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FEATURE STORY

Neutron powder diffractometer team members stand in front of the SuperHRPD beamline building at J-PARC. From left: Dr. Takashi Muroya, Dr. Shuki Torii, Prof. Takashi Kamiyama, Dr. Miao Ping, Dr. Junrong Zhang, Dr. Teguh Yulius Surya Panca Putra, and beamline user Dr. Sang Hyun Lee.

A high resolution, high intensity neutron diffractometer

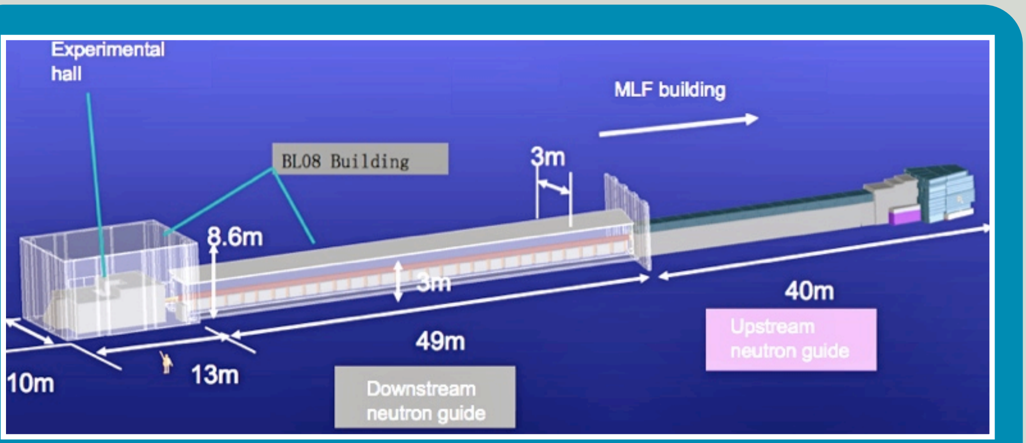
[Neutron diffractometer, SuperHRPD, Industry Application]

The team of neutron diffraction scientists at KEK has constructed the world's highest resolution neutron powder diffractometer, SuperHRPD. In addition to conducting experiments at the frontier of neutron physics, the team puts forth great efforts to involve local industry in their work. Here, read how they managed to achieve this high resolution, and how the world's first industrial involvement in neutron science came about.

Suppose we have a good detector with good resolution to detect neutrons, but we want to make it better? What can we do to improve the resolution? First off, you might like to make your neutron beam pulse compact. What else? Think about how you might measure slightest difference in the wavelengths of neutrons. The answer: move your neutron detector far away from your neutron source so that small differences have time to develop into large differences.

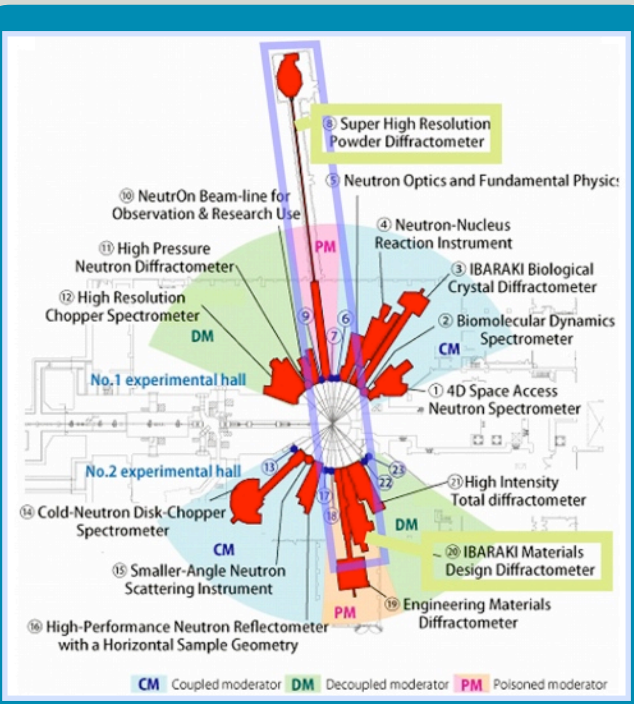
This is the essence of the idea behind the super high resolution powder diffractometer (SuperHRPD) at the Japan Proton Accelerator Research Complex (J-PARC). Normally, a neutron experimental station sits at the end of a beamline which is no more than a few tens of meters in length. The SuperHRPD experimental station sits at the end of a beamline which is 100 meters long. This is one of only two facilities in the world with such a long neutron beamline. Additionally, the SuperHRPD has a specially designed neutron moderator to improve the resolution.

Prof. Takashi Kamiyama of KEK and his team built SuperHRPD to sample powders or crystalline materials using neutron diffraction techniques. In 2008, the beamline broke the world record for highest resolution, achieving a resolution of 0.03 percent of the lattice spacing of the sample. The previous record of 0.05 percent had stood for 20 years, and was held by the Rutherford Appleton Laboratory's ISIS in England. This means that using SuperHRPD, scientists can map the structure of their crystalline samples with a resolution of 0.03 percent of the lattice spacing, a distance which is typically on the order of angstroms (10-10 meters).



The super high resolution powder diffractometer (SuperHRPD) beamline is 100 meters long, helping the SuperHRPD to achieve the world's highest resolution.

Neutrons are particles which are most commonly found in the nuclei of atoms of ordinary matter. They are particles, but according to quantum mechanics, all particles are also waves. For the high resolution diffraction technique, scientists produce free neutrons that are unbound to any nuclei, and send them to a sample 100 meters away. The wavelengths of the neutrons are carefully chosen to be of the order of the atomic spacing in the sample.



A schematic view of neutron beamlines at Material and Life Science Facility (MLF) at J-PARC.

According to quantum mechanics, wavelength, energy, and speed are all different ways of looking at the same thing. If you know any one of these three, you can easily calculate the other two. Faster—and therefore shorter wavelength—neutrons would arrive at the sample earlier than slower, longer wavelength neutrons. By measuring the time-of-flight, scientists can calculate the wavelength of the neutrons. The difference in arrival time increases when the neutrons travel farther. Thus, the long beamline of SuperHRPD improves the resolution.

Bragg diffraction

The mechanism of neutron diffraction is similar to that of X-ray diffraction. When X-rays, high energy electromagnetic waves, hit a crystalline sample, they disturb the clouds of electrons surrounding the atoms within the sample. The disturbed electrons then emit new electromagnetic waves which mix together to produce an interference pattern. The phenomenon is called Bragg's effect. Scientists analyze the interference patterns, and work backwards to get high resolution image of the crystalline structures which produced the pattern.

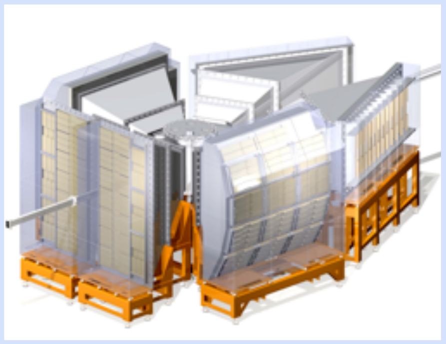
Neutrons are electrically neutral particles, so they cannot affect the clouds of electrons via electric forces. Instead, they interact with nuclei of atoms in the sample via the strong force. Additionally, neutrons have a tiny magnet-like property called spin, and this means they can interact with the magnetic field produced by the clouds of electrons.

An interesting difference between X-ray diffraction and neutron diffraction is that the two techniques are sensitive to atoms of different atomic numbers. X-ray diffraction produces a stronger pattern when there are more electrons. Atoms with larger atomic numbers have more electrons, and so exhibit stronger X-ray diffraction signals. For neutron diffraction, it is not so simple. There is no simple pattern, but in many cases, neutron diffraction is more sensitive to lighter atoms than to heavier ones. Therefore, neutron diffraction gives complementary information to the X-ray diffraction.

Sharp peaks, long travel, better resolution

Powder diffraction techniques, using both X-rays and neutrons, are well established methods. The X-ray powder diffraction technique has been steadily moving forwards for the past 10 years, as new technologies have allowed ever brighter X-ray sources. However, neutron diffraction has not had the same good fortune. With the ISIS upgrade in the UK, the SNS construction in the US, and J-PARC in Japan, this is going to change.

The aim of the SuperHRPD is to build and maintain the world's highest resolution neutron diffraction system for the next ten years. The ability to look into the nano scale structure of a broad range of materials will be crucial to nano-sciences and nano-technologies in the future. The high-resolution, high-intensity neutron diffraction technology is expected to play significant roles in a range of fields from materials to life sciences.



An illustration of the SuperHRPD instrument. A large neutron detector surrounds a vacuum chamber in which neutrons hit and are diffracted by a sample.

For the crystallography device, Kamiyama and his team made use of a well established technology previously developed at KEK. The main structure of the experimental apparatus was simply transported from KEK's Tsukuba campus to J-PARC's Tokai campus.

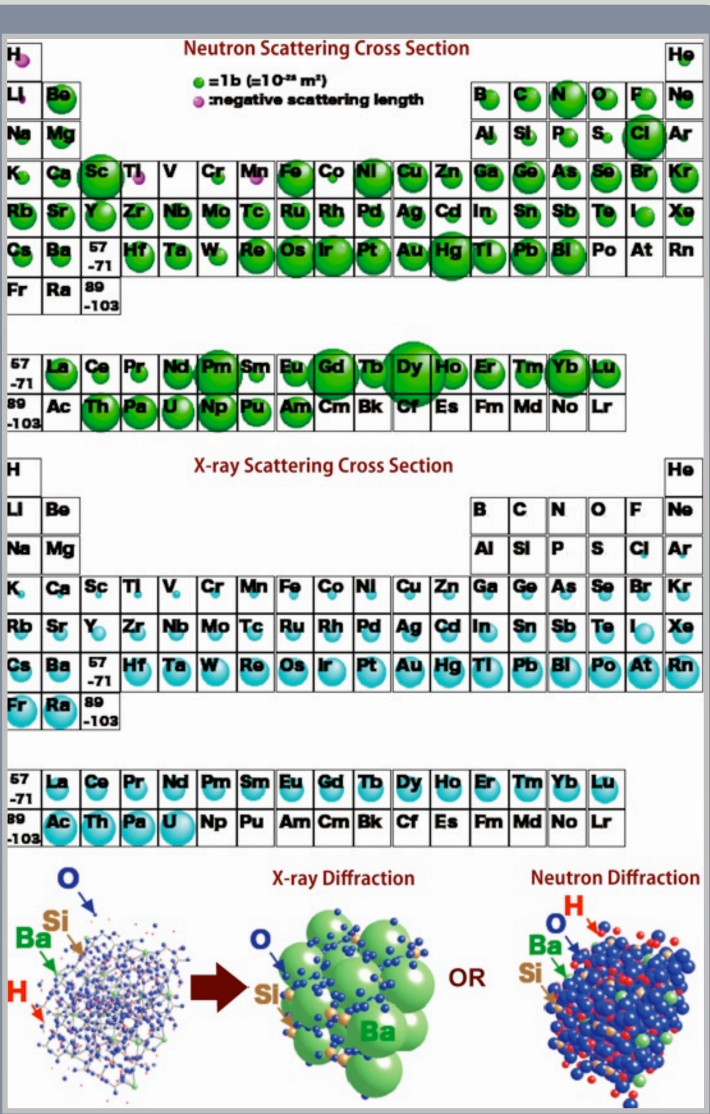
"There are three keys to achieve the high resolution," explains Kamiyama. "The first is the new design of the high resolution moderator in the neutron source, the second is the technology to transport the neutrons for 100 meters, and the third is to minimize detector pixel size."

Ground breaking design vs ground subsidence

A moderator is a device to slow down neutrons. Moderators are made of a type of hydrogen. Because hydrogen atoms have the same mass as neutrons, they can decelerate the neutrons efficiently. The team is interested in neutrons with a broad range of wavelengths, from 0.3 angstrom to 10 angstrom. More energetic, faster neutrons would come out of the moderator quickly, but the ones that take longer to come out are less energetic, slower ones. Therefore, the slower neutrons stretch out the neutron pulse.

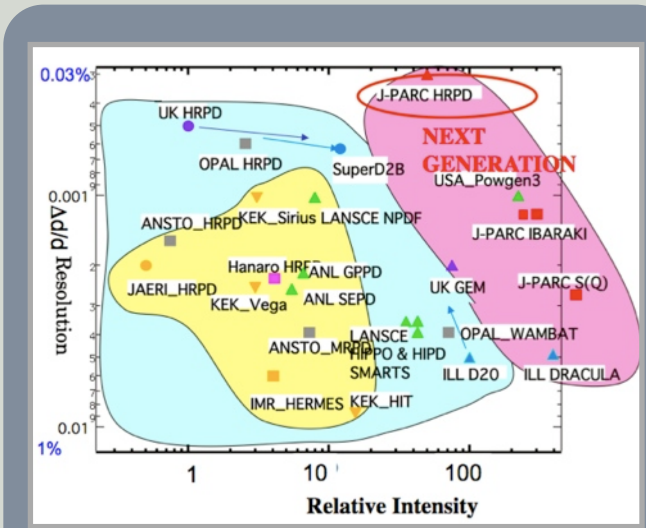
To filter out the lowest energy neutrons, neutron diffraction scientists generally install a slice of material called 'poison' in the middle of the moderator. Energetic neutrons can penetrate through the poison, while less energetic ones cannot. This essentially eliminates the low energy tail of the neutron pulse. Poison is generally placed at the middle so not to give directional preference (neutron beamlines generally stretch out in all directions).

The ingenuity of the J-PARC moderator design is that it positioned the poison much closer to the SuperHRPD beamline, instead of right at the middle, so that the beamline would look at neutrons coming out of much smaller volume of the moderator. This effectively generates narrower pulses. He and the moderator development team ran a number of simulations to understand the effects which various thicknesses, materials, and positions of the poison would have on quality of the beam. The innovative design of the SuperHRPD poisoned moderator produced a system with 1.7 times better resolution than the ISIS, which also has a 100 meter beamline.



Because of the different ways in which X-rays and neutrons interact with atoms, they have different sensitivities for different types of atoms, and therefore provide complementary information.

The length of the 100-meter long neutron beamline isn't unique, but it is a challenging business to build one, especially with the neutron guides made of glass on the fragile and quake-prone soil of Japan. The 40-meter upstream beamline is located in the material and life science building, while the 50-meter downstream beamline stretches out in an annex. On their own, these two structures would subside at different rates. "The original design of the beamline had 40 sections with 40 legs to support the rail," says Kamiyama. "It quickly became clear that it would be practically impossible to adjust every section to the right alignment."



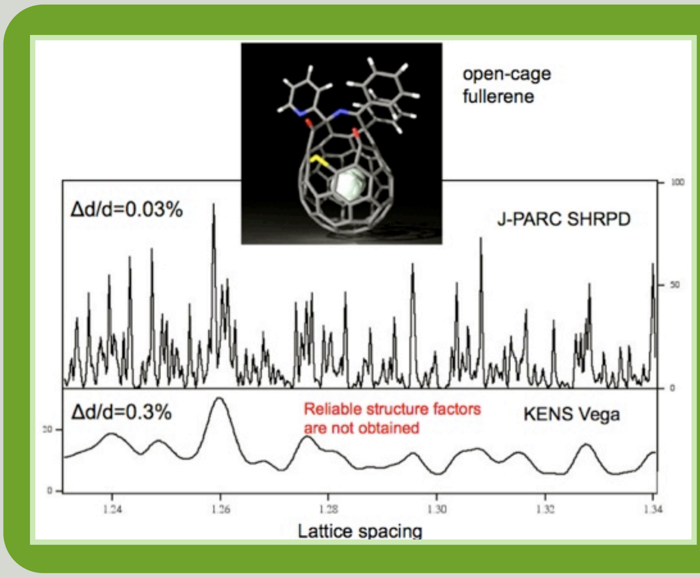
The neutron community is entering the era of the next-generation high intensity, high resolution neutron crystallography.

To solve the issue, the team exercised their ingenuity, and chose to build two rails—one for the upstream beamline and one for the downstream beamline. These two rails are supported by just 6 legs in total, instead of 40. This worked for natural ground subsidence and many earthquakes, but it was not as sturdy as the team had hoped. When an earthquake hit Tokai in June 2008, a part of structure was damaged. "The fight with the fragile soil still continues," Kamiyama says.

Bringing in local industry

There are two powder diffractometer beamlines at J-PARC. One is BL08, the SuperHRPD beamline, that aims to be the highest resolution neutron powder diffractometer beamline in the world. In contrast, the second BL20, the iMATERIA beamline, is designed to be a general purpose neutron powder beamline for materials science and industrial applications.

Just 26.5 meters in length, BL20 is the world's first neutron beamline owned by a local government. It is owned by the prefecture of Ibaraki to promote industrial use of neutron. The BL20 neutron beam is more intense than the BL08 beam, and has a broader range of



A resolution comparison between the SuperHRPD (resolution of 0.03 percent) and KENS Vega (resolution of 0.3 percent). The SuperHRPD can clearly identify the structure of a lattice of atomic size.

neutron energies. “The beamline will be used to study, for example, how to improve battery lifetime and to better understand material capabilities,” says Kamiyama.

Some of BL20’s other notable characteristics are the large size of the sample storage area, and the robot to automate transportation of samples between the storage area and the vacuum chamber. A normal storage area can hold only ten samples, while the storage area at BL20 can hold as many as 700 samples. The robot can take out a sample from a storage cell, bring it to the air lock, and then place the sample inside the vacuum chamber. “The development of robot has not yet been completed, but when it is, iMATERIA will provide a fully automated environment for the structural characterization using neutron to users,” says Kamiyama.

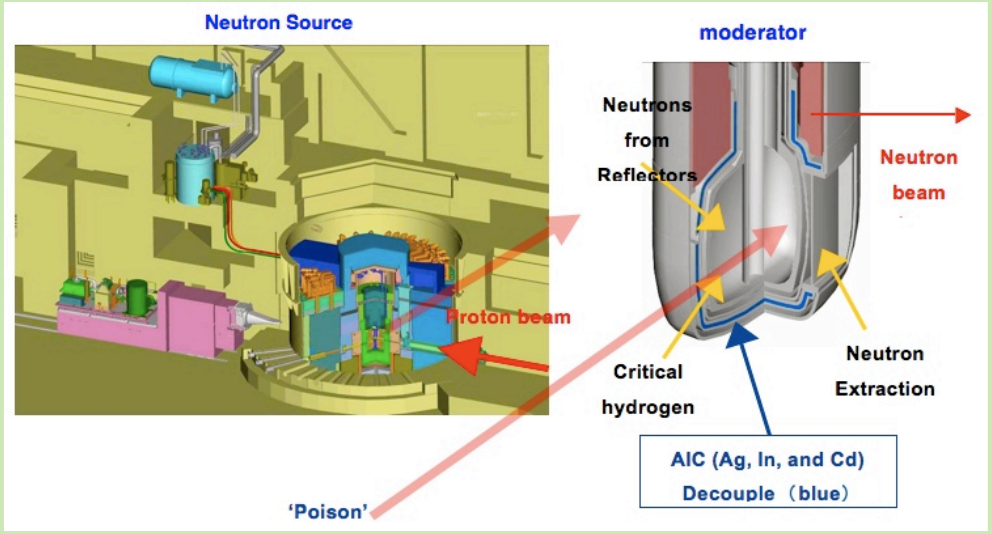
The development of the beamlines was a good opportunity for the newly established collaboration of small local industry, the J-PARC Support Study Group (JSS). The goal of the JSS is to support developing experimental devices at J-PARC. “The SuperHRPD chamber was developed by JSS,” says Kamiyama.

Each vacuum window of SuperHRPD chamber is a square piece of a thin aluminum plate which are 80 centimeters on each side. The windows surround the vacuum chamber at the center of the SuperHRPD device. The thin plates of aluminum are generally sandwiched between two thick and strong frames, which get in the way of the neutrons. To increase the effective area of detectors which look at the specimen located at the center of the vacuum chamber, Kamiyama’s team designed aluminum windows with different sizes, number, and spacing of fastening screws, and found that no frames were required to achieve the required physical strength. The JSS acted on this information, and after many cycles of test-and-modify, the team finally constructed and installed the SuperHRPD chamber with the frameless windows at the BL08 last August.

“The scheme worked,” says Kamiyama. “The resolution as well as the effective area of the detector was also improved, and spurious peaks that were present went away, improving the signal-to-noise ratio.”

Kamiyama and his international team members, Dr. Shuki Torii, Dr. Takashi Muroya, Dr. Junrong Zhang, and Dr. Teguh Yulius Surya Panca Putra, Dr. Ping Miao from Japan, China and Indonesia, respectively, oversee SuperHRPD, while Kamiyama’s pupils now working at Ibaraki University oversee iMATERIA. Now Kamiyama and his KEK members, Dr. Masao Yonemura, Dr. Ryoko Oishi and Dr. Takahiro Morishima work on development of analysis software to take full advantage of both SuperHRPD and iMATERIA.

The two beamlines are now producing large numbers of interesting results. The SuperHRPD can catch the slightest structural changes in crystalline, complex structures, composite materials, and layered structures with very high resolution. The team aims to fully realize the high-resolution and high-intensity capability of the SuperHRPD, producing performances that are comparable to the best resolution synchrotron radiation X-ray diffractometer technology. They believe that this will bring an entirely new type of crystallography to the world.



Neutrons are slowed in the moderator. For obtaining high resolution results, it is critical to employ the right materials at right places. For example, the team’s simulation showed that a partition to cut off the slow end of a neutron pulse, called poison, produces best results when it is located brought closer to the beamline.

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