIMS. Has also held a Chair as Professor of the Institute of Materials Science, University of Tsukuba since 1993, and as Professor of the Graduate School of Pure and Applied Sciences of the same university since 2004. Named NIMS Fellow in December 2004.



Dr. Kazuhiro Hono

<http://www.nims.go.jp/apfim/hono.html>

Ph.D. in Metals Science and Engineering at the Pennsylvania State University (1988). Post-doctoral research associate at Carnegie Mellon University (1988), Research Associate, Institute for Materials Research, Tohoku University (1990), Senior Researcher (1995), Group Head (1995) at National Research Institute for Metals. Director of the Metallic Nanostructure Group, Materials Engineering Laboratory, NIMS (2002). Concurrently appointed as Associate Professor (1999) and Professor (2003) of Materials Science at the Graduate School of Pure and Applied Sciences, the University of Tsukuba (1999). Appointed as NIMS Fellow in December 2004.



<http://www.nims.go.jp/amlaml/english/csag.html>

Dr. Akiji Yamamoto

Doctor of Science. Completed master's course in Dept. of Science and Engineering, Graduate School, Tokyo Institute of Technology (1971). Joined the National Institute for Research in Inorganic Materials (NIRIM) and thoroughly engaged in the development of structural analysis of aperiodic crystal as Researcher and then Senior Researcher. Appointed Director of Crystal Structure Analysis Group, Advanced Materials Laboratory (AML; 2002), NIMS. Named NIMS Fellow in December 2004.

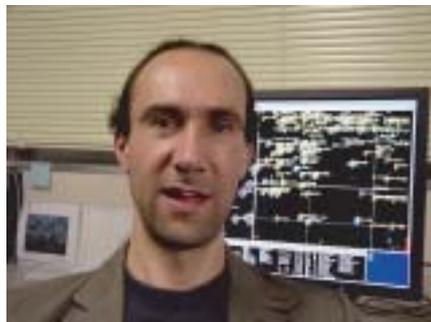


Hello from NIMS

Hello from the Tsukuba Magnet Laboratory at the NIMS Sakura Site, home of the world's highest field strength superconducting magnet for solution state NMR. A little over seven years ago, I took advantage of the STA Fellowship program to come to Tsukuba, where I have since worked and specialized in the field of protein NMR, in particular the use of NMR to solve the three dimensional structures of proteins, and to study their interactions with other biological molecules. The latter is particularly important since it is a protein's binding partners that determine its function, and the interactions between molecules are at the heart of biological processes. Earlier this year, I joined RIKEN, who have a large NMR facility at their Genomic Sciences Center in Yokohama, and collaborate with NIMS in the development of NMR technology. Surrounded largely by very knowledgeable and helpful engineers and material scientists, from Jeol and Kobe Steel as well as NIMS, I am the lucky molecular biologist who gets to use the fruits of their research, the 920 MHz superconducting magnet, the high field strength of which has certain advantages for my own research.

Outside of work, I like to escape from the concrete of the Kantou Plain and the coastal regions, and head to the mountains, where there is still much beauty. Please take a look at the website of the Tsukuba Walking and Mountaineering Club (TWMC), a club I helped to form shortly after my arrival in Japan: <http://eve.bk.tsukuba.ac.jp/twmc/>

Paul Reay (RIKEN GSC Protein Research Group)
Visiting Researcher (Apr. 2004 - May. 2005)
Magnet Development Group
High Magnetic Field Center (Tsukuba Magnet Laboratory)



★ NIMS Winter Events 2004 ★

Crystal Festival (Nov. 25)

- Annual festival of Namiki Site to pray for workplace safety -



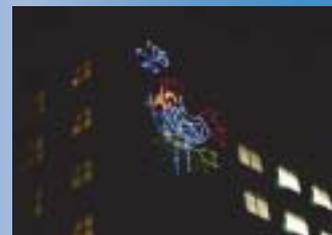
Fuigo Festival (Nov. 8)

- Traditional festival of blacksmith to pray for safety and prosperity -



Xmas & New Year Illumination Display (Dec. 10, 2004 - Jan. 7, 2005)

- Illuminations designed by local students and made by NIMS volunteers -



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