

**Research Center for Structural Materials** 

# Exploring the potential of materials

Multifaceted R&D on structural materials



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Structural materials form the basis of various structures and vehicles vital to daily life, including buildings, bridges, automobiles and aircraft. They must be tough, reliable and durable to ensure our safety.

Although many may think that the ubiquitous presence of structural materials would mean that they have already been studied in detail, their potential remains largely unexplored.

Recent technological advances in nanoscale structural observation have enabled materials researchers to discover that subtle structural differences can influence the performance of materials. In light of these findings, it is vital that processing techniques be developed capable of refining the nanoscale structures of the materials.

The Research Center for Structural Materials (RCSM) at NIMS develops, observes and evaluates materials. A group of experts from various fields, including computational science, has been working together at the RCSM to achieve a thorough and comprehensive understanding of the relationships between processing techniques and the microstructures and performance of materials. The RCSM explores the potential of structural materials to make society safer and more secure.



## Special Talk

## Structural materials supporting sustainable society

Structural materials must withstand weight, vibration and high temperature, and their failure directly endangers people's lives. Kohsaku Ushioda—a Senior Advisor at the Nippon Steel & Sumikin Research Institute Corporation who has been engaged in steel R&D for many years—and RCSM Director Koichi Tsuchiya—who has led structural materials research at NIMS—discussed the current status and future prospects of structural materials research.

## More than 90% of steel material potential remains unexploited

**Tsuchiya** When we refer to "structural materials," the most simple and understandable definition may be "any materials designed to withstand weight and support structures." Structural materials by this definition include materials such as reinforced concrete used in buildings, frames/panels for automobiles and aircrafts, and also, such medical devices as stents used to expand blood vessels.

**Ushioda** Most structural materials familiar to us are large and heavy. However, various other types of structural materials exist in the world and they are a vital component of society. They powerfully support social infrastructure in the background.

**Tsuchiya** From that perspective, the role of structural materials is immense. They are required to be strong, reliable and long-lasting as their failure endangers people's lives. Ushioda The demand for steel—which has been used as a structural material for a long time-to be stronger and to be given new properties is high. However, when steel is strengthened, it may become brittle or difficult to being joined together. Properties of structural materials must be acceptable from a comprehensive, not a partial, perspective. R&D efforts to strengthen steel without compromising other properties and performances have been continued. Although many people might think that everything about steel materials has already been studied, I suspect that less than 10% of the potential of steel materials has actually been exploited. From this viewpoint, steel remains a fascinating material with great potential.

## Key to full exploitation of materials' potential: microstructures

**Tsuchiya** A vital question then is: How can we fully exploit the potential of structural materials? I have come to the conclusion that materials' microstructures are the key factor. For example, the hardness of steels may vary even when their chemical compositions are identical. It was not until the optical microscope enabled us to observe microstructure Steel remains a fascinating material with great potential. —Kohsaku Ushioda

of the steels that we could identify the causes of the differences. Such observation is very important in terms of facilitating the development of materials.

**Ushioda** Recent advances in analytical instruments have enabled visualization of extremely fine microstructures in detail and elucidation of mechanisms by which some materials generate certain properties and performances. The current technological advancements represent only the beginning of our endeavor, however. To facilitate the development of materials, it is important to further increase the accuracy of "eye" functions which enable visualization of materials' microstructures.

**Tsuchiya** Ways by which raw materials are processed affect microstructures of resulting products. Microstructures determine the products' physical properties—such as toughness and strength—and performance. Structural materials R&D is essentially an effort to increase efficiency in various steps of materials development: optimization of processing techniques, microstructures, physical properties and performance. The late Professor Morris Cohen, an American metallurgist, proposed this link, and I am deeply impressed with his concept (see the diagram on p. 5).

There have been active research efforts in recent years to incorporate artificial intelligence (AI) and machine learning into materials development as has been the case at the SIP-MI Laboratory\*. In the near future, a system may be invented which is capable of recommending optimum processing techniques to be used to develop a material with specified desirable functions. However, if such a system is equipped only with input and output capabilities, it would be incapable of determining causes of problems, if any. So, we need to steadily engage in basic research to understand cause-and-effect relationships between processing techniques, microstructures, physical properties and performance, in addition to exploiting AI and machine learning. The RCSM, our research base, has adequate resources-materials experts and equipment-enabling us to take a comprehensive approach to investigating these relationships. These resources represent an advantage of the RCSM.

**Ushioda** The availability of researchers proficient in computational material science constitutes another great appeal of the RCSM.

**Tsuchiya** We also have scientists specialized in the first principle calculations—a relatively uncommon approach in the structural materials field. These specialists and empirical researchers worked together and made new discoveries concerning phase stability of titanium alloys and stability of precipitates in heat-resistant steel. I look forward to more scientific discoveries being made.

## Rivals work together in detailed basic research

**Ushioda** Materials manufacturers are required to develop new materials speedily to survive intensifying international compe-

tition. However, because reliability is the top priority for structural materials, it is vital for structural materials manufacturers to carefully develop materials based on in-depth basic research. From this perspective, I have high expectations for the Materials Open Platform (MOP), which was founded jointly between NIMS and three steel manufacturers in 2017.

**Tsuchiya** Current members of the MOP for the steel industry comprise Nippon Steel & Sumitomo Metal Corporation, JFE Steel Corporation and Kobe Steel, Ltd. Researchers of these organizations have been engaged in joint basic research focusing on the topic of "mechanical efficiency of grain boundaries."

Ushioda "Grain boundary segregation" is currently a hot research topic as well. Impurity elements and additive elements tend to aggregate in boundaries between crystals, which strongly affects properties of materials. Some material-related companies have introduced state-of-art equipment and are observing microstructures of materials and attempting to understand the relationships between grain boundary segregation and materials' properties. However, companies in the industrial sector are unable to perform certain types of basic research projects related to grain boundary segregation, including development of new techniques which enable accurate measurement of grain boundary segregation, determination of relationships between grain boundary segregation and grain boundary character and studies on basic issues related to grain boundary segregation and the strength of materials. They therefore have high expectations for the MOP to undertake these projects. The MOP framework allows NIMS and member companies in the same industrial sector to discuss and identify fundamental issues at the planning stage and carry out in-depth basic research to resolve these issues under the leadership of NIMS. This is a firstever initiative of this kind in Japan and I expect that it will strengthen Japan's international competitiveness in the materials field.

#### Skills, strategies and passion

**Ushioda** I hope that NIMS will continue to serve as a structural materials research hub for years to come. To this end, NIMS should regularly invite researchers from universities and the industrial sector, train them and send them back to their organizations. At NIMS, we respect research activities which are driven by curiosity. Collaboration between company researchers—whose research projects are usually driven by their organizations' interests—and NIMS researchers may produce groundbreaking research results. I hope that NIMS RCSM will lead structural materials research efforts across Japan as a national R&D institute.

**Tsuchiya** I totally agree with you. National R&D institutes and companies need to work together to build a framework for raising the level of the industrial sector and strengthening Japan's international competitiveness. At the individual level, what do you think each researcher can do to facilitate development and practical use of high-quality structural materials?

**Ushioda** Research proficiencies of individual researchers are important, but having sound strategies is of prime importance due to the continuous demand for changes in the types of structural materials in response to changing social needs.

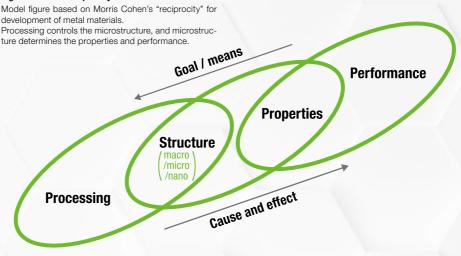
**Tsuchiya** The anticipated advent of the hydrogen society, for example, would change material needs. The current demand for materials which are used at high temperatures in thermal power plants and other facilities may be replaced by demand for materials that are durable at extremely low temperatures.

**Ushioda** Researchers should always have strategies to cope with social change, taking material needs in 5 to 10 years into account, given that development and popularization of materials take a long time.

Simultaneously, it is also important for researchers to be passionate and tenacious. Development of new materials requires new seeds from which they can be derived. However, it is very challenging. Hints for the new seeds may be hidden in unexpected places. I encourage materials researchers to interact and communicate with people specialized in other disciplines.

**Tsuchiya** I believe that interesting discoveries derive not only from knowledge but also from curiosity and sensitivity. I think that these sensibilities, in addition to intelligence, play an important role in R&D activities. From this perspective, communication with people in other disciplines is important. At the RCSM, all

#### Figure: Cohen's reciprocity



pieces of equipment related to structural materials research are located "under one roof," offering researchers with different backgrounds opportunities for active interaction. There is an example illustrating a positive effect of such interaction. I heard that people engaged in polymer materials R&D normally do not observe microstructures of materials. However, after they repeatedly heard metal researchers at the RCSM—such as myself—speaking of microstructures, they now take microstructures into account in their research. I expect that these new insights will lead to interesting discoveries.

However, I have some concerns: young people expressing interest in structural materials have been decreasing. I became a materials researcher because I was fascinated by the beauty of materials' microstructures and by the fact that microstructures and physical properties are closely related. I hope that many young people will discover the excitement of studying structural materials.

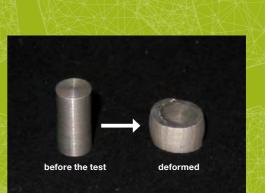
**Ushioda** Materials are indeed alive. Their microstructures sensitively change in response to various processing parameters such as deformation and heat treatment, which sometimes lead to drastic change in materials' properties. There are many aspects of structural materials that are yet to be explored through research and development, so there is a lot of potential in this field for researchers. I hope that many people will pursue career in this field and support society. I have high hopes that the RCSM will create an environment which will stimulate people's interest in the potential of structural materials. (by Shino Suzuki, PhotonCreate)

\*SIP-MI: One of the project in "innovative structural materials" category of "Cross-ministerial Strategic Innovation Promotion Program" led by the Cabinet Office. MI stands for "Materials Integration."

I hope that young people will discover the beauty of microstructures and the excitement of studying structural material —Koichi Tsuchiya

## Unbreakable under compression—— Accordion-like deformable Magnesium alloys

Interest is growing in the use of magnesium (Mg) alloys to further reduce the weight of airplanes and automobiles. Mg alloys now under development that are resistant to early failure when subjected to bending and compression offer great potential. Hidetoshi Somekawa has developed Mg alloys which deform like an accordion, thereby enabling them to have more than three times higher shock absorption properties that Mg alloys in general use can withstand.



Newly developed Mg-Bi alloy tube subjected to compression tests. These cylinderic shaped specimen were compressed by 60% like a folding bellows when pressure was applied at the rate of 0.8 mm/s for 6 seconds.

Expectations are strong for the development of high strength, lightweight materials to save energy and to help slow global warming. Replacing the use of aluminum (Al) alloys in vehicles—one of the most widely used material at present—with lighter and more abundant Mg alloys offers the possibility of reducing vehicle weights by approximately one third.

It is absolutely essential for materials used in infrastructure and automobiles, etc. to be safe and reliable. The role of these materials in ensuring public safety means that they have to be strong and resistant to breakage when subjected to pressure in bending and compression. Although the specific strength (strength per unit of weight) of conventional Mg alloys is high, their shock absorbing capacities are much lower than those of Al alloys. The biggest challenge in putting Mg alloys into practical use, therefore, has been their susceptibility to early failure when subjected to pressure in bending, compression, etc. Hidetoshi Somekawa succeeded in greatly increasing the shock absorbing capacities of Mg alloys after systematically and thoroughly investigating the addition of a variety of elements to Mg.

#### Enhancing the properties of materials by processing them into alloys

Many metallic materials are put into practical use through alloying processes: the addition of other elements to improve mechanical properties (eg, increased strength and/or toughness). In fact, current commercial Mg alloys contain small amounts of Al and zinc for greater strength.

"Previous research had resulted in insufficient understanding of the effects of additive elements on mechanical properties for Mg. Compared to Al alloys, Mg alloys are more difficult to process into complicated shapes and thus had failed to become a major subject of R&D efforts to develop practical alloys," Somekawa said. "However, interest in research on the use of Mg allovs as structural materials has been growing globally for about 20 years due to a steady increase in demand for energy conservation. I decided to study the effect of additive elements on mechanical properties for Mg alloys from a fundamental perspective with the goal of popularizing their use, which had been dismissed by researchers as a 'metallic material that is as brittle as glass.""

### Grain boundary sliding at room temperature: Mg alloys capable of deforming like an accordion

During alloying processes, metallic materials and additive elements are combined and heated until they melt. The liquid is then allowed to cool until it solidifies. The solid is then subjected to wrought processes, such as rolling and extrusion.

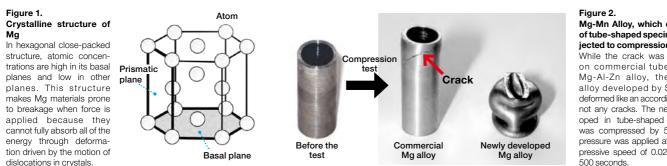
Most metallic materials are composed of nano- and micro-sized crystalline grains

with atomic-level gaps between them called grain boundaries. When these materials are wrought processed, additive elements are more prone to aggregation at these boundaries—a phenomenon called "grain boundary segregation." The type and distribution of aggregated elements are known to greatly affect the mechanical properties, such as strength and shock absorbing capacities of materials.

Somekawa systematically studied the effects of 29 elements (including 15 rare earth elements) which dissolve in Mg on the properties of the resulting alloys.

Somekawa paid particular attention to hexagonal close-packed structure in Mg (Figure 1). Unlike Al alloys and other metallic materials which deform irrespective of the types of forces applied (tension, compression, torsion etc), Mg alloys with hexagonal close-packed structure deform in a significantly different manner in relation to the type and direction of an applied force, making them easily breakable. Somekawa closely examined the atomic radii and electronic arrangements and couplings of elements added to Mg. As a result, he identified manganese (Mn) as a candidate additive element.

"I discovered that the shock absorbing capacities of Mn added Mg alloys increased while their strengths remained equivalent to those of conventional Mg alloys. They were capable of withstanding greater than 50% compression without any cracks by deform-



ing like an accordion (Figure 2)." Further investigation into the deformation mechanism revealed other interesting facts.

"Deformation of metallic materials at room temperature usually occurs by means of a plastic deformation mechanism called dislocation slip," Somekawa said. "However, I found that deformation mechanism of Mg-Mn alloys at room temperature was accompanied by a phenomenon called grain boundary sliding, enabling them to deform in response to both tensile and compression forces."

Deformation of polycrystalline materials occurs when grains slide against each other at their grain boundaries (grain boundary sliding). In usual, this phenomenon is likely to occur in metals at temperatures higher than half of their melting points.

"In theory, grain boundary sliding occurs at room temperature because the bonding states between "Mn-Mg" and "other additive elements-Mg" differ at the grain boundaries," Somekawa said. "I am currently investigating the mechanism in detail."

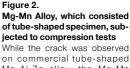
#### Search for new additive elements enabling rapid deformation

While adding Mn successfully increased the shock absorbing capacity of Mg alloys, issues still remain to be addressed. Mg-Mn alloys can exhibit good shock absorption property only when forces are applied slowly; they break when subjected to highspeed impacts (eg, when dropped from a significant height or subjected to a collision).

"A high propensity for grain boundary sliding at room temperature is an important factor in enabling rapid deformation of metallic materials," Somekawa said. "The ideal approach to achieving this point would be to induce grain boundary sliding while

inhibiting grain boundary segregation of added elements. Since the elements, which aggregate at grain boundaries, are fundamentally foreign objects, it would be ideal if we did not need to add them. However, the conversion of "pure" metals into alloys is a necessary means of strengthening them. I therefore searched for alternative additive elements to Mn which do not aggregate at the grain boundaries of the resulting alloys."

Somekawa focused his attention on bismuth (Bi) which, like Mn, can form a solid solution with Mg. However, much smaller amounts of Bi can be integrated into a solid solution with Mg at low temperatures than is the case with Mn. Using the same procedure used to produce Mg-Mn alloys, he succeeded in creating Mg-Bi alloys in which Bi segregation did not occur at the grain boundaries. Mg-Bi alloys exhibited increased grain boundary sliding in the absence of non-Mg elements at grain boundaries.



Ma-Al-Zn allov, the Ma-Mn alloy developed by Somekawa deformed like an accordion and did not any cracks. The newly developed in tube-shaped specimen was compressed by 50% when pressure was applied at the compressive speed of 0.02 mm/s for 500 seconds.

"I found that Mg-Bi alloys are resistant to early failure whether they deform slowly or rapidly," Somekawa said. "The shock absorbing capacity of the new alloy is five times greater than that of commercial Mg alloys and is equivalent to that of middle strength commercial Al alloys."

Somekawa hopes to further enhance the mechanical properties of Mg-Bi alloys until they are appropriate for use in wheelchair and bicycle frames and even automobiles. "Many enhancements need to be made before these alloys can be put into practical use, such as enabling them to absorb compression energy at more high speed regimes, enlarging producted materials and reducing production cost. I will resolve these issues one by one by revising the production process conditions and making other improvements," said Somekawa. (by Kumi Yamada)



## Hidetoshi Somekawa

Group Leader, Lightweight Metallic Materials Group, Research Center for Structural Materials

> H. Somekawa et al., Scientific Reports, vol. 8, 656, (2018), DOI.org/10.1038/s41598-017-19124-w H. Somekawa et al., Materials Transactions, vol. 58, pp.1089-1092 (2017), DOI:10.2320/matertrans.M2017108

## Multiscale microstructure characterization using a single electron microscope

To understand the mechanisms underlying the mechanical properties of metallic materials, microstructure characterization is required. Previously, microstructure characterization at different length scales required the use of multiple electron microscopes, namely, SEM and TEM. Ivan Gutierrez-Urrutia has developed a SEM technique that enables microstructure characterization at a wide range of length scales by the operation of a single electron microscope.

Metallic alloys are polycrystalline materials composed of large amount of crystals. Their mechanical properties, such as strength, ductility and toughness, are known to be controlled by dislocation gliding mechanisms and grain boundary structure. The detailed mechanisms of these phenomena, however, remain still unclear.

To understand these mechanisms, microscope observations are key in addition to theoretical calculations. At present, metallic materials are commonly characterized by optical microscopy, transmission electron microscopy (TEM) and scanning electron microscopy (SEM). The beam source type (light and electrons) and beam energy range (from eV to keV) determine the characteristic field of view and spatial resolution. Therefore, microstructure analysis at wide range of length scales requires the use of multiple microscopes, which is a challenging and time consuming task. To resolve this issue, Gutierrez-Urrutia has developed an electron microscopy technique that enables the observation at a wide range of length scales from millimeters to nanometers, by using a single SEM.

"The mechanical properties of metallic material are associated to several phenomena occurring at a wide range of length scales" Gutierrez-Urrutia said. "Unlike semiconductors and other functional materials, structural metals are heterogeneous polycrystalline materials within a large length scale range, from mm to nm. Microstructure heterogeneity has a strong influence on metal properties. For this reason, observation of a small representative area in a sample material is inadequate in understanding bulk material properties. At the same time, it is also important to closely analyze a small area in a sample at high resolution to fully determine the underlying mechanisms of structural properties. It is indeed necessary to investigate the relationships between phenomena occurring at the macroscale and those operating at the nanoscale. Thus, there is strong demand in



SEM-ECC image of dislocation pile-ups in a 316 L stainless steel

Materials Science for the development of a single electron microscope capable of quantitative microstructure characterization at a wide range of spatial scales—from micro to nano meter— within a sample material."

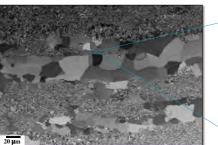
To resolve this issue, Gutierrez-Urrutia has carried out innovative research to add new functionality to SEM.

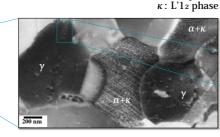
### From Max-Planck to NIMS: finally achieving a multiscale observation technique

The target material that Gutierrez-Urrutia uses as model material to show the functionality of the SEM approach developed is a multiphase Fe-Mn-Al-C Triplex steel (Figure 1).

The SEM technique developed by Gutierrez-Urrutia enables the observation of crystal defects such as dislocations in engineering complex materials as the multiphase triplex steel (Figure 1). The Fe-Mn-Al-C Triplex

Fe-Mn-AI-C Triplex steel





 $\gamma \colon fcc \ phase$ 

 $\alpha$ : bcc phase

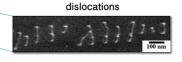


Figure 1. BSE (back scattered electron) - SEM image of the microstructure of a triplex steel composed of a complex mixture of ferrite phase ( $\alpha$ ), austenite phase ( $\gamma$ ) and carbide ( $\kappa$ ) precipitates. The technique developed by Gutierrez-Urrutia enables multiscale microstructure characterization ranging from the micrometer scale suitable for observation of the overall microstructure (image in middle) to the nanometer scale suitable for observation of dislocations (image at right).

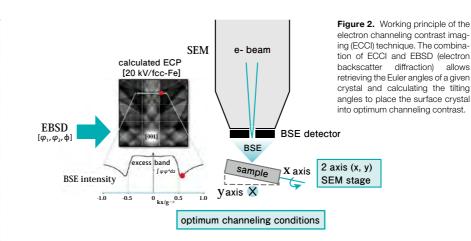
steel-a type of manganese steel-is a lightweight steel composed of a complex mixture of austenite phase (fcc-Fe), ferrite (bcc-Fe) phase and k-carbide precipitates. The characterization of the deformation structure of engineering structural materials at multiple length scales has been commonly performed by combined use of SEM and TEM. However, the use of the SEM technique developed by Gutierrez-Urrutia enables the quantitative characterization of deformation structures and even the analysis of individual dislocations. This SEM technique also offers several major advantages compering to conventional TEM approaches, namely, the efficient and simple sample preparation procedure (TEM requires the manufacturing of extremely thin foils by time consuming techniques), and the large analysis area (TEM observation is limited to the transparent sample area, typically about few µm<sup>2</sup>). How was Gutierrez-Urrutia able to achieve this innovative technique?

"The basic function of SEM is the probing of the sample material with a focused electron beam that is scanned across an imaging area and the subsequent detection of secondary and backscattered electrons (SE and BSE) emitted from the surface sample, thereby forming an image and allowing observation of the sample," Gutierrez-Urrutia said. "The resolution of SEM—about 1 -10 nm—is lower than that of the TEM and is insufficient for detailed crystalline structure observation. However, the wide field of view makes the SEM suitable for quantitative microstructure characterization."

"This advantageous characteristic of the SEM has been exploited by incorporating the electron backscatter diffraction (EBSD) technique for crystal defect imaging by the electron channeling contrast imaging (ECCI) method in the SEM".

The EBSD technique allows crystallographic orientation mapping by indexing the collected EBSD patterns by efficient algorithms. Accordingly, the EBSD technique is an excellent tool to retrieve the tilting angles required to place the sample surface into optimum electron channeling conditions and hence imaging crystal defects with optimum contrast.

"This integrated approach increases the functionality of SEM and enables the acquisition of images of crystal defects at a wide field



of view", Gutierrez-Urrutia said. "Before I joined NIMS, I worked at the Max-Planck Institute for Iron Research, where I conducted joint research with Dr. Stefan Zaefferer to combine the EBSD technique with the electron channeling contrast imaging (ECCI) method." They explored the feasibility of the EBSD technique in retrieving the Euler angles of a given crystal and calculating the tilting angles to place the surface crystal into optimum channeling contrast. For the first time, they developed an efficient SEM approach to obtain images of crystal defects with optimum contrast (Figure 2).

At NIMS, Gutierrez-Urrutia has carried out theoretical research and novel experiments on quantitative dislocation imaging to understand and optimize the imaging conditions for the ECCI method. As a result, he succeeded in developing a multiscale observation technique applicable to different metals such as aluminum and titanium alloys, steels and high-entropy alloys. The use of Gutierrez-Urrutia's technique has led to valuable new discoveries. For example, the characterization of dislocation configurations and nanotwins in lightweight steels and β-Ti alloys -frequently used in aircrafts and medical materials-has allowed the understanding of the underlying deformation mechanisms.

## Seizing active joint research opportunities at NIMS

Gutierrez-Urrutia began fully research and development of SEM techniques in 2008 at the Max-Planck Institute for Iron Research. NIMS—then in close communication with Max-Planck Institute—introduced an ECCI-capable SEM in spring 2013 for the characterization of structural materials. Gutierrez-Urrutia then joined NIMS the following year, in 2014.

"I joined NIMS because it conducts fundamental research on a variety of materials and actively engages joint research with various companies," Gutierrez-Urrutia said. "I have the ambition to develop high-performance SEMs equipped with ECCI functionality. I am pursuing collaboration with world-class electron microscope manufacturers in Japan. I am also attracted to the fact that NIMS has world-class material experts and databases containing experimental data collected over many years. I plan to analyze the correlation between microstructure and NIMS data, and identify underlying physical laws which enables the understanding of materials properties."

(by Kumi Yamada)



Ivan Gutierrez-Urrutia Senior Researcher, Corrosion Resistant Alloy Group, Research Center for Structural Materials

#### Investigating the properties

## Automated evaluation of fatigue properties: Investigating the microstructure of the metals from short crack

There had not been enough data of the formation and growth of short fatigue cracks because of the extremely time-consuming and labor-intensive process. To address this issue, Hideaki Nishikawa has developed an automated evaluation system enabling more than 200 samples to be evaluated for short fatigue cracks annually, and is now ready to study the relationship between fatigue properties and metal microstructures.



Hideaki Nishikawa

Fatigue Properties Group, Materials Reliability Field, Research Center for Structural Materials

## "Like searching for a ring in a soccer field"

Continuous exposure of metals to even relatively small forces (eg, the forces generated by the vibration of a pump, the rotation of an axle or thermal expansion/contraction) from nearby sources may result in eventual breakage. Such breakage has been the cause of serious accidents, including plane crashes and a sodium leak at the Monju Nuclear Power Plant. It is therefore important to evaluate the properties of metals to identify the locations at which short fatigue cracks—signs of breakage—form, and to determine how they propagate.

Nishikawa previously studied metal materials at a private company in connection with analyzing the durability of industrial plant equipment. "It is critical to understand the relationship between fatigue properties and metal microstructures in detail," Nishikawa said. "Safe use of newly developed metal materials cannot be ensured unless their fatigue properties are understood. It is therefore vital to collect data on the development of short fatigue cracks." However, it is difficult to collect this type of data, since we can't figure out where the crack will start. Nishikawa describes this challenge as "searching for a ring in a soccer field."

## Evaluation system utilizing the latest technologies

In the conventional method used to evaluate short fatigue crack growth, a metal test specimen is subjected to vibration for 5 minutes, a plastic material is pressed onto it to make a cast and the plastic surface of the cast is observed under a microscope. Since this evaluation method needs a repetitive, sequential process, Nishikawa has in the past worked through the night to complete it. Nishikawa recalled that the experience was like a training session which tested his patience and grit.

This evaluation method was not a practical means of collecting a sufficient amount of data. Nishikawa therefore decided to develop a faster, automated evaluation process by combining the latest technologies. He eventually succeeded in developing a system composed of many computer-controlled devices capable of automatically performing a series of processes. First, a force is applied to a test specimen a specified number of times. The entire surface of the specimen is then scanned for cracks using an optical microscope and a panoramic photograph of the surface, enabling all cracks to be detected. Many challenges were encountered during development, which Nishikawa was able to overcome using creative approaches. For example, the scanned images were initially out of focus, but he successfully improved autofocus accuracy (Figure 1) and created his own original system (shown on the cover).

Nishikawa also introduced a cutting-edge image analysis technique—digital image correlation analysis—capable of tracking slight surface changes. This technique enables him to observe the various strains present in metallographic structures and in the vicinity of short cracks.

Nishikawa has already made new discoveries using the automated system. He measured short fatigue cracks in carbon steel, a material in widespread industrial use. Once fatigue crack occurs, the crack widens and propagates as force is applied. The growth rates of such cracks had been known to vary widely. Studies have indicated that the relationship between metallographic structures and the ease with which cracks widen may be important in understanding the variability. Evaluation of nearly 100 samples enabled him to achieve these results.

"In future studies, I hope to construct a mathematical formula to summarize the relationship between fatigue and metal microstructures, thereby facilitating the design of safer equipment and the development of fatigue-resistant materials." (by Kaori Oishi)

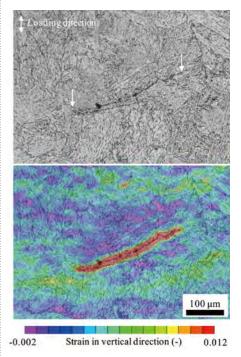


Figure 1. (Top) Short fatigue crack formed in a carbon steel microstructure. Arrows indicate two ends of the crack. (Bottom) Image produced by digital image correlation analysis. The strains around the crack have been visualized.

## Innovative long-term creep tests-Creep life prediction of 10 years in the future

Metal materials used in thermal power plants and petrochemical plants must withstand high temperatures and pressures for decades. In NIMS' creep tests, metal specimens are subjected to constant tensile loads at high temperatures for more than 10 years. Kota Sawada has been studying factors affecting the properties of materials and developing new types of creep tests to meet current industrial needs.



Group Leader, Materials Strength Standard and Technology Grou

## Why does the creep rupture life depend on the year of manufacture?

Although some metal materials are capable of withstanding high pressures at room temperatures, creep deformation can occur for them under a combination of high temperatures and high pressures. They may break after decades of use. Such breakage in an actual power plant may lead to a disastrous accident.

NIMS launched a Creep Data Sheet project in 1966, and have been evaluating the durability of metal materials at high temperatures since then. In creep tests, a tensile stress is continuously applied to metal material specimens at temperatures of 500 to 600°C for 100,000 hours (approximately 11.4 years), and physical changes are then measured. A total of 500 testing machines are currently running. Test results are publicized and used for the formulation of codes and standards for power plant design. Published test results have played a vital role in ensuring the safety of power plants and other structures.

Results of NIMS' creep tests recently revealed that the creep rupture lives of heat-resistant steels differed by a factor of two to three times depending on the year of manufacture despite identical material specifications. Consequently, Sawada and others launched a new joint research project with young researchers in the private sector to identify the causes of these creep rupture life differences, which are of great interest to power plant designers. Detailed electron microscopy analysis of boiler tube found that chromium (Cr) in steel segregates along longitudinal direction of the tubes. This phenomenon was identified as a contributing factor in creep rupture life differences. The material specifications of heat-resistant steels currently indicate Cr content but offer no further Cr-related information. In presentations given at scientific conferences, Sawada and his colleagues have proposed the modification of material specifications that would encourage the production of steels with reduced Cr segregation.

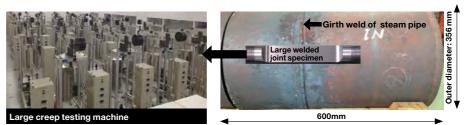
#### Enabling more realistic performance tests using large creep testing machines

Thermal power plants can generate electricity more efficiently by increasing steam temperature and pressure. To ensure the safe operation of thermal power plants under extreme conditions, it is very important to test the performance of steam pipes—the plant component most directly exposed to high temperatures and high pressures—over long periods of time under physical conditions similar to those of real power plants. Sawada felt that this type of test requires the use of large test specimens to complement conventional creep tests.

Sawada's group ultimately selected heat-resistant high-Cr steel used in thermal power plants as a target test specimen to be subjected to the new creep test, and began to construct a database for the assessment of remaining creep life. Test specimens were cut from a welded joint of steam pipe —a relatively weak portion of such piping—that are used in a thermal power plant. The specimens' dimensions were 360 mm in length, 50 mm in width and 25 mm in thickness; larger than specimens used in conventional creep tests. The group also introduced large creep testing machines (Figure 1).

Sawada's group will carry out this research program in collaboration with eight private companies, including power companies, plant manufacturers and material manufacturers. Creep tests will be temporarily stopped for non-destructive inspection every year. The inspection will be performed in a manner similar to periodic plant inspections conducted by the participating companies by adopting the same evaluation categories and methods they use. This methodology will allow collected data to be directly applicable to real power plants.

"Given that the upcoming creep tests will produce final results in approximately 10 years, I am now sounding out various companies in related industries to identify issues they anticipate facing in 10 years. The implementation of the creep tests at a scale as large as this is made possible by the efforts not only of researchers like myself, but also of the engineers who maintain and manage equipment on a daily basis. It is also important to ensure that the next generation continues our efforts." (by Kaori Oishi)

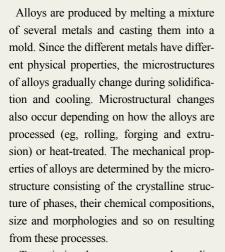


#### THE RESEARCH COLUMN

## Computational thermodynamics plays an important role in developing structural materials

Expediting alloy design using phase diagram calculations

The mechanical properties of structural materials depend greatly on their chemical compositions and the process and heat-treatment conditions. However, countless combinations of compositions and processing conditions exist. The current effort to optimize the conditions has been expedited by elaborate calculations using computers. We asked lkuo Ohnuma about advances in computational thermodynamics that are facilitating the development of structural materials.



To optimize these processes, phase diagrams—which depict the solid, liquid and gaseous phases of alloys in relation to temperature, pressure and alloy composition parameters—have been playing important roles for many decades. When researchers plan to develop a new material, they study phase diagrams to predict the final states of the individual metal elements constituting the alloy product for a given chemical composition and temperature condition. Ohnuma is currently engaged in computational thermodynamics research to facilitate the development of materials.

"Phase diagrams are created based on ther-

modynamic theories and data collected from numerous experiments," Ohnuma said. "The greater the number of metal elements involved, the more complex the phase diagram will be. However, advances in computer technologies have allowed us to perform more accurate and complex calculations of phase diagrams. In particular, recently developed software, including Thermo-Calc, has dramatically enhanced our ability to calculate phase diagrams. These software products—equipped with robust databases to reinforce their calculations and many calculation functions—are capable of estimating the parameters of alloys consisting of as many as 10 or more elements."

"Empirical phase diagrams represent equilibrium states: stable crystalline structures and compositions. However, the strength of many alloys maximizes before they reach equilibrium states. The use of computational simulations enables us to estimate optimum temperatures and thermal treatment durations for producing metastable, non-equilibrium alloys with desirable properties."

In addition, the DICTRA software and phase-field methods have become available for use in simulating the diffusion patterns of individual metal elements and phase transformations and microstructural evolutions in materials. These tools have given computational science an indispensable role in materials development.

## Formation mechanism of LPSO in magnesium alloy identified

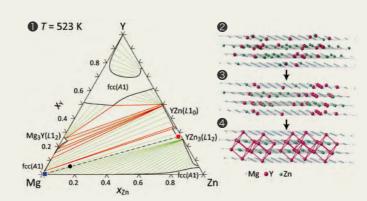


**Ikuo Ohnuma** Group Leader, Computational Structural Materials Design Group, Research Center for Structural Materials

Ohnuma is currently focusing on magnesium (Mg) alloy research. Several years ago, the University of Tokyo Professor Eiji Abe analyzed Mg alloy microstructures using an electron microscope and discovered a new crystalline structure-called a long-period stacking order (LPSO) structure-which strengthens Mg alloys. However, the mechanisms by which the LPSO structure forms in Mg alloys (containing zinc (Zn), yttrium (Y) and other additive elements) had remained unknown. To understand these mechanisms. Ohnuma and Professor Abe's graduate students jointly simulated LPSO structure formation processes based on the computational thermodynamics. As a result, they found that among the three alloy components (Mg, Zn and Y), the interactions between Zn and Y had a particularly significant influence on LPSO structure formation.

"In future studies, I would like to improve the accuracy of the thermodynamic database—which is indispensable in the calculation of phase diagrams—and thereby understand LPSO structure formation mechanisms in greater detail," Ohnuma said. "I hope that these efforts will expedite the practical use of Mg alloys."

(by Kumi Yamada)



(Left) Metastable calcu-Figure. lated phase diagram of FCC phases in the Mg-Zn-Y ternary system aided by the first principle calculations (1). (Right) Schematic diagram showing formation processes of LPSO structure proposed by computational thermodynamics. Zn and Y segregate to stacking faults in FCC structure within  $\alpha$ -Mg (HCP) phase (2). Zn and Y then separate into an Mo phase and a (Zn, Y) phase in the stacking fault planes (3). The (Zn, Y) phase then forms a Zn<sub>6</sub>Y<sub>8</sub>-L1<sub>2</sub> cluster (4).

## INNOVATION THROUGH COLLABORATION IN COMPUTATIONAL THERMODYNAMICS

Special Tripartite Talk: NIMS Award 2017

#### Prof. John Ägren (Royal Institute of Technology, Sweden, Professor)

Holds a PhD from the Royal Institute of Technology (KTH) in Stockholm, Sweden, 1981, and was appointed full professor in physical metallurgy at the same university in 1991. He is one of the developers of Thermo-Calc and DICTRA software, and is the chairman of the directory board since 2001. DICTRA has greatly contributed to the expansion of the practical applicable range of computational thermodynamics from the field of statics to that of dynamics.

#### Prof. Bo Sundman (Royal Institute of Technology, Sweden, Professor Emeritus)

Extes

Professor emeritus of Royal Institute of Technology (KTH). Received his PhD from KTH in physical metallurgy in 1981. From 2006 to 2009 he was at CIRIMAT at Paul Sabatier University in Toulouse and from 2009 at INSTN, CEA Saclay. He was a key developer of the thermodynamic calculation software, Thermo-Calc. He retired from KTH in 2012 and has since worked at INSTN, CEA Saclay and as distinguished visiting professor at the Central South University in Changsha, China.

#### Dr. Kiyohito Ishida (Tohoku University, Professor Emeritus)

Received his PhD from Tohoku University. He worked at Daido Steel for eight years, and then became Assistant Professor at Tohoku University, where he is currently Professor Emeritus. As a pioneer of alloy design and new material development utilizing phase diagrams and computational thermodynamics, he has highlighted the importance of the CALPHAD method and simulations, both within and outside of Japan. He has successfully developed several new alloys such as Co-based super heat-resistant alloys.

The NIMS Award, given annually to the researchers who have made a breakthrough in materials science, went to Prof. John Ågren and Prof. Bo Sundman, both from Royal Institute of Technology (KTH), Sweden, and Prof. Kiyohito Ishida, from Toho-ku University, Japan, in 2017. The three awardees have advanced the field of computational thermodynamics and developed thermodynamic calculation software, both of which are fundamental in structural material design. These developments, as well as the results of their studies on alloy design and its practical applications, play the role of a chart and compass to advance technologies for materials development and the study of materials behavior under various usage environments.

We asked the three professors to retell the story behind their achievement.

#### —First of all, congratulations on your winning the NIMS Award.

**Ishida:** It is a great honor and a real pleasure to receive the NIMS Award. More than 10 years ago, I was a guest researcher at NIMS, and engaged in collaboration work with researchers for a long time. I'm also very happy to have received the award with Prof. Ågren and Prof. Sundman, because as a basic and applied researcher on alloy design and the structure control of materials, based on phase diagrams and microstructure of alloys, I really know that their development of Thermo-Calc and DICTRA had tremendous influence on the development of structural materials.

**Ågren:** Thank you. I'm very grateful to receive the award. Looking at the previous recipients, I am extremely honored to be included among them. I'm very happy about it.

Sundman: Likewise, I'm very honored. I know many of the colleagues who work at NIMS —I have collaborated with many of them, so it is nice to have the opportunity to

see them and to work together again.

—All of you were recognized by the award for advancing the field of computational thermodynamics. One of your achievements was in developing the thermodynamic calculation software "Thermo-Calc." How did you get involved with this project?

**Sundman:** As you know, our colleague (the late Dr.) Bo Jansson, John (Ågren) and I were at first three individual researchers working on different projects, but we found



that we had some things in common-we all needed to do thermodynamic calculations. We decided that each of us could concentrate on one of three parts: thermodynamics, equilibrium calculations, and simulation. So instead of having three separate projects that were likely to die off once we left KTH, we decided to create a homogeneous software that could do many different things. This created interest from other people and, eventually, it became commercially viable. Ågren: Our older colleagues at KTH wereat the time-very sceptical, and said things like: "That really could never work. You have to write small programs for every problem you have-not big programs." But we solved that by dividing big problems into small parts, which were thrown in and out of the computer. It was also good that our supervisor Mats Hillert-who was busy and had little time and yet he was excellentreally took care of us. We didn't ask him if we could collaborate, but then when he saw that we were already working together and that we had created something, he was very happy. Part of the reason for this was that he had created a very nice atmosphere in the lab, and that made it possible for such collaboration to occur.

**Sundman:** But actually, we all left KTH to conduct research elsewhere. It is difficult to

keep the group together for a long time at university level. That was one challenge we faced. In a sense, it is easier to keep a group together if you're all in a company, but then, being in a company also has its challenges.

—And Prof. Ishida, you have developed a number of new alloy materials that are now being applied in industry. You also conducted the thermodynamic analysis for each multicomponent alloy, and developed thermodynamic databases of alloy phase diagrams, which are used widely for materials development. How did you become involved with Thermo-Calc in your research?

**Ishida:** My involvement with Thermo-Calc goes back a number of years. In 1987, the American Society for Metals (which subsequently became ASM International) asked the Japan Institute of Metals and Materials to collaborate on the compilation of Phase Diagrams. It was a big project of some million dollars. There were many category editors assigned for the alloy Phase Diagrams, such as iron-based ones. Professor Taiji Nishizawa and I were to be cobalt category editors. But as at the time there was no committee in Japan to oversee this, Nishizawa and I discussed creating a special committee here for Phase Diagrams.

Eventually, members of the committee came not just from universities but also industry. We asked companies to collaborate with—and support—this project. At that time, Thermo-Calc software had just been created—and it was well-known. When I visited companies, I let them know just how useful Thermo-Calc software was, especially for understanding microstructures. I was acting like a "salesman"—so to speak—for the software. But our main focus was to ask researchers from industry to join, because the opinions from industry were very important.

So I explained the importance of Thermo-Calc software. When I was a student, for example, we could calculate binary phase diagrams, while ternary phase diagrams were difficult and higher order systems were impossible. But real materials like commercial alloys contain many alloying elements, which could be handled by Thermo-Calc software. Fortunately, many Japanese companies took up the software, and were able to apply it to various problems.

**Sundman:** Actually, Japan was the biggest customer for a while.

**Ishida:** Oh, then I must be proud of my contribution to that (laughs).

**Ågren:** It is now used in 69 countries, and if you look into scientific literature there are large number of citations.

### —And this development of the Thermo-Calc led to the development of the DICTRA software, Prof. Ågren?

**Ågren:** The basic idea was to combine thermodynamics and kinetics and to use thermodynamics to extract the driving forces for those reactions. Somehow I felt that if you could only do this, then you could predict a lot of things. And, of course, this was something that you could read a lot about in textbooks, but nobody had really done it. Our supervisor Mats Hillert had had that vision for a long time before me. But at that time they didn't have computers or databases, so it was not possible to do anything (such as to solve equations), except to write down large numbers of equations.

So we developed such code, and I was astonished when we at first applied it to an industrial problem—a joint between two different materials. It worked so well. It was good enough to guide the company we worked with to develop a better joint than they had before.

That success demonstrated three things to me. One was how powerful this technique probably is. The second was how important it is to have industrial collaboration so that you can see real problems—which often are much more complex than those that you may construct yourself. And, the third was to feel the satisfaction that something you had created is helping someone.

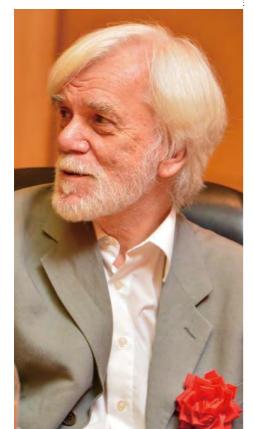
—So, the development of Thermo-Calc and DICTRA was a big revolution in designing materials, and a number of useful materials have been created using them. Now, what are your most recent research interests?

**Sundman:** After working with the software for 30 years, you realize that you may have to rewrite it. So I've been working on Open-



Calphad, a free thermodynamics software. I think it is very useful for academia to have free software that you can compile on whatever machine you happen to be using. You can, for instance, study the algorithms and make improvements, something that is engaging for students. With OpenCalphad, students can access the code and implement new models or develop new ideas.

Ågren: Over the last 10 years or so I have been more involved in what we call Phase Field Modelling, which is nice technology because you can add the effects of stresses, plastic deformation, and other physical effects as well. In addition to that, I have been very interested in high-temperature carburization, and that involves describing thermodynamics and diffusion in rather complex phases. And, finally, for half a year now, I've been working on "Thermodynamics and Kinetics for Beginners." So it will be possible for people to pose the right questions and extract useful answers without knowing all those details, because the general ideas are very simple, but they are hidden in all the equations in textbooks. Ishida: These days, considering the environmental problems that we have today, I again came to think it is crucial to have mutual communication between academia and industry, to know real problems, and to use that knowledge for developing new materials. Kotaro Honda, a famous scientist



in Japan who invented hard magnets in the 1930s or so, had two doctrines. The first is that, in university, research work is the most important activity. The second is the application of science. It was important for him that we not only write papers, but also give birth to new materials for the benefit of society. I agree with Professor Honda's doctrines.

#### 

**Ågren:** I think academia-industry collaborations will be very important because it will teach you, from an academic pointof-view, what the real problems are—and they may be as fundamental as other kinds of problems. This may guide your research to something that is strategically important—not just for companies but also for the future of academia as a whole. This means you won't spend all your efforts on things which are not meaningful.

**Ishida:** I'm afraid that the government tends only to be interested in very popular topics—such a nano technology, superconducting oxides, and so on. But our field—studying phase diagrams or microstructure evolution, for example—is based on fundamental science. In our case, it is very difficult to get research funds. Furthermore, students are often not interested in such fundamental research, and that can be a problem—even in Japan.

**Sundman:** Research funding problems differ country to country. While I'm not directly involved in the funding of universities in Sweden, I know that funding has been cut year-on-year. Although some of that money goes back to research, it has subsequently been allocated to three-year projects. So instead of having long-term funding, which the professor could decide himself, we now have a situation where he has to apply for funding. And, of course, decisions about which large-scale projects to fund are now made by politicians.

Ågren: That is another rather big change happening in Sweden. But I think it is a world-wide trend. Small projects which involve just one person trying to do something new do not get funded anymore. The



trend favors big collaborations with many researchers and industrial partners. This means it is a lot of work to put together such a constellation of stakeholders. And, once you have the funding, there is a lot of management to be done in order to develop the project. That said, most of these developments are very good—students don't feel that they are working in isolation, for instance, and can discuss problems in teams. By contrast, when we started our work, students were often working on their own individual projects, and the fact that we collaborated was because we happened to be sharing the same room.

**Sundman:** It really depends on government and industry funding. What they should ensure is that they recruit good people to work in this field—that is important. I would add that one should ensure good teaching on thermodynamics—even at undergraduate level.

**Ishida:** Yes, I agree with that. Thermo-Calc software is very important, but, in the background, the most important thing is thermodynamics. Since thermodynamics is an integrated physics consisting of many fields of science, I hope the educational environment for thermodynamics gets better in Japan, too.

(Interview: John Amari)

No. 2

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# Science is even more amazing than you think

# Can LEDs solve the food crisis?

**Text by Akio Etori** Illustration by Joe Okada (vision track)

When world population began to grow explosively in the 1960s, food shortages became a serious concern. Like other countries, Japan took the issue seriously and discussed various measures to address it. One idea was to use petroleum proteins: proteins produced from petroleum by fermentation of microorganisms. These proteins would then be extracted and used as food.

Research was conducted to put this concept into practice and edible petroleum proteins were in fact produced. There was a major problem, however: researchers were unable to adequately remove benzopyrene—a potential carcinogen—from the petroleum proteins. The high toxicity of the petroleum proteins led to the abandonment of the concept despite considerable efforts having been made.

Although the project failed, food production dramatically increased and the crisis was overcome through crop cultivar improvement and the use of chemical fertilizers, known as the Green Revolution.

The world population has continued to grow steadily. According to the United Nations, the global population is expected to reach 9.8 billion by 2050, which translates to an increase of approximately 2 billion people over the next 30 years. If the human population continues to grow as projected, the risk of a food crisis will increase.

One way of coping with this issue is to adopt a new crop cultivation method called "speed breeding," which enables agricultural plants to grow several times faster than conventional cultivation. In January 2018, researchers at the John Innes Centre (a plant research institute) in the UK and at the University of Sydney and the University of Queensland in Australia published a method of using light-emitting diodes (LEDs) to facilitate photosynthesis in plants. The method is currently attracting a great deal of attention.

It is widely known that plants grow through photosynthesis. Photosynthesizing plants absorb a large amount of blue and red light while reflecting green light. As such, plant growth is greatly influenced by the wavelengths and duration of the light to which they are exposed. Plant cultivation methods in which plant growth is artificially promoted using sodium vapor lamps—efficient light emitters—are already in practical use. However, this type of lighting emits relatively little blue and red light and is costly as it generates a large amount of heat. By contrast, LEDs offer several advantages: they can emit light of specific wavelengths, they generate only a small amount of heat and they are very energy-efficient.

A joint research team recently developed specially adjusted LEDs capable of emitting high intensity far-red light, and conducted long-term experiments in which agricultural plants were grown under 22 hours of LED light exposure per day. The team was able to grow six generations of wheat, barley, pea (Pisum sativum) and chickpea plants and four generations of canola plants in a single year, significantly increasing the number of times these crops can be harvested when compared to conventional farming practices. In addition, the yield of wheat grown under exposure to LEDs was six times greater than that of wheat grown in farmland and three times greater than that of wheat grown under exposure to sodium vapor lamps.

Continuous cultivation of the same crop within a short period of time often reduces the size of fruits and the number of seeds plants bear. However, that was not the case with the plants subjected to the speed breeding method employed in this research project.

Expedited plant growth may ease food shortages and accelerate a variety of plant-related research. The ability to observe many plant generations within a short period of time would offer scientists opportunities to advance their understanding of crop plant genetics and would be useful in studying plant characteristics, such as the morphologies and flowering periods of various plant species. Some researchers even believe that a combination of the speed breeding method and genome editing techniques, such as those using CRISPR (clustered regularly interspaced short palindromic repeats), will lead to new scientific discoveries. The speed breeding method may provide a way of finding highly effective food crisis response measures.

The global population and food demand will continue to grow. Will humankind be able to overcome these challenges in time by leveraging emerging technologies?

Akio Etori: Born in 1934. Science journalist. After graduating from College of Arts and Sciences, the University of Tokyo, he produced mainly science programs as a television producer and director at Nihon Educational Television (current TV Asahi) and TV Tokyo, after which he became the editor in chief of the science magazine Nikkei Science. Successively he held posts including director of Nikkei Science Inc., executive director of Mita Press Inc., visiting professor of the Research Center for Advanced Science and Technology, the University of Tokyo, and director of the Japan Science Foundation.



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