


NIMSNOW

NATIONAL INSTITUTE FOR MATERIALS SCIENCE

INTERNATIONAL

No. 1
2016



The **NMR**
With the World's Highest Magnetic Field

The NMR

With the World's Highest Magnetic Field

On March 11, 2011, the project team was about to complete the development of an NMR system equipped with world's highest magnetic field.

The goal of exceeding the unprecedented 1,000-MHz magnetic field was just within their reach.

Right at that moment, the menace of nature suddenly devastated years of their work.

After a long while, the project team somehow managed to repair the severely damaged facility and regained the environment for resuming the project.

Then, the team leader who had been giving all he had to the project, fell victim to a fatal illness.

But what was so significant about the 1,000-MHz hurdle that the team overcame one daunting hardship after another to clear it?

What is the NMR expected to bring to future generations?

In this issue, we approach the story behind achieving the world's highest magnetic field based on the testimony of the determined project team members, and feature the history of NMR development.



Special Talk

The Challenge of NMR Beyond

Hideaki Maeda

Director of NMR Facility,
Division of Structural and Synthetic Biology,
Center for Life Science Technologies, RIKEN

1 GHz

Tadashi Shimizu

Director of the High Magnetic Field Station,
Research Network and Facility
Services Division, NIMS

Nuclear magnetic resonance (NMR) systems are essential in a broad range of research such as structural biology, analytical chemistry, and materials science. There has been fierce competition to generate higher magnetic fields for the purpose of realizing high-precision measurements. In 2014, a joint research team including NIMS and RIKEN succeeded in developing an NMR system capable of generating 1,020 MHz (24.0 T)*, the strongest magnetic field in the world. The two project leaders will talk about the journey toward achieving the world's strongest magnetic field and a vision for the future.

* With NMR, the strength of the magnetic field is expressed in Hz. T=Tesla

Radar research gave birth to the NMR system

Shimizu: Our journey to develop the NMR system equipped with the world's strongest magnetic field of 1,020 MHz was quite long and difficult. But first, let's look back on the history of NMR systems.

Maeda: It is not commonly known that the NMR system was created as a by-product of the radar research conducted during the Second World War. Radar, NMR, and cell phones all work by the same mechanism: sending a signal and receiving the returned signal.

Shimizu: Purcell and Bloch were among the scientists who carried out radar research for military purposes in the United States during the war. After the war ended, they returned to universities, and both of them independently discovered the phenomenon called nuclear magnetic resonance (NMR), whereby the nucleus in a magnetic field interacts with an electromagnetic wave of a specific frequency. Then, Varian, who was a student under Bloch at Stanford University, founded Varian Associates in 1947, where he started manufacturing and selling NMR systems. NMR systems started to draw attention in 1950 when it was discovered that molecular structure could be investigated using the system.

Maeda: In 1956, a Japanese company named Japan Electron Optics Laboratory Co. Ltd. (now JEOL RESONANCE; hereinafter referred to simply as JEOL), started manufacturing and selling NMR systems. After about 10 years, Bruker of Germany also entered the market. So Japan was the second country to start manufacturing and selling NMR systems, but Bruker presently has the largest share in the market.

A breakthrough in making superconducting wire

Shimizu: The structure of NMR systems has not changed much so far. The biggest change has been in magnetic-field strength.

Maeda: That's right. With NMR systems, the stronger the magnetic field in which a sample is placed, the cleaner the returned signal will be. During the history of NMR system development, most efforts were spent on generating a more powerful magnetic field. Until the 1970s, the magnetic field had been generated by applying an electric current to

an electromagnet with an iron core wrapped with copper wire. But this magnet did not generate a magnetic field of even 200 MHz. While it was known that a superconducting magnet using a superconductor would create a strong magnetic field, no adequate technology was available to make superconducting wire. The great breakthrough came from Dr. Kyoji Tachikawa of National Research Institute for Metals (NRI; current NIMS). He succeeded in creating superconducting wire which can realize generating a magnetic field higher than 500MHz. Until his development, we used superconductive materials made of niobium titanium (NbTi), and it could generate magnetic field of only 400MHz.*

*Please refer to page 10 for more detail.

"You should not even try it"

Maeda: In the 1990s, the global goal was to exceed 900 MHz. Amid the fierce competition, Dr. Tsukasa Kiyoshi, Dr. Shimizu and other NIMS researchers managed to generate a magnetic field of 920 MHz in 2001, and 930 MHz in 2004. That was brilliant.

Shimizu: These efforts were a part of the "Multi-Core Research Project on Superconductivity" sponsored by the Science and Technology Agency, started in 1988.

Maeda: After these successes, Dr. Kiyoshi and I decided to apply for the program called "Development of Systems and Technology for Advanced Measurement and Analysis," sponsored by the Japan Science and Technology Agency (JST). We submitted our research proposal aimed at generating a magnetic field of 950 MHz. But our proposal was rejected. Then, we submitted another proposal to generate 970 MHz, but it was rejected again. To be accepted, we thought that our proposal needs to incorporate more technical innovation. Consequently, we decided to use a bismuth-based cuprate high-temperature superconductor, which was discovered by Dr. Hiroshi Maeda of NIMS in 1988, and set a goal of exceeding the unprecedented 1 GHz. Third time was the charm for us. Our project, titled "Development of Over-1 GHz NMR Systems" was finally accepted and started in 2008.

Shimizu: However, the cuprate high-temperature superconductor was ceramic, which breaks easily under pressure. That material was far more difficult to handle than NbTi or Triniobium-tin (Nb₃Sn) in creating

thin and long wire and winding it to create a coil. For this reason, we could not realize a high-temperature superconducting magnet during "Multi-Core Research Project on Superconductivity."

Maeda: Using a high-temperature superconducting magnet, we can theoretically create a magnetic field of up to 1.5 GHz. However, when I talked about this magnet at the meetings on NMR, I was often viciously criticized and received such responses as "It won't work. You should not even try it." The magnetic field of NMR systems must be very stable. Honestly, I did not have confidence in achieving a stable magnetic field using a high-temperature superconducting magnet.

Shimizu: It is great that you succeeded in this endeavor. These days, we receive recognition when we have succeeded in achieving something as planned. However, if we could achieve something as planned, then that means the original plan was not very ambitious. Achieving something that seems to be

After exceeding 1 GHz, a new world emerged. We just took the first step into that world.

Hideaki Maeda



impossible is truly great.

Maeda: Since I started working on the R&D of superconducting magnets at Toshiba, my lifework has been the creation of systems that employ the world's strongest magnetic field. At that time, the cutting-edge superconducting technology was high-temperature superconducting magnets. So I wanted to take on that challenge.

Finally exceeded 1 GHz

Shimizu: The "Development of Over-1 GHz NMR Systems" project was conducted by the most talented researchers from NIMS, RIKEN, Kobe Steel, and JEOL. NIMS was responsible for basic design and operation of the superconducting magnet, and Kobe Steel was in charge of the development of a technique to wind the superconducting wire, and created the superconducting magnet. The superconducting wire was developed by Sumitomo Electric Industries. JEOL was responsible for the development and manufacturing of the NMR system, spectroscope, and probe, while RIKEN took charge of developing systems to improve the quality of the power supply and magnetic field, and measuring proteins.

Maeda: When we finally achieved the level of magnetic-field strength, we were filled with emotion. However, we were quite nervous until that goal was actually achieved.

Shimizu: Yes. We slowly raised the strength of the magnetic field day by day over a month. After we reached 1,000 MHz, I was afraid every day that this might be the last time we improve the record. So I took a photo of the record display every day. And on October 17, 2014, we achieved 1,020 MHz at last.

Maeda: We also analyzed the three-dimensional protein structure. We confirmed that the system had much higher sensitivity and resolution than the 700 MHz NMR system. It is important to make sure that the new NMR system indeed fulfills its intended functions in addition to the fact that it generated the world's strongest magnetic field. Only after making this sort of effort, might we deserve recognition from the life science community.

Shimizu: More than 20 years have passed since the launch of the "Multi-Core Research Project on Superconductivity." We finally achieved our goal of generating a magnetic field of more than 1 GHz. This time, we modified a 920 MHz NMR system by replacing a part of its superconducting magnets

with bismuth-based cuprate high-temperature superconducting magnets, as well as adding Nb₃Sn and NbTi superconducting magnets for combined use. If we add more high-temperature superconducting magnets to the system, we can generate an even stronger magnetic field.

Maeda: When I presented these results at a scientific meeting, I showed a slide depicting a footprint on the moon left by Captain Armstrong during the Apollo mission, and said, "That's one small step for a man, but one giant leap for the future." The humor was well received. Since we have proved that a magnetic field greater than 1 GHz can be achieved using high-temperature superconducting magnets, we are now ready to optimize the system.

Shimizu: Now the global goal is to generate a magnetic field of 1.5 GHz. If we achieve that goal, it can be used for the analysis of inorganic materials, which is still impossible today.

Maeda: The magnetic field of 1.5 GHz is quite appealing from the viewpoint of protein analysis. Research in this field is expected to become more efficient due to not only improved sensitivity and resolution of the analysis but also drastically reduced measuring time.

After overcoming many hardships

Shimizu: Looking back on this project, the most serious events we encountered were the Great East Japan Earthquake and the passing of Dr. Kiyoshi, the leader of the project. The earthquake struck right after the NMR system had been assembled. Many of the critical parts were damaged by the disaster. Moreover, Dr. Kiyoshi passed away a year after the earthquake, and I took over his role as leader thereafter. Then, we were told that even if superficial damage was repaired, a full recovery of the system to generate the desired magnetic field could not be guaranteed. But as we had come a long way in this project, and we were so close to completion, we were totally determined to achieve our goal, which could be viewed as a compilation of all of the efforts having been made in superconductivity research across Japan. So, I formed a special team comprising of 13 members and recovered the system in two years. I was really happy when we generated the record-breaking magnetic field. What are your thoughts as you



Japan's superconductivity research is at the top level in global terms. What we need now is to draw up sound strategies.

Tadashi Shimizu

look back on the project now?

Maeda: Despite the fact that our application to the Advanced Measurement Analysis program was rejected twice, we finally delivered the result, which was a great accomplishment. But we cannot simply rest on our laurels. I heard that there are some plans to develop 1.2 GHz NMR systems in the United States and Europe. They might overtake us very soon. To respond to this situation, Japan needs to draw up strategies. We may lose the competition unless we make a step ahead of what others are doing.

Shimizu: Japan is very active in research on superconductivity, and has world-class researchers in basic theory, applied fields, and commercialization. I expect that the technology we have developed will be applied to various fields such as MRI, nuclear fusion, magnetically-levitated trains, and superconductive power lines. However, looking into the future, it is also a fact that Japan is behind Europe and the United States in the aspect of formulating strategies. As NIMS researchers, we intend to address this issue promptly.

(by Shino Suzuki, PhotonCreate)

Key Technology Supporting NMR Systems #1

Never-Ending Quest for New Superconducting Materials

Dream material with zero electrical resistance

Superconductivity is a phenomenon in which the electrical resistance of a material becomes zero. A material that becomes superconductive as it is cooled to a certain temperature is called a "superconductor," and that temperature is called the "critical temperature" (or "superconducting transition temperature (T_c)").

If a superconductor is used as a power line, a large amount of electricity can be transported along it without losing energy. In addition, if a superconducting magnet is created using a coiled superconducting wire, it can generate a far stronger magnetic field than a conventional electromagnet wound with copper wire. Superconductors with these characteristics are applied in various fields such as the energy sector including the transport of electric power, the transportation sector including the magnetically-levitated train, and the biochemistry and medical fields including NMR and magnetic resonance imaging (MRI) systems.

Superconductors with higher critical temperatures

The superconducting phenomenon was discovered in 1911. When mercury (Hg) was continuously cooled, its electrical resistance

suddenly dropped to zero at 4.2 K (K: absolute temperature. 0 K = -273.15°C). Later on, it was found that metallic elements such as niobium (Nb) and lead (Pb) have a superconducting state. In the 1950s, new superconductors with higher critical temperatures were discovered one after another, an intermetallic compound such as NbTi (9.9 K) and Nb₃Sn (18K).

However, in order to cool these metallic superconductors, rare and expensive liquid helium needed to be used. To apply superconductors broadly, superconductors with even higher critical temperatures were surveyed.

After long exploration by many researchers, in 1986, a cuprate was found to have a superconducting state. This was the first discovery of a high-temperature superconductor, which had a much higher critical temperature than metallic superconductors. Amid the situation in which the quest for high-temperature superconductors was a hot topic in science, in 1988, Hiroshi Maeda of NRI discovered that a bismuth-based cuprate had a superconducting state. Its critical temperature was 110 K.

Much attention has been drawn to the cuprate high-temperature superconductor, but it was not just because this superconductor was able to realize superconductivity using liquid nitrogen, a resource available abundantly and inexpensively. When a mag-

netic field of certain strength is applied to a material in a superconducting state, electrical resistance is generated in the material even below the critical temperature. The strength of this magnetic field is called a critical magnetic field. The highest magnetic field generated by metallic superconducting magnets is 23.5 T (approx. 1,000 MHz). On the other hand, cuprate high-temperature superconducting magnets can theoretically generate a magnetic field of up to 50 T (approx. 2 GHz).

New Fe-based high-temperature superconductor

In 1993, Hg-based cuprate high-temperature superconductors with a critical temperature of 134 K were discovered, while among metallic superconductors, magnesium diboride (MgB₂) with a critical temperature of 39 K was discovered in 2001. In 2008, a totally new Fe-based high-temperature superconductor was discovered. Its critical temperature was 58 K. In the future, if we happen to find materials that can enter a superconducting state at room temperature so that the cooling process is unnecessary, rapid progress would be made in the application of superconductors in various fields. The quest for new superconductors continues.

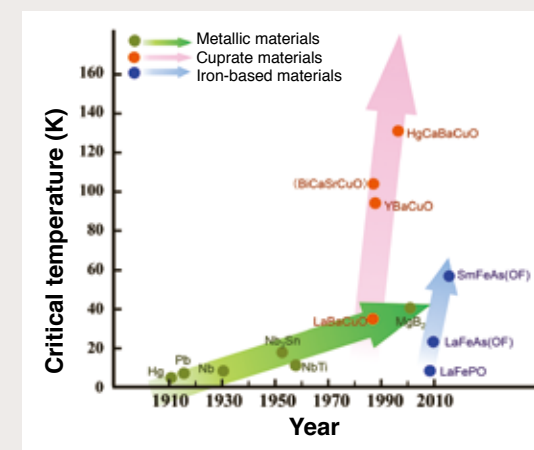


Fig. 1. Discovery of superconductors with progressively increasing critical temperatures



Fig. 2. Bi-based high-temperature superconductor

Dissecting the NMR System

NMR stands for "nuclear magnetic resonance."

The atomic nuclei in a sample placed in a magnetic field absorb and re-emit the electromagnetic wave energy. This phenomenon allows us to find out such things as molecular structure, which indicates how atoms are bound, and electronic state, which determines the physical property of a material. An NMR system consists of a "superconducting magnet" that generates a magnetic field, a "probe" that irradiates the sample with the electromagnetic wave and detects signals produced by nuclear magnetic resonance, a "spectrometer" that controls the generation and irradiation of the electromagnetic wave, and a "computer" that processes the data.



Superconducting joints

When the ends of two superconducting wire coils are connected and if the connection is also superconductive, the state is called a "superconducting joints." When all of the joints are in a superconducting state, the entire superconducting magnet will be in a complete superconducting state and the current applied to the magnet will continue to run permanently without any attenuation. Superconducting joints has not been achieved yet with a bismuth-based cuprate high-temperature superconductor.

Superconducting magnet

- Bi-based high-temperature superconducting magnet
- Nb₃Sn superconducting magnet
- NbTi superconducting magnet

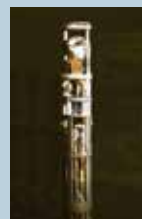
A coiled wire material*1 made of a superconductor*2 whose electrical resistance lowers to zero as it is cooled is called a superconducting magnet. Applying a current to the superconducting magnet generates a strong magnetic field at its center. Conventional NMR systems mainly use superconducting metals, such as NbTi and Nb₃Sn. In the NMR system that achieved the world's highest magnetic field of 1,020 MHz, we used a cuprate high-temperature superconductor called Bi-2223 at the center. Superconducting magnets using high-temperature superconductors can generate a stronger magnetic field.

*1 For details about fabrication of wire rods, see page 10.
*2 For details about the superconductors, see page 7



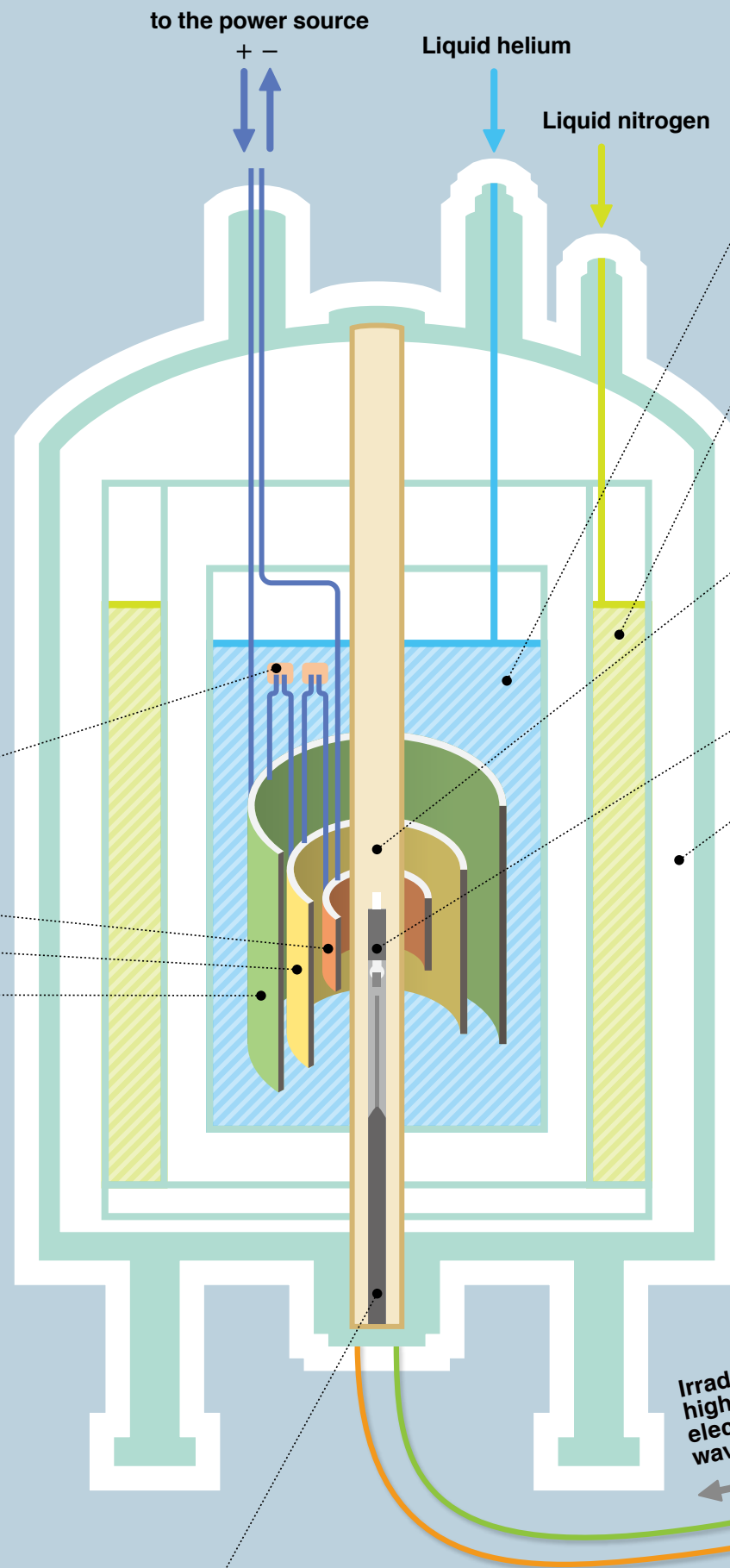
Winding Bi-2223 wire

Bi-2223 wire rods, about 3 km long in total, are wound into a coil of about 10 cm in diameter and about 1 m long. This wire rod looks like a tape about 4 mm wide and 0.2 mm thick.



Probe

The probe irradiates the sample with electromagnetic waves and receives NMR signals generated by the nuclear magnetic resonance phenomenon.



Liquid helium (-269°C)

Superconducting magnets are cooled in a tank filled with liquid helium to maintain the superconducting state. In order to reduce the influence of ambient temperature, the tank is surrounded by liquid nitrogen (-196°C), multiple vacuum layers, aluminum foils, and stainless steel. Liquid helium and liquid nitrogen need to be replenished regularly as they evaporate.

Liquid nitrogen (-196°C)

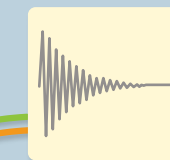


Sample tube

A sample is placed in a narrow tube with a diameter of about 5 mm. High-speed rotation of the tube makes accurate analysis possible.

Spectrometer

The spectrometer controls the generation and irradiation of high-frequency electromagnetic waves. It amplifies the NMR signals received by the probe and sends them to the computer.



NMR Signal

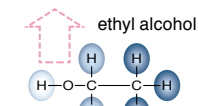
Computer

It carries out the Fourier transformation of NMR signals to calculate the NMR spectrum.



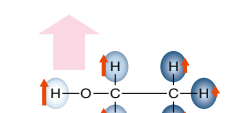
No magnetic field

Each nucleus is a small magnet. When there is no magnetic field, magnets are oriented in a random direction. As such, the total magnetization is cancelled out to zero on average.



Weak magnetic field

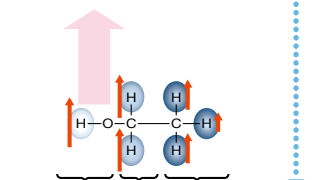
When a magnetic field is applied, magnetization occurs in the direction of the magnetic field (red arrow). Because electrons act to cancel the magnetic field, the magnetization decreases when there are many electrons around the atomic nucleus.



number of electrons : small medium large
magnetization : large medium small

Strong magnetic field

Magnetization is normally very weak. As the magnetic field becomes stronger, magnetization increases, allowing more accurate measurements.



number of nuclei
intensity of the magnetization of hydrogen nucleus

NMR spectral analysis

The horizontal axis of an NMR spectrum represents the intensity of the magnetization of atomic nuclei of the molecule. The stronger the magnetization is, the more the peaks shift to the left, and the weaker the magnetization is, the more the peaks shift to the right. The vertical axis represents the number of nuclei. The NMR spectrum gives us information on the molecular structure,

such as the bonding state of atoms, and electronic state. For example, the NMR spectrum of ethyl alcohol tells us that the ethyl alcohol consists of a hydroxyl group, methylene group, and methyl group, that there is a small number of electrons in the hydrogen nucleus of the hydroxyl group, and that there are many electrons in the hydrogen nuclei of the methyl group.

Processing Superconductors into Wire Materials

Fabrication of wire materials

The discovery of a new superconductor does not lead to immediate practical use. In order to use superconducting materials as power lines or superconducting magnets, they must be processed into thin wire.

It is insufficient, of course, just to process the material into thin wire. Wires used as superconducting magnets are wound into coils, so they must be resilient to bending and pulling. Also, a single wire needs to be at least 1 km long. The key matter concerning superconducting wire is its high “critical current density.” When a certain level of current is applied to a material in a superconducting state, electrical resistance is generated in the material even below the critical temperature. The electric current running in this condition is called “critical current.” Critical current density is calculated by dividing the critical current by the cross section area of the wire rod. The higher the value is, the greater the current that flows through the wire will be, even if a thinner wire is used. This property of superconductors contributes to the miniaturization of superconducting magnets.

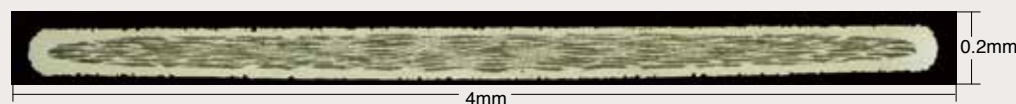
Every time a new superconducting material was discovered, advancement was made in the development of technology to improve the performance of wire materials.

Wire fabrication technology developed at NIMS

Among superconducting materials, NbTi was the first material that was successfully processed into a wire rod. Its high ductility at room temperature was an appealing advantage. Presently, this is the most commonly used superconducting wire rod in the world.

Like NbTi, Nb₃Sn is also a metallic superconductor, but is harder to process. Various methods were studied to establish a technique to fabricate a wire rod. The present major technique is called a “bronze process” developed by Kyoji Tachikawa, a researcher of NIMS at that time. In this process, Nb is introduced into a bronze base material—an alloy of copper (Cu) and Sn—to form cores, and they are heat-treated, resulting in the formation of an Nb₃Sn layer in an interface between bronze and Nb. The wire rods produced with this technique contain numerous, very thin superconducting wires embedded in the base material. The wire rod with this structure is called a multicore wire. The multicore structure enhances the stability of the superconducting wire rod and allows it to be used as a superconducting magnet.

Using hard and brittle material



Bi-2223 wire rod and its cross section

The Bi-2223 wire looks like a tape and it is approx. 4 mm wide and 0.2 mm thick. It has a multicore wire structure in which tens to hundreds of very thin superconducting wires (several microns to several hundred microns thick) are embedded in a silver base material.

The cuprate high-temperature superconducting material called Bi-2223 discovered by Hiroshi Maeda, a researcher of NIMS at that time, was the first high-temperature superconductor successfully fabricated into a wire rod. The material not only has a high critical temperature of 110 K but also has excellent electrical conductivity properties. For this reason, since its discovery, the material has been drawing much attention as a promising superconductor for practical use.

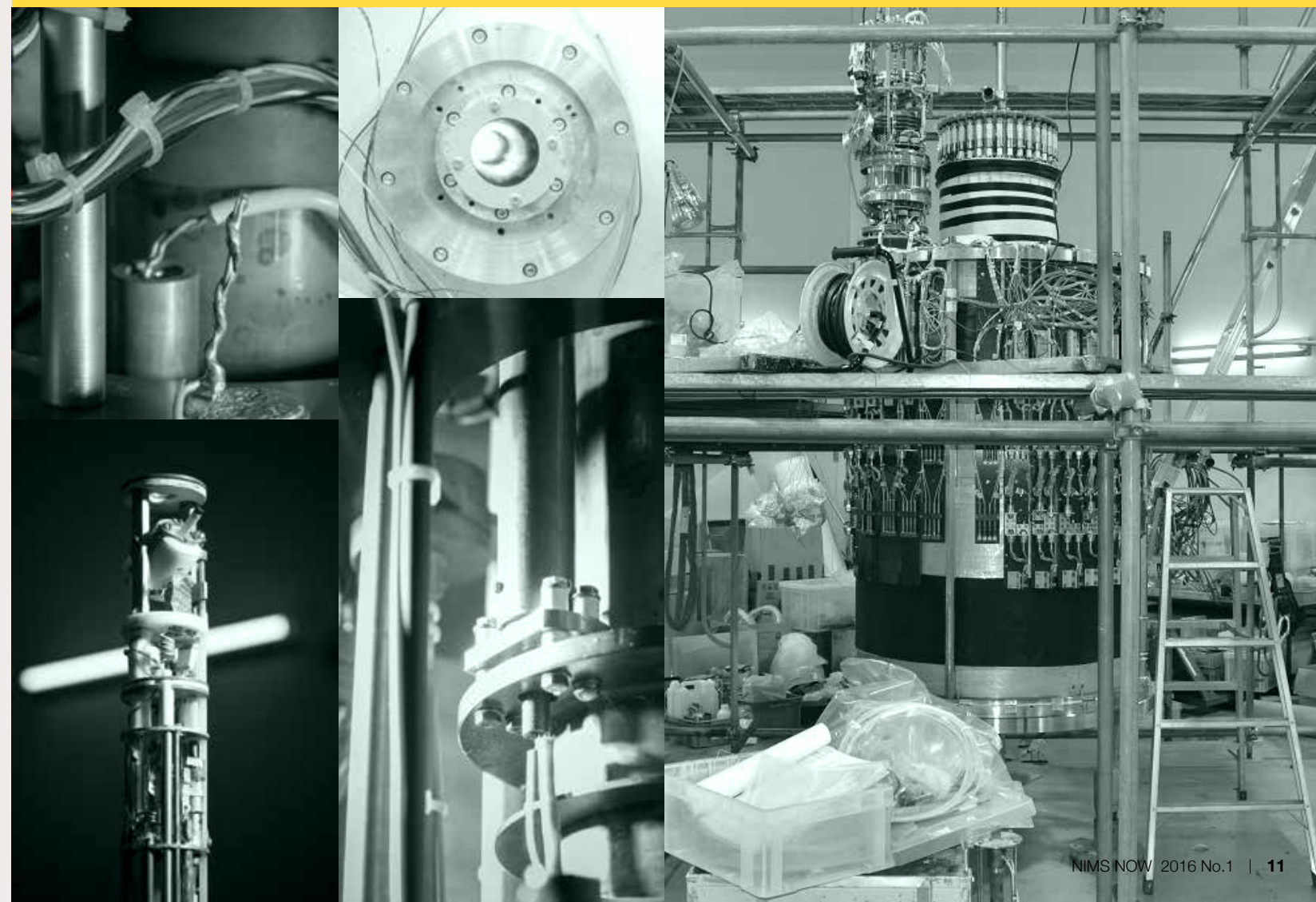
However, the cuprate is hard and brittle, while the material must be resilient to bending, so this issue posed a great challenge. Furthermore, this material has a complex structure consisting of five elements: Bi, strontium (Sr), calcium (Ca), Cu, and oxygen (O). After dealing with great difficulties, Sumitomo Electric Industries successfully commercialized Bi-2223 wire rods in 2005. This wire rod is also used in the high-temperature superconducting magnet installed in the 1,020-MHz NMR system at NIMS. As the wire rod has a very high critical current density, its application in power lines also has been making advancement.

Through continued development of new wire rods and improvement of their performance, we expect that practical use of superconductors will be accelerated and the scope of its application will become broader in the future.

Development History of High-Field NMR System over 1 GHz

Overcoming the Waves of Chaotic Events— Development History of an Over-1 GHz NMR System

On October 17, 2014, about 20 years after the first plan was proposed, the research team finally achieved the world's highest magnetic field, 1,020 MHz (24.0 T), in an NMR system while engaging in the project called “Development of an Over-1 GHz-NMR System.” The project team encountered many challenges while they went through this long tough road, such as catastrophic damage to the nearly completed system caused by the Great East Japan Earthquake, followed by a suspension of the project, as well as the sudden death of the team leader, and the breakout of a global helium crisis. Representing the project team that brilliantly overcame these hardships with creative ideas and a strong will, four of the members now look back on the project.



Devastating Earthquake hit just before completion

Matsumoto: The “Development of an Over-1 GHz NMR System” was a five-year project that started in the fall of 2006. The project was carried out under the strong leadership of Dr. Tsukasa Kiyoshi of NIMS, who suddenly passed away in January 2013. Among the four of us, I was the only team member who was under the direct supervision of Dr. Kiyoshi.

Nishijima: Initially, the project was expected to be completed during fiscal 2011. However, due to the Great East Japan Earthquake, which struck just before the completion, the project was forced to suspend for two years. When post-disaster reconstruction made significant progress and the project was ready to resume at last, Dr. Kiyoshi had suddenly passed away. Because I was affiliated with a different research group at that time, I did not expect at all to join the project. I assumed that I was chosen as a member of the team for my experience in the development of magnets with high magnetic fields in my previous job at Tohoku University. But I was more or less anxious because the concept behind the design of superconducting magnets for NMR was quite different from that of ordinary high field magnets.

Ohki: I was originally an engineer specialized

in measuring samples using an NMR system, and was a member of the team led by Dr. Tadashi Shimizu, who took over the role of the late Dr. Kiyoshi. From the perspective of Dr. Kiyoshi, I was merely an NMR system user, so I also never expected to be involved in the development of an over-1 GHz NMR system (hereinafter referred to simply as over-1 GHz). I was involved in the project because of the Great East Japan Earthquake. As both the over-1 GHz and the 930 MHz NMR system (herein after referred to simply as 930 MHz), which was used by the Shimizu team to which I belonged to, were damaged by the disaster, we had to repair them. Because Dr. Kiyoshi needed to take care of other matters from April 2011, and both systems were developed by Kobe Steel, Dr. Shimizu was appointed as a leader in charge of the repair work and I was also involved in this task.

Hashi: I was also a member of the Shimizu team. Until immediately before the great earthquake took place, I was taking measurements on inorganic materials using the NMR system. I was involved in this project in the same way as Mr. Ohki. I remember that Dr. Kiyoshi and I visited the High Field Magnet Laboratory in Nijmegen, the Netherlands, a long time ago. He was always smiling, and friendly to everyone.

Matsumoto: Dr. Kiyoshi used to say, “This is the second time for me to take on the challenge of developing an over-1 GHz in the last 20 years.” In 2001, Dr. Kiyoshi succeeded in developing a 21.6 T/920 MHz NMR system that generated the world’s highest field NMR at that time. He was actually aiming to achieve over-1 GHz using a superconducting oxide called Bi-2212. However, it was difficult to make a coil with that material, so we used improved Nb₃Sn, a metallic superconducting wire, and achieved 920 MHz. Even today, no one has succeeded in developing practical Bi-2212 yet. This time, we achieved over-1 GHz using a different type of superconducting oxide called Bi-2223. It is very unfortunate that Dr. Kiyoshi could not witness this achievement.

Unexpected aftershocks and sudden death of the team leader

Nishijima: In the morning of March 11, Dr. Kiyoshi was seeing researchers invited

from France to show them the nearly completed over-1 GHz system. Several days later, those researchers contacted us out of concern as to whether the over-1 GHz was okay.

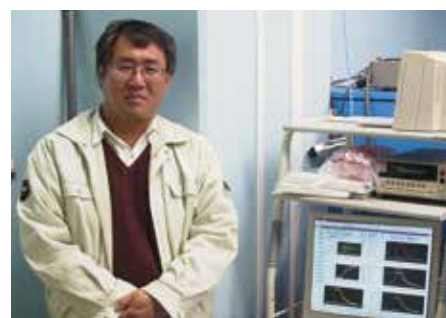
Hashi: After the earthquake, the over-1 GHz was severely damaged, but the magnet of the 930 MHz was still functioning. Once the system is excited, the 930 MHz cannot be demagnetized unless we reduce the current feeding the magnet. However, the socket that needed to be connected to the power supply to reduce the current was found 1.2 m lower than it should be in the system, and another socket situated in the liquid helium was found to be covered with solid air. As such, we could not attach the connector to the socket—a necessary step before plugging



Room temperature bore magnet tube (copper tube of 56mm diameter and 4m long) 10 ton weight shaken by horizontal oscillation of earthquake caused the vacuum break.



Bellows broken by the earthquake



Late Dr. Kiyoshi in 2004, when 930MHz-NMR had been developed.



Gen Nishijima

Senior Researcher, Magnet Development Group, Superconducting Wire Unit, Environment and Energy Materials Division

the socket to the power supply. If this condition was unattended, the magnet could transform into a normal conductor any time and burn, potentially leading to a major accident. The situation was very tense.

Ohki: We needed to dig out the socket while still generating the magnetic field. But we could not see the inside of the system, and needed a CCD camera that could be used even at extremely low temperatures. Dr. Hashi made the camera.

Hashi: Dr. Shimizu came up with that idea. He said, “When I experimentally submerged an LED flashlight into liquid nitrogen, the flashlight still worked. So the CCD camera should also work fine.” (Hashi laughing) Then, we mounted a heater on the CCD camera and placed it inside the system. It worked! Later, that camera played a significant role again in the development of the over-1 GHz.

Matsumoto: It was not until April 2012 when we were finally able to start the repair work. We first worked on the 930 MHz, and then the over-1 GHz. However, we encountered another accident. On December 7, when the repairs for the over-1 GHz were about to be completed, magnitude 7.3 quake struck Japan. The over-1 GHz was again partially damaged. We were quite shocked by the event.

Hashi: As if things weren’t bad enough, Dr. Kiyoshi suddenly passed away in January 2013. While we felt hopelessly lost, NIMS executive vice president said, “The development of the over-1 GHz is

very important, and we definitely want you to continue.” So we first completed the repair work and resumed the project after a two-year suspension, under the direction of the new team leader, Dr. Shimizu.

Nishijima: I also joined the project around the time when it was resumed. But more challenges struck us soon. The “Helium shock” broke out as a result of the greatly reduced helium supply from the United States. From around June 2013, it was becoming difficult to obtain helium in Japan, and this situation interrupted the cooling of the repaired magnet. To deal with this issue, we asked research institutes and companies for support, and managed to fill the system with liquid helium in September. Then, we moved on to the next step.

Ohki: We had another major challenge before that incident, right? The Over-1 GHz needed to be cooled in two steps—with liquid nitrogen and with liquid helium—during excitation. In this procedure, we needed about 10,000 liters of liquid nitrogen, but it would be cumbersome and take time to obtain that amount using regular liquid nitrogen containers. So, we considered the possibility of installing a vacuum insulated pipe to transport liquid nitrogen directly from the tank. However, after learning that this approach would take several months and tens of millions of yen, we gave up the idea. As a last resort, we purchased commercially-available 16-mm-diameter plastic tubes and wrapped them with three 80-m-long insulated pipe with a diameter of about 20 cm. After performing tests several times, we confirmed that liquid nitrogen flew smoothly in the pipe and decided that the pipe was ready for our intended use.

Nishijima: That method cost us less than about one-twentieth of what it would have cost if we had asked professionals to do the job. The way we looked when we were all carrying the very long pipe reminded me of the Nagasaki Kunchi Festival* [laughing]. In addition, as laying the pipe on the floor would take up a lot of space, we bought steel racks, assembled them, and set the pipe at a level above our heads. We had fun doing that.

Hashi: Failure to cool the system twice in September and January 2014 had a major impact on our project. After the failure occurred, we inspected the inside of the system using the CCD camera, which played

Development History of High-Field NMR System over 1GHz



Making a simple insulated pipe for transporting liquid nitrogen



Laying a pipe for liquid nitrogen over steel racks



Kenjiro Hashi

Senior Researcher, High Field NMR Group, Nano Characterization Unit, Advanced Key Technologies Division

*Traditional Japanese festival with a dance of huge dragon manipulated by 10 people.



Shinobu Ohki

Senior Engineer, High Magnetic Field Group,
High Magnetic Field Station, Research Network and
Facility Services Division

a major role during the 930-MHz system repair work. We found a part laying more than 1 m lower than where it was supposed to be, and removed it. It was just a small piece, but if it hadn't have been removed, it could have had a devastating impact on the entire gigantic system. After that, we made good progress in the cooling work, and we were able to conduct electrical tests.

Ohki: Nonetheless, the cooling work was quite challenging. The system generated heat at a much higher rate than expected, and the liquid helium was consumed very quickly. So nearly every day, we took turns refilling 250 liters of helium at a time. We continued this procedure from September 2014 to May 2015.

Finally achieving over 1 GHz

Matsumoto: We also developed a safety device that would prevent the system from failing again if a power failure occurred. We also prepared a system that would call the cell phones of all members when the system detects an error. Under the direction of leader Shimizu who put forward the slogan "We resolutely carry out the project 24/7," we finally made a magnetic field exceeding 1 GHz on October 1, 2014. That day, we took a commemorative photo with great joy. However, from the following day on, we increased the level of the current, aiming to renew the record.

Hashi: On October 17, 2014, we reached a magnetic field of 24 T/1,020 MHz. That was the world-record magnetic field for an

NMR system. However, the magnetic field was unexpectedly unstable, and this condition would not allow us to attain the end goal of collecting protein data with high precision. Consequently, we newly developed a system to correct the magnetic field. With this addition, we had to keep the system running all the time, including the time period between year-end and New Year holidays. We had to work harder to ensure the liquid helium level. So it was quite a relief when the collection of protein data were completed in January of the following year.

Nishijima: On April 14, 2015, when we completed all the measurements with NMR, we also succeeded in generating a magnetic field of 24.2 T. This was the world's highest magnetic field for a practical superconducting magnet at that time. As there was a chance of the superconducting magnet transforming into a normal conductor during the operation, we nervously ran the system.

Ohki: The project was supposed to be completed before May. But to our surprise, on April 15, our cell phones rang when a system error was detected for the first time. It told us that an instantaneous power failure occurred when lightning struck, and all of the members arrived at the facility in

about ten minutes. As we had taken every measure possible in preparation for any kind of disaster, we were able to confirm that the over-1 GHz was running without a problem. This preparedness was the result of having overcome many challenges and having refined our methods.

Hashi: While I feel great that we have achieved over 1 GHz and 24.2 T, as an NMR magnet user, it is more significant for me that we are now able to measure what we could not before. This project aimed to achieve a new magnetic field record. In the future, I also hope to see the development of measurement technologies.

Matsumoto: In reality, the development of over 1 GHz is impossible using metallic superconducting materials. The development was started after the late Dr. Hiroshi Maeda of the then National Research Institute for Metals discovered superconducting oxides in 1988. And it was about 20 years ago when Dr. Kiyoshi put forward the goal of developing the over-1 GHz using the wire material. So the 20-year-old dream finally came true. We would like to continue the work of NMR system development at NIMS and deliver the kind of result Dr. Hashi just explained.

(by Kumi Yamada)

Development History of High-Field NMR System over 1GHz



Commemoration photo of project team members (taken when the operation of the entire system including the NMR spectrometer was verified)

Science is even more amazing than you think (maybe...)

Bardeen-san

In 1911, Dutch physicist Heike Kamerlingh Onnes discovered a phenomenon called superconductivity. At that time, physics was not able to explain the mechanism behind this phenomenon. It was Dr. John Bardeen who theoretically explained the phenomenon in terms of a new discipline called quantum theory.

Dr. Bardeen began to work at Bell Labs in 1945, and in 1948 Bardeen and his coworkers—William Shockley and Walter Brattain—jointly invented the transistor. For this feat, a Nobel Prize was bestowed upon these three physicists in 1956.

In 1951, Bardeen became a professor at the University of Illinois, where he began full-fledged study of a theory for superconductivity. And in 1957, together with Leon Cooper, who was invited from Princeton University, and graduate student John Robert Schrieffer, Dr. Bardeen proposed the BCS theory (named for their initials), which is known as the standard theory of superconductivity.

For this achievement, these three researchers won the Nobel Prize in Physics in 1972. Of many Nobel Prize winners, Dr. Bardeen is the only person who received the prize in physics twice. This fact alone represents his immense contribution to physics.

Bardeen-san was very fond of Japan, and visited the country many times. When he attended the International Conference of Theoretical Physics held in Tokyo and Kyoto in 1953, he paid attention to the electron-phonon theory put forward by Sadao Nakajima, a professor emeritus at the University of Tokyo, who was leading the proceedings of the conference. Based on Nakajima's theory, he published a paper which later became the basis for the BCS theory. He and Professor Nakajima maintained a friendly relationship for the rest of their lives.

With desire to talk with Bardeen-san in person, I visited him in his late years at the University of Illinois at Urbana-Champaign over 30 years ago. There was a monument on campus commemorating his submission of the BCS theory, which concerns the superconducting phenomena, and his consequent second reception of the Nobel Prize in Physics.

Bardeen-san was very smart and friendly. He happily welcomed me, and said, "Let me first show you my lab." We went up the narrow stairs to the top floor. Seeing him move so nimbly, I could not believe that he was nearly 80 years old. I rushed to follow him, out of breath. He pointed to his small lab and guided me inside.

The room looked too plain and ordinary for a two-time Nobel Prize winner.

After telling me some stories related to his past research, he told me, "Nothing would give me more joy than to see the BCS theory being of service to society. In fact, the practical use of NMR—one example of that—is close to being realized, and I would like to show it to you tomorrow, so please come." On the following day, he took me to an NMR system in the university hospital, and enthusiastically spoke of

Written by Akio Etori

Title lettering and illustration by
Shinsuke Yoshitake

its usefulness and potential to me.

As you might know, NMR is currently making a major contribution in the medical field as it is applied to MRI systems, which are used for medical diagnosis and checking the state of the brain.

On a different note, Bardeen-san loved golf. He once said to me, "I recently built a new house on a golf course. Would you like to come by?" The house was not actually on the golf course, but was right next to it, and I could clearly see people playing golf in the course from inside the house. I was afraid that a ball would hit the house, but he did not seem concerned about it at all. He said, "Since I am old now, I usually play only nine holes." He looked like he was heartily enjoying his life.

I left Chicago, thinking about how good-natured Bardeen-san was (despite his social status and great achievements, I think the standard honorific "san" suits him more than sensei in view of his down-to-earth character).

**"san" is a most common Japanese honorific suffix. It often translated as Mr/Ms/Mrs in English. It includes not only feeling of respect but more friendly nuance than "sensei" which stands for Dr/Professor.



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.

1 Inauguration of New President of NIMS

Prof. Kazuhito Hashimoto is appointed new NIMS President on January 1, 2016. He continuously serves as a Professor of The University of Tokyo, Executive Member of Council for Industrial Competitiveness (Cabinet Secretariat of Japan), and Executive

Member at Council for Science, Technology and Innovation (Cabinet Office of Japan). Former President, Prof. Sukekatsu Ushioda resigned as a President on December 31, after his outstanding eight years contribution to NIMS, and now serves as a NIMS Advisor.

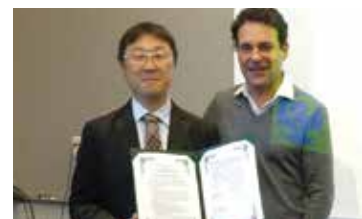


President Kazuhito Hashimoto

2 MANA at NIMS and MoIES at University of Washington conclude MOU

(December 4th, 2015) International Center for Materials Nanoarchitectonics (MANA) at NIMS and Molecular Engineering & Science Institutes (MoIES) at University of Washington (UW) concluded a Memorandum of Understanding. UW, based in Seattle, was founded back in 1861 and is one of the most preeminent universities in the world. Having been a pioneering university in Bio-

engineering and Medicine, UW turned out seven Noble Laureates. MoIES was established in 2007, with top professors and most advanced equipments in the world. Based on this MOU, Biomaterials Unit of MANA will be conducting the research collaboration with MoIES not only in smart biomaterials fields, but in development of many other biomaterials.



Mitsuhiro Ebara, MANA Scientist (left) and Prof. Patrick S. Stayton, Director of MoIES (right)

3 NIMS Signs a Comprehensive Memorandum of Understanding with University of the Philippines System



Quezon Hall at UP Diliman

(December 15th, 2015) NIMS concluded a Comprehensive Memorandum of Under-

standing with University of the Philippines System (UP System). UP System was founded in 1908 and is the most prestigious university in the Philippines. UP System consists of 7 constituent universities on 15 campuses across the country with 60,890 students, among them 14,800 are graduate students, and 5,400 faculty members (as of 2013). UP System has produced 7 out of the 14 Presidents of Republic of the Philippines, 30 out of the 31 National Scientists and a

great number of the leadership in the country. Many faculty members working on materials science are in the Materials Science & Engineering Program (MSEP) at UP Diliman but there are more at other universities such as UP Baguio. A fairly large number of them have experienced staying or working as a postdoctoral fellow at NIMS. This agreement will not only reinforce the existing collaboration between UP System and NIMS in various fields of materials science but also envisage new cooperation and exchange of researchers.

Hello from NIMS

Comparing with USA, a nation of immigrants, nation of Japan is a monolith. This monolithic nation encloses itself with a fence made of laws, culture, language and social groups to keep the foreigners out. Foreigners living in Japan have to spend their life to integrate into this society, however, surprisingly easy in scientific research due to sincere efforts made by scientific institution like NIMS. In NIMS, there is an amazing

program called ICYS, by which talented young scientists from all over the world with different research fields and cultures are "forced" together to do independent research with their own research budget and the freedom. Fortunately, I successfully become an ICYS researcher in the new year of 2015. Joining ICYS brought me a more convenient research environment, both hard environment and soft environment. Currently I am focused on developing a so-called Chop-nod method in surface analysis field in which a whole electron spectrum is

employed as a probe to extend these "old" techniques available for hotspots nowadays such as nanomaterial, energy material.



Da Bo (China)
Jan 2015 – present
ICYS-Sengen Researcher



The Da family enjoy skiing in Hakuba valley.



NIMS NOW International 2016. Vol.14 No.1

National Institute for Materials Science

<http://www.nims.go.jp/eng/publicity/nimsnow/>

© 2016 All rights reserved by the National Institute for Materials Science
photo by Nacása & Partners Inc., Yasuhiro Yasutomo, Michito Ishikawa
editorial design by lala Salon Associates

To subscribe, contact:

Dr. Yasufumi Nakamichi, Publisher
Public Relations Office, NIMS
1-2-1 Sengen, Tsukuba, Ibaraki, 305-0047 JAPAN
Phone: +81-29-859-2026, Fax: +81-29-859-2017
Email: inquiry@nims.go.jp

R100
Percentage of Waste
Paper pulp 100%

