A new framework of bidirectional collaborative research program has been started in 2010 for the study of nuclear fusion reactor materials. By utilizing the environment of the research center at each university, joint research is conducted interactively between the centers and the National Institute of Fusion Science (NIFS). Two topics that were conducted in this framework are described in this paper.

The first topic is a study of a trapping mechanism of hydrogen isotopes in neutron-irradiated plasma-facing materials [1]. One feature of fusion reactor environments is that helium and hydrogen are produced by nuclear transmutation, and the transport and retention of hydrogen isotopes and helium from the core plasma and tritium generated at the blanket occur under neutron irradiation. It is hence indispensable to clarify the effects of neutron irradiation on the behavior of hydrogen isotopes and helium in the candidate materials to assess the feasibility of their use in fusion reactors.

For this, in 2010 a thermal desorption spectrometer (TDS) apparatus (Fig. 1) was installed in a radiation-controlled laboratory at the International Research Center for Nuclear Materials Science (IRCNMS). This apparatus allows us to obtain thermal desorption spectra, thereby enabling the identification and quantification of hydrogen isotopes and helium contained in radioactive materials following reactor or accelerator irradiation. In 2011, the TDS apparatus was equipped with an ion gun to inject hydrogen isotopes or helium into neutron-irradiated specimens.

By using this apparatus, the emission and retention behavior of D was measured in recrystallized tungsten (W) specimens that were first irradiated with 2.8 MeV Fe ions to 3 dpa (displacement per atom) and then exposed to D atoms with very low energy and low flux (fluence: 6.2×10^{22} Dm^{-2}); the result is shown in Fig. 2. For comparison, the results for W specimens irradiated with neutrons or Fe ions and then exposed to TPE (tritium plasma experiment: high energy, high fluence 10^{25} D m^{-2}) are included.

In the case of TPE exposure, neutron irradiated specimens exhibit D desorption from relatively low temperatures to as high as above 1000 K, whereas Fe-ion-irradiated specimens exhibit the completion of D desorption at around 700 K. In the case of specimens with Fe-ion irradiation and D atom exposure, D desorption starts at around 500 K and continues even above 1000 K. The D desorption behavior suggests the existence of trapping sites for...
hydrogen isotopes that are as strong as those in neutron irradiated specimens (de-trapping energy: ~2 eV). These results indicate that 2.8 MeV Fe-ion irradiation can induce strong trapping sites (de-trapping energy: ~2 eV) for hydrogen isotopes and that such strong trapping sites survive under low-flux D exposure but disappear under high-flux D exposure. The de-trapping energy of ~2 eV corresponds to chemisorption entrapment of hydrogen isotope atoms at the inner surface of voids in W.

The second topic is the neutron-irradiation effect on the superconducting magnet systems of fusion devices [2]. A fusion reactor yields many high-energy neutrons, and some of them stream out of the plasma vacuum vessel, penetrate the blanket system, and reach the superconducting magnets that provide high magnetic fields to confine high-energy ionized particles. Under neutron irradiation, the magnet materials are activated and their properties may change.

Changes in the superconducting properties of the superconducting wires and degradation of the interlaminar shear strength (ILSS) of electric insulation materials are the most concerning possible irradiation effects. The superconducting magnet system (Fig. 3), which was installed in the radiation control area of IRCNMS, is a so-called dry magnet working without liquid helium. The magnet generates 15.5 T as the maximum field in a 52 mm RT bore, and it is equipped with Gifford–McMahon (GM) refrigeration with an air-cooled compressor. The magnet system requires only electric power.

A variable temperature insert (VTI) included in the system is a kind of sample holder that has a capacity of 500 A for a sample current. The sample holder is cooled down by thermal conduction with GM refrigeration, and the sample temperature can be controlled from 4.5 K to around 20 K at 500 A. The control system consists of amplifiers and a computer. The control computer is connected to another computer located in a non-radiation-control area by a dedicated internet line. Users are able to change the test conditions from the non-radiation room once an irradiated sample is set up. The 15.5 T superconducting magnet is placed and the VTI is set up on the magnet. Samples irradiated in nuclear reactors will be tested with the new facility, and very important data on the critical current, critical magnetic field, and critical temperature will be obtained.

The two experimental devices described above are open for collaborative works not only with Japanese researchers but also with foreign researchers.

References

Tatsuo Shikama (International Research Center for Nuclear Materials Science)  
E-mail: shikama@imr.tohoku.ac.jp  
URL: http://www.imr-oarai.jp/

Fig. 3 15.5 T superconducting magnet (lower) and variable temperature insert (upper) in a magnetic field shield structure.
The most important problem/issue in today’s world is the creation and management of energy without affecting our environment. Electrical energy is the most important form of energy, and without it our society will collapse. Electrical energy is mostly obtained at the cost of our Earth’s natural resources (coal, oil, etc.), and it sometimes involves dangerous methods of production (nuclear power plants). Therefore environmentally friendly methods of production and proper management of electrical power are very important. For example, in power applications such as transformers and motors, non-oriented silicon steel and oriented silicon steel are mainly used because of their high magnetic flux density \( B \), but the electrical power loss in the Si–steel cores is still high. Core loss \( W \) in silicon steels has been improved, but further improvement seems impossible. With the newly developed soft magnetic amorphous/glassy materials (in our and other laboratories), it is possible to further decrease the electrical power loss, but these materials have lower \( B \). High \( B \) is important for reducing the size and weight of the electrical devices (which is necessary to make them energy efficient). Therefore, development and mass production (at low cost) of nano-crystalline soft magnetic alloys that can simultaneously exhibit high \( B \) similar to Si steel (~2.0 T) and low magnetic core loss \( W \) as amorphous metals is ideal.

We at ARCMG are close to the development of this dream material. The new Fe-rich nano-crystalline soft magnetic material \( \text{Fe}_{85-86}\text{Si}_{1-2}\text{B}_{8}\text{P}_{4}\text{Cu}_{1} \) is composed of ~95 mass% Fe without rare-earth metals [1]. This low-cost soft magnetic material exhibits high magnetic flux density (1.8–1.9 T) along with extremely low core losses (1/2–1/3 of ordinary materials). Fig. 1 shows the changes in magnetic core loss \( W \) at 50 Hz for the \( \text{Fe}_{85-86}\text{Si}_{1-2}\text{B}_{8}\text{P}_{4}\text{Cu}_{1} \) nanocrystalline alloys as a function of maximum magnetic induction \( B_m \). The data for amorphous \( \text{Fe}_{78}\text{Si}_{9}\text{B}_{13} \), highest-grade oriented Fe–3 mass% Si, and non-oriented Fe–3 mass% Si and Fe–6.5 mass% Si alloys are also shown for comparison. These special nano-crystalline soft magnetic materials are produced by the careful crystallization of amorphous/hetero-amorphous metallic alloys. Amorphous/glassy alloys are attractive because of their unique random arrangements of atoms, which result in excellent properties such as soft magnetism, high mechanical strength, and corrosion resistance. Additionally, these metallic glasses have precision micro-/nano-machining capabilities, which make these materials very important for future nanotechnology applications [2]. We have been developing new Fe-based, Co-based, Cu-based, Ni-based, Ti-based, Zr-based, and Mg-based glassy/amorphous alloys for different required functionalities. In the development of these special materials for energy saving or precision machining, alloy design and processing condition optimization are very important. At ARCMG, we have state-of-the-art facilities (automatic, semi-automatic, or manual) to develop such materials (Fig. 2).

Besides the reduction of power loss in electrical...
devices, storage of electrical power is also very important for the next generation of hybrid/electrical cars. Lithium-ion rechargeable batteries are very important. We at ARCMG are also focusing on the growth of single crystals for Li-ion batteries. Spinel-type Li4Ti5O12 (m.p. 1288 K) single crystals for negative electrodes in lithium-ion rechargeable batteries were grown by a NaCl (m.p. 1074 K) flux growth method in air [3]. As shown in Fig. 3, the grown crystals had an octahedral shape with well-developed facets and a flat surface. The TEM image in Fig. 3 indicated that the Li4Ti5O12 crystals were of very high crystallinity without point defects or dislocations. Environmental damage and product costs are thought to be greatly reduced in the flux growth technique using NaCl flux.

In addition to experimental studies, it is also possible to gain a theoretical understanding of various amorphous and nanocrystalline alloys/materials using computer simulations.

For example, a detailed study of the cooling process of Pd42.5Cu30Ni7.5P20 glassy alloy has been done using first-principles calculations, and the results were compared with the data obtained from a synchrotron X-ray diffraction experiment. The calculated densities of states show noticeable changes in the electronic structures of Pd42.5Cu30Ni7.5P20 compositions during a cooling process. The detailed analysis of the atomic and electronic structure of the alloy in the liquid and glassy states reveals the formation of short-range chemical order in the temperature range corresponding to a non-Arrhenius temperature dependence of viscosity [4]. The theoretical group at ARCMG has a great deal of expertise in the application of modern theoretical methods for analyzing important properties of experimentally developed alloys or suggesting new alloys to experimentalists, based on their theoretical understanding.

ARCMG not only is engaged in new cutting-edge scientific research but also focuses on industry-related issues in the development of these new materials. The new Fe-based soft magnetic nano-crystalline alloy developed in ARCMG is an excellent example. Besides the excellent soft magnetic properties ($B_s \sim 1.8 T; H_c \sim 3–5 A/m$), this alloy has low materials (only needs industry-grade materials, without expensive materials such as rare earths) and production costs (can be produced in air), which are extremely attractive for the commercialization of these alloys.

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Akihiro Makino (Advanced Research Center of Metallic Glasses)
E-mail: amakino@imr.tohoku.ac.jp
URL: http://www.arcmg.imr.tohoku.ac.jp/
In-field Heat-treatment Processing for New Materials Development Using the Cryogen-free Hybrid Magnet

High Field Laboratory for Superconducting Materials

In order to realize a hybrid magnet with no need of liquid helium for operation, we have developed a cryogen-free hybrid magnet, consisting of an outer cryogen-free superconducting magnet and an inner water-cooled resistive magnet. This easy-to-operate cryogen-free hybrid magnet is now operating for application-oriented research in high fields up to 27.5 T and at high temperatures up to 1400 K in combination with an electric furnace.

The High Field Laboratory for Superconducting Materials (HFLSM) has concentrated on the three key research fields of “High field magnet technology,” “Materials science in high fields,” and “Basic research under multiple extreme conditions” as cooperative research in the Institute for Materials Research, Tohoku University. The HFLSM has demonstrated the first instances of various kinds of cryogen-free superconducting magnets in the world [1]. A new research field that is called magneto-science has made great progress using a static high field generated by one such unique cryogen-free superconducting magnet. Magneto-science encompasses crystal growth, chemical reactions, biological reactions, and materials processing in high fields. It is especially important to focus on functional materials development from the melt in high fields.

In order to make an easy-to-operate hybrid magnet system, we have constructed a cryogen-free 27.5 T hybrid magnet (28T-CHM), consisting of an outer cryogen-free 8.5 T superconducting magnet and an inner traditional water-cooled 19 T resistive magnet. Fig. 1 shows 28T-CHM, which generated 27.5 T in a 32 mm room-temperature experimental bore [2], and this cryogen-free hybrid magnet surely demonstrates quite flexible machine time for use in long-term experiments.

We also installed a differential thermal analysis (DTA) combined with 28T-CHM. The DTA measurement furnace system consists of Pt crucibles and an alumina support equipped with two pairs of Pt-PtRh thermocouples allowing the temperature difference to be measured [3]. Sample powders and a reference sample of Al$_2$O$_3$ powders were investigated. The maximum temperature was assumed to be 1200°C when the temperature increased in the argon atmosphere.

The motor systems of evolving hybrid electric vehicles require a working potential at high temperatures above 200 °C. The coercivity decrease of a NdFeB magnet has been eased by the addition of Dy [4]. However, this rare-earth metal is now restricted as a strategic material, and in the future it will become quite difficult to obtain important rare-earth metals such as Dy. Therefore, the development of rare-earth-free bulk magnets at the temperature range of 500–600 K is needed as innovative materials science.

On the other hand, it is generally known that MnBi has a high coercivity even at 500 K [5]. However, since Bi and Mn usually remain as individual foreign phases, it is difficult to synthesize single-phase MnBi, and as a result, the development of a high-quality MnBi synthesis process is required. The first-order phase transition through the liquid phase in high fields and the magnetic anisotropy on the crystallographic axis for the MnBi system are expected to produce various functional compositions with in-field heat treatment. This means that the magnetic characteristics can be controlled by the in-field heat treatment. Therefore, it is necessary to make an equilibrium phase diagram for the efficient investigation of materials development in high fields.
Bi–Mn samples with different compositions were made by the method of arc-melting followed by heat treatment. The sample composition was determined by inductively coupled plasma optical emission spectroscopy (ICP-OES). Diffraction peaks from the low temperature phase of ferromagnetic MnBi and Bi were observed in the Bi-rich composition. On the other hand, diffraction peaks from MnBi, Mn, and unreacted Bi were observed in the Mn-rich composition.

The DTA curves for each composition were measured under a zero field. The endothermic peak ($T_B$) from the appearance of the Bi–Mn liquid phase from the ferromagnetic MnBi phase and Bi was observed in all the compositions. This result corresponds to the diffraction peak from Bi observed in the X-ray diffraction measurement. Since unreacted Bi remained in the sample, the endothermic peak from Bi was also observed in the Mn-rich composition. Moreover, an endothermic peak ($T_t$) was observed due to a decomposition reaction accompanied by a magnetic transformation of ferromagnetic MnBi + Bi–Mn liquid phase to paramagnetic Mn$_{1.08}$Bi + Bi–Mn liquid phase. In addition, an endothermic peak ($T_m$) due to the peritectic reaction of Mn$_{1.08}$Bi + Bi–Mn liquid phase $\rightarrow$ Mn + Bi–Mn liquid phase was observed.

The DTA curve measured when a magnetic field was applied to Bi–53at.%Mn is shown in Fig. 2. One notes that the endothermic peaks of $T_B$ and $T_m$ are unchanged in fields up to 18 T, while $T_t$ increases with applied magnetic field. However, the $T_t$ increase clearly deviates from linear behavior above 20 T, as shown in Fig. 3 [6]. We found that the magnetic energy of Mn$_{1.08}$Bi at $T_t$ increases nonlinearly in high fields above 20 T and becomes large, comparable to that of MnBi. These results show that we can generally control the composition, decomposition temperature, and the equilibrium state of ferromagnetic magnetic materials by a high magnetic field if the materials have a large magnetic moment.

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Kazuo Watanabe (High Field Laboratory for Superconducting Materials)
E-mail: kwata@imr.tohoku.ac.jp
URL: http://www.hfism.imr.tohoku.ac.jp
Challenge for Metallic Material Innovation by Collaboration between IMR, Industry, and Government

Kansai Center for Industrial Materials Research

A collaboration project (Kansai Center) between IMR, Osaka Prefecture Government, and enterprises in the Kansai area, sponsored by the Ministry of Education, Culture, Sports, Science, and Technology, was established on April 2011. The Kansai Center strives to create a new value in manufacturing in the metallic material industry and to contribute to human resource development through collaborations with industry and government.

The Kansai Center was established in Osaka as a special unit of the Institute for Materials Research, Tohoku University in April 2011, based on an agreement with Osaka Prefecture Government. The center is sponsored by the government (Ministry of Education, Culture, Sports, Science, and Technology), and it took over the project of the Osaka Center, which was carried out from 2006 to 2011 aiming to support small and medium enterprises in the Osaka area. There are three primary scopes of the Kansai Center. First, we respond to technical problems in enterprises related to metallic materials and their process technologies, and we work with the industry to solve them. Second, the Center introduces academic research output to industry and strives to apply collaborative research with companies. Third, the Center educates next-generation materials scientists and researchers in universities and enterprise about material science applications. The project is conducted under various collaborations with other organizations in order to achieve efficient activities, especially with the Development Center for New Metallic Material at Osaka Prefecture University and the Research Center for Nano-Micro Structure Science and Engineering at the University of Hyogo. There are seven groups in the Center: Environmental Protection and Energy Conversion Materials, Functional Materials for Future Industries, Analytical Science and Technology, Structural Materials for a Low-Carbon Society, Nanostructure Control for Engineering Materials, Advanced Biomaterials, and Structure Design of Green Metallic Materials.

The Center has some unique features and activities. First, the Kansai Center has six full-time university academic staff members (four in Osaka and two in Hyogo) to achieve the work, which is different from other similar organizations at other universities. This enables us to concentrate on collaborative activities with industry and government. Second, the Center has various collaborations with other organizations and supporting industries, such as the Osaka Bay-area Consortium, Research Society for Global Materials Crisis, and General Production Companies, which enables efficient support of industries. Third, seven professors in the Center respond to technical inquiries from industries themselves, and this saves investigation and problem-solving time. Fig. 1 shows the number of inquiries from industries at Creation Core Higashi Osaka (a governmental body where sixteen universities and one college are located to respond to inquiries from industries). It is clear that the Kansai Center occupies the largest portion among the universities, and the number of inquiries to the Kansai Center increase each year.

The Kansai Center has opened technical seminars named “Monodukuri Kisokozaka” on special topics to provide scientific knowledge to technicians and researchers in industry. The seminars are organized by the professors in the Center, and the topics were selected based on industrial needs. Since fall of 2011, the new seminar has started to provide fundamentals of individual metals, such as plasticity, corrosion, and characteristic functions, in addition to history and production. Furthermore, the Center professors provide lectures on materials design, technical skills, and related fundamentals of
materials science to the students in Osaka Prefecture University and Hyogo Prefecture University to demonstrate the importance of material science contributions to industrial applications.

The Kansai Center developed structural and functional materials under collaboration with industries in the Kansai area. Last July, the first application using academic outputs was commercialized by an enterprise in Osaka: Ni-based two-phase intermetallic alloys composed of Ni3Al and Ni3V [1] were applied to the tool materials of friction stir welding (FSW) equipment (Fig. 2). This alloy exhibits extraordinarily high hardness above 700 °C compared to commercial WC hard materials, which is beneficial for heat-resistant materials.

Fig. 3 shows a sink roll with a Ni–Mo–B amorphous spray coating film that exhibits anti-corrosion behavior in hydrochloric acid [2]. The corrosion resistance of the developed Ni–Mo–B amorphous coating film is three times higher than that of Hastelloy C276 in concentrated hydrochloric acid, and its hardness is about the same as Cermet (WC-12Co). The Ni-based amorphous coating is expected to be applied to machine parts under heavy corrosive atmospheres.

Photocatalytic TiO2 prepared by anodic oxidation [3] for water-purification is under development with an enterprise in Osaka. Fig. 4 shows a prototype device (left) and an incorporated Ti lath coated with anodic oxide (right), which could purify the contaminated water.

The Kansai Center strives to innovate in the metallic material industry through collaborations between industry, academia, and government and to promote material science.

Fig. 2 FSW (friction stir welding) equipment (left) and a non-consumable tool composed of Ni-based two-phase intermetallic alloys (right).

Fig. 3 A sink roll with a Ni–Mo–B amorphous spray coating film (lower) and its hardness (upper).

Fig. 4 Prototype device (left) and incorporated Ti lath coated with anodic oxide (right).

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Naoya Masahashi (Kansai Center for Industrial Materials Research)
E-mail: masahashi@imr.tohoku.ac.jp
URL: http://www.kansaicenter.imr.tohoku.ac.jp/index.html
Invention of New Materials by Supercomputer

Center for Computational Materials Science

Simulation is becoming more and more important as a third tool, along with experiment and theory. The research fields of materials design covered by computer simulations are expanding rapidly. The newly implemented supercomputer system HITACHI SUPER TECHNICAL SERVER SR16000 Model M1 has a 300 TFLOPS processing speed, 42.2 TB of memory, and 1.4 PB of disk storage capacity. We are planning to establish the basis to serve this large-scale supercomputer resource as one of the centers within a nationwide project headed by the K computer that form a network of researchers in materials design in Japan as well as worldwide.

The aim of computational materials science is to design and expedite the synthesis of new materials with novel or extreme properties. One of the advantages of computational materials science is that it can guide this exploration by providing efficient strategies. Mutual collaboration between experiments, theories, and computer simulations is important for materials science. Providing stimulating and useful computational environments is the key role of the Center for Computational Materials Science (CCMS). It includes high-performance supercomputers, the latest versions of software, and a high-speed network.

CCMS was developed from the Laboratory of Materials Information Science that was established in February 1989. Its main tasks are as follows:

1. Administration and maintenance of the supercomputing system in this Institute and maintenance of the supercomputing system network.
2. Support the usage of the supercomputing system.
3. Support materials design by supercomputing simulation with parallelization.
4. Support HPCI (High-Performance Computing Infrastructure) and CMRI (Computational Materials Research Initiative). A brief introduction to CMRI is given in the last paragraph.

![Supercomputing system for computational materials science](image-url)

Fig. 1 System configuration.
The supercomputing system at CCMS aims to serve as a solution for researchers in materials design to realize a sustainable world by computer simulation. Fig. 1 shows the system configuration of the supercomputing system at CCMS, which was newly installed in April 2012. The details of each system are as follows:

1. The supercomputer is the HITACHI SUPER TECHNICAL SERVER SR16000 Model M1, which is a shared-memory type parallel machine (water-cooled system). It has a total processing power of 300 TFLOPS composed of 320 nodes each having 980.48 GFLOPS. This is a 40 times speed increase from the previous machine. It has 42.25 TB of main memory, which serves enough computational space for the large-scale computing required by the institute.

2. The application server is an IBM Blade Center HS22, which is a distributed memory parallel machine. It consists of 50 computing nodes and 4 GPFS servers. It is mainly used for software applications.

3. The file server is a HITACHI ENTERPRISE SERVER EP8000, which manages each user's home directory.

4. The disk storage system is a Hitachi Adaptable Modular Storage2500, which consists of a 1.4 PB storage area, sufficient for intermediate storage of large scratch files.

Fig. 2 shows an example of the results of large-scale simulations for the theoretical design of new conductive oxides. Transparent conducting oxides (TCOs) are a unique class of materials that are optically transparent and electrically conducting with a wide variety of optoelectronic applications, such as transparent electrodes in flat panel displays, photovoltaic cells, electrochromic devices, and transparent semiconducting devices. One common TCO is tin-doped indium oxide (ITO), owing to its good conductivity and high optical transparency. At present, joint experimental and theoretical \textit{ab initio} investigations are being performed to predict a new TCO with the prescribed characteristics of interest. \cite{1}

Finally, we introduce CMRI. CMRI was established in 2011 in IMR as a project-oriented section and is one of the three operating institutions of the Computational Materials Science Initiative (CMSI), which was founded through a Grant for Field 2, “New Materials and Energy Creation,” of the HPCI Scientific Program of the Ministry of Education, Culture, Sports, Science, and Technology.

The goal of CMRI is to use supercomputers, among which the K computer boasts the world’s top performance, and the supercomputer system to innovate state-of-the-art computational materials research. Current research topics include first-principles calculations of mechanical properties, interface mechanical/functional relationships, and evolution of microstructure, etc. of various metallic alloy systems. CMRI researchers have been developing their own program codes for these projects, based on electronic structure and statistical mechanics theories.

In recognizing the importance of forming the basic infrastructure that will lead to the next generation of computational materials science, CMRI offers seminars and symposia on parallel computing and collaborative meetings with company researchers. CMRI also promotes training and education in computational materials science for students and researchers in universities and companies. \cite{2}

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Ryoji Sahara (Center for Computational Materials Science)
E-mail: ccms-adm@imr.tohoku.ac.jp
URL: http://www-lab.imr.edu/~ccms/Eng/index.php
Growth and Characterization of Alloys of GaN, InN, and AlN for Optoelectronics by ICC-IMR Visiting Professorship

International Collaboration Center (ICC-IMR)

ICC-IMR is a gateway to IMR from the worldwide community of material science. Those who want to perform collaboration with the groups in the institute can apply for a variety of programs. Here is an example of the achievement on the growth of nitride semiconductors conducted as a guest professorship.

ICC-IMR was founded in April 2008 to succeed the International Frontier Center for Advanced Materials (IFCAM). The mission of the center has extended from an international think-tank of materials science to the center for international research collaborations of the Institute for Materials Research. To achieve this purpose, the program of the center has expanded to include the research collaboration projects by international teams.

As one of the centers of excellence in material science, IMR holds 27 research groups and six research centers. ICC-IMR is working as the gateway for collaborations between international researchers and IMR. At present, the following seven different programs are carried out in ICC-IMR: 1: international project research, 2: visiting professorship, 3: short single research visits, 4: international workshops, 5: fellowship for young researchers, and 6: coordination of international research collaboration. 7: Material Transfer Program. We welcome applications for those international programs from all over the world.

Prof. Baskar, Crystal Growth Centre, Anna University worked at ICC-IMR as a guest professor in FY2011 at the Prof. Matsuoka laboratory. Here is a report of his activities, impressions about IMR, and message for future applicants.

Upon arrival to IMR, Prof. Baskar arranged to have detailed discussions about the facilities available for the growth of InGaN materials on sapphire substrates. The works here have been made very effective by using the two MOVPE systems of the laboratory. He has grown InN and InGaN materials. The epitaxial layers were analyzed by using high-resolution X-ray diffraction (HXRD), photoluminescence (PL), infrared reflectance spectroscopy, scanning electron microscopy (SEM), atomic force microscopy (AFM), and a Hall measurement system. As such, the instruments at IMR are well prepared.

The special growth conditions, such as temperature, high V/III ratio, and high reactor pressure, required for the growth of good quality indium-based nitrides have been easily achieved.

Furthermore, in-situ monitoring systems, such as the Laytech tool, required for understanding of the growth mechanisms of indium nitride in MOVPE, particularly the shape of the initial stage nucleation and the coalescences at higher temperature to obtain two-dimensional growth, have been discussed in detail. The possible use of InGaN for high-efficiency solar cells was also discussed, particularly the issues related to p-type doping, the advantages for concentrated solar cell applications due to strong bonding between nitrogen and indium, and small changes in the band gap with temperature. Those discussions with experts are very useful for progress studying the crystal growth of nitrides.

AlGaN samples with aluminum contents from 20 to 70% were prepared by MOVPE on a two-inch sapphire substrate and cut into 1 cm² and 1 × 2 cm² size samples using a diamond scriber. The samples were cleaned in acetone and isopropyl alcohol. Indium contacts were used to measure the carrier concentration and mobility by a probe method. Though the samples are un-doped, they exhibited n-type conduction with a carrier concentration on the order of 10¹⁷ cm⁻³ up to an aluminum content of 35%. The higher aluminum content samples, above 40%, were highly resistive and the conduction type was difficult to measure. The AlGaN/GaN samples with aluminum content of 22% and thickness of around 80 nm have shown a two-dimensional electron gas (2-DEG) with a mobility of 980 cm² V⁻¹ sec⁻¹. The growth of high-aluminum-content samples on GaN surfaces without cracks and with good crystalline quality is challenging due to a 3% lattice mismatch between AlN and GaN. The samples were analyzed by scanning probe microscopy, and smooth atomic surface steps, pinholes, and micro-cracks were observed depending on the aluminium content. A typical microstructure of an AlₓGa₁₋ₓN/AlₓGa₁₋ₓN/GaN double heterostructure (DH) on GaN/Al₂O₃ observed by AFM is shown in Fig. 1.

Though the thicknesses of the GaN buffer layer and AlₓGa₁₋ₓN barrier are the same, a small thickness variation of 5 nm resulted in micro-cracks, which terminate at pinholes. The micro-cracks are formed due to the relaxation of strain once the critical thickness of the active layer of AlₓGa₁₋ₓN exceeds the limit. Attempts have to be made to grow high-aluminum-content (Al > 40%) AlGaN with high crystalline quality and smooth morphology using an...
AIn buffer with a better lattice match. Once the conditions are optimized for the AlGaN layers, resolution of the doping issues, particularly p-type doping, will be undertaken to develop high-efficiency ultraviolet light-emitting diodes. Prof. Bakar has had much success in these crystal growth studies in the very short term of his visit due to the excellent research conditions of IMR.

During his stay, he visited Nagoya Institute of Technology, Hiroshima University, Kyushu University, and Hokkaido University over 14–20 October 2011. He found that all universities are eager to establish and strengthen the bilateral academic and research cooperation with Anna University, Chennai, India. As there are a large number of Japanese companies in Chennai and discussions are occurring between the government of Japan and Government of Tamil Nadu in Chennai, India, to establish a Japanese industrial park in Chennai, many institutions are looking for a trilateral cooperation between industry in Japan or India, academia in Japan, and Anna University.

Extensive discussions were made to strengthen the semiconductor research at the Crystal Growth Centre of Anna University. It has been proposed to have an India–Japan joint workshop on nitride semiconductors for electronics, photonics, and photovoltaic applications in the year 2012 so that researchers in Japan and India can join the workshop at Anna University to bring forth joint deliberations on future strategies to establish strong collaborations between Indian universities/institutions and Japanese universities. The deliberations will also be useful to strengthen the research on nitride semiconductors for the development of novel devices.

Prof. Baskar said that the ceremony for the declaration of international material science week on 11th October 2011 at IMR, Sendai was a wonderful opportunity to share some of his thoughts about the recent disaster at Fukushima, the collaborative research between IMR and Anna University, the new initiatives in the development of materials at Anna University, and other topics for a better and sustained future for mankind.

He has expressed thanks to IMR, particularly Prof. T. Matsuoka, and Anna University, India, for their support in establishing active collaboration.

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Hiroyuki Nojiri (International Collaboration Center)
E-mail: icc-imr@imr.tohoku.ac.jp
URL: www.icc-imr.imr.tohoku.ac.jp
K. Baskar, Crystal Growth Centre, Anna University
E-mail: baskar@annauniv.edu

Fig. 1 The surface morphology of AlGaN-DH with various active-layer thicknesses. From top to bottom: 20, 15, and 10 nm QW.
Activity of the Integrated Materials Research Center for a Low-Carbon Society

The Integrated Materials Research Center for a Low-Carbon Society (LC-IMR) was founded to promote integrated research in materials science to contribute to achieving a low-carbon society. The main aim of LC-IMR is to promote outstanding projects in the field of materials research, which will contribute to making low-carbon emissions a reality through various perspectives, from energy saving to energy harvesting and storage. We are also making active collaborations with research groups outside our institution. Cooperation between industry, the general public, and the university will be sought through workshops and by actively making information available.

It is important for the field of materials science to contribute to the achievement of low carbon emissions and a sustainable society. LC-IMR was founded in April of 2010 to promote integrated research in materials science towards the achievement of a low-carbon society. Individual members of LC-IMR belong to various materials research fields, such as structural materials, semiconductors, energy storage materials, electronics, and spintronics. We believe that integration of research in different fields is very important for making a breakthrough and thus seek collaborations both among our members and with outside centers/institutes/universities.

In the past two years, we have been working on the following three subjects:

![Fig. 1 Outlook of LC-IMR.](image)
1. Conducting strategic research by individuals.
2. Supporting new research projects through management of internal research funding.
3. Having discussions with industry and government for a better understanding of the needs of our society.

Japanese funding agencies are determining that various strategic research programs in materials science are necessary for our future society. We are being funded and making good progress in those research programs.

LC-IMR is also managing an internal research program funded by IMR to support both promising and challenging research projects, particularly emphasizing on the center’s mission. Examples of the projects selected so far include the strengthening of structural steels for automobiles and the development of new soft magnetic materials and sensor materials.

To publicize the work of our center, we organize an annual workshop and introduce our research activities. We also invite key persons from outside the university, such as industry or government, to discuss how to develop suitable systems for a sustainable society and the expected roles of materials science. Such collaborations are another important part of our activities. We will continue to make efforts to progress in our missions and ask support from everyone in the world.

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Tadashi Furuhara (Integrated Materials Research Center for a Low-Carbon Society)
E-mail: lc-imr@imr.tohoku.ac.jp
URL: http://www.low-carbon.imr.tohoku.ac.jp/eng/index.html
Neutrons for Advanced Material Science

Center of Neutron Science for Advanced Materials

The Center of Neutron Science for Advanced Materials (CNSAM) is a unique neutron facility that has a background of advanced material science at IMR. CNSAM operates two neutron spectrometers for material science in a reactor facility and is also the core of a project to construct a new neutron spectrometer in J-PARC/MLF, which is the world’s brightest neutron source. Moreover, based on the crystal growth technology of IMR, CNSAM is developing novel neutron devices.

Material science using IMR spectrometer and diffractometer in a reactor facility

CNSAM operates a neutron spectrometer and neutron diffractometer for material science in the JRR-3 reactor in the Japan Atomic Energy Agency (Ibaraki, Japan). The powder diffractometer, HERMES, (Fig. 1 upper panel) is an instrument to determine the structures of spins and nuclei in materials with high efficiency. HERMES is one of the most active diffractometers for material science in Japan because HERMES is a powerful tool for investigations of magnetic materials and environmentally applicable materials.

Using HEMRES, we have succeeded in observing the microscopic magnetic characteristics of a composite magnet, Fe52Pt3011B18, based on a metallic glass, by polarized neutron diffraction (PND) experiments. PND is sensitive to small magnetic moments in magnetic materials. Fig. 2(a) shows the PND patterns of annealed Fe52Pt3011B18; I+ (red) and I− (blue) indicate the PND patterns for parallel and antiparallel neutron spins and sample magnetization, respectively. The small difference in peaks means that the peaks include weak ferromagnetic components. Fig. 2(b) shows the difference, I+ - I−, which is proportional to the ferromagnetic components, for different magnetic fields applied to the sample. Note that the ferromagnetic components of FePt remain even at 5 mT, while those of Fe2B disappear, meaning that the difference in soft and hard magnet characters can be directly distinguished by PND experiments.

The IMR neutron triple-axis spectrometer, AKANE, (Fig. 1, bottom panel) is an instrument to observe time and spatial fluctuations of spins and nuclei in materials with high accuracy. AKANE is indispensable for investigating the main issues of novel material science: high-Tc superconductors, metallic antiferromagnets, geometrical spin frustration systems, and so on. Note that observation of spin correlations under high pulsed magnetic fields, which is one of the characteristic technologies at IMR, has already been realized on AKANE.

Spin dynamics in quantum spin systems

Quantum spin systems have attracted much interest due to their non-trivial phenomena. Neutron
scattering experiments provide direct information, which connects experimental and theoretical studies. In the quasi-one-dimensional system CuGeO₃, frustration between antiferromagnetic exchange couplings within the spin-chain is reported to be responsible for the exotic physical properties. To elucidate the nature of the frustration in the spin system, we measured the full excitation spectrum of CuGeO₃ in collaboration with a group at J-PARC. Fig. 3 shows the inelastic neutron scattering intensity, which is directly related to the spin-spin correlation function. Clear continuum excitation exhibiting a quantum spin nature was observed. An intriguing feature was found in the form of a relatively strong intensity on the upper boundary of the continuum. This feature is not expected in a system without frustrated couplings, and the finding triggered a revisiting of the theoretical study of one-dimensional magnets. The observed spectrum was well reproduced by a recent model calculation that included frustrated couplings [2]. Further cooperative study between experiment and theory on quantum magnets proceeds under an operation of J-PARC.

**New projects in J-PARC**

To make a jump in material science, CNSAM is trying to realize a new project: a second-generation spectrometer named POLANO at the world’s brightest neutron beam facility at the Japan Proton Accelerator Research Complex (J-PARC), based on collaborations between Tohoku Univ. and KEK (project leader: K. Ohoyama) (Fig. 4). CNSAM is at the core of the POLANO project. The neutron spectrometer POLANO is characterized by polarized neutrons, which are very sensitive to pure magnetic correlations in magnets. Although we have already realized PND experiments on HERMES, the neutron flux for polarized neutron experiments is not sufficient for novel material science. To overcome this point and to make breakthroughs in material science using polarized neutrons, POLANO will be indispensable. From an applications viewpoint, POLANO will be important to develop rare-earth-free permanent magnet materials and for investigations of the fundamental physics in novel magnets. Since only a few polarized neutron spectrometers exist in the world, this spectrometer will be a key instrument to generate breakthroughs in novel material science in Japan.

In 2011, this project succeeded in gaining authorization by the J-PARC Center. With a goal of beginning construction in 2012FY, detailed engineering designs have already begun.

**References**


Research Centers

Open Laboratory at Cryogenic Center for Wide Research Fields from Frontier Material Science to Low-Temperature Physics

The Laboratory of Low Temperature Materials Science supplies the platforms to perform basic research on materials through the measurement of transport and thermodynamic properties over a wide range of temperatures, from 350 K to 0.01 K. Using a variety of cryogenic equipment, a number of collaborative research projects with scientists from various research fields are ongoing under the technical and educational support of the staff in this laboratory.

Recently, cryogens, such as liquid helium, have been widely used by scientists not only in the fields of low-temperature condensed matter physics and applied physics but also in other fields that are not directly related to low-temperature matter. Most generic equipment for characterizing materials with automatic PC control (for example, SQUID magnetometers, NMR systems, etc.) is now available as commercial products, and one can easily operate it from room temperature down to 1 K without special low-temperature techniques. However, they sometimes require the support of experts on low-temperature science for maintenance, containing initial cooling, keeping the equipment cooled, and troubleshooting.

On the other hand, the special apparatuses for producing very low temperature environments below 1 K have also attracted many researchers working on low-temperature physics and frontier material science, since in many cases, novel quantum phenomena in new devices, materials, and compounds appear in the extreme absence of thermal effects. Experiments below 1 K usually require relatively large-scale facilities, particular techniques, expensive cryogens such as 3He, and a large amount of liquid helium (4He) for long-term cooling. Therefore, it is effective and convenient to keep such apparatuses together with the generic equipment at one division, which works as an open cryogenic center and consists of low-temperature experts.

On the basis of the concepts mentioned above, the Laboratory of Low Temperature Materials Science (LLTMS) possesses many kinds of cryogenic equipment (as listed below) that meet the demands of most researchers and enable them to measure the basic properties of materials as a function of temperature T and magnetic field H.

(1) SQUID magnetometers (× 2 sets):
   for magnetization measurements at \( T = 1.8–350 \text{ K} \) in \( H \) up to 7 (5.5) T.

(2) VSM magnetometer (under reconstruction):
   for magnetization measurements at \( T = 4–300 \text{ K} \) in \( H \) up to 14 T.

(3) Superconducting magnet (SM) and variable temperature insert (VTI) system (× 2 sets):
   mainly for transport measurements at \( T = 1.2–320 \text{ K} \) in \( H \) up to 11 (10) T.

(4) SQUID microscope system:
   for observation of magnetic field distributions on sample surfaces at 2–100 K.

(5) Top Loading 3He cryostat and SM:
   for transport measurements at \( T = 0.26–300 \text{ K} \) in \( H \) up to 8 T.

(6) 3He cryostat and SM with field gradient coils:
   for magnetization measurements by Faraday methods at \( T = 0.3–2 \text{ K} \) in \( H \) up to 8 T.

(7) Dilution refrigerator and SM system:
   for transport and ultrasonic measurements at \( T = 0.02–1.4 \text{ K} \) in \( H \) up to 10 T.

(8) Superconducting vector magnet system with dilution refrigerator or VTI:
   for transport measurements as a function of magnetic field direction in \( H \) up to 7 T.

Taking advantage of the plentiful liquid helium made in the Center for Low-Temperature Science (cryogenic center), much of the above equipment is always kept cooled and open to all scientists of Tohoku University. In addition to in-house research, a number of cooperative projects are now in progress under the careful technical and educational support of the staff of LLTMS. LLTMS contributes to the publication of more than 20 papers per year. The recent topics that the staff of LLTMS have deeply contributed to are:

**Discovery of superconductivity in KTaO3 by electrostatic carrier doping [1]**

By employing an electric double layer transistor configuration for electrostatic carrier doping, superconductivity is successfully induced at the surface of KTaO3, in which superconductivity has
never before been observed (Fig.1).

**Preparation of n-type metallic YBa$_2$Cu$_3$O$_y$ using an electrochemical technique [2]**

Realizing an ambipolar cuprate superconductor is an important issue. Using an electrochemical technique, we succeeded in converting the sign of conduction carriers in YBa$_2$Cu$_3$O$_y$, which is known to be a typical p-type cuprate superconductor with $T_c = 90$ K, from holes to electrons (Fig. 2), to the extent that the new n-type YBa$_2$Cu$_3$O$_y$ shows a metallic transport property.

**Observation of field-induced anomalous magnetic states in a spin-gap material YbAl$_3$C$_3$ [3]**

YbAl$_3$C$_3$ is known to be a rare 4$f$-electron compound that shows a spin-gap state, originating from dimerization of Yb$^{3+}$ pairs. In the detailed measurement of magnetization curves below 1 K, the authors found that the 4$f$-electron dimer state in YbAl$_3$C$_3$ transfers to an unusual field-induced state (Fig. 3), which reveals different properties from that reported in d-electron dimer systems, through a crossover transition.

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**Fig. 1 Superconducting transition observed in the temperature dependence of resistance in KTaO$_3$ with an electric double layer transistor (EDLT) configuration at a gate voltage $V_G = 5$ V. The inset shows the concept of EDLT.**

**Fig. 2 Evolution of electrical resistivity with hole reduction and electron doping by an electrochemical reaction in YBa$_2$Cu$_3$O$_y$ films. The horizontal axis is the carrier number per CuO$_2$ plane.**

**Fig. 3 Field dependence of magnetization of YbAl$_3$C$_3$ in units of $\mu_B$ per Yb atom. Two kinds of field-induced transition from the spin dimer state at low fields are observed around $H = 4.5$ T and 6.6 T at $T = 0.47$ K.**

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**References**


Tsutomu Nojima (Laboratory of Low Temperature Materials Science)
E-mail: nojima@imr.tohoku.ac.jp
URL: http://ltsd.imr.tohoku.ac.jp/
Research Facility for Studying Physical and Chemical Properties of Radioactive and Nuclear Materials

Laboratory of alpha-Ray Emitters

More than 170 species of radioisotopes and nuclear materials can be used in the Laboratory of Alpha-Ray Emitters. This facility is one of the important centers in Japan for studying the physical and chemical properties of radioactive materials, especially for alpha-ray emitters such as actinide elements, as well as fission and fusion reactor materials. The facility has been used by many researchers from all over the country for preparation of samples and for chemical and physical measurements of a variety of radioactive materials.

In March 1960, a radioisotope (RI) handling facility was officially approved by the Science and Technology Agency (currently, Ministry of Education, Culture, Sports, Science, and Technology) as ‘SHIDAI 337’ and installed in the basement of Build. #1, IMR. The facility was relocated to the present building in 1978. The Laboratory of Alpha-Ray Emitters is currently approved for the use of more than 170 species of radioisotopes, including alpha-ray emitters such as actinide elements and nuclear materials (Fig. 1 shows the uranium metal stored in the facility). The number of such facilities is quite limited in Japan; the others are JAEA (Japan Atomic Energy Agency) and the International Research Center for Nuclear Materials Science (Oarai) of IMR. The Laboratory of Alpha-Ray Emitters has, therefore, an important role in the study of actinide elements in Japan, and this activity and capacity extends international research collaborations.

The facility is composed of two areas: the radiation-controlled area and the non-controlled area. The controlled area consists of three rooms for chemical experiments, three rooms for physical experiments, one room for radiation measurements, one contamination test room, and rooms for storage of RI and nuclear materials. Each chemical room has local exhaust ventilation systems for the treatment of various chemicals.

The radiation-detection equipment that can be used in this laboratory are two gamma-ray spectrometers (high-purity Ge semiconductor detector), a liquid scintillation counter, and an alpha-ray spectrometer (silicon detector). A mono-arc furnace, tetra-arc furnace, electrical discharge machine, X-ray diffractometer, and IP reader are used for the preparation and identification of nuclear material alloys, such as UCoGe. Two inert-gas glove boxes, a CHNS element analyzer, an ICP emission spectrophotometer, an FT-IR, an UV–visible spectrophotometer, a powder X-ray diffractometer, and an X-ray single crystal structure analysis system are installed in the laboratory. A transmission electron microscope has been used to study the irradiation effects of nuclear materials. In recent years, a physical property measurement system (PPMS) was installed and used for the physical study of actinide compounds (Fig. 2).

In recent years, this laboratory has produced several outstanding achievements. The noted work introduced here includes (i) a study on the physical properties of uranium metallic compounds based on preparation of very fine single-crystals, and (ii)
the synthetic study of molecular-based magnets including f inner-transition elements, i.e., actinides and lanthanides.

The interplay of ferromagnetism and superconductivity has been a central topic of condensed matter physics. Recently, we presented the first example in which superconductivity is induced by ferromagnetic spin fluctuations in the ferromagnet UCoGe [1-2], and we further suggested the possibility that a spontaneous vortex state emerges as a compromise of the two competitive orders; a vortex induced by spontaneous magnetization can exist even at zero external magnetic field (right panel of Fig. 3), in contrast to a regular type-II superconductor (left panel of Fig. 3).

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Fig. 3 Schematic illustration of superconducting phase diagrams.
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Lanthanide and actinide complexes were widely investigated as candidates for quantum nano magnets, such as a single-molecule magnet (SMM) or a single-chain magnet (SCM), in which each molecule shows superparamagnetic behavior below $T_c$. We have synthesized the first example of a structurally designed Er(III) SMM (Fig. 4 and Fig. 5) [3].

The fission and fusion reactor materials intensively investigated are (1) Zr–Nb binary alloy development for advanced nuclear fuel rods, (2) irradiation behavior of ferritic–martensitic steels for fusion first walls, (3) vanadium-based alloy development for fusion (collaborating with NIFS), and (4) microstructural evolution and mechanical property change in steels irradiated with high-energy ions, performed at the Cyclotron Radioisotope Center (collaborating with School of Engineering).

References


Kenji Shirasaki (Laboratory of alpha-Ray Emitter)
E-mail: kshira@imr.tohoku.ac.jp
Determination of Released Trace Elements in Static Immersion Test for Characterization of Metallic Biomaterials

Analytical Research Core for Advanced Materials

Our research group conducts elemental analysis of various materials to determine major, minor, and trace levels of metallic and non-metallic elements. This report introduces the analysis of metallic biomaterials, one of our recent activities. The amounts of metallic ions released from the materials using static immersion into simulated body fluids (SBF) were determined. Sensitive and accurate determination of trace metallic elements in SBFs could be performed by inductively coupled plasma optical emission spectrometry (ICP-OES). By using a H₂SO₄-fume pre-treatment method, it was possible to determine concentrations of elements in SBFs on a ppb order.

Metallic biomaterials have been widely employed by orthopedists or dentists in clinical practice. Each of the metallic biomaterials has been applied to appropriate parts of the human body. However, when the metallic biomaterials are implanted for a long time, metallic elements may be dissolved into the surrounding tissues. The resulting toxic influence of metals released from metallic implants has been demonstrated [1, 2]. It has been recently reported that not only allergies but also several medical disorders have been caused by these metallic elements. Allergy responses are especially sensitive to trace amounts of the metallic elements. Accordingly, metallic biomaterials that have high corrosion resistance (high safety for the human body and low metal release) are required; therefore, the evaluation of corrosion resistance is important for their development.

The corrosion resistance of metallic biomaterials is evaluated by using static immersion into a simulated body fluid (SBF) and then assessing the metallic ions in the SBFs. Therefore, trace metallic elements in the SBFs should be noted when investigating the properties of the metallic biomaterials. Moreover, in order to evaluate the trace elements released by the static immersion test, it is necessary to determine μg L⁻¹ (ppb) levels of trace metallic elements in the SBFs. The concentrations of various elements in SBF solution samples have been determined by inductively coupled plasma optical emission spectrometry (ICP-OES, Fig. 1), inductively coupled plasma mass spectrometry (ICP-MS, Fig. 2) and atomic absorption spectrometry (AAS, Fig. 3) [3, 4].

ICP-OES has analytical advantages, such as the simultaneous determination of elements with a wide dynamic range. However, the operating conditions of the ICP are influenced by the matrix elements included in a sample solution [5, 6] or the viscosity of the solution. SBFs include a large amount of alkali metal salts (NaCl, KCl, etc.) and/or organic compounds (glucose, amino acids, etc.) and have a high viscosity. This could alter the signal intensities of the analytes, and thus it is difficult to obtain precise and accurate results in the analysis of elements in the SBFs. Accordingly, it is vital to select an optimal sample pre-treatment method before analysis of the elements using ICP-OES.

In order to develop a simple and easy procedure for high-sensitivity, precise, and accurate
determinations of trace metallic elements in the SBFs by ICP-OES, we investigated a H$_2$SO$_4$-fume pre-treatment method [7 - 9]. By using this method, the viscosity of a sample solution can be decreased and the solution can be stabilized in acidic condition. Moreover, the organic compounds contained in the SBFs can be removed. Thus, it is possible to determine the concentrations of elements in the SBFs to a ppb level.

SBFs include high concentrations of salts, such as NaCl, KCl, and several alkali or alkaline earth metals. The authors investigated the influences of these matrix elements on measurements by ICP-OES. Solutions containing analytes (2 mg L$^{-1}$) and NaCl (8 g L$^{-1}$) were prepared, and the emission intensities of the analyte elements were measured by ICP-OES. Their intensities and intensity ratios (elements/Y) are shown in Fig. 4. Both the intensities and the intensity ratios are normalized to unity when containing no NaCl. When containing NaCl, the intensities for Al and Pd were more than 1, but the intensities for the other elements investigated were less than 1. The intensity ratios for Au, Ca, Cr, Ti, V, and Zn were nearly 1. In this case, an internal standard correction method by using Y can be applied for these elements; however, it is not effective for the other elements such as Al and Pd. Na has a serious influence on the measurement of such elements by ICP-OES, and therefore, it is necessary to prepare matrix-matched standard solutions for calibration to ensure accurate determination.

We determined the elements released from SUS 316 or Ti–6Al–4V alloys by static immersion tests. These materials were respectively immersed into lactic acid in a PFA bottle at 310 K for 20 days in ambient atmosphere. After removing the materials, the elements released into the lactic acid were determined by ICP-OES with the H$_2$SO$_4$-fume treatment. It was possible to determine concentrations of the released elements at a ppb level by using the H$_2$SO$_4$-fume treatment method.

References

Tetsuya Ashino (Analytical Research Core for Advanced Materials)
E-mail: ayustet@imr.tohoku.ac.jp
URL: http://bunseki-core.imr.tohoku.ac.jp/