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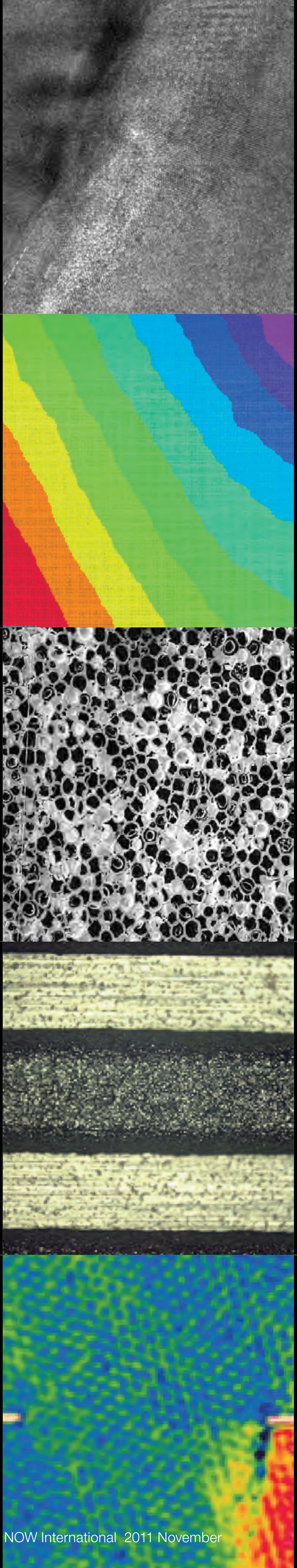
No.9
2011 NOVEMBER

A Toolbox for the Future

–Evolving Hybrid Materials



A Toolbox for the Future

–Evolving Hybrid Materials

Hybrid materials are combinations of multiple materials
in which the properties of those materials are retained and utilized.

The "Lightweight High-performance Hybrid Materials Project" at NIMS
strives to achieve an aim of realizing the "Hybrid Material Technology Tools".

Enhancing its usability as a versatile tool
by elucidating the various properties of hybrid materials,
not only with know-how alone, but with R&D achievements of NIMS.

Our technology tools will be indispensable for those users
who try to combine and create new materials and structures.

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The Necessity of Development Tools for Hybrid Materials and their Practical Application

NIMS Fellow
Unit Director, Hybrid Materials Unit, Environment and Energy Materials Division
Yutaka Kagawa

What are "hybrid materials"?

The expression "hybrid materials" is used as a general term for composite materials, porous materials, laminated materials, and other materials with similar features. Among these, our project focuses on hybrid materials which are used as structural materials.

As key features of the project, "Multiscale structures from the atomic level to the structure itself" and "Ingenuity in utilizing the properties and shapes of materials themselves" are incorporated in the materials research.

The basic technologies to create hybrid materials can be considered to comprise "Combinations of materials with different properties," "Interfaces created by the combination of different materials," and "Residual stress generated by the combination of materials," among others.

Methodology of deriving hybrid materials from property requirements = Construction of tools

Because a hybrid material is a single material which is produced from diverse materials, its properties are also diverse. Therefore, the objects of research and development will differ greatly, depending on the actual use environment.

For example, when considering application as an actual structural material, because the use environments for aircraft and automobiles are different, the combinations of materials used will naturally differ, and the properties required in parts where adhesion or joining is necessary will also be different.

Conversely, however, when the properties which are required in a material are specified, this leads to a methodology of determining "what one material can be produced by combining which materials in which ways." That methodology itself should not be controlled by the use environment.

In this project, we think of that methodology in terms of "technology tools." To give these tools the greatest possible generality, all technologies are constructed assuming materials which will be used in the lightweight moving bodies of the near future, which is the object of research and development.

Concretely, assuming that hybrid materials will be used in the lightweight moving bodies of the near future, we identify the problems and the key technologies for that case. Our aim is to construct technology tools which do not depend on the type of material by using the specializations of project members to solve those problems.

Research objectives and the "toolbox."

Therefore, members set themes considering 3 points in particular, these being (1) Technology which can be used independently regardless of the type of material, (2) Reflection of the opinions of hybrid material users, and (3) Inclusion of elements of research as basic and generic technologies.

As a result, the following should be mentioned as absolutely necessary issues when using hybrid materials: "Evaluation of mechanical properties of connections, adhesion, and interfaces between dissimilar material," "Evaluation of thermal properties, including interfaces," "Methods of reducing the coefficient of thermal expansion of materials utilizing shape and material combinations," and "Development of new materials considered useful in future hybrid materials, e.g., lightweight porous materials, etc." These were adopted as respective research topics. Furthermore, "Soft joining processes" and "Joining processes based on properties of living organisms" have also been incorporated in research and development as new adhesion and joining technologies for the future which are not simply extensions of existing technologies.

To propose the use of technologies which make the maximum use of the features of hybrid materials, the project will also carry out research based on multiscale dimensions and research with a wide time axis, extending as far as proposals of new concepts that break through the existing technologies and lead to the technologies of the near future.

As a distinctive feature of this project, unlike research on designated materials, the project does not target clearly-defined material properties. Instead, the objective is to create a "toolbox" which will enable engineers using hybrid materials to develop structural components more quickly.

The results obtained in this project are expected to be used widely as basic and generic technologies for materials.

Yutaka Kagawa Dr. Eng. Completed doctoral course at the Waseda University Graduate School of Science and Engineering in 1984. He is now a NIMS Fellow and the Unit Director of the Hybrid Materials Unit. He is also Professor of the Research Center for Advanced Science and Technology (RCAST) at the University of Tokyo.

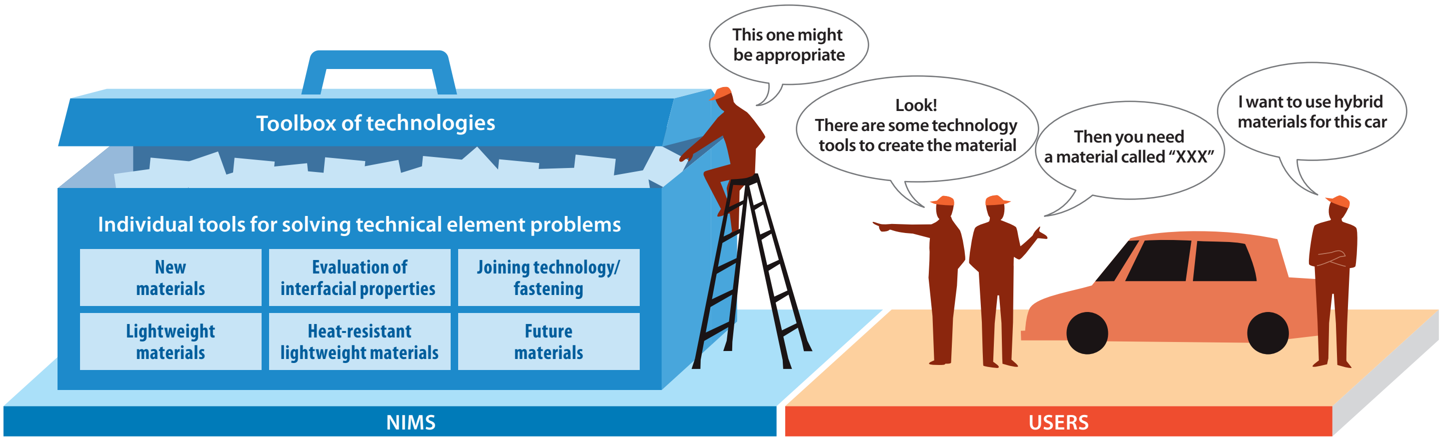


Figure Image of technology tools for hybrid materials and the NIMS "toolbox" concept

Tool for Evaluating Mechanical Properties of Hybrid Materials

Multiscale Characterization of Mechanical Properties

Composite Materials Group, Hybrid Materials Unit,
Environment and Energy Materials Division

Yoshihisa Tanaka

When external factors, i.e., heat and mechanical force, act on a material, there is a possibility that the material may fail due to microscopic concentration/accumulation of local strain, even if the deformation of the material is macroscopically uniform. For this reason, investigation of the linkage between microscopic strain concentration and macroscopic strain is important in reliability evaluation technologies. Until now, deformation measurement techniques have comprised contact methods such as a strain gage, displacement gage, etc., noncontact interferometry such as moire and speckle techniques, and a method of measuring nano-strain under the scanning electron microscope. Recently, a metal crystal strain measurement technology using electron backscattered diffraction (EBSD) and other advanced techniques have been proposed, but because each of these is applied to characterization at different specimen scales, integrated measurement of deformation and strain from the nanometer to the millimeter scale has not been possible. This article introduces a multiscale deformation/strain measurement technology for cases in which external forces act on materials with complex hierarchical structures. This technology was realized by developing a multiscale pattern combining patterns of different scales.

As one example of a material having a hierarchical structure of nano to millimeter scale, the following figure shows an example of multiscale deformation/strain measurement when a bending load acts on a laminated carbon fiber reinforced plastic (CFRP) hybrid material with different fiber orientations. This is a measurement of the macroscopic deformation behavior and local shear strain around carbon fibers having a diameter of 5μm when an in-situ 3-point bending test was performed in a field emission scanning electron microscope. The bending deformation behavior in the indenter, shear sliding deformation of the lamination interface, and local nano deformation and strain distribution in the microscopic region can be seen.

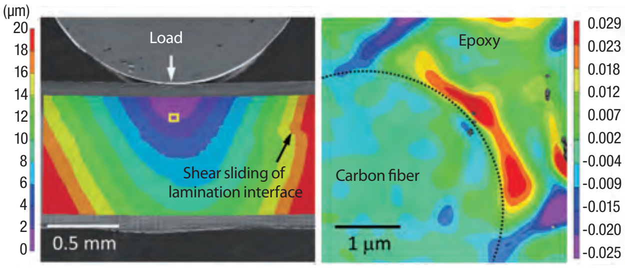


Figure (left) Macroscopic displacement distribution in a bending test of CFRP, and **(right)** shear strain distribution around the fiber in the region indicated by the square in the figure on the left.

This multiscale measurement technology can also be applied to measurements of thermal strain and residual stress, and is expected to contribute to reliability engineering, for example, in elucidation of the mechanism of damage initiation and evolution in materials with hierarchical structures and elucidation of the interfacial failure mechanisms in semiconductor devices, biomaterials, etc.

Yoshihisa Tanaka Dr. Eng. Completed doctoral course at the Graduate School of Engineering, University of Tokyo in 1999. Joined the National Research Institute for Metals (NRIM), a predecessor of NIMS, as a Researcher in 1989. He is now a NIMS Principal Researcher.

Tool for Evaluating Thermal Properties of Hybrid Materials

Measurement and Simulation of Interfacial Thermal Resistance

Composite Materials Group, Hybrid Materials Unit,
Environment and Energy Materials Division

Yibin Xu

Numerous interfaces exist in hybrid materials. When heat is transmitted through these interfaces, the phonons and electrons which carry heat are reflected or scattered at the interfaces, causing thermal resistance. As there is a possibility that this interfacial thermal resistance largely controls the thermal conductivity of the material as a whole, this is an important element in control of heat conduction characteristics. However, interfacial thermal resistance depends on a variety of factors, not just limited to the crystal structure and properties of the basic material, but also the method of joining at the interface, among others. For this reason, accurate estimation of the quantity of interfacial thermal resistance is difficult.

Therefore, we developed a frequency-domain thermoreflectance method, as illustrated schematically in Fig. 1, which enables high accuracy measurements of interfacial thermal resistance at thin film/thin film and thin film/substrate interfaces in order to obtain an accurate grasp of the quantity of interfacial thermal resistance and elucidate the material and structural elements which influence this property.

A temperature change of frequency ω in the thin film is generated using an alternating current or periodic oscillation laser, and that change passes from the thin film through the interface and propagates into the substrate. The temperature change at the thin film surface is detected by the change in the reflectivity of the probe laser. Because its amplitude and phase depend on the thermal conductivity and specific heat of the thin film and substrate as well as interfacial thermal resistance, the interfacial thermal resistance can be obtained if the thermal properties of the thin film and substrate are known.

Figure 2 shows the relationship between the interfacial thermal resistance between an Au thin film and single crystal sapphire (a-Al_2O_3) substrate and the particle size of the Au, as measured by this method. The straight line in the figure is the theoretical value of the Au/sapphire interfacial thermal resistance calculated using a phonon scattering model. When the Au particle size is large (200 nm), the measured and theoretical values of interfacial thermal resistance are in good agreement. However, when the Au particle size is smaller than the phonon mean free path (approximately 70 nm), interfacial thermal resistance increases as the particle size becomes smaller. The specimen with a particle size of 50 nm was the only specimen which was subjected to an annealing treatment and also the only specimen in which Au_2Al, which is a compound of Au and Al, could be detected by X-ray diffraction. From this result, it can be understood that interfacial thermal resistance is reduced by chemical bonding.

Because this measurement technique is applicable to various hybrid materials, it can be used as an effective technology tool for future hybrid materials. Application to heat-radiation design of electronic devices, LEDs, etc. is also expected.

Yibin Xu Dr. Eng., Ph.D., Informatics. Completed the doctoral course in Materials Science at the School of Engineering, Shanghai Institute of Ceramics, Chinese Academy of Sciences in 1994. Ph.D. Researcher at the National Industrial Research Institute of Nagoya in 1995, Researcher in the NIMS Materials Information Technology Station in 2002, and Principal Researcher of the same organization in 2007. Present position since 2011.

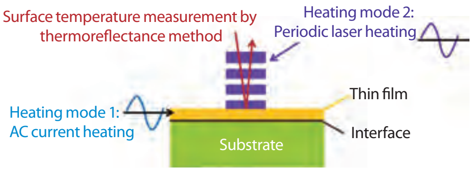


Fig.1 Experimental system for measuring the interfacial thermal resistance between a thin film and substrate and the principle of measurement.

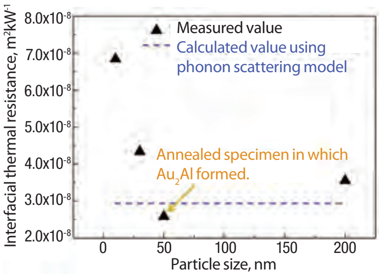


Fig.2 Relationship of interfacial thermal resistance between an Au thin film and single crystal sapphire substrate with Au particle size.

Tool for Lightweight Hybrid Materials Development

Low Thermal Expansion, Light-weight, Hybrid Lattice Structures

Composite Materials Group, Hybrid Materials Unit,
Environment and Energy Materials Division

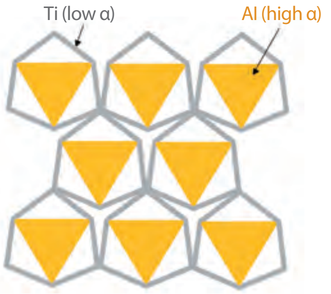
Christopher Mercer

In many applications, structural materials systems are subjected to large temperature gradients during service. Because of thermal expansion, these thermal gradients can impart large stresses on the structure. A good example of this is the outer skin of a future hypersonic vehicle, where frictional heating at velocities in excess of Mach 3, can lead to temperatures of several hundred degrees. The thermal gradients produced can generate extreme forces on the underlying attachments and components. Ultimately, structural failure can occur. Therefore, the purpose of this research is to design and fabricate hybrid lattice structures, comprised of two dissimilar materials, that allow control of thermal expansion behavior. An example of such a lattice is shown in Fig.1. It consists of a framework made from a material with a low coefficient of thermal expansion (CTE), e.g. titanium, and triangular inserts composed of a material with a higher CTE, e.g. aluminum. During heating, the inserts expand more than the framework and push against it. The framework members are forced to rotate at the nodes. This rotation counteracts the lengthways expansion of the individual framework members, and the nodes remain invariant. As a result, low or zero thermal expansion is achieved.

The concept has been successfully demonstrated for bi-metallic lattices consisting of a Ti-6Al-4V framework with Al 7075-T6 inserts. Experimental results are in very good agreement with finite element predictions. The next stage of the research to extend the concept to carbon and glass fiber reinforced polymers. By controlling the orientation of the fibers relative to the lattice geometry, it is possible to precisely control the thermal expansion characteristics.

Finally, in addition to exhibiting low thermal expansion behavior, the lattices must also possess good mechanical properties, such as high strength and stiffness, and low weight, since they are to be employed in structural aerospace applications.

Christopher Mercer Completed doctoral course in the Department of Materials Science and Engineering of The Ohio State University, U.S.A. in 1996. Prior to this he was employed as a technologist at Rolls-Royce, United Kingdom. Since receiving his Ph.D., he has held research positions at The Ohio State University, Massachusetts Institute of Technology, Princeton University and the University of California Santa Barbara. He joined NIMS as a Senior Researcher in 2008.



Schematic illustration of a low thermal expansion lattice structure.

Tool for Realizing Hybridized New Functions:

Material Property Control Using Porous Metal

Composite Materials Group, Hybrid Materials Unit,
Environment and Energy Materials Division

Satoshi Kishimoto

Porous metals are sponge-like materials consisting of a cellular structure with high porosity. These porous materials are manufactured by a variety of methods, including foaming the metal, bonding numerous hollow metal spheres, baking metal powder in a sponge-like form, and plating metal on a sponge-like polymer which is intrinsically porous.

Because porous metals have this distinctive form, they exhibit low density and have shock absorbability. Taking advantage of these properties, some attempts have been made to use porous metals, for example, as materials for spacers between the hood and engine in automobiles in order to reduce the impact on the human body (in particular, the skull) during traffic accidents.

We have developed a technology for encapsulating a material (e.g., polymer, metal, ceramic, etc.) within the cells (pores) of porous metals, and have also developed a technology for producing porous metals in which the size of the cells is changed stepwise in the thickness direction. When a polymer is encapsulated, this material is produced by impregnating the polymer in a porous metal with interconnected open cells. On the other hand, when a metal or ceramic is encapsulated, the powder particles of a metal or ceramics are coated by a metal and then this porous metal is fabricated by sintering the metal-coated particles (Fig. 1). Porous metals in which the cell size (diameter) is successively changed can also be manufactured if particles of different diameters are coated with metal and arranged in the order of size in the thickness direction (Fig. 2).

When polymers or ceramics are encapsulated in the cells of these porous metals, the stiffness and strength of the material are improved, and if the cell size and cell wall thickness is also changed, physical properties such as the elastic modulus, coefficient of thermal expansion, thermal conductivity, etc. can be controlled. Thus, we are now engaged in research on the manufacture of materials having the optimum physical properties in order to produce hybrid materials.

Satoshi Kishimoto Dr. Eng. Entered NRIM in 1984, and was appointed Senior Researcher in 1993. Joined NIMS as Principal Researcher in 2001 and became NIMS Chief Researcher since 2009.

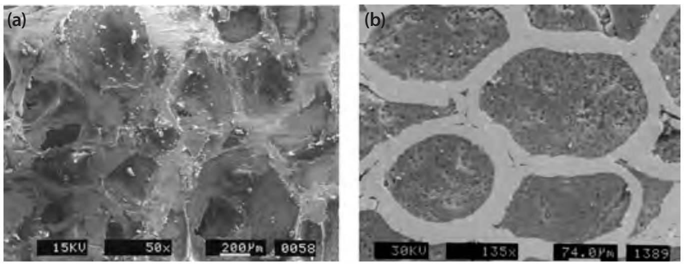


Fig.1 Scanning electron microscope images of materials with cellular structures. **(a)** Cellular metal (stainless steel) encapsulating a polymer (polyurethane), and **(b)** cellular metal (Ni-P alloy) encapsulating a ceramic (silicon carbide).

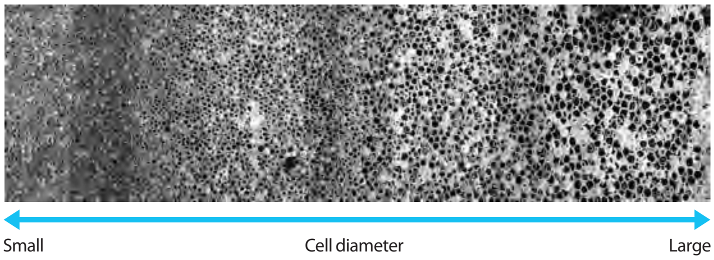


Fig.2 Porous metal (Ni-P alloy) with graduated cell diameter.

Tool for Lightweight High-Reliability Functional Materials

Research on Hybrid-Type Polymer Composites

Composite Materials Group, Hybrid Materials Unit,
Environment and Energy Materials Division

Kimiyoshi Naito

Fiber reinforced polymer matrix composites (FRPs) excel in high specific strength and high specific stiffness. However, the low fracture strain, low interlaminar shear strength and low fracture toughness of composite materials are drawbacks. For example, delamination and cracking occurs easily when an object such as a stone is simply dropped on the composite. Thus, improvement of impact resistance is an unsolved problem. Composite materials also tend to fail suddenly, which is a serious safety issue.

Fiber-metal laminates (FMLs), which are hybrids of FRPs and metals, were developed to solve these problems. FMLs not only compensate for the weaknesses of FRPs, but also suppress the crack propagation of metals, since FRPs have excellent fatigue characteristics. Thus, FMLs overcome the weaknesses of both metals and polymers. Among FMLs, hybrids of glass fiber reinforced polymer matrix composites and aluminum alloys have already been applied practically.

On the other hand, one issue for hybridization of FRPs and metals is control of residual stress during manufacturing. Figure 1 shows the distinctive fracture behavior of an FML hybrid comprising an aluminum alloy, glass fiber reinforced polymer matrix composite (GFRP), and carbon fiber reinforced polymer matrix composite (CFRP). Delamination failure has occurred due to the differences in the coefficients of thermal expansion between the constituent materials and the poor interlaminar shear properties of the FRPs.

In this research, a new a material design technology was developed, which targeted a body with a lightweight structure for use in next-generation electric vehicles. The interfacial bonding configuration of the FML was varied in 2- or 3-dimensions, making it possible to control the interfacial fracture resistance and the interlaminar shear properties of the FRP (Fig. 2). The failure behavior of this hybrid material was clarified experimentally and analytically using a variety of evaluation techniques. Based on these results, we are now developing a hybrid material design technology for lightweight, high reliability hybrid materials with high impact resistance (crashworthiness) and fatigue resistance.

Kimiyoshi Naito Dr. Eng. Completed doctoral course at the Doshisha University in March 1998 and was employed in the Mitsubishi Electric Corporation, Kamakura Works, Sagami Factory. Joined NIMS since February 2005 and is currently a Senior Researcher.

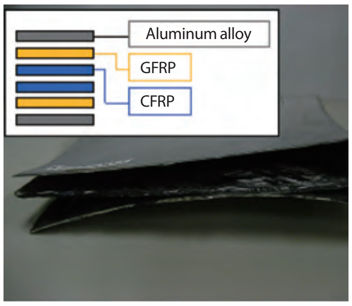


Fig.1 Hybrid material comprising an aluminum alloy, GFRP, and CFRP. Dramatically improved impact resistance and fatigue resistance are expected as a result of hybridization. However, in this example, delamination failure of the hybrid material occurred due to the differences in the coefficients of thermal expansion between the constituent materials and the poor interlaminar shear properties of the FRP.

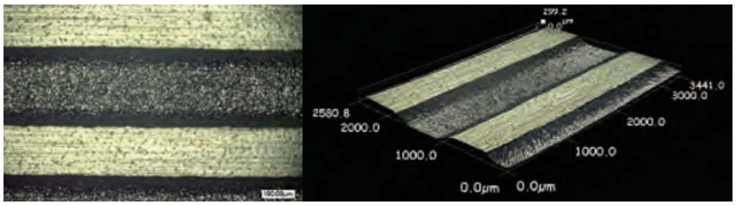


Fig.2 Example of variation of bonding interface configuration. (Left) Optical microscope photograph of the surface of an aluminum alloy, and (right) a 3-dimensional image using the optical microscope. The author attempted to control the interfacial fracture resistance and interlaminar shear characteristics of the FRPs by forming a stepped profile at the bonding interface.

Tool for Reliability Evaluation/Joint Interface Evaluation and Analysis

Reliability Evaluation Technology for Lightweight, High Strength Composites

Unit Director, Hybrid Materials Unit,
Environment and Energy Materials Division

Yutaka Kagawa

Composite Materials Group, Hybrid Materials Unit,
Environment and Energy Materials Division

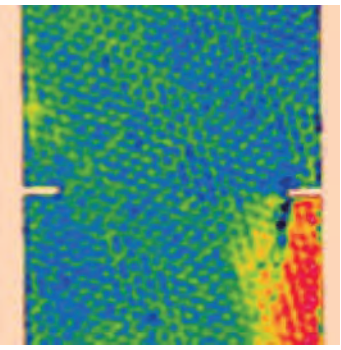
Yoshihisa Tanaka

The applications of lightweight composite materials with excellent mechanical properties are expanding, beginning with carbon fiber reinforced plastics (CFRP) and carbon fiber reinforced SiC matrix composite materials, which have a large specific modulus and specific strength. These materials themselves have histories of more than 30 years, and future expansion of their application as structural materials is predicted. When these composite materials are employed as structural components, adhesion and joining techniques for different materials, such a pairs of composites, composites and metals, etc. are of key importance. Historically, adhesion and joining techniques have been fields where know-how was necessary. Typically, completely different results are obtained with different combinations of object materials. The aims in this research are to establish a quantitative evaluation/analysis method for the problem of delamination of the adhesion/joining interface between different materials and a nondestructive technique for detecting delamination.

The following figure shows an example of detection of delamination, by measuring changes in heat conduction, for a fiber reinforced composite consisting of laminated textiles. Delamination has occurred in the composite in the yellow and red areas.

As shown by this example, delamination which is not visible from the surface can be detected by accurately measuring changes over time in the temperature at the surface of a composite material, when the surface is irradiated with pulsed white light. At the same time, by performing a dynamic analysis of the delamination phenomenon, the energy release rate necessary for delamination of the interface can be obtained. This measurement technology provides a noncontact, nondestructive evaluation technique, and as an analysis technology, it is useful in the evaluation of material properties and realizing interfaces using property values which do not depend on know-how.

Yutaka Kagawa For profile, see p. 4.
Yoshihisa Tanaka For profile, see p. 5.



Example of detection of the area where delamination has occurred in a laminated fiber reinforced composite material.

Tool for Net Shape Lightweight/ Heat Resistant Materials

Research on Hybrid Ceramic Materials

Composite Materials Group, Hybrid Materials Unit,
Environment and Energy Materials Division
Shuqi Guo

It is well known that the brittleness of ceramic materials can be overcome by hybridization with ceramic fibers which possess strength. This type of strengthening, or reinforcement has been demonstrated in a large number of materials over the past 20 years. However, in order to create not only experimental materials, but also practical materials, the establishment of a manufacturing process which satisfies the three requirements of short time, low cost, and high reliability is key.

Within the field of fiber reinforced ceramics, high expectations have been placed in recent years on materials in which short carbon fibers are dispersed in silicon carbide (SiC), as wear resistant materials for use in severe environments, such as brake disks for high speed trains, automotive materials, etc. However, an energy efficient, high speed manufacturing technology is essential for achieving practical application of these materials.

In the manufacturing process developed in this research (Fig. 1), firstly, short carbon fibers, SiC particles and phenolic resin are mixed to a homogeneous mass, and a carbon fiber reinforced plastic green body (CFRP) which is extremely close to the shape (i.e., a "net shape" or "near-net-shape" compact) of the final product is produced. Next, the green body is heated to high temperature in inert atmosphere, causing carbonization of the polymer, in order to produce a porous SiC-modifying carbon/carbon preform. Then, the porous body is put together with liquid silicon under vacuum at temperatures above the melting point of silicon. After the liquid silicon is infiltrated into the porous short fibers body, the Si reacts with the C in the compact, forming a hybrid material of carbon fiber and SiC.

The same high strength observed in monolithic ceramics cannot be obtained in this hybrid material. However, as shown in Fig. 2, the hybrid has sufficient damage tolerance so as not to break when a nail is driven into it. This property can be obtained because the function of accumulating micro-damage in the material is imparted to the material by hybridization of carbon fibers and SiC.

Shuqi Guo Ph.D. Completed doctoral course in the Graduate School of Engineering of the University of Tokyo in 1997. He was a Researcher in the Institute of Industrial Science (IIS) of the University of Tokyo, JSPS Fellow, and NEDO Fellow before being appointed to his present position as Principal Researcher at NIMS in April 2005.

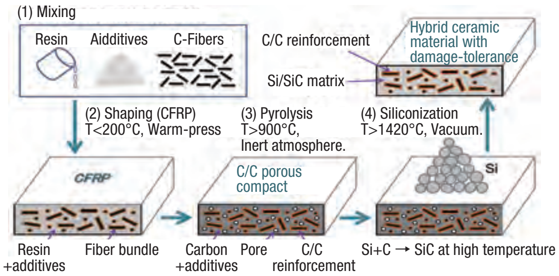


Fig. 1 Schematic diagram of energy efficient, high speed net shape manufacturing process.

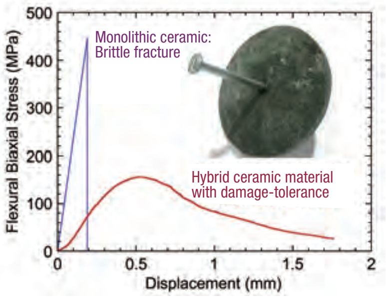


Fig. 2 Stress-displacement curve of disk-shaped hybrid ceramic material.

Tool for Design of Hybrid Ceramics

Design Method for Lightweight, Tough Ceramic Structures

Unit Director, Hybrid Materials Unit,
Environment and Energy Materials Division
Yutaka Kagawa

Carbon fiber reinforced SiC matrix composite (hybrid) material is a lightweight, heat resistant ceramic material. As a material with improved fracture resistance, which is a drawback of SiC, it is considered a promising material with a wide range of application fields, from aerospace to automobiles. In parallel with the development of a process technology for this material, analysis of its mechanical properties, taking into consideration the use application environment, has become important.

One key feature of this hybrid material is the mechanism by which it demonstrates its crack-resistant properties: by accumulation of micro-cracks in the material. To maximize this mechanical property, it is necessary to make skillful use of the non-uniformity of the microstructure in the material to obtain consistent functioning of the mechanism which prevents fracture of the material. Therefore, we are investigating the mechanism in which the inhomogeneity of the material microstructure results in micro-cracking and theoretically optimizing the mechanism which prevents fracture.

Based on the phenomenon that micro-cracks occur in the material, a knowledge of the amount of damage accumulated in the material, and an understanding of the link between the amount of damage and the safety of the material, is important for determining the reliability of the material. The model shown in Fig. 1 is a "mosaic model," which is used in theoretical analyses of non-uniform microstructures. The composite microstructure for realizing the target property can be obtained by incorporating the properties of the respective constituent phases and the interfacial mechanical properties between the constituent materials.

This theoretical consideration can be applied generally to hybrid materials which show similar properties. For example, by introducing the optimum composite microstructure or hybrid structure, even ceramic materials that display brittle fracture behavior can be widely useful as structural materials with new functions, and can be handled in the same manner as metallic materials. The concept of predicting properties and then fabricating materials (Fig. 2), rather than fabricating the material and then investigating its properties, is particularly useful in speeding up the development process for hybrid materials, in which the possible combinations of materials are unlimited.

Yutaka Kagawa For profile, see p. 4.

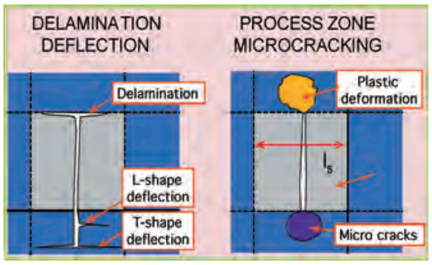


Fig. 1 Mosaic model used for analysis of the mechanical properties of fiber reinforced ceramic composite materials.

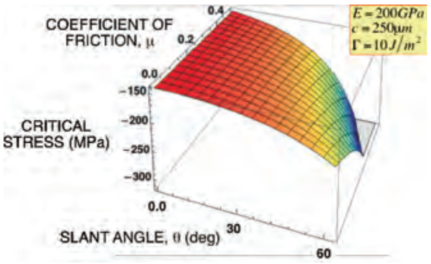


Fig. 2 Conditions for propagation progress of an unstable crack from a micro-crack, showing an example of the relationship of the compressive stress applied to the material, the slant angle relative to the axis of compression of the micro-crack, and the coefficient of friction of the crack surface.

Tool for Future Homo/ Heterogeneous Material Joining Process

Environment-friendly Hybrid Bonding Technology at Low Temperature in Ambient Air

Interconnect Design Group, Hybrid Materials Unit,
Environment and Energy Materials Division
Akitsu Shigetou

In the field of microelectronics, there is a strong demand for integrating organic/biocompatible substrates and conventional LSIs, which is obtained with single assembly (bonding) method, in order to create a multi-functional high-speed flexible system. For this, discrete layer structures, where diverse materials (semiconductors, glass, polymers, metals, etc.) show their surfaces on the same plane, have to be bonded vertically at the same time (Fig. 1). Additionally, the process temperature has to be lowered to, say, 150°C at least, to compensate for severe difference in thermal expansion among the materials. It is also inevitable to perform the bonding sequence in ambient air and non-toxic condition for the sake of high throughput and biocompatibility. In order to obtain good bondability on the mixed surfaces with different bond mechanisms in such an adsorptive condition at low temperature, and to ensure electric conduction at the metal-metal interface simultaneously, we have to quantitatively modify chemical binding state and morphology (physical contact) of the outermost adsorbate layer to create a bridging function. Many possible candidate materials have been proposed for such a bridging layer. However, in this study, emphasizing the advantage of low toxicity and high applicability of water, we proposed a novel bonding technology, named "vapor-assisted surface activation method." In this, a compatible and stable bridging layer is created on the surfaces of multiple materials in single process by quantifying adsorption/desorption behavior of water molecules on atomically clean surfaces, which results in highly controlled chemical/physical structure of aqueous compound. Up to now, we have realized homo/heterogeneous interconnection among Cu, glass, polyimide, etc., at 150°C in ambient air, regardless of the combination of materials (Fig. 2). Some of these results won the technical awards for high feasibility to 3D hetero-integration of future electronics.

Akitsu Shigetou Dr. Eng. Completed the doctoral course in the Department of Precision Engineering, School of Engineering, the University of Tokyo. She then worked as a Research Fellow at the Research Center for Advanced Science and Technology (RCAST), and as an Assistant Professor, at the same University. She has been a Senior Researcher at NIMS since September 2007.

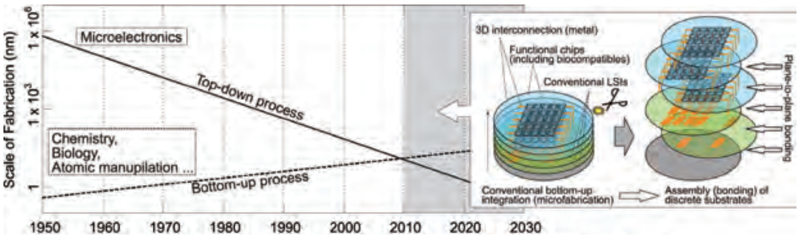


Fig. 1 Loadmap of organic/inorganic hetero integration for electronic devices.

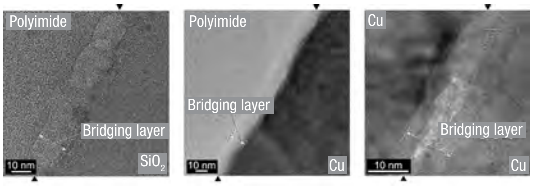


Fig. 2 Transmission electron microscope images of homo/heterogeneous bond interfaces of Cu, SiO_2, and polyimide obtained at 150 °C and atmospheric pressure by vapor-assisted surface activation method.

Tool for Design of Functional Interconnection

Reversible Bonding/Adhesion Technologies Learning from Nature

Group Leader, Interconnect Design Group,
Hybrid Materials Unit,
Environment and Energy Materials Division
Naoe Hosoda

Bonding/adhesion technologies are constantly evolving in tandem with the development of new materials. Today, the role of bonding/adhesion technologies is not limited simply to joining two substances. Technologies are now being developed which impart functions to the joint, including electrical conduction, heat conduction, optical properties, vibration properties, etc.

In next-generation bonding technologies, development of ultra-high functions for joint interfaces will be required as a technology for supporting advanced technologies. Control of the bonding/adhesion and disassembly and recyclability will also be new requirements as technologies supporting resource circulation-type manufacturing.

Our group is developing new bonding technologies which respond to these needs. This article introduces the development of technologies for control of bonding/adhesion and debonding.

"Bonding and debonding" means control of phenomena which are essentially complete opposites, in that a joint must be strong during use but must also be easy to debond or disassemble. In fact, there are organisms in the natural world which skillfully control these phenomena. Organism which move on surfaces in 3 dimensions, such as leaf beetles, jumping spiders, and house geckos, have evolved legs or feet with excellent adhesion/debonding properties. We are engaged in the development of technologies for controlling adhesion and debonding at the nano, micro, and macro sizes by researching the mechanism of the appendages of these tiny creatures.

The mechanism by which leaves fall from plants that naturally lose their leaves in autumn is also an example of control of adhesion and debonding. From the time when buds form, falling leaves have a layer with a separation mechanism, called an "abscission layer," at the leafstalk. When it receives a signal of the timing of separation, this part grows, causing self-separation. We are also engaged in research on incorporating this mechanism in bonding technology.

We hope to further develop the results of this research as a technology supporting resource circulation-type manufacturing, and to ensure that this work is useful.

Naoe Hosoda Ph.D. (Science) Acquired doctoral degree at the University of Stuttgart. She was an Assistant Professor at the Research Center for Advanced Science and Technology (RCAST), the University of Tokyo and Associate Professor of the Department of Precision Engineering, also at the University of Tokyo, prior to her present position as Group Leader of the Interconnection Design Group. She has held concurrent positions as Manager of the Learning from Nature Cluster, Associate Professor of the University of Tsukuba, Visiting Associate Professor of Shibaura Institute of Technology, Lecturer at Niigata University, and Visiting Researcher at the Max-Planck Institute for Metal Research.

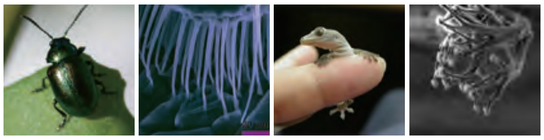
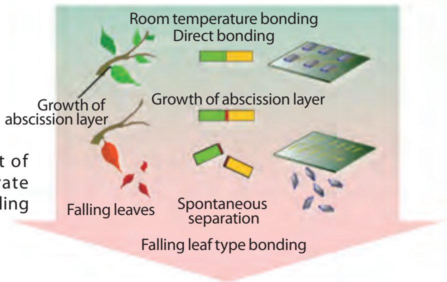


Fig. 1 Attachment devices of the leaf beetle *Gastrophysa viridula* (Coleoptera, Chrysomelidae) and the gecko *Gekko japonicus*

Fig. 2 Development of interfaces that separate spontaneously like falling leaves.



Success in Development of High Temperature/ High Speed Gigacycle Fatigue Tester

Materials Fatigue Group, Materials Reliability Unit,
Environment and Energy Materials Division
Yoshiyuki Furuya

High speed vibration occurs in the turbine blades of jet engines and gas turbines during operation. Because the frequency of this high speed vibration is several kHz (several 1000 cycles/second), the number of fatigue cycles exceeds 1 gigacycle (10^9 cycles) in approximately 10 days. However, gigacycle fatigue due to this type of high speed vibration cannot be evaluated adequately with the conventional technology, as the frequency used in normal fatigue tests is on the order of 10 Hz (10 cycles/second).

In this research, an ultrasonic fatigue test technology using a frequency of 20 kHz (20,000 cycles/second) was used. Ultrasonic fatigue testing is one type of axial fatigue tests, in which cyclical tension and compression are applied to a test piece using the phenomenon of resonance in longitudinal, i.e., axial vibration. As merits, in addition to the fact that the fatigue test reaches the gigacycle region quickly, the frequency used in the test is also close to that in actual equipment.

However, the ultrasonic fatigue test is normally performed at room temperature, and it is no easy matter to perform testing at high temperature, assuming the operating environment of turbine blades and similar parts. It is not possible simply to attach a heating device, as the ultrasonic fatigue test uses the phenomenon of resonance, and the condition of resonance changes at higher temperatures because Young's modulus (material constant) is affected by temperature.

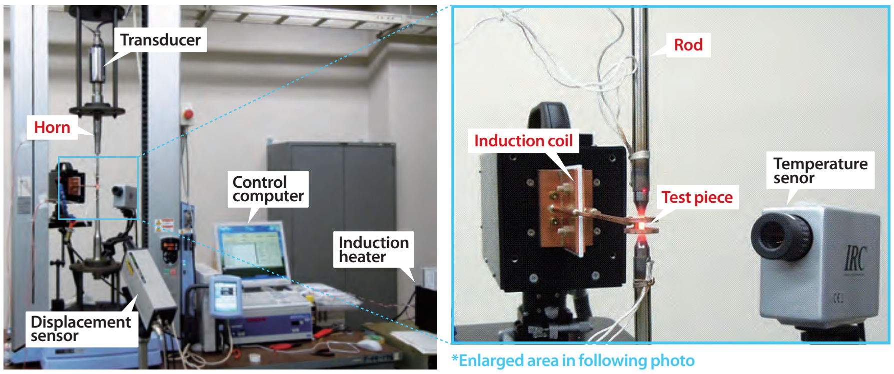
This device development research was carried out by making various improvements to a commercial ultrasonic fatigue tester, which was used as a base. Fig. 1 shows photographs of the developed device. An induction heater and various types of sensors were added, and parts which required improvement were independently designed and fabricated. New software for performing stress calculations was also developed. Although this development was a process of repeated trial and error, as a result, we succeeded in developing a device which is capable of controlling all parameters with good accuracy under high temperature conditions. The range of temperatures in which testing is possible using this device is roughly 500-1500 °C.

The results of tests which were performed using the device are shown in Fig. 2 and Fig. 3. Fig. 2 shows the results of a test at 650 °C using heat-resistant steel for boiler use; Fig. 3 shows the results of a test at 1000 °C using a turbine blade material.

The test with the boiler material was a preliminary test to identify problems with the device. The results were in good agreement with the results of an ordinary fatigue test (1-800Hz, no frequency effect), confirming that highly reliable results can be obtained.

Likewise, in the test with the turbine blade material, which is the real target, the results were in good agreement with those of a conventional fatigue test (performed only at 10Hz, frequency effect unclear). This results shows that appropriate results can also be obtained under a severe condition of 1000 °C.

As can be understood from these results, a high temperature ultrasonic fatigue test device which provides high reliability and can be used at 1000°C was successfully developed.



*Enlarged area in following photo

Fig. 1 Photographs of the developed device. Independently developed parts are shown in red. Software for the personal computer used for control and the measurement methods for the displacement sensor and temperature sensor were also developed independently.

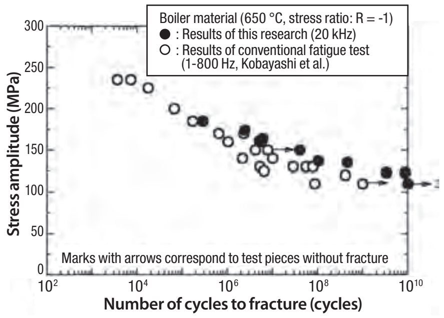


Fig. 2 Results of test performed at 650°C using heat-resistant steel for boiler use. The results obtained with the developed device are in good agreement with the comparison data (values in the literature).

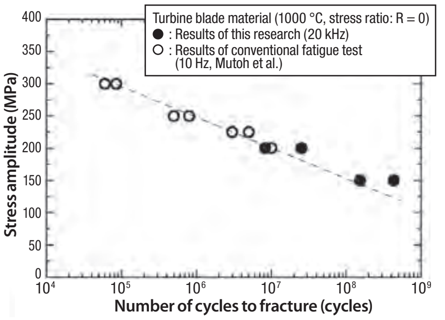


Fig. 3 Results of test performed at 1000 °C using turbine blade material. The results obtained with the developed device are in good agreement with the comparison data (values in the literature).

Yoshiyuki Furuya Dr. Eng. Completed the doctoral course at the Kyushu University Graduate School of Engineering. He was employed as a researcher at NRIM in 2000, became a NIMS Researcher in 2001, and was appointed as a NIMS Senior Researcher in 2005. He was appointed to his present position in 2011.

Novel Approach for Synthesizing Reduced Nano-Oxides: Low Temperature Reduction of Nanosized TiO_2 without Particle Growth

Nano Interface Group, Nano Interface Unit,
Nano-Green Field, MANA
Satoshi Tominaka

ICYS-MANA
Yoshihiro Tsujimoto

Essential nanomaterials for a low-carbon society

Fuel cells are important energy devices for reducing CO_2 emissions, and thus highly-functional materials for improving their performance are developed worldwide. For the effective improvement of the power generating efficiency of fuel cells, electrode materials with a large surface area, which enhances the reaction extracting electricity from chemical substances, are necessary. The development of nanomaterials is indispensable for this. For example, if 1 cm^3 of electrode material is divided into small particles with a size of 1 nm^3, the area of the material will be increased by a factor of 10 million (Fig. 1). As this shows, use of nanosized material is effective for increasing electrode surface area.

We are engaged in research focusing on titanium oxides, which have high chemical durability as electrode materials for fuel cells. Among the titanium oxides, titanium dioxide (TiO_2) is the most stable, and improved durability can be expected in comparison with carbon materials which are used as fuel cell electrodes. Normally, however, TiO_2 is not an electrical conductor. Reduced titanium oxide, which contains less oxygen than TiO_2, is an attractive material, as it displays high conductivity and its surface is known to be passivated with native TiO_2.

In the synthesis of reduced titanium oxide, treatment in which TiO_2 is reduced at a temperature on the order of 1000°C is normally used. Because the melting point of rutile type TiO_2 is more than 1800℃, it can be considered stable even in a reduction reaction at 1000℃. However, in actuality, it is difficult to obtain nanoparticles of reduced titanium oxide from nanosized TiO_2, as the particles grow during the reduction reaction. The melting point of the substance is considered to be reduced significantly at the nanosize. It has also been reported that 30 nm particles of TiO_2 grew to double their original size when heated for 15 days at 465 °C. Thus, it is necessary to deepen our understanding of the thermal stability of the substance in order to obtain a nanosized material.

Removing oxygen atoms with maintaining shape

Therefore, we studied an approach which is different from that used to date, namely, removing oxygen atoms from nanoparticles of TiO_2 at low temperature with maintaining the shape of those particles. The experimental procedure is simple: TiO_2 nanoparticles is mixed with calcium hydride, which works as a strong reducing agent even at low temperature, and then is heated at 350℃, which is roughly 600℃ lower than that in the conventional method. As shown in Fig. 2, we succeeded in synthesizing a material absorbing visible light, this meaning that the titanium oxide changed to the reduced type, in which electrical conductivity can be expected. Moreover, the nanostructure of the reduced one was the same as that of TiO_2 precursor. Although we are currently investigating the detailed mechanism, we have ascertained that stable bonds which maintain crystallinity and nanostructure even while oxygen atoms are removed exist in the crystal, resulting in gradual change in the crystal structure from TiO_2 to Ti_2O_3.

The merit of our technique is the possible synthesis of nanosized reduced titanium oxide from nanosized TiO_2, which had been studied by numerous researchers. In other words, it is not necessary to develop a special technique for synthesizing nanosized reduced titanium oxide from raw materials such as titanium chloride or the like. Moreover, this technique is expected to be applicable to the synthesis of other TiO_2 nanostructures, for example, TiO_2 nanowires. Such nanostructures have the merit of large surface area, and also make it possible to form electrodes with excellent electrical conductivity. We are currently engaged in a further analysis of the reaction mechanism and electronic state from the atomic level, in order to establish a synthesis method for highly-functional titanium oxides as materials useful for society.

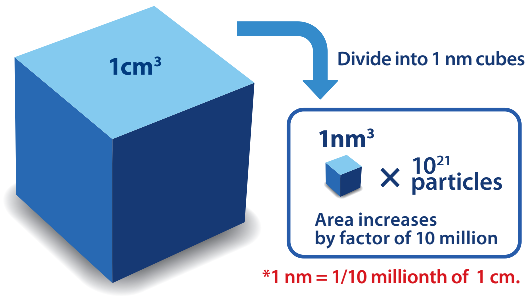


Fig. 1 Surface area of nanomaterials

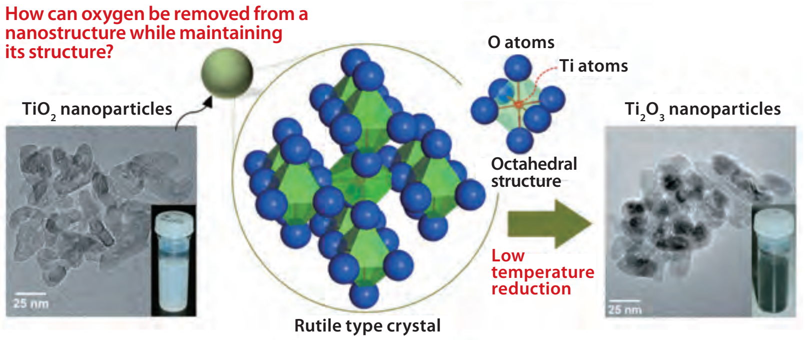


Fig. 2 Transmission Electron Microscope (TEM) images of TiO_2 nanoparticles (left) and reduced titanium oxide synthesized by the low temperature reduction method (right)

Satoshi Tominaka (right) Dr. Eng. Completed the doctoral course at the Graduate School of Science and Engineering, Waseda University in 2009 and joined NIMS in 2010 as a MANA Scientist. His specialties are physical chemistry and electrochemistry. He is engaged in research on nanomaterials and development of devices in the field of fuel cells, etc.

Yoshihiro Tsujimoto (left) Ph.D. (Science) Completed the doctoral course at the Graduate School of Science, Kyoto University in 2009. Was a Japan Society for the Promotion of Science (JSPS) Research Fellow prior to joining the International Center for Young Scientists (ICYS) of MANA in 2010. He is engaged in development of new functional materials, centering on the transition metal oxides.

NIMS NEWS

1 Workshop in Budapest on "Metal Oxide / Polymer Nanocomposite and Application"

(Sept. 19-21, 2011) A 3-day "Workshop on Metal Oxide / Polymer Nanocomposite and Application" was held in Budapest, Hungary. It was held jointly by the Research Institute for Technical Physics and Materials Science, Hungarian Academy of Sciences and the Nano-Electronics Materials Unit at NIMS as part of the bilateral joint research program of the Japan Society for the Promotion of Science (JSPS). With the participation of Tohoku University, Budapest University of Technology and Economics, Johannes Kepler University (Linz, Austria), and the Leibniz Institute of Polymer Research (Dresden, Germany), which are engaged in joint research with the two host organizations, a total of 26 researchers participated in the event.

The workshop began with a Keynote Address by Prof. István Bársony (Director of the Research Institute for Technical Physics and Materials Science), followed by a lecture by Prof. Niyazi Serdar Sariciftci of Johannes Kepler University. A wide range of topics were also discussed by young researchers from the various countries, and researchers from the NIMS side also presented a number of reports, mainly on the basic properties of the materials, covering the functions of ZnO as a semiconductor, its interfacial properties with organic materials, the mechanism of formation of organic-semiconductor interfaces, and other topics.


Attendees of the workshop

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2 Minister of Science and Technology Development in Zimbabwe, Visits NIMS

(Sept. 29, 2011) Minister of Science and Technology Development in Zimbabwe, Hon. Heneri Dzinotyiweyi, visited NIMS accompanied by the Ambassador to Japan, H.E. Stuart Harold, and other two delegates to discuss possible cooperation with NIMS and to study the operation of NIMS.


Minister (right) and Ambassador(left)

The Minister showed a great interest in NIMS' efforts on materials research and achievements since becoming an Independent Administrative Institution as well as the inter-linkages between NIMS and industry and acceptance of students and young scientists from abroad.

The Minister toured laboratories of computational materials science and also nanomaterials research. The Minister holds a Ph.D. degree in Mathematics and also a professorship at University of Zimbabwe, so he was strongly interested in the research activities and asked many questions during the laboratory tour.

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3 NIMS new partnership

(Oct. 10, 2011) Structural Materials Unit and Materials Reliability Unit in NIMS (Tsukuba, Japan) and Max-Planck-Institut für Eisenforschung GmbH (Düsseldorf, Germany) agreed to a Memorandum of Understanding (MOU) on the research and development in the field of structural materials.

Max-Planck-Institut für Eisenforschung GmbH conducts basic research for an improved understanding on iron, steel and related materials, such as nickel, titanium and intermetallic phase alloys. The institute currently consists of three departments (1. Microstructure Physics and Metal Forming, 2. Interface Chemistry & Surface Engineering, 3. Computational Materials Design), and each department has the latest research facilities and top-level scientists. Through their inter-department activities, coupling experiments and calculation, world-level fundamental research is running there to pursue mechanisms of materials properties.

We are expecting a further development of structural materials research based on the MOU.


Prof. D. Raabe(left) and Dr. K. Tsuzaki(right)

Hello from NIMS

Dear NIMS NOW readers,

Living and working in Japan has been one of the best experiences of my life! I joined NIMS in January 2011 as a postdoc and have discovered a lovely working environment with many friendly colleagues. As a member of the International Center for Young Scientists I work independently with guidance from some expert mentors, which is great training for an academic career. We have lots of support from English-speaking staff and there are also classes for learning Japanese. Life in Tsukuba includes lots of cultural activities and parties with international friends. Many people also take the chance to visit all the beautiful sites of Japan, such as Kyoto and Nara, and the nearby Nikko National Park. Of course, one of the best things about Japan is that there is always the chance to relax in the Onsen at the end of the day!


Skiing on perfect powder in Nagano – the site of the 1998 Winter Olympics


Looking out over the beautiful volcanic scenery in the Aso-Kuju National Park

Zoe Schnepp (UK)
ICYS
January 2011-Present

NIMS NOW International 2011. Vol.9 No.9
National Institute for Materials Science
http://www.nims.go.jp/eng/publicity/nimsnow/

cover image: room temperature bonding machine and the gecko *Gekko japonicus* for reversible bonding technology research

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Mr. Tomoaki Hyodo, Publisher
Public Relations Office, NIMS
1-2-1 Sengen, Tsukuba, Ibaraki, 305-0047 JAPAN
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