Research Centers

IMR KINKEN Research Highlights 2014

Novel Device for Irradiation-Related Materials Science and a Mysterious Phase Transition in the Actinide compound URu₂Si₂ International Research Center for Nuclear Materials Science

A novel irradiation-rig was developed by a collaboration between IMR and SCK•CEN, the Belgian nuclear research center, that provides five different neutron doses at the same neutron-flux and irradiation temperature. In order to advance Actinide science, ⁹⁹Ru nuclear magnetic resonance measurements were performed on tetragonal URu₂Si₂ for the first time, and the nature of its phase transition, which has been a long-standing mystery in heavy-fermion physics, was revealed.

Neutron irradiation using materials testing reactors is commonly performed to study the effects of radiation on materials. To obtain a more precise understanding of irradiation-dose dependencies, identical irradiation conditions, i.e., irradiation rate (neutron flux), neutron spectrum, and irradiation temperature, are necessary across different doses. We have developed a novel irradiation rig using the BR2 reactor at SCK•CEN, Belgium's nuclear research center, in a collaboration between IMR and SCK•CEN.

The materials-irradiation research program of the Oarai Center was conducted at SCK•CEN's BR2 reactor after the suspension of JMTR in 2006. A new irradiation rig system, the "Lifting Basket in the Experimental Rig for the BR2 Thimble tube sYstem, or "LIBERTY", is being developed under the framework of the collaboration between IMR and SCK•CEN. LIBERTY consists of one shroud tube containing up to five needles in which samples, thermo-couples, and an electrical heater are placed. Each needle will be lifted from the bottom to the top position of the shroud separately when the required dose for that needle is achieved.

LIBERTY has the following advantages: 1) Each needle can be independently lifted up above the reactor core level when the specified fluence is reached, while the other needles remain in neutron flux. 2) Each needle can be separately instrumented; electrical heating wires and thermo-couples can be placed onto the needles to control the specimen temperature during irradiation. 3) Relatively large specimens, such as compact tensile specimens, can be tested. LIBERTY is now installed in BR2, and the irradiation will be performed in FY2014 for collaborative experiments with SCK•CEN researchers.

The other achievement that the Oarai Center wishes to highlight is in actinide studies. The nature of the second-order phase transition at $T_0 = 17.5$ K, the so-called "hidden order (HO)," of the tetragonal compound URu₂Si₂ has been a long-standing



Fig. 1 Crystal structure of URu₂Si₂ and the temperature dependence of both the Knight shift and η obtained from the ⁹⁹Ru-NMR spectra [1].

mystery in heavy-fermion physics. Despite intensive research efforts, the order parameter, i.e., the broken symmetry of this low-temperature phase, has not been identified for more than 25 years. We performed ⁹⁹Ru magnetic resonance nuclear (NMR) measurements on this compound for the first time to test if the local rotational symmetry at the Ru(4d) site is broken or not. A high-quality (low residual stress) prepared single-crystalline sample was in collaboration with Hokkaido University, and the NMR measurements were performed at the University of Hyogo. Careful analyses of the asymmetry parameter \mathbf{n} of the electric field gradient at the Ru(4d) site in the ⁹⁹Ru NMR spectra show that η remains zero to within an accuracy of ~0.001, providing a new useful piece of information that the symmetry change of HO is extremely small, even if it exists.

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Recent Progress in the Development of Advanced Materials

Cooperative Research and Development Center for Advanced Materials

he center continues to investigate and develop new materials with important functional properties that have important roles in key thematic areas such as energy, transportation, housing, and healthcare. Here, we highlight our recent research activities in the study of nanoporous materials for effective gas storage/separation applications.

Innovative and cost-effective gas storage/ separation technologies should address the following important concerns: capture, separation, transport, and long-term storage. Metal-organic framework (MOF) materials contain two main components: metal ion connectors and organic linkers (see Fig.1), and they have high porosity and highly moldable framework structure. The versatility of MOFs permits the rational design and assembly of materials having novel topologies and exceptional storage and separation properties. Due to the regularity of MOF structures, it is easy to both build periodic structural models and to perform simulations that are very helpful finding the search for new materials with the desired characteristics.

In the Development Research Division of CRDCAM, we estimate the important properties of various MOF materials using highly accurate methods in order to accelerate the realization of novel materials, hand-in-hand with experiments. In collaboration with experimentalists, we determined the enhanced sorption of acetylene on Cu₂(pzdc)₂(pyz) using extensive first-principles calculations; the enhanced sorption is attributable to the double hydrogen bond support between the acidic acetylene proton and its acceptor basic site on the channel surface [1].

The sorption of different guest molecules (both R and S isomers) into the pores of Zn₂(bdc)(l-lac)(dmf) was also modeled. The results revealed the process



Fig. 1 Schematic representation of an MOF framework.



Fig. 2 Charge density distribution for a CO-coordinated MOF with (a) CO and (b) N₂ in small channels. Red represents an excess and blue a depletion of electrons.

of interaction between the porous coordination polymer host and the encapsulated molecules that provides a qualitative explanation for the observed sorption enantioselectivity [2].

Recently, a new soft, nanoporous MOF material with accessible Cu²⁺ metal sites that selectively adsorbs CO via adaptable pores has been studied using first-principles calculations in collaboration with experimentalists. The calculations showed a remarkable distinction in the adsorption properties of the MOF with respect to CO and N₂ separation [3].

It was found that the excess charge localized on the adsorbed CO molecules, and electron depletion observed in the MOF framework, indicated favorable attraction conditions between CO and the host structure (Fig. 2 (a)). the electron depletion localized on both N₂- and CO-coordinated MOFs indicated repulsion between N₂ and the host framework. This is in agreement with energy calculations that showed a difference in absorption energies between these two cases.

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Development of the World-Record-Holding 20 T cryogenfree superconducting magnet

High Field Laboratory for Superconducting Materials

A 20 T cryogen-free superconducting magnet (20T-CSM) was developed by upgrading the existing 18T-CSM. The 20T-CSM broke the world record for the highest magnetic field produced by a CSM. The innermost coil was made of high-strength and high-performance Bi₂Sr₂Ca₂Cu₃O_y (Bi₂223) high-temperature superconducting tapes. The increase in the current density resulting from the improvement in both the in-field critical current and the mechanical strength of the Bi₂223 tapes is the key technology that made the 20T-CSM possible.

Since the first practical cryogen-free superconducting magnet (CSM) was developed at the HFLSM, IMR, Tohoku University in 1992, magnetic fields produced by CSMs have been becoming stronger steadily, as shown in the top image of Fig. 1. Most of these advanced devices were created at the HFLSM.

The 18T-CSM is routinely utilized for high-field research such as a magnetization measurements and in-field material processing at the HFLSM. It consists of an inner Bi2Sr2Ca2Cu3Oy (Bi2223) coil, a Nb3Sn middle coil, and a NbTi outer coil. To make the 20T-CSM, the middle Nb₃Sn coil and the Bi2223 inner coil were replaced. A bronze-route Nb3Sn coil with a low ac-loss was adopted to reduce hysteresis loss during field ramping. A Bi2223 tape with a Cu alloy reinforcement was utilized for the new inner coil. The new Bi2223 tapes were produced by Sumitomo Electric Industries, Ltd. using an advanced controlled over-pressure (CTOP) process. Because of its high- J_c and high mechanical strength, the magnetic field contribution of the Bi2223 coil was improved from 2.5 T to 4.5 T under a background field of 15.5 T by the low-temperature superconducting outsert (Nb₃Sn and NbTi) coils. After the upgrade of the 18T-CSM, the HFLSM successfully reached 20 T in a 52 mm roomtemperature bore, which is a world record for a cryogen-free superconducting magnet.

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Fig. 1 (top) Progress in cryogen-free superconducting magnets by year. Closed circles were achieved by HFLSM-IMR. (bottom) Photo of the 20 T cryogen-free superconducting magnet. The insets show the cross-sections of the superconducting wires and tapes.

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Scientific Breakthrough toward the Creation of New Industrial Materials Kansai Center for Industrial Materials Research

Industrial Materials Research (IMR) has launched Kansai Center, a comprehensive Institute-wide effort that pairs IMR's research groups across the innovation spectrum to solve today's industrial challenges and address tomorrow's global energy problems. The center was established in April 2011 based on an agreement between IMR, Osaka Prefecture Government, and enterprises in the Kansai area sponsored by government, taking over the Osaka Center.

The Kansai Center was established in Kansai as a special unit within the Institute for Materials Research (IMR) at 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 took over the responsibilities of the Osaka Center, which carried out its mission from 2006 to 2011. The mission of the Osaka Center was to support small and mediumsize enterprises in the Osaka area, and the area covered by the Kansai Center expanded from the original Osaka area to encompass the entire Kansai area. The Center has three missions: First, to solve technical problems that industries have struggled to resolve. Solving such problems is occasionally through accomplished collaboration across universities, research institutions, fields, and institutional boundaries. Some of these activities developed in collaborative partnerships with industry. Second, the Center introduces academic output to industries, with the aim of applying such output to society. Currently, about 40 enterprises have contracted to engage in joint research on projects of mutual interest, and the Center has vibrant patenting/licensing activities, including 11 new invention disclosures. Third, the Center helps educate next-generation materials scientists and researchers in universities and enterprises. To this end, we have organized a bimonthly forum named "Monodzukuri Kisokoza," which focuses on special topics regarding materials and processing.

Projects are conducted through broad collaborations between the government, universities, research institutions, and other organizations. Among them, the Development Center for New Metallic Material in Osaka Prefecture University and the Research Center for Nano-Micro Structure Science and Engineering at the University of Hyogo are close partners in collaborative projects. The Center's staff participate in educating students at both the universities. We have seven groups at the Center:



Fig. 1 Fraction of inquiries (2010–2013) from industries at the Creation Core Higashi-Osaka. The area in red corresponds to the Kansai 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 four venues to cover the Kansai area: the Osaka office at Osaka Prefecture University, the Hyogo office at University of Hyogo, the Sendai office in IMR, and Monodzukuri Business Information Center Osaka (MOBIO) in the Creation Core Higashi-Osaka, a governmental body where sixteen local universities and one college are located to facilitate collaboration with various industries.

Technical inquiries from industry are handled solely by the staff, which helps reduce investigation and problem-solving times. Figure 1 illustrates the number of inquiries from industries to the Creation Core Higashi-Osaka. It is apparent that Kansai Center occupies the majority portion among the universities, and the number of the inquiries is about 500. About 30% of all inquiries are from enterprises located in areas other than Kansai.

The Kansai Center will continue to innovate in the metallic materials industry through collaborations between the related organizations and promote materials science research.

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New Sprouts in Computational Materials Science

Center for Computational Materials Science

The Center for Computational Materials Science (CCMS) at IMR supports various computational studies on materials science by providing users with an efficiently tuned super computer, the SR-16000, and a number of powerful software. The total number of users in FY2013 reached nearly 150. Introduced here are two examples of recent activities in computational materials science sprouting from CCMS. "First-principles calculations of a ternary phase diagram at 0 K" was performed by N. Drebov et al. [1], and "Dislocation behavior clarified by discrete dislocation dynamics simulation combined with phase-field method" was a unique theoretical work by a graduate student, Mr. Y. Yamada, at IMR.

First-principles calculations of a ternary phase diagram at 0 K*

In a computational search of ferromagnetic phases and their magnetic properties like the Curie temperature, thermodynamic stability is necessary to establish their existence. The primary indication of stability is given by the formation energy relative to that of the pure elements, but competing phases are typically neglected there. Therefore, we calculated ternary phase diagrams to find new ferromagnetic phases.

Figure 1 [1] shows the result of the calculation of the Ce-Fe-Si system, including the hard magnetic candidate CeFe10Si2. In the framework of the present approximated total energies, the proposed phase is part of the convex hull of the ternary system and, in this way, is also stable.

Dislocation behavior clarified by Discrete Dislocation Dynamics simulation combined with Phase Field Method**

In order to gain insight into the plastic deformation mechanisms in an alloy, it is necessary to understand dislocation behavior. However, this is a difficult task in practical alloys due to complex interactions among dislocations. To resolve this problem, we performed discrete dislocation dynamics simulations (DDDs) combined with the phase-field method (PFM), whose basic procedure was proposed by Rodney et al. [2]. First, we calculate the microstructure associated with the orderdisorder transition by PFM. Then, we introduce dislocation loops and simulate dislocation motion using DDD. Figure 2 shows the typical behavior of dislocation loops. The dislocations are pinned by the ordered phase, and some pairs of dislocations are connected by anti-phase boundaries in the ordered phase. Additionally, multiplication of dislocations by cross slip is observed.



Fig. 1 Ce-Fe-Si phase diagram at 0 K [1].



Fig. 2 Behavior of dislocation loops in the microstructure predicted using the phase-field method.

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Experimental Demonstration of a Persistent Current

International Collaboration Center (ICC-IMR)

In this research, we will demonstrate that combining an epitaxial FePt nanopillar with perpendicular magnetic anisotropy and a nonmagnetic nanoring is an alternative method of generating a spin-polarized persistent current in a non-magnet. Even though such a spin-polarized persistent current was proposed theoretically almost 20 years ago [1], there has been no experimental demonstration to date. This device would open up new research horizons as a spin source for quantum computation.

The quantum phases of charged particles in mesoscopic structures have been investigated, and interference and oscillatory behavior was observed to be induced by application of an external field [2]. For instance, electrons traveling along semiconductor or normal metal rings threaded by a magnetic flux acquire a quantum dynamical phase that produces interference phenomena such as the Aharonov-Bohm (AB) and Altshuler-Aronov-Spivak (AAS) effects. In addition, when the spin of the electron rotates during its orbital motion along the ring-shaped path, the electron acquires an additional phase element known as the geometrical or Berry phase.

A new nanofabrication method for producing a quantum device on a MgO(001) substrate consisting of a 300-nm-inner-diameter nonmagnetic nanoring with a 70-nm-diameter FePt nanopillar inside by using electron-beam lithography and Ar-ion milling has been successfully developed. The nanoring is 100 nm wide and contains 15 nm Cr and 5 nm Au layers to improve the adhesion onto the MgO substrate. As shown in Fig. 1, the center nanopillar is designed to provide a nonuniform magnetic field in the nanoring in its remanent state after perpendicular saturation. Such a nonuniform field is theoretically



Fig. 1 SEM image of the examined device.



Fig. 2 Magnetoresistance measured at 25 mK with increasing (blue) and decreasing (red) applied magnetic field.

expected to induce a persistent spin current in the nanoring [1]. Four contacts were fabricated near the nanoring for measurement of the induced current.

Figure 2 shows magnetoresistance curves measured at 25 mK. A clear oscillation is observed in the range of 80–100 mT, which is larger than that estimated from the nanoring diameter (20–60 mT). This may be caused by the presence of multiple electron paths or universal conductance fluctuation within the 100-nm-wide nanoring.

Around zero field, a symmetric increase in the magnetoresistance with increasing field was observed, which proves the existence of weak anti-localization.

In summary, we have successfully fabricated a quantum device consisting of a ferromagnetic nanopillar enclosed by a nonmagnetic nanoring. As the first step toward the demonstration of a spin-polarized persistent current, we observed AB oscillation and weak anti-localization in the nanoring below 5 K.

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Neutrons for Advanced Materials Science

Center of Neutron Science for Advanced Materials

he Center of Neutron Science for Advanced Materials is a unique neutron facility that performs novel materials science research at IMR. This center operates two neutron spectrometers for materials science in a reactor facility, and is also constructing a new neutron spectrometer in J-PARC/MLF, which will provide the world's brightest neutron beam.

The Center of Neutron Science for Advanced Materials was established in 2010 to perform novel materials science using neutrons. In particular, this center is at the core of a project on building a new neutron spectrometer in Japan, the Proton Accelerator Research Complex (J-PARC), in a collaboration with Tohoku University and the High Energy Accelerator Research Organization (KEK). This neutron spectrometer, named POLANO (Fig. 1), aims to observe spin dynamics using the polarization analysis technique, which is very sensitive to pure magnetic correlations and latticespin couplings [1,2]. POLANO will be indispensable in the investigation of current issues in materials science, such as multiferroic materials, spin frustration systems, and multipolar orderings in rare earth compounds.

The construction of POLANO began in FY2013. The focusing guide tubes for neutron transport, the radiation shields, and some important neutron devices such as 360 neutron detectors and 3 neutron choppers have already been installed. By the end of FY2014, the POLANO vacuum chamber, with a radius of 2 m, and the polarization analyzer for scattered beams are going to be installed. The first polarized neutron beam is expected in early FY2015.

To obtain an accurate polarization analysis, precise magnetic field designs along the beam pass from the polarizer to analyzer (~2m) are indispensable. Thus, we have begun the quantitative estimation of the required magnetic fields using finite element methods and the super computer at the Center for



Fig. 1 A conceptual drawing of POLANO [1,2].





Computational Materials Science at IMR. Figure 2 shows an optimized result of the magnetic field distribution around the sample position in the POLANO. We also developed a method for the estimation of changes in neutron polarization along the beam pass in the calculated magnetic fields based on a quantum mechanical treatment. From these calculations, we succeeded in determining the alignments of the spin devices which provide a neutron polarization of over 95% through the beam pass.

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Cores with High Saturation Magnetic Flux Density and Low Core Loss

Research and Development Center for Ultra High Efficiency Nano-crystalline Soft Magnetic Material

Nanocrystalline Fe-rich Fe84.3Si4B8P3Cu0.7 cores with almost the same saturation magnetic flux density, as well as lower core loss, as Si-steels were formed by consolidating powders using spark plasma sintering. This success in fabricating a bulk core from powders is promising for the manufacture of electronics/electrical devices with low energy-consumption.

Modern society is faced with many issues, such as the development of environmental safeguards, energy conservation, and the prevention of global warming and climate change. Nowadays, more and more attention has been given to curbing CO₂ emissions by saving energy. Of all the functional advanced materials, nanocrystalline soft magnetic alloys, such as FINEMET, NANOPERM, and HITPERM have been considered as appropriate candidate materials for realizing energy savings. However, the simultaneous achievement of high saturation magnetic flux density (B_s) and low core loss (W) has not been obtained to date in existing soft magnetic alloys.

Given these circumstances, members of this center have found a number of new nanocrystalline soft magnetic alloys called "NANOMET" [1]. These are Fe-rich alloys with compositions Fe83.3-86Si1-4B8-10P2-4Cu0.7-1 and are prepared by crystallizing an unusual as-guenched nanohetero-amorphous phase that includes a large amount of extremely small bcc Fe (less than 2-3 nm in size) grains. The characteristics of these NANOMET materials include B values that reach 1.7-1.8T (at 800 A/m), which is almost comparable to commercial oriented silicon steel. Additionally, NANOMET materials exhibit extremely low W, typically 1/2-1/3 times that of the highestgrade oriented silicon steel and about one order of magnitude smaller than those of non-oriented silicon steels at a maximum flux density of 1.7 T. Thus, NANOMET has the potential to be used in applications requiring higher B and lower W than ever.

This report includes recent research on applications for NANOMET in products. Our group has succeeded in fabricating bulk specimens of nanocrystalline materials of the Fe-rich alloy, Fe84.3Si4B8P3Cuo.7, 12 mm in diameter and 2–5 mm in thickness [2]. The bulk cores were made from ribbons that were first crushed into powders and subsequently compacted using spark plasma sintering (SPS). The bulk cores possessed relative densities up to ~88.3%. Sintered cores are mainly composed of grains of α -Fe with sizes 10–27 nm precipitated in the amorphous matrix. Similar to wound cores, sintered-powder cores have high M_{s} , but their total core loss (W_1) is significant. This disadvantageous aspect was eliminated by the





addition of SiO₂ (<2 mass%) powders, which significantly reduce W_t at frequencies f > 1 kHz.

When compared with values reported in the literature, the present core samples exhibit much lower W_t than nonoriented electrical steel sheets and other commercial powder cores (f > 1 kHz), as shown in Fig. 1. We believe our core materials can produce better performance if used in power electronic/ electrical devices operating over a frequency range of 1–100 kHz.

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Ideal Two-Dimensional Superconductor Induced by Electric Field Laboratory of Low-Temperature Materials Science

Recently, we succeeded in converting an insulating surface of SrTiO₃ to a superconducting surface using only the electric field effect and employing an electric double-transistor configuration. Here, we shed light on the basic properties of the electric field induced superconductor, namely two dimensionality and effective thickness.

Two-dimensional (2D) electron systems that are induced by the electric field effect are attractive areas for the exploration of new physical properties of condensed matter because of the ability to control conduction carriers without changing disorder, and because a unique electric state is expected in a static electric field. In particular, the realization of electric field-induced superconductivity has been a long-standing challenge owing to the need for high carrier densities. Recently, it was reported that the superconductivity can be induced electrostatically on the insulating surface of SrTiO3 at 0.4 K (the same as the bulk critical temperature) by employing an electric double-layer transistor (EDLT) configuration (Fig. 1(a)) [1]. While EDLT techniques are currently applied to versatile materials, most basic properties of superconductivity in EDLT remain unclear. Here, we perform measurements of the effective superconducting thickness in SrTiO3-EDLT at various gate voltages $V_{\rm G}$ [2].

The dimensionality and effective size of the superconducting carriers (Cooper pairs) can be investigated using the anisotropic properties of the upper critical magnetic field Hc2. Figure 1(b) shows the typical dependence of H_{c2} on the magnetic field direction θ in SrTiO₃-EDLT for V_G = 3.5 V (an induced carrier density of 1.0×10^{14} cm⁻²), which was obtained using the measurements of the resistive transition as a function of the magnetic field H at 0.1 K. The $H_{c2}(\theta)$ curve with a sharp cusp rather than a round peak at $\theta = 90^{\circ}$ (for H parallel to the conducting plane) can be explained perfectly using the 2D Ginzburg-Landau model (black solid curve in Fig. 1(b)), indicating that an ideal 2D superconductor is induced using our device. The effective thickness deff, which was estimated from the temperature dependence of H_{c2} for both parallel and perpendicular conditions, is ~10 nm, which is much smaller than the coherence length (size of Cooper pair) of ~45 nm, and is consistent with the 2D nature of $H_{c2}(\boldsymbol{\theta})$.

Surprisingly, the measured d_{eff} at various values of V_{G} was found to be almost invariant, which



Fig. 1 (a) Top view of the planar-type electric double-layer transistor device used in this work. (b) Angular dependence of H_{c2} at 0.1 K for V_{G} = 3.5 V. The inset shows the magnification around θ = 90° (parallel to the surface).

contradicts the calculation results obtained using a triangular quantum-well model. This may be a unique nature of superconductivity in SrTiO₃–EDLT.

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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 most important centers on the planet for studying the physical and chemical properties of radioactive materials, such as actinide compounds. Many researchers visit this facility every year from several leading universities and institutes in the country to prepare a variety of materials and carry out their chemical and physical measurements.

The present building, built in 1978 as a successor to the former IMR facility in the basement of the old Building 1 since 1960, is approved for the use of more than 170 radionuclides and elements: alpha-ray emitting actinides, for example. This capability of this facility deserves attention because of the scarcity of similar facilities in Japan; the others include JAEA (Japan Atomic Energy Agency) and The International Research Center for Nuclear Materials Science (Oarai) of IMR. Therefore, this laboratory occupies an important position for the study of actinide elements, not only in Japan, but internationally.

This facility is composed of two areas; a radiation-controlled area and a non-controlled area. The controlled area consists of three chemistry laboratories, three physical laboratories, a room for radioactivity measurements, a contamination test room, and storage rooms for RIs and nuclear materials. Each chemistry lab is equipped with local exhaust ventilation systems, which enables the use of various kinds of chemicals. In the radioactivity measurement room, two gamma-ray spectrometers (with high-purity Ge semiconductor detectors), a liquid scintillation counter, and an alpha-ray spectrometer (with Si detector) were installed for radiation detection purposes.

Slow magnetic relaxation phenomena at a single-molecule level realized by highly anisotropic magnetic susceptibility tensors of one or more f-block element(s) are of great importance because of the potential applications of this class of material to leading-edge information technologies. Extensive studies on metal complexes with a 4f magnetic center have been conducted, whereas the effect of dipole-dipole interactions between the f-electronic systems on the magnetic relaxations remains unclear. We have developed a novel compound incorporating two highly anisotropic f-block ions, in which the magnetic centers are subject to well-

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Fig. 1 (a) Plots of $\chi M''$ against the applied ac magnetic frequency in the presence of an external magnetic field $H_{dc} = 1000$ Oe. (b) Calculated Zeeman diagram for the $J_z = \pm 6$ states of the dinuclear complex.

defined anti-prismatic ligand fields. As shown in Fig. 1 (a), the presence of the intramolecular f-f interaction promotes magnetic relaxations, provided that an external static magnetic field (H_{dc}) of 1000 Oe is applied. A theoretically simulated Zeeman diagram indicates that the appropriate H_{dc} induces admixing of the wave functions (the blue hatched region in Fig. 1 (b)), leading to enhanced magnetic relaxation via quantum tunneling pathways between two orthogonal wave functions.

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Analysis of Inorganic Materials using a Wavelength-Dispersive X-ray Fluorescence Spectrometer System

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X-ray fluorescence spectrometry helps ascertain the bulk elemental compositions of inorganic materials. These rapid and direct measurements give semi-quantitative information. Furthermore, wavelength-dispersive X-ray fluorescence systems and the availability of homogenous calibration specimens like borate glass beads provide accurate and precise results for both major and minor elements.

X-ray fluorescence (XRF) spectrometers are often applied to inorganic materials for nondestructive elemental analyses. Without any calibration standards, the instruments provide semiquantitative results automatically using the fundamental parameter calculations based on a fluorescent X-ray spectrum.

On the other hand, a wavelength-dispersive XRF spectrometer (Fig. 1), along with a specific sample preparation technique, is useful for determining major and minor components in inorganic materials. In general, quantitative XRF analyses require bulk solid specimens and several standards. Several types of specimens are commonly employed in XRF. For instance, a solid plate with a polished surface, loose powder that is packed in a vessel with a polymer film seal, a pellet or a briquette of pressed powder in a support ring, or a glass bead where the sample powder is fused and vitrified together with an alkali borate flux [1].

The application of XRF to solid inorganic materials has some advantages. Calibration standards of reference materials or pure chemicals are available for full quantification. Furthermore, the glass bead has a homogeneous body because of the melting preparation processes. Several Japanese Industrial Standards (JIS) involve wavelength-dispersive XRF with glass bead preparation, such as iron ore, Portland cement, lime, refractory products (e.g., argillaceous, alumina, magnesia, and zirconia), etc. The preparation of the glass bead requires pulverization of the inorganic sample, mixing the resulting powder with a flux, heating the mixture at around 1300 to 1500 K in a platinum-alloy crucible, homogenizing the melt, and vitrifying the mixture in a mold by air-cooling. The obtained glass beads provide fluorescent X-rays without severe matrix effects. Several materials are suitable for the XRF calibration, such as commercial reference materials and in-house standard samples. Additionally, analysts can produce synthesized standards from pure metals and reagents [2–4].



Fig. 1 Wavelength dispersive X-ray fluorescence spectrometer.

The Author adapted glass beads for the following XRF applications: 42 elements (Na, Mg, Al, Si, P, K, Ca, Sc, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Ga, As, Rb, Sr, Y, Zr, Nb, Sn, Cs, Ba, La, Ce, Pr, Nd, Sm, Gd, Dy, Er, Yb, Hf, Ta, W, Pb, Th, and U) by using 0.4–2.2 g of a mixture of silicate minerals and glasses (geochemical rocks) [3] and 10 elements (Na, Mg, Al, Si, P, K, Ca, Ti, Mn, and Fe) by using 11 mg of a sintere mixture of weathered silicates (ceramics) [4].

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