from Tsukuba, Japan to the world

NIMS NOW International

National Institute for Materials Science

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New Year 2007

President's Greeting

Prof. Teruo Kishi NIMS President

First, I would like to wish all of our colleagues and friends a very happy New Year.

Last year, NIMS completed its 1st Mid-Term Program, which covered the period from April 2001 to March 2006. The 5-year period of our 2nd Program began in April 2006.

The national government's 3rd Science and Technology Basic Plan also began in April 2006, and "Nanotechnology and Materials" will continue to be one priority field under this new plan. The new Plan places particularly strong emphasis on innovation. Looking at history, materials have frequently caused revolutionary changes in the times, and from this viewpoint, we are confident that our efforts to pioneer fundamental/basic research in the materials field is the most effective road to achieving innovation.

In promoting our 2nd Mid-Term Program, we have prepared three reports intended to define the past, present, and future positioning of NIMS. Looking back over the last 5 years, in "Research Accomplishments/NIMS 21," we selected 21 interesting research achievements from our 1st Program. Because we have now substantially achieved our numerical evaluation targets, such as the number of published papers, we focused on truly challenging and innovative results. Looking to the long-term future, in "NIMS Policy Paper 2020," we expectedly attempted to predict the ideal form of NIMS in the year 2020, On the other hand, "Materials Science Outlook 2006" takes a birds-eye view of current materials research/ nanotechnology research worldwide in order to clarify the positioning of the 20 projects which NIMS is carrying out under its 2nd Program. To gain a better understanding of our work, I invite all of our friends to peruse these three volumes.

As our organization for promoting research, in addition to the 20 Centers which will carry out the 20 projects mentioned above, we have also created 2 Laboratories to conduct germinal research and 8 Stations, which are responsible for research infrastructure. In the future, we also aim to establish a unit which we are tentatively calling the Interdisciplinary Materials Development Center to make our facilities available to both internal and external researchers.

With the aim of becoming the world's foremost materials research institute, we are engaged in the creation of new materials, practical application of materials, and construction of the materials information infrastructure through strengthening industry/academic/governmental collaboration and international collaboration. For this, a research environment with a high degree of freedom and the cooperation of researchers with a high level of independence inside and outside of NIMS are indispensable.

In closing, I would like to request your continuing guidance and support in the coming year, and I offer my very best wishes for the success of all of your efforts.

* Please visit http://www.nims.go.jp/eng/news/ for downloading NIMS publications including "Research Accomplishments/NIMS21" and "Materials Science Outlook 2005". English version of "NIMS Policy Paper 2020" and "Materials Science Outlook 2006" will be ready in spring.

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A Visit by the President of Thailand's NSTDA and Conclusion of MOU with the MTEC

Dr. Sakarindr (right) and President Kishi.

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Creation and Functionalization of Novel Nanotubes and Nanosheets at NMC

Takayoshi Sasaki
Managing Director
Nanoscale Materials Center (NMC)

The Nanoscale Materials Center (NMC), which was established in the Nanoscale Materials Field of NIMS, aims to create nanoscale materials that are expected to exhibit unique physical properties resulting from miniaturization and morphological control to the nanometer order. In addition, we develop advanced applications of these materials in fields such as IT, environmental protection, and bioscience. The Center comprises of the following four research groups, and is conducting research with a particular focus on 1-dimensional nanotubes and 2-dimentional nanosheets, as exemplified in the **figures**, from the fundamental/basic standpoint.

1. Soft Chemistry Group
2. Soft Ionics Group
3. Nanomaterials Synthesis & Analysis Group
4. Nanotubes Group

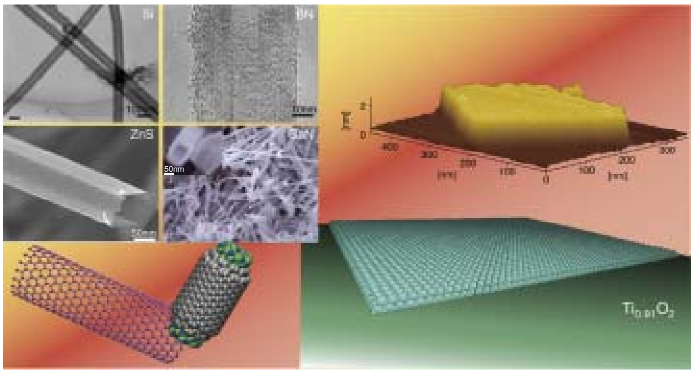

Fig. 1 (Upper left) TEM images of various nanotubes (Si, BN, ZnS, GaN). (Upper right) AFM image of oxide nanosheet on substrate. (Lower) Structural models of boron nitride (BN) nanotube and its metal-included form (left) and titanium oxide nanosheet (right).

The Center has been accumulating unique synthetic techniques in "soft chemistry," which utilizes the chemical reactivity of inorganic materials around room temperature, and the world's top class electron microscope observation techniques. These superiorities of the center are fully utilized in systematic creation of novel non-carbon nanotubes and oxide and hydroxide nanosheets which possess various functionalities such as semiconducting properties, ferromagnetism, permittivity, and the like. Furthermore, the Center's objectives also include the development of nano-hybrid materials with sophisticated functions, which are difficult to realize with mono-phase materials, by precise integration and hybridization of these nanoscale materials at the nano level. The research activities of each group of NMC are introduced in the following pages.

For more details:
http://www.nims.go.jp/nmc/index-e.html

NIMS News

NIMS Exhibited an Academic Journal "STAM" and a Portal "e-materials.net" at EMRS and MRS Fall Meetings

The Materials Information Team of the NIMS International Affairs and Public Relations Office set up a promotional booth at the 2006 EMRS Fall Meeting held on Sept. 4 - 8 at Warsaw Institute of Technology, Poland, as well as at the 2006 MRS Fall Meeting held on Nov. 27 - Dec. 1 in Boston. The total numbers of attendees at the EMRS and MRS meetings were about 1,000 and 6,000, respectively. Booth visitors numbered roughly 30 per day at both meetings. The team promoted the English language peer-review journal "Science and Technology of Advanced Materials" (STAM) that is edited by an international editorial board and published by NIMS with a partner publisher, Elsevier Ltd. In this year STAM has published five special issues focused on projects from the Center of Excellence program that is sponsored by Japanese government. Those issues have established STAM as a journal that grasps and disseminates the "current status of materials research in Japan". We distributed more than 400 copies of these unique issues of the journal at the EMRS and MRS meetings.

We announced and advertised the following upcoming journal contents: (i) a special issue entitled "Superconductivity in Diamond and Related Materials" which is edited by Dr. Y. Takano *et al.* and (ii) an invited review paper, "New Families of Mesoporous Materials" by Dr. Ariga *et al*. There were many requests for sample copies or reprints of the above-mentioned issue and article.

The team also displayed promotional banners and posters and demonstrated a portal-site "e-materials.net" on-line, which is one of our main services. The site is provided to scientists to support and foster research activities. NIMS plans to open the beta version of the web-portal to researchers, and to continue to develop and improve it throughout 2006/2007.

2006 MRS Fall Meeting: Prof. R. P. H. Chang* of Northwestern University visited the STAM/Portal-site booth. (*Prof. Chang is the General Secretary of International Union of Materials Research Societies (IUMRS) and established the Materials World Network.)

The Human Resources Development Office and International Young Scientists Center of NIMS cooperated in displaying posters and distributing pamphlets. Many visitors were interested in NIMS research positions and there were many inquiries.

A new trend could be seen in the MRS exhibition; institutional research activities, facilities and network activities were newly exhibited in addition to the usual corporate exhibits of equipment and raw materials. Institutional and corporate recruiting activities were very popular. Many people carefully read job offers and lined up for interviews. We will continue to present our exhibit in the future, in cooperation with HRDO and ICYS, placing importance on further publicizing NIMS research activities and promoting the exchange of research information.

For more details: http://www.e-materials.net/stam/index_e.html

Synthesis and Integration of Novel Nanosheets to Develop Advanced Functional Materials

Takayoshi Sasaki, Nobuo Iyi
Akio Watanabe, Yasuo Ebina
Minoru Osada, Tadashi Ozawa
Soft Chemistry Group
Nanoscale Materials Center (NMC)

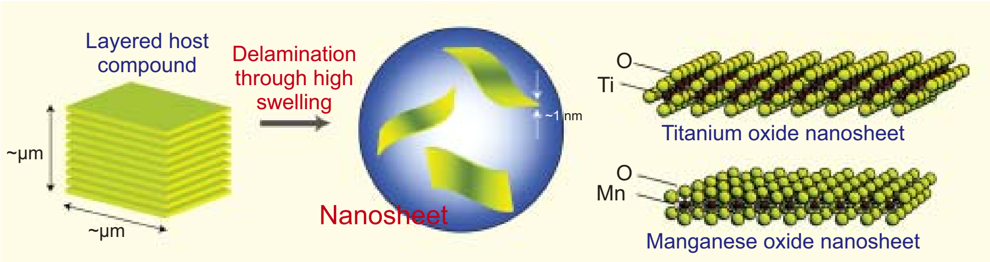

Fig. 1 Conceptual diagram of the production of nanosheets by delamination of a layered host compound to its elementary layer. This figure shows a part of the material, and the material extends substantially infinite in the lateral direction at the scale of the figure.

Many compounds with crystal structures consisting of stacked layers, as exemplified by graphite and mica, are known. The Soft Chemistry Group is conducting research on nanosheets obtained by a unique technique of delaminating layered host compounds to their elementary, single-layer form by chemical procedures. (**Fig. 1**) The first and foremost property of nanosheets is its morphology. Because the minimum basic unit of the layered structure is extracted, these materials normally have a lateral size of micron or larger relative to their thickness of around 1 nm, which is equivalent to several atoms, and display extremely high 2-dimensional anisotropy.

The most important objectives of the Group are to perform the challenging task of delaminating various layered host compounds and to synthesize diverse oxide and hydroxide nanosheets. Because the thickness of nanosheets is in the range where the properties truly unique to the nano size appear, including quantum confinement effects and others. Thus, elucidation of their physical properties is one critical issue. Moreover, because nanosheets remain largely unexplored, this is a class of materials with high potential for the discovery of novel properties and phenomena. In fact, the Soft Chemistry Group has already discovered a succession of extremely interesting properties, including high permittivity and room-temperature ferromagnetism resulting from doping with cobalt and other magnetic elements in titanium oxide nanosheets (**Fig. 2**). As an even more fascinating feature of nanosheets, when these are utilized as carefully stacked "building blocks," it is possible to construct diverse nanostructures. Furthermore, because a colloidal suspension can be obtained by dispersing nanosheets in a liquid medium, it is possible to perform these operations in a liquid phase. This enables various types of nano-level structural control, such as nano-thin films, which can be formed with a thickness precision of 1 nm, hollow shells with an inner void volume of more than 90 %, and fine particles having an extremely high surface area.

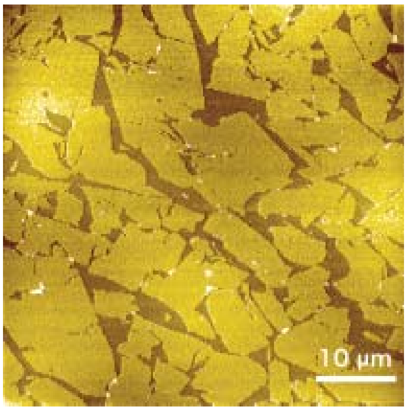

Fig. 2 AFM image of Fe-doped titanium oxide nanosheet. Dense tiling of a substrate surface with a 1 nm thick nanosheet is possible. The obtained ultra-thin film functions as a transparent magnet at room temperature.

We are continuing our efforts with the ultimate aim to develop a new technology called "wet process nanotechnology," in which desired functionalities are realized by artificial integration and hybridization of nanosheets consisting of 2-dimensional crystals with molecular-level thinness as planned.

For more details: http://www.nims.go.jp/softchem/index-e.html

NIMS News

< Continued from p.1

A Visit by the President of Thailand's NSTDA and a Conclusion of MOU with the MTEC

Dr. Paritud (left) and Dr. Shinohara, Group Leader of NIMS Corrosion Group.

(November 14, 17, NIMS) -- Dr. Sakarindr Bhumiratana, the President of Thailand's National Science and Technology Development Agency (NSTDA), visited NIMS on November 14. The NSTDA belongs to Thailand's Ministry of Science and Technology and is responsible for four national laboratories, the National Center for Genetic Engineering and Biotechnology (BIOTEC), National Metal and Materials Technology Center (MTEC), National Electronics and Computer Technology Center (NECTEC), and National Nanotechnology Center (NANOTEC). President Sakarindr and President Kishi of NIMS held informal discussions in an exchange of information and opinions on science and technology policy in the two countries and the promotion of cooperation between NSTDA and NIMS. Dr. Sakarindr also observed work in research groups, including corrosion, metal fatigue, biomaterials, ultra-fine structural analysis, etc.

In particular, MTEC and NIMS Corrosion Group of the Materials Reliability Center signed an MOU on corrosion research on November 17 and will begin joint research in this area. MTEC conducts a wide range of materials research including metal materials, ceramics, high-polymer materials, and biomaterials. Dr. Paritud, President of MTEC, discussed with President Kishi about the science and technology policies and the future collaboration between the two institutes. The two institutes are planning to promote exchanges of researchers and research information and to carry out cooperative research beginning by the study of "The influence of environmental factors on metal corrosion".

Creation of Nanoscale Ionic Materials

Kazunori Takada, Renzhi Ma
Soft Ionics Group
Nanoscale Materials Center (NMC)

In contrast to electronics, in which electrons play the key role, phenomena related to ions are termed "ionics." It goes without saying that the ions in ionic materials display large mobility. As a result, for example, when two species of ionic conductors are placed in contact, ions migrate from one side to the other, forming ion vacancies and interstitial ions in a region of several nanometers at the contact interface. Because ion conduction phenomena which differ from those in the bulk appear at this interface, the discovery of novel properties can be expected if ionic materials themselves are reduced to the nanometer scale. The Soft Ionics Group is engaged in research on ionic materials in the nanoscale region, beginning with reduction of such materials to the nanoscale size.

Because electrodes and electrolytes are joined in ionic devices such as batteries and chemical sensors and ions flow across the interface, research on the interfacial region of ionic materials is extremely important. Through research on such interfaces, we succeeded in realizing high power in an all-solid-state lithium battery. The lithium ion battery has become an indispensable device in today's information society. However, securing safety is an important issue because lithium ion batteries, unlike other cells, use a flammable organic solvent electrolyte. Safety can be improved dramatically by using a ceramic electrolyte in place of the organic solvent electrolyte, but a large current cannot be obtained. As a result of our research on interfacial ionic conduction, we introduced a third ionic conductor at the interface between the cathode ($LiCoO_2$) and the ceramic electrolyte several nanometers in thickness as a buffer layer (**Fig. 1**), and succeeded in realizing high speed ion conduction at this interface. Although this battery has a completely solid-state composition, it displays high-rate performance comparable to that of lithium ion batteries using a liquid electrolyte (**Fig. 2**). Research on this all-solid-state lithium battery was carried out on commission from Japan's New Energy and Industrial Technology Development Organization (NEDO).

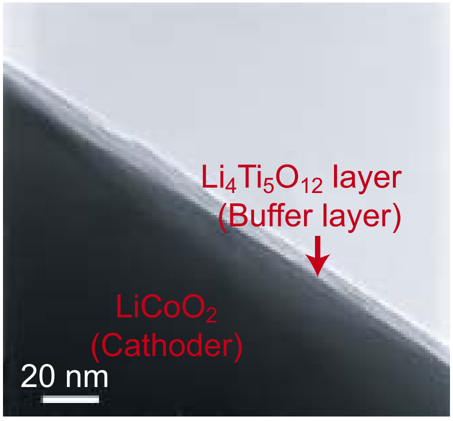

Fig. 1 TEM image of buffer layer ($Li_4Ti_5O_{12}$).

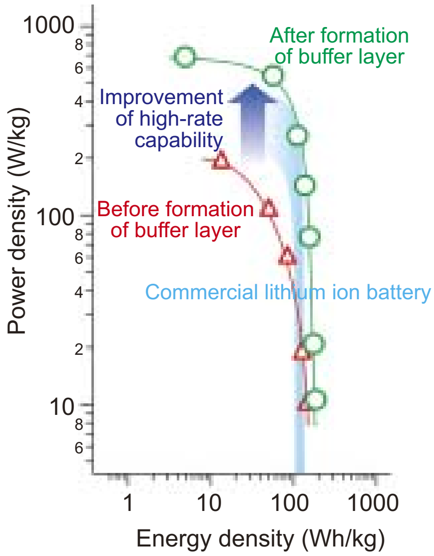

Fig. 2 High-rate performance of all-solid-state lithium battery.

For more details: http://www.nims.go.jp/softionics/index-e.html

In-situ Electrical and Mechanical Probing of Nanotubes inside a 300 kV High-Resolution Transmission Electron Microscope

Dmitri Golberg, Masanori Mitome
Nanotubes Group
Nanoscale Materials Center

The Nanotubes Group launched a full-range study on mechanical and electrical properties of carbon, boron nitride and other inorganic nanotubes (NTs), like B-C-N, ZnO, ZnS *etc* with the aid of a TEM-compatible "Nanofactory Instruments" piezo-driven STM-TEM holder. By using this holder it is possible to physically contact an individual NT, to electrically probe it, as well as to manipulate and deform it, all inside TEM. Importantly, all conventional TEM and HRTEM operations, *e.g.* high-resolution imaging, spatially-resolved electron diffraction, elemental mapping using electron-energy loss (EELS) and energy dispersion X-ray (EDX) spectroscopies are possible during all stages of probing. This is an appealing advantage of the present technique over many pre-existing studies. So far, most of the mechanical and electrical property evaluations of NTs have been performed in atomic force microscopy (AFM) or scanning electron microscopy (SEM) setups which suffer from a limited spatial resolution. This makes precise analysis of the particular NT structural features impossible and mechanics/physics behind them unsolved. Moreover, even if the time- and effort-consuming electrical or mechanical tests on an individual NT have successfully been accomplished, there has been always a huge

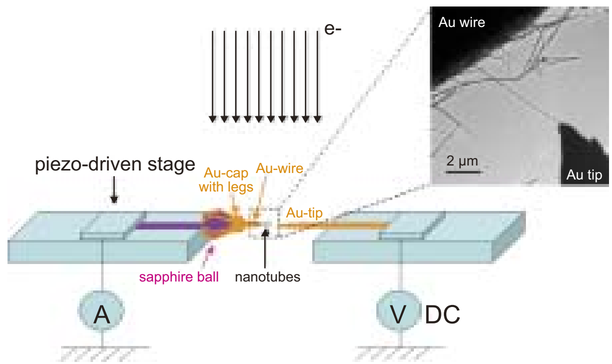

Fig. 1 A sketch of a STM-TEM piezo-holder utilized for the electromechanical measurements of nanotubes. The inset shows a view of the layout inside TEM.

Synthesis of Novel Nanoscale Materials and Their Functionalizations

- Recent Achievements in BN Nanotubes -

Yoshio Bando, Ryutaro Souda
Takao Mori, Chengchun Tang
Nanomaterials Synthesis and Analysis Group
Nanoscale Materials Center (NMC)

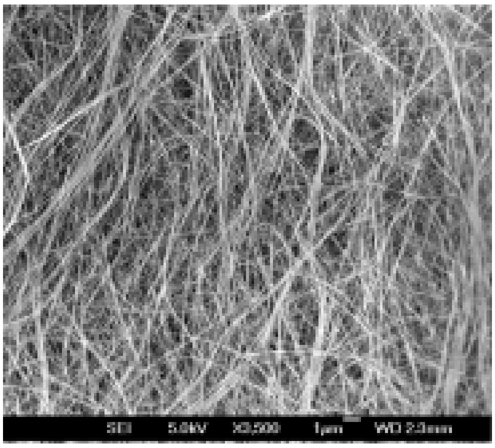

Fig. 1 High purity BNNT synthesized by carbon-free CVD process.

Because nanoscale materials, as represented by carbon nanotubes (CNT), can be made to manifest novel functions not seen in the bulk, high expectations have been placed on their application to electronics, bioscience, etc. as new materials which hold the key to nanotechnology. The Nanomaterials Synthesis and Analysis Group conducts research in which we search for novel nanotubes, nanowires, and other substances whose existence was unknown heretofore and elucidate their structures and functions. In particular, we are devoting great effort to research aimed at mass synthesis of high purity boron nitride (BN) nanotubes and manifestation of functions in this material.

In comparison with CNT, BN nanotubes (BNNT) have high heat resistance and oxidation resistance, and also have excellent qualities in terms of insulating properties and heat conduction, but until now, these BNNTs had the drawback of being difficult to synthesize. However, we recently developed an innovative chemical vapor deposition (CVD) process which does not use carbon as the starting material, and succeeded in synthesis of impurity-free high purity BNNTs (**Fig. 1**). The results of this research revealed that BNNTs are a hydrogen storage absorbent, and with fluorine doping (F-BN), their properties can be changed from their essential insulator property to a semiconductor property (**Fig. 2**).

The applications of BNNTs include composites with polymers. It is possible to improve various properties of polymer films, including their strength, heat resistance temperature, etc., simply by mixing several wt.% of BNNTs in the polymer. As new materials which are transparent and lightweight, and also have high heat conduction, strength, heat resistance, and insulating properties, high expectations are placed on these hybrid materials in applications such as IC boards and heat-resistant polymers, among others.

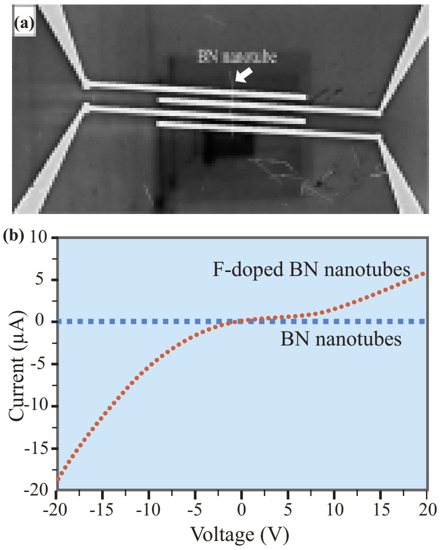

Fig. 2 Synthesis of semiconductor by F-doping of BNNT. When measured by the 4-probe method (a), the pure BNNT (b) (blue) is an insulator which does not pass an electric current, but its characteristics change to those of a semiconductor as a result of fluorine doping (red).

For more details: http://www.nims.go.jp/synthesis/index-e.html

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degree of uncertainty as to what particular structure, morphology and/or NT chemical composition they are related to and how a given NT was affected during the data collection.

Figure 1 shows a general setup of a piezo-driven TEM-STM holder. The gold wire with attached NTs is inserted into the movable part of a holder with the NTs exposed outwards. The etched gold wire (0.25 mm in diameter) having a sharp etched tip is used as a counter-contact/electrode. This electrode may be biased during holder manipulation up to ±140 V. The *in-situ* TEM electromechanical experiments were performed in a JEM-3100FEF Omega-filter 300 kV field-emission HRTEM.

Figure 2(left and middle) displays an example of multi-walled BNNT mechanical probing. NT was gently squeezed between the two gold contacts/electrodes. The tube exhibits superb flexibility which was not *a-priori* expected for a layered partially ionic BN compound. The reversible bending deformation was found to be truly elastic with no traces of a residual plastic deformation up to reversible bending angles exceeding 70°. The highly corrugated BN tubular layers in the vicinity of a reproducibly appearing kink fully restore its original shape after reloading. A kink position on the BNNT was persistent. The characteristic *I-V* curve simultaneously taken on the unloaded BNNT is shown in **Figure 2(right)**. BNNT is a perfect insulator up to the voltages of approximately ±25-30 V. At voltages of more than ±30 V BNNTs start to display passing currents exceeding several tens of nA. The electric transport was reversible and did not lead to any morphological destruction of BNNTs.

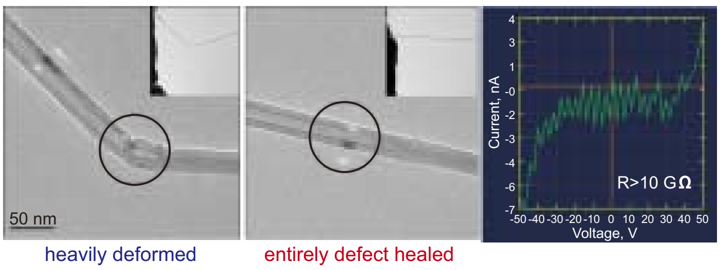

Fig. 2 In situ TEM deformed (left) and relieved (middle) BN nanotube. Highly corrugated BN tubular shells fully restore their original shape after reloading. A representative I-V curve taken on unloaded BN tube is shown on the right. This displays the true characteristics of an electrical insulator.

Finally it is emphasized that *in-situ* TEM evaluation of NT electromechanical properties is very important as far as prospective structural, electrical or functional applications of NTs, and their integration into modern nanotechnology, *e.g.* nanoscale sensors, actuators and nano-electromechanical systems (NEMS) with integrated electronic/optical functions are concerned.

For more details: http://www.nims.go.jp/nanotube/index-e.html

Hello from NIMS

A Learning Experience

My name is Joseph but everybody calls me Joe. I am from Dublin and am going through the final year of my honours degree in Chemistry from Trinity College Dublin.

Of the many benefits one has studying chemistry the best I feel is the opportunity to travel. Through a combination of "Connections" and an outrageously exaggerated CV, I was able to get a place on the NIMS internship program all the way on the other side of the world. I was on paper there solely to conduct my final year research project with a view to producing new materials for use in fuel cells.

But you don't travel just to work in different labs so I took every opportunity I could find to get to know the mix of cultures that is Tsukuba. I found the series of cultural activities set up by ICYS to be most useful and that everybody I met in ICYS and NIMS in where always willing to help. I suppose that which I have taken back with me most is the great impression of all the people that I have found in Tsukuba from the ever helpful secretaries of the ICYS office to the barflies of Corkheads Aussie bar. I am certain that I will not meet such a mix of people or fit in so well again.

So what is next for me? Well the project did not go down too badly. So (fingers crossed) I should pass the degree and hopefully go for further study. Will Tsukuba ever see the likes of me again? There should be a new batch of Irish students before this year is out and you never know, with an even more unbelievable CV I may just possibly be back.

Joseph Govan (Trinity College, University of Dublin, Ireland)
September, 2006 - December, 2006 (NIMS Internship Program)
Fuel Cell Materials Center, Organic Nanomaterials Center &
International Center for Young Scientists (ICYS)

[Myself (left), the other interns and Prof. Bando on the last day]

[Myself (second on right) and the rest at the ICYS Kendo class]

NIMS News

Report on "Prof. Sir Kroto's Science Class in Tsukuba" at NIMS

Prof. Kroto with participants.

(December 9, NIMS) -- An event called "Prof. Sir Kroto's Science Class in Tsukuba" was held in Sengen Site for elementary school students in grades 1-4 and their guardians. A total of 95 children and 74 guardians attended.

The International Center for Young Scientists (ICYS) organized this science education program, which was conducted by Professor Sir Harold Kroto (Professor at Florida State University), who is a Nobel prize winner in Chemistry (1996) and an Executive Advisor to the ICYS. Prof. Kroto has held science classes for children in many of the world's countries. As part of these activities, this program was held so that children in the Tsukuba area could understand the interest and wonder of science. In spite of inclement weather on the day of the event, a large number of children and their parents participated. After listening to Prof. Kroto's lecture with keen attention, the participants completed a soccer ball-shaped "Bucky ball" (fullerene model) while enjoying friendly direct contact with the professor. This lively and pleasant program concluded after a Q&A session with some questions that seemed to stump even Prof. Kroto for a moment.

For more details: http://www.nims.go.jp/icys/event/Kroto2006/

NIMS National Institute for Materials Science

PUBLISHER
Dr. Hisao Kanda

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