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Feature

Rare Metals

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In Brief



AIST Bare Metal Task Force

What is "rare metal" ?

For the past few years, we frequently hear of "the rare metal problem" or "the rare metal crisis". The terminology, "rare metal", is not an academically defined one, and there is no consensus on which element it pertains. In Japan, recently, the term is often used to refer to the 47 metal elements shown in Figure 1, according to the definition set by the Ministry of Economy, Trade and Industry. Sometimes, the 17 rare earth elements are counted as one kind, and the total is counted as 31. There are a total of 89 existing elements in the natural world, and therefore, it can be said that over half of the elements are rare metals.

Elements as titanium, manganese, chromium, which are found in abundance in the earth's crust, are also considered to be rare metals. This is because manganese and chromium have been essential elements for the industrial world since its early days, used as additives to enhance the properties of iron. Titanium is considered "rare" because it is a difficult metal to produce as high technology is required for refining the abundant ore in form of titanium oxide. On the other hand,

from historical circumstances, gold and silver, which have been in existence since ancient times, are not called rare metals.

From "vitamins of industry" to "lifeline of industry"

Rare metals have been called "vitamins of industry" and their importance in industry has been recognized for some time. However, recently, the industry has become highly dependant on products that can not be made without using rare metals so that they are becoming "the lifeline of industry".

Here are introduced some representative examples of products in which rare metals play important roles, or in which they will do so in the near future.

Figure 2 shows the relationship between rare metals and the automobiles of today as well as the energy-saving cars with low environmental load presently being developed. It is evident that no matter what type of automobile is developed, rare metals will become more and more important.

Another example is liquid crystal television. The production of liquid crystal television is rapidly growing and its screen

is getting larger and larger. An extremely rare metal, indium, is indispensable as an element of the transparent conductive film (ITO) of liquid crystal television panel.

Why are rare metals problematic?

Although the importance of rare metals is increasing, the supply is not necessarily stable and the prices are recently rising sharply which causes a big problem. The points that follow can be considered as its international background.

• With the rapid industrialization of highly-populated developing countries as China and India, the sudden increase of the demand for all resources (and its future demand estimation)

• The sudden demand increase of certain rare metals needed for new products as liquid crystal television and hybrid cars (including many energy-saving devices / devices with low environmental load)

• The large influx of speculative money into the comparatively small rare metal market

• The export restrictions / restraints by the countries with resource reserves as part of

	group 1	group2	group3	group4	group5	group6	group7	group8	group9	group10	group11	group12	group13	group14	group15	group16	group17	group18
period 1	1																	2
	Н				30 rare i	netal ele	ments		17 rare	earth eler	nents							He
period 2	3	4			excludin					t in rare m			5	6	7	8	9	10
	LI	Be			CACIDUIN	s rui c cui	1010						В	С	N	0	F	Ne
period 3	11	12											13	14	15	16	17	18
	Na	Mg											AI	Si	Р	S	CI	Ar
period 4	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36
	K	Са	Sc	Ti	V	Cr	Mn	Fe	Со	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr
period 5	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54
	Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Те		Xe
period 6	55	56	57~71	72	73	74	75	76	77	78	79	80	81	82	83	84	85	86
	Cs	Ba	lanthanoid	Hf	Ta	W	Re	Os	lr	Pt	Au	Hg	TI	Pb	Bi	Po	At	Rn
period 7	87	88	89~103															
	Fr	Ra	actinoid															
			lanthanoid L		58	59	60	61	62	63	64	65	66	67	68	69	70	71
					Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu
		actinoid		89	90	91	92											
				Ac	Th	Pa	U											

Figure 1: 47 rare metal elements





Figure 2: An example of rare metals used in automobiles with low environmental load

Figure 3: An image of the integrated research aimed for by the rare metal task force

their strategy to develop domestic industry.

Such phenomena as the globalization of the economy and the development of modularized products have made possible the explosive increase of the productivity of many products. This also heightens the danger of sudden demand-supply imbalance in rare metal markets.

Included in the rare metals whose unstable supply is apprehended is the following: ① elements whose reserves and quantity of production are extremely small, and whose ore-deposits are located in a handful of countries (platinum group elements, tungsten, heavy rare-earth elements), ② elements produced only in extremely small amounts as by-products when other resources are mined, and therefore, of which it is very difficult to increase production to meet the needs (such as indium which is mostly produced as by-product of zinc).^{[1][2]}

AIST rare metal task force and its goal

In order for Japan, a resources-poor country, to continue valuing "monozukuri" (manufacturing), create new industry and maintain its position as an advanced industrial country in the future, it needs to alleviate the supply instability of rare metals and to heighten its level of rare metal resource security.

Furthermore, problem solving by development of new technology is believed to lead to big chances for creation for our industry.

The measures adoptable by public research institutions are the diversification of resource supply options through search for new ore-deposits, the development of resource-saving technologies and alternative material technologies and the buildup of recycling technology. Moreover, to suppress speculation, it is important to strive for dispatch of effectual information.

AIST is the only institution in Japan that

is organizationally carrying out the abovementioned research and development measures. Moreover, although the research done may not be intentionally on rare metals, a large number of researchers are engaged in technological development using the special characteristics of rare metals. However, previously, as many research units had been engaged in individual problems according to their own specialties, coordination and dispatch of information necessary for tackling the rare metal problem were not enough.

The rare metal task force was organized in 2006 to actively tackle the rare metal problem by promoting field-integrating research topics which straddle several research units in our institute. Figure 3 shows an image of an integrated research that the rare metal task force is aiming for.

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Rare Earth Magnet without Use of Heavy Rare

High-performance magnet used in many high-tech devices

Over the years, Japan has continuously led the world in the development of highperformance magnet. In particular, the performance of Nd-Fe-B magnet has far exceeded all other magnets. It is continuously being improved and its performance is being upgraded.

Nd-Fe-B magnet is used in high-tech devices and machines as hard disk voice coil magnets (VCM), mobile-phone speakers / vibration motors, DVD optical pickups, industrial robots and magnetic resonance imaging devices (MRI) ,etc.^[1]. In recent years, magnet-using motors for hybrid and electric vehicles are drawing attention. There is, however, a problem as car motors are said to operate in high temperatures of about 200 °C. Since it is difficult to install a cooling system for car motors, heat resistant property is required.

The importance and scarcity of dysprosium (Dy)

Indicators of the characteristics of permanent magnets are coercive force, remanent flux density, and maximum energy product. A magnet with high maximum energy product is considered to be a highperformance magnet. Nd-Fe-B magnet which is a high-performance magnet has a drawback of dramatically decreasing coercive force with the rise of temperature. In order to improve this aspect, it has become apparent that a few % addition of dysprosium (Dy) is effective. Presently, Dy has become an essential element for Nd-Fe-B high-performance magnet as it helps to maintain coercive force even at high temperatures. Automobile magnets which are used in harsh environments need to maintain their performance at high temperatures, and in order to do so, it is said that 3 times the amount of Dy needed at normal temperature has to be added. However, the addition of Dy decreases the remanent flux density. As the maximum energy product is set by the relation between the coercive force and the remanent flux density, excessive addition of Dy will decrease the maximum energy product of the magnet.

The rare earth element resources in the earth crust are eccentrically-located and its majority is produced in China. There is a need to reduce its usage as, especially compared to light rare earth elements as neodymium (Nd) and samarium (Sm), heavy rare earth elements including Dy are unevenly distributed to a dramatic degree, their output low, and their prices are about 3 times that of Nd^[2]. In FY2007, the Ministry of Economy, Trade and Industry has started a project to develop a magnet with reduced usage of Dy. With the recent increase in demand for magnets, especially with the production increase of environment-friendly hybrid cars, it is thought that the usage of Nd-Fe-B magnets will increase all the more hereafter. As the present important position of Nd-Fe-B magnet is thought to be set for the time being, this project is very important.

The possibility of rare earth magnet that does not use heavy rare earth elements

While reducing the usage of Dy, research for high-performance magnet that does not

use Dy needs to be promoted. At present, Sm-Fe-N magnet which uses a light rare earth element, Sm, is promising as a magnet equivalent to that of Nd-Fe-B^[3]. This magnet, said to be of the same performance as Nd-Fe-B, is thought to be operative in relatively high temperatures as its Curie temperature is about 150 °C higher than that of Nd-Fe-B. Moreover, it is corrosion resistant and, therefore, its surface does not need to be coated with other metals as Nd-Fe-B magnet, which is also advantageous. There is, however, one drawback that it breaks down into samarium nitride and iron in high temperatures of about 600 °C . This causes great problems when manufacturing magnets.

Magnet is made by consolidating alloyed magnetic powder, and there are two basic types of sintered magnet and bonded magnet.



Figure 1: The relationship of sintering temperature with remanent flux density (Br), coercive force (Hcj) and sintering density of Sm-Fe-N powder Sm-Fe-N breakdown is happening around the dotted line temperature.

Sintered magnet is made by sintering magnetic powder in high temperatures, and this method produces a dense magnet. The bonded magnet, on the other hand, is made by bonding the magnet powder by resin. This means that the magnetic material is left in powder form and resin fills in the voids. Because of this difference, even though the same amount of magnetic powder is used, sintered magnets elicit higher performance than bonded magnets. However, bonded magnets with resin can be molded into various shapes, and thus, each magnet is used for different purposes.

When developing a high-performance magnet, sintering method is advantageous, but it is extremely difficult to sinter magnetic powder as Sm-Fe-N breaks down in high temperatures. For this reason, all Sm-Fe-N magnets on market are of the bonded type.

Changing the sintering process

Our research group is trying to make a Sm-Fe-N sintered magnet. With the conventional sintering method, the pre-pressed solidified magnetic powder is sintered in a furnace. With this method, Sm-Fe-N is exposed to high temperature for a relatively long time that it is more likely to breakdown. We have attempted to make a dense Sm-Fe-N sintered magnet while suppressing possible breakdown by using a new sintering method, the pulsed current sintering process ^[4] which sinters in a short time while applying pressure.

To make dense sintered components, important factors are the sintering temperature and the applied pressure. Generally speaking, by raising the degree of factors, the denser the sintered component becomes. Figure 1 shows the relationship of sintering temperature with remanent flux density (Br), coercive



Figure 2: The relation between sintering density and maximum energy product of Sm-Fe-N Marks of A and A indicate when sintering density is changed depending on applied pressure and sintering temperature, respectively.

force (Hcj), and sintered density when the sintering temperature of Sm-Fe-N powder is changed. Its density increases as the sintering temperature rises. On the other hand, its magnetic property remains stable up to 500°C , but when the temperature exceeds this, it declines and its coercive force especially becomes almost nil. This is because the breakdown of Sm-Fe-N has occurred. In this manner, the magnetic property of Sm-Fe-N does not gradually decline according to temperature rise, but characteristically remains the same up to a certain temperature level and declines in one stroke as the temperature rises beyond. To overcome this characteristic, it is necessary to sinter at a temperature which does not trigger breakdown while applying high pressure. Figure 2 shows the relationship between the density and the maximum energy

product $((BH)_{max})$ taken from experimental results. As by increasing pressure and sintered density, maximum energy product can be increased, low-temperature highpressure sintering can achieve good results. However, although it is possible to make highperformance magnet with this method, only simple-shape sintered components can be made and it is not suited for mass-production. The production cost is also estimated to be extremely high. Presently we are doing research and development for fabrication and sintering of optimal powder, with production cost reduction and process development adapted to mass-production in mind.

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Palladium Dependency Reduction / Alternative Hydrogen Separation Membrane

Problems with platinum group metals

The platinum group metals, as platinum and palladium, are metals that are vital for areas concerning the environment and energy. Almost half of the world production volume is used as exhaust purification catalyst. Recently, hydrogen is regarded promising as a clean energy medium, and platinum group metals are also crucial for the production and utilization of hydrogen. Presently, most of the hydrogen is made from resources as natural gases by chemical reaction, and platinum group metals are used as catalysts. Fuel cell that efficiently generates electricity from hydrogen and oxygen uses platinum as catalyst. In this manner, in order to realize a hydrogen powered society, it is thought that a large amount of platinum group metals is needed.

On the other hand, the quality of the mineral vein of platinum metal is low, and 1 ton of rock is needed to attain 1 gram of pure metal. Therefore, the natural environment is destroyed in mining as well as in disposal of most of the mined rocks. Moreover,

enormous amount of energy is consumed in treating the mined rocks. It is ironic that the production of platinum group metals necessary for dealing with environmental / energy problems is adversely causing an environmental / energy problem.

Making high-purity hydrogen with palladium

Metal membrane made of palladium can be used to sieve hydrogen from mixed gas. Its separation mechanism is shown in Figure 1. A hydrogen molecule is dissociated into two atoms on the membrane surface, dissolved within the membrane, diffused by weaving through metal atoms, recombining on the other side of the membrane and thus permeating through it. Molecules other than hydrogen cannot permeate the membrane as it is difficult for them to dissociate, dissolve and diffuse. In manufacturing semiconductor and LED, super high-purity hydrogen is needed, and palladium membrane is used to produce 99.9999999 % pure hydrogen.

Unlike distillation which involves gas-

liquid transformation, this separation process has good energy efficiency, and is thought promising for hydrogen production for fuel cells. By supplying high-purity hydrogen obtained through metal membrane, the use of platinum as fuel cell catalyst can be reduced. A fraction of the platinum group metal catalyst will also become unnecessary for the production of hydrogen.

However, palladium is also part of the platinum group. AIST is doing various research concerning palladium dependency reduction / alternatives. Part of the research is introduced here.

Palladium dependency reduction / alternative technologies

① Reduction of palladium usage by thinning the membrane

The basic method for reducing palladium usage is reducing the thickness of the membrane.

When the membrane thickness becomes 5 μ m, one tenth of those on market, the hydrogen permeation rate per unit area



Figure 1: Principle of hydrogen separation through metal membrane



Figure 2: Palladium thin membrane of 5 μm thickness formed over stress relief interstice of porous support

will become ten times larger. Therefore, the required membrane area for the same quantity of permeated hydrogen will be one tenth; and consequently, the required palladium quantity can be reduced drastically to one hundredth of the present level.

To strengthen the thin membrane, a porous support is needed. However, it is extremely difficult to make a defect-free thin palladium membrane on top of a porous support because of the irregularity of its surface. At AIST, we have succeeded in making a defect-free palladium thin membrane by first forming a thin polymer layer on a porous support, plating the palladium on the layer and then removing the polymer layer (Figure 2). It has become clear that, as the interstice is left where the polymer layer existed, the palladium is not strongly restrained, and thus long-term stability is enhanced. Efforts are presently being made to evenly reduce membrane thickness (1~5 µm), to shift to palladium (Pd) alloy represented by Pd₆₀Cu₄₀, and furthermore, towards practical use as assembling into a module.

(2) Thinning of membrane made from vanadium, an alternative of palladium

Vanadium is a metal known to have high hydrogen permeability and is more abundant than palladium. Up to now, research had been done on vanadium membrane made by metal rolling; however, many processes as alloying, heat treatment, surface polishing were needed. At AIST, we have succeeded in forming a thin vanadium membrane on a porous metal support by a simple process of using vapor deposition (Figure 3). Using chlorine-free raw material, the effect to the metal support is little, and it is an environmentally-friendly process.







Figure 4: Principle of single-roller liquid quenching method, and amorphous alloy made by this method (Photo provided by Mitsubishi Materials Corporation)

By covering the support thinly with vanadium, its hydrogen permeability can be demonstrated. Further efforts will be made to reduce membrane thickness, to improve performance by alloying, with improvement of production in mind.

③ Membrane of non-palladium amorphous alloy

As shown in Figure 1, hydrogen dissolves as it permeates the metal membrane, but it is generally known that metal becomes fragile because of hydrogen (hydrogen embrittlement). Consequently, the biggest problem in the development of nonpalladium membrane material was to overcome the possibility of destruction and separation ability loss. With this in mind, we focused on amorphous alloy, known for its toughness, as possible membrane material. As a result, we found that amorphous alloy made of zirconium and nickel can be used as hydrogen separation membrane. In addition, although normally membrane made of nonpalladium metal needs palladium covering, we found that this membrane allows hydrogen to permeate through without it. Therefore, it is a promising complete alternative to palladium. Furthermore, with amorphous alloy, it is possible to produce a large surface membrane by using the singleroller liquid quenching method (Figure 4). Through collaborative research with a corporations, we are working toward improvement of performance, enlargement, and durability enhancement.

In view of contributing to simultaneously solving problems of resource / environment / energy, AIST will continue to do research development of hydrogen separation membrane.

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Recycling of Rare Earth Elements

Recycling of rare earth magnet

Rare earth magnet, especially of neodymium, iron, and boron system, has excellent magnetic property. It is used in hard disc drive (HDD), hybrid car motor, magnetic resonance imaging system (MRI), and acoustic equipment, and will continue to be important material for our daily life.

This magnet uses rare-earth elements such as neodymium and dysprosium. Japan depends entirely on foreign countries for rare-earth supply and imports almost all from China. In order to secure steady supply of rare earth elements, it is important to do resource exploration / development in the countries other than China, as well as to develop rare earth element recycling technology for domestic use.

In the manufacturing process of rare earth magnet, $20 \sim 30$ % is scrapped from cutting and breakage, and this is already

being recycled. However, for the magnets used in the products on market, there is little recycling done. With recovery of open market scrap in mind, at the Research Institute for Environmental Management Technology, we are studying the separation technology of rare earth elements by primary concentration using particle separation followed by chemical (hydrometallurgical) method.

Recovery of rare earth magnet powder from HDD

It is important to concentrate magnet in a solid state in order to recycle rare earth elements efficiently, because the weight percentage of magnet in a whole HDD is only 2~3 %. For the recycling of HDD, we are thinking of a two-step selective comminution where the package is initially crushed, and then only the rare earth magnet powder is selectively ground



and recovered from the magnet containing fraction, which was not crushed in the first step (Figure). This process can concentrate and recover rare earth magnet powder efficiently within the general recycling process for small domestic appliances, and can promote the economical efficiency of the following metal recovery process by dissolution and extraction.

Metal separation by hydrometallurgical process

Raw material recovered from the primary concentration process is dissolved in mineral acid such as sulfuric acid and hydrochloric acid; however, as magnet contains over 70 % iron, huge quantity of acid is necessary to dissolve the iron. We are searching for a method by which only rare earth elements can be efficiently dissolved, while suppressing iron dissolution as much as possible. For the metal separation and purification from the dissolved solution, the application of solvent extraction is being considered. By using the solvent extraction, dysprosium, a heavy rare earth element, and neodymium, a light rare earth element can be separated relatively easily, and nickel plated on the surface of the magnet and boron are left in the solution. We hope to recycle the extracted rare earth elements, by recovering them as chemical compounds and reusing them as raw materials for the magnet.

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Fluorescent material as light source of home information appliances / illumination

Today instruments which change electricity to light as display / lighting devices are essential to our lives. With these devices, the fluorescent material plays important role of converting invisible ultraviolet rays / electron beams generated by electricity into visible light. With high-intensity fluorescent lamp and backlight of liquid crystal display, white light emission is achieved by mixing blue, green, and red high-intensity fluorescent materials, and europium and terbium are used in large quantities. Recently with rare earth, however, the prices of fluorescent materials are rising, and it cannot be completely denied that there is a risk that the supply may be cut short. Therefore, the recycling of fluorescent material has become an important issue.

Recycling of rare earths of fluorescent material

In recent years, the quantity of recycled fluorescent material is increasing due to the improvement of recovery rate of fluorescent lamps from businesses. However, as part of it is deteriorated, and the mixture rate of the three colors varies according to product, little recovered fluorescent material is used in new products that emphasizes performance. Therefore, in order to improve recycling rate, there is a need for technology reseparation into the original colors, and to sort out the deteriorated fluorescent material. For this purpose, we are developing a technology that physically separates these materials by using the different physical property of each



fluorescent material.

With the deteriorated parts of fluorescent material that are difficult to recycle, they can be reused as elements by dissolving in solution and then by extracting europium / terbium. However, with fluorescent material for lamps and plasma display, there are many oxide substances that cannot be extracted by acid, and there is a need to develop a preprocessing method to make their extraction possible. As one of such methods, we are considering vitrification by partly adding oxide composition. Another consideration is the above-mentioned technology by which rare earth is efficiently extracted at low cost from acid extracted solution

The future of technology for fluorescentmaterial containing rare earth

For measures concerning rare metal of fluorescent material, in short terms, the

development of recycling technology is effective. However, for middle to long terms, technology concerning reduction of quantity of rare earth being used in fluorescent materials, and rare earth element alternatives are important issues. For this purpose, we are in the process of developing highly-efficient fluorescent material with little rare earth by using porous silica glass, and of developing highly-efficient fluorescent substance which uses metal other than rare earth.

With the technological development for recycling / reduction of rare earth usage / alternatives, a framework by which fluorescent substances can be steadily obtained at a low price will be established, and "light from electricity" will continue to be steadily supplied in our lives.

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Tomoko Akai

Rare-Earth: Resource Exploration and Development

Introduction

The demand for rare metals, along with other metals, is increasing more and more every year. It has been often said that petroleum will only last for another 30 years, but its life-span, despite the demand increase, has not been shortened and has been maintained. This is due to the efforts made to discover, develop new petroleum deposits and to make them productive. The same can be said of rare metals, and continuous efforts are made to extend its life-span. However the quantity of consumed resources of today is totally different from that of 30 years ago. Therefore, it is extremely difficult to secure resources of the same number of years, and a breakthrough and innovation is vital in exploration / development of resources.

Take rare-earth as an example, the total quantity consumed in the entire world from 1900 to 2006 was 2.2 million tons, with the consumption increasing yearly, and it is estimated that the demand in the next 30 years will be 8 million \sim 10 million tons^[1] (Graph). Therefore, in order to maintain rare-earth life-span of 30 years, it is necessary to

secure reserve quantity 4~5 times the amount of consumed resources in the last 100 years.

Because, with rare-earth, the consumer and production countries are limited to specific areas, it has not drawn much attention and resource quantity survey and mineral exploration / development have not been actively pursued worldwide. Japan, however, is the second largest rare-earth consuming country following China, and we, Mineral Resource Research Group, have been doing resource quantity survey since 2005 of rareearth which is essential for Japanese hightech industry.

Exploration of rare-earth resources

Rare-earth is a general term for 17 elements consisting of 15 elements of lanthanide series with scandium and yttrium added. The disclosed amount of rare-earth reserve thus far is a collective sum of all elements. However, the proportion of each element in the deposits varies, and there is, for example, no database on where and how much neodymium and dysprosium used in rare-earth magnet exist in reserves.



Quantity of production and future demand of rare-earth elements—Quantity of production is based on the data of the US Geological Survey (http://minerals.usgs.gov/minerals/pubs/commodity/rare_earths/stat/). The red line shows the production growth if it increases at the same rate as it has so far. The blue line shows the demand growth over the next 30 years if it increases at the rate of the last 10 years (1.7 times).

We started by making this database. As many of the rare-earth mines do not disclose detailed data on each element (or they do not possess any data), it is not possible to construct an accurate database on literature documentation. We made the database by exploring deposits for ore material sampling in countries around the world as China, the world's largest rare-earth producing country (Photo 1), and others from Africa to South America, and by making our original chemical evaluation. As a result, it has become clear that; (1) there is at least about 9 million tons of rare-earth reserves in the world (of which approximately half is in China), (2) reserves for heavy rare-earth elements as dysprosium and terbium are extremely limited. The clarified quantity of reserves corresponds to the estimated quantity of world demand over the next 30 years.

Search for new supply source of rare-earth

Of the rare-earth elements, development of new supply sources of heavy rare-earth elements (as dysprosium and terbium) is an important issue as the prices are continuously increasing due to urgent demands. Presently, heavy rare-earth elements are supplied from weathered granite deposits in the southern part of China. This type of deposit has only been found in China. We did geoscientific research of this deposit type and found that three conditions are necessary; (1) presence of reduced granite rich in heavy rareearth elements. (2) tropical to semitropical temperature and high humidity which causes the formation of thick weathering crust, (3)environment in which minerals containing heavy rare-earth elements disintegrate.

Having received Grant-in-Aid for Scientific Research from Japan Society for the Promotion of Science, we, along with the University of Tokyo and Kyushu University, are exploring for granite weathering crust in areas that fulfill these conditions, and have found promising areas in Southeast Asia^[2].

Generally speaking, a long period as 10 years and large development funds are needed for resource exploration and development^[3]. Development of new mines also goes hand in hand with burdening the environment. In order to rapidly and economically supply heavy rare-earth elements, and to minimize the environmental load, we are considering the possibility of recovering rare-earth elements as by-products of base metals and non-metallic minerals from deposits already under development.

One candidate is apatite which is the raw material for agricultural fertilizers. Collecting ores from the major apatite deposits in the world, and measuring the quantity of rareearth contained within, it became clear that approximately 170,000 tons of rare-earth elements (oxide conversion) was contained in apatite produced annually in the world. This amount exceeds the world annual rareearth production output (=the amount of consumption) of 2006 which was 130,000 tons. If rare-earths are recovered during the process of producing phosphoric acid from apatite, it is possible to dramatically increase the rare-earths supply.

In Japan, it has been known that there exists some ferromanganese ores rich in heavy rare-earths found in stratified manganese deposits^[4]. Of the total rareearths found in ferromanganese ores (1800 ppm average), heavy rare-earth elements consists of 20 %, and this richness in



Photo 1 Bayan Obo, China: world's largest rare-earth mine. Mineral ore reserves of around 57 million tons, rare-earth oxide grade 6%.

heavy rare-earths is its distinction amongst rare-earth reserves. Unfortunately, the manganese deposits in Japan are all very small and have already stopped mining, and therefore, it is not possible to recover any rare-earth elements. An issue today is to find a large-scale stratified manganese deposit rich in rare-earths. AIST, along with Council for Geoscience, Republic of South Africa, and Japan Oil, Gas and Metals National Corporation (JOGMEC), signed an agreement to do collaborative research of rare metal resources in the Republic of South Africa. As a first step, we are exploring for potential rare-earths in the stratified manganese reserve in the Karahari area, which is one of the largest deposits in the world (Photo 2).

Towards resource development of rare-earth elements

In order to develop rare-earth resources, it is necessary to not only do basic exploration and research of resources but also to do beneficiation tests and economical survey for development / production. In 2008, the



Photo 2 Mr. Ramontja, CEO of South Africa Council for Geoscience (left), Dr. Foya, Director of Mineral Resource Development (right) and writer (center).

Ministry of Economy, Trade and Industry (METI) started "Basis Project for Rare Metal Resource Development Promotion" with a 1.24 billion yen budget. With this project, Japan intends to strengthen relations with countries with resources with hopes on securing its rights for resources by using the latest results of geological science. It hopes to do so by strategically targeting countries of Africa, Central Asia, the Pacific Rim region which, although expected to have potential for mineral resource endowment, have not had sufficient exploration activities done. Within this project, exploration of new ore refining technology / recovery technology for uncollected rare metals in residue and for new types of rare metal deposits will be considered.

Collaborating with JOGMEC which is responsible for carrying out this project, we would like to propose new areas and subjects of exploration.

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Research Hotline

UPDATE FROM THE CUTTING EDGE Apr.-Jun. 2008

The abstracts of the recent research information appearing in Vol.8 No.4-6 of "AIST TODAY" are introduced here, classified by research area. For inquiry about the full article, please contact the author via e-mail.

Life Science & Technology

Non-invasive evaluation of arterial stiffness using conventional oscillometric blood pressure measurement A simple method for evaluating arterial stiffness

We have developed a simple and non-invasive method for evaluating arterial stiffness using oscillometric blood pressure measurement. The arterial pulse waves of the brachial artery were derived from a cuff wrapped around the upper arm during inflation and deflation of the cuff. The peak-to-peak amplitudes of the individual pulse waves were plotted. The pattern of pulse amplitude changes was different between compliant and stiff arteries: a compliant artery showed a clear peak whereas a stiff artery showed an unclear or no peak. Based on these findings, we developed indices and algorithms to distinguish the patterns of pulse amplitude changes and then applied these novel indices to arterial stiffness measurement. Using these indices, arterial stiffness can be evaluated through conventional oscillometric blood pressure measurement.

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Arterial pulse wave derived from a cuff wrapped around the upper arm.

Top panel: a pressure inside the cuff during inflation and deflation of the cuff.

Middle panel: Arterial pulse wave obtained by filtering the pressure inside the cuff.

Bottom panel: Peak-to-peak amplitude of the arterial pulse wave.

A melt-treatment of asbestos by heating with infrared light Possible application to an easy and safe treatment for asbestos on site

We have developed a new on-site method to melt noxious industrial wastes including asbestos easily using infrared radiation from halogen lamps. The harmful property of asbestos originates from its long and thin fiber shape. So far, it has been very difficult to make the waste innocuous since the melting temperatures of asbestos are very high (~1500 °C). Estimated stock of various kinds of waste containing asbestos in Japan is about 108 ton, and its treatment may take a long period of about 100 years using present facilities. If our technique is employed, all relevant cost can be fairly reduced.



(left) Asbestos (chrysotile) before melt and (right) asbestos after melt.

Nanotechnology, Materials & Manufacturing

A new inorganic binder

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Nanoelectronics Research Institute

AIST TODAY Vol.8, No.4 p.28 (2008)

Aiming to develop an environment-friendly ceramic process with the binder

We have developed a new inorganic binder with both rigidity and flowability for extrusion technique. It is based on the hydration of ρ -Al₂O₃ (Al₂O₃ • nH₂O, n \doteq 0.5), one of the alumina hydrates. Under optimum processing conditions for the amount of this binder and hydration condition, alumina rods and tubes were successfully prepared by the extrusion technique. Conventional extrusion technique requires the use of an organic binder to impart flowability and rigidity to the starting materials. However, the organic binder must be completely burnt out, which results in emission of CO₂. The inorganic binder is a promising material for conversion into ceramics during heating. Furthermore, during this process, CO₂ is not emitted from the binder. Thus, inorganic binders are expected to contribute to the environment-friendly ceramic manufacturing process.



Alumina rods and tubes prepared by the new environment-friendly ceramic manufacturing process with the new inorganic binder.

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AIST TODAY Vol.8, No.4 p.29 (2008)

High yield separation of metallic and semiconducting carbon nanotubes

Quite simple, quick and scalable method

We have developed a novel separation method of metallic and semiconducting single wall carbon nanotubes (SWCNTs) by agarose gel electrophoresis. We used SWCNT-dispersed gel as a sample of gel electrophoresis and found a drastic improvement in the separation (Figure). In this method, most of metallic SWCNTs moved out from the starting gel and formed the frac. 2, while semiconducting SWCNTs remained in the starting gel (frac. 1). As a result, almost all SWCNTs applied to gel electrophoresis were separated into respective electronic types. Since the new method is quite simple, quick and scalable, it could be suited for industrial production.



Nanotechnology, Materials & Manufacturing

Novel manufacturing technology for magnesium alloy sheets Full-dry hot pressing technology using a diamond-coated die

We have developed a full-dry pressing technology without lubricant for hot pressing of magnesium alloy plates using a diamond-coated die prepared using CVD. Magnesium alloy sheet can be pressed only at elevated temperatures. Therefore, special lubricant that withstands such high temperature is indispensable. Along with raising lubricant costs, oil mist generated from lubricants at high temperature degrades the work environment. Serious problems related to environmental concerns also emerge, such as the necessity of product cleaning after processing and disposal of wash water. Moreover, burned lubricant requires frequent maintenance of dies, which pushes up costs.

Dry pressing technology with a diamond-coated die needs no lubricants using the characteristic of ground diamond surfaces with a very small friction coefficient. Moreover, excellent heat resistance of diamond demonstrates satisfactory lubrication performance and coating durability up to a high temperature of 320 °C, which is a pressing temperature of magnesium alloys of poor workability. Dry manufacturing without lubricant realizes a long-cherished ambition in this field.

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AZ31, thickness 0.8 mm

Non-combustible alloy AMX602, thickness 0.8 mm

Drawn cups after dry hot drawing using a diamond coated die and a blank holder. (left:AZ31, thickness 0.8 mm, right:non-combustible alloy AMX602, thickness 0.8 mm)

Production of thick-film thermoelectric devices using centrifugal force

One step forward to realization of high-efficiency thermoelectric devices

We have developed a new process, "centrifugally pressurized solidification", utilizing uniform pressure by centrifugal force, and produced thermoelectric thick films. This process enables the production of thick films with microstructure close to single crystal, leading to thermoelectric performance of a practical level. A thermoelectric generation device comprising thick films is characterized by its ability to function as a cooling fin and keep a sufficient temperature difference for thermoelectric generation even by natural cooling, and the ability to be applied to curved structure such as exhaust pipes. The newly developed centrifugally pressurized solidification not only produces thermoelectric thick films close to a single crystal but also simplifies the manufacturing process and increases the yield as compared to the conventional method.



Nanotechnology, Materials & Manufacturing

A novel and practical method for pressureless sintering of boron carbide

New process greatly lowers the cost of manufacturing the hardest and lightest engineering ceramics

Boron carbide (B_4C) has the highest hardness (HV = 38 GPa) and lowest density (2.52 g/cm³) among all practical engineering ceramics. However, its sinterability is poor due to the strong covalency of B-C bonding. Densification of B_4C to high densities is usually achieved by hot pressing, which makes the manufacturing cost high and precludes the formation of complex shapes. In this study, we have developed a new method of pressureless sintering of B_4C . When placed in an Ar gas atmosphere containing gaseous Al compounds, B_4C green bodies can be sintered to over 95 % of theoretical density under 2200 °C. When B_4C green bodies are doped with less than 1 % of sintering additives such as Si and W, even higher sintered densities can be achieved. Because they have high sintered densities and contain minimum amount of deleterious secondary phases, the B_4C ceramics prepared by this method have high hardness and mechanical strength. This simple and efficient pressureless sintering method will greatly lower the manufacturing cost of B_4C ceramics and promote their industrial applications in the fields, for example as sandblast nozzles, mechanical seals, hard disk substrates, and other components requiring wear-resistance and light weight.

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AIST TODAY Vol.8, No.6 p.23 (2008)



Schematic drawing of the developed sintering technique

Development of an efficient tandem type dye-sensitized solar cell

New architecture of dye-sensitized solar cells for higher conversion efficiency

Dye-sensitized solar cells are expected to be next generation solar cells because of possible low cost production. In order to improve the solar light to electricity conversion efficiency, we examined a tandem type dye-sensitized solar cell. In a tandem cell, the top cell converts shorter wavelength light energy to electricity, and the bottom cell converts longer wavelength light energy. We have developed a new process of preparation of highly transparent nano-structured titanium oxide film electrode for the top cell and multi-layered film electrode for the bottom cell. The conversion efficiency of 11.0 % was demonstrated by using conventional Ru complex dyes as sensitizers. Higher conversion efficiency will be realized when we develop high performance sensitizers optimized for tandem type solar cells.



Metrology and Measurement Technology

Accurate measurement of the volume of silicon spheres Interferometeric determination of the sphere volume for the redefinition of the kilogram based on an atomic mass

Many of the units of physical quantities, such as frequency, length, voltage, resistance, and time are defined using physical laws or quantum effects which are considered to be universal and immutable. In contrast, the kilogram, the unit of mass, has continued to be based on a material artifact. The Avogadro constant is a fundamental physical constant that expresses the number of atoms or molecules contained in 1 mol of a substance. Therefore, an accurate determination of this constant would enable the kilogram to be defined based on an atomic mass. An interferometer equipped with a direct optical frequency tuning system has been developed for a determination of the Avogadro constant by the X-ray crystal density method. The volume of 1-kg silicon spheres is determined by this interferometer with a relative standard uncertainty of 3.0×10^{-8} , being the smallest uncertainty achieved for the silicon spheres to date. At present, an international project for the precise determination of the Avogadro constant role in this project. An isotropically enriched silicon crystal is used in this project to further increase the accuracy of the Avogadro constant. This would realize the redefinition of the kilogram.

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An interferometer equipped with a direct optical frequency tuning system to determine the volume of 1-kg silicon spheres. A 1-kg silicon sphere is placed in the center of the interferometer. The diameter of the sphere is about 94 mm.

Accurate quantification for the environmental pollutants at trace levels

Accurate quantification using ¹³C-labeled internal standards

We are developing a certified reference material for the accurate quantification of polycyclic aromatic hydrocarbons (PAHs) at trace levels (< 1 μ g/g). PAHs are formed through incomplete combustion and are transported for long distances. It is therefore necessary to monitor the levels of emitted PAHs not only in highly contaminated samples, but also in less-contaminated environmental samples. To develop matrix-type certified reference materials, isotope-dilution mass spectrometry (IDMS) is commonly applied for the accurate quantification of not only PAHs but also other pollutants. The IDMS method is based on the principle that internal standards with stable isotope behave in the same manner as the target (native) compounds throughout analytical processes. However, it is not true for the quantification of PAHs at trace levels using deuterium-labeled PAHs although this is true for those at high levels (> 1 μ g/g). In the light of this fact, we used ¹³C-labeled PAHs as internal standards to develop the matrix-type certified reference material of PAHs at trace levels.

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Metrology and Measurement Technology

Development of an optical lattice clock Toward the next-generation atomic clock

We are developing an optical lattice clock toward realization of the next-generation atomic clock. Optical clocks have an intrinsic superiority over the present Cs microwave clock, which is the definition of the SI second, because they can split time with a much higher rate. These ticks can be precisely counted by using an optical frequency comb, the inventors of which were awarded the Nobel Prize in 2005. The long remaining problem to search for the next-generation optical atomic clock can be solved by the revolutionary idea of the optical lattice clock proposed by Prof. Katori of the University of Tokyo in 2001. At AIST, we started developing the optical lattice clock using ytterbium atoms. This innovation will lead to the redefinition of the second in the near future.

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Conceptual image of the optical lattice clock

The 41st Science Advisory Committee of International Science and Technology Center

The 41st Science Advisory Committee (SAC) of the International Science and Technology Center (ISTC) was held from February 18 to 20 at AIST Tsukuba. The ISTC Secretariat is located in Moscow, and its objective is to support researchers of the former Soviet Union with contributions from such countries as Japan, the United States, the European Union, Canada and Korea. SAC is a committee that recommends proposed projects. Dr. Yutaka Murakami, Senior Research Scientist of AIST, is a cochairman, and 15 members of SAC and ISTC Secretariat participated.

On the first day, ISTC related officers of the Ministry of Foreign Affairs and the Ministry of Economy, Trade and Industry attended, and Vice-President Masakazu Yamazaki of AIST gave an opening speech. During the next two days, focusing on recommendation of proposed projects, SAC activities and future action policies were discussed. On the last day, the outline of AIST was presented, and views were exchanged on possibilities of collaboration with Russia. Lastly, there was a tour of the research centers of nanotechnology and photovoltaics, and the three day schedule came to a close.

This meeting was a step toward working effectively with ISTC Headquarters in promoting collaborative research between AIST and Russia in the future.



AIST Vice-President Yamazaki greeting the members

A Visit by Thailand Minister of Science and Technology

On March 7, AIST Tsukuba and Tokyo Headquarters were visited by Mr. Wutipong Chaisang, Thailand Minister of Science and Technology, Minister's advisor, representatives of MOST (Thailand Ministry of Science and Technology), of NSTDA (National Science and Technology Development Agency) and TISTR (Thailand Institute of Scientific and Technological Research) both of which had signed comprehensive agreements with AIST, and of Royal Thai Embassy in Japan. There were a total of 26 people, including the press.

The Minister is highly interested in BTL (Biomass To Liquids), and although he had only recently become a cabinet member of Prime Minister Samak Sundaravej' s new administration, he strongly wished to visit AIST during his visit to Japan. At Tsukuba, after a welcoming speech by the then Senior Vice-President Tatsuo Katsura,



Minister Wutipong, President Yoshikawa, Vice-President Yamazaki and others (Tokyo Headquarters)

the Minister and the party moved to Biomass Technology Research Center and Research Center for New Fuels and Vehicle Technology, and keenly observed the research sites. At Tokyo Headquarters, a courtesy call was made to President Hiroyuki Yoshikawa, and strengthening of the future cooperative relations was expressed in environmental and energy fields especially related to Biomass. At the same time, the Minister expressed his gratitude to the President for his valuable advice as chairman of the NSTDA International Advisory Board. Special reference was made to NAC2008 (NSTDA Annual Conference) of March 24 to be attended by Her Royal Highness Princess Sirindhorn where the Minister looked forward to meeting the President again.

This AIST visit by Minister Wutipong was very significant in maintaining the relations of research cooperation between the two countries.



Minister Wutipong, then Senior Vice-President Katsura and others (Tsukuba)

Report on "nano tech 2008" International Nanotechnology Exhibition & Conference

"nano tech 2008", International Nanotechnology Exhibition & Conference was held from February 13 to 15 at Tokyo Big Sight.

"Nanotechnology" taken as a core technology, and "the fascinating world of nanotechnology from cutting-edge technologies to newest products" being the catchphrase, numerous exhibits as Nano Bio Expo 2008, ASTEC 2008, METEC '08, neo functional material 2008 were simultaneously held. The closing time was especially extended for one hour to enable as many people to attend. According to the sponsor report, 522 enterprises including 198 from 23 foreign countries participated, with 884 booths. The number of visitors is increasing by the year, and there were over 49,000 visitors at this event, showing that it is not only good for information gathering but also serves as a place for business matching.

AIST exhibited 28 research results from leading research areas as nanomaterial, nano fabrication, nano metrology, nano sensor, nano bio, and social receptivity. During the



Awarding ceremony

exhibition, in order to deepen understanding of AIST, we not only presented our research results but also tried to actively e x c h a n g e in for mation with the visitors. In the afternoons, presentations of AIST technological seeds were made within the booths, in order to disseminate research results for more concrete collaboration among industry, academia and



AIST booth

government as in joint research and technological transfers.

Furthermore, at this event, novel and pioneering forms of technology were selected among exhibitors and commended. AIST was honored with the nanotech Special Award and was given the award on the last day of the exhibition. The reason for the award was that "AIST succeeded in developing carbon nanotube ink that has three primary colors, by separating, with high purity, carbon nanotubes (CNTs) into metallic CNTs and semiconductor CNTs. This has led to great progress in electronic applications. The Institute has also succeeded in developing a mass synthesis approach for organic nanotubes composed of amphiphilic compound which serve as materials with new inclusive functions such as drug delivery systems (DDS), and this was a major step toward application in bioindustry."

4,200 printed pamphlets of all AIST exhibits were gone by the morning of the last day which shows the surprising number of visitors we had. Our staff was constantly kept busy responding to enquiries. We would like to thank all those who visited our booths.

Thailand NSTDA International Advisory Board and NSTDA Annual Conference

The International Advisory Board meeting of NSTDA (National Science and Technology Development Agency), a core research organization of Thailand that has signed a comprehensive agreement with AIST, was held in Thailand from March 20 to 22, and President Yoshikawa chaired the meeting. There were discussions on a wide range of topics which are also of AIST concerns, as the balance between basic research and applied research, the matrix management of the organization and interdisciplinary research, strengthening of collaboration among industry, academia, and government, human capacity development, and special research topics of the future.

NSTDA Annual Conference was held at Science Park on March 24, and Her Royal Highness Princess Sirindhorn and Mr. Wutipong Chaisang, Minister of Science and Technology were present. President Yoshikawa gave a keynote lecture titled, "Science and Technology for Society", in which he presented the role and the direction that science and technology should take. Minister Wutipong had visited AIST on March 7 and had exchanged views with the President. NSTDA and AIST hold a workshop every year, and President Sakarindr Bhumiratana of NSTDA is a member of AIST Advisory Board. This Board meeting and conference helped to further strengthen the intimate research collaboration between the two organizations.



Members of the NSTDA International Advisory Board

Exhibition at Hannover Messe 2008

AIST exhibited at "Hannover Messe 2008", the world's largest trade fair held from April 21 to 25 at Hanover, Germany. At this Hannover Messe, as Japan was the official Partner Country, we exhibited 32 R&D results and products of technological-transfer venture corporations, a number 3 times that of last year.

During the official walk on the first day, Chancellor Angela Merkel of Germany visited the Japan Pavilion and viewed the humanoid robot HRP-2. Moreover, Japan Special Envoy, former Prime Minister Shinzo Abe, and Mr. Masashi Nakano, Senior Vice Minister of Economy, Trade and Industry (METI) visited AIST booth, and viewed the therapeutic robot, Paro, switchable glass / mirror, and claybased film, Claist.

During the fair, over 4,000 people visited the AIST booth, and there were over 400 technological consultations, and around 20 business negotiations concerning purchase of development devices and agency contracts.



AIST booth at Hannover Messe 2008

Symposiums were also held along with the exhibits.

Helmholtz-Gemeinschaft, the leading research association of Germany, Forschungszentrum Jülich and Forschungszentrum Karlsruhe, both members of the association, each signed a comprehensive agreement with AIST to promote research collaboration on 21 and 22. To commemorate this, "Germany-Japan Research Collaboration Workshop for Sustainability" was held on 22, and Senior Vice Minister Nakano gave a congratulatory speech.

In addition, co-sponsored with Japan External Trade Organization (JETRO), "Japanese-German Photovoltaic Energy Initiative Symposium" was held on 24, and there were reports and panel discussions by Japanese and German administrations, corporations and research institutes. There were over 180 participants, far exceeding expectations, which demonstrated the high interests of both countries in the field.

Furthermore, "Japanese-German Business Summit", "German-Japanese Roadmap Symposium", "1st German-Japanese Summit on Mobile Robots & Autonomous Systems" organized by other organizations were held, and they were attended by Senior Vice-President Akira Ono of AIST, Director Toshimi Wada of Collaboration Promotion Department, and Dr. Takanori Shibata and Dr. Osamu Matsumoto, Senior Research Scientists.

With the exhibition and symposiums, international technological transfer and research collaboration expansion in the future are anticipated.

Cover Photos Above: Minister Wutipong, President Yoshikawa, Vice-President Yamazaki and others (p. 18) Below: Bayan Obo, China (p. 19)



ADVANCED INDUSTRIAL SCIENCE AND TECHNOLOGY (AIST)

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