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Research Center
for Structural
Materials

Pushing the limits

Pushing the limits

Structural materials—which form the backbones of buildings, infrastructure and equipment—are increasingly important in ensuring that structures survive earthquakes and other disasters and in global efforts to achieve carbon neutrality.
These issues require structural materials to be fully functional in extremely severe environments.

For example, improving the fuel efficiency of aircraft and thermal power plants will require lighter weight materials with greater heat resistance.
Next-generation energy infrastructure will have to deal with embrittlement caused by low temperatures and hydrogen exposure.
Moreover, these new technologies will need to be sufficiently durable to help society become more sustainable over the long term.

The NIMS Research Center for Structural Materials carries out basic research with the goals of contributing to the industrial sector,
meeting social demands and protecting people's lives.
Its activities include developing structural materials capable of effectively functioning in extreme environments and advanced evaluation and analytical techniques.

Data：
Organizational structure of the Research Center for Structural Materials (RCSM)

Managing Director: Takahito Ohmura

Number of permanent researchers: 59

Materials Manufacturing Field

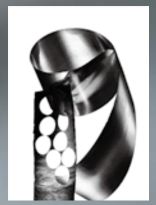
- Polymer Matrix Composites Group
- Ceramic Matrix Composites Group
- Fatigue Resistant Alloy Design Group
- Light-weight Metallic Materials Group
- High Temperature Materials Group
- Smart Interface Group
- Additive Manufacturing Group
- Anisotropic Materials Group
- Corrosion and Protection Group
- Thermomechanical Processing Group

Materials Evaluation Field

- Creep Property Group
- Fatigue Property Group
- Cryogenic Fatigue Group
- Steel Research Group
- Corrosion Research Group
- Welding and Joining Technology Group
- Mechanical Properties Group
- Microstructure Analysis Group
- Computational Structural Materials Group

Cover Story

Magnesium foil



Magnesium (Mg) is intrinsically brittle and occurs fracture easily. Hidetoshi Somekawa (Group Leader, Light-weight Metallic Materials Group) focuses on grain boundary sliding—a phenomenon in which adjacent grains slide against each other—as part of his in-depth research on Mg crystalline structures. Through these efforts, Somekawa has succeeded in greatly improving Mg's workability and capacity to be processed into foils. The round hole in the foil in the cover photo was cut by Toshihiko Mandai (Senior Researcher, Research Center for Energy and Environmental Materials). He used the round piece of foil as a component in a prototype Mg battery cell. By using Mg foils as negative electrodes, Mandai achieved an Mg battery with a capacity approximately 20% greater than that of existing Mg batteries.

<Article in ChemistryViews referenced>
https://www.chemistryviews.org/ultrathin-magnesium-metal-anodes/

Metallic specimen fractured via slow strain rate tensile test—a test specially designed to study the impact of hydrogen exposure on metallic materials under cryogenic conditions (see p. 15 for details).

Key Projects

The Research Center for Structural Materials has been carrying out research and development in two project categories.

#1 Create structural materials for carbon neutral technologies capable of functioning properly under extreme conditions

Effective decarbonization efforts will require advanced structural materials that can perform under extreme conditions (e.g., embrittlement-causing cryogenic temperatures and hydrogen exposure) over the long term. NIMS' precision materials design includes meticulous control of a variety of parameters, including composition, phase distribution across microstructures, arrangement of defects and inhomogeneity (e.g., dislocations and segregations) and interfacial structures. NIMS develops production processes to achieve these precise controls and maximize the performance of materials.

Project leader

Makoto Watanabe

Designing advanced structural materials capable of functioning under extreme conditions

In 2008, NIMS developed a heat-resistant nickel (Ni)-based superalloy which has since been used in jet engines. This is a good example of NIMS' success in overcoming the extreme environments in which materials are used through precision design. Researchers working on this project will develop a next-generation Ni-based superalloy by reducing the amount of rare earth elements used to create the original alloy without compromising its ability to withstand temperatures as high as approximately 1,100°C . Another subproject is extending the service lives of seismic dampers for large buildings developed by NIMS by investigating metal fatigue mechanisms. In addition, the project team will work on improving the workability and other properties of lightweight metals, such as magnesium and aluminum, to satisfy various technologies' weight reduction requirements. Other subprojects focus on the development of nonmetallic materials that can be used in decarbonization efforts, including polymer matrix composites that decompose in response to external stimuli and ceramic matrix composites (CMCs) with enhanced heat resistance.

See Research 1 on **p. 6**
See Research 3 on **p. 10**

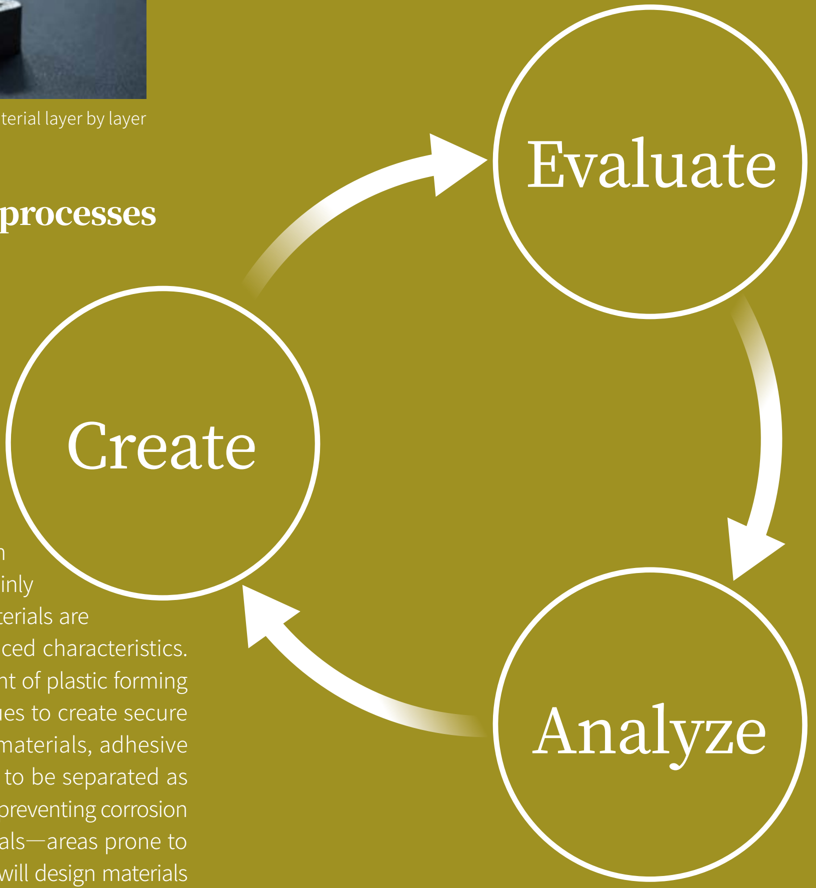
Aircraft engine turbine blade made of the heat-resistant nickel-based superalloy developed by NIMS（photo : Naohiro Tsukada)

Three-dimensional object created by depositing a material layer by layer using laser powder bed fusion technology

Refinement of **production processes** to maximize material performance

The microstructures and characteristics of metals are determined by their production processes, such as molten metal cooling time, metal processing techniques and heat treatment temperatures and duration. Precisely controlling production processes is therefore crucial in creating materials with desirable characteristics. This subproject mainly focuses on processes in which different materials are combined into multi-materials with enhanced characteristics. This ongoing effort involves the development of plastic forming processes and thermal treatment techniques to create secure interfacial structures between dissimilar materials, adhesive technologies that enable joined materials to be separated as needed and coating technologies capable of preventing corrosion at the interfaces between dissimilar materials—areas prone to developing corrosion. In addition, this team will design materials using metal additive manufacturing—a technology that has been attracting considerable interest in recent years. Data science and numerical analysis will be actively incorporated into these R&D activities. The team will also develop technologies able to continuously update and suggest improvements to materials production processes as well as predict resulting crystalline structures and material characteristics.

See Research 2 on **p. 8**



#2 Improve reliability of structural materials to make society more resilient

All materials are destined to eventually degrade. It is feasible, however, to extend the service lives of materials by better understanding their degradation and property appearance mechanisms. NIMS will advance its materials evaluation testing—which has been carried out continuously for decades—to enable it to generate data more relevant to extending the lives of existing structures and determining ways of safely introducing hydrogen energy and other new technologies. In addition, computational science and microstructural analysis will be actively used to elucidate these mechanisms.

Project leader

Hideki Katayama

Upgrading materials **evaluation testing** to generate reliable data and better understand materials' properties

"Creep", "fatigue" and "corrosion" are well-known as the typical factors in the degradation of metallic materials. NIMS has been carrying out research for many years to clarify these mechanisms while also evaluation testing materials systematically and collecting data on them. More recently, this project team has been developing creep and fatigue testing techniques for materials fabricated using metal additive manufacturing and creep testing techniques to assess the effect of fluctuating temperatures and stresses on materials used in thermal power plants to better meet the needs of the industrial sector. The team is also working to improve the accuracy of AI-based predictive techniques to determine the corrosion risk of target materials using weather data. In addition, the team is investigating degradation mechanisms at welds of metallic structures leading to structural fragility by in-situ observation using synchrotron radiation. The project will also work on establishing material evaluation methods to overcome the issues related to hydrogen energy utilization, such as hydrogen embrittlement, in which hydrogen penetrates into the metal and makes it brittle, and cryogenic fatigue degradation, in which metal fatigue becomes pronounced at the cryogenic temperatures required for the retention of liquid hydrogen.

Rusting iron

See Research 4 on **p. 11**

Understanding the **mechanisms** of degradation and property appearance using advanced analytical and predictive technologies

Understanding the mechanisms of degradation and property appearance of metallic materials requires an investigation of phenomena on a multi-scale and unraveling the correlation with the parameters obtained. The Research Center for Structural Materials is equipped with state-of-the-art analytical and observational instruments and computer simulation tools vital to tackling this and other subprojects. The research teams in this project will develop advanced analytical techniques (e.g., three-dimensional analysis using different types of electron microscopes) and advanced predictive techniques (e.g., simulation techniques capable of sequentially predicting the compositional structures of sample materials at scales ranging from nano to macro). In addition, the team will investigate ways to strengthen steel materials by studying the influence of lattice defects (e.g., grain boundary dislocations) on the mechanical properties of steel materials. For this purpose, the team will observe crystalline deformation processes in situ under a transmission electron microscope and conduct other studies.

See Research 5 on **p. 12**
See Research 6 on **p. 13**

Autoclave used for CFRP fabrication. To create a CFRP, a stack of prepreg sheets composed of carbon fibers impregnated with resin is first placed in a mold. The stack is then sealed by covering it with a film material and heated under high pressure to cure the resin. This fabrication method enables the production of high-quality CFRP with very few voids and other defects within it, making it suitable for use in aircraft components and for other purposes that require high reliability.

RESEARCH 1

Making strong, lightweight, environmentally friendly CFRPs

Kimiyoshi Naito

Leader,
Polymer Matrix Composites Group

Fiber reinforced plastics (FRPs) are strong, lightweight, heat-resistant materials. Expectations are growing for FRPs to be incorporated into aircraft to reduce weight. Kimiyoshi Naito is an expert in polymer matrix composites, including carbon fiber reinforced plastics (CFRPs) composed of resins and carbon fiber. He has been engaged in a challenging effort to develop recyclable CFRPs. We asked Naito about his strategies.

Increasing FRP waste due to non-recyclability

Polymer matrix composites consist of a polymer (e.g., a resin) and other materials. They can be stronger and lighter than metals. Fiber reinforced plastics (FRPs) are a well-known example. Plastics are inherently hard and brittle. However, incorporating fibers into plastics creates FRPs considerably stronger than plastics alone. Common types include glass fiber reinforced plastics (GFRPs) and carbon fiber reinforced plastics (CFRPs).

FRPs have significant advantages over metals. GFRPs are cheaper and as strong as metals. CFRPs are lighter than aluminum—one of the lightest metals—yet 10 times stronger than iron. Moreover, unlike metals, FRPs do not rust.

According to Naito, FRPs also have major disadvantages.

"Wind turbine power generation blades are made of GFRPs," Naito said. "When they crack and become unusable, they go to waste disposal facilities and can never be recycled. The number of these 'blade graveyards' is increasing. It's ironic that wind power generation—a technology that is supposed to be environmentally friendly—is polluting the environment. Although it's technically feasible to melt GFRPs' plastic components by burning them at high temperatures to extract and recycle the glass fibers, this is not economical as glass fibers are cheap. This is why these damaged blades are discarded. At the same time, recycling CFRPs is costly and high temperatures destroy their carbon fiber components. For this reason, most out-of-service CFRPs are disposed of in landfills. Growing public awareness of environmental conservation has been vigorously driving research efforts to find ways of recycling FRP components (i.e., fibers and resins)."

Naito has launched a project to develop environmentally friendly, recyclable CFRPs in line with NIMS' fifth medium-to-long-term (MTLT) plan, which went into effect in April 2023.

Strategies backed by experience

Naito envisions designing CFRPs whose resin and carbon fiber components can be separated using small amounts of energy.

"With regard to the raw plastics used as CFRP components, I'm considering naturally occurring resins that decompose in response to external stimuli (e.g., heat and electromagnetic radiation) or the activities of microorganisms," Naito said. "I'm also thinking of using naturally occurring carbon fiber to reduce the environmental impact. I'm particularly interested in vitrimers, a type of resin. When heated in a specific manner, vitrimers soften as their intermolecular crosslinks rearrange. In fact, I just began a research project in this area with the University of Tokyo Professor Kohzo Ito (a NIMS Fellow), an authority in vitrimer research. NIMS' Polymer Matrix Composites Group is responsible for combining fibers and resins into composites and evaluating them. Our group has ample expertise and experience developing composites, including materials design, production process development, joining of different types of materials, evaluation and analysis. Knowledge of all of these steps is vital to producing larger composite samples."

Naito has focused on composite materials research since joining NIMS in 2006.

During the implementation of NIMS' second MTLT plan (April 2006–March 2011), Naito created a CFRP composed of nanoparticles dispersed within a resin matrix that demonstrated high tensile and impact strength.

Naito succeeded during the third MTLT plan period (April 2011–March 2016) in developing a strong, lightweight composite composed of lightweight metals (i.e., aluminum and titanium alloys) and a high-strength FRP. During this process, he developed adhesives to join these component materials and optimize their shapes.

While the fourth MTLT plan was in effect (April 2016 March 2023), Naito developed adhesives vital to creating multi-materials.

Naito chooses from various FRP fabrication techniques depending on a material's ultimate purpose. He uses an autoclave (photo on p. 6) to create FRPs that need to meet high reliability requirements and uses cheaper production methods (e.g., an oven and press) when creating lower-grade FRPs.

Naito also carries out various mechanical evaluations of FRPs by performing impact, fatigue and creep testing and other tests under various environmental conditions (e.g., low and high temperatures). He procured most of the lab equipment needed to evaluate resins, composites and nanoscale interfacial structures himself, including a scanning electron microscope (SEM) and an atomic force microscope (AFM), both of which are equipped with load application devices. He also estimates FRP strength by analyzing experimental data using the finite element method (FEM), a numerical analysis technique.

Interfaces greatly influence materials' properties

Naito, who has studied FRPs and other polymer matrix composites from many different angles, was convinced that interfaces are the key to improving their physical properties.

"When metals and FRPs are joined together using adhesives, they form interfaces (see the illustration at left end in the figure)," Naito said. "Even within an FRP, there are interfaces between its component fiber bundles and the resin. Furthermore, the fiber bundles themselves contain interfaces between the individual fibers and the resin. Designing and controlling these interfaces is crucial in improving the performance of composites. Taking the polymer matrix composite I developed during the third MTLT plan period as an example (see the illustration at right end in the figure), the adhesion strength between the nanoparticles and the resin needs to be just right in order to optimize the composite's physical properties. If the adhesion is too strong, the fibers will break, while weak adhesion can result in a composite weaker than the resin alone. I'm finally in a position to propose optimum FRP design guidelines and evaluation/analytical techniques after spending years creating, evaluating and analyzing FRPs. My current goal is to advance research and development into the environmentally friendly, recyclable CFRPs aforementioned."

Lightweight FRPs vital to the hydrogen economy

Public interest is high in hydrogen energy. Liquid hydrogen is expected to be a promising clean energy source for aircraft.

Reducing the weight of aircraft fuel tanks is an important issue. Because hydrogen is a significant fuel candidate, using FRPs for this purpose has been a focus of active R&D. FRPs have advantages over metals as liquid hydrogen tank materials because they are more resistant to hydrogen embrittlement and have superior thermal insulation properties—important, given that hydrogen liquefies at the extremely low temperature of -253°C.

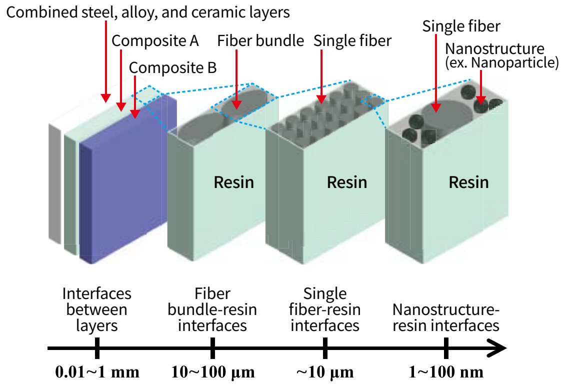
The physical properties of FRPs under cryogenic conditions are not well known. Naito therefore plans to evaluate these properties.

"NIMS has been making progress on the research and development of hydrogen liquefaction technologies," Naito said. "In addition, the Research Center for Structural Materials has been testing materials under cryogenic conditions. I plan to closely collaborate with these research teams to formulate FRP design and production process guidelines in harmony with efforts to promote hydrogen energy utilization."

Naito has been vigilant about meeting changing social demands.

CFRP sheets fabricated using an autoclave. By changing the prepreg sheet stacking schemes, carbon fibers within the sheets can be stacked in different orientations, allowing Naito to control the strength, thermal conductivity and other properties of the resulting CFRPs. For example, the CFRP sheet on the left has continuous fibers woven both lengthwise and crosswise, making the sheet resistant to tensile force applied in those directions but stretchable diagonally. The middle CFRP sheet contains short fibers oriented randomly, causing it to exhibit consistent physical properties in all directions. Finally, the CFRP sheet on the right contains continuous fibers running lengthwise, making it resistant to tensile force applied in that direction but susceptible to tensile force applied in a crosswise direction.

Figure. Interfaces in different polymer matrix composites



RESEARCH 2

Metal additive manufacturing: capable of fabricating reliable structural components

Masahiro Kusano
Senior Researcher,
Additive Manufacturing
Group

Metal additive manufacturing is an emerging material fabrication technology. Products fabricated using this technique have microstructures and physical properties different from those made using conventional methods. Understanding these differences is crucial to optimizing process conditions, which vary depending on a manufactured product's intended purpose. Masahiro Kusano has been working to identify optimum process conditions using simulations and machine learning.

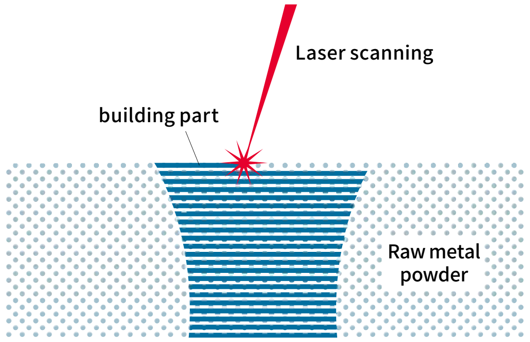
Issues unique to metal additive manufacturing

Metal additive manufacturing (AM) has very attractive characteristics, including the ability to flexibly design and fabricate parts with complex, unconventional geometries (e.g., consolidation of multiple conventional components into a single part). Currently, forging or casting followed by machining and welding is used to produce components for automotive engines, aircraft and so on. If metal AM can be used to fabricate such components, manufacturing yields and costs would be improved. The strengths of metal AM are particularly apparent when fabricating parts from difficult-to-process materials, such as nickel (Ni)-based alloys and titanium (Ti) alloys.

Laser powder bed fusion (L-PBF), a metal AM method, is a process in which a raw metal powder is spread over a platform and then irradiated and melted with a focused, high-powered laser (figure 1). An L-PBF machine repeats these operations as directed by CAD (computer-aided design) software until a near-net shape product is fabricated. Since metals solidify quickly and crystal grains grow epitaxially during the process, metal AM fabricated microstructures are significantly different from those achieved using forging and casting. As these microstructures determine the properties of a material, it is important to understand how they form during the L-PBF process.

Figure 1. Manufacturing an object using laser powder bed fusion (L-PBF)

An L-PBF machine alternately spreads metal powder and scans and melts it with a laser until a near-net shape product is fabricated as designed by CAD software. A focused laser beam (≲ 100 µm in diameter) allows products with great dimensional accuracy to be made.



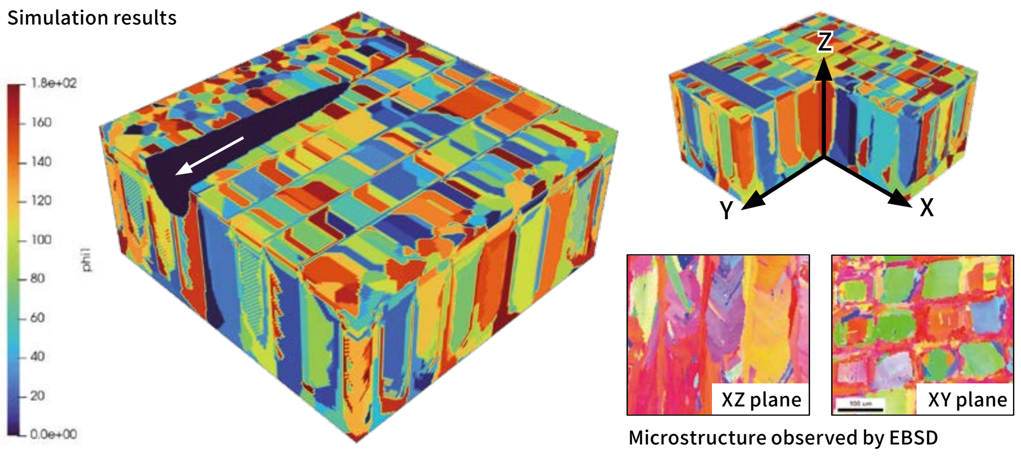


Figure 2. Simulated microstructure compared with experimental observation

The novel, newly developed program simulates three-dimensional grain growth during the solidification process after laser scanning. The left and upper right figures show simulated crystal orientation maps of a nickel-based alloy. The grains are directed along the building direction (Z-direction). Similar figures on the bottom right show the XZ and XY cross sections observed using electron backscatter diffraction (EBSD).

"L-PBF hasn't yet been widely adopted for manufacturing products that require high quality and reliability (e.g., aircraft parts) because it is still challenging to control microstructures during the L-PBF process," Kusano said. "To achieve this microstructural control, we need to investigate the effects of various process parameters such as laser power and laser scanning speed on the thermal histories."

Experimental and numerical approaches

"Both numerical analysis and experimentation can be used to study the relationships between processes, microstructures and material properties," Kusano said. "It is very costly and time consuming to fabricate, observe and evaluate a large number of samples with different process parameters. I therefore use both experiments and numerical analyses to understand the relationships."

Kusano performed a thermo-fluid analysis of an L-PBF process by modeling powder spreading, the fluid dynamics of the melt pool and heat transfer within the material. Based on this analysis, he developed a computer program to simulate microstructure growth. The simulated 3D animation shows crystal grains growing along the building direction (figure 2). Kusano used this simulation to search for optimum laser scanning parameters to produce desired material properties.

"I also use machine learning to predict the tensile properties of Ti alloy microstructures fabricated using L-PBF with various process parameters," Kusano said. "I prepared a dataset by extracting microstructural features (e.g., crystal grain diameters and aspect ratios) from microscopic images and conducted tensile strength tests. I then performed multiple linear regression on the dataset and made some progress in identifying the process parameters and microstructures that influence tensile properties." Predicting mechanical properties is difficult even when Ti microstructures have been prepared using better understood forging and casting processes. Thus, machine learning-based prediction could greatly improve the reliability of Ti alloys made using L-PBF.

Another factor affecting product reliability is defects that may form during the L-PBF process that may unexpectedly damage a manufactured product even if its geometry is exactly as designed. "Defects are mainly classified as voids and cracks that form through different mechanisms," Kusano said. "Voids are known to be controllable by adjusting laser energy density. However, effective methods of suppressing cracking have yet to be discovered. Crack formation in Ni-based alloys is known to be influenced by laser power, scanning speed, and preheating temperatures. Cracking mechanisms have also been found to differ depending on the stage of the L-PBF process (e.g., fast solidification after laser scanning, thermal cycling during fabrication of subsequent layers and post-heat treatment processes). I'm currently studying the cracking mechanisms of Ni-based alloys by conducting experimental fabrication and detailed observations, and performing numerical analyses to simulate thermal histories, local stress and strain and microstructures."

Developing L-PBF with automatic process parameter control

"If my numerical analysis and experimental research is successful, I hope in the future to add a feedback control system to an L-PBF machine," Kusano said. "For example, I'm developing a thermal analysis program with a feedback control subroutine to maintain the surface temperature of a target. Experimental fabrication will then be conducted using predetermined process parameters based on the simulated results." Most commercial L-PBF machines do not currently have such systems. Kusano will continue to pursue research to develop novel and reliable structural materials.

RESEARCH 3

Ceramic matrix composites: a revolutionary aircraft material

Hideki Kakisawa

Group Leader, Ceramic Matrix Composites Group

Ceramic matrix composites (CMCs) are heat-resistant, lightweight materials. They have been used in some aircraft engine components. Hideki Kakisawa has been working to improve CMCs' heat resistance while exploring new applications for them by developing techniques to evaluate their performance and that of their coating materials. We asked him about his R&D activities.

Increasing commercial use

A ceramic matrix composite (CMC) is composed of ceramic fibers embedded in a ceramic matrix. Although ceramics themselves are hard and brittle, fiber-reinforced CMCs are resistant to cracking. Even if a CMC cracks, the fibers it contains will prevent the crack from growing, preventing unexpected, sudden fractures. Lighter weight than metals and superior to them in heat resistance, CMCs make promising aircraft materials.

"In fact, non-oxide CMCs were adopted in 2016 for use in high-pressure jet engine turbine components for commercial aircraft," Kakisawa said. "Their use in combustion chamber inner walls and other components is also being considered. In addition, oxide CMCs have begun to be used in jet engine exhaust nozzles. Currently available non-oxide CMCs are able to withstand temperatures as high as 1,200 to 1,300°C. To further improve their heat resistance, we have been developing techniques to evaluate CMCs and their coating materials."

Real-time observation at high temperatures

The CMCs for use in jet engine turbines become significantly more susceptible to corrosion with increases in the temperature of the combustion gases that rotate turbines. To address this issue, the surfaces of these CMCs are sealed with environmental barrier coatings (EBCs).

"The internal temperatures of high-pressure turbines rise and fall repeatedly as an aircraft takes off and lands," Kakisawa said. "Rapid heat cycles cause strain because thermal expansion rates of the CMC and EBC components are different. This in turn causes the EBC to crack and separate, exposing the CMC to corrosion. It had been impossible to know when EBCs had been damaged by high temperatures and heat cycles. This is because at high temperatures, materials emit thermal radiation in the form of intense light, making them difficult to observe. To overcome this problem, we developed an original optical microscope capable of observing these materials at high temperatures (figure 1)."

Using this microscope, Kakisawa's team succeeded for the first time in the world in successive microscopic observation as their temperatures increased from room temperature to 1,400°C (figure 2). Moreover, the team was able to quantify the strain that developed in the samples by performing digital image correlation (DIC) analysis. The team also succeeded in capturing the very moment at which a crack was generated in an EBC—a significant achievement that may provide clues to the development of higher-performance EBCs.

"Replacement of metals with CMCs in key components responsible for passengers' lives could be a turning point for Japan," Kakisawa said. "Western nations have been leading jet engine development, but if this shift really proceeds, Japan's influence in this area may increase as it is the pioneer of ceramic fiber and a major producer."

Kakisawa's team has been conducting a number of research projects in collaboration with domestic heavy manufacturers with the aim of developing CMCs and EBCs that meet the requirements for practical use in aircraft.

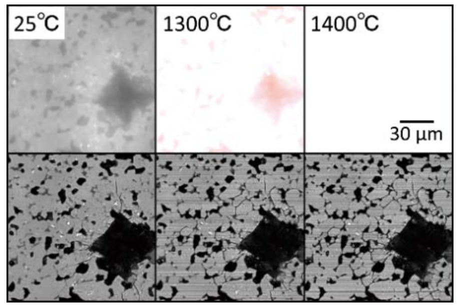


Figure 2. In-situ observation using a high-temperature optical microscope

A ceramic surface observed at different temperatures. Using a general-purpose optical microscope causes the images captured at high temperatures to be overexposed due to intense thermal radiation (upper images). By contrast, the high-temperature optical microscope Kakisawa' s team developed enables images to be captured with appropriate brightness and contrast at surface temperatures as high as 1,400° C (lower images).

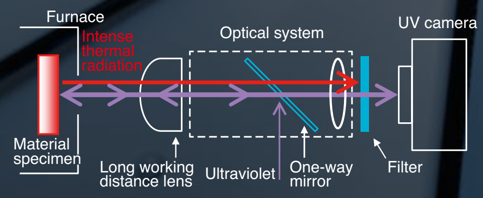


Figure 1. Schematic diagram of the high-temperature optical microscope developed by Kakisawa' s team

A heated material specimen is first irradiated with ultraviolet light. The UV light reflected by the specimen is then captured by the UV camera. The heated specimen also emits intense thermal radiation which is blocked by a filter placed in front of the camera. The surface of the specimen has a minute heat resistant speckle pattern developed by Kakisawa' s team. Successive images are taken of the specimen while tensile force is applied to it. By analyzing these images using a digital image correlation (DIC) technique, the displacement of the minute pattern due to the force applied is then quantified, determining strain distribution across the specimen.

RESEARCH 4

Solidification cracking—a cause of severe weld failure—caught in the act!

Tomoya Nagira

Group Leader, Welding and Joining Technology Group

Welding is an indispensable technique used to construct a variety of structures. Solidification cracking—the formation of shrinkage cracks during the solidification of molten weld metal—shortens the lives of welded structures. Tomoya Nagira has been investigating solidification cracking mechanisms using a synchrotron x-ray imaging technique at the SPring-8 facility.

Alloy samples used in Spring-8 experiments

Observing weld formation processes obscured by the welding arc

Arc welding uses electricity to create enough heat to melt and join pieces of metal. Solidification cracks are a type of defect that can develop during welding. Although solidification cracking has been the subject of research for more than half a century, the details of its formation mechanisms remain unknown. This is primarily because solidification cracking had been impossible to observe as it occurs within a flash of light and because molten metal solidifies very quickly.

To overcome these issues, Nagira came up with a plan for in-situ observation of weld formation using a synchrotron x-ray imaging technique at SPring-8—the world's largest synchrotron radiation facility. SPring-8 is able to generate synchrotron x-ray beams with among the highest spatial and temporal resolutions in the world.

Nagira hoped that this in-situ observation scheme would enable him to take real-time, detailed measurements of events occurring during the weld formation process. He developed a device capable of observing a metallic specimen being subjected to arc welding at high resolution and installed it in a SPring-8 beamline. In 2019, he used this setup to carry out in-situ observations of welds made of an iron-manganese-silicon alloy developed by NIMS. This experiment revealed for the first time in the world that phase transformation—the change from one crystalline structure to another—may be an important key to containing solidification cracking (figure).

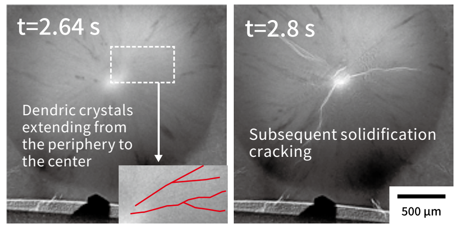
"No one had previously attempted in-situ observation of arc welding using a synchrotron x-ray imaging technique," Nagira said. "Developing an observational device for this specific purpose was therefore a process of trial and error. More than a decade of our R&D into in-situ observation technology made this achievement possible."

Predictions based on observational data

"Our long-term goal is to develop welding techniques capable of fully preventing solidification cracking," Nagira said. "For the time being, we aim to formulate guidelines on how to reduce solidification cracking."

The novel x-ray imaging system Nagira developed has generated a wealth of valuable weld-related data. Nagira hopes to ascertain weld fracture mechanisms caused by solidification cracking by compiling x-ray imaging data into databases to make it available for data-driven research.

Alloy composition susceptible to solidification cracking



Alloy composition resistant to solidification cracking

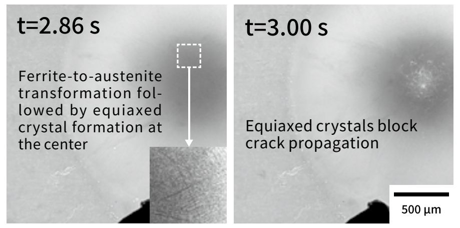


Figure. Small areas of two iron-manganese-silicon alloy specimens with different composition ratios were subjected to arc welding. The subsequent melting and solidification processes were then monitored every 0.02 seconds using an x-ray imaging technique. Solidification cracking occurred in the specimen whose austenitic phase remained unchanged during the melting and solidification process (upper images). By contrast, the specimen that underwent phase transformation from ferritic to austenitic was able to contain solidification cracking (lower images).

RESEARCH 5

Large-area, high-resolution, three-dimensional imaging of materials using multifunctional microscopes

Toru Hara

Group Leader,
Microstructure Analysis Group

Hara operating a PFIB-SEM. A PFIB is applied to a material specimen to remove individual atomic layers from its upper surface one at a time. After each layer is removed, a two-dimensional image of the exposed surface is captured using an SEM. The resulting sequence of 2D images is then combined to construct a 3D image of the specimen.

Structural materials degrade over time as they undergo various damaging processes, such as deformation, cracking and corrosion. Toru Hara uses his expertise in electron microscope-based analysis to study the mechanisms behind these processes through various structural materials research projects.

Sophisticated electron microscopy

The physical properties of structural materials are greatly influenced by their microstructures, which can be characterized at scales ranging from nanometers to millimeters. State-of-the-art analytical technologies play a very significant role in understanding the relationships between microstructure and physical properties.

“For this purpose, we have developed a number of advanced electron microscope-based analytical technologies, including an orthogonally-arranged focused ion beam scanning electron microscope (FIB-SEM) and a plasma focused ion beam-SEM (PFIB-SEM; photo above),” Hara said.

These electron microscopes are capable of observing a large three-dimensional area of a sample material at high resolution. They function similarly: an ion beam is applied to a specimen to remove thin layers from its upper surface. After each layer is removed, an SEM image of the exposed surface is captured. This process produces a sequence of hundreds of cross-sectional images. These images are then processed by a computer to construct a three-dimensional image of the specimen with its microstructure. This image can be used to analyze various microstructural features, including compositions and crystalline orientations.

“We set up our first orthogonally-arranged FIB-SEM in 2011,” Hara said. “Some researchers had since expressed an interest in observing even larger areas of sample materials at high resolution. To meet this demand, we introduced a PFIB-SEM in 2020. The PFIB-SEM is able to observe a three-dimensional area of a specimen approximately 1,000 times larger than the FIB-SEM is capable of imaging. This was made possible by its ability to remove a specimen’s surface layers considerably faster.”

Discoveries that contradict widely accepted metallic fatigue mechanisms

Using the PFIB-SEM, Hara’s group solved an old mystery. Metals develop microcracks through repeated use. These cracks grow over time, eventually leading to fracture. Although this process had long been known, the mechanisms behind it had been poorly understood.

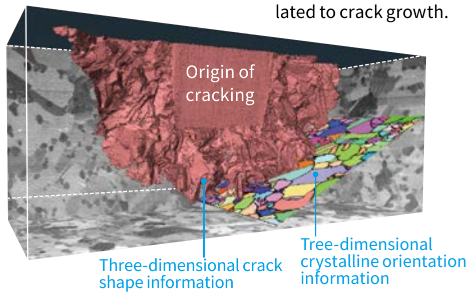
“We tackled this issue in collaboration with Hideaki Nishikawa (Principal Researcher, Fatigue Property Group),” Hara said. “Our group intentionally induced cracking in a nickel-based alloy specimen, collected FIB-SEM image data from the cracked specimen and processed the data using the computer program Nishikawa developed to construct a three-dimensional image of the specimen. By analyzing this image, we identified the metallic crystal planes conducive to crack growth for the first time (figure).”

This discovery surprised many experts in the field as the results challenged conventional wisdom.

Hydrogen-related materials are one of NIMS’ priority research areas. A key issue for these materials is hydrogen embrittlement—the weakening of metals when penetrated by hydrogen atoms, making them susceptible to fracture. Hara’s group plans to investigate hydrogen embrittlement mechanisms in collaboration with other materials researchers.

“The approach I’m considering is to prepare metal samples that have and have not been penetrated by hydrogen and compare how they develop cracks,” Hara said. “There are many other ongoing structural materials development projects that require in-depth observational analysis. I want to contribute to these R&D projects using the PFIB-SEM and other leading-edge technologies.”

Figure. Three-dimensional image of an alloy specimen in which cracks were intentionally induced. The image shows crystalline microstructures and orientations related to crack growth.



(For details, see NIMS NOW vol. 23, no. 2, p. 10)

RESEARCH 6

Accurate simulation of heat-resistant material microstructures based solely on fundamental physical laws

Ryoji Sahara

Group Leader,
Computational Structural
Materials Group

Materials used in aircraft engines and other systems that operate at high temperatures need to be heat resistant and durable. Experimentally evaluating the physical properties of these materials is time-consuming and costly. Ryoji Sahara has been developing accurate computer simulation techniques that can be used to expedite property evaluations and materials development.

Calculation errors: a primary source of inaccuracy in simulations

Strength, toughness and other properties of heat-resistant metals are closely related to their microstructures, which can be characterized at various scales ranging from atomic and electronic movement to macroscale microstructures. Controlling these properties therefore requires an understanding of complex microstructures at multiscale. Computer simulations are indispensable for predicting microstructures that may enable materials to exhibit desirable physical properties.

Atomic bonding states and electronic states can be accurately predicted by quantum mechanics-based first-principles calculations. However, because these calculations work properly only at extremely small spatial and temporal scales, it is difficult to apply them to macroscale microstructures even with the aid of a supercomputer. Phase field (PF) models are a viable tool for solving macroscale problems, although they also have a disadvantage: the magnitude of the calculation errors from these models substantially increases when applied to materials used at high temperatures. According to Sahara, a common way of correcting PF models has been substituting some of their parameters with experimentally measured values.

“Although conventional PF models can be used to verify experimental results, they cannot be used to make predictions,” Sahara said. “I wanted to develop simulation techniques capable of making predictions without needing to incorporate experimentally measured values by combining first-principles calculations and PF models. To achieve this, I teamed up with Yokohama National University.”

Technique accurately simulates heat-resistant materials’ microstructures

Sahara’s team collaborated in formulating the so-called potential renormalization theory (figure 1)—a framework for incorporating first-principles calculations suitable for solving atomic-scale problems into a PF model suitable for solving macroscale problems. Based on this theory, the team then developed a first-principles PF method capable of simulating the complex microstructures of heat-resistant materials based solely on fundamental physical laws without relying on experimentally estimated parameter values. This method was published in 2019 as the world’s first simulation technique of its kind. If efforts to determine the microstructures of heat-resistant materials using this method prove successful, they would facilitate investigation into the physical properties (e.g., strength) of these materials.

“We actually used this method to simulate the microstructures of heat-resistant nickel-aluminum alloys and the Ti-6Al-4V titanium alloy (a.k.a., Ti64),” Sahara said. “We verified that the calculated values were mostly consistent with experimentally measured values for all of the dozen alloys with different compositional ratios we studied (figure 2).”

In future research, Sahara’s team plans to expand the range of materials to which the first-principles PF method can be applied and simulate their microstructures. These include interstitial solid solutions—formed by small atoms of a solute filling in the empty spaces between the atoms of a solvent in a crystal lattice—and high-entropy alloys that are potentially stronger and tougher and superior in heat resistance to conventional alloys.

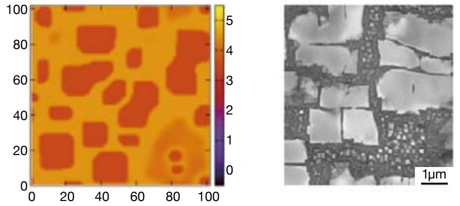


Figure 2. Comparison of theoretically predicted and actually observed nickel-aluminum (Ni-Al) alloy microstructures

Microstructures of alloys composed of 82% Ni and 18% Al which have been heated to the temperature of 1,027 ℃. (Left) Microstructure simulated by the first-principles phase field method. (Right) Microstructure observed under an electron microscope. The boxy shapes of the deposits were properly simulated.

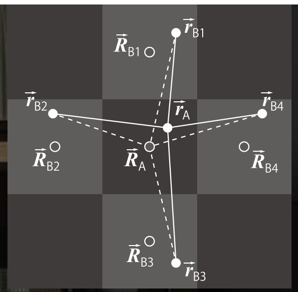
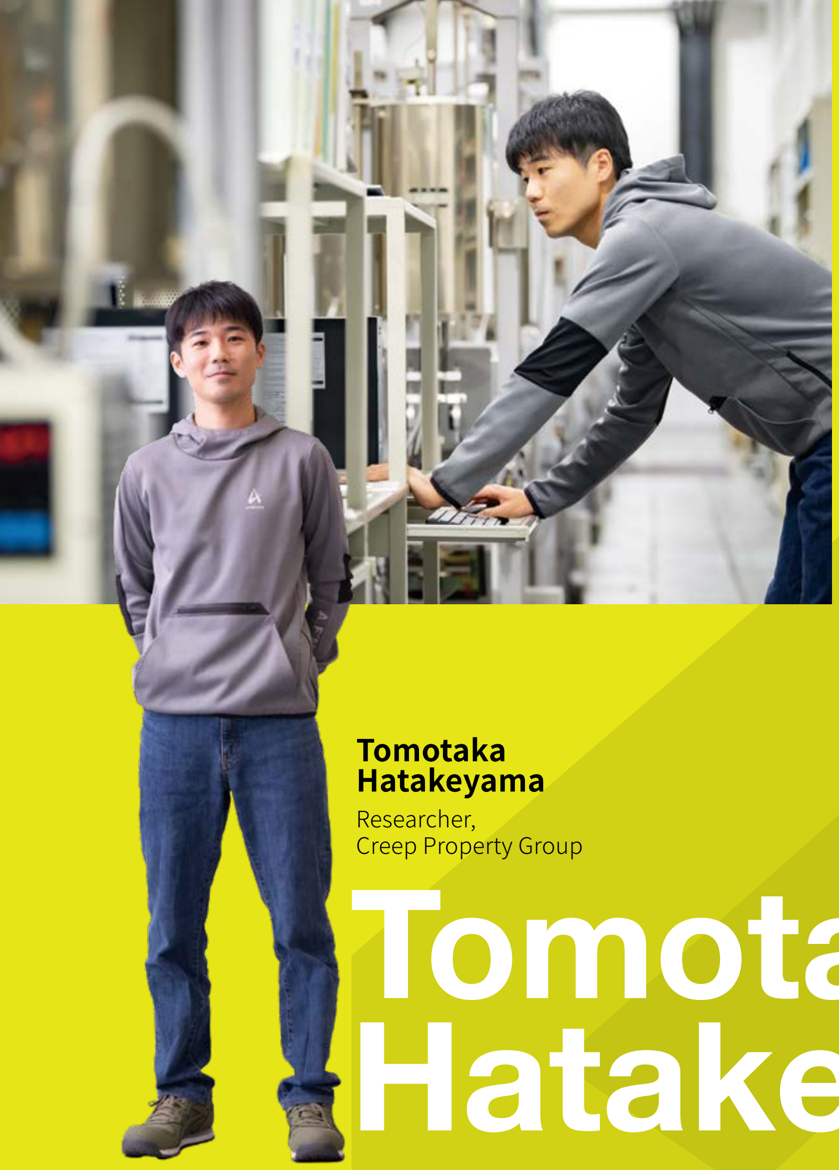


Figure 1. Diagram illustrating the potential renormalization theory concept

The potential renormalization theory assumes that the potential (represented by solid lines) of a given atom ($\vec{r}_A$) with internal degrees of freedom, which constitutes a crystalline lattice (the square cell at the center), is influenced by its neighboring atoms ($\vec{r}_{B1}$~, $\vec{r}_{B2}$, $\vec{r}_{B3}$ and $\vec{r}_{B4}$). This atomic potential is supplanted by the potential (represented by dashed lines) at the center of the same lattice ($\vec{R}_A$). This method incorporates first principles into a phase field model without reducing its accuracy in solving macroscale problems.

Leaders of the future

We highlight research projects being carried out by three young researchers as part of NIMS' efforts to develop next-generation structural materials.



Tomotaka Hatakeyama

Tomotaka Hatakeyama
Researcher,
Creep Property Group

Effect of microstructure on the long-term creep strength of L-PBF-manufactured heat-resistant steel

Laser powder bed fusion (L-PBF) is a revolutionary metal additive manufacturing process capable of fabricating complex structures in net shape (see the schematic diagram on p. 8). However, L-PBF has not been adopted for the manufacturing of mechanical parts that need to meet high reliability requirements. To ensure that L-PBF products satisfy these requirements, the fundamental relationships and mechanisms affecting their microstructure and physical properties need to be investigated.

I am currently conducting long-term creep testing on heat-resistant steel samples (modified 9Cr-1Mo steel) manufactured using L-PBF. During the testing, a constant tensile force is applied to steel test pieces while they are exposed to high temperatures (500–700℃). The elongation of the test pieces and the time to fracture are then measured. This steel has conventionally been processed through casting, rolling and heat treatment, allowing it to form an entirely martensitic microstructure. By comparison, the microstructure of L-PBF products is composed of a complex mixture of martensite and ferrite phases resulting from repeated localized heating and rapid cooling. This two-phase microstructure can be transformed into a homogeneous martensitic microstructure similar to that produced using conventional methods by subjecting it to a heat treatment. I am trying to determine whether L-PBF products with the two-phase microstructure or homogeneous martensitic microstructure are equally reliable as the products made with conventional methods through long-term creep testing. If my research produces evidence that L-PBF products are equally reliable, this manufacturing process may find a significantly wider range of applications.

Kazuho Okada

Kazuho Okada
Researcher,
Steel Research Group

Achieving both high-strength and high-hydrogen-resistance in low-/medium alloy steel by controlling microstructure

Hydrogen embrittlement—the phenomenon by which metals and alloys become brittle due to the hydrogen introduction—is a very challenging issue in efforts to reduce automobile weight for exhaust gas regulations. This is because of a well-known trade-off—resistance to hydrogen embrittlement decreases with increasing the strength of steel, and vice versa. To overcome this issue, I have been investigating the effects of hydrogen on deformation and fracture behavior.

Recently, I succeeded in improving the hydrogen embrittlement resistance of low-carbon martensitic steel (a typical high-strength steel) without compromising its strength by increasing carbon segregation at specific grain boundaries using a simple heat treatment. Increasing carbon segregation at the boundaries presumably strengthens the bonding between grains and suppresses the hydrogen accumulation at the boundaries, improving the resistance against hydrogen-related fracture at the grain boundaries. I am also exploring other approaches to developing fracture-resistant steel without sacrificing its strength.

Slow strain rate tensile test being conducted at the temperature of liquid nitrogen (see also the photo of a metallic specimen fractured via this test on p. 2)

Kentaro Wada

Kentaro Wada
Researcher,
Cryogenic Fatigue Group

Amassing low-temperature hydrogen embrittlement data to facilitate hydrogen infrastructure material design

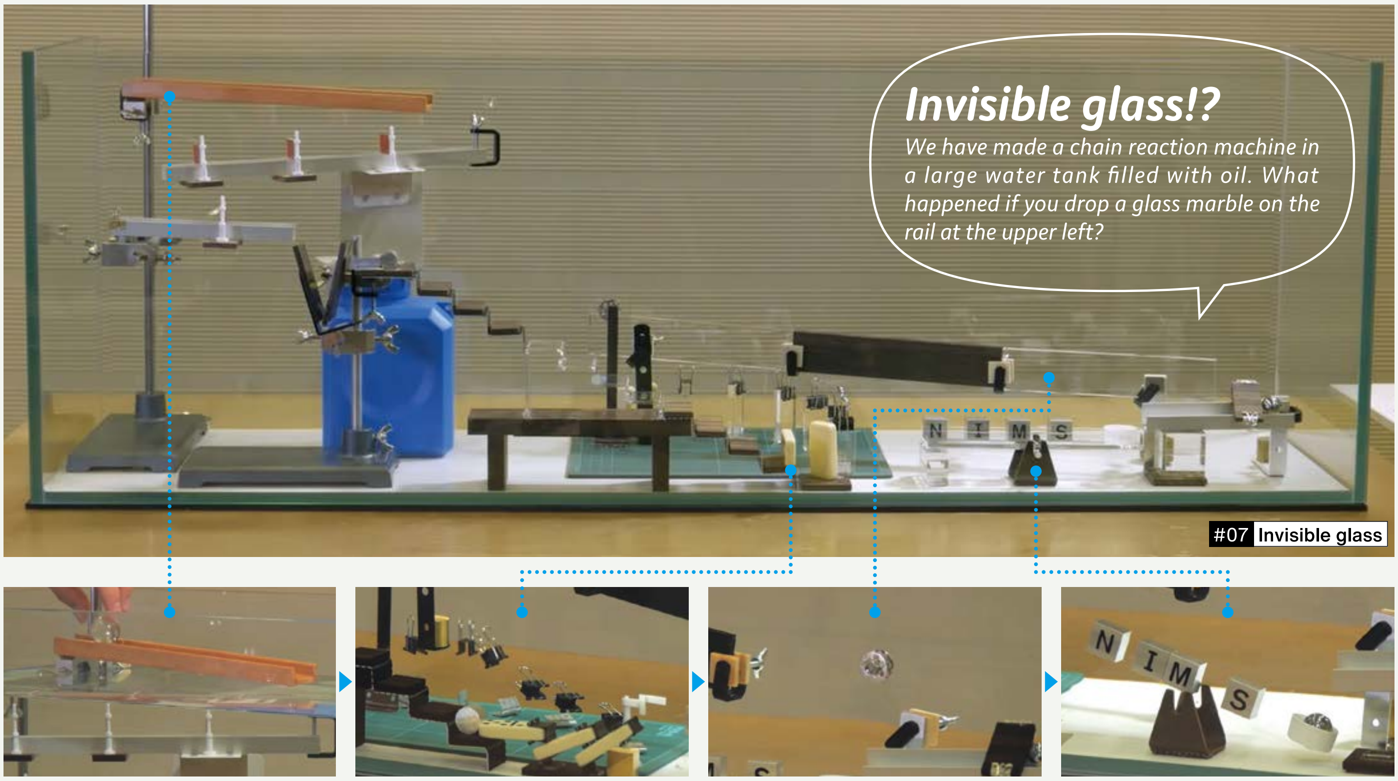
My project is similar to the research projects being conducted by Dr. Kazuho Okada (lower left). I am also evaluating the resistance of metallic materials to hydrogen embrittlement at low temperatures. Materials which are applicable for hydrogen infrastructure are limited due to a lack of hydrogen embrittlement resistance data. Because hydrogen can be transported most efficiently in a liquid state at -253° C, the properties of hydrogen infrastructure materials need to be evaluated under cryogenic conditions. It is urgent to generate evaluation data and make it available for materials design.

The slow strain rate tensile test is one of the tests I have been carrying out. For this test, I prepare metallic specimens that have been charged with hydrogen, put them in a cooling bath (e.g., liquid nitrogen with a boiling temperature of -196 ℃) and apply a tensile load to them until the fracture. I have been analyzing testing results to determine hydrogen embrittlement processes under cryogenic conditions. I also investigate the mechanisms behind the hydrogen embrittlement in metallic materials by observing fracture surfaces by using a scanning electron microscope and other technologies. In addition, I have been collecting other types of data (e.g., fracture toughness and fatigue properties) indispensable to the design of hydrogen infrastructure products. Through these activities, I hope to contribute to the development of hydrogen-compatible metallic materials.

A steel specimen is immersed in a salt bath (i.e., high-temperature molten salt), which is a basic heat treatment for controlling steel microstructure. The physical properties of steel can vary widely depending on the treatment temperatures and cooling rates.

A Message to Future Scientists

— YouTube video clips by NIMS and EUPHRATES —



Invisible glass!?

We have made a chain reaction machine in a large water tank filled with oil. What happened if you drop a glass marble on the rail at the upper left?

#07 Invisible glass

Drop a glass marble...

Invisible dominos?

Invisible rail??

Invisible seesaw???

What is “A Message to Future Scientists” ?

“A Message to Future Scientists” is a YouTube video clip series created jointly by NIMS and EUPHRATES Ltd. (a group of specializing in creative work, including NHK’s educational TV programs). We have added an English subtitle option to these clips.

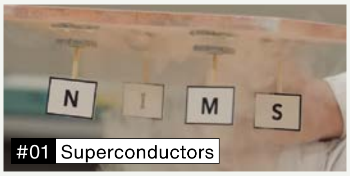
This scientific video series has been popular in Japan with a cumulative view count exceeding 7 million. These clips demonstrate the various intriguing scientific phenomena and unique materials NIMS has developed using fascinating images that are also entertaining and beautiful.

An English subtitle option makes it accessible to a broader global audience with an interest in science. We hope you enjoy it!

"A Message to Future Scirntists" video clips

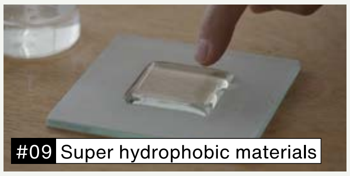
List of “A Message to Future Scientists” video clips

We have released 16 video clips so far.



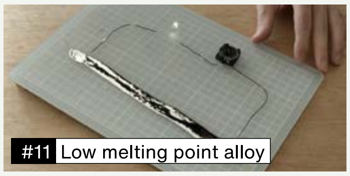
#01 Superconductors

#02 Shape memory materials

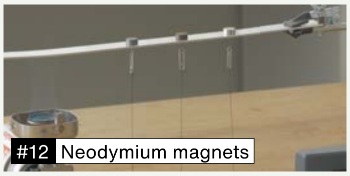


#09 Super hydrophobic materials

#10 Thermal expantion

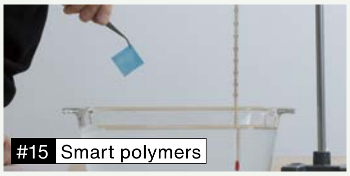


#11 Low melting point alloy



#12 Neodymium magnets

#13 Aerogel heat insulator



#15 Smart polymers

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photo by Michihito Ishikawa　editorial design by Barbazio Inc.
Front cover photo: magnesium foil (see p. 2 for details)

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