NIMS NOW INTERNATIONAL

NATIONAL INSTITUTE FOR MATERIALS SCIENCE

2023 No. 2

Research Digest 2022

Research Digest

2022

NIMS significant research accomplishments are published as press releases
with the potential to lead to the creation of novel practical materials.
Of the many R&D results we published in 2022, we selected 10 highlights.
For a broader and deeper understanding of recent materials research at NIMS,
please check out the complete collection of press releases on our website.

2022 NIMS Press Release INDEX

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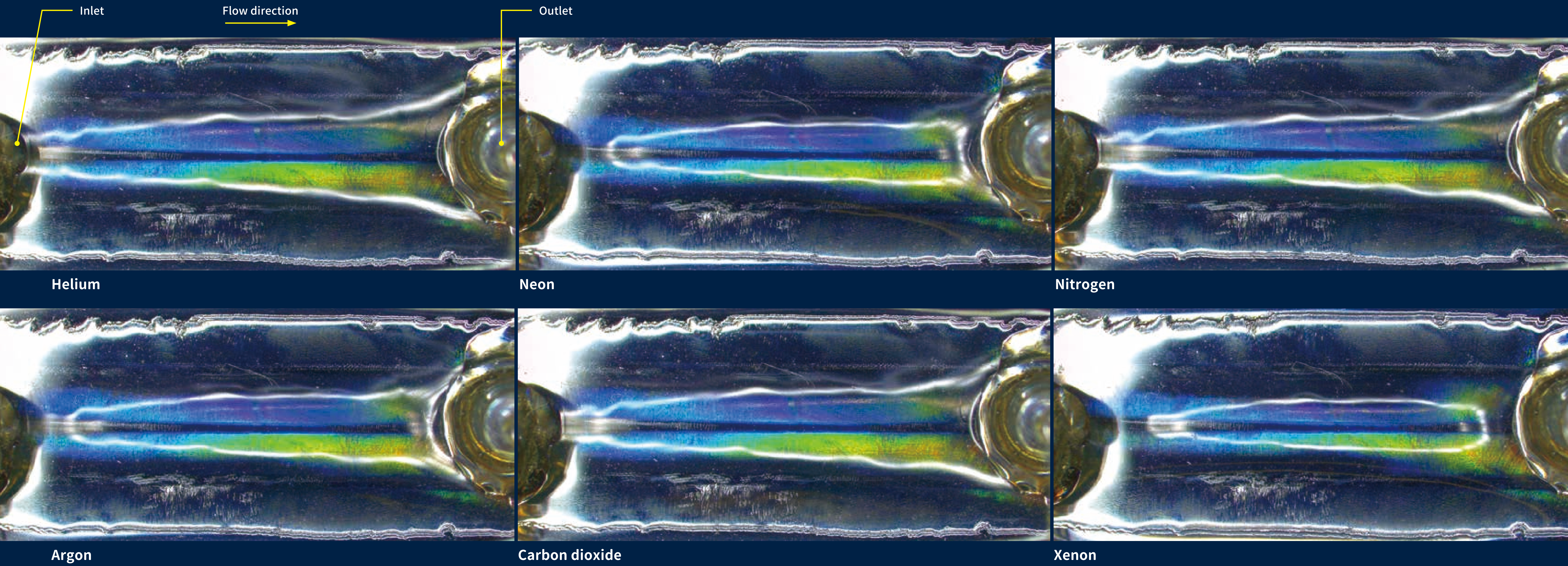
NEWS!

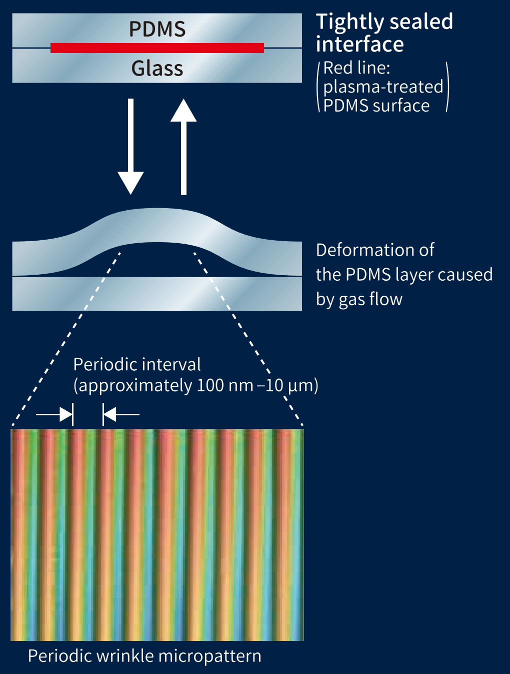


01 Colorfully revealing the invisible

We are surrounded by transparent, colorless gases. When these gases flow through a special device developed by Dr. Kota Shiba, the device is able to image them in iridescent colors. The slightly different coloration patterns associated with various gases can then be used to identify them.

This gas imaging device is composed of a glass substrate and a polydimethylsiloxane (PDMS) layer—a soft silicone material—with a tightly sealed interface between them. Before these two layers are combined, part of the PDMS surface is treated with argon plasma to harden it. The area surrounding the hardened surface is then treated with oxygen plasma to make it adhesive. Finally, the treated PDMS layer is tightly attached to the surface of the glass substrate, completing the device.

The device is easy to use. A gas of interest is forcibly introduced into the PDMS–glass interface. As the gas passes through the interface, the argon plasma-treated, hardened PDMS surface forms a periodic wrinkle micropattern which causes iridescent colors (i.e., structural coloration). The structural coloration is produced when the micropattern interferes with incoming light. The periodic intervals of the micropattern change with the viscosity and density of the gas introduced. This change causes different structural coloration patterns to be displayed which can be used to distinguish different gas species under consistent flow conditions. This gas imaging and identification device is inexpensive and user-friendly and requires no power source or additional, more sophisticated equipment. It may potentially have wide-ranging applications, including healthcare and artistic applications.



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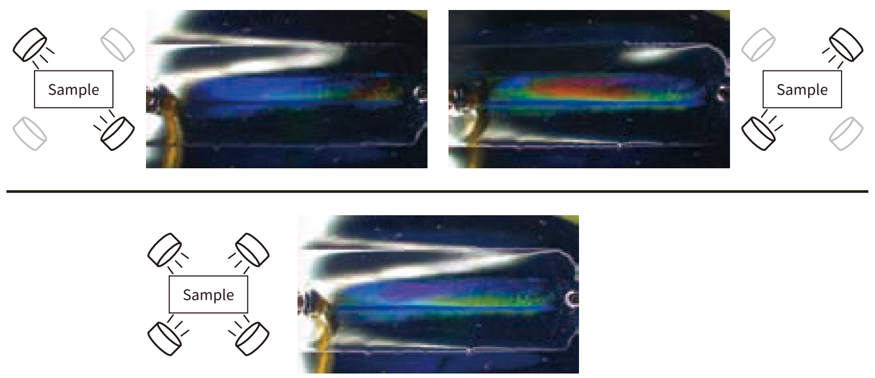
What is structural coloration?



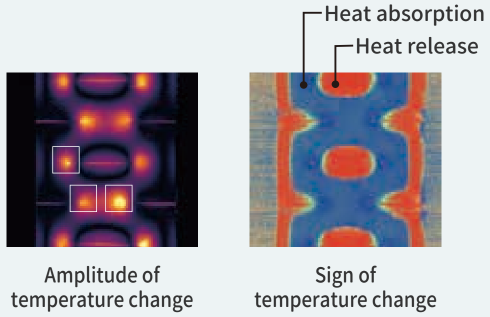
Structural coloration is produced by microscopically structured surfaces as they interfere with visible light. This coloration varies depending on the angles at which incident light interacts with the micropatterned surface. Shiba's device can demonstrate this phenomenon. When the device—through which nitrogen gas (N_2) is flowing—is exposed to light from different angles, it exhibits different structural coloration patterns (top two photos). Simultaneous exposure to light from four different angles causes the device to exhibit a combination of four different coloration patterns (bottom photo). Because each gas is associated with a unique structural coloration pattern, this device with four different light sources is effective in identifying different gases.

02 Heat manipulation inspired by traditional Japanese paper crafts

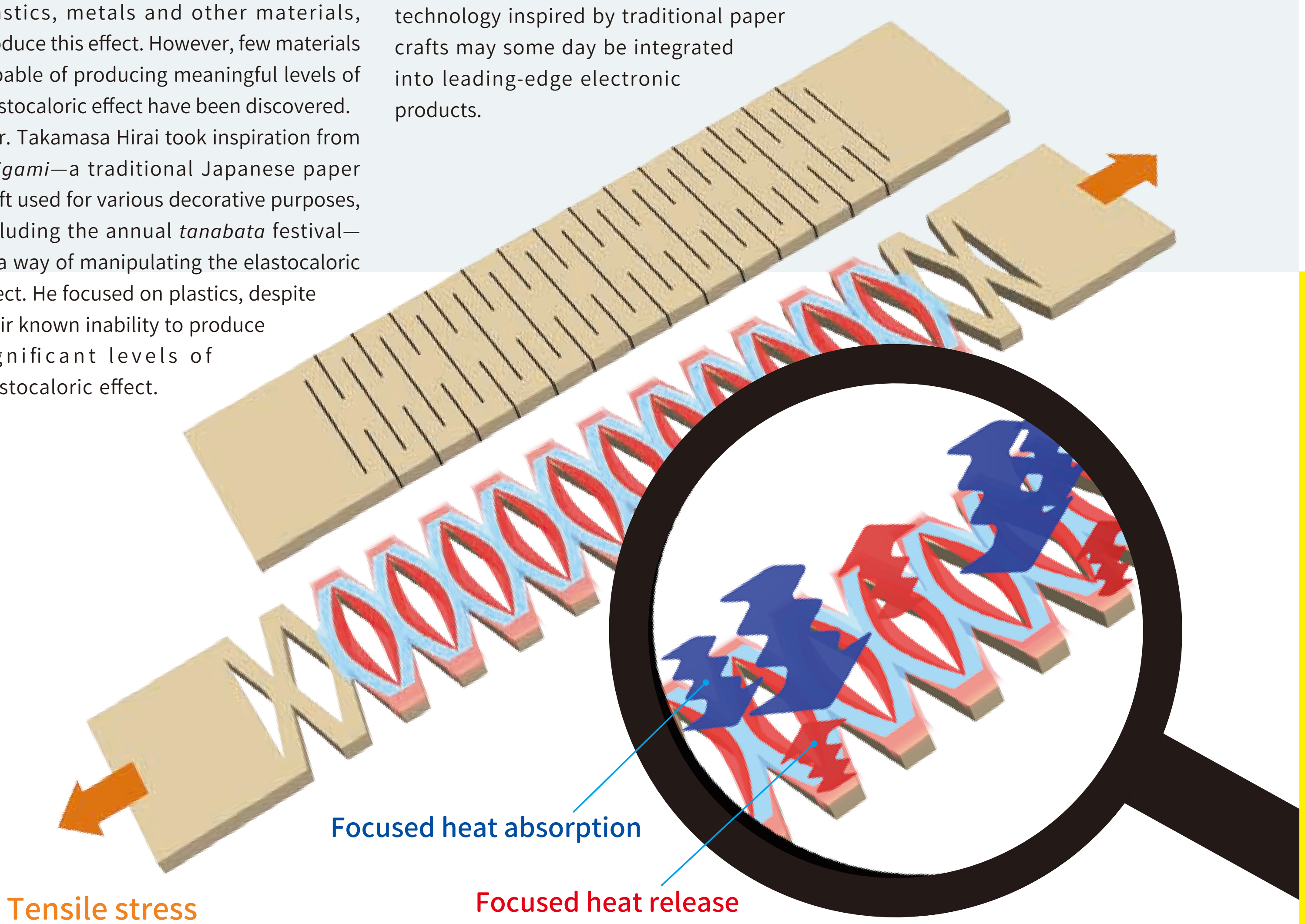
Electronic devices are becoming more sophisticated through miniaturization and greater functionality. However, the heat generated by these elaborate devices poses a major challenge. Excess heat often impedes device performance and leads to device failure. Refrigerators are able to cool their interiors by compressing and circulating a refrigerant gas. Integrating similar cooling mechanisms into much smaller devices is difficult. One potential approach to this issue is the use of the elastocaloric effect—absorption or release of heat by stretching or compressing the solids. Most of the solids, e.g., rubber, plastics, metals and other materials, produce this effect. However, few materials capable of producing meaningful levels of elastocaloric effect have been discovered.

Dr. Takamasa Hirai took inspiration from *kirigami*—a traditional Japanese paper craft used for various decorative purposes, including the annual *tanabata* festival—as a way of manipulating the elastocaloric effect. He focused on plastics, despite their known inability to produce significant levels of elastocaloric effect.

By processing a plastic sheet into *kirigami* patterns, Hirai succeeded in significantly increasing the localized elastocaloric heating and cooling performance of the material. The possible ingenuity of these position-resolved thermal managements on the sheet is wide-ranging. For example, specific regions in the sheet can be made to absorb or release heat or certain regions can be made considerably cooler than others by modifying the incision patterns in the sheet. In addition, *kirigami* processing enables even hard materials not only to stretch but also to bend to a greater extent using a smaller amount of force. This technology inspired by traditional paper crafts may some day be integrated into leading-edge electronic products.



A *kirigami* pattern was first cut in a polystyrene sheet—a type of plastic—which was then pulled from both sides to stretch it. The change in temperature across the stretched sheet was then measured using lock-in thermography. As a result, certain areas of the sheet were found to focus temperature changes (e.g., areas marked in white in the upper image). In addition, a phase image (the lower image) revealed that some parts of the sheet absorb heat while others release it depending on the distribution of internal stress.



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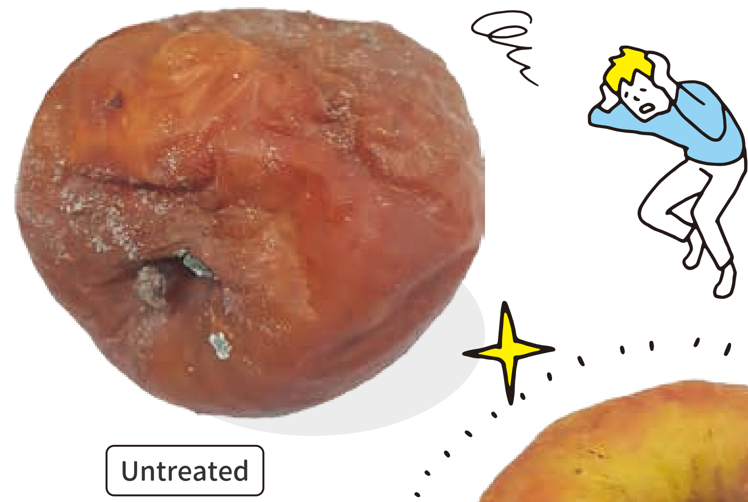
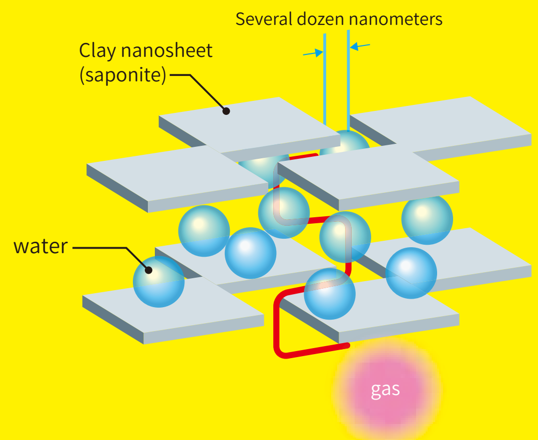


03 Clay nanosheets offer a new long-term apple preservation method

Food waste—including fresh produce waste—is a pressing issue. One approach to easing this problem is to develop technologies to preserve fresh produce for extended periods of time. Dr. Miharu Eguchi has fabricated a food coating film composed of clay nanosheets 1 nm in thickness made of silicon, oxygen and aluminum. The clay nanosheets are first dispersed in water and then applied to the surface of fresh produce to form a very thin coating over it. To assess the film's long-term food preservation capability, Eguchi conducted experiments on apples.

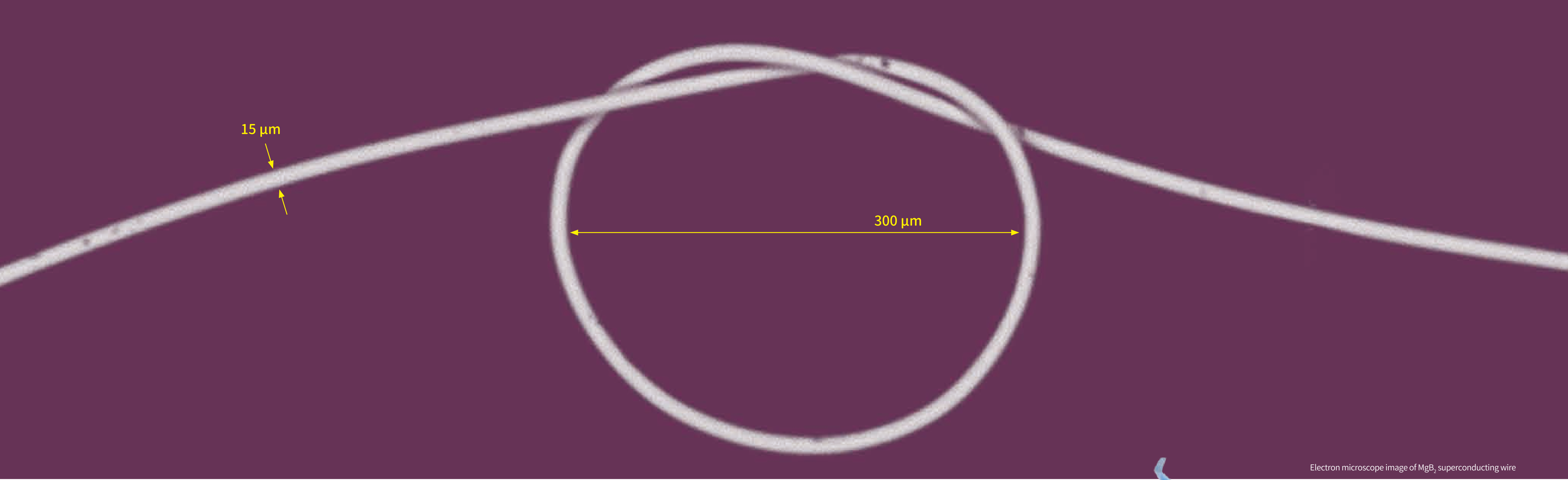
About three months into the experiment, apples that were untreated or covered in cling wrap had decayed or grown mold, while those coated with the clay film were free of decay.

The size of these nanosheets is the key to their effectiveness. When fresh produce, including apples, is supplied with abundant oxygen, it respires at a high rate, promoting ripening and mold growth. On the other hand, when the oxygen supply is completely cut off, it suffocates and spoils. Eguchi selected clay nanosheet particles several dozen nanometers in size, enabling pores of the ideal size to be created between the nanosheets, through which optimum amounts of oxygen are supplied to preserve fresh produce. In addition, the water content of the clay film enables it to come into tight contact with the apple it coats, presumably preventing the apple from either releasing or being exposed to ethylene gas—a cause of rotting—and preventing it from emitting insect-attracting odors. This clay film may help reduce food waste in the future.



Clay film structural schematic

Clay nanosheets retain water. Because the water molecules trapped in the gaps between the nanosheets have polarized electrical charges, these molecules effectively block undesirable gases (e.g., ethylene and insect-attracting odor compounds) from penetrating the clay film while allowing optimum amounts of smaller oxygen molecules to penetrate it.



Electron microscope image of MgB_2 superconducting wire

04 World's thinnest MgB_2 superconducting wire at 0.015mm in diameter:
transforming a hard, brittle compound into a flexible wire

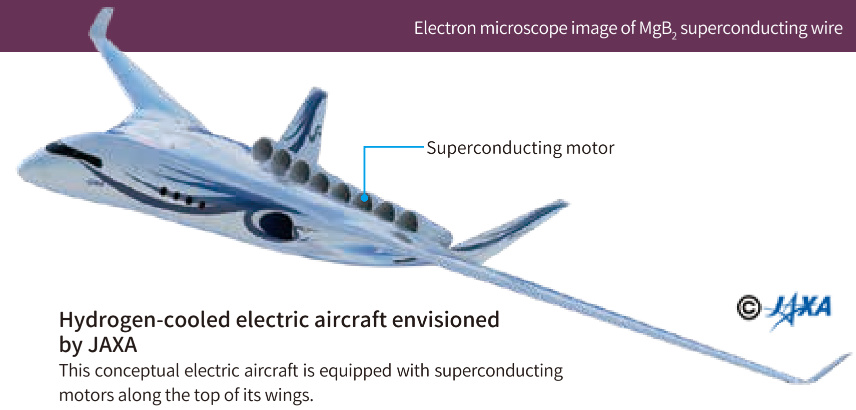
This photo shows a looped magnesium diboride (MgB_2)[*1] superconducting wire[*2] only 300 µm (0.3 mm) in diameter, somewhat resembling *mizuhiki*—a traditional style of Japanese decorative knot-typing. MgB_2 is fundamentally a hard, brittle compound similar to pencil lead in texture. Dr. Akihiro Kikuchi, his collaborators and Meiko Futaba Co., Ltd. jointly developed a technique to process MgB_2 into a wire 15 µm (0.015 mm) in diameter. This ultrafine wire is amazingly flexible and can be bent without breaking.

MgB_2 superconducting wires are expected to be vital to the development of superconducting magnets. A superconducting magnet is able to produce very strong magnetic fields when large electric currents flow through its coils of superconducting wire. Because superconducting wire coils are complex in shape and vary widely in size depending on the application of the magnets, the development of flexible MgB_2 wires capable of meeting these requirements was eagerly awaited.

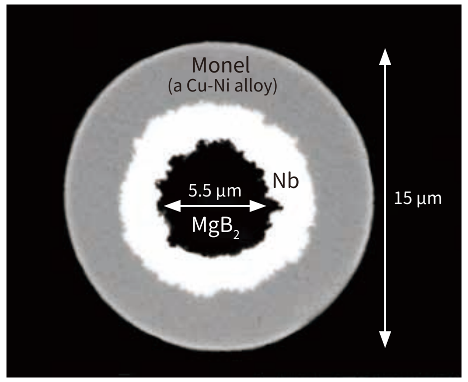
Expectations are growing for the development of superconducting motors driven by superconducting magnets. Widespread use of zero-emission superconducting motors to replace technologies driven by fossil fuels may potentially reduce CO_2 emissions. However, a major issue—alternating current (AC) losses in superconductors—needs to be resolved before these motors can be successfully developed. As the direction of the current flow in the motor alternates, the associated magnetic fields fluctuate, causing some of the power to dissipate as heat. To reduce AC losses, Kikuchi's group twisted several ultrafine MgB_2 superconducting wires together to create a thicker twisted wire. This twisted wire is more effective in reducing the extent to which magnetic flux lines travel than a non-twisted wire of the same thickness. The twisted wire is also conducive to controlling the electric currents generated between the adjacent ultrafine wires that compose it. These and other advantages make the MgB_2 wire energy-efficient. Kikuchi's group also made many other improvements—including insulating the ultrafine MgB_2 superconducting wire with a highly electrically resistant alloy—thereby significantly reducing AC losses in MgB_2 superconductors.

Japan Aerospace Exploration Agency (JAXA) is currently carrying out research on electric aircraft propelled by superconducting motors. The MgB_2 superconducting wire is expected to be an effective component of these motors because it is able to transition into a superconducting state at the relatively high temperature of 20 K—the temperature of liquid hydrogen—and it is very light as it is composed only of light chemical elements. NIMS and JAXA plan to begin joint research in the near future.

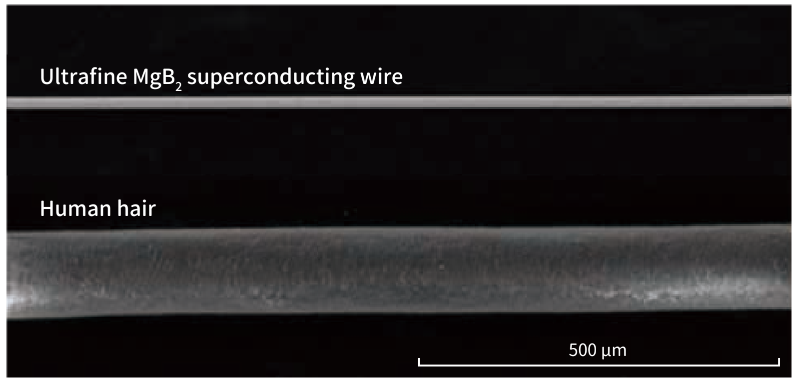
*1 Magnesium diboride (MgB_2) is an intermetallic compound made of magnesium and boron. An Aoyama Gakuin University research group discovered in 2001 that MgB_2 becomes superconductive at 39 K.
*2 Superconductors are materials whose electrical resistance disappears at their critical temperatures.



Hydrogen-cooled electric aircraft envisioned by JAXA
This conceptual electric aircraft is equipped with superconducting motors along the top of its wings.
Liquid hydrogen is used to cool the motors, enabling them to generate a fast-moving jet of air to propel the aircraft.



Cross-section of the world's thinnest MgB_2 superconducting wire
This wire is created by first filling a niobium tube with a mixture of magnesium (Mg) and boron (B) powder materials and then enclosing it in a Monel (a high-strength alloy of nickel and copper) tube. Monel is used to make the wire more compatible with the wire drawing process, while the niobium tube is used to prevent chemical reactions from occurring between the mixed powder and the Monel. After undergoing the wire forming process, the wire is thermally treated at 650°C, causing the Mg and B to react and convert into an MgB_2 superconductor. The MgB_2 core is only 5.5 µm (0.0055 mm) in diameter, as fine as a strand of spider silk.

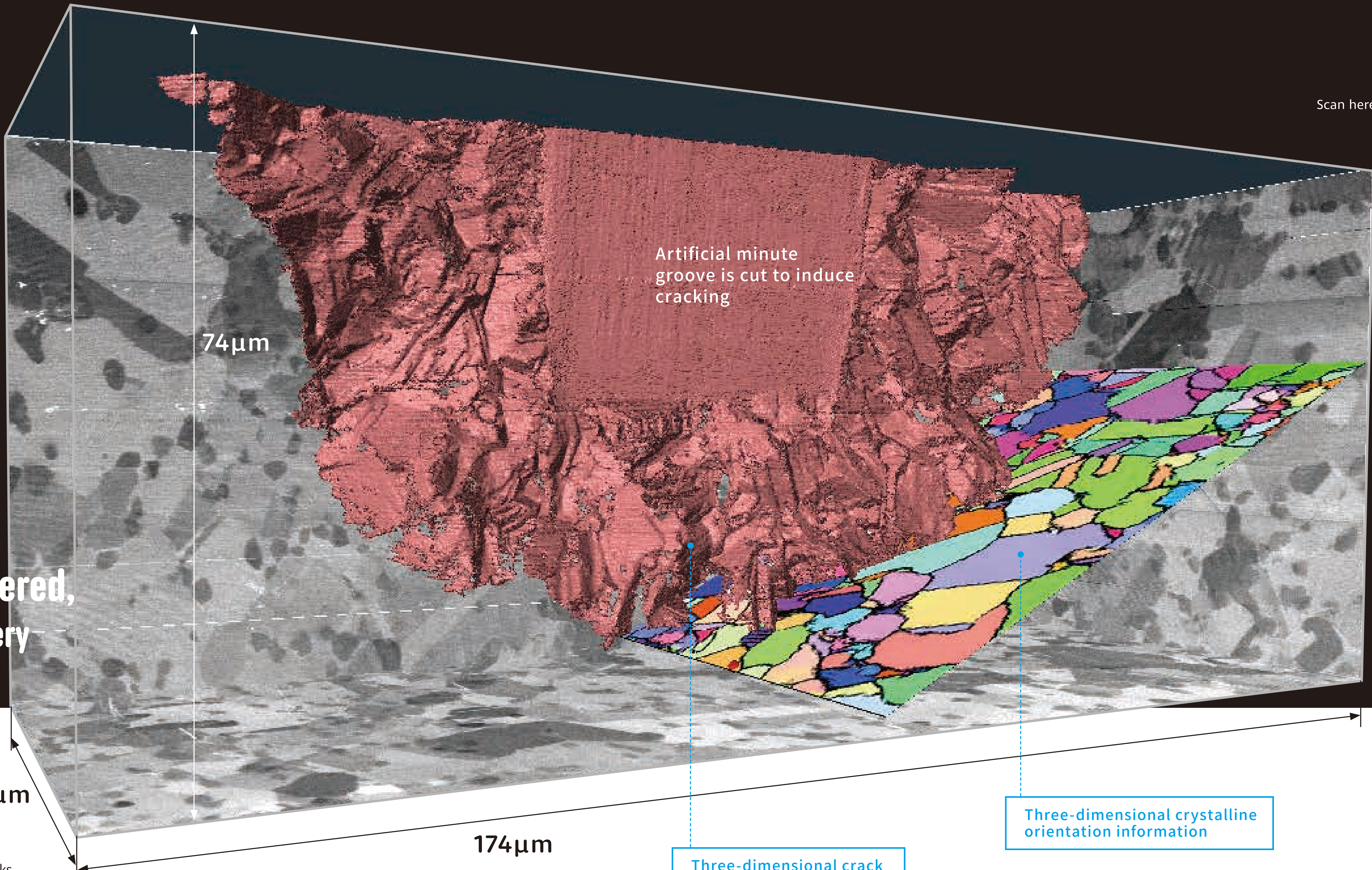


Comparison between the MgB_2 wire developed by Kikuchi's group (top) and a human hair
A human hair is 80–100 µm in diameter while the MgB_2 superconducting wire is only 15 µm in diameter. Alternating current (AC) losses—a key indicator of a superconducting motor's energy efficiency—can be categorized into hysteresis losses, coupling losses and eddy-current losses. Hysteresis losses can be reduced by making the cross-section of a superconducting wire smaller, and coupling losses can be greatly reduced by twisting several superconducting wires together.

05

Metallic fracture mechanisms uncovered, ending a 50-year mystery

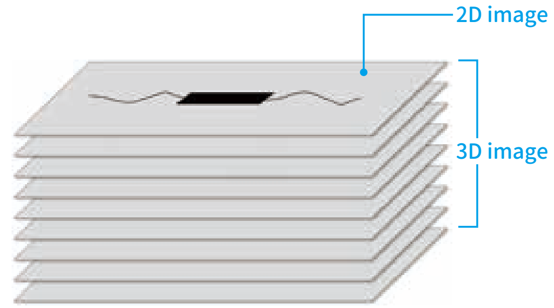
Scan here for details ▸



Metallic materials that are repeatedly subjected to external forces develop tiny cracks. These cracks slowly propagate and eventually cause the metals to fracture. This fatigue fracturing in metals has been a major cause of serious accidents. Predicting the fatigue lives of materials is therefore important in preventing accidents from occurring. Crack growth mechanisms have long been researched with the goal of improving the accuracy of these predictions. During the 1970s, scientists gained an in-depth understanding of the mechanisms that produce microscopic cracks in the early fatigue life stages and those behind the large cracks that result in fracturing in the final stage. However, the intermediate stages (i.e., the mechanisms by which microscopic cracks grow into larger ones) remained unknown.

Dr. Hideaki Nishikawa worked to unravel this long-standing mystery in collaboration with Dr. Toru Hara—a materials analysis expert—and other researchers. Nishikawa focused on a nickel-based superalloy which NIMS developed for use in aircraft engines. The team prepared a specimen of this superalloy, created artificial cracks in it and took various measurements from it in an innovative way using a PFIB-SEM*, a leading-edge analyzer. As a result, the team succeeded for the first time in three dimensionally imaging 200 micrometer-class cracks, including their growth traces and the orientations of crystals (lattice orientations) around them. The crack growth mechanisms revealed by this image analysis clearly deviate from the commonly accepted theory, reminding Nishikawa of the saying, "A picture is worth a thousand words."

As a crack grows in a metal, it destroys a succession of individual crystalline grains. This mechanism was previously thought to be driven mainly by a tensile force generated within crystalline grains. However, Nishikawa' s team found that the major force driving crack growth was actually a shearing force along the diagonal slip planes of crystalline grains. Nishikawa hopes to make society safer by making fatigue life predictions about materials more reliable.



*PFIB-SEM: A plasma focused ion beam (PFIB) is applied to a specimen to remove its atomic layers one by one from its upper surface. After each layer is removed, a two-dimensional image of the exposed surface is captured using a scanning electron microscope (SEM). The resulting sequence of 2D images is then combined to construct a 3D image of the specimen.

Image showing the orientations of crystals around the traces of crack growth

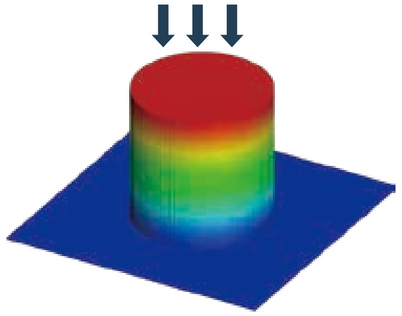
A majority of cracks were found to propagate along the (111) crystalline plane known as the slip plane (shown in blue).

06 Simple and economical 3D laser printing technology for fabricating single-crystal metallic parts

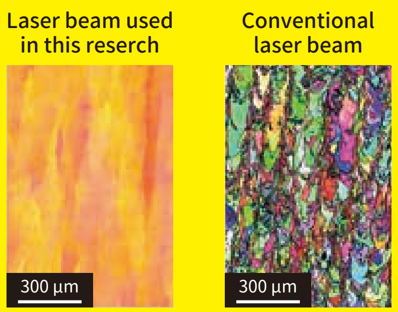
Aircraft engines are becoming more complex structurally as they undergo modifications to reduce their CO_2 emissions. Expectations are growing for the development of 3D printing technologies capable of easily and economically fabricating computer-designed metallic engine parts in complex shapes. Among the different 3D printing technologies available, laser powder bed fusion*—an additive manufacturing process in which a laser beam is used to melt a raw material in powder form—is more user-friendly and cost-efficient than electron beam melting. This is because laser powder bed fusion can be performed in the air rather than in vacuum. However, this technology also had a disadvantage: it was only able to manufacture polycrystalline objects (i.e., objects composed of randomly oriented crystals). A polycrystalline structure is significantly more susceptible to grain boundary fractures at high temperatures than a single-crystal structure with a continuous and orientation-aligned crystalline lattice. Therefore, the fabrication of strong, heat-resistant, single-crystal parts is desirable in aircraft engines.

Laser powder bed fusion was previously able to produce only polycrystalline structures because the conventional laser melted powders with a large temperature gradient across the powder bed surface. When powder particles melt at a wide range of temperatures, crystals grow in various directions, making grain boundaries in their lattices and prone to developing defects during solidification. To resolve this issue, Dr. Tomonori Kitashima and his collaborators adopted a flat-top laser for melting a powder material at a uniform temperature across a powder bed surface area. Using this laser, the team actually succeeded in processing a nickel powder material into a single-crystal structure. This 3D printing technique is applicable to a wider range of materials, and it could potentially be used to manufacture most mechanical aircraft components in the future.

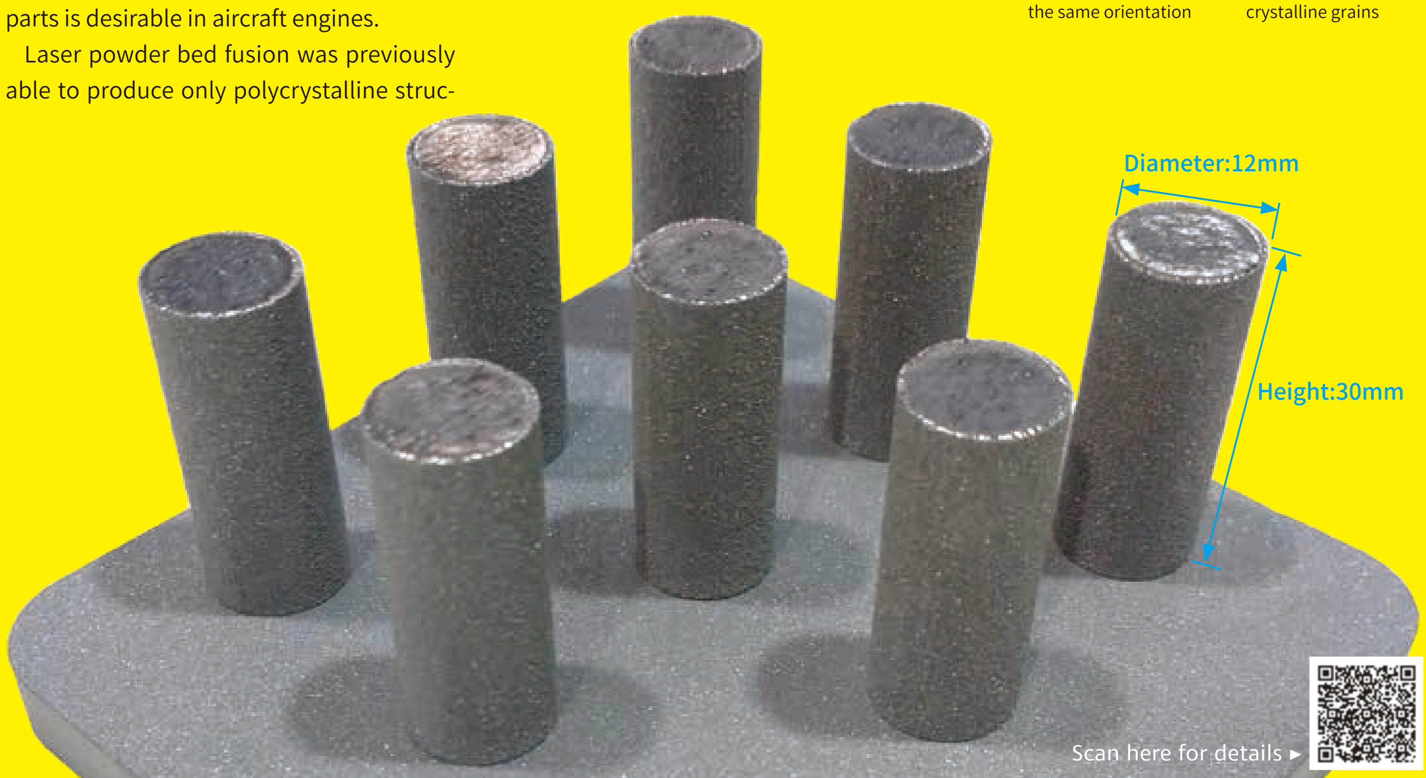
*Laser powder bed fusion: A 3D printing technique in which a laser beam is applied to a bed of a metallic powder raw material, causing it to melt and fuse together. After the layer of powder material solidifies, another layer of powders is fed on its top and irradiated with a laser beam again. This process is repeated until an intended shape is formed.



Kitashima and his co-workers developed a technique to apply a flat-top laser to cause a uniform melt region in laser powder bed fusion 3D printing. This technique enabled the heat to propagate planarly and shallowly at the surface of a metallic powder raw material, reducing strain in the melted material and allowing its crystals to grow in the same direction. As a result, the production of single-crystal structures has become feasible.



Grains with the same orientation | Less oriented crystalline grains



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NEWS!

30,000-ton press used to synthesize hexagonal boron nitride (h-BN) crystals

07 NIMS researchers with exceptional crystal synthesis skills win Clarivate Citation Laureate awards

Takashi Taniguchi
Fellow/Director
International Center for Materials Nanoarchitectonics (WPI-MANA)

Kenji Watanabe
Chief Researcher
Electroceramics Group
Electric and Electronic Materials Field
Research Center for Functional Materials

Hexagonal boron nitride (h-BN)—as transparent as ice—is a crystalline compound of boron and nitrogen. Only Drs. Takashi Taniguchi and Kenji Watanabe at NIMS are able to synthesize h-BN crystals of amazing purity. They are among the small number of researchers from various countries selected as Clarivate Citation Laureates in September 2022.

Every September, Clarivate Plc, a scientometrics company, honors researchers who have made significant contributions in their scientific fields. Twenty researchers from four countries received awards in 2022. Clarivate surveyed a total of about 55 million research publications from around the world and selected the winners based on the number of times their publications were cited. Taniguchi and Watanabe are among the winners of this extremely fierce competition.

The h-BN crystals created by Taniguchi and Watanabe have enabled breakthroughs in research on two-dimensional materials, including graphene. Graphene, which consists of a single layer of carbon atoms, is often referred to as a "dream material" because its electronic and thermal properties can be manipulated with a high degree of precision. However, accurately measuring its performance had been difficult because its extremely thin structure makes it very susceptible to the influence of the material with which it comes into contact. The individual layers of h-BN crystal Taniguchi and Watanabe fabricate serve as atomically flat insulators less susceptible to impurities. Their h-BN crystals have been found to be ideal substrates for graphene research because they do not interfere with graphene's performance, including the movement of its electrons.

The reputation of Taniguchi and Watanabe's h-BN crystals began to spread rapidly soon after the first recipient group of the crystals published their graphene research. Their h-BN crystals are now in very high demand among two-dimensional materials researchers around the globe. The total number of research papers in which Taniguchi and Watanabe are listed as coauthors now exceeds 1,700.

New discoveries have been made in many two-dimensional materials research projects in which Taniguchi and Watanabe's h-BN crystals have been used as substrates, including new quantum physical properties. The two NIMS researchers are now aiming to synthesize larger h-BN crystals with even fewer defects to enable even more amazing scientific discoveries.

Drs. Taniguchi and Watanabe's contributions recognized:
Fabrication of high-quality hexagonal boron nitride crystals which have enabled a revolution in research on the electronic behavior of two-dimensional materials

Virtually perfect h-BN crystal with a purity of 99.9999%

NIMS as a collaboration facilitator

Materials open platforms (MOPs): bringing rival companies together

In 2017, NIMS began organizing MOPs—frameworks for promoting basic research collaboration between competing private companies in the same industries. Two new MOPs have recently been launched in addition to the previously established chemical and pharmaceutical MOPs.

MOP

08 All Solid-State Battery MOP now in full operation

Scan here!! ▶

Participating companies JX Nippon Mining & Metals Corporation | JFE Steel Corporation | SUMITOMO CHEMICAL Co., Ltd. | TAIYO YUDEN CO., LTD. | DENSO CORPORATION | TOYOTA MOTOR CORPORATION | NGK SPARK PLUG CO., LTD. | Mitsui Mining & Smelting Co., Ltd. | Mitsubishi Chemical Corporation | Murata Manufacturing Co., Ltd.

The All Solid-State Battery MOP was formally launched by NIMS and 10 private companies to develop next-generation rechargeable batteries—with a focus on oxide all solid-state batteries*—on May 1, 2020. The COVID-19 pandemic severely restricted in-person interaction. Nevertheless, steady progress was made on preparing the MOP through such activities as lab equipment procurement and online workshops. As a result, a face-to-face research collaboration environment was organized and the MOP members have begun working together closely on June 7, 2022.

Some aspects of all solid-state battery R&D are hugely costly, including the development of technologies needed to control and analyze interfacial structures within rechargeable batteries and to identify new solid electrolyte materials. The common goal of the All Solid-State Battery MOP is therefore to collaboratively develop technologies that would be difficult for independent companies to achieve through individual efforts. NIMS will share its more than two decades of experience in developing all solid-state batteries and its sophisticated battery research-related equipment with the other MOP members in the hope of contributing to Japan's efforts to make society more energy-efficient and sustainable.

*Oxide all solid-state batteries: all solid-state rechargeable batteries are very safe due to their entirely solid composition. All solid-state batteries equipped with oxide electrolytes have many advantages over the sulfide all solid-state batteries already in practical use. For example, oxide all solid-state batteries are highly compatible with existing battery production processes and no toxic gas is emitted during their production.


Laboratory for All Solid-State Battery MOP

09 Launch of the magnet MOP

Scan here!! ▶

Participating companies TDK Corporation | Daido Steel Co., Ltd. | Shin-Etsu Chemical Co., Ltd. | Proterial, Ltd.*1

NIMS and four magnet manufacturers founded a magnet MOP on May 30, 2022. For the decade ending in FY2021, NIMS served as the Elements Strategy Initiative Center for Magnetic Materials (ESICMM) with support from MEXT and conducted basic theoretical research on heavy rare earth element-free magnets*2. In addition, NIMS developed lab instruments vital to the microstructural analysis of magnets and built materials databases and a network of researchers. These efforts made under the ESICMM framework have been passed on to the magnet MOP participants, who are now building infrastructure for materials design by updating the materials databases and developing data-driven techniques.

In addition, NIMS has been increasing its collaboration with university researchers via its cross-appointment system. NIMS is also running a membership system called the "Magnet Partnership" as a means of exchanging opinions with non-MOP member magnet-related companies. Moreover, NIMS has built a mechanism which helps it promptly identify foreseeable technical issues relevant to the magnet industry and properly incorporate them into MOP projects. NIMS has been making these multifaceted efforts to boost the international competitiveness of Japan's magnet industry.

*1 Proterial, Ltd.: Formerly Hitachi Metals, Ltd.
*2 Heavy rare earth element-free permanent magnets: Existing strong permanent magnets contain dysprosium or terbium—a heavy rare earth element—to increase their coercivity. Efforts are underway to develop magnets capable of retaining strong, persistent magnetic fields without adding heavy rare earth elements.


At the signing ceremony

10 NIMS, Yamaguchi City and Mori Urban Planning: promoting a wellbeing-conscious community

Scan here!! ▶

According to the World Health Organization (WHO), wellbeing is a contented state in which a person is physically, mentally and socially happy and healthy. On November 18, 2022, NIMS, the City of Yamaguchi and Mori Urban Planning Corporation concluded an agreement to conduct collaborative projects aiming to create new wellbeing industries. The three organizations will work together to create and support new healthcare and other industries using the materials and technologies developed by NIMS, thereby promoting the development of Yamaguchi City. As one of its first initiatives, the joint team plans to assess the usability of a wellbeing-related technology developed by NIMS.

The quick-response, high-sensitivity perspiration (or moisture) sensor developed by Dr. Jin Kawakita will be a main player in the first stage of this collaborative project. Previous research demonstrated that this sensor is able to effectively measure the rate of perspiration from the fingers. The research also found that measured perspiration rates can be used to estimate bodily hydration levels, indicating that the sensor may be useful in detecting signs of dehydration and heatstroke. To further improve the sensor's accuracy, additional perspiration rate data will be collected from people working out at a sport club in Yamaguchi City. This experiment will target a larger number of subjects across a wider age range than previous studies and perspiration rates and other measurements (e.g., body temperatures, weights, heartbeats) will be taken from them before and after their workouts. The correlation between these parameters will be statistically analyzed and the results will be used to develop effective dehydration and heatstroke prevention measures.


Amount of sweat being measured using the perspiration sensor

Researchers LIST


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Olfactory Sensors Group
Electric and Electronic Materials Field
Research Center for Functional Materials


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Takamasa Hirai
Researcher
Spin Caloritronics Group
Research Center for Magnetic and Spintronic Materials


03
Miharu Eguchi
Senior Researcher
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Research Center for Structural Materials


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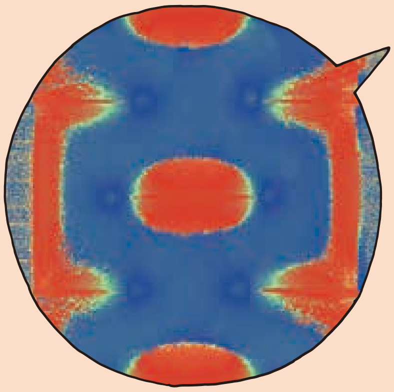

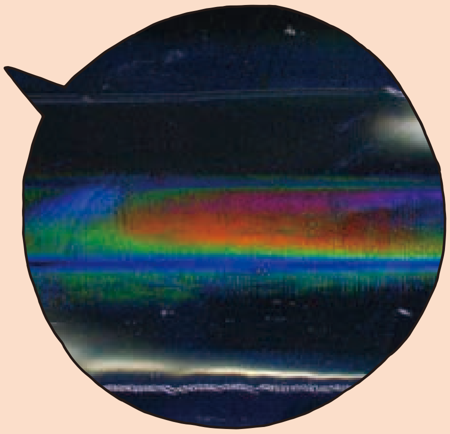

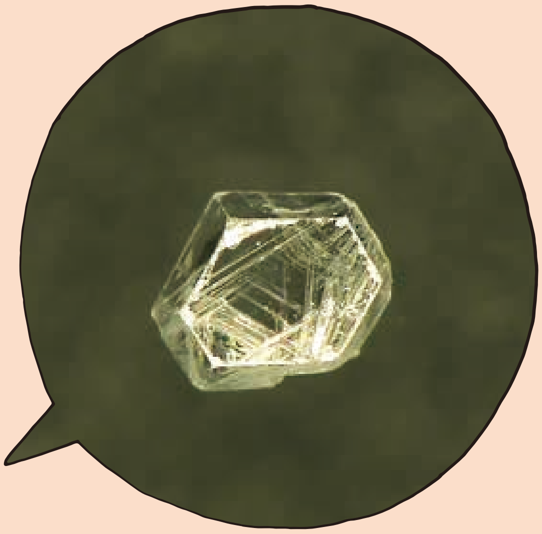

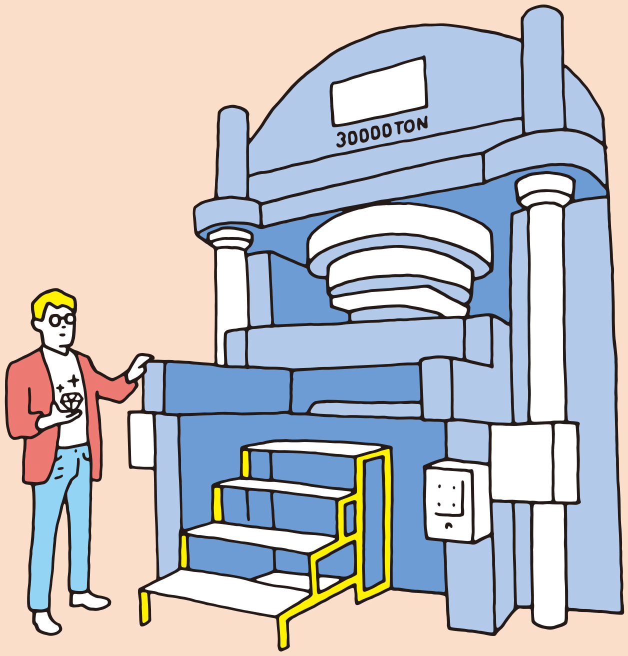

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