NIMS NOW International

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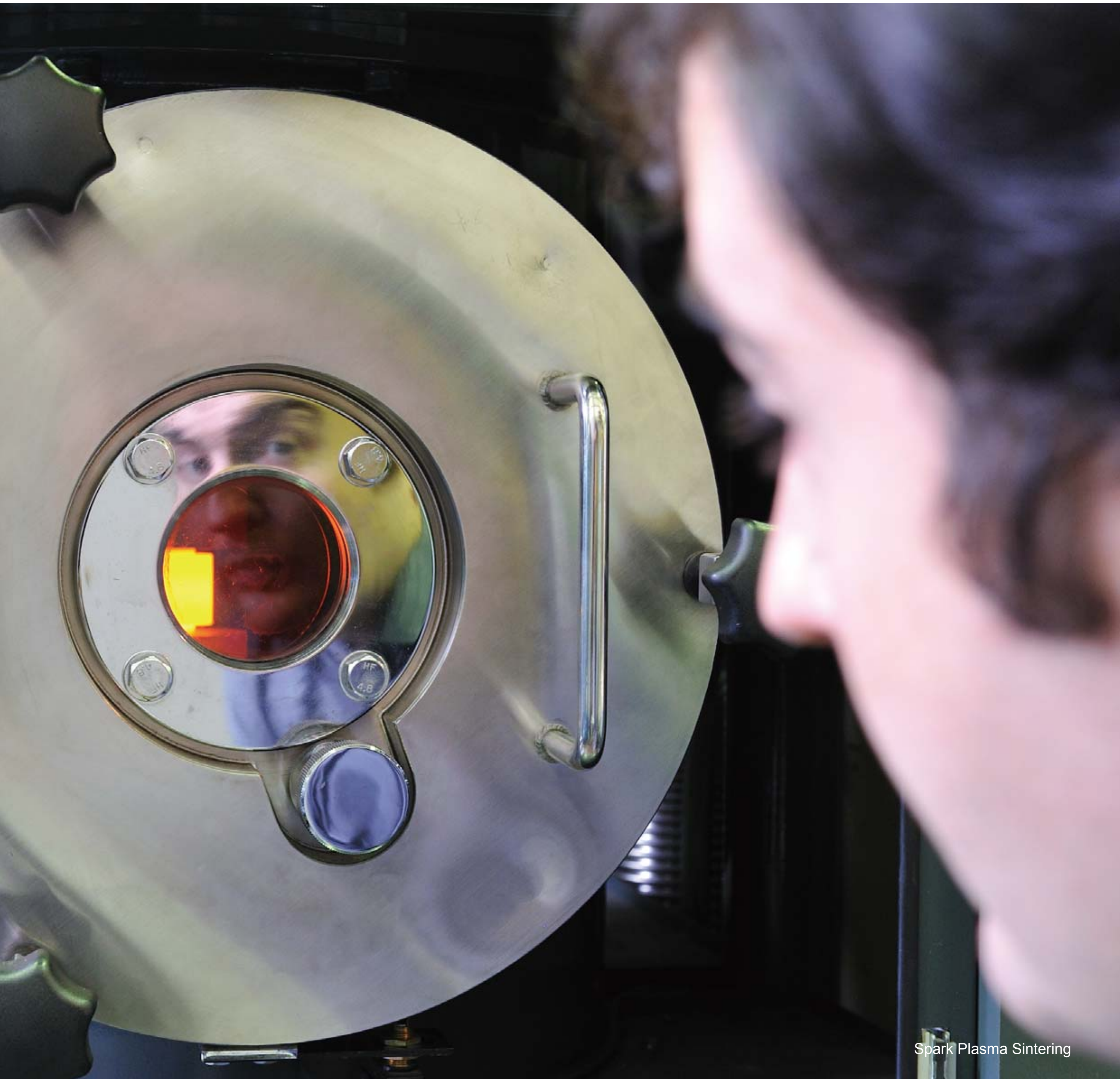
Innovative Ceramics



Spark Plasma Sintering

Innovative Ceramics

Today's ceramics are becoming increasingly multi-functional materials, but the potential of ceramics as materials is still unknown. Applications are expanding into areas which would have been unimaginable only a few years ago, with the commercial application of SiAlON phosphors to LEDs at the top of the list. The Nano Ceramics Center takes full advantage of the outstanding process development and characterization technologies of NIMS as it continues to shine new light on ceramics.

Aiming at radically advanced and multi-functional ceramics.

Yoshio Sakka
Managing Director, Nano Ceramics Center

In recent years, the development of new devices supporting advanced industries such as the semiconductor/IT industries, environmental industries, nuclear power, aerospace, and others, and development of equipment, higher efficiency, and reduction of environmental loads have been strongly required.

The aims of the NIMS Nano Ceramics Center are to radically improve the fundamental properties of ceramic materials, such as optical and magnetic functions, heat resistance, and high strength, and to create multi-functional "innovative ceramics" by intentionally superimposing or refining those properties.

In addition to pursuing various nanoparticle processes and developing advanced versions of those processes, the Center is integrating all steps from the construction of guidelines for nanostructural design based on the mechanisms responsible for the manifestation of functions to the synthesis/evaluation of new functional materials, and is achieving new development through mutual collaboration.

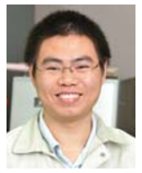
The key element technologies for realizing this are (1) synthesis of nanoparticles with homogeneous composition and controlled crystal size, (2) nanoparticle arrangement with uniform particle size, integration, and dispersion control, and fabrication of ceramics with regular pore structures and utilization of that space, (3) high order structural control from the micrometer to the nanometer order, and (4) nanostructure design based on theoretical/experimental study of the relationship between local structures and the manifestation of object functions.

In particular, applying electrical, magnetic, electromagnetic, stress, and other types of external stimulation to the reaction field is effective for realizing advanced nanoparticle processes. In all cases, leading research on individual processes is carried out at NIMS, and as a result, NIMS boasts a high potential in all areas. Concrete element technologies include a technique for creation of nanoparticles using reactive thermal plasma, a technique for production of high purity non-oxide nanoparticles using precursors, a technique for production of high-performance non-oxide nanoparticles by a vapor phase process, advanced sintering techniques such as spark plasma sintering (SPS) and others (→ p. 6), a technique for textured development of feeble magnetic ceramics using a strong magnetic field (→ p. 4), film-forming by electrophoresis deposition (→ p. 8), a technique for fabrication of lamellar ceramics, and a technique for fabrication of regular porous ceramics by anodic oxidation (→ p. 9).

This Special Feature introduces recent achievements of the Nano Ceramics Center. We hope that the publication of these results will also lead to many collaborative projects and joint research.

Yoshio Sakka Ph.D, Joined the National Research Institute for Metal (NRIM; predecessor of NIMS) in 1983, and current position from 2006. Professor at University of Tsukuba. Principal Investigator at the International Center for Materials Nanoarchitectonics (MANA).

Bio-inspired Machinable Nanolayered Ceramic:
the Unprecedented Fracture Toughness and Strength



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Inspiration from nature.

It is rather difficult to believe that someone can produce ceramic materials with the bending strength of 1200 MPa, fracture toughness as high as 18 MPa•m$^{1/2}$ and machinability comparable to that of steel.

In fact, to date, the strength and toughness of inorganic ceramics are very difficult to enhance simultaneously. Usually, these two factors show an opposite tendency. The intrinsic brittleness of ceramics also limits their wide application.

The twin quests for reinforcement and ductility of ceramics have pushed many scientists to take inspiration from nature. Several millions years of natural selection and evolution permitted natural plants and animals to develop optimized microstructures survive in severe environment conditions. The nacre shell microstructure consists of submicrometer scale layered grains with the plates aligned orderly[1]. Thus, the microstructure configuration justifies its excellent tensile, compressive and shear behaviors.

As in the case of the nacre shell, in order to simultaneously increase the fracture toughness and the bending stress, it is indispensable that the microstructure exibits[2]:

-weak grain boundary interfaces.
-large and elongated grains.

Making nacre like microstructure.

Recently, nanostuctured artificial nacre was reproduced by sequential deposition of polyelectrolytes and clays[3]. The bulk hybrid ceramic-based materials increased by four times the strength, while the fracture toughness was comparable to those of aluminum alloys. Unfortunately, the introduction of organic materials prohibits employment at high temperatures.

MAX phases in which M is transition metal, A is A group element, X is C or N, and *n* is 1-3, because of their nanolayered structure, are ideal candidates to reproduce nacre's microstructure. Among the MAX phases, Nb_4AlC_3 has been selected for investigation. As shown in **Fig. 1**, the weak bonding between Al atom layers with Nb and C atom layers contributes to the easy dislocation formation and their slipping which induce the development of kink bands in the grains presenting the "quasi-plastic" behavior.

Up to date, the strong magnetic field technique is the only available method to align nanolayered Nb_4AlC_3 particles. The strong magnetic field alignment technique[4] permitted to obtain highly *c*-axis oriented Nb_4AlC_3 grains, while the high temperature annealing generated plate-like grains. The nacre shell-like microstructure with the layer stacking from nano-scale to milli-scale was obtained.

Increasing fracture toughness and strength.

Fig. 2a-c show the mechanical properties and toughening mechanisms of textured Nb_4AlC_3 ceramic. It is seen that the bending strength of samples are as high as 1184.9 ± 283.3 and 1219.2 ± 108.6 MPa when tested parallel and perpendicular to *c*-axis (**Fig. 2a**). Also, as shown in **Fig. 2b**, the fracture toughness when tested parallel and perpendicular to c-axis are as high as 17.9 ± 5.16 and 11.49 ± 1.38 MPa•m$^{1/2}$ respectively. In comparison with those of untextured Nb_4AlC_3 ceramic, the bending strength and fracture toughness have been increased by factors of 3.5 and 2.5, respectively.

Undoubtedly, the microstructure design justifies the mechanical responses. The single-edge notched bending (SENB) samples tested along the *c*-axis direction present the zigzag fracture mode. The zigzag fracture surface corresponds to high surface energy transformed from the mechanical energy. Additionally, the investigation of the microcosmic zigzag fracture surface revealed pull-out grains distributed on the whole surface (**Fig. 2c**), which means that the toughening mechanisms possibly correspond to crack deflection which increases the surface energy and crack bridging which lowers the stress intensity factor at the crack tip.

Furthermore, the toughening mechanisms were evaluated by the precracking method. After testing, the sample still keeps the initial shape without complete fracture. The crack emanates from the notch and is prone to propagate along the symmetrical direction owing to the weak interfaces in the grains and at the grain boundaries in the textured ceramic. The crack branches can effectively absorb the mechanical energy for inhibiting the fast, catastrophic and straight crack propagation. The SEM micrograph in **Fig. 3** clearly shows the toughening mechanisms due to crack deflection, grain pull-out and bridging. The plenty of weak interfaces promote the crack deflection[5]. This result is ascribed to the orderly alignment of Nb_4AlC_3 plate-like grains.

Development of shell-like ceramics with high performance.

Fig. 4 shows the diagram of the flexural strength and fracture toughness of textured Nb_4AlC_3 ceramic in comparison with those of other advanced ceramics. It is observed that both the strength and toughness of textured Nb_4AlC_3 ceramic are the highest. It means that the present tailored ceramic possesses unprecedented mechanical properties. The strong magnetic field alignment technique method successfully permitted the design of optimized microstructure with enhancement of both the strength and toughness. Undoubtedly, the as-prepared texture ceramic is a good candidate to be applied in the structural fields. Additionally, its applicability can be extended to high temperature fields. In fact, Nb_4AlC_3 ceramic retains ambient bending strength up to 1400°C without any degradation. In addition, it is extremely significant that the easy machinability of Nb_4AlC_3 is its intrinsic character for the shaping of complex structural parts.

Our fundamental investigation will guide the future development and design of shell-like ceramics with high performance.

Chunfeng Hu, Ph.D, Joined MANA in the Nano-Materials Field starting from 2008.

Yoshio Sakka, (Profile is on P3)

Salvatore Grasso, Ph.D graduate student at Doctoral Program in Materials Science and Engineering, Graduate School of Pure and Applied Sciences, University of Tsukuba.

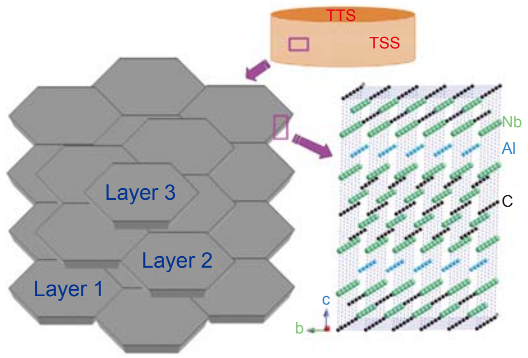
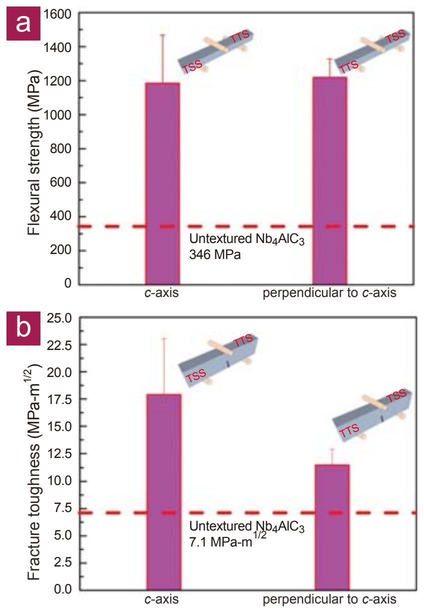


Fig.1 Microstructure of textured Nb_4AlC_3 ceramic.
Schematic map of tailored nanolayered Nb_4AlC_3 ceramic, showing the orderly stacking of grains whose *c*-axes are perpendicular to the textured top surface (TTS).



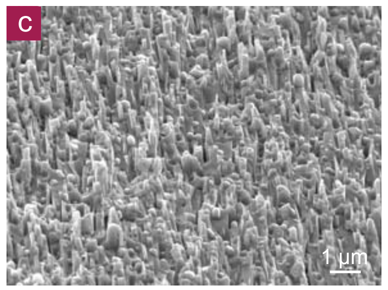


Fig.2 Mechanical properties and toughening mechanisms of textured Nb_4AlC_3 ceramic.
a and **b**, bending strength and fracture toughness of tailored Nb_4AlC_3 ceramic tested on the different directions in comparison with those of untextured Nb_4AlC_3 ceramic, indicating the marked enhancement of both strength and toughness. **c**, scanning electron microscope (SEM) image of pull-out grains on the fracture surface, indicating the possible toughening mechanisms of crack deflection and grain bridging.

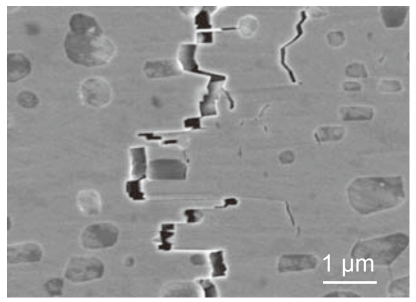


Fig.3 *In situ* propagating crack induced by the precracking method.
The zigzag propagation of the crack shows the typical crack deflection, grain pull-out and crack bridging features.

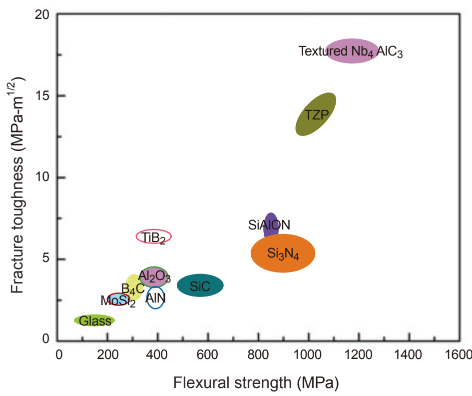


Fig.4 Flexural strength and fracture toughness of textured Nb_4AlC_3 ceramic compared to those of other advanced ceramics.
The tailored Nb_4AlC_3 ceramic possesses the highest bending strength and fracture toughness.

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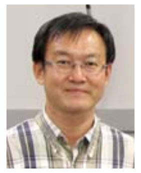
5) Becher, P. F. Microstructural design of toughened ceramics. J. Am. Ceram. Soc. 74, 255-269 (1991).

A New Spark Plasma Sintering Method for Synthesising of Ultra-Fine-Grained Transparent Ceramics

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Spark plasma sintering method newly developed at NIMS.

It is possible to obtain high strength and transparency simultaneously, when we sinter polycrystalline ceramics to a high density with maintaining a fine grain size. Existing models predict that transparency equivalent to that of single crystals is possible, when the volume fraction of pores is reduced to 0.01% for a grain size of smaller than 100nm.

Many useful functions can be expected in such transparent ceramics: strength, hardness, thermal conductivity, refractive index, heat resistance and plasma resistance higher than those of glasses and resins. In addition, transparent ceramics are superior to single crystals in isotropy and homogenous properties, formability and mass-productivity. Other optical properties such as laser emission may also be expected by nanometer-sized structural and compositional design. In order to obtain new functions in nanometer-sized transparent ceramics, NIMS has been trying to develop a new method using a spark-plasma sintering (SPS) technique. The method, for which the heating processes are different to those of conventional one, has enabled us to synthesize ultra-fine grained and high-strength transparent ceramic materials.

Synthesis of ultra-fine-grained and high-strength transparent ceramics.

The SPS method is known as a technique for enhancing densification processes by utilizing the spark discharge among particles and Joule heating that are exerted by a pulse current applied to the compact of the raw material through the graphite die (**Fig. 1**). Rapid heating is a characteristic of this technique.

We have found, however, that controlling the heating rate noticeably lower than usual SPS technique leads to full densification under strongly suppressed grain growth. We also found that this method enables the full densification at a temperature by 200-500 °C or more lower than that of usual SPS technique. The full densification at lower sintering temperatures results in highly fine-grained transparent ceramics (**Fig. 2**).

Applying our method to high purity Al_2O_3 and $MgAl_2O_4$, we have obtained high-strength transparent materials: the strength reached the highest level or two times higher than that obtained by other techniques with an optical inline transmission of 45-70%, the value of which was first achieved by SPS. For densification of undoped Y_2O_3, a sintering temperature higher than 1400°C is necessary for conventional SPS, and this technique has not been successful for obtaining transparency nor translucency. The present new method, however, has first attained translucency and fine-grain size (<200nm) at a very low sintering temperature of 950°C. We have also found that the sintering temperature of the present technique can be further lowered by doping a trace amount of adequate cation to the grain boundaries.

Our research includes theoretical and experimental analyses on sintering phenomena and relating basic mechanisms. We have placed special attention on the relationship between the grain size or porosity and the optical scattering or absorption that relates closely to translucency or transparency, the relationship among the local state of chemical bonding at grain boundaries, diffusion and the low temperature sintering described above and the relationship between nanostructures appearing during sintering and densification mechanisms. Basic information newly obtained from the analyses has provided potential guidelines for new synthesizing methods and for obtaining new functions in nanocrystalline ceramic materials.

Keijiro Hiraga (center left), Dr. Eng. Completed degree at the Graduate School of Engineering, Tohoku University. Joined NRIM in 1978. Visiting Scientist at the Max-Planck Institute in 1990-1991. Director of the Fine-grained Refractory Materials Center since 2002. Visiting Professor at the Graduate School of Chemistry, Hokkaido University since 2008.

Byung-Nam Kim (left), Dr. Eng. Completed degree at the Graduate School of Engineering, the University of Tokyo. Research Associate in the Faculty of Engineering, Tokyo Metropolitan University and the Research Center for Advanced Science and Technology, University of Tokyo. Joined NRIM in 1998. Visiting Scholar at the University of Pennsylvania 2003-2004. Has held present position since 2006.

Koji Morita (center, right) Dr. Eng. Completed degree at the Interdisciplinary Graduate School of Engineering Sciences, Kyushu University. Research Fellow of Japan Society for the Promotion of Science (JSPS). Joined NRIM in 1997. Present position since 2008. Currently Visiting Scholar at the Technischen Universität Darmstadt (Germany).

Hidehiro Yoshida, Dr. Eng. Completed degree at the Graduate School of Engineering, the University of Tokyo. Prior to joining NIMS, was a JSPS Research Fellow and Research Associate at the Graduate School of Frontier Sciences, University of Tokyo. Joined NIMS in 2004 and has held present position since 2008.

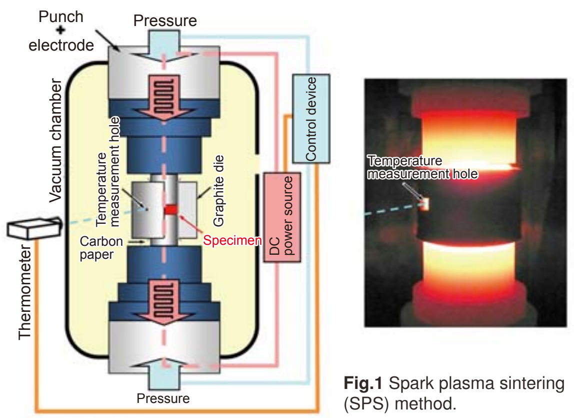

Fig.1 Spark plasma sintering (SPS) method.

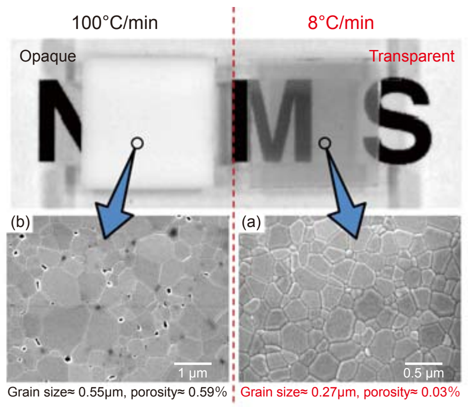

Fig.2 Comparison of the conventional SPS (left) and the low-rate-heating SPS (right) newly developed in this study. High-purity undoped Al_2O_3 was sintered at 1150°C for 20min.

Current Status of SiAlON Phosphors

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In recent years, white LEDs have been actively adopted in the fields of lighting and displays as a response to energy saving needs. In lighting, light-bulb types LED lighting is used as a substitute for conventional incandescent lights, and in displays, liquid crystal displays using LED backlights are the main stream. The white LED is a light emitting device comprising a blue LED tube and a phosphor. The phosphor is a key material which determines the spectrum of the LED. Durability and a color developing property are required in phosphors for LED applications. SiAlON phosphors are NIMS-developed materials which meet these requirements.

The material SiAlON is a crystal consisting of the elements Si, Al, O, and N. As a ceramic, SiAlON was an object of research as a heat-resistant material in the past. The Nitride Particle Group proposed phospors based on a new concept, using these crystals as the host, and has synthesized phosphors with high light emitting efficiency and excellent stability. In comparison with the conventional oxide phosphors, SiAlON phosphors have the distinctive features of (1) excellent durability, (2) favorable temperature properties, (3) visible light excitation, and (4) large degree of freedom in material design SiAlON materials are continuing to become the world standard as phosphors for white LED applications in use under severe conditions.

The phosphors developed by NIMS include CASN ($CaAlSiN_3$; red), α-SiAlON (yellow), β-SiAlON (green), aluminum nitride (blue), and others (**Fig. 1**). These materials are mass-produced and marketed by chemical makers and are used practically in a variety of industrial products.

Conventional white LEDs actually emit bluish-white light, and it was difficult to obtain warm light like that produced by incandescent light bulbs. Lighting with the color of conventional light bulbs was made possible by increasing the red-light component, which was achieved by adding CASN to this type of LED. Because white LEDs using CASN and β-SiAlON produce light consisting of the three primary colors of light, red, green, and blue, color reproducibility is improved if this type of LED is used in liquid crystal backlights (**Fig. 2**). Therefore, television makers are increasing the percentage of products using LED backlights in place of the conventional cold cathode fluorescent lamp (CCFL) technology, and it is generally thought that LEDs will be used in all large-scale liquid crystal panels within several years.

In research on phosphors, the search for substances which can serve as new host crystals is important, but this is still a largely unexplored field. Several years in the future, it is expected that a large number of new phosphors will have been discovered and will be advancing toward practical application. NIMS intends to continue to be a leader in research on new phosphors. To maintain our position at the top of this field, we will search for new substances, develop processes, elucidate light-emitting properties, and propose materials of a level that can be used in industry.

Naoto Hirosaki, Dr. Eng. Joined Nissan Motor Co. in 1980 and the National Institute for Research in Inorganic Metals in 1998. Currently Group Leader of the Nitride Particle Group and Non-Oxide Ceramics Group. Professor in the cooperative field of the Department of Frontier Materials, Graduate School of Engineering, Nagoya Institute of Technology.

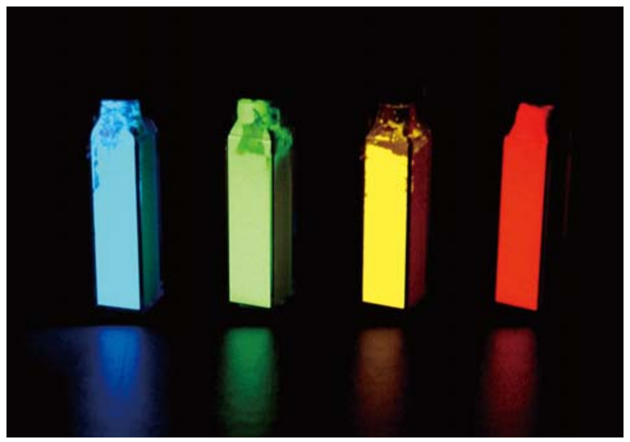

Fig.1 Phosphors developed by NIMS.

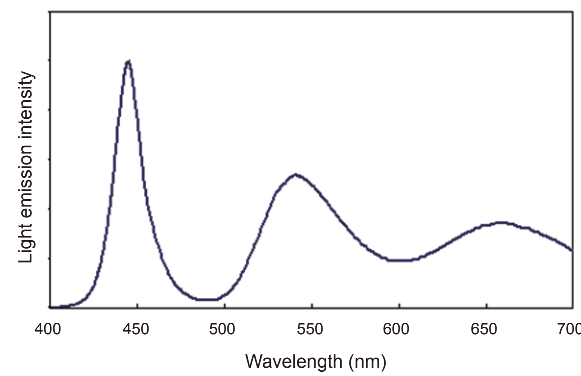

Fig.2 LED for use as liquid crystal backlight comprising the three primary colors of light (red, green, blue).

Ceramic Forming Process Using Electrophoresis of Colloidal Particles

Tetsuo Uchikoshi
Fine Particle Processing Group,
Nano Ceramics Center

A ceramic colloid process capable of producing complex shapes.

The state in which particles with diameters on the order of 10^{-7} to 10^{-9}m are dispersed in a gas or liquid is called a colloidal system. Because colloidal particles are larger than solute molecules and ions and always have an electrical charge, they have distinctive features, including Brownian motion, Tyndall scattering, dialysis, electrophoresis, etc., and exhibit phenomena such as coagulation and salting out.

Submicron to nano-sized ceramic particles dispersed in liquids such as water and non-aqueous solutions display precisely the same properties as colloidal particles. Thus, it is possible to obtain dense solidified compacts by promoting aggregation of charged dispersed ceramic particles by an appropriate method. This forming method is called the colloidal process and is used as a method of manufacturing ceramics with complex shapes.

Features of ceramic film-forming by electrophoretic deposition.

We are engaged in research on processes which apply the phenomenon of electrophoresis of charged colloidal particles in liquids as a forming technique for ceramic films. Charged particles in a liquid display the behavior which depends on the potential gradient of the electrical field. When an electric field is applied to a suspension of ceramic particles by inserting + and – electrodes into the suspension, the particles in the space filled with the liquid are electrophoresed and deposited on the electrode surfaces.

Because the particles do not follow the straight line representing the shortest distance between the electrodes, but rather, migrate along the electric field lines, uniform coating on a curved or irregular substrate surface and on a patterned electrode are possible with a little ingenuity in the arrangement of the electrodes. The particle layer deposited by this method has a density equal to or greater than that consolidated by cold isostatic pressing (CIP) at 200MPa, and densification is possible at lower temperatures. Fabrication of laminates with a controlled film thickness is also easy.

Expanding applications of the electrophoretic process.

The electrophoretic deposition process is increasingly applied to fields where application had been difficult in the past. This is possible as a result of the development of techniques for the preparation of suspensions suitable for the electrophoretic deposition process, deposition of particles on nonconductive substrates, suppression of electrolysis of aqueous solutions by applying DC pulse current, among other advances. Recently, oriented ceramic films and oriented laminated composites, crystal orientation in which is controlled, have been fabricated successfully by combining a magnetic orientation technique using a high magnetic field and the electrophoretic deposition process.

We are engaged in research aimed at achieving high functions and diversity in various ceramic materials by manipulating colloidal particles in liquids under electrical and magnetic fields in order to control the microstructure of the formed compacts, including environment and energy-related materials such as anode-supported fuel cells, photoelectrodes for dye-sensitized solar cells, photocatalyst films, and thermoelectric modules, medical materials such as dental crown materials, and structural materials such as ceramic veneer materials, among others.

Tetsuo Uchikoshi, Dr. Eng. Joined NRIM in 1987. Visiting Scientist at McMaster University (Canada) 1997-1998. Present position (Chief Researcher) since April 2007. His specialty is the physical chemistry of ceramic powders.

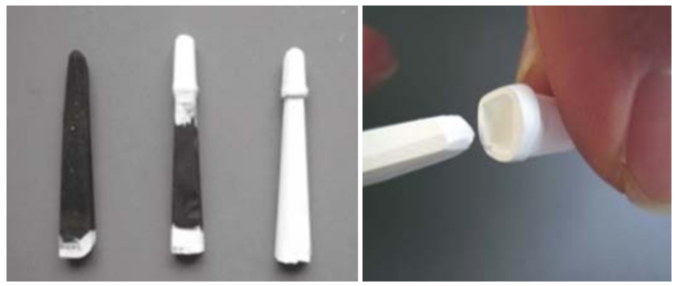

Fig.1 Example in direct shaping of ceramics by electrophoretic deposition using conductive polymer-coated non-conductive ceramic substrates. This technique can be utilized for fabricating various shape-controlled ceramics, such as dental crown.

Fig.2 Multicolor light emitting devises prepared by laminating yellow green and red SiAlON phosphors. The electrophoretic deposition process enables easy emission color tuning by controlling the thickness of each phosphor layer.

Porous Ceramics Applicable to Fabrication of Diverse Nanostructures

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Group Leader,
Functional Glass Group,
Nano Ceramics Center

Hiroyo Segawa
Functional Glass Group,
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Anodic oxidation alumina enables fabrication of diverse structures.

Anodic oxidation alumina is one type of porous ceramic containing oriented nanometer-sized pores. Due to the interactions which occur during the reaction process, a regular porous structure is formed without using a die. This anodic oxidation technique, which now has a history of more than 40 years, has been developed and improved as an industrial mass production technology for improving the chemical resistance of aluminum (alumite) and manufacturing colored aluminum sashes, etc.

The Functional Glass Group of the NIMS Nano Ceramics Center is expanding this anodic oxidation technology to a technique for forming films on the surface of glass, and is also engaged in research to realize advanced technology for the fabrication of more diverse nanostructures.

The pore sizes which can be produced span a wide range, from several nm to 1000nm. Pore shapes from the ordinary cylindrical type to conical and barrel shapes, etc. are also possible, and compounds can be introduced into the pores by the sol-gel method or electrodeposition, enabling use of the pores as nanometer-sized molds.

It is possible to obtain a structure (array) of standing rods or tubes of nanometer size on a glass surface by dissolving the mold, utilizing the difference in the solubility of anodic oxidation alumina and the compound introduced into the pores. Expected applications include photocatalysts in which a TiO_2 film is added to the inner wall of pores and high density magnetic recording media containing embedded magnets.

Aiming at application to industrial production processes.

Fig.1 shows scanning electron microscope images of Ni nano-cone arrays with various aspect ratios (ratio of height to diameter) fabricated using the anodic oxidation alumina mold technique described above. Because a large aspect ratio can be obtained, application to non-reflective films, sensors, etc. is expected. A structure of aligned vertical pores is obtained by applying a DC voltage, whereas a completely different layered structure is formed if an AC current is applied.

Next, **Fig. 2** shows scanning electron microscope images of examples of layered structures fabricated using various acid solutions. This structure resembles the structure of bivalve shellfish. In these shellfish, a stronger structure is formed by division of the shell material into cells within the layers. At present, we are investigating techniques for fabricating more complex nanostructures similar to the spiral shells of conches, methods of controlling the layer thickness and introducing compounds between layers, and related topics. Lamellar anodic oxidation films are considered applicable to sensors and optical devices.

The development of a composite control method for anodic oxidation technology employing DC and AC voltages and techniques for fabricating various porous materials and nanostructures with higher accuracy are also expected. Moreover, because anodic oxidation technology is an industrial mass production technology, it is thought that the technology developed in the present work will be useful as an industrial production technology for nanomaterials.

Satoru Inoue, Dr. Eng. Completed the master's course in the Department of Chemical Engineering, Graduate School of Engineering, Tokyo Institute of Technology. Research Associate and Associate Professor at Tokyo Institute of Technology. Prior to appointment to present position in 2006, was Senior Researcher at the National Institute for Research in Inorganic Metals (NIRIM; predecessor of NIMS) and Group Director in NIMS.

Hiroyo Segawa, Dr. Eng. Completed the doctoral course in the Department of Inorganic Materials, Graduate School of Engineering, Tokyo Institute of Technology. Assistant professor at Oita University, Japan Science and Technology Agency (JST) Fellow, and Assistant Professor, Graduate School of Engineering, Tokyo Institute of Technology. Appointed to present position at NIMS in 2009.

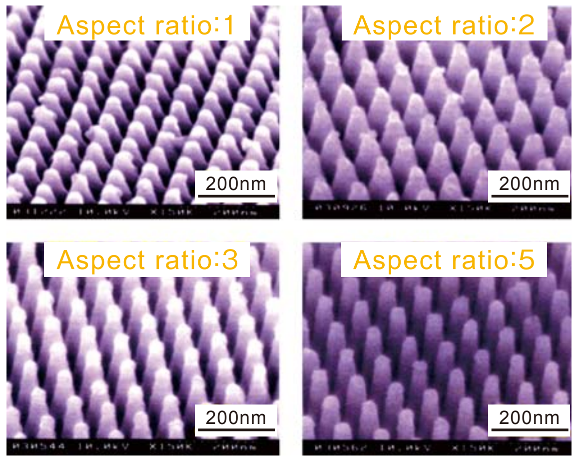

Fig.1 Scanning electron microscope images of Ni nano-cone arrays fabricated using various anodic oxidation pore arrays as molds.

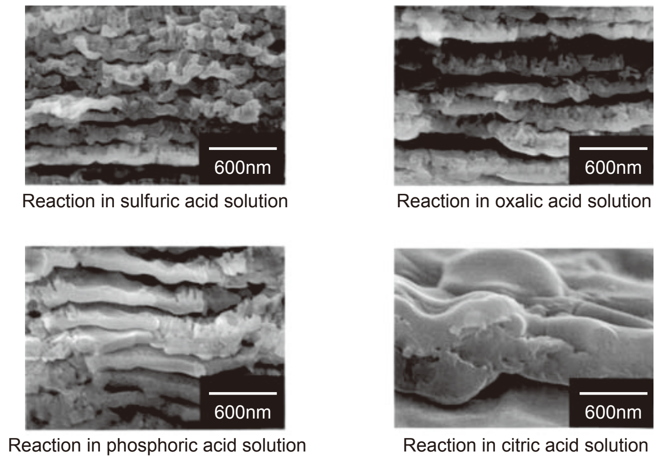

Fig.2 Scanning Electron microscope images of lamellar anodic oxidation alumina fabricated by anodic oxidation applying an alternating current in aqueous solutions of various acids.

Aiming at Development of Multi-Functional Ceramics by Open Research

Yoshio Sakka Managing Director, Nano Ceramics Center

In its R&D activities, the Nano Ceramics Center gives high priority to an open, innovative environment which works in collaboration with private companies. Dr. Yoshio Sakka, the Center's Managing Director, discusses the fascination of multi-functional nano-ceramics and the future goals of the Center.

—— What kinds of research lead to more advanced nano-ceramic materials?

We carry out research from the standpoint that nano-ceramics are created from powders. A good powder consists of nanoparticles which are fine, homogeneous in size, and have a special structure. What is important is not only simply improving the properties of the powder. In materials, the aim is to produce bulk materials and films. Therefore, it is essential to form a dense, pore-free structure when in the compaction process by applying heating. Although we have adopted a method of dispersing powders in solutions, our researchers have also developed a technique for forming an ordered pore structure and introducing atoms into those pores. Our work is not limited to the conventional process, but also involves the use of an electrical and magnetic fields. But it isn't enough just to purchase equipment for this purpose. The researcher's own ingenuity and improvements are also key factors. This kind of skill is extremely important.

—— How did you develop an interest in ceramics?

I'd been engaged in research on the diffusion of oxides since my student days, so it was necessary to produce high purity, pore-free sintered compacts. Since these efforts weren't very successful with commercial powders, I observed powders with a scanning electron microscope and found that the cause was the large size of the particles. Based on this, I began making fine high purity powders. At the time, there was a researcher who was interested in nacre, which has a lamellar structure of calcium carbonate and protein. This structure shows both excellent strength and toughness, and can be formed at room temperature. Creation of a structure like that of seashells became the ideal. I also made a special effort to study with this professor. Although this topic is also discussed elsewhere in this Special Feature, this is one example in which learning from the structures of living beings provided an approach for research.

—— Can you describe your goals for the NIMS Nano Ceramics Center?

The basic goal is process development, but we are also developing innovative ceramics using those processes. In other words, our first goal is to further improve the top data, but our second goal is to create materials which have two or more special properties by careful control of the microstructure. For example, a material with a combination of strength and transparency, and also adding thermal conductivity to those properties . . . Of course, there are also cases where two properties are mutually contradictory. Therefore, it is extremely important to clarify the relationship between microstructure and properties. For this, computational science and characterization techniques are necessary.

—— Your final aim is practical application, I suppose.

Once research has advanced to a certain level, it should be carried out with a private company. This is because we cannot reach the final stage of practical application by ourselves. In that sense, we very much welcome people from general companies. From the viewpoint of private companies, NIMS may seem to have a high threshold, but today, we are actively working to create an atmosphere in which it is possible to do research together. If we can open up our research in this way, we may be able to conceive completely unexpected applications. If the country that applies this development is Japan, we'll be extremely happy, but since the results should be finally for the entire world, we hope that every country will accept them.

—— By the way, what are your hobbies?

I don't have any particular hobbies, but I enjoy reading books in various fields. When I was younger, my quota was to read one book a day. I was strongly impressed by the fact that there was somebody in Japan like Nanami Shiono, who wrote *Stories of the Romans* [a 15-volume history that was completed in 2006]. We have many writers who would enjoy a higher international reputation if they weren't Japanese.

—— What are your impressions of young researchers? NIMS also has a large number of foreign researchers.

Outstanding Japanese researchers make steady progress in their research, even if left to their own devices, and there's no problem at all in this regard. But people who aren't like that tend to be somewhat conservative. If they aren't told what to do, they won't do anything at all. This group of young Japanese has been nicknamed the "waiting-for-instructions tribe." In contrast, almost of the non-Japanese researchers seem to be aggressive, perhaps because they have a sense of crisis. One example is the problem of doctorates. In Korea and the United States, you can't expect to become a research leader without a doctorate, but in Japan, a doctorate doesn't make that much difference, and salaries are also basically the same. Moreover, since the number of research posts in Japan has decreased significantly, only some excellent researchers can find such positions.

The fact that technical personnel are not treated with importance is another problem. In recent years, those people have been active as technical leaders in Korea and China. Japan is currently No. 1 in materials-related fields like ceramics, but there's no knowing what the situation will be like 10 years from now. As a last line of defense, I think the government will have to create research bases, and provide equipment and train people who can do research.

Yoshio Sakka (Profile is on P3)

FACE Interview

Expanding the Possibilities of Ceramics

Naoto Hirosaki Nitride Particle Group, Nano Ceramics Center

SiAlON phospors are used in white LEDs, and the NIMS Nitride Particle Group is a leading force in the development of these essential materials. Group Leader Naoto Hirosaki continues to be deeply involved in research on ceramics, and is also constantly aware of the need to transfer technologies to industry. Dr. Hirosaki discusses his work in this FACE Interview.

—— First, how did you become interested in research related to LEDs?

Originally I had no interest whatsoever in LEDs. My specialty was SiAlON, and I had devoted many years to research on that material. SiAlON was the material for key components in engines and high temperature materials, and appeared to have absolutely no relation to LEDs. I myself, and the laboratory to which I belong, had been involved in research on heat-resistant materials from the days of the National Institute for Research in Inorganic Metals (NIRIM), which was one of the predecessor organizations of NIMS. As a result of this work, this material had reached actual application in engines and other fields. From around 2000, we began considering new applications from various angles. The idea of using SiAlON in LEDs was first proposed in this process. Thus, this work began as a new development of SiAlON, so I also began as a total newcomer to the field of LEDs.

—— When did you first become involved with ceramics?

I finished university and decided on my first job 30 years ago. At just the same time, there were moves to use ceramics in automobile engines. I thought this seemed interesting, so I joined a private-sector automobile company, where I was involved in research and development on ceramic engines.

—— What were your impressions of research and development in a private company?

Even though we say development, because it's a private company, the purpose is commercialization of products. As a result, even though the research itself went well, it couldn't be used by the company. Because I'd had that experience, I was happy that our work on LEDs could actually be used. In the case of structural materials, a total evaluation is necessary. This means the material won't be used if it can't satisfy a wide range of requirements, including heat resistance, strength properties, reliability, cost, and response in case of trouble. Price is particularly important. In that sense, metals are naturally rival materials, and even today, ceramics are only used in special applications.

—— So LEDs are different from engines.

Yes. LEDs don't have to satisfy a total evaluation, they only have to have one excellent property. If they can achieve the target of producing a certain color with a certain efficiency, that is sufficient. Of course, they also have to maintain that performance.

—— How was the idea of applying SiAlON to LEDs conceived?

Because there were no previous examples, we simply decided to give it a try. Phosphors are based on the phenomenon in which atoms such as europium and cerium included in a crystal emit light. Because we had also investigated materials containing rare earth elements in connection with SiAlON as a heat-resistant material, we thought that SiAlON might emit light if europium was added. At first, the light was dim, but we have now reached a point where quite wide use is possible. This is the result of convergent research by assembling a team of experts in various fields, including phosphors, synthesis, light-emitting devices, and others.

—— How do you see NIMS as a research institute?

As a research organization, it is an ideal environment with a large degree of freedom in research. Because that derives from NIRIM, I feel that the culture is unchanged. Accordingly, I'd like to tell young researchers to be self-assertive and do the research that they really want to do. It's alright if you don't pay too much attention to what other people think. Although I myself am always stimulated by the outstanding ideas that young people have because they're young (laughter).

Naoto Hirosaki (Profile is on P7)

NIMS Advisor Prof. Teruo Kishi Selected for Distinguished Life Membership by the ASM

The ASM (American Society of Materials International) has selected Prof. Teruo Kishi, who served as the first President of NIMS and is currently a NIMS Advisor, to receive a Distinguished Life Membership. The award ceremony was held in Pittsburg on October 29. In particular, this award recognizes Prof. Kishi's contributions in connection with the establishment of the Nanotech Network (Nanonet) and the World Materials Research Institute Forum (WMRIF), among other achievements. Prof. Kishi is the fourth Japanese selected for this honor in the past 50 years, following Soichiro Honda, Kazuo Inamori, etc. During the past year, Prof. Kishi also received the Japanese Society for Non-Destructive Inspection Award in June, was named an Ostwald Fellow by the Federal Institute for Materials Research and Testing (BAM; Berlin, Germany) in August, and received the Contribution Award of the International Conference on Fracture in October.

NIMS Signed a Comprehensive Collaborative Agreement with University College London of England

On October 28th, President Prof. Sukekatsu Ushioda signed a Comprehensive Collaborative Agreement (a sister institute agreement) with University College London, which was founded in 1826 as a radically different university, opened to people of all beliefs and social backgrounds. UCL is one of the world's leading multidisciplinary universities, ranked 4th in the QS Quacquarelli Symonds Ltd. World University Rankings (2009 and 2010), with more than 4,000 academic and research staff for 6,200 graduate and 11,400 undergraduate students. UCL has produced 21 Nobel prize laureates besides strong connections with world-famous scientists like C. R. Darwin, A. Graham Bell and Sir A. J. Fleming.

This agreement will not only reinforce the existing collaboration between UCL and NIMS in the field of computational materials science, organic nanomaterials and photocatalytic materials, but also envisage new collaboration and exchange of researchers.

The wilkins Building in UCL

Results of the 10th NIMS Forum

The 10th NIMS Forum was held on October 20th at the Tokyo International Forum convention center. As the 10th event in the series, this year's Forum also marked an important milestone. The content of the exhibition further promoted the theme of "Materials research responding social needs," which was a keyword, and was received a high evaluation from visitors.

Opening greetings by President Ushioda were followed by oral presentations on recent directions in research by the Managing Directors of various NIMS Centers, and introductions to the recent trends at the Innovative Center of Nanomaterials Science for Environment and Energy (ICNSEE) and International Center for Materials Nanoarchitectonics (MANA) and activities in connection with industry-IAI- collaboration.

The poster session featured 92 items in 13 categories, including "Next-generation fuel cells and power generating materials," "Environmental recycling/resource utilization," "Next-generation semiconductors," and others. A large number of important research achievements were introduced, attracting keen interest from many visitors. Notable topics included the development of permanent magnets which do not use the rare earth element dysprosium, the discovery that silver phosphate is a high-activity photocatalyst under visible light, and the fabrication of fundamental logic devices using graphene.


Greetings by President Ushioda


Enthusiastic visitors gathering at the poster session

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