ISSN 1346-602X

*Full Research* in Society, for Society

# AIST
National Institute of Advanced Industrial Science and Technology
# TODAY
International Edition

2013
2013-3
No.49

FEATURE

## Rare Metals 3
Measures for Unstable Supply of Rare Metals Vital to the Development of Next-Generation Vehicle Industries

## Research Hotline
UPDATE FROM THE CUTTING EDGE（April−June 2013）

## In Brief

# Rare Metals 3

— Measures for Unstable Supply of Rare Metals Vital to the Development of Next-Generation Vehicle Industries —

## Trends in Rare Metals in the Turbulent World Situation and Challenges for the Future

### Uncertainty in the global economy

With the world's political and economic landscape undergoing major changes, there have been several events that have significantly influenced the supply and demand of rare metals during the past few years.

The first event was the deceleration in growth and destabilization of the global economy caused by the European sovereign debt crisis, which has continued since 2010 in the wake of the Lehman Shock. Since the late 1990s, the global economy has been changing dramatically due to various factors such as increases in production capacity and sustainable market expansion resulting from rising income levels in emerging nations such as China and the Southeast Asian countries, and the repeated emergence and collapse of economic bubbles in developed countries. Under these circumstances, the prices of rare metals, whose markets are small, are quite volatile because they are susceptible to the rapid, profit-oriented movements of speculative transactions, although the long-term trend of prices is upward.

### Exposure of the risk involved in rare-earth resources

The second event was the dispute between Japan and China over the collision of a Chinese fishing boat with a Japanese patrol vessel that took place near the Senkaku Islands on September 7, 2010. After this incident, China stopped its exports of rare-earth resources to Japan for more than three months, which increased their prices until they reached a peak in July 2011. The highest price of dysprosium recorded at that time was about 10 times that in August 2010. The exposure of such a supply risk surprised the world, and many countries began efforts to reopen rare-earth mines, while several projects were initiated to develop technologies for alternative materials or reduce the use of rare earths. After some time, prices decreased to a certain extent, but as of July 2012 they were still at much higher levels than before the price hike. Japan, the U.S., and the EU have filed a joint case with the World Trade Organization against China's export restrictions on rare-earth resources.

### Impact of the shale gas revolution

The third event was the ongoing large-scale production of shale gas, an unconventional form of natural gas, in the U.S. Expectations that the U.S. would become a major importer of natural gas in the near future have been replaced by the possibility of the U.S. becoming a major exporter, a development that is referred to as the shale gas revolution. This revolution is expected to also affect China and the EU because the supply and demand situation for fossil fuels will become less tight, and thus increases in energy prices, one of the decisive factors restricting the economic growth of emerging countries (which are major growth engines of the global economy) will be avoided, at least for the time being. At the same time, eased supply and demand conditions for fossil fuels, and the fact that natural gas emits smaller amounts of $CO_2$ than petroleum during combustion, will slightly lessen the motivation for the dissemination of next-generation automobiles, so that the risk involved in rare-earth resources, which are used in such automobiles, will emerge a little later than expected.

### Rapid globalization of industry changing the meaning of resource supply

The fourth event was the accelerated globalization of production and consumption of industrial goods. In the past, many large Japanese companies manufactured products in Japan and exported them abroad. Due to the yen's sharp appreciation and the rapid expansion of emerging markets, they have been increasingly manufacturing products overseas, which are then sold in those overseas markets, to secure profits. In such cases, large amounts of rare-metal materials need to be supplied to many factories abroad. For example, more than half of the total production of four-wheeled vehicles by Japanese manufacturers is now carried out at overseas plants. To put it the other way around, securing the supply of rare metals for domestic production alone is no longer sufficient for Japanese auto manufacturers doing business worldwide.


For inquiries about this article : Materials Research Institute for Sustainable Development http://unit.aist.go.jp/mrisus/index_en.html

### Research activities related to rare metals for next-generation vehicles

Focusing on the Japanese auto industry, which has a strong competitive edge but faces fierce competition, the feature articles of this issue of *AIST TODAY Intl. Ed.* are dedicated to the research activities of AIST's Rare Metal Task Force in relation to rare metals for next-generation vehicles, ranging from resource exploration to the development of technologies for substituting, reducing the use, and recycling of rare metals. More specifically, we present here the results of research on heavy rare-earth elements that are used in the permanent magnets of motors for electrified vehicles, tungsten used in tools for high-performance and high-precision machining, and platinum-group elements used in catalysts to purify vehicle emissions. We hope you find the articles in this feature interesting.

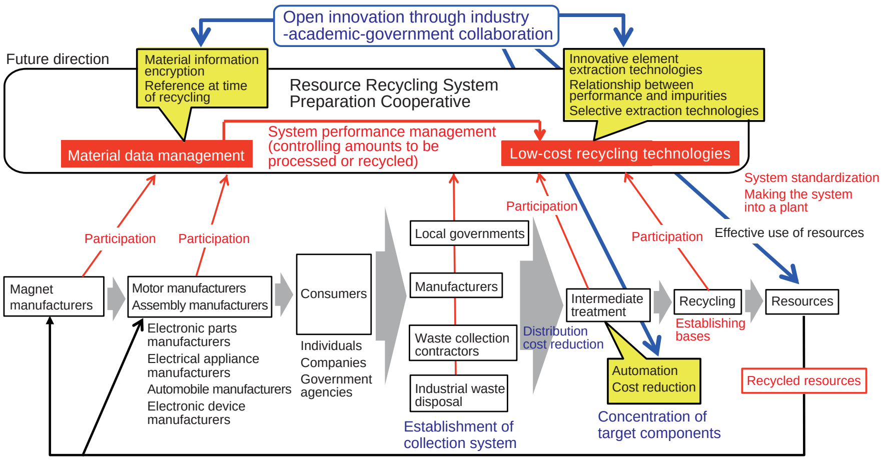

Director
Materials Research Institute for Sustainable Development
**Mamoru NAKAMURA**


# Securing Resources for the Development of Alternative Materials

### Securing rare metals essential for the production of high-performance materials

For many years, Japanese companies have produced competitive products for the global market by developing energy-saving materials and systems and applying them to industrial products. Now, however, with the expanding demand for resources due to the industrialization of countries around the world, ensuring the stable procurement of resources, particularly rare metals or constituent elements, has become indispensable for Japanese companies to continue to produce superior materials and systems.

For example, when producing neodymium-iron-boron (Nd-Fe-B) magnets, one of the high-performance magnets essential for energy-efficient motors, the heavy rare-earth element dysprosium should be added to provide heat resistance. Dysprosium is very rare, as its distribution is extremely uneven, and its price has been climbing. On the other hand, demand for these high-performance magnets has been increasing further because of the dissemination of energy-saving appliances, electrification of vehicles, and diffusion of wind power generation. Although the development of technologies for reducing the use of such heavy rare-earth elements or using alternative materials is being extensively conducted in Japan, more time is required to put these technologies into practical use. In order to respond to the rapidly increasing demand for heavy rare-earth elements, the establishment of a new resource recycling system, which combines resource recovery from used products with technologies to reduce material usage, is the most urgent task.




**Technological challenges for construction of a resource recycling system (high-performance magnets as an example)**

### AIST's recommendations toward the establishment of a resource recycling system

AIST has set up the Rare Metal Task Force, consisting of researchers working in various fields related to rare metals such as the supply and demand balance, resource exploration, recycling, alternative materials, and technologies to reduce material usage, in order to promote comprehensive discussions beyond their respective specialties. The Task Force has so far proposed effective countermeasure technique for each individual rare-metal resource.

In fiscal 2011, a project to examine practical resource recycling systems was established under the Council on Competitiveness-Nippon (COCN) in cooperation with industry. A report on its activities is available on the COCN website, including technical challenges, future directions, and social systems for recycling resources, with a particular focus on a heavy rare-earth element used for magnets and lithium for secondary batteries as resources requiring immediate action.

Industry is strongly urging AIST to develop technologies for the establishment of a comprehensive resource recycling system including (1) efficient identification of target resources in used products, (2) effective and innovative separation and recovery for target resources, and (3) peripheral technologies such as joining, surface treatment, and so on, which never cause deterioration of recovered resources. In response to these requests, AIST and the Task Force have initiated a research project for the establishment of a resource recycling system as shown in the figure, using the example of high-performance magnets as a model. Based on discussions at COCN, utilizing seed technologies developed by universities from the initial phase of the project, and in collaboration with the private companies and government sectors, AIST hopes to propose a practical social system for the recycling of resources that can be achieved as early as possible. AIST wishes to widely invite participants who would like to contribute to these research activities. If you are interested, please contact AIST's Materials Research Institute for Sustainable Development.

Deputy Director
Materials Research Institute for
Sustainable Development
**Keizo KOBAYASHI**

# Magnets Free of Heavy Rare-Earth Elements
# – Fabrication of Sm-Fe-N bulk compact and its characteristics –

## High-performance magnets for next-generation vehicles

Next-generation vehicles, such as hybrid and electric vehicles, do not use a gasoline engine as their main power source. Such vehicles have been rapidly spreading in recent years, to the extent that we can no longer really refer to them as "next-generation" vehicles. Among numerous advanced technologies applied in next-generation vehicles, the key is interior permanent magnet (IPM) motors, which are small but highly efficient motors. As the name indicates, magnetic materials are used to achieve the performance of the motor. Currently, most IPM motors use neodymium-iron-boron magnets, the strongest type of magnet.

High-performance magnets used for automobile motors must be able to withstand heat and a reverse magnetic field, and generate a high magnetic field. However, the Nd-Fe-B magnet is, in fact, weak against heat by nature and its magnetic performance drops sharply at temperatures exceeding 100 °C. To improve its heat resistance, dysprosium (Dy), a heavy rare-earth element, needs to be added. Since Dy is rarely found in the Earth's crust and is produced in very few countries, it faces the risk of depletion and rising prices. Against this backdrop, both the public and private sectors in Japan are conducting research to reduce the amount of Dy used, and have recently succeeded in developing a technology to reduce the amount of Dy to be added by nearly half, to 4 % by weight. Even so, given the current dissemination of next-generation vehicles worldwide and the expanded use of IPM motors in wind power generators, the risk of Dy depletion is still present. The development of Dy-free high-performance magnets is therefore an urgent necessity.

## Sm-Fe-N magnets as a Dy-free magnet candidate

Under such circumstances, our research group is now focusing on samarium-iron-nitrogen (Sm-Fe-N) compounds for the realization of a Dy-free magnet. As the comparison in Table 1 shows, both the spontaneous magnetization, $J_S$,


For inquiries about this article : Materials Research Institute for Sustainable Development http://unit.aist.go.jp/mrisus/index_en.html

and anisotropy field, $H_a$, of Sm-Fe-N compounds are equivalent to or higher than those of the Nd-Fe-B compound. No other compound has such high levels of both $J_S$ and $H_a$, suggesting that Sm-Fe-N compounds have the highest potential as a Dy-free magnet material. In particular, since the Curie temperature, $T_C$, of Sm-Fe-N exceeds that of Nd-Fe-B by more than 150 °C, Sm-Fe-N is expected to exhibit higher heat resistance without the need to use any heavy rare-earth elements.

There is a major problem with Sm-Fe-N, however. IPM motors normally use magnets that are produced by sintering powdered magnetic materials, but Sm-Fe-N is thermally decomposed at around 550 °C, making it difficult to use as a magnetic material subject to sintering under high temperatures.

**Table 1 Magnetism of representative compounds**

| | $J_S$ [T] | $H_a$ [MA/m] | $T_C$ [°C] |
|---|---|---|---|
| $Nd_2Fe_{14}B$ | 1.61 | 6.13 | 312 |
| $Sm_2Fe_{17}N_3$ | 1.57 | 20.69 | 476 |
| $Sm_1Fe_9N_{1.5}$ | 1.70 | 6.13 | 520 |
| $Sm_1Co_5$ | 1.07 | 32.07 | 747 |
| $Sm_2Co_{17}$ | 1.22 | 5.17 | 917 |
| FePt | 1.43 | 9.15 | 477 |
| Ferrite | $< 0.5$ | $< 1.60$ | $< 650$ |

$J_S$ : Spontaneous magnetization $H_a$ : Anisotropy field $T_C$ : Curie temperature

## Development of low-temperature sintering technology

In order to sinter Sm-Fe-N powder below its decomposition temperature, we have developed a low-temperature sintering technology called the high-pressure current sintering method. Detailed information on this technology is given elsewhere.[1] Briefly, raw powder filled in a mold are heated and sintered by applying a current to the mold under high pressure. While the conventional sintering method densely consolidates powders by heating them to high temperatures, causing mass transfer, this high-pressure current sintering method employs high pressure to densify the powders and the electric current is used to mainly bond the powder particles, thus achieving sintering at a low temperature. In reality, in order to sinter under high pressures exceeding 1 GPa with a high level of reproducibility, the development of various technologies was required such as current-sintering devices that can precisely control high pressures, and molds that can withstand high pressures.

## Sm-Fe-N isotropic bulk magnet

The high-pressure current sintering method has enabled us to sinter and consolidate isotropic Sm-Fe-N powders at 400 °C, which is below their decomposition temperature. Previously, unless a special method was used such as compacting powder with explosive and impact pressures, the relative density of sintered Sm-Fe-N compact was about 85 % at most, but a density of 90 % or more has been achieved using this new technology. As a result, $(BH)_{max}$, an indicator of magnet performance, is 135 kJ/m$^3$, the world's highest level for isotropic magnets.

Besides magnet performance, Sm-Fe-N sintered magnets are expected to provide high heat resistance. As shown in Fig. 1, the magnetic field (magnetic flux density) of an Nd-Fe-B magnet sharply decreases when it is heated, and the addition of Dy can reduce this loss of density. Meanwhile, although the magnetic flux density of a fabricated Sm-Fe-N magnet is very low at room temperature due to its isotropic nature, its rate of reduction with respect to temperature is also low, and is equivalent to the magnetic flux density of an Nd-Fe-B anisotropic magnet with 3 % Dy at 200 °C. Figure 2 shows this in an intuitive manner. When an Nd-Fe-B magnet (at the tip of the arrow) is heated by a dryer, its magnetic property decreases and many of the iron balls attracted by the magnet fall. In contrast, this is not the case for a Sm-Fe-N magnet.

## Aiming at the development of high-performance Dy-free sintered magnets

It has been demonstrated that Sm-Fe-N exhibits excellent heat resistance without the use of any heavy rare-earth element once it is sintered. If we can fabricate Sm-

Fe-N anisotropic sintered magnets with a magnet performance higher than that of isotropic magnets, they may be able to be used as heavy rare-earth-free magnets comparable to Nd-Fe-B anisotropic magnets. Recent research results[2] have highlighted the need to develop powders suited to sintering for the production of anisotropic sintered magnets, which is one of our current research topics.

Phase Engineering for Advanced Materials Group,
Materials Research Institute for Sustainable Development
**Kenta TAKAGI**

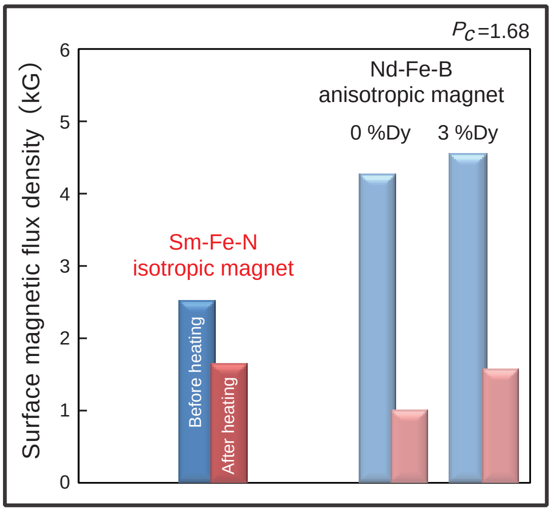



**Fig. 1 Comparison of changes in magnetic flux density between Sm-Fe-N isotropic magnet and Nd-Fe-B anisotropic magnets when heated (at 200 ℃)**

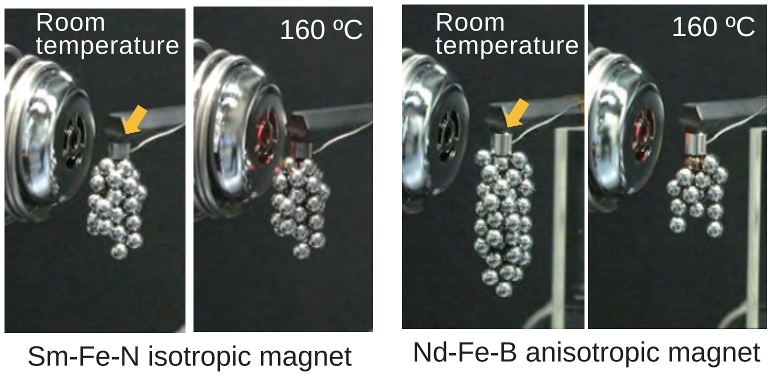



**Fig. 2 Number of iron balls attracted by heated Sm-Fe-N and Nd-Fe-B sintered magnets**

References
[1] K. Takagi *et al.*: *AIST TODAY*, 12 (6), 22 (2012).
[2] K. Takagi *et al.*: *J. Magn. Magn. Mater.*, 324 (15), 2336-2341 (2012).

# Recycling of Rare-Earth Elements from Neodymium Magnets

## Recycling of neodymium magnets

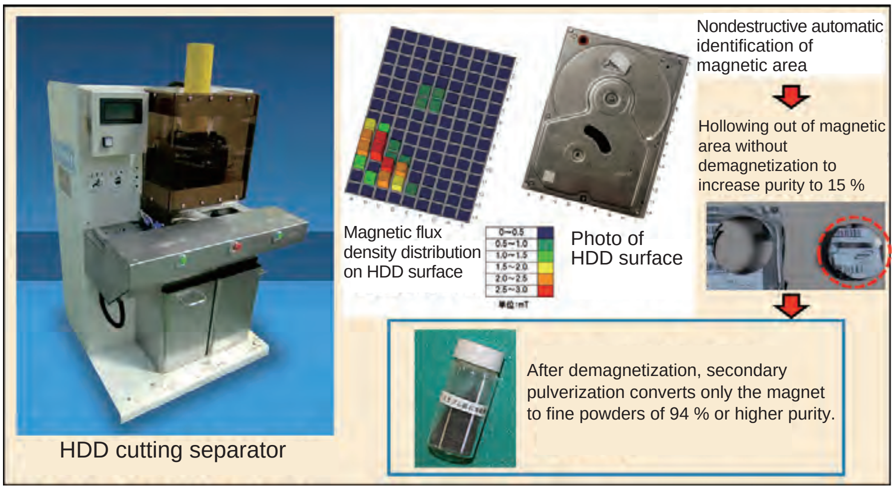
Neodymium magnets are indispensable to our daily lives as they are used in motors for hard disk drives (HDDs), hybrid vehicles, industrial robots, etc. Among five elements designated by the Japanese government as priority elements for recycling, neodymium magnets contain two: neodymium and dysprosium. The recycling of neodymium magnets therefore needs to be accelerated. Neodymium magnets are mostly used in electrical or electronic appliances such as HDDs and air-conditioning compressors. Since the individual neodymium magnets used in these applications are small in size, generally they are manually recovered from used appliances.

## Recovery of magnetic alloy materials from HDDs

In an attempt to establish an economically feasible mass processing and recycling system, the Research Institute for Environmental Management Technology is studying how to mechanize and automate such manual operations. We have jointly developed with the private sector a special HDD cutting separator to recover magnets from HDDs. This separator detects leakage magnetic fields on the surface of the HDD in a nondestructive manner to locate the magnet then hollows out the area containing the magnet, thereby increasing the purity of the magnet obtained from 1 or 2 % to approximately 15 %. The targeted area is then subjected to demagnetization and secondary pulverization to selectively pulverize the magnet only, producing magnetic alloy materials (magnetic powders) with a purity between 94 and 97 %.

### Separation of rare-earth elements through a hydrometallurgical process

Our group is also studying methods to chemically treat magnetic powders recovered from HDDs in the manner described above to separate and recover neodymium and dysprosium contained in magnets. In this process proposed jointly with Japan Oils, Gas and Metals National Corporation and Tohoku University, magnetic powders are heated and oxidized to convert iron, which accounts for more than 60 % of the magnet, to iron oxide, which is poorly soluble in acid, so that only rare-earth elements are selectively dissolved in the acid. To separate and purify metals from the solution, the solvent extraction method is used. The appropriate selection of the extractant and pH enables us to first extract only the heavy rare-earth element dysprosium. According to our experiments, the extraction percentages of dysprosium and neodymium after the first operation were 97 % and 6 %, respectively. The small amount of neodymium thus extracted can be removed by washing the organic phase. A pure dysprosium solution can be obtained by back-extracting the washed organic phase with acid, regenerating the extractant at the same time. After the extraction of dysprosium, neodymium can be extracted from the remaining solution, and, in the same manner, a pure neodymium solution can be obtained by back-extraction. When oxalic acid is added to the solution containing these rare-earth elements, they are precipitated and separated as rare-earth oxalates, which are then calcined to recover highly pure rare-earth oxides.




Two-stage pulverization process for recovery of magnetic powders using an HDD cutting separator

Advanced Recycling Technology Research Group,
Research Institute for Environmental Management Technology
**Tatsuya OKI**

Deputy Director
Research Institute for Environmental Management Technology
**Mikiya TANAKA**

## Efforts to Reduce the Amount of the Platinum Group Metals Used in Diesel Exhaust Purification Catalysts

### Need to develop technologies to reduce the use of platinum group metals

The supply of platinum-group metals (PGMs), such as platinum (Pt), palladium (Pd), and rhodium (Rh), is unstable because they are produced only in limited regions of the world such as South Africa. The major industrial application of these PGMs is in exhaust purification catalysts. Since they are expensive, research and development of technologies to reduce their amounts used in three-way catalysts for gasoline-fueled vehicles, for which demand is strong, has been conducted over the years, and the results are being applied to actual vehicles. On the other hand, R&D is lagging behind in the field of catalysts for large-sized diesel vehicles such as trucks. Since long-distance transportation both in Japan and other countries depends on large-sized diesel vehicles, technologies to reduce the use of PGMs for their exhaust purification catalysts should also be developed.

### Targeted materials in NEDO project

A joint project, "Development of Technology to Reduce the Use of PGMs in Diesel Exhaust Purification Catalysts" (2009-2013), is conducted by AIST, catalyst and material manufacturers, and universities under the framework of the Rare Metal Substitute Materials Development Project commissioned by the New Energy and Industrial Technology Development Organization (NEDO). The Research Center for New Fuels and Vehicle Technology, Research Institute for Innovation in Sustainable Chemistry, and Materials Research Institute for Sustainable Development of AIST are participating in this project.

Figure 1 shows a schematic diagram of an exhaust purification catalyst system for large-sized diesel vehicles. A diesel oxidation catalyst (DOC) is placed at the inlet of the exhaust gas to oxidize hydrocarbons (HCs), carbon monoxide (CO), and nitric oxide (NO). In the second stage, a diesel particulate filter (DPF) is inserted to effectively collect and burn soot, and in the third stage, selective catalytic reduction (SCR) is employed to selectively reduce nitrogen oxides (NOx) to nitrogen. Of these three catalysts, the DOC and DPF use a large amount of PGMs. The above-mentioned joint project aims to reduce the amount of PGMs used in these two catalysts by 50 %.

## Research work at the Materials Research Institute for Sustainable Development

Next, let’s look at the research and development of technologies to control catalyst degradation by using composite nanoparticles, which the Materials Research Institute for Sustainable Development is working on under this project. The amount of PGMs used in a catalyst is determined by the catalyst performance not at the initial stage, but after long-term use, because catalyst metal particles gradually coagulate during use due to various factors such as exposure to high temperatures. Since the surface area of the metal is important for the catalytic reaction, the performance will deteriorate if the metal surface area is reduced due to the coagulation of metal particles. Therefore, a large amount of PGMs has to be used at the beginning in order to satisfy emission standards even after long-term use.

Our research group is working to control the degradation of catalyst performance by using composite nanoparticles consisting of two or more metal elements supported by a porous carrier. The theory behind this is to control the coagulation of metal particles by combining elements that can effectively control the movements of metals under high temperatures and carrying them in the fine pores of the catalyst carrier (Fig. 2). Applying this technology, we have developed a DOC that uses 40 % less PGMs but whose performance is at the same level as commercially available catalysts. For the remaining two years of the project, we plan to further develop this technology and establish a mass production method for the DOC.

Catalytic Nanomaterials Group,
Materials Research Institute for Sustainable Development
**Yutaka TAI**

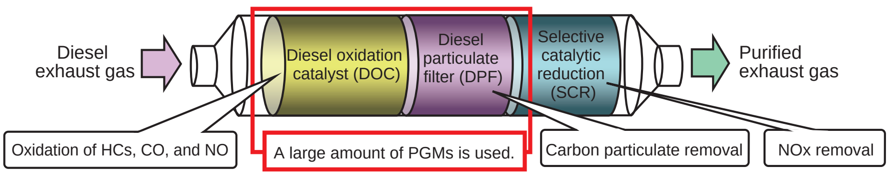



**Fig. 1 Schematic diagram of exhaust purification catalyst system for large-sized diesel vehicles**

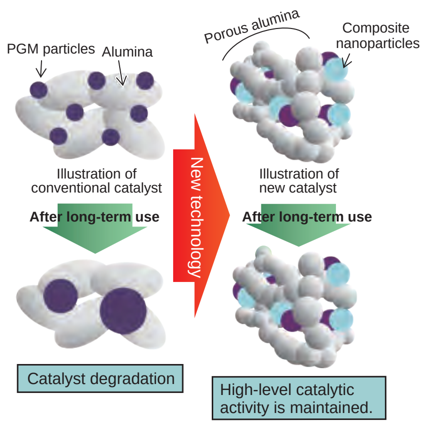



**Fig. 2 Control of catalyst degradation by technology to carry composite nanoparticles**

 For inquiries about this article : Materials Research Institute for Sustainable Development http://unit.aist.go.jp/mrisus/index_en.html

# Development of Tool Materials Using Smaller Amounts of Rare Metals
# – Addressing new work materials such as CFRP and improvement of cermet performance –

## Development of WC-FeAl as a hard material to replace cobalt

In recent years, carbon-fiber-reinforced plastic (CFRP) has been attracting attention as a structural material for transportation equipment such as aircraft and automobiles. Compared with other major structural materials, CFRP is light, highly rigid, and strong. Aircraft fuel efficiency has been improving as a result of reductions in aircraft weight, and in the case of Boeing's 787, which went into service in the autumn of 2011, CFRP accounts for 50 % of the weight of the aircraft. In addition to rigidity, CFRP is required to satisfy stringent standards in terms of precision and surface roughness when used as a structural material. Currently, CFRP is cut using rotating tools, but cutting gives rise to several problems such as an increase in cut surface roughness when the cutting feed rate is increased, possible delamination, and the expense of the tools. These problems are hindering the wider use of CFRP.

Carbide tools or diamond-coated carbide tools are normally used for cutting CFRP. Since CFRP itself is very rigid and strong, the blade edges easily wear out and coating films often come off, significantly shortening the life of the tools. Moreover, plastic tends to degrade during cutting due to the heat generated by the friction between the tool and the CFRP. The materials used for CFRP cutting tools must therefore have high hardness and high thermal conductivity, as well as excellent adhesion with coating films.

The Materials Research Institute for Sustainable Development is conducting research on tungsten carbide-iron aluminide (WC-FeAl), a new hard material, in which cobalt (Co), a rare metal used in WC-Co cemented carbides, is replaced with an intermetallic compound, FeAl. WC-FeAl has excellent oxidation resistance and high-temperature strength, as well as relatively high thermal conductivity; for example, 71 W/mK in the case of WC-15vol%FeAl. The main feature of WC-FeAl is good adhesion with carbon films (Japanese Patent No. 4997561). When a diamond-like carbon (DLC) film was formed on WC-FeAl substrate by sputtering and evaluated for adhesion by scratch testing, its peel strength was found to be up to 30 % higher than that of WC-Co substrate (Fig. 1). We also experimentally fabricated a diamond-coated drill (Fig. 2) and bored a CFRP plate with it. We confirmed the high potential of the drill as a CFRP piercing drill because, when compared with commercially available diamond-coated carbide tools, it created fewer burrs around the bore, no coating film came off, and the blade edge was less nicked. In the future, we will try to identify how such high adhesion is generated and will conduct durability tests in order to put WC-FeAl to practical use.

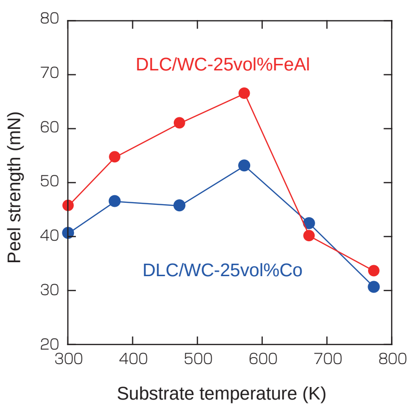



Fig. 1 Adhesion evaluation of DLC film on WC-25vol%FeAl substrate

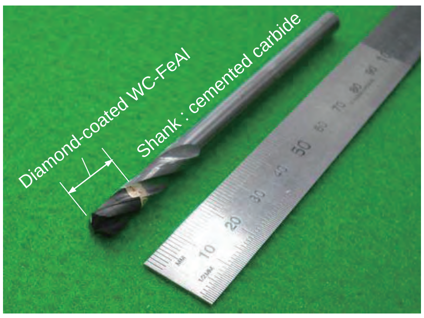



Fig. 2 Experimentally fabricated diamond-coated WC-FeAl drill

## Development of a highly tough cermet as an alternative to tungsten

Tungsten (W), used in WC-Co cemented carbides, is a typical rare metal. W is mostly produced in China and its prices are still at high levels due to China's export restrictions. Under these circumstances, it is very important to develop technologies to reduce the amount of W or to find a substitute for W in carbide tools. The Materials Research Institute for Sustainable Development is working on the development of hard materials that do not use W, or more specifically, cermet-based materials. The basic composition of a cermet (a composite material consisting of ceramic and metallic materials) is titanium-carbonitride nickel (Ti(C,N)-Ni) with molybdenum carbide ($Mo_2C$) added to improve sinterability; i.e., (Ti(C,N)-$Mo_2C$-Ni). Since cermets are less reactive with iron, they are used for finishing steel materials. On the other hand, cermets have lower fracture toughness than cemented carbides, and their applications remain limited.

Our group has found that adding certain metal elements to Ti(C,N)-$Mo_2C$-Ni can improve its fracture toughness. New cermets that we have developed exhibit increases in toughness of up to 40 % compared with these of conventional cermets with the same degree of hardness (Fig. 3, Patent Application No. 2011-254201). This improvement in cermet toughness, which had been a major issue, is expected to pave the way for the replacement of carbide dies with the cermets.

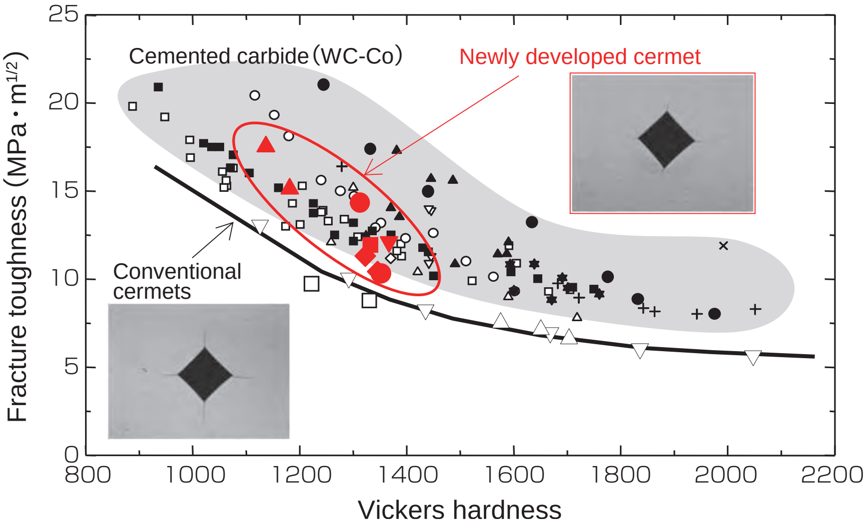



Fig. 3 Relationship between fracture toughness and Vickers hardness of newly developed and conventional cermets

Advanced Integrated Materials Research Group,
Materials Research Institute for Sustainable Development
**Akihiro MATSUMOTO**

# Rare Metals Opening the Way to the Future – Tungsten and platinum-group elements –

## Introduction

The rare-earth crisis, which has become a serious problem since 2009, reminded us of the constraints on stable supplies of unevenly distributed rare-metal resources. Tungsten and platinum-group elements are rare metals that are mined only in limited areas of the world, as is the case with rare-earth elements. Since these metals are now, and will continue to be, indispensable to industry (Table 1), it is necessary to secure stable and sufficient supplies of such resources to meet future demand.

## Tungsten

Tungsten is widely used in both commercial and military applications. Tungsten carbide, a typical cemented carbide, is the major secondary product, and is used in a wide range of fields including construction, metalwork, mining, and oilfield development (the gas and oil drilling industries). In addition, tungsten compounds are used as catalysts, and tungsten alloys and pure tungsten metal are used in electrodes, wires, and heating and lighting equipment. In terms of use by region, the production of tungsten carbide accounts for approximately 70 % of total tungsten consumption in Europe, steel and alloys account for about 30 % in China and Russia, grinding machines account for 41 % in the U.S., while in Japan, tungsten is mostly consumed for producing chemical compounds to be used as catalysts. As much as 81 % of the world's tungsten resources are produced in China (Fig. 1).[1]

On the other hand, the Chinese government has imposed restrictions


For inquiries about this article : Institute for Geo-Resources and Environment http://unit.aist.go.jp/georesenv/english/index.html

on exports of tungsten since 2005, on the grounds of depletion of high-grade mineral deposits in the country and the setting aside of a quota for domestic consumption. As a result, world tungsten production decreased in 2007. The consolidation of small- and medium-sized mines in China has given China Minmetals Corporation alone a share of approximately 50 % of the total production in China.

## Geological background: Exploration, mining, and mineral processing

Scheelite ($CaWO_4$) and wolframite ($(Fe,Mn)WO_4$) are important minerals that contain tungsten. Approximately 60 % of tungsten is mined as scheelite (about 30,000 tons per year). Past studies have indicated that tungsten deposits in the form of scheelite are usually found around ilmenite-type granite rocks (reduced magma due to crystallization differentiation).[2] In China, tungsten deposits are concentrated in the southern part of the country where ilmenite-type granite rocks are widely distributed (Fig. 2). Scheelite deposits are found in relatively deep places and many of them are skarn deposits, which are formed by hydrothermal alteration of limestone (Shizhuyuan in China, Nui Phao in Vietnam, Cantung in Canada, and King Island in Australia). These mineralogical characteristics are one of the important guiding factors in mineral deposit exploration.

For many tungsten deposits, underground mining is the principal mining method unless the ore bodies are distributed near the surface. As many ore bodies are deposited in the shape of lenses (skarn deposits) or veins (vein deposits), open-cut mining is too costly and not appropriate in such cases. As mentioned earlier, scheelite and wolframite are the two important minerals that contain tungsten. The former is refined by gravity concentration, and the latter by magnetic concentration since it is magnetic. Generally, interim products (mainly ammonium paratungstate, or APT) are produced from mineral concentrates by chemical treatment and exported.

| Product | Major producers (2009) | Applications |
|---|---|---|
| Tungsten | China (81 %) | Carbide tools, catalysts |
| Platinum | South Africa (56 %) | Catalysts, fuel cells, accessories |
| Palladium | Russia (26 %) | |

Table 1 Producers and applications

## Platinum-group elements

Among the platinum-group elements, platinum (Pt, atomic number: 78) and palladium (Pd, atomic number 46) are found only in small amounts in the Earth's crust, at 10 ppb and 1 ppb, respectively. Deposits of platinum-group resources in the world are estimated as follows from a geological standpoint: Bushveld (South Africa, 79.7 %), Great Dyke (Zimbabwe, 10.4 %), Norilsk (Russia, 8.2 %), Stillwater (U.S., 1.4 %), and Sudbury (Canada, 0.3 %) (Fig. 3).[3]

Total deposits of platinum-group elements are estimated to be about 76,000 tons, the equivalent of a 300-year supply based on annual global consumption (about 250 tons in 2010), out of which 90 % of the platinum is found in South Africa, 90 % of the palladium is located in Russia, and nearly 80 % of annual production is supplied from these two countries (Fig. 4).[4] In order to secure stable supplies of platinum resources in the future, Japan should intensify its alliance with South Africa and increase its presence in the field of resources through collaborations among industry, academia, and government.

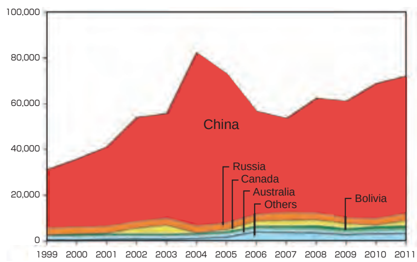



Fig. 1 China's tungsten production is overwhelmingly large.[1]


Mineral Resources Research Group,
Institute for Geo-Resources and Environment

**Hiroyasu MURAKAMI**
**Tetsuichi TAKAGI**

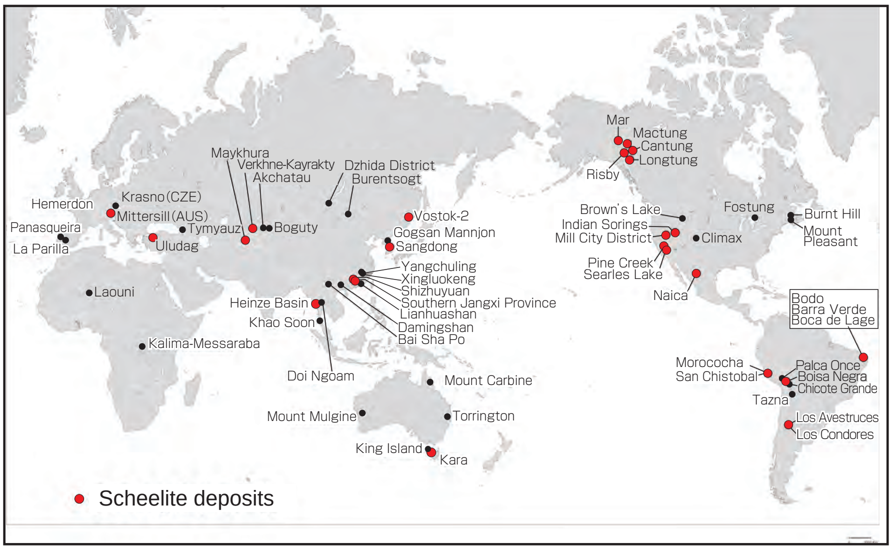



**Fig. 2 Global tungsten deposits are concentrated in southern China.**

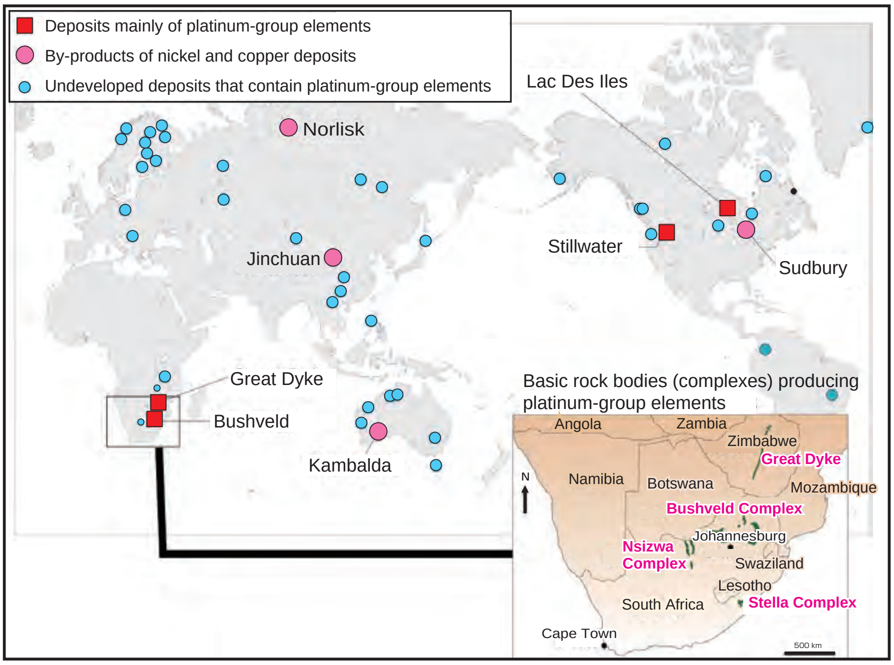



**Fig. 3 Deposits of platinum are widely distributed throughout the world but very few are economically feasible to mine.**

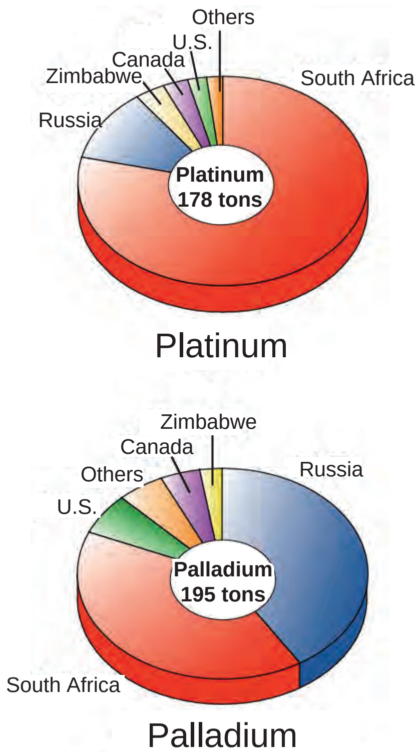



**Fig. 4 Most platinum and palladium is produced in South Africa and Russia, respectively (production in 2009[4]).**

## References


[1] U.S. Geological Survey: *Mineral commodity summaries 2011*, 176-177 (2011a).

[2] S. Ishihara: *J. Geol. Soc. Japan*, 77, 441-452 (1971).

[3] Y. Kita: *Metal Resource Report 2005*, 3, 27-41, JOGMEC (2005).

[4] U.S. Geological Survey: *Mineral commodity summaries 2011*, 120-121 (2011b).




For inquiries about this article : Institute for Geo-Resources and Environment http://unit.aist.go.jp/georesenv/english/index.html

# Research Hotline

# UPDATE FROM THE CUTTING EDGE

## Apr.-Jun. 2013

**The abstracts of the recent research information appearing in Vol.13 No.4-6 of "AIST TODAY" are introduced here, classified by research areas. For inquiry about the full article, please contact the author via e-mail.**

Environment and Energy

## Immobilizing Pt nanoparticles inside the pores of metal-organic framework

### Highly active catalysts for hydrogen generation from hydrides

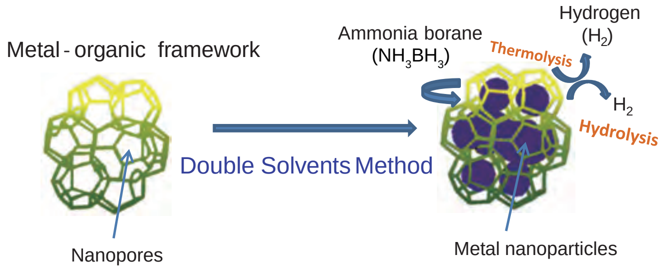
We have developed a double solvent method to immobilize ultrafine Pt nanoparticles inside the pores of a metal-organic framework (MOF), MIL-101, without aggregation of Pt nanoparticles on the external surface of the framework. TEM and electron tomographic measurements clearly demonstrated the uniform three-dimensional distribution of the ultrafine Pt nanoparticles throughout the interior cavities of MIL-101. The resulting Pt@MIL-101 composites exhibit excellent catalytic performance for both hydrolysis and thermolysis of ammonia borane to effectively generate hydrogen, which encourages the application of ammonia borane as a promising hydrogen storage material for mobile fuel cell systems. The present results also bring light to new opportunities in the development of high-performance heterogeneous catalysts by using functionalized cavities of MOFs as hosts for ultrafine metal nanoparticles.

**Qiang XU**

Research Institute for Ubiquitous Energy Devices

q.xu@aist.go.jp

AIST TODAY Vol.13 No.5 p.14 (2013)

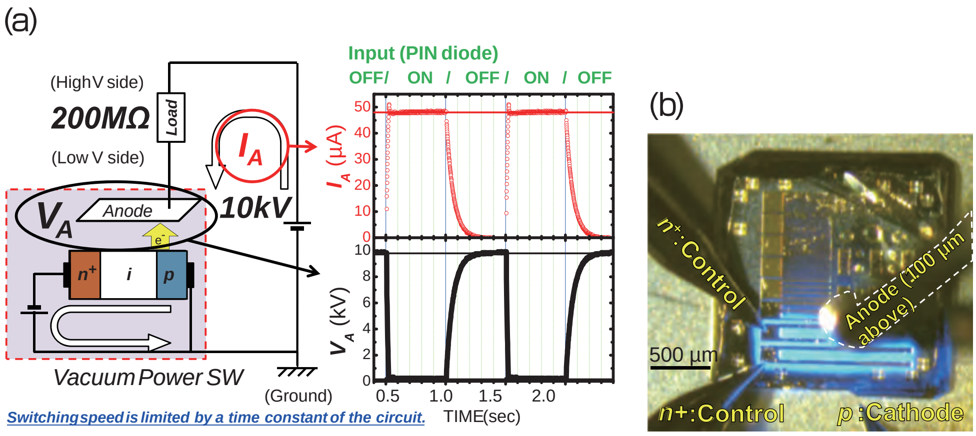



**Schematic representation of synthesis of Pt nanoparticle inside MOF nanopores using double solvents method**

# High-voltage vacuum power switch for smart power grids

## Innovation with unique properties of diamond semiconductors

We have developed vacuum power switches rated at 10kV, building electron emitters (essentially PIN diodes) from hydrogen-terminated diamond, whose negative-electron affinity enables it to supply a large amount of current across a high vacuum when it is turned on. We also calculated that the technology could be scaled up to 100kV or higher, making the approach a promising one to control smart grids.




10 kV switching result of a vacuum power switch (a), and a top view image (b)


**Daisuke TAKEUCHI**

Energy Technology Research Institute

d.takeuchi@aist.go.jp

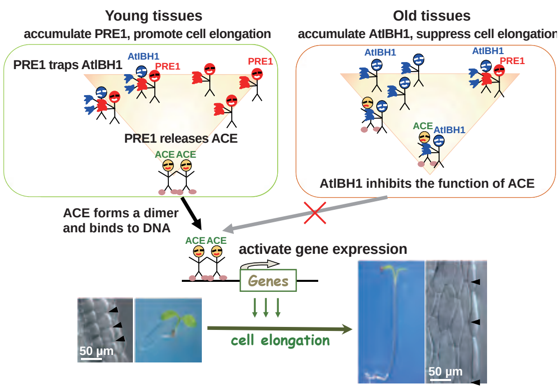

AIST TODAY Vol.13 No.5 p.15 (2013)



# A system that regulates height and size of leaves and seeds in plants

## Tri-antagonistic bHLH transcription factors regulate cell elongation in plants

In plants, basic helix-loop-helix (bHLH) transcription factors play important roles in the control of cell elongation. Two bHLH proteins, PACLOBTRAZOL RESISTANCE1 (PRE1) and *Arabidopsis* ILI1 binding bHLH1 (AtIBH1), antagonistically regulate cell elongation in response to brassinosteroid and gibberellin signaling, but the detailed molecular mechanisms by which these factors regulate cell elongation remain unclear. Here, we identify the bHLH transcriptional activators for cell elongation (ACEs) and demonstrate that PRE1, AtIBH1, and ACEs constitute a tri-antagonistic bHLH system that competitively regulates cell elongation. In this system, ACEs directly activate the expression of enzyme genes for cell elongation by interacting with their promoter regions. AtIBH1 negatively regulates cell elongation by interacting with the ACEs and thus interfering with their DNA binding. PRE1 interacts with AtIBH1 and counteracts the ability of AtIBH1 to affect ACEs. Therefore, PRE1 restores the transcriptional activity of ACEs, resulting in induction of cell elongation.




A tri-antagonistic basic helix-loop-helix system regulates cell elongation in plants.


**Miho IKEDA**
mi-ikeda@aist.go.jp

**Masaru TAKAGI**
m-takagi@aist.go.jp

Bioproduction Research Institute

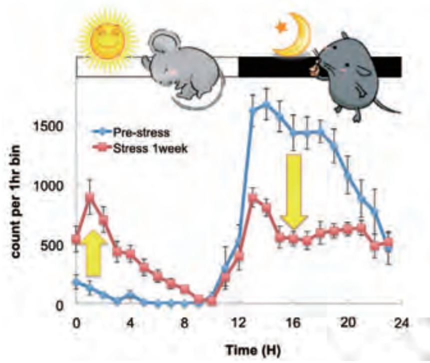

AIST TODAY Vol.13 No.5 p.16 (2013)

# How to develop an animal model of stress-induced sleep disorders

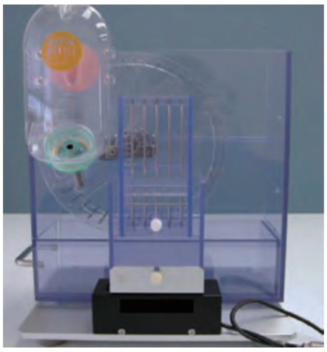
## Aiming at prediction of sleep disorders and elucidation of their mechanism

Chronic and social defeat types of stress induce depression and post-traumatic stress disorder (PTSD) with circadian rhythm abnormalities. So far, an appropriate experimental system that can assess circadian locomotor activity during continuous exposure to a stressor has not been established. We developed a novel system called PAWW (Perpetual Avoidance of Water on a Wheel) that expose mice to continuous stress. Continuous PAWW stress reduced the duration of daytime sleep of stressed mice, especially during the first half of the light period, and increased nighttime sleepiness. Continuous PAWW stress also simultaneously obscured sleep/wake and locomotor activity rhythms. These sleep architecture phenotypes under stress are similar to those of patients with insomnia. Circadian gene expression in the liver and muscle was unaltered, indicating that the peripheral clocks in these tissues remained intact.

Koyomi MIYAZAKI
Biomedical Research Institute
k-miyazaki@aist.go.jp
AIST TODAY Vol.13 No.6 p.20 (2013)




Diurnal rhythm of wheel running behavior of PAWW stressed mice



Cage for generating sleep disorder model mice (SW-15S-SD)



# Development of *Euglena*-based bioplastics

## Euglenoid constituent ratio of the plastics is ca. 70 %.

*Euglena*-based bioplastics were synthesized by introducing a long-chain fatty acid obtained from a lipid (wax ester) derived from *Euglena* to a polysaccharide (paramylon, β-1,3-glucan) produced by *Euglena*, a natural polymer consisting of glucose molecules. Euglenoid constituent ratio of the resulting bioplastics is approximately 70 %. Features of the bioplastics include high thermal plasticity and heat resistance. That is, their thermal plasticity is comparable to that of conventional bioplastics (polylactic acid and nylon 11), cellulose acetate with plasticizer, and petroleum-based ABS plastic. Heat resistance of the *Euglena*-based plastics is higher than that of these reference plastics.

Motonari SHIBAKAMI
Biomedical Research Institute
moto.shibakami@aist.go.jp
AIST TODAY Vol.13 No.6 p.21 (2013)

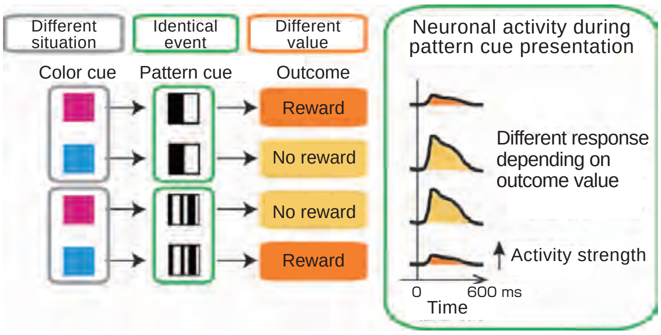

Production process of *Euglena*-based bioplastics

# Neuronal activity underlying flexible recognition of events

## For understanding the neural mechanisms of cognitive decline

The importance of the perirhinal cortex for reward-related information processing has been suggested. To examine whether or not neurons in this cortex represent reward information flexibly when a visual stimulus indicates either a rewarded or unrewarded outcome, we examined neuronal activity in the macaque perirhinal cortex using a conditional-association cued-reward task. The task design allowed us to study how the neuronal responses depended on the animal's prediction of whether it would or would not be rewarded. Two visual stimuli, a color stimulus followed by a pattern stimulus, were sequentially presented. Each pattern stimulus was associated with both rewarded and unrewarded outcomes depending on the preceding color stimulus. We found an activity depending upon the upcoming rewarded and unrewarded outcomes during the pattern stimulus presentation. The result indicates that the activity of perirhinal neurons flexibly signals the meaning of a stimulus independent of the identity of the stimulus.

Yasuko SUGASE
Human Technology Research Institute
y-sugase@aist.go.jp
AIST TODAY Vol.13 No.6 p.22 (2013)




A conditional association cued reward task to examine neuronal activity in the perirhinal cortex



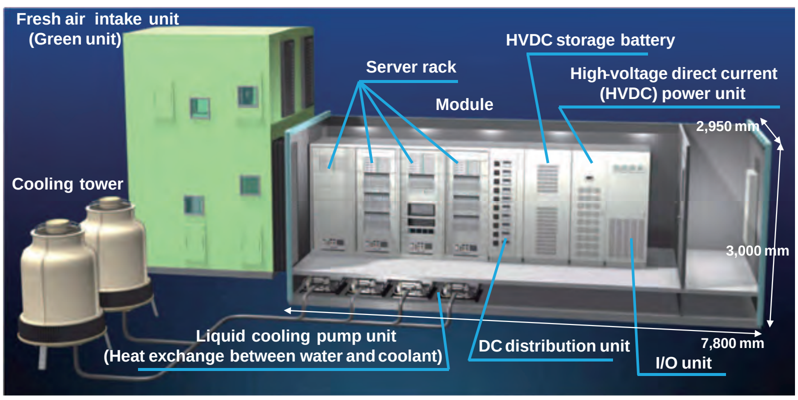
# Building a new modular green data center

## Realizing 30 % less power consumption than conventional modular data centers

We have developed a new modular data center by combining advanced energy-saving technologies to optimize the energy-use efficiency of the whole data center, realizing 30 % less power consumption than conventional modular data centers.

The four remarkable features of the modular data center are a liquid-cooled fan-less server, an air-conditioner-less data center with a fresh air intake unit, high-efficiency power supply, and energy-saving operation of the server. We also propose functional PUE (Power Usage Effectiveness) which is an evaluation metric of the power consumption efficiency by separating into power supply cooling, and information processing functions, instead of conventional PUE.

Satoshi ITOH
Information Technology Research Institute
satoshi.itoh@aist.go.jp
AIST TODAY Vol.13 No.4 p.8 (2013)




System configuration of modular green data center

# Applying semiconductor films on highly liquid-repellent surfaces

## High performance organic polymer transistor realized

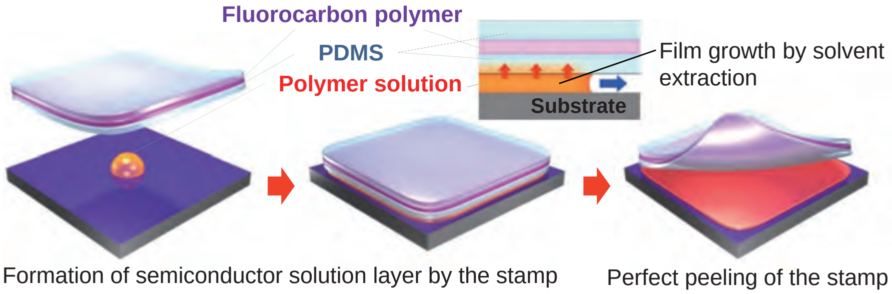
We have developed a manufacturing technology for highly uniform thin films of organic polymer semiconductors without material loss by applying the semiconductor solution on a highly hydrophobic surface that strongly repels the solution. It is known that conventional solution processes for thin-film production are not compatible with highly hydrophobic surfaces, although the employment of such surfaces as the gate dielectric surfaces is quite effective in improving the stability of thin-film transistor (TFT) characteristics. In this study, a new "push coating" technique was developed, where a solution of organic polymer semiconductor is compressed with an original silicone-rubber-based trilayer stamp to wet the hydrophobic surfaces uniformly by way of the capillarity. It causes almost no material loss, in striking contrast to any other solution processes. The technology allows remarkably simple production of high performance TFTs that are indispensable building blocks for information terminal devices such as electronic papers.

**Tatsuo HASEGAWA**
t-hasegawa@aist.go.jp

**Toshikazu YAMADA**
toshi.yamada@aist.go.jp

Flexible Electronics Research Center

AIST TODAY Vol.13 No.4 p.9 (2013)




Schematic of the film production process by the push coating technique



# Wide-area indoor camera tracking of a mobile device

## Toward realizing maintenance support systems using augmented reality

One of the essential requirements of augmented reality (AR) is the technique of localization. To achieve robust and global localization in wide areas, we have developed a method for estimating photo-shoot location and orientation based on virtualized reality environment models. Previously, model-based localization methods with photos were not efficient for covering wide areas in terms of modeling costs. Therefore, by applying our previous modeling method, we have developed a new efficient modeling method with involvement of data for image-based matching.

Figure 1 shows a framework for the localization. In the framework, at first, an image shot by a tablet is sent to the server. Next, in the server, the input image is compared to model data to estimate photo-shoot location and orientation. Finally, an estimated result is sent to the tablet. Figure 2 shows examples of experimental results. In the experiments, users can see labels on posters, and also can get additional contents by touching the labels.

**Koji MAKITA**

Center for Service Research

k.makita@aist.go.jp

AIST TODAY Vol.13 No.5 p.17 (2013)

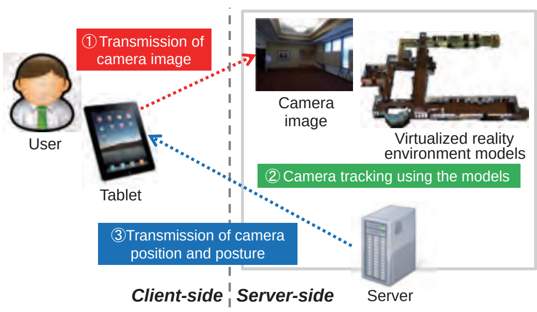



Fig.1 Outline of the camera tracking system

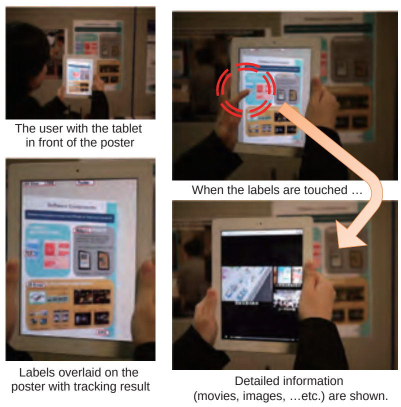



Fig.2 Experiments using a poster

# FinFET with the world's smallest characteristics variability

## Contributing to lower power consumption and higher performance of integrated circuits

We have developed a prototype of a 14 nm-generation 3D transistor (FinFET) with the world's smallest variability of characteristics (Fig. 1). A primary cause of the characteristics variability in a FinFET is the variability of physical properties of the metal gate electrode material. An amorphous metal material for the gate electrode that has a small variability of physical properties has been developed and the prototype FinFET was fabricated using the material. The variability of the electrical characteristics for the FinFET with the amorphous metal gate showed better variability than that of the FinFET with a conventional polycrystalline metal gate, and achieves the world's smallest characteristics variability. With integrated circuits beyond the 14-nm generation, including SRAMs, major issues have been the hindrance to performance improvement and the reduction in yields, both due to the characteristics variability of elements. The present results are expected to contribute to solving these issues.

**Takashi MATSUKAWA**
Nanoelectronics Research Institute
t-matsu@aist.go.jp
AIST TODAY Vol.13 No.6 p.23 (2013)

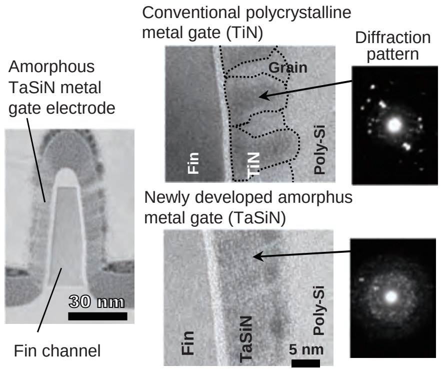



Fig.1 Comparison between the developed FinFET with the amorphous TaSiN metal gate and the conventional FinFET with the polycrystalline TiN metal gate

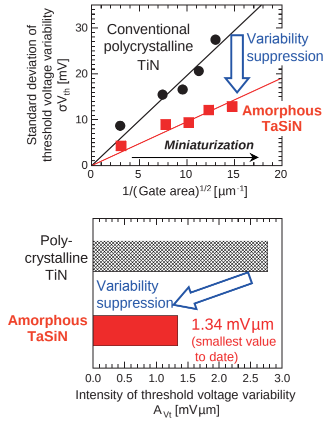



Fig.2 Pelgrom plots comparing the threshold voltage variability of the amorphous TaSiN metal gates and the conventional polycrystalline TiN metal gates

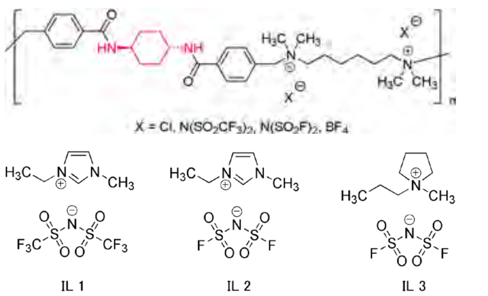


# Gel-forming materials for ionic liquids that work at very low concentrations

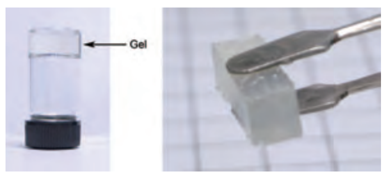
## Additives gelatinizing ionic liquids with excellent retention of their ionic conductivities

Ionic liquids (ILs) are attractive materials because of their unique properties such as low volatility, thermal stability, nonflammability, intrinsic ionic conductivity, and electrochemical stability. ILs are expected as electrolytes in electrochemical devices. The gelation procedure may become a convenient method to obtain conductive quasi-solid electrolytes, which are often preferred in practical applications to avoid electrolyte leakage. We have developed novel gel-forming materials derived from *trans*-cyclohexane-1,4-diamine for ionic liquids. They can gelatinize a variety of ionic liquids at very low concentrations (0.9-20 g/L). The gel-sol transition temperature are greater than 100 ℃ in the concentrations over 50 g/L for IL 2 and greater than 125 ℃ in the concentrations over 20 g/L for IL 3. The gelatinized ILs exhibit high mechanical strength along with the rapid recovery in rheology measurements. In addition, the ILs retain almost their ionic conductivities after gelation.

**Jun'ichi NAGASAWA**
Nanosystem Research Institute
j.nagasawa@aist.go.jp
AIST TODAY Vol.13 No.4 p.10 (2013)




Structure of the developed gelator (above), and the structure of ionic liquids for the examination (below)




The gel of ionic liquid 1 formed in a mold

# Development of high-definition infrared color night-vision imaging technology

## Clear high-definition color video recording of objects in darkness

We have developed 3CCD full high-definition (HD) infrared color night-vision imaging technology. Infrared night-vision cameras are used widely to record images in darkness, but for a long time only monochromatic images have been available. In fields where night-vision recording is indispensable (e.g. security, on-board vehicle cameras, and nocturnal wildlife observations), HD cameras are beginning to be used to collect detailed image information, but still as monochromatic images. If color images that are similar to those under visible light can be recorded by using only infrared light, then information that is qualitatively different from that currently available can be obtained and new developments in the above-mentioned and other fields can be expected.

**Yasushi NAGAMUNE**
nagamune.y@aist.go.jp
**Takashi TOKIZAKI**
t-tokizaki@aist.go.jp
**Toshitaka OTA**
ohta-t@aist.go.jp
Nanosystem Research Institute

AIST TODAY Vol.13 No.5 p.18 (2013)


**The newly developed HD infrared color night-vision camera**
The black rectangular block on top of the camera is the infrared projector.


**Example of images of objects taken in darkness by using the newly developed infrared color night-vision camera**



# Analysis of trace light elements with X-ray absorption fine structure

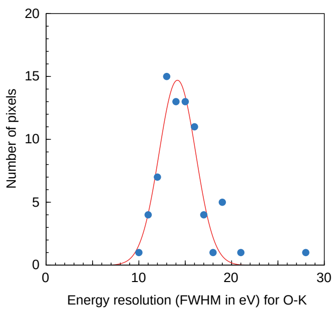
## Nano-structure of an SiC semiconductor revealed by a superconductor

A trace amount of light elements are indispensable for functional materials or devices. Nitrogen is a typical donor dopant for a compound semiconductor, SiC. However, doping into SiC is very difficult compared with monoatomic Si, and thus it is still one of the difficulties for realizing energy-saving semiconductor devices. A lack of nano-structure analyzing method for trace light elements was prohibiting green innovation. Here, we report a new instrument using superconductivity and successful lattice site determination of the nitrogen dopant implanted in SiC.

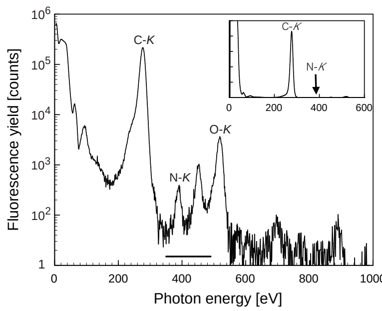
X-ray absorption fine structure spectra at the nitrogen K-edge were measured by using a superconducting X-ray detector that enables to separate the weak N-*K* line from the strong C-*K* line. Comparison between the experiment and first-principle calculation revealed that the nitrogen atoms occupy the C sites.

**Masataka OHKUBO**
Research Institute of Instrumentation Frontier
m.ohkubo@aist.go.jp

AIST TODAY Vol.13 No.4 p.11 (2013)




**Pixel number distribution of energy resolution for the oxygen *K* line (525 eV)**



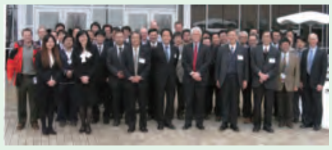

**An example of the detection of trace nitrogen dopant atoms in SiC**

## Workshop with the National Renewable Energy Laboratory of U.S.A.

A workshop with the National Renewable Energy Laboratory (NREL) was held on March 12, 2013, in Golden, Colorado, U.S.A. Vice-President Yabe and researchers from the environment and energy department as well as other fields of AIST attended the workshop, along with officials from METI led by Director Shinji Tokumasu of the International Affairs Office.

The workshop

A memorandum of understanding on comprehensive research cooperation was concluded in May, 2008, between AIST and NREL, and collaborative researches relating to solar power generation and biomass refinery have been conducted at both institutes. The main purpose of the workshop was to confirm the bilateral research results on a range of renewable energy technologies, and to provide opportunities for researchers to exchange technical and scientific ideas. About 50 researchers of both countries participated in this workshop.



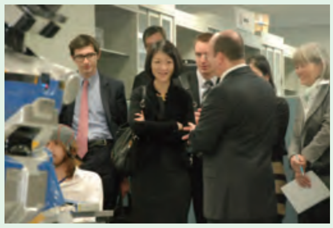
Participating members

Overviews of the two institutes were followed by several research presentations concerning photovoltaic technologies, biomass energy, hydrogen energy, and energy network technologies. In addition, the U.S. policies for supporting dissemination of technologies were explained.

After the workshop, the participants visited the Energy Systems Integration Facility (ESIF) which was newly constructed in the grounds of NREL. It is an experimental facility to build integrated systems which efficiently utilize various energy resources. The mission of ESIF is in common with that of Fukushima Renewable Energy Institute of AIST which is scheduled to be opened in April, 2014. Further collaborations between the two institutions are expected in the future.

## AIST Tsukuba Receives Visit from French Minister for Economic Regeneration, with Responsibilities for Small and Medium-sized Enterprises, Innovation and the Digital Economy

On March 29, 2013, Ms. Fleur Pellerin, French Minister for Economic Regeneration, with Responsibility for Small and Medium-sized Enterprises, Innovation and the Digital Economy, and Mr. Christian Masset, Ambassador of France to Japan, visited AIST Tsukuba to inspect CNRS-AIST JRL (Joint Robotics Laboratory), where collaborative research project has been conducted between AIST and the Centre National de la Recherche Scientifique (CNRS).



Minister Pellerin (center) receiving explanations

Senior Vice-President Ichimura gave a speech covering the overall relationships with France including the visit to AIST by Mr. Louis Schweitzer, Special Representative of French Minister of Foreign Affairs for French-Japanese Partnership, the attendance of AIST Advisory Board Meeting by Dr. Alain Fuchs, President of CNRS, and the participation in STS Forum by Ms. Genevieve Fioraso, French Minister of Higher Education and Research. Vice-President Kanayama introduced the active research collaboration that has been conducted in a complementary manner between the two institutions combining the superior hardware of AIST with the superior software of CNRS.

Visitors and JRL/AIST members

Updates on the project status, the staff members, and the research activities were reported by Japanese and French managers responsible for JRL. A demonstration of the humanoid robot undergoing development also took place.

Minister Pellerin showed strong interest in this project. Various views and ideas were enthusiastically exchanged on collaborative robot researches with other organizations, how to operate the robot, and the utilization of research results.

**Cover Photos**
Above: Iron balls attracted by heated Sm-Fe-N sintered magnets (p. 6)
Below: Experimentally fabricated diamond-coated WC-FeAl drill (p. 9)

AIST TODAY
International Edition
2013-3 No.49
Published in September, 2013

AIST NATIONAL INSTITUTE OF ADVANCED INDUSTRIAL SCIENCE AND TECHNOLOGY (AIST)

Website and Publication Office, Public Relations Department
National Institute of Advanced Industrial Science and Technology (AIST)

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-ml@aist.go.jp URL: http://www.aist.go.jp/index_en.html



AIST11-E00003-9