

# AIST

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MESSAGE

## President's Message Thoughts for the New Year

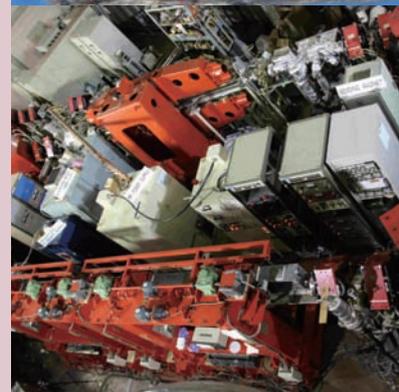
FEATURE

## Rare Metals 2

## Research Hotline

UPDATE FROM THE CUTTING EDGE (October–December 2009)

## In Brief



# President's Message: Thoughts for the New Year



**Tamotsu Nomakuchi**

President

National Institute of Advanced Industrial Science and Technology (AIST)

## 1. Introduction

I hope you began the New Year 2010 with novel thoughts in a fresh ambiance. Last year drastic changes took place in the administration in Japan and the first nonwhite President assumed office in the US. Let us hope these changes will pave paths to a safe and sustainable society.

Last year also brought me considerable changes, such as assuming the presidency of AIST. During the nine months after my taking office, I have enjoyed inspiring discussions with members of universities, companies, and public institutions, of both domestic and overseas, as well as conversations with the directors of research units and those of departments for research support and management within AIST. Through such communications, I kept pondering over the meaning of “In Society, For Society”, the motto in the AIST Charter.

## 2. AIST Open Lab 2009

We held the second AIST Open Lab on October 15 and 16, 2009. There were more than 3,000 visitors during the two-day event, approximately the same number as last year, mainly from the private sector. Though AIST continuously announces its research strategies of each research field to the public, and makes effort to disseminate the research results, to understand AIST, I found it particularly effective

for our guests to actually visit the research sites and to directly discuss with the researchers. I myself visited some sites, and was encouraged by seeing many researches of huge possibilities. Also, at every corner I felt enthusiasm and vitality, possibly because of strong motivation and awareness of the issues of many participants from industry. I believe AIST's message was well delivered that we put high priority on realizing a low-carbon society and a safe, secure, and healthy society.

In spite of the present status which is still bound by expenditure cutbacks and is far from economic recovery, many people visited the AIST Open Lab from industrial society. I believe there were two reasons for their visits. One is that the industry now has big expectations toward AIST for its leading and fundamental researches, as I wrote in AIST Today No. 32. Another is that the research policy of AIST, “Integration for Innovation --Technology to Society” has begun to be recognized and valued widely among them.

During the event, I met many executives of various companies and learned anew that they are indeed charged with various problems. They seek to solve the pressing problems, to meet the new industrial standards, and to search future courses in their business enterprises. Though it is beyond our capacities to meet all of such demands of the companies, I am convinced of our mission to contribute to

solving diverse issues by sharing our concerns with industry, and by the “*Full Research*” that we promote by covering from basic research to product realization research.

In my key note lecture on the first day of the AIST Open Lab, and also in “AIST Today” No. 33, I mentioned that I have come to realize anew that the significance of R&D for industry, academia and public sectors is intrinsically different, though collaboration of the three sectors is quite often referred to. For “Industry” the motivation or driving force of R&D would be business needs, while for “Academia” it would be intellectual curiosity though it is stipulated in the Fundamental Law of Education that the missions of universities are education, research and social contribution. Well then, what would be the significance of R&D to the public sector? It should be the needs and demands of the nation and society. We have piles of issues to be solved for the sustainable development of society. Many of these issues, however, could not be dealt with only by industry that focuses on economic activities, or by universities that respect academic freedom. It is the role of the “public sector” to stand up and meet such challenges. The AIST Open Lab is the very occasion for people involved in “industry” and “academia” to gain deeper understanding of AIST researches. Collaboration based on our mutual understanding would be the most dynamic driving force of open innovation of Japan.

The questionnaires filled in by the participants gave us invaluable comments and requests on AIST’s research and public relations. I recall that some comments noted that the exhibition of successful businesses created in cooperation with small and medium enterprises should have been announced in more effective ways. Many of the visitors expressed hopes for continuation of the AIST Open Lab. From the next time on, it would be even more meaningful and well-organized based on our experience.

Here, I would like to mention the one-day AIST Open Houses. They are regularly held to introduce research activities to the local communities to deepen their understanding of AIST, and at the same time to offer young generation chances to be in direct contact and become familiar with the latest achievements in science and technology, and thus to encourage them to acquire better understanding. It also attracts visitors from industry and

universities, since the latest researches are also exhibited. Last year, we had 6,000 participants in Tsukuba and 14,000 in the whole AIST network including the regional centers. I myself visited the Open House at AIST Tokyo Waterfront, and was impressed by an elementary school pupil who was touching “Paro”, the therapeutic robot by AIST, and looking into kaleidoscopes with full curiosity. The recent tendency of young people’s distancing away from science has been a concern of society. However, with enjoyable and easy-to-understand presentations of the wonders of science and technology, we are helping to increase “little scientists”. Therefore, we should also continue to make the AIST Open Houses better.

“AIST is not good at publicizing its research results. Since significant researches are conducted, AIST should disseminate the results in more efficient ways.” This is the comment I once received from the external peer reviewers. I had a similar impression myself when I was in industry. Once I began to see AIST from the inside, however, I recognized our people’s steady and solid endeavor. However, besides their academic publications, we need to publicize more to the general public to gain deeper understanding of our research. This is what the reviewers intended to point out. Now, a serial column introducing characteristic researches at AIST appears every Monday in *Nikkan Kogyo Shimbun*. We should seek to increase such chances for dissemination.

### 3. ISO General Assembly

The 32<sup>nd</sup> General Assembly of International Organization for Standardization (ISO) was held in Cape Town, South Africa, from September 16 to 18, 2009. I attended it for the first time, as the chairman of Japan Industrial Standardization Council, which I had assumed in February 2009, before I joined AIST.

I had a strong impression that international standards are deeply related to the sustainable development of the world economy. At the opening session, representatives from International Electrotechnical Commission in charge of electrical standardization, and Telecommunication Standardization Unit of International Telecommunication Union in charge of communication standardization, and also of World Trade Organization (WTO) participated.

The WTO representative emphasized that international standardization had become indispensable for the sound development of world trade.

It was also impressive that many discussions focused on how we could contribute through our standardization activities to addressing the present global issues such as climate change. In short, the harder the issue is, the more effective and useful standardization would be in sharing our awareness of problems and in planning efficient R&D investment.

A proposal by International Energy Agency for the realization of a low-carbon society also attracted attention. It proposed to share a highly reliable common calculation method called Measurable, Reportable, Verifiable Method, or MRV Method, in calculating the carbon emission in the world, which is similar to the idea Japan has developed.

My previous conviction that international standards nowadays have come to play extremely important roles in national science and technology strategies and industrial strategies, and also in business strategies of companies, is now strengthened.

Though these few years Japan has been emphasizing international standardization, and has increased occasions of acting as a convener country etc., we are still behind Europe and USA, while recent activities of China and Korea should not be easily ignored. Fortunately, at this ISO General Assembly, Mr. Takeda, Director-General of the Japanese Standards Association, was elected Vice-Chairman of ISO. He is an alumnus of the Ministry of Economy, Trade and Industry, and has been recognized in the world with his years of expertise in international standardization organizations. Japan should send out more of such experts who can actively serve in international scenes. Even in the field of standardization, there exists a sort of North-South issue in differences of approaches by industrialized countries and developing countries. Industrialized countries consider standardization to be essential for fair development of the world economy, whereas developing countries regard it to be indispensable for globalizing their own industry and request industrialized countries for appropriate share of cooperation. It is a welcome situation that Japanese persons play leading roles in this area, in terms of international contribution. Therefore, we should steadfastly support them.

Recently, AIST has been active in international standardization, and many of our colleagues serve as conveners of ISO technical committees. In the past, technologies that were already mature were frequently standardized. These days, however, the interest is to have more efficient R&D by applying standardization with regard to new emerging issues such as climate change. This tendency widens fields where AIST could be increasingly involved.

Here I would like to make a small digression. The “Consortium on fabrication and characterization of solar cell modules with long life and high reliability,” established last autumn, plans to propose new technologies related to evaluation and diagnostic methods as international standards, as well as to reinforce production competence of the 31 participating component makers. I think it is important to have the standardization targets in a plan from the start as in this case. Generally speaking, our traditional method of manufacturing has been to create the very best product which no body else could imitate, while relying on the effort of standardization of others. However, this method will not catch up with the world today, where technology evolves and globalizes drastically. One may temporarily enjoy the top position for sometime, but quite soon will be left behind if the technology is not standardized. Therefore, we are in the age where proposing our own standards is extremely important as part of our research strategy.

Let me extend my discourse further. Those who know AIST appreciate the value of its role as a calibration agency for various measurements related to physics, chemistry, and biology. AIST is well known as a depository of prototypes of kilogram (and meter), while it is worldly renowned for its activities as the National Metrology Institute of Japan (NMIJ), for standards of seven base units of length, mass, temperature, time, electric current, luminous intensity, and amount of substance. Measurement instruments are produced based on the results of tests and calibrations of NMIJ, AIST. The high prestige in the quality of Japanese products in the world market is established firmly upon such efforts of AIST.

Recently AIST is widely expected to expand its functions to establish and disseminate standards to cover integrated technologies, the expansion of targeted

technology fields, and the increase of the safety and risk awareness. In order to meet such social expectations, we will make efforts to utilize AIST's advantages as a comprehensive research institution.

#### 4. Collaboration with USA and South Africa

Early May 2009, I visited USA to conclude memorandums of understanding (MOUs) of research cooperation with five institutes of the US Department of Energy (DOE):

- \* Sandia National Laboratories (SNL),
- \* National Renewable Energy Laboratory (NREL),
- \* Los Alamos National Laboratory,
- \* Lawrence Livermore National Laboratory,
- \* Lawrence Berkeley National Laboratory, and with
- \* National Institute of Standards and Technology (NIST) of the US Department of Commerce.

NREL and NIST clearly proclaim their missions, of renewable energies and standards respectively in their names. The others are comprehensive research institutes covering wide fields with their individual specialties. I visited cutting-edge research facilities of nanotechnology and admired the advanced equipment and the research environment. I had an occasion to discuss with Dr. Steven Chu, Secretary of Energy, and gathered that he would be emphasizing not only nanotechnology, but also necessary fields for the realization of a low-carbon society such as new materials, renewable energies, and storage batteries. AIST cannot simply stand still and give way to others. We welcome challenges of our good rivals in fair and friendly competition. After the visit to the US, new programs for exchanges of information and researchers are being organized, based on the MOUs. Researchers sent from AIST would learn immensely in the US, working together with excellent researchers from all around the world. Reciprocally, trainees will be accepted at AIST from the University of New Mexico of the state of New Mexico where SNL and other collaborating laboratories are located.

Prior to the aforementioned ISO General Assembly, my colleagues and I visited three national research institutes of South Africa in September:

- \* Council for Science and Industrial Research (CSIR)
- \* Council for Geological Science (CGS)
- \* Mintek

The three institutes and AIST held the first workshop. CSIR is a comprehensive research institute, CGS is the institute of geological survey, and Mintek is an institute specialized in resources and mines. Though I was able to attend only the first day of the workshop, I heard that there were good discussions and exchanges of information with coordinators and researchers from AIST. We were impressed by their enthusiasm to promote the basic capacities for industrial development making use of their natural resources. Their interest in promoting collaboration with AIST is clear, to which we hope to respond, and already some joint researches are underway. High governmental officials that I met the day before the workshop showed strong interest in renewable energies (utilization of photovoltaics and solar heat), catalyst technologies to improve conversion efficiency in coal liquefaction and fuel cells. I would like to extend my gratitude to Ambassador Ozawa and his colleagues at the Japanese Embassy in South Africa for their generous understanding and support of our mission. Though we are geographically far apart, we hope to enhance collaboration with the institutes.

#### 5. Obiter Dictum

For more than two years I have served as a member of the Council for Science and Technology of the Ministry of Education, Culture, Sports, Science and Technology. There have been discussions on the present tendency of young researchers not wishing to go to universities or research institutes overseas to explore their potentials. The reasons might be that studying abroad would not merit in their career development, or that there is no need to go abroad because of our improved research environment, or that exchanges of information and researchers are much easier nowadays with the advanced traffic systems and communication methods. The decisive reason given by the majority of the members, however, was that the "spirit of challenge" or the "feeling of yearning" of young people is no longer vigorous. This is indeed a regrettable tendency for our country, and encouragement measures should be devised. While universities are expected to reform their laboratories, AIST is going to foster qualified researchers that can lead the world.

# Rare Metals 2

## Trends in Rare Metal Issues and Efforts of the Rare Metal Task Force

### Rare metal issues

While there is widespread concern over the crisis of the limited availability of energy resources, we must also recognize the seriousness of the problems facing the supply of rare metals due to the fact that most of rare metal deposits are located in extremely limited countries and that development of their substitution is more difficult than in the case of energy, which has several alternatives. Although Japanese manufacturers heavily depend on rare metals, domestic production (especially onshore production) is extremely limited. Below are some of the reasons why issues related to rare metals have become critical.

#### • Demand

① The rapid economic development of countries with massive populations, such as China and India, is increasing demand for rare metals.

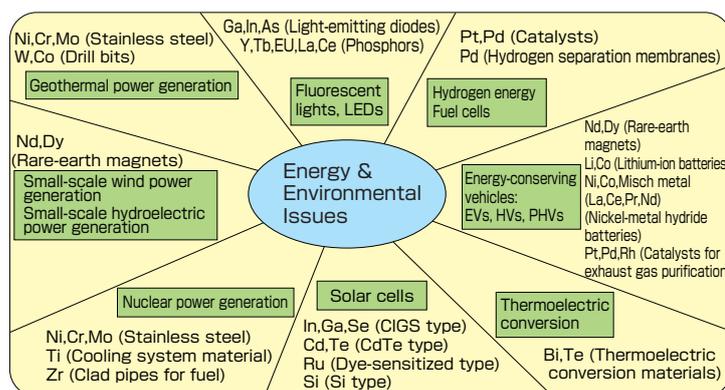
② The IT revolution, globalization of mass production systems (modularization and low labor costs), and huge amounts of capital that can be freely moved around the world have made a rapid increase in global productivity (i.e., a rapid increase in consumption of natural resources) possible.

③ Demand has increased for new products that require rare metals, such as EV and HV, because of policies to deal with energy and environmental issues (“green rare metals”).

#### • Supply

④ An oligopoly for some of the rare metal resources has been created by China and some major mining companies.

⑤ The development of new mineral deposits faces longer lead times and higher



Energy & Environmental Issues and Rare Metals

costs due to resource nationalism and environmental protectionism.

⑥ Mining of low-grade deposits, which become necessary as high-grade deposits are depleted, require substantial amounts of energy. With existing technologies and rising energy cost, the usability of low-grade deposits is limited.

#### • Speculation

⑦ Enormous speculative funds are searching for alternative investments that can replace investments in stocks and land. Investment in natural resources is a strong candidate.

Because of the above factors, demand for rare metals will rise continuously with a high probability of a surge in the short term, while a rapid increase in their supply is likely to become more and more difficult.

### Status during and after the financial crisis

The prices of many rare metals have declined with the fall in demand due to the recession, which began with the financial crisis in the autumn of 2008. However, the above-mentioned factors have not changed at all. In fact, the prices of some rare metals

for which there is a strong oligopoly have not decreased. Tight supply and demand of rare metals can be expected with economic recovery, or with the mere anticipation of a recovery. If the supply of even one type of rare metal becomes limited, the manufacturing of products that use it will be constrained. This could cause major damage to an industry as a whole, particularly if the industry is highly competitive internationally.

### Efforts of the Rare Metal Task Force

The objective of the AIST Rare Metal Task Force is to support Japan's economic security by developing urgently needed technologies that will have a major impact on the entire flow of rare metals, such as resource exploration, technologies to reduce rare-metal consumption, technologies to develop the substitutes for rare metals, and recycling technologies, utilizing AIST's research potential and accumulated technological know-how.

Director  
Materials Research Institute for  
Sustainable Development  
**Mamoru Nakamura**

# Exploration of Rare Metal Resources

## Background of rare metal exploration

There are two types of metal resources. One type is consumed in massive quantities as vital industrial materials. These include iron, copper, and aluminum, to name only a few. The other type, referred to as rare metals, sees very little consumption in quantitative terms but is essential for high-technology industries. In particular, rare-earth elements (REE) are critical metals for magnets in electric motors used for hybrid vehicles and consumer electronics. As such, their consumption has been increasing every year, and a stable supply of the REE is vital to Japan's industrial development. Unfortunately, however, the stability of their future supply is in question, because more than 90 % of REE are produced in China and the supply is easily impacted by the political, economic, and mining industry conditions there. With the above as the backdrop, the Mineral Resource Research Group of the Institute for Geo-Resources and Environment (GREEN) is conducting resource explorations overseas to diversify the supply chain of REE outside of China. Details of the REE exploration activities of the research group are discussed in the May 2008 issue<sup>[1]</sup>, as well as on the pages 4 and 5 of this issue. Here, I will discuss the concepts behind this undertaking.

## What is resource potential evaluation?

The aim of the resource exploration activities of the Mineral Resource Research Group is to evaluate the resource potentials



**Field investigation at Canada's Thor Lake deposit**  
Long shadows are seen even at midday because the site is located in the Arctic.



**Mineral ore at Thor Lake**  
Abundant rare-earth elements are contained in the reddish-brown mineral (bastonite).

of yet-to-be-developed mineral deposits. The term *resource potential* refers to the prospects for developing a mineral deposit based on the grade and total reserve of the ore of the targeted element. A resource potential needs to receive an evaluation of high quality in order to minimize the risks associated with mine exploitation and make stable production possible. Generally, the quantity of ore reserves in a deposit is calculated based on information obtained from the three-dimensional structure of the deposit and the ore distribution in terms of its grade, which can be gathered from surface exploration, drilling surveys, and chemical analyses. In the case of a REE deposit, however, it is of critical importance to understand the form of existence of the REE concerned in addition to the above aspects. For example, if REE is contained within carbonate minerals, it can be easily extracted with acid treatment, but if it is contained in refractory minerals such as zircon, extraction will be difficult to justify in a conventional business case study.

Therefore, those parts that are difficult to extract must be excluded from the calculated quantity of the ore reserve. An accurate resource potential evaluation of a rare earth deposit should thus include mineralogical information on how many parts per million of REE are contained within what mineral.

## Future developments

The overseas resource exploration activities of the Mineral Resource Research Group are being conducted in collaboration with private enterprises and the Japan Oil, Gas and Metals National Corporation (an independent administrative institution). We are in the process of targeting several promising REE deposits, and are moving forward with the survey work in the hope of starting a new mine by 2014.

Institute for Geo-Resources and Environment  
**Tetsuichi Takagi**

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[1] Y. Watanabe: *AIST TODAY*, 8 (5), 18-19 (2008).

# Types and Characteristics of Rare Earth Deposits

## Current status of REE resources

Rare earth elements (REE) are considered to be seventeen elements consisting of lanthanoids (lanthanum to lutetium), yttrium, and scandium<sup>[1]</sup>. Demand for REE has been increasing in recent years for use as catalysts in automotive applications and as rare earth magnets. It is predicted that the demand will continue to rise as hybrid vehicles become more prevalent. However, more than 96 % of REE resources are imported from China<sup>[2]</sup>. In particular, heavy rare earth elements (HREE) have lower crustal abundance compared with light rare earth elements (LREE). In addition, the types of deposits that supply HREE are limited. Therefore, a discovery of new mineral deposits is essential for stabilizing the supply of REE resources (especially HREE resources)<sup>[3]</sup>. We are currently investigating resource potential in various REE deposits .

## Types of deposits supplying REE resources

Deposits that supply rare earth resources<sup>[4]</sup> can be divided into two categories; igneous deposits formed by igneous rocks or associated hydrothermal activity and weathering deposits created from weathered rocks (Fig. 2). Examples of igneous deposits include carbonatite deposits, alkali rock-related deposits, and hydrothermal iron

deposits. Carbonatite which is an igneous rock consisting mainly of carbonate mineral is an important source of REE resources. China's Maoniuping deposit and the Mountain Pass deposit in the U.S.A. are two of the major carbonatite deposits. Alkali rocks often occur with carbonatites, and can form HREE-rich deposits. Canada's Thor Lake deposit is representative of the alkali rock-related deposits. A hydrothermal iron ore deposit has many characteristics. China's Bayan Obo deposit, which is the largest deposits in the world, is a well-known hydrothermal iron deposit.

Examples of weathering deposits include laterite deposits, ion-adsorption type deposits, and placer deposits. Laterite here refers to red soil that becomes rich in REE due to decomposition of carbonate minerals by weathering. Enrichment of REE by the weathering process is a marked characteristic of this type. Australia's Mount Weld deposit is a representative example. Ion-adsorption type deposits exist only in southern China. Most of HREE supply comes from this region. This type of deposit is characterized by REE adsorption onto the surface of clays and other materials in weathered granite soil. The Longnan deposit and Xunwu deposit are representative examples of such deposits. Placer deposits are formed by sedimentation

of concentrated heavy minerals through weathering and erosion of rocks in locations such as seashores. They are rich in ilmenite, but also often contain monazite, which is one of REE minerals. The Kerala region in southern India and Richards Bay in South Africa are examples of areas of placer deposits.

## Minerals containing REE

The ore minerals containing REEs consist of carbonates, phosphates, oxides, and ion-adsorption clays. Bastnaesite [REE(CO<sub>3</sub>)F] is a representative REE mineral among carbonate minerals and is contained in carbonatite deposits. Monazite [(REE,Th)PO<sub>4</sub>] is representative of phosphate minerals. It is commonly found in placer deposits and igneous deposits. The oxides include loparite [(REE,Na,Ca)<sub>2</sub>(Ti,Nb)<sub>2</sub>O<sub>6</sub>], which is contained in alkali rocks. With ion-adsorption clays containing kaolinite [Al<sub>2</sub>Si<sub>2</sub>O<sub>5</sub>(OH)<sub>4</sub>] and other materials, as REE are electrically adsorbed onto the mineral surfaces, they can be recovered by ion exchange using a mild acid without decomposing the minerals themselves. Although there are several silicate minerals containing REE, they are rarely used as resources because of the high cost of extraction due to their generally tough crystal structure.

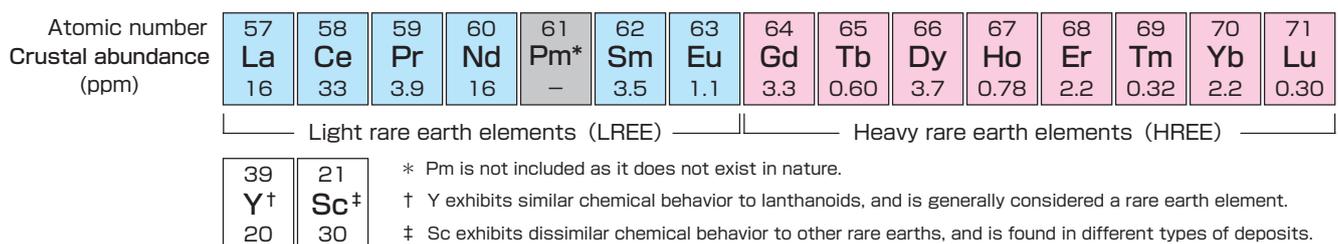
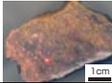


Fig. 1 Types of rare earth elements (REE) and crustal abundance

	Types of deposit	Representative examples of deposits (country)		Dominant REE resources
Igneous deposits	Carbonatite deposits	Maoniuping (China) Mountain Pass (U.S.A.) - currently not operating		LREE
	Alkali-rock-related deposits	Lovozero (Russia) Thor Lake (Canada) - not yet developed		LREE HREE
	Hydrothermal iron ore deposits	Bayan Obo (China)		LREE
Weathering deposits	Carbonatite-derived laterite deposits	Mount Weld (Australia) - not yet developed		LREE
	Ion-adsorption type deposits	Longnan and Xunwu (China)		LREE HREE
	Placer deposits	Kerala (India) Richards Bay (South Africa) - not yet developed		LREE

**Fig. 2 Types of REE-supplying deposits and their characteristics**  
The photos show REE ores and deposits.



**Fig. 3 Outcrop of weathered crust of granite in Laos**  
Ion-adsorption type REE mineralization is partly identified.

### Challenges surrounding REE resources

We need to continue studying REE deposits, because systematic studies of such deposits are relatively uncommon in the world compared with those targeted at base-metal and precious-metal ore deposits. Identification of HREE deposits and of REE minerals are two of the challenges facing research on REE deposits. The first challenge is to identify and discover deposits capable of supplying HREE, which are relatively rare compared with LREE. For example, ion-adsorption type deposits, which supply HREE, were previously only found in China,

but such mineralization has begun to be locally discovered in other countries<sup>[5]</sup> (Fig. 3). We also need to study deposits that may be enriched in HREE, such as alkali rocks and placer deposits. The second challenge is to clarify the existence form of REE minerals, and quantitatively evaluate them. Although REE are contained within a variety of minerals, the majority of silicate minerals are difficult to utilize as resources. We also know that minerals containing REE occur between the grains and cracks of other minerals<sup>[6]</sup>. Therefore, we need to evaluate whether REE exist within minerals that are suitable

for mineral processing and refining, and whether they can be extracted economically. Resolution of the above issues will lead to the development of more REE deposits.

Institute for Geo-Resources and Environment  
**Kenzo Sanematsu**

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# Reduction of Platinum Usage by a Core-Shell Structure

## Necessity of reducing the use of platinum in fuel cells

Fuel cells, which have been attracting attention as a clean energy source in recent years, can be classified into several different types depending on the electrolyte used. Among the types of the fuel cells, the polymer electrolyte fuel cell (PEFC), which uses a proton-conducting polymer membrane as the electrolyte, can operate at a low temperature below 100 °C. Thus, power sources for portable electronic products and automobiles are among the targeted uses of PEFC.

There are however, issues associated with this type of fuel cell, specifically because it operates at a low temperature. Generally, the rate at which a chemical reaction takes place rapidly decreases with lowering temperature. A highly active catalyst is therefore required in order to ensure a sufficient reaction rate. In a PEFC, an oxidation reaction of hydrogen occurs on the anode and a reduction reaction of oxygen takes place on the cathode. With the existing technology, platinum must be used as the catalyst for both electrodes.

Platinum is a very scarce element, as is well known. Worldwide annual production of platinum is approximately 230 tons, and reserves of platinum-group elements are estimated to be 63,000 tons.<sup>[1]</sup> At the current pace, these reserves would last for close to 200 years. However, it is said that once PEFCs become the mainstream, platinum use will increase to 10 times the current level or more depleting the reserves in less than 20 years. The issue of the supply of platinum-group elements is

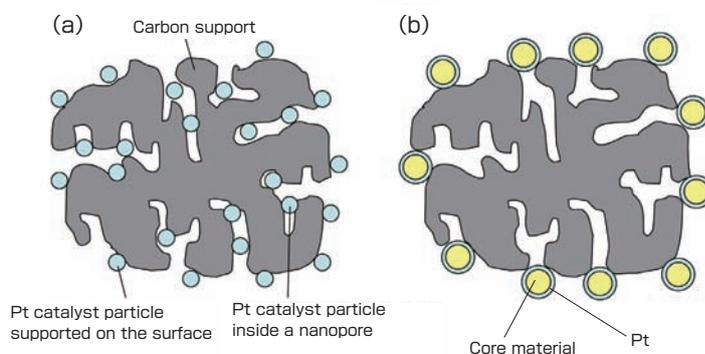
extremely serious, especially considering the fact that approximately 80% of these elements is exclusively distributed in the Republic of South Africa<sup>[1]</sup>.

## Core-shell catalyst technology

With the above as the backdrop, there are various efforts worldwide to significantly reduce the use of platinum in PEFCs. The Catalytic Nanomaterials Group of the Materials Research Institute for Sustainable Development is examining the effectiveness of "core-shell technology", which is one of the methods for reducing the use of platinum as an electrode catalyst. The application of core-shell technology to

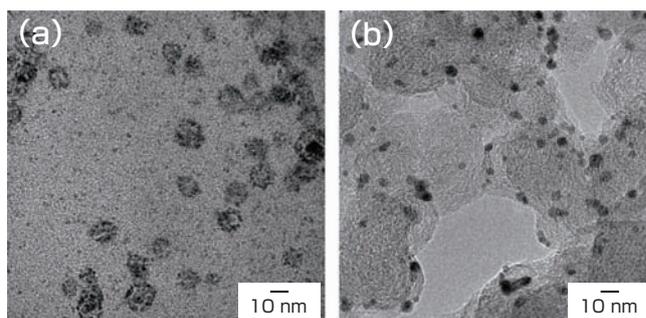
a catalyst involves replacing the interiors of the metal particles (core), which do not directly contribute to the catalytic reaction, with a different material, while leaving the surfaces of the particles (shell) consisting of a highly active material (platinum).

Increasing the specific surface area of metal particles generally improves the efficiency of a supported metal catalyst. Hence, it is a common practice to reduce the diameter of the metal particles. However, reducing the diameter does not yield the desired effect with a PEFC. The key is the support material that holds the metal particles. Ultrafine carbon particles forming a substance called carbon black

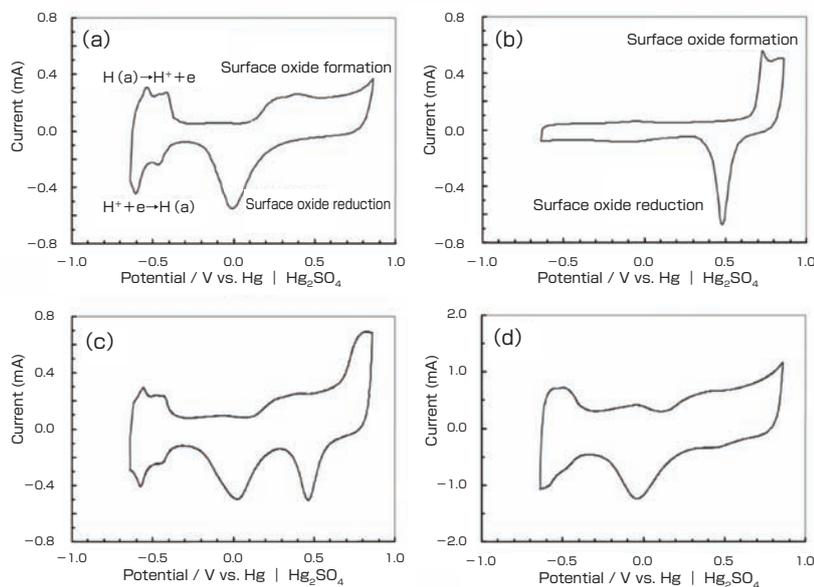


**Fig. 1 Platinum catalyst particles supported on carbon black**

(a) If the particle diameter is too small, the particles become trapped in the nanopores and do not contribute to the reaction.  
(b) The platinum utilization can be effectively improved by the core-shell technique without reducing the particle diameter.



**Fig. 2 (a) TEM image of catalyst nanoparticles with silver core and platinum shell (b) TEM image of catalyst nanoparticles with gold core and platinum shell supported on carbon black**



**Fig. 3 Cyclic voltammograms of**  
**(a) platinum nanoparticles**  
**(b) gold nanoparticles**  
**(c) mixture of platinum and gold nanoparticles**  
**(d) gold core-platinum shell nanoparticles**

are used as the catalyst support in a PEFC due to their conductivity. These carbon particles have a lot of nanopores, and if the metal particles are too small, they become trapped in these pores. The trapped particles are unable to contribute to the reaction (refer to Fig. 1(a)). Core-shell technology is one of the methods to improve the platinum utilization without reducing the particle size, avoiding the above phenomenon (Fig. 1 (b)). The core material used would be cheaper and with less risk of supply disruption than platinum.

### Production and evaluation of core-shell catalysts

Figure 2 (a) shows a transmission electron microscope (TEM) image of nanoparticles, produced by a method called liquid-phase reduction, that are each

made up of a silver core and a platinum shell. Several particles with a diameter of approximately 10 nm can be observed. The lighter colored interior is silver, and the darker colored surface is platinum. Figure 2 (b) shows a TEM image of nanoparticles on a carbon black support, consisting of gold cores and platinum shells. The diameter of the majority of the particles is from 2 to 4 nm, similar to that of commercially available platinum catalysts. It is difficult to directly verify the core-shell structure using a TEM image, because the atomic numbers of gold and platinum are close to each other. However, we can verify the structure electrochemically, as follows. Figures 3 (a), (b), (c), and (d) show the results of cyclic

voltammetry of platinum nanoparticles, gold nanoparticles, a mixture of platinum and gold nanoparticles, and gold core-platinum shell nanoparticles, respectively, all on a carbon black support. The plots (cyclic voltammograms) obtained in this method show a unique wave shape for each sample, based on the electrochemical characteristics of the surface materials. Apparently, (d) shows an almost identical wave shape to (a), even though the core-shell particles contain similar amount of platinum and gold. It can therefore be concluded that the nanoparticles are in the core-shell structure with platinum covering most of the outer surface.

Factors that will determine the performance of core-shell catalysts include the nanoparticle diameter, core material, core-to-shell ratio, metal loading, and production method, to name only a few. Our group will systematically alter these variables and evaluate the performance. In particular, we will focus on the reduction of platinum use, and explore the limits and possibilities of core-shell technology.

Materials Research Institute for Sustainable Development

**Yutaka Tai**  
**Wataru Yamaguchi**

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# Development of Iron-based Thermoelectric Material as an Alternative to Bismuth Telluride Alloy

## Massive amounts of waste heat

In Japan, several hundred million kL (crude oil equivalent) worth of primary energy is consumed annually. However, in the process of converting primary energy into secondary energy such as electricity, more than 60 % of the primary energy is released into the atmosphere as waste heat. Effective use of this massive amount of waste heat is being sought against the recent social background, such as concerns over the future supply of energy and the emergence of environmental issues.

However, it is difficult to effectively utilize waste heat by means of conventional energy conversion techniques—driving a turbine to create electricity, for example—because the sources of waste heat, such as power plants, factories, and automobiles, are widely scattered, and moreover the majority of this waste heat is at a low temperature of less than 200 °C. In order to effectively utilize this scattered, small-scale, low-temperature waste heat, "thermoelectric generation," which has a conversion efficiency independent of the

energy scale and is capable of generating electricity from thermal energy of any temperature, may provide a solution.

## The rare metal issue in thermoelectric power generation

Thermoelectric generation utilizes the temperature difference between thermoelectric materials to generate electricity. Principally, a basic component of thermoelectric generation system is an electrical circuit consisting of connections of p-type and n-type thermoelectric materials in series, as shown in Fig. 1. Such a setup can generate electricity when one end of the thermoelectric materials is heated using waste heat, thus making recovery and reuse of the energy possible. An example of a highly effective thermoelectric material for temperatures of 200 °C or below is an intermetallic compound using bismuth (Bi) and tellurium (Te). Active research has been conducted on Bi-Te thermoelectric material, and devices with a conversion efficiency of several percent have already

been developed.

However, both Bi and Te are rare metals. In addition, Te has lower estimated reserves than platinum, and Bi is unevenly distributed. The use of these materials as the basic elements in thermoelectric power generation devices, which would be required in large quantities to utilize waste heat sources that are scattered in various locations, may therefore be problematic. Moreover, the Bi-Te system can only be used in a limited range of environments because of its low chemical durability against oxidation and low mechanical strength. Hence, at AIST we are developing iron-based thermoelectric materials that are less resource-restrictive as replacements for the Bi-Te compound, in order to promote effective utilization of low-temperature waste heat for practical applications.

## Research and development of iron-based thermoelectric materials

$\text{Fe}_2\text{VAI}$  alloys with a Heusler crystal

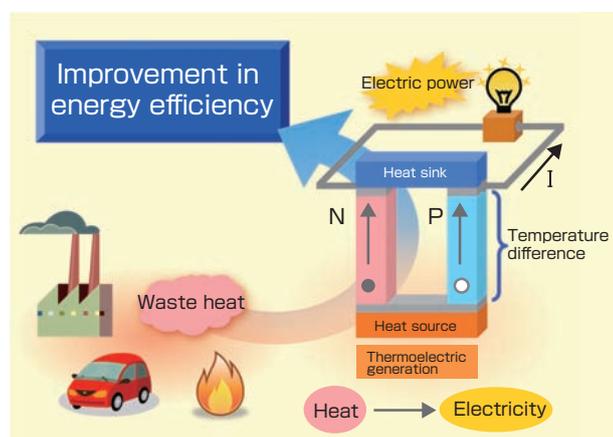


Fig. 1 Concept of waste heat recovery using thermoelectric generation device.

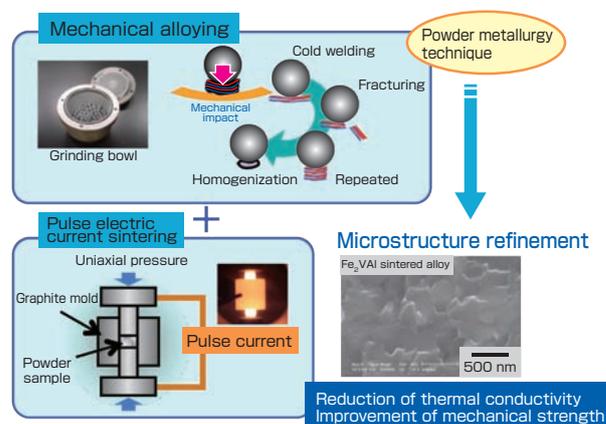
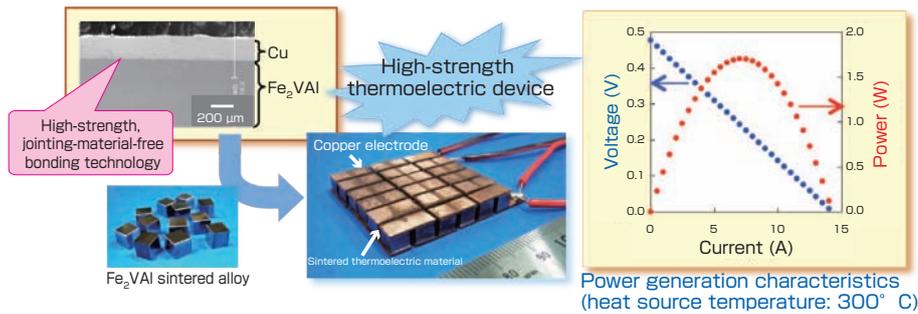


Fig. 2 Powder metallurgy technique and microstructure of  $\text{Fe}_2\text{VAI}$  sintered alloy



**Fig. 3 Thermoelectric device developed with direct electrode joining technique and its power generation characteristics**

structure exhibit superior power generation performance comparable to that of conventional thermoelectric materials, such as the Bi-Te system, at room temperature by regulating valence electron concentration via partial substitution of elements. They are stable materials, with a high melting point of 1500 °C along with good oxidation resistance. However, Fe<sub>2</sub>VAl alloys are not suitable for practical application as they are because of their relatively high thermal conductivity, resulting in the difficulty to obtain large temperature difference and in a low energy conversion efficiency.

AIST has therefore been attempting to enhance the thermoelectric properties of Fe<sub>2</sub>VAl alloys by controlling microstructure through a powder metallurgy technique, as shown in Fig. 2. First, we succeeded in obtaining the alloyed powder, which has the nanometer-sized internal structure, by using a mechanical alloying method. We achieved this by determining the conditions that allow stable repeated mechanical pulverizing and

mixing. Next, the mechanically alloyed powder was sintered rapidly using a pulse-current sintering technique to suppress grain growth during heat treatment and to obtain a fine microstructural bulk material. We were then able to obtain sintered alloy made of nanometer-order crystal grains. In the microstructured sintered alloy, we succeeded in lowering the thermal conductivity through a scattering effect at the grain boundaries. The structural refinement also improved the durability of the material by dramatically increasing its mechanical strength.

### Development of devices for practical application

Electrode connection is one of the most important factor of research for the development of practical thermoelectric devices. For the construction of conventional thermoelectric devices, jointing materials, such as solder, were used for the connection between electrode and thermoelectric material. However, this technique spoils the high

mechanical strength characteristic of Fe<sub>2</sub>VAl alloy, because jointing materials usually have a low melting point and low mechanical strength. We succeeded in developing a technique for directly joining Fe<sub>2</sub>VAl sintered alloy with copper electrodes that has high mechanical strength (Fig. 3). We have verified that as much as 1.7 W of power can be obtained when the thermoelectric device is used with a heat source of 300 °C. Moreover, the electrical resistance at the connection is negligible, resulting in minimal power loss at the connection.

With its comparably higher mechanical strength, the newly developed thermoelectric device can potentially be used in harsh environments such as the internal combustion engines of cars, which are subjected to vibration and extreme heat cycles. In addition, because of the reduction of reinforcements and other unnecessary parts with this new jointing technique, simplification of the structure along with increased design freedom can be realized when designing for practical applications. The device is economical and suitable for mass production because it is made up of elements that are cheaper and have a more reliable supply compared with conventional thermoelectric materials. We hope that it will contribute to the dissemination of thermoelectric conversion technology. AIST will continue its efforts to enhance the performance of thermoelectric materials and explore applications for waste heat.

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Materials Research Institute for Sustainable Development

**Masashi Mikami**

# Development of Highly Effective Extractant for Platinum Group Metals

## Separation and purification processes using solvent extraction

In recent years, platinum group metals (PGMs) have been increasingly used in industry, e.g., electrical systems and devices, catalysts, etc; therefore, it has become important to recover PGMs from their wastes. The solvent extraction (liquid-liquid extraction) method is mainly employed for separation and purification processes of the PGMs. This method is based on the partition of a solute between two immiscible phases (i.e., aqueous and organic phases). Typically, an organic compound (extractant) with high affinity to a specific metal diluted with an organic solvent (diluent) is used as an organic phase. Hence, the successful outcome of solvent extraction depends entirely on the selection of a suitable extractant. Moreover, for industrial applications, separation properties like extraction rate and durability need to be considered in addition to basic capabilities such as extractability and selectivity.

Currently, much of the metal recycling from PGM-containing wastes utilizes separation and purification processes consisting mainly of solvent extraction

that were developed by mining companies. In particular, processes based on that developed by INCO (Fig. 1)<sup>[1][2]</sup> are widely used. In the INCO process, silver chloride (AgCl) is precipitated at the leaching stage using chlorine gas (Cl<sub>2</sub>) + hydrochloric acid solution (HCl). Next, ruthenium (Ru) and osmium (Os) are separated by distillation, and gold (Au), palladium (Pd), platinum (Pt), and iridium (Ir) are separated by solvent extraction. Lastly, rhodium (Rh) is recovered from the residual solution. The extractants used here are excellent for the selective separation of PGMs, but many of them have issues concerning extraction rate, durability, etc. Another drawback is economic inefficiency due to the long retention period of rhodium, which is the most expensive of the PGMs. We are developing PGM extractants, and have discovered effective organic compounds for extracting palladium or rhodium.

## Rapid separation of palladium using an oxidation-resistant extractant

Di-*n*-hexyl sulfide (DHS) is one of the most popular extractants used in industry for the palladium separation. DHS exhibits a good capability for the

separation of palladium over platinum, but the palladium extraction is very slow. In addition, the palladium extractability with DHS deteriorates with its long-term use. A new palladium extractant with a high extraction rate and superior durability is therefore desired. We have found that thiodiglycolamide (TDGA) shows excellent extractabilities for palladium<sup>[3][4]</sup>. TDGA has a sulfide having a high affinity with palladium and two *N,N*-disubstituted amides, which is simply synthesized and soluble in various organic solvents. As seen in Fig. 2, the extraction rate of palladium with TDGA is extremely high compared with that with DHS. TDGA can also selectively extract palladium over platinum. Although the extraction percentage (*E*%) of palladium with DHS is severely reduced by contact with a strong acid solution (0.75 mol/L nitric acid + 2.25 mol/L hydrochloric acid), that with TDGA hardly changes. The FT-IR spectra show sulfoxide formation by the oxidation of the sulfide in DHS after contact with the strong acid solution, but not in TDGA, indicating that TDGA is more durable than DHS. Additionally, TDGA exhibits similar properties to DHS in regard to the back-extraction and loading capacity. Thus, TDGA has good properties for practical use along with its prominent basic extractabilities; we are now studying practical applications for this extractant.

## Development of a new extractant for rhodium

In the separation and purification

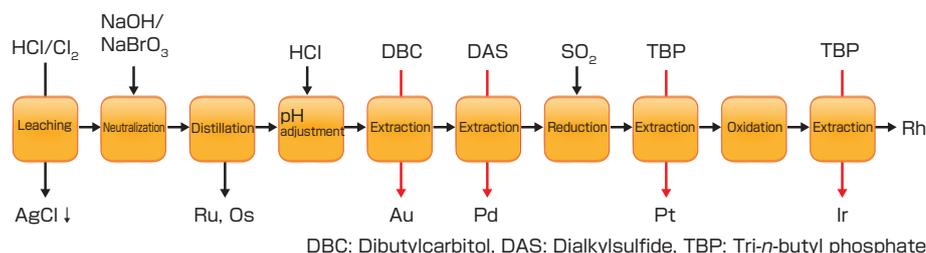
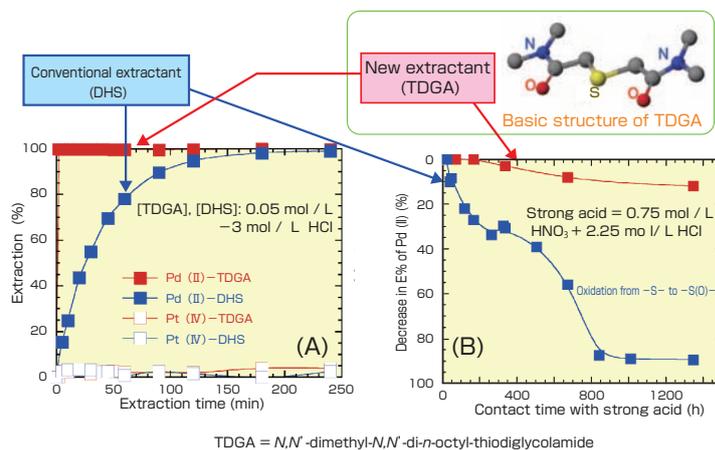


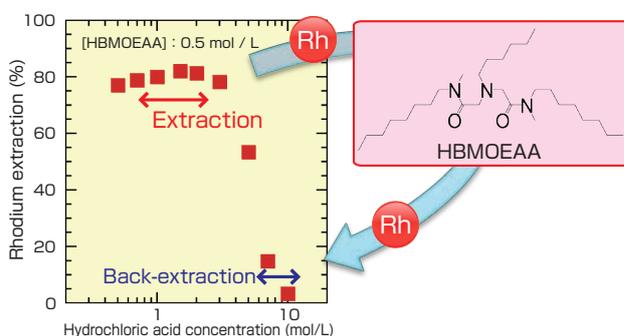
Fig. 1 Metal separation and purification process by INCO  
Solvent extraction is mainly used.

processes of PGM, as mentioned earlier, rhodium is recovered from the residual solution after the other PGMs are separated. This is because there has been no effective rhodium extractant developed for industrial use. Rhodium exists as anion complexes in relatively highly concentrated hydrochloric acid solutions, which are nearly impossible to extract. Generally, the application of an ion-pair extraction would be considered for extracting anion complexes, but additional effects to the ion-pair reaction seem to be required for rhodium extraction.

We synthesized a tertiary amine containing two *N,N*-disubstituted amides: *N-n*-hexyl-bis(*N*-methyl-*N-n*-octylethylamide)amine (HBMOEAA). We then studied the extraction behavior of the PGMs (rhodium, palladium and platinum) from hydrochloric acid solutions. As shown in Fig. 3, HBMOEAA extracts 80% of rhodium at 1 to 2 mol/L hydrochloric acid. This *E%* value is the highest ever obtained in rhodium extraction from relatively highly concentrated hydrochloric acid. As for the palladium and platinum extraction, high *E%*s from 0.5 to 10 mol/L hydrochloric acid are obtained, whereas rhodium is hardly extracted at about 10 mol/L hydrochloric acid. This means that palladium, platinum and rhodium are simultaneously extracted at 1 to 2 mol/L hydrochloric acid, and then rhodium alone is back-extracted into the aqueous phase by bringing the organic phase into contact with a 10 mol/L hydrochloric acid solution. This result of recovering



**Fig. 2 Comparison of palladium extraction using conventional extractant DHS with that using new extractant TDGA (A: extraction time, B: stability against strong acid)**  
 Compared with DHS, TDGA can selectively extract palladium more rapidly and the extractability for palladium is less affected by the strong acid.



**Fig. 3 Rhodium extraction from hydrochloric acid solution with HBMOEAA**  
 Extraction/back-extraction can be carried out by adjusting the concentration of hydrochloric acid.

rhodium before palladium and platinum is groundbreaking. The next research will focus on the extraction mechanisms and the characteristics required for practical application.

Research Institute for Environmental Management Technology  
**Hirokazu Narita**

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## UPDATE FROM THE CUTTING EDGE

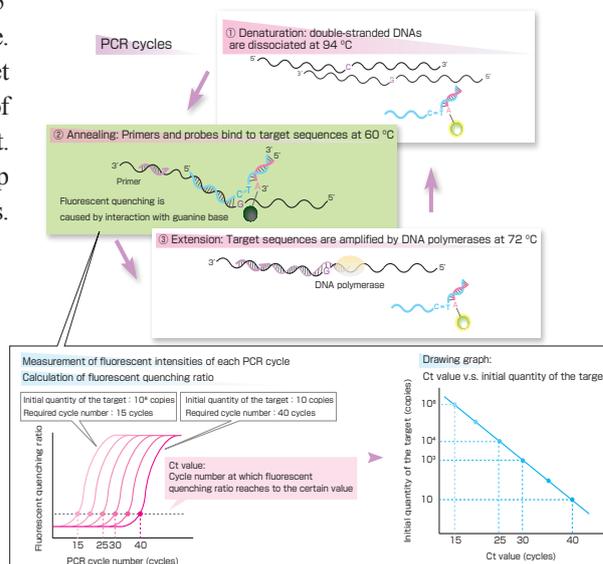
Oct.-Dec. 2009

The abstracts of the recent research information appearing in Vol.9 No.10-12 of "AIST TODAY" are introduced here, classified by research areas. For inquiry about the full article, please contact the author via e-mail.

Life Science and Biotechnology

### Universal quenching probe system: flexible, specific, and cost-effective real-time polymerase chain reaction method Application expected to quantitative detection of viruses

We have developed a flexible, specific, and cost-effective real-time polymerase chain reaction (PCR) method called the universal QProbe system. In this method, a quenching probe (QProbe) and a joint DNA are used. The QProbe is a singly labeled oligonucleotide with a fluorescent dye that is quenched via electron transfer between the dye and a guanine base at a particular position. The joint DNA has the target specific sequence on the 5' side, and the complementary sequence to the QProbe on the 3' side. When the QProbe/joint DNA complex hybridizes with the target in PCR, the fluorescence of the dye is quenched. The amount of fluorescence quenching is proportional to the quantity of the target. This method substantially reduces the cost of real-time PCR setup because the same QProbe can be used for different target sequences. This method can be applied to the quantitative detection of viruses.



Naohiro Noda

Institute for Biological Resources  
and Functions

noda-naohiro@aist.go.jp

AIST TODAY Vol.9 No.10 p.18 (2009)

Scheme of new method for gene  
quantification

## Discovery of early rising effect of a ketogenic diet on mice

### A low-carbohydrate diet affects the circadian clock

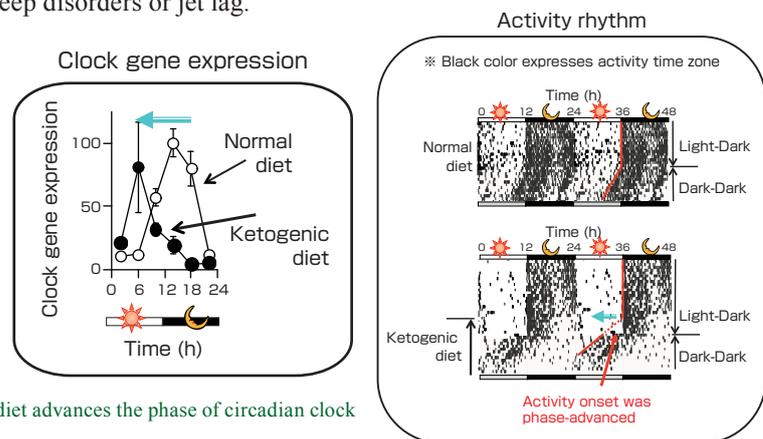
We have found that the circadian clock of mice can be controlled by feeding a low-carbohydrate diet. A low-carbohydrate, ketogenic diet was administered to mice for 14 days and subsequently, the expression of a clock gene which is a marker of the biological clock was analyzed. The clock gene was expressed 4 to 8 hours earlier in the mice on low-carbohydrate diet than in the control mice. Since a mouse is a nocturnal rodent and its circadian rhythms are strongly influenced by light, it is difficult to observe the behavior of mice in a light-dark (day-night) cycle environment. Hence, the mice were placed in constant darkness and the effects of the diet on their behavioral patterns were recorded. The mice on the low-carbohydrate diet woke up earlier (i.e., they became early risers) than the control mice. The forward shift of activities was also observed in the case of mice with DSPS (delayed sleep-phase syndrome; mice with DSPS tend to oversleep), who were fed the low-carbohydrate diet. We anticipate that this low-carbohydrate ketogenic dietary treatment would be a new therapy for sleep disorders or jet lag.

**Katsutaka Oishi**

Institute for Biological Resources  
and Functions

k-ooishi@aist.go.jp

AIST TODAY Vol.9 No.11 p.20 (2009)

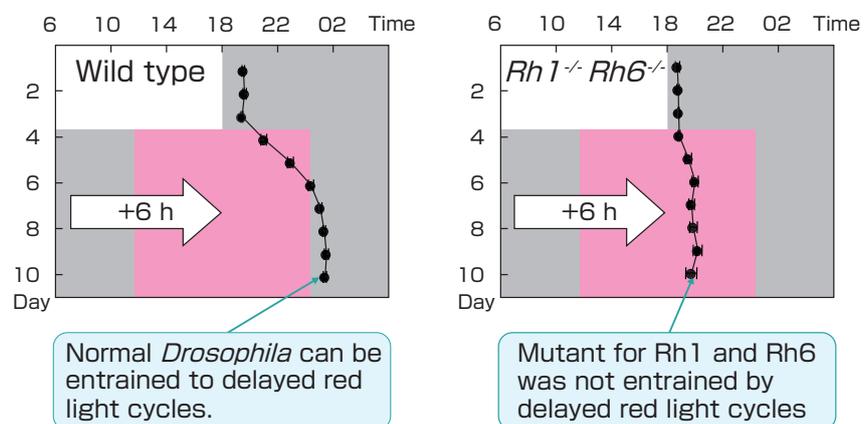


Ketogenic diet advances the phase of circadian clock in mice.

## An invisible-light-entrainment mechanism of the *Drosophila* circadian rhythm

### Re-entrainment of *Drosophila* rhodopsin mutants to red-LED light

Like most other insects, *Drosophila* visual system is insensitive to red light. However, *Drosophila* circadian rhythms can be entrained by red light. Here, we discovered the corresponding organs and rhodopsins responsible for the circadian entrainment of *Drosophila* to red light using rhodopsin mutants and red light emitting diodes (LEDs). The entrainment by red light was not observed in eyeless mutants of *Drosophila*. We also found that double Rhodopsin 1 (Rh1) and Rhodopsin 6 (Rh6) mutants were not entrained to red light, while single Rh1 mutants and single Rh6 mutants were both entrained to red light. These results indicate that *Drosophila* can entrain the circadian rhythm to red LED light through Rh1 and Rh6 in compound eyes.



**Shuji Hanai**

Institute for Biological Resources  
and Functions

s-hanai@aist.go.jp

AIST TODAY Vol.9 No.12 p.18 (2009)

Entrainment of normal and rhodopsin mutant by red light  
Off-set of locomotor activity was shown

## Multimedia content retrieval using automatic speech recognition

### Sub-word based realization of speech search without any restriction of vocabulary, grammar, or language

We have developed a technology to retrieve multimedia content directly using speech from collections of speech and/or multimedia data.

In addition, we have released it to the public on an open website for a verifying experiment since last year (<http://www.voiser.jp>). This system searches for keywords in speech contained at such multimedia content at video sites and speech sites on the Internet, and is characterized by the ability to search without a dictionary and to use any word as keyword without restriction. The ability was realized by unique universal-code based recognition units that AIST has been developing for speech recognition. The system breaks down the speech into sub-phonetic segments (SPSs) for recognition, and applies search processing to the coded SPSs to achieve high search performance. The system makes it possible to search a large amount of multimedia content containing proper nouns and new words on the Internet in real time without any further maintenance. Practical application of this technology enables users to efficiently extract multimedia information and enormously expands the possibility of creating new values for a huge amount of unused multimedia content.

**Shi-wook Lee**

Information Technology Research Institute

[s.lee@aist.go.jp](mailto:s.lee@aist.go.jp)

AIST TODAY Vol.9 No.12 p.19 (2009)



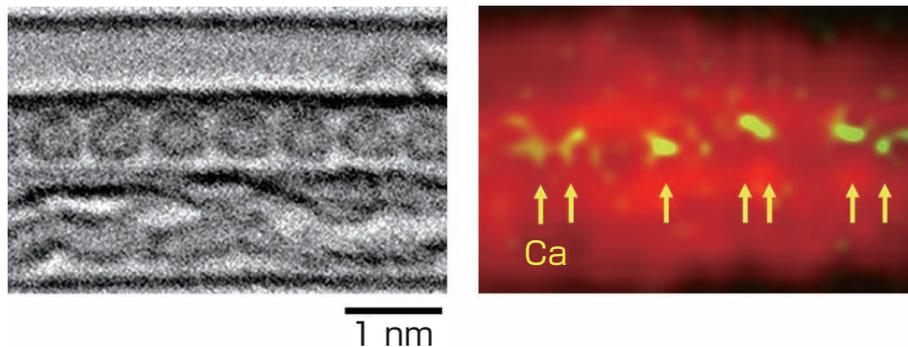
**Demonstrative website which can do pinpoint search of video contents**  
(<http://www.voiser.jp>)

## Nanotechnology, Materials and Manufacturing

## Imaging of single calcium atoms using electron energy-loss spectroscopy

### Low voltage microscope enables structure analysis without destruction

We have developed a low-voltage electron microscope with a newly designed aberration correction system. The accelerating voltage of transmission electron microscope has been substantially reduced to 60 kV from 200-300 kV which used to be required to obtain the resolution of 0.1 nm. Here we demonstrate the first example of low-voltage microscopy imaging of individual molecular structure without massive destruction. Combined with electron energy-loss spectroscopy (EELS), the single atoms encaged inside carbon nanotubes have been successfully identified. Single calcium (Ca) atoms inside the nanotubes were unambiguously identified for the first time using EELS.



**Scanning transmission electron microscope (STEM) image of Ca peapod (left) and its chemical map (right)**  
Ca map (yellow) and carbon map (red) are shown. Seven Ca atoms are indicated by arrows.

**Kazutomo Suenaga**

Nanotube Research Center

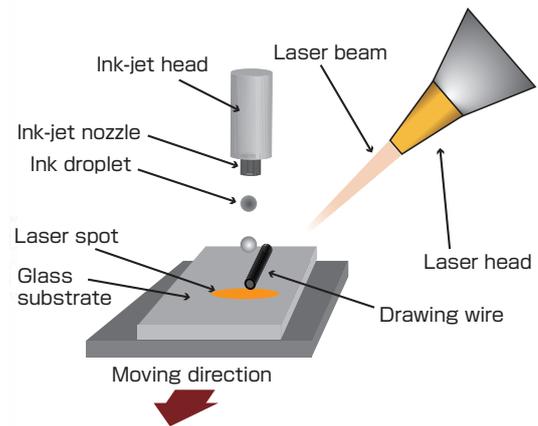
[suenaga-kazu@aist.go.jp](mailto:suenaga-kazu@aist.go.jp)

AIST TODAY Vol.9 No.10 p.19 (2009)

## Successful formation of fine and high aspect ratio patterning with laser assisted ink-jet printing

### Industrial ink-jet printing for low resistance wiring on various kinds of substrates

In order to reduce the line width and to improve the patterning throughput on ink-jet printing, a CO<sub>2</sub> laser was irradiated during printing. This method is named laser assisted ink-jet printing (LIJ). The drawing conditions such as droplet size, ejection frequency, laser irradiation power, and stage speed were optimized. As a result, fine line patterns of silver (Ag) with 7~10 μm width and over 10 μm thickness were obtained by using the 25 μm droplets in diameter, which were ejected from a single nozzle head. The aspect ratio and the drawing speed of the Ag line reached over 1:1 and 10 mm/sec (60 cm/min) without a re-coating process. The line width with laser irradiation was 20~25 times smaller than that without laser irradiation. The thickness with laser irradiation was over 10 times larger than that without laser irradiation.



Laser assisted ink-jet printing

**Jun Akedo**

Advanced Manufacturing  
Research Institute

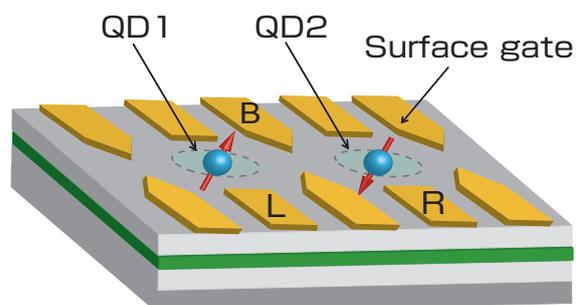
akedo-j@aist.go.jp

AIST TODAY Vol.9 No.10 p.20 (2009)

## Electrical measuring method for quantum states

### Applications in quantum information processing are expected

We have established the theoretical foundation for a method to electrically measure the quantum superposition of the two spin states (singlet and triplet) of two electrons captured in a semiconductor double quantum dot. With conventional methods, only the probability of singlet and triplet spin states could be measured. Since the new method can also detect the quantum-mechanical relative phase of the two states, the two-electron spin states are fully measured. This method is valuable not only in the context of basic physics but also as a means for confirming the initialization of the quantum states and reading out the computed results that are necessary to develop a quantum information processing device. Therefore, this method is expected to contribute to the development of quantum information processing devices based on semiconductor artificial molecules.



**Schematic illustration of a double quantum dot**

Electrons are represented by the blue spheres and their spins are indicated by the red arrows. The regions where electrons are confined are represented by the dotted circles. Surface gates are indicated by the yellow plates. The double quantum dot is connected to the source and drain electrodes.

**Hiroshi Imamura**

Nanotechnology Research Institute

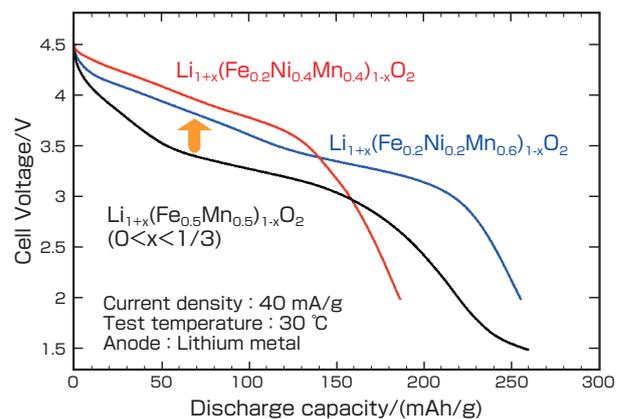
h-imamura@aist.go.jp

AIST TODAY Vol.9 No.11 p.21 (2009)

# Development of cobalt-free oxide positive electrode material for lithium-ion batteries

## Realization of high discharge voltage close to that of existing positive electrode material

We have developed two cobalt-free positive electrode materials,  $\text{Li}_{1+x}(\text{Fe}_{0.2}\text{Ni}_{0.2}\text{Mn}_{0.6})_{1-x}\text{O}_2$  and  $\text{Li}_{1+x}(\text{Fe}_{0.2}\text{Ni}_{0.4}\text{Mn}_{0.4})_{1-x}\text{O}_2$  ( $0 < x < 1/3$ ) containing a cheap and environmental-friendly element, iron (20 % of total transition metal content), using the coprecipitation – hydrothermal – calcination method. Both materials have not only high specific capacity of above 180 mAh/g, but also high average discharge voltage of 3.5–3.7 V. The observed discharge voltage is comparable with those of existing positive electrodes like  $\text{LiNi}_{1/2}\text{Mn}_{1/2}\text{O}_2$  and  $\text{LiNi}_{1/3}\text{Mn}_{1/3}\text{Co}_{1/3}\text{O}_2$  (4.0 V) and higher than that of our previously developed material  $\text{Li}_{1+x}(\text{Fe}_{0.5}\text{Mn}_{0.5})_{1-x}\text{O}_2$  (3.0 V). We believe that the utilization of iron in oxide-based positive electrodes will be accelerated by using these newly developed materials.



**Mitsuharu Tabuchi**

Research Institute for Ubiquitous  
Energy Devices

m-tabuchi@aist.go.jp

AIST TODAY Vol.9 No.12 p.20 (2009)

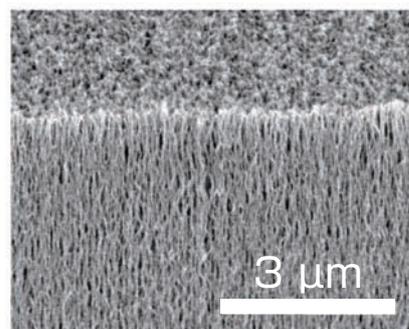
Initial discharge curves of two newly developed positive electrodes (red and blue) and a previously developed one (black) after charging up to 5 V

Metrology and Measurement Science

# A black coating developed using structured carbon nanotubes

## An excellent light absorber and thermal radiator like a black body

We have developed an excellent black coating which absorbs light almost perfectly across a very wide spectral range from ultra-violet to far-infrared. This black coating is fabricated with vertically-aligned single-walled carbon nanotubes (SWCNTs) which are directly synthesized on substrates by *Super-growth*, a novel synthesis method developed by AIST. Owing to its nanometer-scale structure, the SWCNT coating exhibits high light absorption rate of 98-99 % uniformly in an extensive spectral range from 0.2  $\mu\text{m}$  to 200  $\mu\text{m}$ . This optical feature is quite similar to the black body, a theoretical material that absorbs all incident light. From an industrial viewpoint, this black coating will contribute to improvements in solar energy conversion, infrared detector, heat release in electronic devices etc. Metrologically, better black coating is also expected especially in the infrared region in order to fully cover the Planckian curve.



Scanning electron microscope (SEM) image of the black coating

**Kohei Mizuno**

Metrology Institute of Japan

k-mizuno@aist.go.jp

AIST TODAY Vol.9 No.10 p.21 (2009)

# Simultaneous emission of infrared free electron laser light and a quasi-monochromatic X-ray beam

## Development of a two-color light source of infrared light and X-ray with variable energy

We have developed a free electron laser (FEL) with a compact storage ring in the infrared region. The wavelength of infrared FEL light was in the region of 0.84–1.50  $\mu\text{m}$ . The maximum power of the infrared FEL light transmitted through an optical cavity was about 1.6 mW, and the intracavity power was about 5W. We have also developed intensive quasi-monochromatic X-ray by using FEL Compton backscattering. The yield of the generated X-ray beam is roughly  $10^6$  photons/s with the energy of 1.2-2.1 MeV. A prospective application of the present system would be a high-flux two-color light source of infrared light and an X-ray with variable energy.

If applied to the accelerator such as an energy-recovery linac, we would be able to realize a near monochromatic X-ray source with the energy of over 0.3 MeV and the yield of  $10^{12}$  photons/s, of the level not easily attainable even in a large synchrotron radiation facility. This type of X-ray beam would offer new measuring methods for magnetic material research, and would also contribute to the development of an ultra-high density magnetic recording system.

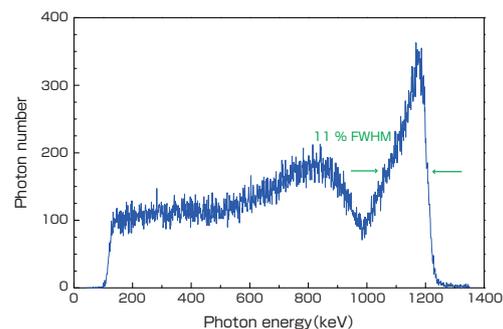
**Norihiro Sei**

Research Institute of  
Instrumentation Frontier

sei.n@aist.go.jp

AIST TODAY Vol.9 No.11 p.22 (2009)

Example of an energy spectrum  
of an X-ray beam by Compton  
backscattering



# High-precision frequency measurement using an optical fiber link

## Remote absolute frequency measurement of a strontium optical lattice clock

The precise measurement of time and frequency, which has the smallest uncertainty ( $10^{-15}$ – $10^{-18}$ ) of all types of measurement, is of great interest for a wide range of applications including basic science, metrology, broadband communication networks and navigation with the Global Positioning Systems (GPS). The recent development of optical frequency measurement based on femtosecond combs has stimulated the field of frequency metrology, especially research on optical frequency standards. One major challenge for scientists working on high-precision frequency standards and measurements is to deliver and compare state-of-the-art clocks at different locations. Here we demonstrate a precise frequency measurement over a physical distance of 50 km between Tokyo and Tsukuba using a phase-stabilized 120 km optical fiber link and coherent optical transfer. The transition frequency of the strontium optical lattice clock at the University of Tokyo is measured to be 429228004229874.1(2.4) Hz. The results demonstrate the excellent functions of the intercity optical fiber link, and the clear potential of optical lattice clocks for use in the redefinition of the second.

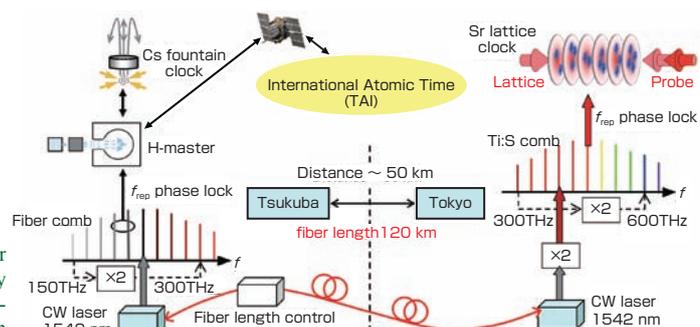
**Feng-Lei Hong**

Metrology Institute of Japan

f.hong@aist.go.jp

AIST TODAY Vol.9 No.11 p.23 (2009)

Optical carrier transfer  
and absolute frequency  
measurement using a 120-  
km fiber link between  
Tsukuba and Tokyo



# Precise characterization of nanomaterials by pulsed field gradient nuclear magnetic resonance method

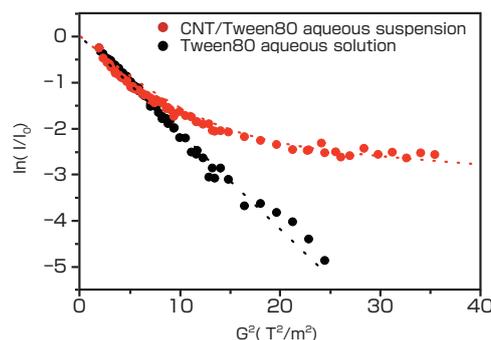
## Accurate size determination and observation of diffusion phenomena of nanomaterials in solution

Pulsed field gradient nuclear magnetic resonance (PFG-NMR) spectroscopy has been developed as a method for quantitative measurements of self-diffusion coefficients of nanomaterials. It is possible to distinguish the individual diffusion components by monitoring NMR signals at different chemical shifts simultaneously.

Accurate diffusion coefficients were obtained using special NMR cells and the precise size determination of nanomaterials was accomplished by extrapolation methods varying both the concentrations of the nanomaterials and the surfactants in aqueous solution. In addition, the slow diffusions of solvent and surfactant molecules in colloidal nanoparticle aqueous dispersion were directly observed. The slow diffusions of molecules were attributed to the strongly adsorbed molecules on the nanomaterials and the amount of bound molecules was estimated (Figure). Our improved PFG-NMR method has promising potentials in the field of the characterization of functional nanomaterials and their nano-toxicity assessments.

### PFG-NMR spin-echo signal attenuation plots for Tween80 molecules

The attenuation plot of Tween80 is approximately a straight line for the Tween80 aqueous solution, indicating one diffusion mode of Tween80 molecules in this solution. The observed signal decays of the Tween80 molecules were nonlinear in CNT/ Tween80 aqueous suspension, indicating some distribution of the diffusion coefficients of the Tween80 molecules. The slow diffusion of Tween80 molecules was attributed to the strongly adsorbed molecules on the CNT in the aqueous suspension.



**Haruhisa Kato**

Metrology Institute of Japan

h-kato@aist.go.jp

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## In Brief

### President Nomakuchi's Visit to the Republic of South Africa —Workshop Held by four Research Organizations—

AIST, during the visit of Mr. Akira Amari, the then Minister of Economy, Trade and Industry to the Republic of South Africa in November, 2007, concluded a memorandum of understanding with Japan Oil, Gas and Metals National Corporation and Council for Geoscience of South Africa (CGS), and has been engaged in collaborative research on rare metal resource evaluation (article in AIST TODAY, 2008-No.28).

In September 2009, when AIST President Tamotsu Nomakuchi attended the General Assembly of the International Organization for Standardization (ISO) in Cape Town, nine AIST researchers accompanied him to Pretoria. President Nomakuchi held separate talks with the Director-General of the Department of Science and Technology and the tops of CGS, Council for Science and Industrial Research (CSIR), and Mintek. A joint workshop was also held by the four organizations.

CSIR could be seen as AIST of South Africa, and is mainly promoting "Type 2 Basic Research" centered on national projects in the fields of environment, energy, materials,

manufacturing technology, ICT, and life science. Mintek is actively engaged in industrial application research focusing around the fields of metallurgy and metallic materials. During this workshop, future research cooperation was discussed, not only in geology where research collaboration is already in operation, but also in the fields of environment, energy,



Dr. Sibusiso Sibisi, President and CEO of CSIR (back row second from right) and AIST President Nomakuchi after their meeting

nanotechnology and materials.

On the first day of the workshop, President Nomakuchi gave an opening speech, followed by overview presentations by each of the four organizations, and this deepened our understanding of each other. On the second day, working sessions in the three fields were held in which participating researchers gave research presentations that furthered our mutual understanding. There were also discussions on the possibility of research cooperation. Lastly, all participants assembled to hear reports of the sectional discussions, and discussed the overall ways of cooperation in the future.

AIST researchers also visited the research facilities of the three organizations and deepened their understanding of the research equipment and environment.

Concerning this visit to South Africa, the Embassy of Japan in South Africa greatly helped in the arrangements with the three organizations, and Mr. Toshiro Ozawa, Ambassador of Japan to the Republic of South Africa and his Embassy gave us total backup during our visit. The expectations of both government members towards AIST's research cooperation are high, and with this visit, we will further advance research cooperation with South Africa.

## Thai Minister of Science and Technology Visits AIST Tsukuba —Ceremony for the Renewal of Comprehensive Memorandum of Understanding —

On October 7, 2009, Dr. Kyunying Kalaya Sophonpanich, Minister of Science and Technology of Thailand visited AIST. After exchanging views with President Nomakuchi on the research activities of AIST and science and technology, she took a tour of the Research Center for New Fuels and Vehicle Technology and Science Square Tsukuba. Dr. Sakarindr Bhumiratana, President of the National Science and Technology Development Agency of Thailand (NSTDA), and Mr. Surapol Vatanawong, Acting Governor of the Thailand Institute of Scientific and Technological Research (TISTR) were also present. A ceremony for the renewal of the comprehensive memorandum of understanding (MOU) signed 5 years ago by the two organizations and AIST was held, and the cooperation into the future was reconfirmed. There were 24 people accompanying the minister including executive officials of the Ministry of Science and Technology of Thailand, NSTDA, TISTR, members of the Royal Thai Embassy in Japan, and people of the press.

Minister Kalaya who was awarded a PhD in nuclear physics in the UK is well-versed in science and technology, and she made comments and asked questions on research activities of AIST with great interest. AIST has had active exchange with NSTDA and TISTR based on the comprehensive MOU signed on November 25, 2004. We have deepened our cooperation through exchange of researchers, Japan International Cooperation Agency (JICA) technical

trainings, management level communication, collaborative researches, and regularly held workshops (six already held), centered on environment, energy, information technology, nanotechnology, and materials. It was extremely meaningful that the renewal of the MOU was completed during the visit of Minister Kalaya.

AIST, along with NSTDA and TISTR, is working on a five-year Japan Science and Technology Agency (JST)/JICA Project "Innovation on Production and Automotive Utilization of Biofuels from Non-food Biomass" starting this year. The Thai side was much interested in such topics as the introduction of non-food biofuels in the transport sector, climate change mitigation measures, manufacturing technology of transportation fuel, engine evaluation, and fostering biofuel researchers who can work autonomously. Minister Kalaya listened attentively to the presentations at the relevant research sites of AIST.

Through this visit by the minister and the renewal of the comprehensive MOU, we reconfirmed the importance of promoting mutual collaboration. Through strengthening research collaboration between Thailand and AIST, we aim to contribute to the prevention of global warming, environmental improvement, and to solving the energy problem, as well as the economic development not only in the two countries but also in Asia.



AIST President Nomakuchi (left) and Science and Technology Minister Kalaya Sophonpanich of Thailand (right)



The renewal of the comprehensive MOU (front row from left: NSTDA President Sakarindr, AIST President Nomakuchi, TISTR Acting Governor Surapol)

# Third Workshop between AIST and Department of Biotechnology of India

Under the Joint Statement Towards Japan-India Strategic and Global Partnership of December, 2006 signed by the prime ministers of India and Japan, AIST concluded a comprehensive memorandum of understanding (MOU) with the Department of Biotechnology (DBT) of the Ministry of Science and Technology of India in February, 2007. Based on this MOU, it was agreed that research cooperation would be promoted between DBT and AIST in a matching fund system in the three fields of bioinformatics, medical glycoscience, and cell engineering, and the first workshop between AIST and DBT was held in Tsukuba in January, 2008. Later, upon the project proposal of the Computational Biology Research Center, AIST, DBT publicly sought organizations in India; an international selection committee was organized; four subjects and six organizations from over 50 applications were selected; and collaborative researches were started in 2009.

At the second workshop held in Hyderabad in November, 2008, there were project proposals on medical glycoscience and cell engineering made by AIST, and exchanges have been promoted between researchers of AIST and of research organizations under DBT.

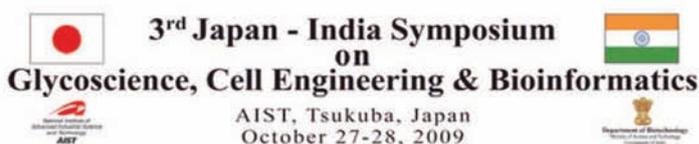
In response to these achievements, the third workshop was held in Tsukuba on October 27 and 28, 2009. For the symposium held on October 27, there were over 90 participants, and there were presentations of the research of both countries on the three topics of medical glycoscience, cell engineering, and bioinformatics. At the poster session, there was much serious discussion, and there was active

interaction among the researchers.

At the bilateral meeting held on October 28, we confirmed the specific progress made on these individual themes of collaboration, and discussed ways of further advancing the research cooperation in the future.

In bioinformatics, there were reports on the progress and future developments of the collaborative research by researchers of AIST and participating research organizations of India. In medical glycoscience and cell engineering, information exchange was started with research organizations under DBT based on the project proposal made by AIST in February, 2009. This workshop served to advance research exchange and coordination among researchers of AIST and participating research organizations of India. It was confirmed that specific research contents would be reviewed by December, 2009, and that the individual research cooperation agreements were planned to be concluded in 2009.

When the first bioinformatics project proposal was made, DBT selected research organizations through open-application in India. DBT has evaluated the results of interaction among researchers encouraged through the three workshops, and has announced that it will financially support specific parties that have already advanced research exchanges. At the same time, DBT has indicated its intentions to distribute budget likewise to universities in India which already have active ties with AIST, thus strengthening the support system for research exchange with AIST.



**3<sup>rd</sup> Japan - India Symposium**  
on  
**Glycoscience, Cell Engineering & Bioinformatics**  
AIST, Tsukuba, Japan  
October 27-28, 2009



Symposium attendees and logo

## Cover Photos

Above: Mineral ore at Thor Lake (p. 7)

Below: The storage ring NIJI-IV dedicated to free electron lasers (p. 21)

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Publication Office, Public Relations Department  
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AIST Tsukuba Central 2, 1-1-1 Umezono, Tsukuba, Ibaraki 305-8568, Japan  
TEL: +81-29-862-6217 FAX: +81-29-862-6212 Email: [prpub@m.aist.go.jp](mailto:prpub@m.aist.go.jp) URL: <http://www.aist.go.jp/>

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