WPI-AIMR NEWS
Volume 10
August 31, 2010
Cover: Snapshot of long-lived spatial heterogeneities, projected onto a x-y plane, obtained by numerically solving the Tokuyama nonlinear stochastic equation for density fluctuations in a colloidal suspension at a liquid state with volume fraction \( f=0.466 \) and a supercooled-liquid state with \( f=0.566 \), where the relaxation time is of order \( 10^2 \) and \( 6\times10^4 \), respectively. (Michio Tokuyama, WPI-AIMR)
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Progress of AIMR during April-August of FY 2010

Yoshinori Yamamoto
Director, WPI-AIMR, Tohoku University

Since volume 9 of WPI-AIMR News was delivered to you in April, four months have passed. You may be thinking that volume 10 should have already been delivered to you because four issues per year were published in the past. We have published (1) AIMResearch and (2) WPI-AIMR News through our office.

AIMResearch is the print version of the website "AIMResearch" in which specialists of Macmillan create abstracts on the top-notch research results that WPI researchers published in top journals. This print version is published once a year because the website is renewed every month. In addition to those two publications, (3) Tohoku WPI Tsu-Shin, an outreach magazine for the public, was launched in June along with MEXT's request that we enhance our outreach activities in order to let citizens and students know of the activities of this WPI research center. Accordingly, it seemed that quarterly publication of WPI-AIMR News would be too busy for us, and we have decided to publish WPI-AIMR News three times per year instead. I hope you will understand this change. Anyway, it is a pleasure for me to now deliver volume 10 to you.

In the middle of July, the third follow-up meeting of the WPI program was held in Tokyo. All the follow-up committee members, including foreign members, two Nobel laureates, the program director, the five program officers, and several MEXT members gathered at the interview room and listened to the presentation of all five WPI centers. This is the most important meeting for evaluating the activities of the WPI centers, and the evaluation report has just appeared on both MEXT and JSPS website. After the review meeting was over, Professor Kawasaki and Professor Chen gave lectures on their own research results in front of the committee members. I believe this was a very good opportunity for us to let them know what kind of top-notch materials research projects are currently being undertaken at AIMR. At the banquet for this meeting, the Minister of Education, Culture, Sports, Science and Technology, Mr. Tatsuo Kawabata, gave a speech and in it stated that MEXT understands the importance of the WPI program and, therefore, the 6th WPI center has been decided to be at Kyushu University even under the present-day financial problems of the Japanese Government. This is very good news for all of us, and I expect the continuous and strong support of MEXT for the WPI program.
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Finally, it is a pleasure for me to announce that Professor Masaru Tsukada has become the Editor-in-Chief of *WPI-AIMR News*, since the previous Editor, Professor Toshio Sakurai, left his position at AIMR. On this occasion I would like to thank Professor Sakurai very, very much for his tremendous effort on the inauguration of WPI-AIMR and launching *WPI-AIMR News*. Now the summer vacation season has already begun and you may receive volume 10 after the vacation is over. Let’s all make a good start to the semester in September.
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Interviews
Merry meet, many fusion

Professor Adschiri (A): I like to ski and I think there are two ways to enjoy skiing. The first is to ski fast and cool in front of a lot of people. The other is to stumble and plow through freshly fallen snow which no one has yet to set foot in. You don't know what you will find there, but you may see beautiful scenery in front of you or look behind to find that others have followed you creating a ski slope on which everyone can enjoy skiing. To put it simply, it's either competing to be No.1 or pursuing a path to be a unique "only one." Both are important, but in my research, I take the "skiing in the freshly fallen snow" approach.

I was born in Niigata. I moved around a lot because of my father's job as a banker. I went to different nursery schools and during elementary school, I transferred school every year. As a child, I kept thinking "not again!" but I became good at making friends. How you socialize with people is in part due to your personality based on DNA, but I believe it mostly relates to how you grew up. Someone pointed this out to me once, and looking back at my life. I agreed.

Professor Komatsu (K): Children are highly adaptable so I suppose they think and act according to their surroundings.

A: It was hard going from place to place, but I had fun. I actually have an elementary school reunion next week. All of my classmates are in completely different fields, but in a sense it is good to meet with people in a different environment because it gives you the chance to change your way of thinking.

K: That is important. It's difficult to become friends with people in different fields as an adult, but for children it's the opposite. You just have to meet each other and you become friends. Learning that there are ways of thinking other than your own and that there are so many kinds of people in the world will be an advantage later on in life. I'm sure the same is true for your research.
Interview with Professor Tadafumi ADSCHIRI, WPI-AIMR Principal Investigator

“Merry meet, many fusion”

Professor Adschiri (A): I like to ski and I think there are two ways to enjoy skiing. The first is to ski fast and cool in front of a lot of people. The other is to stumble and plow through freshly fallen snow which no one has yet to set foot in. You don’t know what you will find there, but you may see beautiful scenery in front of you or look behind to find that others have followed you creating a ski slope on which everyone can enjoy skiing. To put it simply, it’s either competing to be No.1 or pursuing a path to be a unique “only one.” Both are important, but in my research, I take the “skiing in the freshly fallen snow” approach.

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K: That is important. It’s difficult to become friends with people in different fields as an adult, but for children it’s the opposite. You just have to meet each other and you become friends. Learning that there are ways of thinking other than your own and that there are so many kinds of people in the world will be an advantage later on in life. I’m sure the same is true for your research.
A: I think so too. Take this room for example. A professor of humanities at a certain university once told me that holding classes at a café from time to time gives birth to completely different ideas than when he holds classes in a classroom. Cafés have music playing, greenery, and pictures. I tried to design my office with that in mind. I have pictures hanging even in the hall. It’s good to have experiment results and newspaper articles pinned up, but I thought it might be good to have a research environment in which students and staff can come up with ideas from a different perspective. It is important to place yourself in an environment where you use a different part of your brain.

I entered the University of Tokyo and my professor was Dr. Daizo Kunii. When you blow air into sands from below, the sand-bed enters into a state similar to boiling a liquid. In this state, solid sands behave like a fluid and move from one reactor to the next. This state is called “fluidization”. This technology led to the current petrochemical technology for processing large amounts of petroleum with solid catalysts. This is a technology which makes a new process treating the solid matter like liquid, and Dr. Kunii was the authority of this field. Originally this technology was born in U.S., but he made it possible to design the reaction that occurs inside the reactor for the first time.

K: By changing the media, you can modify the specific gravity.

A: That’s right. I said “similar to boiling a liquid”, but we can create the boiled situation without bubble and the fluid situation where all the sands are blown away. This determines the performance of whole process.

K: When we apply pressure on solids, it will not spread out equally, but in case of liquid the pressure works equally by Pascal’s principle, and also we can control the stream of a magnetic or electric field.

A: Creating a magnetic field and controlling it is state-of-the-art research. That is exactly the idea. When we know too much the research surrounding us, we cannot escape from it. On the contrary, when someone of different field says “Can’t you do it like this?”, it can be a trigger to change our original idea.

K: What you might call fusion.
A: Fusion can be achieved even by yourself if you change your surroundings. During the oil crisis 30 years ago, I was doing my doctoral thesis on gasifying coal in a fluidized bed in Kunii laboratory. Coal reacts with high temperature steam to from carbon monoxide and hydrogen. My research was on the conversion process of the internal structure of coal slowly changed into gas. By the time I finished my doctoral course, research on materials became active also in process engineering and I then started research on converting hydro carbon gas into solid carbon through chemical vapor deposition (CVD). It was very easy as I only had to do the exact opposite of what I had been doing up until then. I had to observe how the structure of carbon, the solid, changed. Even in this case, we can find a fusion of the totally different researches.

Unfortunately, close to the end of my research, Dr. Takehiko Furusawa, who was Dr. Kunii’s protégé, passed away at the young age of 49. I had worked as a researcher at the Japan Society for the Promotion of Science for a year after earning my doctorate and had just become an assistant at the lab. So in my second year, he was gone all a sudden. Because the professor was gone, the lab was shut down. I had a year’s grace, but that year passed by quickly as I helped students transfer to other labs. So I did not write a single paper that year.

There were many companies who invited me to work for them as I was struggling. I had almost made up my mind to go into the private sector, but Dr. Kunio Arai and Dr. Shozaburo Saito invited me to come to Tohoku University. So I decided to do research there and came to Tohoku University in 1989. I remember thinking to myself that as long as you do your work diligently, someone will notice and lend a helping hand.

At Tohoku University, I did research on supercritical high-temperature-high-pressure gas, which is the foundation of my current research. In chemical engineering, property estimation is necessary for developing and designing a process. At high pressure, especially at the supercritical point, where gas no longer condenses at a certain temperature, a condition where highly pressurized gas remains gas and does not turn into liquid under pressure is created. Under the condition, the properties change greatly so the property estimation becomes difficult.

Just around this time, Dr. Saito made Tohoku University the center of this research. I started research along with my boss, Dr. Arai. Changing the density of fluid changes
the solvent power, so utilizing this property, we did research on material separation. Since I moved from researching the reaction of coal to fluid properties and equilibrium, not only my research itself, but my whole mindset changed. I had to specialize in thermodynamics, which I was not very good at during my student days, so it was tough, but I studied hard because the professors gave me the chance to work.

If I had to try something, I thought it would be better to start from scratch rather than hold on to what I had up until then, so I quit my research on coal and started new research. At the time, supercritical research was all about research on separation and no one was researching “supercritical-reaction.” You lose sight of the people in the same field. That was why I was able to achieve fusion on my own. It was “supercritical-reaction-target material.” Coal, heavy oil, biomass, waste disposal, organic synthesis reaction; all of these. Also, the nanoparticles which are what I am current researching. I started research on all of these simultaneously from 1989.

K: I see. By discarding your old research you were able to get a fresh look at things.

A: Exactly. If I had continued with my old research, I never would have thought of it. The moment I made the decision to concentrate on supercritical research, I felt as if I knew exactly what I had to do.

Dr. Kunii’s research theme was “research on the methodologies for creation.” He says that the mindset for creation is calculation. Inventions originating completely from scratch are extremely rare. He says new ideas come from multiplying, adding, subtracting, and dividing old ideas. They may come from a transposed matrix or a transform matrix. He taught me that I always need to keep this in mind to come up with new ideas. The idea I brought up earlier was exactly that so it was an easy choice for me to make.

The other thing is to not think just by yourself. My main research theme right now is making nanoparticles in supercritical water. This was an idea I came up with together with my student. In supercritical water, substances are dissolved in highly pressurized, high density steam. Most metal oxides are dissolved. The earth itself is made in this way. The crust of underwater volcanoes is in a high temperature, high pressure state so subterranean water is always in a supercritical state.
I’m getting off topic here, but I once had the opportunity to talk to a professor of geophysics. I had always thought that magma was molten rock (lava), but it’s actually rock which has melted in supercritical water. This makes the water take on characteristics of rock, creating rock-like water, which keeps dissolving the surrounding rock. Magma is a state in which a lot of rock is dissolved in a small amount of water. Magma exists and flows even at a temperature lower than the melting point of rock. When magma rises suddenly, the highly pressured steam returns to a normal atmospheric pressure all at once and expands. That is a hydrovolcanic explosion. The Kanto loam layer is actually made up of natural nanoparticles. The melted rock that erupted from Mount Fuji and Mount Asama was depressurized and stopped melting and all of it was deposited. The particles deposited are the Kanto loam layer. As it slowly accumulated, mineral veins emerged. Because deposits occur starting with substances with lower melting points, you will always find the same type of substance in a single mineral vein.

K: But in the Kanto loam layer, you find small crystals such as pyroxene and olivine instead of powder. At a stage in which they can still be purified, they erupt along with the magma and changes in conditions cause them to change as well.

A: Yes. That’s exactly what naturally synthesized nanoparticles are.

There was a student who just happened to be doing similar research. Apparently he really did not enjoy his research and came to me for advice. He was making silica and at solubility of 1 wt%, even with a lot of water, all you would be able to make was about a pinch of silica. He told me he wondered if that would actually benefit anyone. To be sure, being able to melt a lot is one thing, but it is no fun if you end up with just a pinch of silica. We started discussing this and ended up getting drunk together.

When you think of solubility, all you think of is metal salts. “But wait, if metal salt can be dissolved into water, maybe that is all we need,” I said. At these words, the student suddenly woke up from his drunken stupor and went to the library for research. He came back and said to me “No, that won’t work. Metal salt will thermally
decompose in high temperatures and become metal oxides.” Since his aim was to make metal oxides, I jokingly told him that maybe that was a good thing, but the student quickly switched to equipment that would allow metal particles to flow and react in high temperatures. That was the first original nanoparticle synthesis process at Tohoku University. I examined it under an electron microscope and thought to myself how beautiful it was.

He recreated what nature does, not by depressurizing, but by rapidly raising the temperature. He made the metal salts react by letting it flow while simultaneously rapidly raising the temperature. This results in creating almost all the same thing. Using a pressure vessel called an autoclave with extremely thick walls like a cannon, the temperature rises gradually. By letting it all flow as a fluid like petrochemicals, he made them react inside and uniform, pretty crystals formed. This is the result that came out of a talk with a student. It is not only your own ideas that are important, but also interaction with others.

K: There are various kinds of interaction; between different generations, those who are learning and those who are teaching, those who are drunk, etc.

A: At the time I had just started working there so I was able to think from a fresh perspective without being completely immersed in that research. The free-thinking must have emerged from there not being a relationship of seniority. That is what fusion is. I still work with young post doctorates and when we do research on the same floor in different fields, it is always very refreshing. There is a high potential for new ideas and technologies to be born. In that sense WPI provides a great place for fusion.

K: Fortunately there are four very different disciplines, so it might be interesting to really flex the brain muscle to achieve fusion.

A: Seven years ago, I moved to the Institute of Multidisciplinary Research for Advanced Materials (IMRAM) from the School of Engineering in Aobayama and launched a new lab. Fortunately, all the positions were still open and it seemed like it could be an environment in which I could work together with associate professors, assistant professors and technical staff. So I decided to try something completely new again.
After having researched supercritical fluids for nearly 15 years, I didn’t think I would be stimulated by people who had been doing similar research. I looked for people who had what I didn’t have, people in fields such as computer science, biotechnology, and materials science. It was a gamble, but I thought I would try to make a place to fuse new ideas together to create something like “supercritical fluids-bio-materials-computer science,” similar to what I had done when I came to Tohoku University in 1989.

At first, it was as if I was talking to people who used a different language. Especially for bio research, there are so many abbreviations in that field. For the first couple of years, we couldn’t understand each other, so we held an educational seminar in our lab where we would give lectures on the very basics: bio, materials, computer and supercritical. Because it was all things I wanted to learn, things entered my mind smoothly, unlike when I was a student reluctantly attending classes. We kept throwing ideas at each other trying to find out if we could do certain things.

Dr. Umetsu, who is now an associate professor at the School of Engineering, built a bridge between the nano and bio fields for us. The peptides we use to recognize the particles we make are formed from a chain of more than ten amino acid molecules. For example, for zinc oxide we would have to find a peptide that would only stick to zinc oxide, and one that would only bond to the (100) sides of the zinc oxide. Dr. Umetsu prepared $10^9$ different types of peptides and experimented with them all. That was something. Viruses infect cells. He injected a little bit of the DNA of each of the $10^9$ different peptides into the viruses. He looked at which peptides would stick on the Zn O viruses. But it’s is hard to find the ones where only one or two have bonded. However, we are talking about viruses. So, you infect this virus to E. coli bacteria.

**K:** A virus is clump of genes, an aggregate of nucleic acids.

**A:** Yes, viruses are not alive. They are not living organisms. They have DNA inside them and work to inject that DNA elsewhere. Cells injected with that DNA are confused into thinking the alien DNA is their own and thus multiplies the DNA, causing bacterial or viral infection. For example, even if only one or two virus peptides latch on to the E. coli, it will help the virus to multiply until the virus grows very large and if we take a sample of the virus after that and look at its DNA, we can see which peptides
are attached to it. This method is known as phage display. We have found many peptide that can recognize inorganic nanoparticles using this method.

At times like this, device people usually come up with completely different ideas. They usually ask, “Can’t you put the peptides indicated on the foot of the virus on the head, hands and shoulders of the virus as well?” We can do it. According to the way device people think, it’s better to indicate everything here— for example, “here a source, here metal, here a drain and then on the opposite side, attach a semiconductor particle and a dielectric around that space there.” They will say, “Can’t we make this circuit into any design we like?” We have some particles, and when the particles are lined up in a free-form shape they form a circuit. If you want to line up the particles in a particular way, it’s better to make living organisms line them up. The idea is that if you change the necessary blueprints, the DNA of the organisms, you can get the particles to line up on their own. Living organisms are the only things which can cause particles to go where they want them to go. And while living organisms are constantly regenerating on their own DNA, things like the positions and shapes of eyes, noses and mouths hardly ever change.

Last year, a man who participated in WPI as a Junior PI, Dr. Teizer, utilized this idea, and attempted to find out what would happen if we used this method of using living organisms to make particles move. Living organisms move. Muscles move. There are things called kinesins which move above proteins called micro-tubulins. The thought was, what if we placed particles in these kinesins? Would they take the particles with them along the micro-tubulins? We came to this idea thanks to interaction with people from a different field.

K: That’s interesting. Like one of Aldous Huxley’s mechanisms using living organisms to move muscles, but making living organisms do the work. And all of it can be controlled by people.

A: It would be great if we could put this to practical use by joining our activities together with others little by little. This was an example of a fusion research with bio, but also with materials and computer science, we found fusion in our lab. Not only in our lab, but also with the outside, we found a fusion: More than two years ago, a mathematics professor, Dr. Kotani, and professor at Meiji University, Dr. Sunada, attempted to try to fuse their work with ours. That was the
time I first felt how amazing math really is. The research involved taking clusters shaped like the Mitsuya Cider logo, made up of three lines separated by 120 degrees which intersect at a point, and seeing what shape they would form if they were connected. On just hearing 120 degrees, chemists, and particularly those of us like myself who work with carbons, immediately thought that “120 degrees equals $sp^2$ hybrid orbital of carbon.” This is the carbon that graphite and graphene sheets are made of. I remember thinking that it was obvious that if you connected particles in the shape of the Mitsuya Cider logo that you would get a pattern made up of a series of rhombi. However, these mathematicians were thinking in three dimensions. And if you bend the shapes upward, and then bend them another 120 degrees, it seems you get a decagon. This was a shock. This was a totally new idea. Mathematics provided us with a new way of looking at things.

Sometimes when you meet someone from a different field you discover something so surprising it is like you have been hit on the back of the head. I think that this kind of shock is extremely important. With both fullerene and carbon nano-chips, we are talking about things that have been around for a long time. Discovering things that have been around for a long time and looking for these things are two completely different processes. Once people think that something exists, they will find it, because they will go looking for it. If you don’t think to look for something you won’t see it. What is important is that the new ideas you find be used to synthesize new materials into your research. This too is a type of fusion.

K: That wasn’t something you were taught, but something that you learned on your own I think. You didn’t move around for the three years of high school then?

A: I went to Toyonaka High School in Osaka. Luckily I didn’t change schools for those three years. I was a cocky student even though I almost never studied. Teachers used to get mad at me because I often wrote criticisms of their lectures in my notebook. I enjoyed being made to stand up and told to leave. But anyway, fortunately I would enter the University of Tokyo.

We were separated into different courses according to our sophomore year grades. At
that time, only those with good grades could enter applied physics, electric engineering or aerospace courses. On the other hand, my major, chemical engineering, had strange teachers promising to teach things like “methodologies for creation” so a lot of people with poor grades came to these courses. Even now, many of the people that I deal with are these kinds of people. However, among them are people who became the CEO of their company around age 40. They were different from people who have done nothing but study I think.

K: Just like in the world of living organisms, diversity is important, isn’t it? Diversity has helped living organisms to continue on for hundreds of thousands of years. I mean, if it was just fish, everything would have disappeared long ago.

A: It’s all about diversity which has come about through cross-fertilization though, basically. I think that this is especially why meeting a lot of different people is important.

K: During the Meiji Enlightenment, a lot of different people came to Japan from many different countries, and as things jumbled together we really grew, didn’t we?

A: Yes. At the end of the Edo period, those with ambition began trying to do what they wanted with their lives more and more. They didn’t just interact with those in their field, but exchanged opinions with many different people, found their ambition, and tried to accomplish something. Things weren’t separated into set groups, but were more free-flowing. Comparing it to entry into university, we now have the AO (Admission Office) route. I think that such interesting ideas are great.

K: I heard this story from a friend of mine from Stanford University. It seems that they purposely form their student body out of those with good grades and those with poor grades. Apparently this leads to a more effective education in the end.

A: I think that’s absolutely true. In such a situation you are forced to make you own environment. I have the feeling that for me, happiness is meeting many different people.

K: Even if you do meet people though, if you can’t absorb anything from them or really see who they are it doesn’t mean anything. And you as well have something which
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A: It's all about diversity which has come about through cross-fertilization though, basically. I think that this is especially why meeting a lot of different people is important.

K: During the Meiji Enlightenment, a lot of different people came to Japan from many different countries, and as things jumbled together we really grew, didn't we?
A: Yes. At the end of the Edo period, those with ambition began trying to do what they wanted with their lives more and more. They didn't just interact with those in their field, but exchanged opinions with many different people, found their ambition, and tried to accomplish something. Things weren't separated into set groups, but were more free-flowing. Comparing it to entry into university, we now have the AO (Admission Office) route. I think that such interesting ideas are great.

K: I heard this story from a friend of mine from Stanford University. It seems that they purposely form their student body out of those with good grades and those with poor grades. Apparently this leads to a more effective education in the end.
A: I think that's absolutely true. In such a situation you are forced to make you own environment. I have the feeling that for me, happiness is meeting many different people.

K: Even if you do meet people though, if you can't absorb anything from them or really see who they are it doesn't mean anything. And you as well have something which will be absorbed. The problem for young people today is how to find their own themes.

A: It is just like what I said about ski slopes at the beginning. People should do research in a field expressly because no one has done much research in that field yet. If I find out that someone in the laboratory is doing research into something that is already being researched, I try to convince them to gather their courage and stop that research. With new snow, it doesn’t matter if you are good at skiing or not. You might fall down or fall on your behind, and there might be a lot of holes in the field, but it doesn’t matter. Because no one is looking at you. You can steadily go where you want to go.

I spoke about one of our nano-particle synthesis methods earlier, supercritical hydrothermal synthesis. This method was born out of a discussion with a student. For students as well, it is important that those with more experience encourage their classmates below them to come up with new ideas themselves.

I can’t do anything as grand as teach ambition, but I do think that students are always watching our actions. When they see someone working really hard on something, they begin to understand what it’s all about. For this reason, I have called many people to my new laboratory and had them study organic fusion. I think that their studies and ambition will meet up eventually.

In a supercritical high-temperature-high-pressure environment, oil and water completely mix together. Oil and water are known as a classic example of two things that just don’t get along, but in a supercritical environment they completely mix. If organic and non-organic synthesis reactions occur in the midst of this environment, I think that we will be able to do some amazing things. Oil and water only mix in this kind of environment, and that makes me think that in a supercritical environment especially we will be able to achieve these synthesis reactions. This is another challenge for us. In supercritical environments we have produced materials that are hybrids between organics and inorganics. Because we were able to create such materials, mixes of organics and inorganics – well, let me show you a polymer. This might look like a plastic, but actually its 70% zirconia with nanoparticles added to it. Although it may not look it because it is clear and flexible, it has a high refractive index.
When you think of ceramics you think pottery, but this is also a ceramic. Except it is completely mixed with non-organic nanoparticles.

K: You might even be able to make a polarized filter I suppose.

A: I think that we will be able to. For instance, I think we might be able to do it through diffraction grafting. We are talking with the research group of Terunobu Miyazaki, the expert in magnetism, to see if we can’t make a translucent magnetic film. Dr. Hitosugi and Dr. Mizukami proposed to explore a Faraday effect on the film. Dr. Hashizume and his group as well are asking if we can’t incorporate this in their devices – interacting with different people really is interesting, isn’t it?

K: Yes, it’s really interesting. We really must do it.

A: Currently a 4 billion yen national project “Super Hybrid Materials” is underway at the IMRAM. By adding boron nitride to thermal conductive materials, we can make paper-thin films with thermal conductivity 10 times better than what was once possible.

K: This is a fusion of materials. Type II diamonds have several tens of times higher thermal conductivity than copper does. So adding powdered diamond will raise thermal conductivity even more. I was actually the one who found that type II diamonds are mostly found among smaller diamonds. The discovery of type II was announced about 100 years ago by three physicists in England. Type II diamonds are extremely transparent with regard to ultraviolet light and their thermal conductivity is in a class of its own. However, they are extremely rare in nature. Only about one in every 200 diamonds are type II and therefore, they are expensive. However, when mining diamonds, sand containing diamonds are run down a grease table and the sand is washed away, but small diamonds that can become abrasive compounds stick to the grease. One day I was examining them through ultraviolet absorption, and I discovered that more than 10% were type II diamonds that are transparent in ultraviolet light. After I published this in Science, the discovery was put into practical use at the Bell lab for heat sinks.

A: I will use that idea right away.

K: Later, S and other companies were able to synthetically produce type II diamonds.
A: I was actually supposed to go to a private company and to make synthetic diamonds.

K: I think you are more suited to the university environment. You were able to expand your research because you were at a university.

A: Currently I am challenging to synthesize metallic glass nanoparticles by a chemical route. First of all, it’s important to try, especially in fusion research, because communication will lead to new ideas. Answers may emerge from completely different fields with someone saying “normally, we do it like this, but we tried it this way and came up with something interesting.” I am now achieving fusion with many professors within the WPI.

K: Finally, let me ask you why you decided to go into the sciences.

A: I have a great deal of curiosity for everything. I like things that have a solid logic behind them. I’ve always liked mathematics and science since elementary school as well as puzzles and mathematical quizzes. While I was studying in high school, I realized that what I had thought to be clearly logical up until then, was actually not. I don’t really know if I am a man of science though.

K: That is fine. You won’t know by the time you die.

A: I actually also enjoy cultural things such as paintings and music. So, I might be classified as science-oriented, if I had to choose between science and humanities, but I think everything is fusion.

K: Indeed. And you head in the direction for which you are best suited.

Thank you for your interesting interview today. I would have liked to have heard these stories a long time ago.

A: Chit chat is more interesting. I had a fun time.
K: Thank you.

Interviewer: Prof. H. Komatsu

At Adschiri Lab,

Advanced Materials Processing Building

IMRAM, February 16th, 2010
出会が生みだすフュージョン研究

阿尻：スキーの話ですが、スキーにも楽しみ方が二種類あると思います。一つは大勢の人
前で格好よく速く滑る。もう一つは、誰も踏み入れたことのない新雪の中を、尻もちを
たきながら進んでいく。何があるかはわからないけれど、その先にきれいな景色が見えた
り、振り返ると後ろからいろんな人が来ていて、皆が楽しむゲレンデになっていったりす
るような楽しみ方です。簡単に言うとナンバーワンを目指してしのぎを削って探求をする
か、オンリーワンを目指すかですね。両方とも非常に重要ですが、どちらかというと、新
雪を滑るような研究が私の目指すところです。

私は新潟で生まれました。銀行マンだった父の都合で、引っ越しが非常に多かったです。
幼稚園も変わりましたし、小学校では毎年引っ越します。子供心に「またかよ」と思いま
したが、友達をつくるのが上手くなりました。人とつながる仕方はDNAで決まった性格
もあるでしょうが、生い立ちによるところも大きいと思います。ある時人からそれを指摘
され、自分の生い立ちを振り返ってなるほどと思いました。

小松：子供は非常に柔軟ですから、環境に応じて考えるのでしょうか。

阿尻：各地を渡り歩いて苦労もしましたが、楽しかったです。実は来週も小学校の同窓会
があります。同級生は皆全然違う分野ですが、考え方をチェンジする意味でも違う環境の
人と会うのはいいですね。

小松：それは大事ですね。大人になってフィールドの違う人とはなかなか仲間になれませ
んが、子供は逆です。顔合わせていると仲間になる。自分の考えとは別の考え、世の中に
はたくさんいろんな人がいるということは、後からプラスになりますね。それが今の研
究にも通じているのではちょっと。

阿尻：そうですね。例えばこの部屋にしてみてもそうです。ある大学の文系の先生が、ゼ
ミを時々喫茶店でやると、教室でやるとは全然違う発想が出てくると言っていた。よ
って喫茶店にはＢＧＭが流れている、緑があり、絵が飾ってある。教授室を作る際、そんな場
所を意識しました。廊下にも絵を飾っています。実験結果や新聞記事が掲示されているの
でもいいですが、学生やスタッフが見て少し違う発想がパッと出てくる研究環境もいいと思
いました。違う頭を使う環境に身を置くことが大切ですね。

高校から東大に入りました。先生は国井大蔵先生です。流動層といって、砂を下から
空気で吹き込むとちょっとによく動く発想が出て来ると言っていました。よ
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高校から東大に入りました。先生は国井大蔵先生です。流動層といっただ、砂を下から
空気で吹き込むとちょっと液体のように沸騰したような状態になり、一つのリアクターか
ら別のリアクターにその砂を動かすことで、液体と同じように移動させられます。この技
術によって、触媒を使って大量の油を処理する今の石油化学技術ができました。固体を液
体と同じように扱える新しいプロセスをつくり上げる装置工学で、国井先生はその大家です。もともとはアメリカでできた技術ですが、国井先生がその中で起こる反応を初めて設計できる形にしました。

小松：媒体を変えすることで比重を変えることもできますね。

阿尻：そのとおりです。先ほど沸騰するような、と言いましたが、沸騰でもバブルができない状態や、全部吹っ飛んでいくような流動など流動状態をつくることができる。それがプロセス全体のパフォーマンスを決めたりします。

小松：固体に圧力をかけても均等にかかりませんが、液体であればバパスカルの原理で均等にかかりますし、電場、磁場の流れを制御できますね。

阿尻：磁場をかけて制御することは研究室で最後の方に最先端の研究として行われていました。まさにその発想です。周りの研究を知り過ぎてしまうと、そこから抜けられない。逆に、全く違う分野の人が「こんなことはできないのか」と言う方が、発想そのものを変えられます。

小松：それこそフュージョンですね。

阿尻：自分一人でも環境を変えればフュージョンはできるのです。30年前のオイルハウスの頃、ドクター論文で流動層の中で石炭をガスにする研究をしました。炭素と水蒸気を反応させ、COと水素に変える。固体が小さしずつガスに変わっていく際の内部構造の研究です。ドクターが終わった頃には材料の研究がアクティブになり、CVD（Chemical Vapor Deposition）今度はガスから固体をつくる研究を始めた。今までとは全く逆のことをやればいいので、とても簡単にでした。石炭の構造、固体の構造がどう変わるのかを見ればよかった。

ところが研究をしていた最後の頃、面倒を見てくださっていた、国井大蔵先生のお弟子さんの古沢健彦先生が、49歳の若さで亡くなりました。私はドクターを卒業して1年間学振の研究員をした後に助手になったのですが、2年目になってしまいました。そうすると、上の先生がいなくなった研究室はお取りつぶしです。1年間の猶予はありませんが、学生の配属を替える世話で1年間が過ぎてしまいました。ですからそのときの1年間は論文ゼロです。

何にもできず困っていると、「うちに来ないか」と言って下さった企業がたくさんありました。ほとんど企業に行く予定でしたが、東北大学の新井邦夫先生と斎藤正三郎先生がいらして、東北大学に誘って下さった。それだったらお世話になろうかな、と平成元年に東北大学に来ました。きちんと仕事をしていれば見ていて拾ってくれる人もいるのだと思いましたね。

東北大学に来てからは今研究の基礎になる、超臨界、高温高圧のガスの状態の研究をしました。化学工学はそのプロセスの開発・設計の学問で物性推算が必要になります。高圧、特に臨界点において、その温度以上では凝縮しなくなる、圧力をかけても液体にならずに高圧のガスのままでいられる状態ができる。この近辺のところは物性が大きく変化す
るので、感度が一番高いわけです。

ちょうどその頃、斎藤正三郎先生がアメリカから研究を輸入し、東北大学を日本の中の出発点にしました。超臨界の物性研究がスタートし、私のポスだった新井邦夫先生とその研究に取り組みました。密度を変えれば溶かす力を変えられるので、それを使って物の分離に関する研究をしました。石炭で反応の研究をしていたところから物性、平衡論ですから、研究内容はもちろんです、発想そのものがあればいます。学生の頃にあまり得意ではなかった熱力学を専門にするので大変な思いをしましたが、せっかく拾っていたので一生懸命勉強しました。

チャレンジするのであれば、今までのものにしがみついているよりゼロスタートの方がいいと思い、石炭の研究をやめて新しい研究をスタートしました。それまで超臨界を研究している人は、分離の研究ばかりで、「超臨界×反応」の研究はありませんでした。同じ分野にいる人は見えなくなる。だから一人フュージョンができたわけです。「超臨界×反応×対象物質」ですね。石炭や重質油、バイオマス、廃棄物処理、有機合成反応など全てです。それから、現在研究しているナノ粒子合成です。平成元年に同時並行で片っ端から全部やることになりました。

小松：なるほど。古いしがらみをパッと捨てて転換したことで、見る目がフレッシュになったわけです。

阿尻：そのとおりだと思います。今までの研究をしていたら、この発想は出てきませんでした。全部超臨界でやると決めた瞬間、自分がやらなければならない仕事が見えてきた気がします。

国井先生は「創造のための方法論的研究」を研究テーマに掲げていました。創造のための発想とは演算だということです。真にゼロスタートのオリジナルな発明は非常に稀で、新しい発想は既にあるアイデアの掛け算や足し算、引き算、割り算、転置行列だったり変換行列だったり、そういった演算でできていることが多いそうです。それを常に頭に入れて新しい発想を出しやすいと教えられました。先ほどの発想はまさにそれを適応した、自分にとっては簡単なことでした。

もう一つは、自分だけで考えないことです。今のメインテーマは超臨界の水の中でナノ粒子を作る研究ですが、これは学生と一緒に出てきた発想です。超臨界の水では、高圧高密度の水蒸気の中に物が溶けていきます。金属酸化物は大体溶ける。もっとも地球はそうしてできています。海底火山の地殻は高温高圧状態ですから地下水が超臨界です。

脱線してしまいますが、地球物理の先生と話す機会がありました。マグマは岩が溶けたもの（溶岩）だとばかり思っていたのですが、そうではなく超臨界の水の中に岩が溶ける。そうすると、岩の性質を持った水、岩らしい水ができ、周りの岩をどんどん溶かし込んでいく。少しの水にくたくさんの岩が溶け込んだ状態が溶岩、マグマです。岩の融点よりも低くてもマグマが存在して流動している。それが突然上に上がってくると、高圧の水蒸気が一気に普通の大気圧の水蒸気にに戻りますから、全部膨張する。それが水蒸気爆発、火山爆
発です。関東ローム層がありますが、それは「自然のつくったナノ粒子」です。富士山、浅間山の噴火で溶けていた岩が一気に全部減圧されて溶けなくなったり、全部析出するしかないので、それらを微粒子として析出したのが関東ローム層です。徐々に上がってくると鉱脈になります。溶解度の低い順番に析出していてから、同じ鉱脈には必ず同じようなものが析出しします。

小松：ただ、関東ローム層の中には、輝石やカンラン石など、パウダーではなく小さな結晶になっています。まだ精製できるような条件が、マグマからいきなりドーンと上がってしまって、何かで条件が変わりながら何か変わっていっているのですね。

阿尻：そのとおり。まさに自然が合成したナノ粒子そのものだと思います。

仲：ちょうどそれに近い研究をしていた学生がいました。彼はその研究がとても嫌だったらしく、私に相談に来るのです。作っていたのはシリカですが、溶けるとしてもせいぜい1wt％で、大量の水を扱って耳かき1杯程度しかできません。そんなものが本当に役に立つのかと言うのです。確かに大量に溶けるものならばまだしも、大変なプロセスを動かして耳かき1杯のシリカだったらそれは面白くないと2人で議論し始め、おしまいには飲み会になった。

溶けるといえば金属塩ぐらいしかない。でも待てよ、金属塩が溶けるのだったらそれでいいんじゃないかと言うと、酔っていたはずの学生がパッと図書館に行って調べた。そして「先生、だめです。金属塩は高温にしてしまうと熱分解して金属酸化物に変わります」と言う。目的が金属酸化物をつくる研究だったのだからそれでいいんじゃないかと私は半分冗談も含めて投げやりに言ったが、その学生はすぐに金属塩が流せる高温場で反応ができるよう装置に切り替えました。それが初めての東北大学オリジナルのナノ粒子合成プロセスです。電顕で見たらきれいで、こんなにきれいな粒子ができるのかと思いました。

自然と同じことを、減圧ではなく急速に温度を上げる方法でやったのです。流体を流しながら急速に温度を上げて反応させていました。これによってほとんど同じものができる。オートクレーブという非常に分厚い大砲の筒のようなお釜だと、徐々に温度が上がっていきます。それが、石油化学のように全部流体として流し、中で反応させていく方法を使ったことで、均一できれいで、こんなにきれいな粒子ができるのではないかと思いました。

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小松：ジェネレーションの違う者同士、教育を受けている人間と、教育をする、でき上がった人とのインタラクションが大事ですね。

阿尻：私も入ってきたばかりだったので、その研究に完全に染まり切っていないフレッシュな頭で物を考えられた。上下の関係ではなくて、自由な発想が出たのでしょう。それなら、フロアで違う分野の研究をやる時は全てフレッシュですね。新しい発想や技術が生まれる可能性を大きいに秘める気がします。WPIではそういう面でいい融合の場を提供してくれていると
と思います。

小松：幸いなことが多いディシプリンが三つあるから、本当に頭を柔軟にして融合したら面白いかかもしれないですね。

阿尻：７年前に青葉山の工学部から多元研に移り、また新しく研究室を作ることになりました。幸いにしてボストが全部空いており、准教授、助手、技官の３名と一緒に仕事をする場ができそうで、その時も全く新しかったことしようと思いました。

超臨界で20年近く研究をされてきたので、似たような研究をしていた人が来てもなかなか発想の刺激にはならないと思い、自分にないもの、コンピューターケミストリー、バイオテクノロジー、マテリアルサイエンスの人を呼びました。博打でしたが、平成元年に東北大に移ってきた時と同じように「超臨界×バイオ」など、新しい発想の融合の場を作ろうと思ったのです。

ただ、バイオは略語が多く、違う国の人が詰まっているようでした。初めの１、２年間はお互いに言葉が通じず、基礎の基礎からレクチャーをする教育機関を作りました。自分が知りたいというから、無理やり授業に出ていた学生の頭をとっていくとどんどん頭に入っていた。こんなことはできないのかとお互いにどんどん投げかけをするのです。

そこでは、現在工学部で准教授として活躍している梅津君が、ナノとバイオの架け橋をしてくれました。我々がつくる粒子を認識するペプチドはアミノ酸が十数つつながったものです。例えば酸化亜鉛だったら酸化亜鉛しか、しかもその（100）面しかくっつかない、そういうペプチドを見つけてくるのです。彼は、10の９乗個、10の９乗種類のペプチドを準備して、それを全て実験しました。これはすごいですね。遺伝子操作ができる。ウイルスは細胞に感染するのですが、そのウイルスのDNAを、10の9乗種類の違うDNAを少しずつウイルスの中に入れていきます。入れたウイルスをこの粒子にくっつくかどうかを見るのです。洗い流せばくっついたかどうかがわかるので、これは簡単です。しかし、１個や２個しかくっついていないものを見つけるのが大変なのです。ところが、これはウイルスですので例えば大腸菌に感染させてやってと。

小松：ウイルスは遺伝子の固まり、核酸の集合体でしょう。

阿尻：そう、ウイルスは生きていない、生物ではありません。自分の中にDNAを持っていて、そのDNAを注入する機能を持っている。注入された細胞は自分のDNAだと勘違いし、自分の細胞の中でどんどん増えていき、ウイルスだらけになる。これが細菌感染・ウイルス感染です。たとえ１個か２個しかいないなくても、ウイルスを大腸菌に感染させると大腸菌がどんどんウイルスを増やしてくれる。その後にウイルスを取り出してDNAを調べれば、どんなペプチドがくっつくのかが見える。この方法がファージディスプレイです。この方法を使っていろいろなものを作りました。

こうした時、デバイス屋さんはまた違った発想をします。ウイルスの足に提示させていくペプチドを、頭や手、肩にもくっつけるかと言われたのです。これができることですね。彼の発想によれば、ここにいろんなものを提示させておけば、ここにソース、金属で
すね、ドレイン、反対側にして、このところに半導体の粒子をくっつけて、この間のところには誘電体をつける。こういう回路とかなんとか自分で勝手に並べられるのではないかと。我々は粒子を持っています。ナノ粒子を自由な形に並べられたら回路ができる。自在に並べるには生物に並べさせればいい。必要な設計図であるDNAを書きかえればその粒子を自在に並べられるという発想です。粒子を自在に自分の思いどおりの場所に置くことを唯一できるのが生物です。生物は自分のDNAで常に代謝しているわけですが、目、鼻、口の位置、形はほとんど変わりません。

昨年Junior PIとしてWPIに参加したTeizer先生もそういう発想をつなげて、今これをさらに動かしてみたらどうかとなっています。生物は動いている。筋肉もそうです。キネシンとはチューブリンというタンパクですが、その上を走るのです。そこに粒子を入れておけばこの粒子を連れて回ってくれるのではないか。発想が違う分野の人が出てきたときにでき上がってきます。

小松：それは面白い。オルダス・ハクスリーの筋肉を動かすメカニズムをうまく使って、生物にそれをやらせる。それを人間が全部操るということですね。

阿尻：少しずつ組み上げていって応用ができればいい。現在彼らは、別の場所で活躍していますが、隔れていても場所は関係ありません。一度フュージョンを経験した人はまた違う場所でも自分なりのフュージョンをしています。

二年ほど前から数学の小谷先生、明治大学の砂田先生と数学とのフュージョンをしていきます。数学のすごさを初めて感じました。三ツ矢サイダーのような120度の角度をつなげていくとどんな形になるのかという研究です。120度と聞いただけで、化学の人、特に私のように炭素を扱っていた人は、「これは炭素でいえばグラファイト構造、グラフェンシートができています。三ツ矢サイダーの構造はつなげていけば六角形が敷き詰められた形になるの当然だと思っています。しかし彼女たちはこれを3次元で考えたのです。上に120度で曲がって、さらにまた曲がり、十角形になるらしいです。これはショックでした。我々は装置の設計などで数学には慣れていたつもりですが、発想そのものが全く新しい。数学が新しい視点を与えてくれました。

違う分野の人と出会うことで頭をたたかれたようなショックを受ける、これが大切だと思います。フラーレンやカーボンナノチューブも同じで、昔からあったわけです。あったことと、それが見つかったこと、探し出すことは違います。あると思った瞬間に皆が探し出すから見つかるのです。見ようと思って見ないと見えない。大切なのはそういう発想が新しい材料を合成する研究に向かわせてくれたことです。そういう意味でもフュージョンですね。

小松：それは誰からも教わらない、自分自身が学び取った方法論ですね。高校は三年間かわらなかったのですか。

阿尻：高校は大阪の豊中高校で、幸いにして三年間にかわりませんでした。ほとんど勉強をしないのに生意気で、先生の講義の講評をノートに書いてよく怒られていました。立たさ
れたり、「出ていけ」と言われありましたが楽しんでいました。

大学では２年間の成績で各コースに振り分けがありました。当時は応用物理や電子工学、航空宇宙には成績のいい人しか入れない。私が入った化学工学は、「創造のための方法論を教える」なんてことを教える変な先生がいて、成績の悪い人もたくさん来るわけです。これでどうこう言えるわけではありませんが、四十幾つで社長になった人もいますし、勉強だけをした人とはちょっと違うと思います。

小松：生き物の世界と同じで多様であることが大事ですね。生き物が何百万年も続いているのは多様性があるからで、これが魚だけだったらもっと消えてしまいますね。

阿尻：基本的には交配して変わっていった中での多様性ですね。だからこそ、いろいろな人たちと出会える場が大切なのでしょうか。

小松：明治の文明開化した頃はいろいろな国から人が来て、ごちゃごちゃしながらも伸びていきましたよね。

阿尻：そうですね。末段の頃から、志を持った人が自分でやりたいことをどんどんチャーでンジしていました。同じ分野の中だけでなく様々な人と意見交換をしながら、最後には自分の志を見つけて挑戦していった。輪切り的な採択ではなく、もっとフリーな形です。入試の方法としてＡＯ入試が出てきていますが、そういった面から見ると確かにそうですね。

小松：スタンフォード大学の友人から聞いたのですが、わざわざ成績のいい人と悪い人とを組も合わせているそうです。その方が最終的には効率のいい教育ができるそうです。

阿尻：そのとおりだと思います。そういう場を自分の周りでもつくらなければいけないですね。私は幸せにもそういういろんな人たちと出会えた気がします。

小松：出会っても、ご本人に吸収力や見る目がなければ意味はありません。自分自身が吸収するものを持っていたのですね。今の若い学生の問題は、どのように自分でテーマを見つけ出すかですね。

阿尻：初めにスキのゲレンデの話をしましたが、同じことだと思います。他人にほとんど研究されていない分野だからこそやる。研究室では、誰かがしているとわかったら勇気を持ってその研究から撤退しようと言っています。新雪であればどんなに下手でもいいのです。転んでしもうちをついて穴があいているとしても、全然気にすることはない。周りに見ている人はいませんから。着実に自分たちが行きたいところに進めます。

先ほどご紹介した超臨界水熱合成という我々のナノ粒子合成法、これは学生とのディスカッションから生まれた方法です。学生でも新しいものを自分で生み出せると後輩たちに伝えなければいけませんね。

志を教えるなどという大それたことはできませんが、学生は我々の背中を見て生きていると思います。チャレンジしている姿を見て、「なるほど、こういうものなのか」と学ぶ。だから、新しい研究室ではいろいろな人を呼んできた他に、有機の合成の研究もしていました。いつかこれを二つ結びつけることができると思っていました。

超臨界の高温高圧の水の中では水と油が完全に混ざっている。「水と油」は仲の悪い代
名詞ですが、超臨界では完全に混ざり合います。混ざり合った中で有機合成反応も無機合成反応も起きるならば、とてつもないものができると思いました。水と油が混ざるのはこの領域しかありませんから、超臨界でこそできるものがあるはずだと思ったのです。これも一つのチャレンジでした。それでできたのが有機と無機がハイブリッドした材料系です。これができたことで、有機と無機、例えばポリマーですが、ちょっとお見せしましょう。

小松：偏光フィルターもできるかもしれない。
阿尻：できると思います。例えば回折格子をつくるようなこともできるでしょう。それから、磁性の専門の宮崎照宣先生のグループと、フィルムにして透明に磁石はできないかという話になっています。橋詰先生たちはデバイスに加工できないのかとコメントしてくださいますし、やはり違う人たちが出てくると面白いですね。

小松：本当におもしろい。実際やらなきゃいけないな。
阿尻：今、40億のナショナルプロジェクトが多元研の中で動いているのですが、熱伝導材料、ボロンナイトライドをこの中に入れることで熱伝導が今までの10倍高い紙のようなフィルムを作っています。こういうものもできると思っていませんでした。ただの紙に見えますが、熱伝導が今までの10倍です。
小松：これは材料のフュージョンですね。タイプⅡダイヤモンドは、熱伝導は銅の十数倍高いので、ダイヤモンドのパウダーを入れたら熱伝導率がさらに上がります。タイプⅡが小さいダイヤモンドに多いことは実は私が発見しました。タイプⅡダイヤは100年くらい前にイギリスで3人の物理学者が連名で発表しています。タイプⅡは紫外線にすごく透明で熱、伝導がすばらくされてい。ところが、自然の中に大へん稀で200個に1個ぐらいしかなく、非常に高いと言われていました。ところで鉱山からダイヤモンドを取ってきてグリーステーブル上にダイヤモンドが入っている砂を流すと、砂は洗い流され、研磨剤になる小さな砂粒のようなダイヤモンドがグリースにつかまります。ある時それを紫外線吸収で調べたら、なんと紫外線に透明なタイプⅡがなんと10%以上あった。このことを「Science」に発表したら、ベル研究所でヒートシンク(熱吸収板)に使うアイデアが実用化されました。
阿尻：それは早速使わせてもらおう。
小松：タイプⅡは、その後、S社などで人工合成できるようになりました。
阿尻：私は、民間企業に行く予定でそこで人工ダイヤモンドを作ろうと思っていた。
小松：大学の方が先生に合っています。大学だからこそ広げられるのです。
阿尻：今は、メタリックガラスのナノ粒子をつくってそれを分散させる研究をしています。まずはやってみることが大事です。特にフュージョンリサーチは、やっている間にコミュニ
名詞ですが、超臨界では完全に混ざり合います。混ざり合った中で有機合成反応も無機合成反応も起きるならば、とてつもないものができると思いました。水と油が混ざるのはこの領域しかありませんから、超臨界でこそできるものがあるはずだと考えました。これも一つのチャレンジでした。それでできたのが有機と無機がハイブリッドした材料系です。これができたことで、有機と無機、例えばポリマーですが、ちょっとお見せしましょう。これプラスチックスのように見えますが、これには70%ジルコニアのナノ粒子が入っています。透明でとてもそうは見えないですが、屈折率が高いのです。セラミックスとは陶器ですね、これそのものなので、無機がナノ粒子であってポリマーと完全に混ざります。

小松：最後に一つ、理系に進んだ理由を教えてください。
阿尻：ロジックのしっかりしたもののが好きだったからです。小学校の頃から数学や理科、それからパズルや数学的なクイズも好きでした。特に必ず答えが出るものの方が好きでした。実際には、受験勉強をしているうちに、今までシャープさと考えていたことはちょっと間違っていたと気付きました。本当に理系なのかどうかはよくわからないですね。

小松：それがまだいいのです。死ぬまでわからないです。
阿尻：どちらかといえば、絵や音楽など芸術的なことも好きです。ですから理系、文系という言い方をすると理系になるのかもしれませんが、やっぱりすべてフュージョンなのではないでしょうか。

小松：そうですね。その中で性に合った方に進むわけです。
阿尻：本当にこうした話の方がおもしろいですよね。楽しいひとときでした。
小松：ありがとうございました。

2010年2月16日
東北大学多元物質科学研究所素材工学研究棟 PI阿尻雅文教授室にて

小松 総

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Interview with Professor Kazue KURIHARA, WPI-AIMR Principal Investigator

“When the “Maybe Girl” Commits herself….”

Administrative Director Iwamoto (I): First, as you were appointed the first female Principal Investigator of WPI-AIMR back in April, I should have come earlier to have this interview. So Professor, I understand that you were not born in Sendai.

Professor Kazue Kurihara (K): I was born in Tokyo.

I: And you also grew up in Tokyo?

K: That’s right. In fact, I lived with my parents in Tokyo right up until I went to the United States to be a post doctorate fellow.

I: So you are a true Edo Tokyoite.

K: Well, not strictly speaking an Edo Tokyoite as Shinagawa is considered uptown Tokyo.

I: And you graduated from the Faculty of Science, Ochanomizu University?

K: Yes, from the Department of Chemistry in Faculty of Science.

I: And I suppose you were interested in science from an early age?

K: Not really. If I would have had to choose when I was young between the humanities or science I think I would have said the humanities because I enjoyed reading books and writing essays. However, rather than taking a sort of rational approach to the humanities, I wanted to study in fields that were a little more empirical, such as history or archeology. That was the basic image I had of myself. But my parents told me I wouldn’t be able to make a living in those fields, and the image they had for me was to study medicine or pharmacology if I entered a science department or perhaps to study law if I entered a humanities department. As I originally wanted to be
an archeologist, it was a major concession for me to enter the Department of Chemistry in Faculty of Science.

I: That was certainly a major concession.

K: I did also want to study science a little too. It was at that time that the position of science writer was slowly starting to become quite fashionable.

I: Yes, I remember.

K: As I was interested in book publishing, I thought it would be good idea to study science and then to work at a publishing company. That was how I felt at the time, and so I continued through education in a sense of liberal arts. A lot of my classmates enjoyed in amateur radio or joined science clubs.

I: But in your case, you wanted to help communicate scientific knowledge to the general public?

K: Not really. I had no so much experience of science outside of studying it at school, so I wanted to know a little more about the science around me. As for humanities, I quite often used to go to the library and research and study them.

I: So you had continued from the Faculty of Science on to its Master’s course at Ochanomizu University. Your specialty had been formed from your Master’s degree to the School of Engineering, the University of Tokyo.

K: I was actually a member of a humanities circle when I was at university. Quite a few of the other members were studying aesthetics and philosophy, and so whatever we were discussing they were always on a completely different level to me. So based on this experience I decided to give more emphasis to the things that I had been studying. We conducted a student experiment in physical chemistry in my third year which I found very interesting. Starting from measurement – and this applies just as much today – we can understand the nature of molecules. Even today, I remember this experiment clearly; we measured the dielectric constant to understand the molecules’ dipole moments, and by observing the properties of the solution we were able to understand in detail the qualities of the substances dissolved within the solution.
thought that these kinds of measurements were very interesting.

I: So this was your first encounter with measurement.

K: From then on I found the student experiments in physical chemistry extremely interesting and so I went to the physical chemistry laboratory. At that time, I still did not intend to become a researcher in the future and so at graduate school I remained in the same physical chemistry group and studied interface chemistry in the laboratory for my Master’s degree. It was during my Master’s degree that I realized I found research based on observation to be the most interesting and so carried out the kind of research that is based on observation and measurement, and published one paper. So even when I was a student I was able, to a certain extent, to do research that appealed to me. As a result I found my research interesting, and although originally I thought I wanted to be an archeologist, I think that perhaps what I really wanted was to be a researcher.

I: So from the start you had a passion for research?

K: My classmate said that it was uninteresting if at least one person in the class did not go on to do a doctorate and encouraged me to continue on with my studies. At that time, this classmate talked to an associate professor who was her supervisor that I should continue onto a doctoral program. The two of them discussed it and suggested to me that I might enter the University of Tokyo for studying at the group of a professor who was giving a course at Ochanomizu University.

We knew there had been an example of entering the doctoral program at the School of Engineering in the University of Tokyo from another university. So, I applied for and entered an industrial physical chemistry laboratory because their research was similar to interface chemistry. It was not really so spontaneous, though I did my absolute best to pass the tough entrance examination, then study at doctoral program.

I: I thought it interesting when you said that in your case, it was precisely because you were a member of a humanities club that you decided to do your absolute best and study science. It seems that what you wanted was to do something based on observation that could be empirically verified.

K: That’s right. I have wanted to be an archeologist or a researcher since high school,
and I think they are somewhat connected with what I do today, precision measurements.

I: Although archeology is within humanities there is certainly lot of field work required.

K: Science means proof and it can be hard work acquiring this proof. In some ways the precise measurements required are something like a sport. That is why I enjoyed it.

I: So you continued on to a doctoral course; what was your PhD about?

K: It was on biomimetics, but what has been a common theme throughout my career has been the self-organization of molecules. My master's thesis was about micelle, which is the aggregation of surfactants (surface active agents) and is therefore the principal ingredient in detergent, and liquid crystals. After that, my doctoral thesis was about the lipid bilayer membrane. I used a biomembrane model for my research. I modeled leaf photosynthesis by adding chlorophyll $a$ into the model membrane and then irradiating light. Various elements of this research were the first of their kind in Japan. My sub-supervisor had researched the lipid bilayer membrane in the United States, and we were the first in Japan in using the lipid bilayer membrane in chemistry and conducting researches on reactions within the lipid bilayers.

I: Fundamentally speaking, biomimetics is replicating, which is to say mimicking, living organisms.

K: That’s correct. It is the creation of artificial systems that mimic functions within living organisms. So an example of biomimetics in its purest form is an airplane, as a plane mimics a bird. However, that is not at a molecular-level mimetics; mimicking leaf photosynthesis would be biomimetics at the molecular level. Photosynthesis occurs from a reaction between sunlight and the chlorophyll in the chloroplast membrane in a plant leaf. In the same way, the goal of the research that I was involved in at that time was to convert light energy into chemical energy and then collect this energy within a chemical compound. This has recently once again become an important area of research as it can contribute to achieving a low-carbon society. The oil shocks were taking place when I was involved in this area of research.

I: That’s right; the first oil shock was in 1973.
K: This area of research was attracting a lot of attention after the first oil shock as it was the first into energy conversion systems. It was also about that time when the field of biomimetics began to emerge. Research into biomembrane models and enzyme models had just begun, and the materials used in interface chemistry were exactly the same as those used in areas such as biomembrane modeling in biomimetics. As interface chemistry deals with structures made up of aggregations of various molecules, I was involved in this area of research from the start.

I: Currently, there are various scientists within WPI-AIMR that are researching interfaces; it became a globally important field at the end of the 1970s, didn’t it?

K: That’s right. Although the meaning of interface chemistry changes over time, it is a scientific discipline with quite a long history. Aggregate structures of lipids and surface active agents were used for biomembrane model in the 1970s, as lipids have the same kind of structure as surface active agents. It was exactly at that time that the ideas were emerging about organizing molecules and designing various functional structures, such as to control the distance of a reaction. This was the first time these ideas had been expressed and they serve as the starting point for the development of the different nanotechnologies of today.

One such starting point was research into molecular self-organization, while another was research into enzymes. The background to this research was that X-rays had revealed the structures of living organisms, biomembranes, and enzymes. This was extremely significant for the research occurring at this time as it enabled these new materials to be created.

I: So new methods of creating materials, and also methods of observation, were being realized.

K: Of course today we are at a considerably more advanced stage, but that period of time was the starting point. Today, nano-level materials are being used in industry and interfaces have come to be considered extremely important. Although previously really only a small number of people were involved in this field, today everyone is involved and consequently the outermost boundaries are constantly being pushed further and further back.
I: Is it related one to your research themes, surface force measurement.

K: I received my doctorate in 1979. My doctoral thesis was based on photoreaction experiments, and when I then went to America as a post doctorate fellow I entered the laboratory of the biomimetics specialist Professor Fendler, who was proposing the concept of membrane mimetic chemistry and researching how to create superior materials that mimicked biomembrane functions. Professor Fendler’s laboratory had many instruments for measuring fast light reactions. Among them was a Laser Flash Photolysis system, which at that time was the most advanced research instrumentation available. I wanted access to this equipment so joined Professor Fendler’s laboratory.

Research had commenced into the creation of nanoparticles in special environments. So next I began creating nanoparticles within organized molecular structures. I returned to Japan, and spent two years at Department of Synthetic Chemistry, Kyoto University, in the laboratory of Professor Iwao Tabushi, who was the leading researcher in the field of biomimetics.

During this part of my career I began to think about the research I wanted to do for my career. Up until that point, I had created or designed various functional materials. I was not synthesizing molecules but rather combining component molecules to design a functionalized system. As I was originally a physical chemist, my research involved measurements; namely, measuring and identifying characteristics of its advantageous functions. As I do not synthesize molecules, my approach for improving the functions could be rather limited when I discover interesting functions. Rather than this type of research, the research themes that I wanted to pursue are those that would lead me a special stage after accumulation of experiences, say, for 10 years.

I thought that I might find it in Physical Chemistry. I encountered surface force measurement when I was trying to find someway of understand molecular nanoarchitectures and self organization. Self organization indicates the properties demonstrated by a molecular aggregation, and as the molecules interact to aggregate, I thought it would be interesting to use surface force measurement to try to understand this system. The laboratory in Sweden that I had moved to
had installed surface force measurement equipment and I was able to conduct a joint research on this measurement.

I: That was at the Institute for Surface Chemistry in Sweden?

K: Yes, but in Japanese we call it Institute for “Interface” Chemistry.

I: Professor Fendler who directed your research when you were in the United States was then the Texas A&M professor?

K: Yes, during my stay in the US, he moved to Clarkson University with his group in exactly the same set up.

I: You stayed in the US for a total of three years, and then went to Sweden, is that right?

K: Yes. More precisely, I returned to Japan for two years, and then two years in Sweden.

I: Apart from the research results you achieved, what significance do you think your experience of researching overseas had in forming you as a person?

K: Professor Fendler frequently said to me that I needed to commit myself.

I: What did he mean by commit yourself?

K: He meant to clearly state my opinion. I was prone to saying “maybe” so much that I acquired the nickname, the “maybe girl.” So I was told I had to commit myself, that is, once I had decided what my opinion was I had to clearly state it. In Japan I am often the only woman in a group of men, but whether male or female, in Japan there are not many people who clearly speak their mind in front of other people.

I: Yes, you’re right.

K: So, he started to get really anxious about this, about the number of times I was told to commit myself. So I was made fun of because I did not speak my opinion, they said that I was the “maybe girl.” But thanks to this experience I think today I am able to
clearly state my opinion if necessary, particularly when I am talking to foreigners.

I: I also spent a long time living overseas, but inevitably Japanese people are prone, like you mentioned, to saying “maybe,” or “perhaps.” There are many instances when it would have been better to have the confidence to speak my mind. Foreigners can say some very extreme things quite directly, and to some extent this way of speaking would still be considered unacceptable in Japanese society.

K: Yes, we can’t speak like that in Japan.

I: There are some things we cannot accept, but I think there are attitudes in Japan that need to change. This is an extremely important point.

K: For example, when I went to Sweden, it was through Bilateral Programs of the Japan Society for the Promotion of Science. After the end of stay by this scheme, I remained in Sweden, but I felt that the salary for my research position was quite low. So I discussed it with the director of the institute, I said that I felt my salary was rather low. As Japanese do not generally discuss salaries, if I had still been in Japan I would not have wanted to have this discussion. But I had heard that overseas, a person’s salary reflects how much he or she is appreciated by their employer. So I said I would like a raise because I wanted to stay there for a long time; as a result, it was doubled.

I: Without question, these kinds of discussions take place overseas.

K: Yes, I spoke after fully considering what I wanted to say. I often remember it as an extremely valuable experience. When overseas, you can get your point across by thinking things through and then stating the main thread of your argument.

I: Yes, I agree. Of course the reason your salary was doubled was because of your ability as a researcher, but you are right that when overseas you have to speak your mind. If you don’t say anything it is assumed you are happy with the current situation. If you have even a slightly different opinion, you will be respected if you clearly state that opinion; I think this a trait that Japan needs to learn.

K: So perhaps one of the most important lessons I learnt from living overseas was not to be a “maybe girl” but to commit myself. In addition, although this is also the case
in Japan, I have many friends in places throughout the world through conducting research. I have friends in many universities overseas, and when I am attending an academic conference and I meet an acquaintance whose appearance has changed and I am not instantly sure who they are. I am able to work out who they are by mentioning some peoples’ names and reminisce about them.

I: That is a priceless resource to draw on.

K: Yes it is.

I: So, after you returned from Sweden, you joined Professor Toyoki Kunitake at Kyushu University and became a group leader of ERATO.

K: To a certain extent, I continued with my research into molecular films that I began when I was a student. I researched molecular recognition, displaying at the molecular film on the water surface. Actually, molecular recognition is the specialty of Professor Tabushi I mentioned earlier. So it was natural for me to research molecular recognition of monomolecular film when I was in Professor Tabushi’s laboratory then continued it at ERATO. At ERATO, the group theme was pursued in a way that I created a form and it was then developed by other team members who synthesized new molecules.

I then began carrying out surface force measurements at ERATO. I had heard that Professor Kunitake was also interested in surface force measurements, so we decided to pursue this area when he found out that I was also interested in it. We knew there was a prototype device for sale in Australia, so we bought it and began our research. Even though I was a physical chemist, up until that point I had never assembled a large-scale complicated instrument. Although we had bought the device, we could purchase only the main chamber, the main components; we still had to source and assemble all the parts, which was before we even started carrying out the measurements that were themselves relatively difficult. Although I had had the opportunity to take a look at the instrument, I had never had one operational. In Japan, I had to assembly it myself while searching one by one for all the parts, constantly asking questions to lots of different people. But finally I was able to begin the sub-theme surface force measurements.
I: A pretty tough start.

K: We purchased and installed the first upgrade model that was built in the work shop of the Australian National University. I had seen the previous version of the model in Sweden, and I knew that it was difficult to use and needed upgrading, but you are in for a tough time if you buy equipment that is not up and running. I went to Australia to see the device in action, as two weeks training period included in the price of the machine. I saw that it worked, thought it would be a good machine, and bought it. As it was a brand new model, the purchase agreement entitled us to a replacement if it did not work. I am able to build this device today thanks in large part to how extensively I was able to study it at that time.

I: Did they show the device so openly there?

K: No, normally I would not have been able to see it. They wanted to show me some tests of the device to confirm that it was a good device in full working order. A person responsible for developing new instrumentation is a good person who is passionate about research, and I knew him from my time in Sweden. Perhaps in some part thanks to him, subsequently I was able to build the instrument myself.

I: So to recap what you have said, of course your research field is interfaces and it was while you were accumulating measurement data, as you previously said, you acquired just the main chamber of this instrumentation and you had to work out by yourself what you needed to add to it.

K: Yes. It was the first time that I had assembled such a large piece of equipment, and once it was assembled I then carried out the surface force measurements at ERATO as a sub-theme. After we had completed them, the quality of the data was such that it could be quoted in text books. As this was the first time this device had been operational in Japan, I think that we made some senior researchers very happy, and some of them traveled all the way to Kyushu to see it. When the ERATO project ended Nagoya University offered me a position with which I could carry out the same measurements in their Biophysics Laboratory, and so I became an associate professor there.

I: So you moved to Nagoya University?
K: Yes. I was moving to their Biophysics Laboratory, which had previously been called the Precision Measurement Laboratory, so I felt an affinity with it. Fortunately by coincidence, I was employed there because the professor of precision measurement was interested in my measurements.

Nagoya is a Mecca of physical measurements such as electron microscopes, x-rays, and moreover the Applied Physics Department had a lot of experiences in building instruments and had an excellent workshop. So I felt I would be able to build equipment there.

I: About 10 years ago you moved to Tohoku University. Are you still carrying out measurements here?

K: Yes, and thanks to doing these measurements my skill in developing new instruments and methods is also improving.

I: Could you explain about your development of the shear resonance measurement method?

K: Conventionally, surface force measurement involves measuring normal forces that act vertically. This method uses horizontal oscillation to measure shear response. By adopting the resonance measurement we could further heighten the level of sensitivity and improve detection levels. It is used to observe friction and liquid flow, by placing a liquid between two solids, in the scientific disciplines of rheology and tribology.

I started doing these measurements at Nagoya when the atomic force microscope (AFM) was developed. Although, I am a little unsure about the extent to which AFM can be used to measure surface force, it is quite easy as a measurement method. On the other hand, surfaces force measurements are extremely precise, but they are quite time consuming. I thought about what could not be done without using surface force measurement.

At that time, I thought it would be shear measurements for liquids confined between two large surfaces, which cannot be measured with an atomic force microscope. I
wanted to build a device to carry out the measurements, and at first I thought that we could measure shear force in a conventional way. However, something like a resonance vibration effect occurred and accurate measurements were impossible. So we then monitored the effect in order to check how this resonance vibration was occurring.

No one else was using the shear resonance measurement method, it was entirely original. But ultimately we decided to try to use it while monitoring how well it functioned and we found that it enabled us to achieve some excellent measurements. Despite the fact that no other researchers were advocating this method, it enabled us to observe a large signal extremely easily and moreover to measure a range of properties simultaneously. Therefore, I am actively using this method and I expect it to become the standard method of measurement in this field.

I: WPI-AIMR has a variety of approaches for materials science, but I think this measurement method will play an indispensable role in the creation of new materials.

K: Today, we are especially seeing in many different locations a number of materials that are more detailed and more precise and there is a demand for confining liquids in very tight spaces. These kinds of composite compounds are important in a wide variety of fields, particularly when creating materials. To put it very simply, even when discussing wettability, when combining the properties of surfaces with those of liquids it is extremely important to correctly observe how these properties change in narrow spaces. As a result we get a comparatively large number of applications for joint research from companies.

I: Changing the subject, congratulations on your selection in 2009 as the President Elect of the International Association of Colloid and Interface Scientists. Is this the international organization for researches in colloid and interface chemistry?

K: That’s correct, it is the only one.

I: Does it have a lot of members?

K: No, member numbers are not that high, but all the main researchers in the field are members. There are about 400 members and it is the umbrella organization for other
groups around the world. Most of the European colloid scientists belong to the European Colloid and Interface Society, which is a subsidiary organization. The Division of Colloid and Surface Chemistry, which is part of the Chemical Society of Japan, and the Division in the American Chemical Society of which the current President is a member of, are also both subsidiary groups. Also, the Australian group falls under its umbrella.

I: I see. And you are the chair person of the Division of Colloid and Surface Chemistry, the Chemical Society of Japan?

K: Yes. Actually, I have been also a council member of the International Association of Colloid Scientists for 6 years, and when my term ended I was recommended for presidency by the German. There was one other candidate, and I was selected by election. I have been active to find ways of moving the organization forward as an umbrella organization and also as a forum for international exchange, and I think these efforts have been appreciated. Also, my research field is surface force measurement, which is one of the most important concepts in colloid-related fields, so I think this was another reason I was elected.

I: I heard that in the near future this organization will be holding a major international conference in Sendai.

K: Yes, it is in 2012.

I: So the year after next; are you busy with preparations?

K: Extremely busy. We are jointly sponsoring the conference with the Science Council of Japan. We also want to ask to WPI-AIMR to jointly sponsor the event.

I: Yes, thank you for that.

K: As it is two years away, from this summer we must think really carefully about the structure of the event.

I: By the way, you are so busy at the moment; do you have time for hobbies?

K: No, at the moment I have no hobbies. I read a little. But I find doing different work very diverting, and what is most enjoyable for me is being in the laboratory doing research.

I: So, this is the happiest moment, I guess.

K: Throughout my career, I have moved from position to position so by the time I was 42 years old I had experienced just about everything I could. Also, when I was at Nagoya I started to build new equipments mostly by myself. So compared to the career of a typical university researcher I think I have a lot of diverse experiences on the research front line. So although these days I am very busy, I am happy because I think that the young researchers value the time we spend together researching and discussing.

I: That is very important. I know, Professor, that you were awarded a prize by The Society of Japanese Women Scientists. Do you have any advice for female or young researchers?

K: I have one piece of advice. I think that everyone can achieve a very original research. First, you should think about what are the most fundamental issues in your field. Then, you should what you can do at the leading edge of this field. If you combine your conclusions in these two areas then I think you should soon be able to conduct new, leading-edge research. You don’t have to be a research genius.

I: Very often, we are prone to feeling that we are not very original, that is not the case with you?

K: No, I don’t think that is the case with me. But of course observational skills are necessary. If I look back on myself when I was young, I think I had quite good powers of observation. However, the majority of what I do today is the result of an accumulation of leading-edge research and the research done daily over a long period. Also, there is probably a fixed perception of female researchers, and women tend to think there is a ceiling stopping them rising above a certain point. I think they should imagine this ceiling to be a little higher than they themselves think it is. Unexpected things can happen. When I was a student, it was unthinkable that a woman could preside over a standard laboratory in a large university. So I thought I would finish my
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life as a researcher having always helped someone else. But research is like a game of catch. I thought that I should enjoy receiving back from somewhere else the ball that we ourselves threw even if I would be an assistant. The world of research is changing and I hope that women can work on a level playing field with men, or even in some cases have the playing field balanced in their favor.

I: Yes, the research world is changing at a dizzying speed. It is not wise to set for yourself something as a fixed constant, you should try to change it.

K: But it is precisely for that reason that it is vital to strengthen our abilities.

I: Thank you very much for taking time out of your busy schedule to talk with me.
WPI-AIMR PI 栗原：和枝教授に聞く
(Interview with Professor Kurihara – Japanese version)

Maybeガールがコミットするとき

岩本：本年4月にWPI-AIMRの初の女性主任研究者に就任されたのでもっと早くインタビューに
来ると思っていたのですが、今日になり失礼しました。先生のお生まれは仙台ではないと伺い
ました。

栗原：ええ、東京です。

岩本：東京生まれの東京育ち。

栗原：はい、そうです。私は博士研究員でアメリカに行くまで実はずっと親元から離れたこと
がなかったのです。

岩本：では、江戸っ子ということで。

栗原：江戸っ子ではないですね。東京っ子ですが、品川なので東京の山の手ですね。

岩本：学部は、お茶の水女子大学の理学部卒業ですね。

栗原：理学部化学科ですね。

岩本：小さいころからやはり理科がずっとお好きだったのですか。

栗原：いいえ、私はどちらかというと本を読む事や、文章を書く事が好きでしたので自分は文
系だと思っていました。ただ、文系でも余り理屈でやるようなものよりは、歴史や考古学といっ
た、少し実証的なものをやりたいと思っていたました。どういうわけかそういう具体的なイメージ
がありました。けれども、両親はそういうものをやっても食べていけないと言い、理学部や理系
でも多分薬学部、医学部とか、あるいは文系だったら法学部などをイメージしていたようです。
私はもともとが考古学なので大分譲って理学部化学にしました。

岩本：大きな譲歩ということですね。

栗原：自分も少し理系も勉強したかったのです。当時は結構格好のいい職業として理系ライタ
ーというのが少しずつ出ていていた時代なんですよね。

岩本：ああ、そうでしたね。

栗原：ですから、本の出版とか、そういうものには関心があったので、理系のことをやって、
将来は、出版社のようところに勤めて本を作るのもいいかなとか、そういう気持ちがあって、
非常に教養的な意味で進学しました。周囲には無線をやっていたり理科クラブに入っていたりと、
やはりそういう同級生が多かったですね。

岩本：先生の場合にはむしろそういう科学の中身を一般の人を広く知らせたいというお気持ちはあったのですか。

栗原：いや、自分が今まで余り系の学問は学校の勉強以外はしたことがなかったので、少しそういうものをきちんと知りたいという気持ちがあったんですね。結構文系のことは図書館に行って調べたり勉強していましたので。

岩本：それから、理学部化学科から修士もお茶の水女子大学に進まれたわけですが、専門性というのはだんだん修士、それから東京大学の工学系研究科の博士課程に行くあたりで決まってきたわけですか。

栗原：私は大学の時、実は文系のサークルに入っていました。けれども、そこでは、美学や哲学とかをやっている人たちが随分いて、何か議論しているとやはり全然議論のレベルが違うので、あるときに、自分で勉強したものを大事にしてみたいなと思ったのと、3年のときに物理化学の学生実験をやったときに、ああおもしろいと思いました。計測から——これは今に通じているけれども——計測して、それから分子の性質がわかるというところです。今でも記憶に残っていますが、誘電率の測定をやって分子のダイポール、双極子モーメントがわかりますが、それでも溶液の性質を見てその中に溶けているもののさらに細かい性質がわかるということから、こういう計測はおもしろいと思ったのです。

岩本：計測との出会いですね。

栗原：それがきっかけで、物理化学の学生実験はとてもおもしろかったので、物理化学の研究室に行きました。そのときは将来研究者になるというつもりはまだなかったので、大学院はそのまま物理化学の研究室という、界面化学分野の研究室の修士に行きました。修士の時には、観察というものに基づく研究というもののがおもしろくて、一つ論文になる研究を観察をもとにしてできました。自分の研究が学生でも多少できたわけです。それで研究がおもしろくなったりということと、あとはもっとと考古学をやりたいというように、そもそも少し研究者というものになりたかったかもしれないですね。

岩本：もともと研究熱心でいらしたのですね。

栗原：だから、それがたまたま化学だったということと、あとは同級生が、クラスの中で1人くらいドクターにいかないとおもしろくないよねと言って、進学を勧めてくれたのです。そのときに、一緒に勉強会をやっていた助教授の先生に彼女は博士課程に進学したら良いと思っていますのだけれどと同級生が話して、勝手に彼らが相談して、私に進学したらと言って、そのときに非
栗原：そうですですね。だから、考古学をやりたいと思っていた気分と研究をやりたい気分というのは、高校生のときからあったので、そういうものと今やっている精密測定というのはやはりどこかものところではつながっているのかなと思いますね。

岩本：確かに考古学は人文系にしてはかなり数値も必要ですね。

栗原：サイエンスは実証なので労働としての大変さはあるけれども、きちんと測っていくというのはある意味ではスポーツみたいなところがありますよね。だから、それもよかったなと思いますね。

岩本：それで、博士課程に進まれて、博士論文は、どういう内容ですか。

栗原：バイオミメティックなんですけれども私の場合ずっと共通しているのは、分子の自己組織性というもののです。修士論文はミセルと液晶です。ミセルというのは界面活性剤が集合しているからで、それと液晶。それから、博士論文は脂質の二分子膜といって、生体膜のモデルです。それもこれにクロロフィルを入れて光を当てて葉の光合成のモデルのような研究をやりました。これも日本で初という要素がいろいろあって、脂質の二分子膜を米国で習ってきた先生が研究室にいて、日本の化学分野では一番最初に脂質二分子膜での反応の研究をやりました。

岩本：バイオミメティックということは、あたかも生物であるかのような、生物を真似するかのようなものですね。

栗原：そうですね。生物の中で起こっている機能を真似て人工的な系を作ること、だから、バイオミメティックの一番単純なのは、例えば飛行機だとも言えます。飛行機は鳥を真似ています。ただ、それは分子レベルのミメティックではないです、分子レベルで真似のなら、例えば葉の光合成の真似があります。植物の葉では葉緑体の膜の中にクロロフィルがあり光がたると、それで反応を起こして光合成が進む訳ですが、それと同じように光を当てて、光のエネルギーを化
学エネルギーとして、それを化合物に蓄えたいという、そういう研究がそのころからあって、その研究に携わりました。また最近いろいろ、低炭素化社会実現という文脈でそういう研究がまた出てきていますけれども、当時もうオイルショックの後ですから。

岩本：そうですね、第1次オイルショックが1973年でしたね。

栗原：オイルショックの後なので非常にそういう研究が注目されていました。エネルギー変換系の一番最初の研究ですね。それから、バイオミメティックもちょうどそのころに出てきた分野でして、生体膜のモデルや酵素モデルは、そのころに研究として始まった分野なので、界面化学の分野で扱う材料がちょうどバイオミメティックの生体膜モデルなどに共通するものなのです。いろいろ分子を集めた集合系みたいなものを界面化学で扱う訳で、最初からそういう分野で研究をやってきました。

岩本：界面というのは今WPI-AIMRでいろいろな研究者の方がやっていらっしゃいます。1970年代の終わりからやはり界面というのは世界的に重要な分野だったのですね。

栗原：そうですね。時代によりそれぞれ意味が違っていると思いますけれども、界面化学というのは結構古い学問ですが、1970年代には生体膜のモデルとして、脂質は界面活性剤と同じような構造をしたものなので、脂質や界面活性剤の集合系が使われていました。ちょうどその当時に分子を組織化していろいろな機能系を設計するという考え方、例えば反応する距離をコントロールするとか、そういうような考え方も出てきて、ちょうどそれらがいろいろな今のナノテクにつながる考え方のはしりになっています。

一つは分子の自己組織性ということだと思いますし、もう一つは、酵素は必ずしも自己組織性ではないのですが、酵素も含めいろいろな生体の機能を真似て良い材料を作ろうという研究のほうりがその頃から出てきました。背景には生体の構造や生体膜の構造、酵素の構造をX線で解明したとか、そうした研究がその当時出てきたことが大きいのです。材料も作れるようになってきました。

岩本：そういう材料を作る方法も出てきたし、観察する手段も出てきたということですか。

栗原：もちろん今の方がずっと進んでいますけれども、その当時がはしりです。今はナノレベルの材料が工業的にも使われるようになって、界面が大事だと言う風になってきています。それから、多分その当時は本当に一部の人だけがやっていたのが、今はみんながやっている。だから、先端はもっと深くなっていましょう。

岩本：今おっしゃったことは、先生の研究テーマの一つの表面力測定と関係するのですか。

栗原：私は1979年に博士号をとりましたが、そのときは光反応の実験を博士論文でやって
いったので、その後アメリカへ博士研究員として行った研究室の先生はやはりバイオミメティックをやる、メンブレミメティック・ケミストリーを提唱していたフェンドラー先生といって、生体膜の機能を真似た良い材料を作るという、そういう研究をやっていた方でした。その先生のところにはいろいろな光の高速反応を測る装置があって、中でもレーザーフラッシュフォトリシスという当時としては先端の研究手段でした。それをやりたくてそういう装置を持っている先生のところに行きました。当時は、特殊な場でナノ粒子を作るという研究もはじまってきて、今度はナノ粒子も分子組織体の中で作る研究をやって、日本へ戻ってきて、そのころそういうバイオミメティックでやはり当時のトップ研究者と言われた京都大学合成化学科の田伏岩夫先生の研究室に2年ほど行きました。

そのうち、自分らしい研究は何かを考えるようになりました。それまで私は、いろいろな機能材料を一応作っていました。作っていたというより、系の設計をしていた訳です。材料を合成するのではなくて、組み合わせて機能系を設計することをやっていましたが、自分は元々物理化学なので、何か計測をしてこの特性はこういう性質を持っている、こんなにいい機能があるということを測る研究だったのです。私は合成ではないので、おもしろい機能を見出しても、それをだんだん深めていくというアプローチがとれないので、そういう研究よりは10年やったら10年後はもうちょっと違うことができるような研究テーマをやりたいと思いました。それで、分子組織体とか自己組織性がわかる手段として何かいないかなと考えていたときに表面力測定に出会いました。自己組織性は分子が集まる性質ということで、相互作用により集まる訳ですから、そういう系を理解するために表面力測定というのは面白いのではないかということです。その後スウェーデンの研究所に行った時に、その研究所にはその測定装置を導入したところだったので、ちょっと共同研究をさせてもらって、どんな測定法かということがわかってきた。

岩本：スウェーデンの表面化学研究所ですか。
栗原：界面化学研究所と日本では呼ばれています。直訳すると確かに、surface chemistry、表面化学ですけどね。
岩本：先程のお話で、アメリカで研究された時のフェンドラー先生というのはTexas A&Mの恩師ですね。
栗原：そうです。在米中にはクラークソン大学に先生が移られ、研究室も同じ構成で移りました。
岩本：アメリカには都合3年ぐらい滞在し、その後スウェーデンに行かれたのですか。
栗原：はい。日本に2年戻ってきて、スウェーデンに2年です。
岩本：外国で研究しという経験は、研究の成果はもちろんのこと、ご自身の人間形成にとってどんな意味があったと思われますか。
栗原：私はフェンドラー先生には頻繁にコミットしろと言われました。
岩本：コミットしろというのは。
栗原：意見をはっきりと言え、ということなのです。私はすぐMaybeと言ったらしいんです。
おかげで、もしかすると私は結構はっきりとした意見を、特に外国人に対して言えるようになったような気がするんです。
岩本：そうですね。
栗原：だから、それがすごく気になったらしく、とても頻繁にコミットと言われましたね。ですかから、Maybeガールだ、おまえはMaybeガールだとかかわれていました。
岩本：私も外国に長い間暮らしましたけれども、どうしても日本人は先程おっしゃったPerhapsだとかPerhapsだということが多い。もっと自信を持って言え良いのにといhogとところがあります。
栗原：外国人は結構乱暴なことを断定的に言ったりして、その辺はまたある意味日本人としても、日本の中でもそれを全部通すということがまだなかなかできないですよね。
岩本：受け入れられないということがあるので、むしろ日本の風土を変えていかなくてはいけないと思います。でも、それは非常に重要な点ですね。
栗原：あとは例えばスウェーデンにいたときは、最初は日本学術振興会の二国間交流で行き、それが終了しても引き続き滞在しましたが、先方がお給料が安かったのです。それを所長に交渉して、どうもこれは安いのではないかと言いました。日本ではお給料については議論しないということが慣習であるので、私は日本でこういう議論をするつもりはなけれども、どうも外国では給料はその人にいてほしいというものを反映していると聞いている。それだから、このお給料だと私はここにずっといたいという気持ちにはならないので上げてほしいと言ったら上がったのです。倍ぐらいになりました。
岩本：大変なスタートだったのですね。
栗原：導入したのは、オーストラリアの国立大学の工作室で作った改良型1号機でした。私は先にWPI-01號を導入してきた Kazumasa Watanabeのところから聞いたのです。
岩本：そうですね。もちろん今のお話は先生の実力があったからですけれども、外国ではやはりはっきり言わないといけません。何も言わないとそれに満足していると思われるし、やはりちょっとでも違うのではないか、こういうことは違うというのはやはりはっきりしていこうのが導かれますし、日本も本来は見習わなくてはいけないと思います。
栗原：だから、Maybeでなくコミットしろと言われたのは、それは多分一つの外国にいたときの大きな教訓ですね。
あとは、私は国内もそうですか、随分色々なところで研究させていただいたので友達が大勢います。どこの大学にも友人がいて、外国でも、学会等に出席すると知人に会って風貌が変わって誰かわからないでしょうし、「誰々」と言わざるが偽善しか話ができるというときが結構ありますよね。
岩本：それは貴重な財産ですね。
栗原：そうですね。
岩本：ところで、先生は、スウェーデンから帰っていらっしゃって九州大学の国武豊喜先生のところでERATOのグループリーダーになられましたね。
栗原：ここでは単分子膜といって、ある意味で学生のときからの続きです。水の上にやはり界面活性剤に似たような合成の分子を並べて分子認識の研究をしていたました。実は分子認識というのは、前にお話した田伏先生のお家芸です。私は田伏先生のところにいたときにやはり単分子膜の分子認識の研究を、ちょっと違う形ですけれどもしていました。ERATOではグループテーマで、私が最初に形をつくると、ほかの人たちは新しい分子を合成して展開しました。
表面力測定はERATOで始めました。表面力測定は国武先生も関心を持っていましたのです。それで、私が関心があるということを知ってやってみようかと、オーストラリアでその装置の原型を売り出してしまったので、それを買って始めました。私はそれまで物理化学といっても計測器を大きく組む事はやったことはありませんでした。ところが、装置は売っていますが、メインチャンバー（主容器）というものだけ、メインコンポーネントだけ売っていて、あとは全部自分でそろえて組まなければいけないし、測定も割合難しいものでした。私はたまたま少し見せてもらったけれども、実際は動かしたことはなかったので、国内で組むのに全部部品を一つずつ捜したりいろいろな人に聞いたりしながら自分で組んで、サブテーマで表面力測定を始めました。
岩本：大変なスタートだったのですね。
栗原：導入したのは、オーストラリアの国立大学の工作室で作った改良型1号機でした。私は先
行モデルをスウェーデンで見ていたんですが、使いにくいということを知っていたので、よくすべきだと思っていたのですが、動かない装置を買ってしまったら後で大変ですよ。どう動くのか見せてほしいと言って装置の価格に含まれていたトレーニング期間2週間を使ってオーストリアに行き、見てもらい、「ああ、動きそうだだから、ではこれ買って」ということで、それで買ってました。新しいモデル1号機ですので、もし動かなかったら替えるという契約もしました。

栗原：それだけオープンに見せてくれるものなのですか。

岩本：いや、普通は見せないでしょう。だから、それは絶対動くのだということをテストで見せようとしたので。どういうふうにテストするのかを、たまたま新しい装置を開発した人を私はスウェーデンにいるときから知っていたということもあって、それもあったのでしょうが、とても丁寧に見せてくれた。また、その人が人柄のいい研究好きの研究者だったので。だから、以後、装置を作れるようになったのは多分それがすごく大きいです。

栗原：はい。初めて大きな装置を自分である程度組み立てたことになるのですが、ERATOで表面力測定をサブテーマでやっていて、終わりのところには教科書に引用されるような新しいデータもとれるようになったのです。日本では初めて動いた装置というので随分シニアの先生方が喜んでくださって、中には九州まで見に来てくれた先生もいました。その後、名古屋大学で、そうした測定をやっているなら、生物物理工学研究室で雇ってくださるということで助教授になりました。

岩本：それで、名古屋大学に移られたのですね。

栗原：そうですね。ですから、名古屋では生物物理の研究室に行ったのですが、その研究室の前の名称は精密計測だったときいて縁を感じました。話ができ過ぎていますが、だから精密測定の先生が興味を持っていて雇われたことになります。名古屋は電顕、X線と非常に物理計測のメカみたいなところで、応用物理は装置作りがすごく盛んな学科だったので、学科で立派な工作室を持っていて、だからそこで装置を作るようになったのが装置作りの初めです。

岩本：それで10年ほど前に東北大学にいらして、今もそういった測定ですか。

栗原：そうです。やっていて、だんだんおかげさまで装置作りも腕が上がってきました。
岩本：共振ずり測定法も開発されたと伺っていますが、これはどういうことですか。
栗原：普通の表面力測定は垂直に働く力、ノーマルフォースを測るのに対し、それを面を平行にずって、ずることでのレスポンスを測るというので、場合によっては垂直力がかかっているところをこういうふうにすることでさらに感度よく検出できるという場合もあるのです。もともとは固体の間に液体を挟んで、摩擦とか液体の流れを見るとか、学間分野でいうとレオロジーとかトライポロジーとかいう、分野に近いような測定です。
名古屋で測定を始めた時、原子間力顕微鏡が出てきました。これがどの程度の表面力測定に使えるか少しわからない部分があったのですが、測定法としてはかなり楽でした。一方、表面力測定の精度はすごくいいのですが、やはり手間のかかる測定なので、その中で表面力測定でないとできない残る部分は何だろうと考えました。
その時、大きな表面に液体をはさみずる測定は残るのだろうと思ったわけです。これは原子間力顕微鏡ではないの。それをやりたいと思って装置を作って、装置ができて、最初は普通に応力測ろうと思っていたのですが、何か共振のすそみたいなものがかかってきたので、まずは共振がどういうふうに出てくるのかチェックしようということでモニターし出したのがきっかけです。共振法というのはだれもやっていてなくて、オリジナルメソッドなのですが、実は結果的に言うとそれをモニターしようと思ってやりたら、とてもきれいに測れそうだということになり、できた方法です。ほかの人たちはそういう方法を提案していなかったのですが、大きいシグナルを見るので非常に簡単で、しかもいろいろな性質が同時に測れるというので、私はこれが標準的な方法になるのではないかと期待して一生懸命やっています。
岩本：WPI-AIMRでは、材料科学についていろいろなアプローチでやっていくところですから、先生のこういった測定方法は新しい材料を考えていく上で本当に不可欠なものでしょう。
栗原：今は特にどこでもいろいろな材料が微細・精密になっていて、狭い空間に液体を入れなければいけないというような要求もあるようです。そういう複合化というのも色々な分野で大事で、特に材料を作るときにはそうです。非常に単純には濡れ性ということについても、表面の性質と液体の性質との組み合わせとか、狭い空間になったときの性質の変化をきちんと見ていくということがとても大事だということで、割合、企業からの共同研究の申し込みも多いです。
岩本：話は変わりますが、2009年からは国際コロイド界面化学者連盟（International Association of Colloid and Interface Scientists）のプレジデント・エレクトだそうで、おめでとうございます。これはそういうコロイドや界面化学の研究者の国際的な団体ですか。
栗原：そうですね。これが唯一のものです。
岩本：かなり会員数も多いのですか。
栗原：いや、会員数は多くないのですが、主な研究者はほとんど入っていて、会員数は400名くらいです。ヨーロッパのコロイド科学者の大体は、ヨーロピアンコロイドというものに所属していますが、その上の組織ですし、日本化学会の中にコロイド界面化学部会がありまが、それも傘下に入っていますし、アメリカの化学会の部会もありますので、現会長はアメリカ化学部会の会長ですが、その他も含め、アンブレラオーガナイゼーションです。
岩本：そうですか。先生は日本化学会のコロイド及び界面化学部会の部会長ですよね。
栗原：はい。実は、国際コロイド化学者連盟では理事を6年間させていただいて、任期終了時になお彼氏が推薦いただいたのが、選挙で当選させていただきました。私はアンブレラオーガナイゼーションとか国際交流とか、そういう面でうまく進める方法はないかということを一生懸命やってきましたので、そういうところを評価されたのだと思います。また、研究も表面力の測定というのはコロイド関連の分野では最も要になる概念の一つなので、そういう分野の研究をやっているからだと思います。
岩本：この組織は近々仙台で何か大きな国際会議を開催されると聞きましたが、
栗原：2012年です。
岩本：再来年ですか。その準備とかお忙しいですね。
栗原：もう大変なんです。日本学術会議と共同主催に採択いただきまして、WPI-AIMRにもそのうちに共同主催のお願いに行くつもりでいます。WPI-AIMRにもその
岩本：はい、それはありがとうございます。
栗原：ちょっとまだ2年後くらいなので、今年の夏あたりから1年ぐらい本当にちょっと組織を考えないといけないですね。
岩本：話はまだ変わりますが、先生はこれだけお忙しい中で趣味は何ですか。
栗原：現在は趣味といえるものは、ありません。本は少し読んでいますけれど。ただ、違う仕事をしていただいているのが気分転換ですし、研究室で研究をしているのが一番楽しい。
岩本：それはある意味で一番幸せかもしれないですね。
栗原：私は長く流動ポストという経歴なので42歳くらいまで自分で全部実験していました。その後も名古屋のときは装置を全部自分で組んでいましたから、そういう意味で普通の大学の一般の研究者に比べると現場が長いので、今は忙しいですけれど、一緒に議論しながら研究をやるということに関して若い人が大事にしてくれるので幸せと思っています。
岩本：それは重要なことです。先生は、日本女性科学者の会の賞も受賞されていますけれども、女性研究者あるいは若手研究者に対するアドバイスとしたら、どういうことになりますか。
栗原：一つは、私は、だれでも非常にオリジナルな研究はできると思っています。それは自分の分野の非常に基本的な課題で何がわかっていないのかを考えて、それから最先端で何ができるかを考えて、それを組み合わせてちょっと考えてればだれでもすぐ先端の新しい研究ができるのではないかと。天才性という話ではない気がします。
岩本：とくに自分のオリジナルティーがないのだと思うのがちですが、そんなところはないと。
栗原：そんなことはないと思います。ただ、もちろん観察力は必要です。私は若い時の自分を思うとかなり観察力があったかもしれないですが、例えば今やっているのはやはり先端と毎日の積み重ねでやっているところが多いですね。
あと、女性研究者ですが、従来からの形というのはあるかもしれないですけど、女性はこのくらいといった限界などは、世の中は動いているのだから、限界は自分の思うより少し先にあると思ってやったらいいのではないかと思います。思いもかけないようなことってありますね、私が学生の頃でしたら大きな大学の研究室を女性が主宰するというのは考えられないことでしたから、私は一生だれかの手伝いをして研究者としては終わるだろうと思っていた。それで研究というのはキャッチボールするもので、自分の投げた球はどこから返ってくるのかは楽しくてそれでいいと思っていました。でも、今はこんなに世の中が変わってきて、女性でも普通にやれるし、あるいは場合によっては普通以上にやることが期待される場合もありますし。
岩本：そうですよね。世の中自体がもうどんどんすごい速さで動いていますからね。何がある一一定的分みたいものも自分で設定してしまうと、かえっていけませんね。
栗原：でも、だからこそ、実力を蓄えていうところが大事ではないでしょうか。
岩本：長時間にわたりまして貴重なお話をいただきどうも有り難うございました。

２０１０年７月５日
東北大学多元物質科学研究所材料物性総合研究棟 PI栗原研究室にて
岩本 渉

２０１０年４月１日からインタビューは、小松教授から岩本事務部門長へ交代しました。
News Update
In April 2010, WPI-AIMR decided to further promote outreach activities. Dr. S. Ikeda was assigned as Outreach Manager, a position responsible for planning and promoting WPI-AIMR’s outreach activities. The aim of the outreach is to facilitate communication and mutual understanding between WPI-AIMR and the public. On June 19, 2010, the press release regarding “promotion of science and technology communication with the public” was issued by Tatsuo Kawabata, Minister of State for Science and Technology Policy (concurrently holds the position of Minister of Education, Culture, Sports, Science and Technology) and the Council for Science and Technology Policy. The press release specifies that the research projects based on public funds of over 30 million yen will be evaluated not only in their research results but also in their outreach activities. Now WPI-AIMR has to meet accountability standards for the public, and show how our research activities are important in society and what excellent results will be obtained.

The outreach team of WPI-AIMR issued the first volume of our outreach magazine in June. The magazine was named Tohoku WPI Tsu-Shin. All articles are written in Japanese for the public distribution. Volume 1 contains greetings from the director, an explanation of “WPI”, and a brief introduction of ongoing research projects at WPI-AIMR. The memorial cover of the first issue was illustrated with a painting drawn by an elementary school pupil who lives in Miyagi Prefecture. We hope that this painting acts as a mediator between citizens and WPI-AIMR. The star seems to represent an atom and the stork seems to represent a molecule, and they just seem to be flying out of the universe (the material world). The column “Communication Square” at the last page of the magazine invites the questions and comments from the public. Tohoku WPI Tsu-Shin volume 1 has been mailed to all elementary schools and junior high schools in Sendai City and all high schools in Miyagi Prefecture. The magazine has been placed at Sendai City Information Centers, Sendai Science Museum, and Sendai Astronomical Observatory. It has also been distributed to participants in the scientific events shown on the next page. The magazine will be issued three times a year, in June, October and February. So far, WPI-AIMR opened a booth at the following three events.
WPI-AIMR Outreach Activities

In April 2010, WPI-AIMR decided to further promote outreach activities. Dr. S. Ikeda was assigned as Outreach Manager, a position responsible for planning and promoting WPI-AIMR’s outreach activities. The aim of the outreach is to facilitate communication and mutual understanding between WPI-AIMR and the public. On June 19, 2010, the press release regarding “promotion of science and technology communication with the public” was issued by Tatsuo Kawabata, Minister of State for Science and Technology Policy (concurrently holds the position of Minister of Education, Culture, Sports, Science and Technology) and the Council for Science and Technology Policy. The press release specifies that the research projects based on public funds of over 30 million yen will be evaluated not only in their research results but also in their outreach activities. Now WPI-AIMR has to meet accountability standards for the public, and show how our research activities are important in society and what excellent results will be obtained.

The outreach team of WPI-AIMR issued the first volume of our outreach magazine in June. The magazine was named Tohoku WPI Tsu-Shin. All articles are written in Japanese for the public distribution. Volume 1 contains greetings from the director, an explanation of “WPI”, and a brief introduction of ongoing research projects at WPI-AIMR. The memorial cover of the first issue was illustrated with a painting drawn by an elementary school pupil who lives in Miyagi Prefecture. We hope that this painting acts as a mediator between citizens and WPI-AIMR. The star seems to represent an atom and the stork seems to represent a molecule, and they just seem to be flying out of the universe (the material world). The column “Communication Square” at the last page of the magazine invites the questions and comments from the public. Tohoku WPI Tsu-Shin volume 1 has been mailed to all elementary schools and junior high schools in Sendai City and all high schools in Miyagi Prefecture. The magazine has been placed at Sendai City Information Centers, Sendai Science Museum, and Sendai Astronomical Observatory. It has also been distributed to participants in the scientific events shown on the next page. The magazine will be issued three times a year, in June, October and February.

So far, WPI-AIMR opened a booth at the following three events.
“Science and Technology Festa in Kyoto” (June 5)

WPI-AIMR made a presentation at the “Science and Technology Festa in Kyoto” held at Kyoto International Conference Center on June 5, 2010. The five WPI centers gathered to present the WPI program and their research activities. At the same venue, Prof. Adschiri, PI of WPI-AIMR, was awarded “The Prize of the Minister of Education, Culture, Sports, Science and Technology in the field of Cooperation among Government, Industry and Academia” and also opened his booth at the Festa.

“The City of Academia ‘Sendai-Miyagi’ Science Day 2010” (July 11)

WPI-AIMR opened a booth at the “City of Academia ‘Sendai-Miyagi’ Science Day 2010” held at Kawauchi North Campus in Tohoku University on July 11, 2010. About twenty elementary and junior high school students tried to make transistors using organic semiconductors and studied a cutting-edge research topic.

“Tohoku University Open Campus 2010” (July 28 - 29)

Tohoku University Open Campus was held on July 28 and 29, and WPI-AIMR opened a booth in the Physics A Building, Aobayama Campus to show Tohoku University’s activities to high school students. Chemical experiments (reactions and synthesis) and physical experiments (transistor characteristics) were demonstrated, and an introduction of WPI-AIMR was offered using posters and magazines.

For details of these events, please visit the “News and Information” page of our website.

Profile of Outreach Manager

Susumu Ikeda graduated from the Faculty of Science, Tohoku University in 1990. After working at cement companies, he moved to the University of Tokyo in 1995. After obtaining a Ph.D. in 2000, he worked at the University of Tokyo as a postdoc, research associate and assistant professor. In 2008, he moved to WPI-AIMR as an assistant professor. His wide research experiences in mineralogy, petrology, cement chemistry, surface science (metals, semiconductors and insulators), organic semiconductors, and electronic devices are helpful in informing the public about variety of research topics currently in progress at WPI-AIMR.
The 1st WCU/AMS (POSTECH)-WPI/AIMR (Tohoku University) Joint Workshop at POSTECH, June 18, 2010

M. Yamaguchi

The 1st WCU/AMS (POSTECH)-WPI/AIMR (Tohoku University) Joint Workshop was held at POSCO International Center in Pohang University of Science and Technology (POSTECH) in the Republic of Korea on June 18, 2010. POSTECH, founded by world-class steel company POSCO at Pohang, is a leading university of the Republic of Korea in the field of science and technology. The purpose of the workshop was to promote collaboration and exchange of researchers between WPI-AIMR and WCU-AMS based on the agreement signed this March at Sendai during the 2010 WPI-AIMR Annual Workshop. Both programs possess a common purpose in the development of new frontiers in the materials sciences. Seven PIs visited from WPI-AIMR with a student who obtained a PhD from POSTECH and is now working with Professor Adschiri, and six PIs from POSTECH gave talks.

The workshop opened with welcome remarks from Professor Kimoon Kim (POSTECH), WCU-AMS director, explaining the WCU-AMS program in Korea, and announcing the agreement and future plans. The introduction of WPI-AIMR program was then provided by WPI-AIMR director Professor Yoshinori Yamamoto.

The first scientific talk in the morning session was given by Professor Kimoon Kim. He showed a number of cucurbituril derivatives including polymer shells, which can form inclusion complexes with various organic host molecules. Such novel compounds can be applied to ion channels embedded in a lipid bilayer, targeted drug delivery, and immobilizing a protein on a solid surface.

The second talk was given by Professor Kyo Han Ahn at the department of chemistry, POSTECH. He discussed molecular recognition and sensing of anionic species such as carboxylates or amines for biochemical study and disease diagnosis. The molecular probe method was integrated to a gold nanoparticle sensing system and polymerized liposome sensing systems.

Professor Kingo Itaya of WPI-AIMR presented a new method to study surface structures. A laser scanning confocal microscope combined with a differential
interference contrast microscope (LCM-DIM) method allows the observation of a single atomic step on Si(111) and Si(001) in an NH₄F solution. The atomic process of metal electroplating was revealed by this method, which can be applied to the production of electronic devices based on organic semiconductors.

Professor Moon Jeong Park from WCU-AMS was the only female speaker in this workshop. She talked about poly (styrenesulfonate-b-methylbutylene) membranes comprising hydrophilic phases with widths ranging from 2 to 5 nm to control water retention. Such ion-containing polymer electrolyte membranes (PEMs) can be used in fuel cells.

Professor Tadafumi Adschiri of WPI-AIMR described organic-inorganic hybrid nanoparticles. Organic modified nanoparticles were synthesized in supercritical water taking advantage of organic molecules and metal salts aqueous solutions being miscible. The hybrid nanoparticles show high affinity with the organic solvents or polymer matrix, which facilitates their applications.

After lunch, the afternoon session was started with a lecture by Professor Masatsugu Shimomura from WPI-AIMR. He discussed novel biomimetic surface materials, honeycomb-patterned polymer films, which have two-dimensional regular arrays of micro/nano pores. Such surfaces show very small light reflectance and a super-hydrophobic nature.

Professor Danil N. Dybtsev (POSTECH) joined WCU-AMS program from Russia. He presented enantiopure porous coordination polymers containing zinc metal, which can be used for chiral separation or fine purification of optically active molecules.

Professor Naoki Asao of WPI-AIMR discussed the use of nanoporous gold in catalysis particularly in the oxidation reaction of alcohols to aldehydes and ketones in the presence of molecular oxygen. The monolithic materials consisting of a three-dimensional network of ligaments with diameters on the order of 10 to 50 nm were shown to be effective for this transformation.

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Professor Hee Cheul Choi of WCU-AMS showed methods for constructing nano- to micro-structures using vapor phase reaction (vaporization-condensation-recrystallization) and the solution phase (drop-drying). Using these techniques, hexagonal and helix structures were constructed.

Professor Toshio Nishi from WPI-AIMR discussed new and practical soft materials useful for the society in view of the environment, energy, and safety. As examples, development of ultra-low rolling resistance tire compounds and seismic-elastomeric isolators was described.

The last talk in this workshop was given by Professor Masaki Kawano, who recently joined POSTECH as a WCU professor from the University of Tokyo. He developed the structure determination method of crystalline compounds by powder X-ray analysis, not by single crystal X-ray diffraction. A porous coordination network was formed by kinetic effect, and the method was applied to analyze the substances.

On the day preceding the workshop, there was a Soccer World Cup Game between the Republic of Korea and Argentina in South Africa. The game was shown on a large display in a student hall, and many students enjoyed the game until late at night. Unfortunately, the Korean team lost the game, although the game itself was very nice. On the next day, professors and students were discussing the game. Such close interactions of professors and students are a merit of POSTECH. I had an opportunity to work with POSTECH graduate students, and found them to be enjoyable, polite and enthusiastic.

POSTECH is located in the suburbs of Pohang, where most of the students and professors live. We stayed at the guest rooms in the POSCO International Center, which were surprisingly well equipped. We hoped to have such facilities in our university. It is also notable that the historic city of Gyeongju, the capital of the Silla Kingdom for almost a thousand years, is located a 30 minute car ride from Pohang.

Professor Kim visited WPI-AIMR this July after the workshop in order to discuss further interactions and to give talks in Aobayama and Katahira. Several collaboration programs appear to be starting between Tohoku University and POSTECH. It is highly likely that WPI-AIMR and WCU-AMS will be excellent partners in the East
Asian region for the promotion of research in the field of materials sciences.

(Workshop’s Photos)
WCU Project and POSTECH-AMS

W. Iwamoto

The WCU Project

The Government of the Republic of Korea launched in 2008 the national project called “World Class Universities” in order to meet a critical need to foster research in key growth-generating fields, and to ensure quality in the development of next-generation human resources. The project invites global top-level scholars and researchers to develop world-class academic programs and departments in Korean universities, which will ultimately lead to the creation of world-leading institutions of higher education in. This project was initiated with the purpose of promotion of quality and excellence, and to increase the competitiveness of higher education institutions. It also represents the idea of “from brain drain to brain gain”.

The Project focuses on supporting new growth-generating fields that have the potential to the spearhead national development. Examples include NBIC (NanoBioInfoCogno) fusion technology, disruptive technology, breakthrough technology, energy science, bio-pharmaceutics, etc. Priority is also given to inter-disciplinary fields that cut across the fields of basic sciences, humanities and social sciences, which will contribute to national, social and academic development.

The WCU project invites the participation of international scholars and researchers according to three project types: 1) recruiting scholars to establish new academic departments or specialized majors in Korean universities, 2) employing foreign scholars at existing academic programs, and 3) inviting distinguished world-class scholars to teach and research in Korea. The proposals are submitted to panel reviews and international peer reviews. In the case of type 1), 32 proposals were selected so far through the 1st and 2nd round selection processes.

Different from WPI program in Japan, the WCU Project focuses not only on research, but also education. Moreover, the Project also covers the linkage with humanities and social sciences.

POSTECH and AMS

Pohang University of Science and Technology (POSTECH) was established in 1986 as the first research-oriented university in Korea with the strong support of POSCO, a top global steel production company. With 10 departments and 57 research centers, the University now has 244 faculty members and 780 researchers together with 3,057
students (figures are as of 2009). The global partners of POSTECH consist of over 70 universities in 19 countries. It is notable that Tohoku University signed an Academic Exchange Agreement with POSTECH in September 2000.

POSTECH was selected in all three project types of the WCU Project, the number of selected proposals totaling 6. The three proposals selected in the first type, i.e. the new academic department establishment, have progressed to the opening of the Division of Advanced Material Sciences, the Division of Integrative Biosciences and Biotechnology, and the Division of IT Convergence Engineering.

The Division of Advanced Material Sciences (AMS) headed by Professor Kimoon Kim focuses on fundamental research for the development of new functional materials and device platforms (Figure 1), based upon four research areas, namely emergent materials, self-assembled nano hybrid materials, self-assembled soft materials, and emerging device systems. (Figure 2)

![Figure 1](image1)

AMS now has 14 faculty members (PIs) in the areas of physics, chemistry, and materials science. As the WCU Project requires 30-40 % of them to come from abroad, there are 5 foreign researchers as faculty members (1 German, 1 French, 1 Russian, 1 Japanese from The University of Tokyo and 1 Korean member who just finished 3 years of research activities at UC Berkeley). From last year AMS receives 15 post-graduate students. The Korean Ministry of Education, Science and Technology provides about 3 million US dollars to AMS through the WCU Project annually. This subsidy is used for an annual salary and lab establishment expenses for each foreign scholar, and research grants for all the faculty members.

While AMS has a great advantage in collaborating with POSTECH Biotech Center, the National Center for Nanomaterials Technology, and the Pohang Accelerator Laboratory in POSTECH, it also constitutes global network with the Max Planck Institutes, the Asia Pacific Center for Theoretical Physics, RIKEN SPring-8 Center and WPI-AIMR, with which AMS signed an Agreement on Academic Exchange last March. (Figure 3)

AMS makes great efforts to integrate various disciplines not only in research, but also in education which is most challenging. On the other hand, AMS involves as much as possible foreign faculty members who cannot necessarily stay a whole year in Korea.

This article is written based upon the brochures of the Korean Ministry of Education, Science and Technology, POSTECH and AMS, together with precious information provided through interviews with Professor Kimoon Kim, Director, WCU-AMS to whom I express my sincere gratitude for spending his time in spite of the tight schedule of his short visit to WPI-AIMR from 19 to 22 July.
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Prof. Kimoon Kim
Dr. Akihisa Inoue’s lecture
at reception of Acta Materialia Gold Medal

Dr. Akihisa Inoue who is a member of International Advisory Board of WPI-AIMR and President of Tohoku University was awarded “Acta Materialia Gold Medal”. The presentation ceremony was held at the WPI-AIMR Annual Workshop on March 26, 2010 in Sendai. Dr. Thaddeus B. Massalski, Professor of Carnegie Mellon University and Executive Secretary of Acta Materialia, Inc, handed over the Gold Medal to Dr. Inoue.

Following the ceremony, Dr. Inoue gave a special lecture titled “Development and Applications of Bulk Glassy and Nonequilibrium Crystalline Alloys”. The summary of the lecture is as follows:

Unlike crystalline solids amorphous alloys do not have a periodic structure which results in unique properties. Bulk glassy alloys are produced at cooling rates of 1-100 K/s. The lowest critical cooling rate for glass formation is 0.067 K/s which is about 15 million times smaller than that required for ordinary amorphous alloys.

For example, Pd-Cu-Ni-P glassy alloy ingots produced in our laboratory are the largest monolithic metallic glassy objects obtained up to date owing to exceptionally high glass-forming ability of the alloy and application of a special processing technique.
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For example, Pd-Cu-Ni-P glassy alloy ingots produced in our laboratory are the largest monolithic metallic glassy objects obtained up to date owing to exceptionally high glass-forming ability of the alloy and application of a special processing technique.

Glassy phase transforms to a super-cooled liquid on heating before crystallization. In this region the strain rate sensitivity exponent m~1.0 which indicates the achievement of the ideal super plasticity. Therefore we can obtain a very large elongation of 22,000% in the super-cooled liquid region (ΔT_x).

For the past two decades we accumulated a large amount of data on bulk glassy alloys and we produced large scale bulk glassy alloys with critical diameters of over 2 cm in a variety of alloy systems as shown in the Table.

These alloys consist of three or more elements, have a significant atomic size mismatch above 12% and negative heat on mixing. Based on these guiding rules, we obtained a variety of metallic bulk-glassy alloys mentioned above.

Initially scientists believed that metallic glasses form dense random packed structures. Later it become clear that they consist of efficiently packed atomic clusters. Molecular dynamics simulation based on plastic crystal model representing the clusters can explain the structure of metallic glasses.
By using the ordinary photo-register coating process we can produce a nickel die. By pressing the glassy alloys to the die in the super-cooled liquid region we can obtain the diffraction grating of glassy alloys which has the same shapes as resistant structures of the grating.

By precise control the incident and the refractive diffraction angles and the intervals, one can obtain red, green and blue colors. By using these spectroscopy systems we open up the new hologram technologies based on bulk glassy alloys. Now we are challenging these hologram technologies.

Some of bulk metallic glasses deform plastically under compression at room temperature (RT). $Zr_{70}Ni_{16}Cu_{6}Al_{8}$ glassy alloy exhibits RT plastic elongation of about 3 % upon tensile test. This is the first example of such a behavior for a monolithic bulk (massive) metallic glass.

This alloy also shows a very high fracture toughness of 110 MPa-m$^{1/2}$ which is also maintained after heat treatment. This alloy shows a very long shear-band length in front regions of the pre-fatigue cracks as well as very large crack chip opening displacement.

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We also worked on the recording media and achieved 25 nanometers for pitch size and 12 nanometers for bit size corresponding to recording density of one terabit per square inch. We are now approaching almost 2 terabit per square inch and the pitch size is 18 nanometers.

Fe-Cr-based metalloid type soft magnetic glassy alloys have been commercialized with the commercial name of Liqualloy by Alps Electrical Corporation. The picture shows Liqualloy consolidated magnetic cores for choke coils of the power supplies and their performance.

Glassy alloy diaphragms for pressure sensors are produced by net-shaped casting process. These glassy alloy diaphragms have smaller size and demonstrate higher sensitivities as well as much higher pressure endurance zones in comparison with standard steel pressure sensors.

We also obtained Ti-based glassy alloy pipes with a diameter of 2 mm and wall thickness of 0.2 mm. These pipes are suitable for applications for sensing element to detect the Coriolis force of liquid or gas flowing inside the Ti-based glassy alloy pipes subjected to reinforced oscillations.
Kawasaki Group’s paper was selected for the June issue of the Virtual Journals of Ultrafast Science

The paper by Prof. Kawasaki’s group published in the Applied physics Letters 96, 211904(2010), has been selected for the June issue of the Virtual Journals of Ultrafast Science.

The summary of the paper is as follows:

“Exciton properties and ultrafast dynamics in CuScO\textsubscript{2} thin films”

F. Liu\textsuperscript{1,2}, T. Makino\textsuperscript{3}, H. Hiraga\textsuperscript{3}, T. Fukumura\textsuperscript{1}, Y. Kong\textsuperscript{2}, M. Kawasaki\textsuperscript{1,3,4}

\textsuperscript{1}Institute for Materials Research, Tohoku University, Sendai, 980-8577, Japan
\textsuperscript{2}School of Physics, Nankai University, Tianjin 300071, China
\textsuperscript{3}WPI Advanced Institute for Materials Research, Tohoku University, Sendai, 980-8577, Japan
\textsuperscript{4}Japan Science and Technology Agency, CREST, Tokyo 102-0075, Japan

Cuprous delafossites Cu\textsubscript{4}O\textsubscript{2} (e.g., A = Al) are peculiar and promising materials because the holes (having positive charge) play a role in realizing a high conductivity while maintaining a good optical transparency [1]. This is significantly different point from other wide-gap materials because many wide-gap semiconductors, such as ZnO, have natural tendency to be grown under fairly high residual n type. The crystal structure of delafossite Cu\textsubscript{4}O\textsubscript{2} consists of an alternating stack of Cu ion layers and O\textsubscript{2} octahedron layers as shown in the inset of Fig. 1. Replacement of the A site element with Sc has successfully led to two more intriguing properties; a direct-transition nature (4.36 eV) and strong excitonic effect (380 meV). These properties are advantageous from the viewpoint of optoelectronic applications (such as a light-emitting diode). The main panel of Fig. 1 shows an 80-K optical absorption spectrum. It also revealed the optical characteristics such as wider direct-band-gap, much larger exciton binding energy, and much weaker phonon-exciton coupling than those of ZnO. This can avoid the instabilization of exciton even at room temperature. Table 1 compiles the binding energies of exciton for representative oxide materials.

We have previously that the binding energies of exciton are enhanced from the bulk values with a decreasing in the quantum well layer thickness in ZnO/MgZnO quantum well heterostructures, as a result of quantum-confinement effects [4]. The low-dimensionality inherent to CuScO\textsubscript{2} is considered to be a reason of such a large binding energy of excitons.

![Fig. 1. Absorption spectrum for CuScO\textsubscript{2} thin films. The red [Disc.], blue [Cont.] lines represent the results of the ground and continuum states of excitons. Inset shows its crystal structure.](image)

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<th>Table 1: Bandgap energies ($E_{g}$) and binding energies of exciton ($E_{\text{ex}}^b$) for representative oxide materials</th>
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<td>$E_{g}$[eV]</td>
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70
Femtosecond pump-probe experiments were performed for this exciton system [5] because none of its dynamical properties is known. The main dynamical features were shown in Figs. 2(b) and (c) and summarized in the following two aspects: exceptionally slow thermalization time in a scale of 10 ps and relatively small nonlinearity accompanied with the dispersive lineshape around the excitonic resonance. These findings can be consistently interpreted in terms of remarkably large binding energy of exciton and of weak phonon interaction. It should be emphasized that the observed time-resolved nonlinearity can be simply explained only by the screening effect of Coulomb potential for the excitons. In other words, these dynamical studies on its optical nonlinearity revealed that CuScO$_2$ can manifest itself as a model substance for excitonic nonlinearity in a naturally low-dimensional structure because the data could be simply explained in terms of excitonic screening effects.

References:

Dr. Konstantinos Georgarakis’s paper was published in “ESRF Highlights 2009”

The paper “Detection of deviation from ideal solution behavior in Zr-Cu metallic glasses with addition of Al” by Assistant Professor Georgarakis was selected for “ESRF Highlights 2009” issued in February 2010. The Highlights selects to feature only 76 articles out of about 1500 papers published annually on the works carried out at the European Synchrotron Radiation Facility (ESRF). The Summary of the paper is as follows:

Metallic glasses, made by carefully cooling liquid alloys so that they solidify without crystallization, are metastable materials with unusual structures and remarkable properties. Thanks to an irregular, tightly packed internal arrangement of atoms, metallic glasses are moldable like plastic when heated to the glass transition temperature, yet stronger than typical crystalline alloys at room temperature [1,2].

The most accessible method for determining the atomic structure of metallic glasses is the analysis of their diffraction of X-rays. Whereas conventional X-ray beams are completely absorbed near the surface, high energy, high flux radiation from synchrotron sources penetrates even thick samples, providing a deeper understanding of the unique internal structure of metallic glasses.

Using real space pair distribution functions (PDFs) derived from high precision x-ray diffraction data we attempt to probe the internal structure of Zr-Cu metallic glasses and investigate the effect of a third element (Al) in their atomic structure. The results indicate that Zr-Cu binary metallic glasses can be satisfactorily approximated as ideal solid solutions because of the relatively weak interactions between Zr and Cu atoms, figure 1a. Whereas only very thin glasses can be cast from binary Zr–Cu liquid alloys, addition of a third strongly interacting component, aluminum (Al), to the liquid alloy leads to easier glass formation such that they can be cast to thicknesses up to several millimeters without crystallization.

Ternary Zr–Cu–Al metallic glasses deviate markedly from ideal solution behavior, figure 1b. Zr–Cu–Al glasses are comprised of finite regions of atomic order, providing the extra support needed to form thicker glasses. This phenomenon is believed to be due to strong attractive interactions between the sp-electrons of
aluminum and the d-electron shell of the larger zirconium atoms. In addition, Al has an intermediate atomic size between those of Zr and Cu atoms. Thus, addition of Al promotes chemical short range ordering in the liquid, improves the local packing efficiency and slows down long range atomic diffusion required for crystallization, leading to increased glass forming ability (GFA) [3]. The atoms arrange in clusters resembling icosahedra, which pack together tightly in the glass, figure 1c, as observed in a previous study [4].

In spite of the similar atomic size of Cu and Ni, Zr–Ni binary metallic glasses exhibit different structural arrangements in the short range order (SRO) and their structure cannot be approximated by an ideal solution approach, contrary to the case of Zr–Cu. The highly negative enthalpies of mixing between Zr–Ni atoms, corresponding to strongly attractive interactions, induce CSRO in Zr–Ni metallic glasses. As in Zr-Cu, addition of Al in Zr–Ni metallic glasses can increase significantly the stability of the supercooled liquid state [5].

Figure 1: Gaussian fitting of the first PDF peak for a) Zr$_{50}$Cu$_{50}$ and b) Zr$_{50}$Cu$_{40}$Al$_{10}$ metallic glasses. Thick black curves: experimental PDFs, thin red curves: calculated partial PDFs derived from the experimental total PDFs, thin blue discontinuous curves: Gaussian partial PDFs scaled to the weight factors of an ideal solution and thick discontinuous light blue curves: expected total PDFs based on the weight factors [3], c) schematic diagram showing how the atoms in Zr–Cu based metallic glasses form icosahedral clusters that pack tightly together by sharing faces (right) or edges (left) [4].

References
The 1st WPI-AIMR Awarding Ceremony

The 1st WPI-AIMR Awarding Ceremony based on the WPI-AIMR Awarding System* established in April 2010 was held at the occasion of WPI-AIMR Joint Seminar in Tohoku University on July 9.

From the applications submitted by the end of June 2010, the four laureates, Associate Professor Hitosugi, Associate Professor Nakajima (absent because of his mission), Assistant Professor Ueno, and Research Associate Fujinami, were selected and commended by Director Yamamoto.

* the WPI-AIMR Awarding System

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<th>Implementation Guidance for the WPI-AIMR Awarding System</th>
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<td><strong>1. Outline</strong></td>
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<tr>
<td>WPI-AIMR Awarding System aims to give a Certificate of Achievement and extra prize for further enhancing cutting-edge research in their respective fields to the researchers who delivered great performance on their research that fulfills our purpose and received an award from outside organizations.</td>
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<td><strong>2. Applicant Eligibility</strong></td>
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<td>WPI-AIMR Award is given to the researchers working in WPI-AIMR at Sendai excluding PIs immediately when they won the prize from foreign and domestic research organizations, academic societies, academic journals, or administrative organizations, and Institute Director evaluates their work is worth awarding.</td>
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<td><strong>3. Selection Process</strong></td>
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<td>In the case a researcher won a prize, he/she shall submit Notification of Award and the photocopy of the notice or the certificate of the award to Institute Director. Institute Director evaluates the awarding organization and the significance of the award comprehensively, and then Executive Committee conducts screening to make a final decision.</td>
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<td><strong>4. Awarding</strong></td>
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<td>The praiseworthy researchers are given a Certificate of Achievement and extra prize. The extra prize is the necessary expenses for the research activity such as travel expenses and expendable supplies. It is set to up to 300,000 yen, however, the amount is subject to change depending on each case.</td>
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Professor Ikuhara (PI) honored with Humboldt Research Award

Prof. Ikuhara, PI of WPI-AIMR, wins Humboldt Research Awards, bestowed by the Alexander von Humboldt Foundation in Germany, an organization that promotes academic co-operation between excellent scientists and scholars from abroad and from Germany. The award is granted in recognition of a researcher's entire achievements to date to academics whose fundamental discoveries, new theories, or insights have had a significant impact on their own discipline and who are expected to continue producing cutting-edge achievements in the future. Prof. Ikuhara becomes the 3rd Japanese winner in the field of materials science. The award ceremony will be held at Bamberg in Germany on March, 2011, and the reception will be given by the President of the Federal Republic of Germany on June, 2011.

Prof. Ikuhara is well known internationally recognized scientist in the field of interface science and grain boundary engineering in materials. So far, he has intensively studied the atomic and electronic structures in various ceramic interface mainly by transmission electron microscopy (STEM, HREM, EDS, EELS), and quantitatively determined the structures by combining the first principle theoretical calculations. Based on the microstructural characterization, he undertook a variety of research concerning microstructure control and high temperature mechanical properties and functional properties of ceramic materials, and achieved numerous successful results. In particularly, his research concerning grain boundary related phenomena took a world-leading role in this field of research.

The Third Series of WPI-AIMR Joint Seminars FY2010

The third series of the WPI-AIMR Joint Seminars started from April, 2010. The aim of this seminar series is to enhance mutual scientific communications among research staffs in WPI-AIMR to promote further fusion researches and the seminars are organized and managed by younger research staffs in WPI-AIMR. The Sessions are not constituted only by one way talks based on established research results, but also lively discussions and exchange of ideas.

Lineup of the third series of WPI-AIMR Joint Seminars (April to July)

1st Seminar, Apr. 9, 2010
Prof. Y. Yamamoto "How can we achieve our mission of WPI?"

2nd Seminar, May 14, 2010
Chen Group Organizer: T. Fujita, Moderator: K. Akagi
T. Fujita "Introduction"
T. Fujita "Nanoporous Metal and Composite"
A. Hirata "Applications of Super TEM / STEM installed in WPI"
D. Pan "Mechanical Behavior of Bulk Metallic Glasses"

3rd Seminar, May 21, 2010
Adschiri Group Organizer: D. Hojo, Moderator: T. Makino
S. Takami "Overview"
D. Hojo "Dispersibility of Nanocrystals --How do we utilize NCs?"
T. Togashi "Fusion Research Field of Biochemistry and Nanotechnology --Biological application of magnetic nanocrystals"
S. Harton "Directed Assembly of Fe3O4 Nanocrystals using Block Copolymers"

4th Seminar, Jun. 4, 2010
Esashi and Gessner Group Organizer: Y. Lin, Moderator: A. Lahiri
Y. Lin "Introduction/Overview"
Y. Lin "MEMS bonding technology with MGs and nanostructure"
J. Lee "MG materials for the MEMS structure; The Freestanding PZT Film for MEMS using Ge sacrificial layer"
N. Kaushik "Application of metallic glass thin films in patterned recording media"
S. Yoshida "MEMS-based data storage system using conductivity-switching polymers"
M. Muroyama "Networked Tactile Sensing Systems for Human Assist and Friendly Robots"
5th Seminar, Jun. 25, 2010
Hitosugi Group  Organizer: T. Hitosugi, Moderator: T. Fujita
T. Hitosugi "Overview"
T. Ohsawa "Current Issues on Atomic-scale Nature of Functional Metal Oxides"
K. Iwaya "STM for Fusion Research"
S. Shiraki "Magnetism of 1D- and 2D arrays of magnetic atoms and molecules on surfaces"

6th Seminar, Jul. 9, 2010
Ikuhara Group  Organizer: S. Tsukimoto, Moderator: Y. Lin
S. Tsukimoto "Introduction/Overview"
L. Gu "Zooming into an Atomic World: The Choice of Aberration-Corrected Transmission Electron Microscopy (TEM)"
M. Saito "Atomic-Scale Characterizations of Functional Materials by TEM/STEM"
Z. Wang "Introducing First-Principles Calculations into the State-of-the-Art Electron Microscopy: an In-Depth Looking at Materials"

7th Seminar, Jul. 23, 2010
Louzguine Group  Organizer: C. Qin, Moderator: S. Mizukami
C. Qin "Introduction/Overview"
Q. Zhang "Design and fabrication of bulk metallic glasses"
N. Chen "Development of new bulk metallic glasses with unique properties"
A. Caron "1. “Effect of minor composition changes in the Zr-Al-(CuNiCo) bulk metallic glassy system”  2. “Application of atomic force acoustic microscopy in materials science”"
S. González "Mechanical properties of Mg-Li-Cu-Y multiphase composites"
The 2\textsuperscript{nd} Seminar (Chen Group)
Nanoporous Metal, Advanced TEM/STEM, and Bulk Metallic Glass

Organizer: T. Fujita (Advanced Functional Materials Lab., BMG)

After brief introduction of Joint Seminar of WPI-AIMR, the three major research subjects of Chen lab are introduced. Three subjects are (1) nanoporous metal and composite, (2) applications of super TEM/STEM installed in WPI, and (3) mechanical behavior of bulk metallic glasses. The summary of three subjects is as follows:

(1) Nanoporous metals (NPMs) made by dealloying represent a class of functional materials with the unique structural properties of mechanical rigidity, electrical conductivity, and high corrosion resistance. They also possess a porous network structure with feature dimensions tunable within a wide range from a few nanometers to several microns. Coupled with a rich surface chemistry for further functionalization, NPMs have great potential for applications in heterogeneous catalysis, electrocatalysis, fuel cell technologies, biomolecular sensing, surface-enhanced Raman scattering (SERS).

(2) Basic functions and applications of super TEM/STEM installed in WPI will be introduced. The super TEM/STEM enables us to see atomic scale structural and chemical information through HAADF, EDS, and EELS techniques. We will show the preliminary experimental results obtained from nanoparticle, nanoporous metal, graphene oxide, and bulk amorphous materials.

(3) The widespread enthusiasm for research on bulk metallic glasses is driven by both a fundamental interest in the structure and properties of disordered materials and their unique promise for structural and functional applications. Unlike the case for crystalline materials, the disordered and nonequilibrium nature of metallic glasses causes their underlying deformation mechanisms to be poorly known. A definite correlation between mechanical behavior and the atomic/electronic structures of metallic glasses has not been established. To this presentation, the micromechanisms of mechanical behavior of metallic glasses and present a brief overview of the current understanding of their strength, ductility, and plasticity at the microscopic and atomic scales.

References
The 2nd Seminar (Chen Group)
Nanoporous Metal, Advanced TEM/STEM, and Bulk Metallic Glass
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The 3rd Seminar (Adschiri Group)
Utilization of Metal Oxide Nanocrystals ~ Disperssion and Application
Organizer: D. Hojo (Organosoft and Hybrid Materials Lab., Soft Materials)

Dispersibility of nanocrystals (NCs) in solutions is one of the critical issues to utilize NCs. Due to high surface energy of NCs, NCs are easily aggregated in the solution preventing from exhibiting true nanometer-size effects of NCs [1]. The mechanism of dispersion of NCs in a solution was mentioned in terms of the interaction of NCs and the solution. The method of making NCs accessible to the chemical reaction as a new molecular system, which consists of organic molecules and inorganic solid surfaces, was also a topic of the talk [2].

We chose two topics for application of NCs in this seminar. One is related to the biological application and other is related to polymer technology.

The biological application is one of the promising applications of magnetic NCs. We introduced why magnetic nanocrystals were focused on the biological field and why the NCs can be used as a drug carrier. In addition, we also showed our research activity on the nanobio-technology field, such as a topic of biomolecules applying to nanostructured inorganic materials.

Polymer nanocomposites have gained considerable attention in recent years due to the ability to improve properties such as mechanical strength, electrical conductivity, and permeability/selectivity with relatively low-cost polymers. More recently block copolymers (BCPs), polymers with chemically different segments in long sequences or “blocks,” have been used as templates to direct assembly of NCs within BCP nanodomains. We discussed the production of magnetic/paramagnetic thin films (~100 nm) using magnetite (Fe₃O₄) NCs within block copolymer templates. By controlling the interactions between the NC surface, through ligand chemistry, and the two blocks of the BCP, this same technology should be able to simultaneously direct assembly of the NCs and BCP nanodomains in the presence of a magnetic field as well as allow reversible “switching” of domain directionality.

References
Micro/Nano Electro Mechanical Systems (MEMS/NEMS) is the integration of mechanical elements and electronics on a common silicon substrate through micro/nano fabrication technology. Multitudinous functionalities of components of sensors and actuators could be realized with various materials by using compatible micro/nano machining processes [1-2]. MEMS was born from semiconductor technologies, and over last tens years many functional materials (e.g. piezoelectric materials, CNTs, polymers, SiC etc.) have been adjected into this field and have opened a new and large regime for MEMS application [3-4].

MEMS materials laboratory in WPI-AIMR aims at developing practical MEMS devices and technologies using novel materials as well as nanostructures. Bulk metallic glasses are well-known with their superior properties, and have been employend to device structures, patterned recording media, and bonding intermediate. Furthermore, a scientific well-studied random porous nanostructure is used for industrially standrad MEMS process to enhance conventional bonding functionality. PZT is a famous actuation material utilized for movable MEMS devices. The new process for free-standing films was developed using Ge sacrificial layer and has realized thick films fabrication. Conductivity-switching polymers have shown their valuable roles on MEMS-based data storage system as recording media, which is expected to become the future data storage system with a recording density beyond 1 Tb in². Forward future application of smart integrated MEMS and LSI, tactile sensor systems of human assist and friendly robots are developed for safety/smooth interaction between humans and robots.

MEMS is continuously meeting new materials and creating colorful functionalities.

References
The 5th Seminar (Hitosugi Group)
Atomic-scale Characterization of Perovskite Oxide Thin Films
Using Scanning Tunneling Microscopy


Atomically-controlled thin film growth of transition-metal-oxide heterostructures has opened up new perspectives for surface and interface physics. For example, metallic and magnetic interfaces have been discovered between two nonmagnetic perovskite insulators [1,2]. To further explore unique functionalities at these surfaces and interfaces, more attention should be paid to the electronic structure of the substrate surfaces at the atomic scale.

Among a wide variety of substrates, strontium titanate SrTiO$_3$ has been most commonly used for growing perovskite heterostructures. However, atomic-scale understanding of the surface structures has been still incomplete.

Scanning tunneling microscopy/spectroscopy (STM/STS) are ideal probes for this purpose. We have recently constructed a new STM combined with pulsed laser deposition (PLD), which enables low temperature STM/STS measurements of thin films without exposing their surfaces to air.

We have revealed that SrTiO$_3$(001) substrate prepared by a typical annealing treatment resulted in disordered surface on an atomic scale [3]. To obtain more ordered surface, homoepitaxial SrTiO$_3$ thin films were grown in different growth mode on this disordered substrate, and their surface structures were characterized by STM [3,4]. It was found that the atomic-scale surface structure including oxygen vacancy strongly depends on not only the growth condition but also the film thickness. Based on these results, we can provide a microscopic picture of the near-surface stoichiometry of thin films, which could affect the electronic properties at the SrTiO$_3$-based interfaces.

References
Atomic-resolution characterization of structures is requisite for understanding of the atomistic mechanisms of physical phenomena related to various properties in functional materials and electronic devices. In recent characterization technologies, there has been remarkable progress by Scanning Transmission Electron Microscopy (STEM) utilizing the spherical aberration corrector, resulting in enhancement of the special resolution (~1 Å). In addition, the light elements such as carbon, lithium (Li) and oxygen, which compose Li ion battery and organic compound etc., are possible to observe directly using the STEM technique, e.g. Super-TEM facility installed in WPI-AIMR. The STEM technique enables us not only to observe typical microstructure but also to identify the locations of atom/dopants, and analyze the local electronic state in materials. Thus, TEM/STEM is one of the most powerful characterization techniques.

Accordingly, both fundamentals and applications of the TEM/STEM were introduced and our recent (fusion) researches were demonstrated in this seminar. As for fundamentals, the mechanics, image formation and merit/demerit of the TEM/STEM were explained minutely by Lin Gu. As for fusion researches, M. Saito introduced the latest research topics: (i) structure of nano-wiring fabricated in MgO bicrystals [in collaboration with Schluger group], and (ii) direct evidence of B-site ordering of Fe and Ta ions in double-perovskite Sr$_2$FeTaO$_6$ thin films with unique magnetic properties [in collaboration with Kawasaki group] [1]. Z.C. Wang demonstrated the important advance for merging the state-of-the-art STEM with the high-precision first principle calculations in the atomic-scale determination of complex structures as well as in relating that structure to its properties on a quantum level based on our recent researches [2, 3]. As a consequence, the atomic-structure characterization using STEM could play an important role in rapid progress in the fusion researches in WPI-AIMR.

References
The 7th Seminar (Louzguine Group)
Development of New Bulk Metallic Glasses with Unique Properties

Organizer: C. Qin (Advanced Functional Materials Lab., BMG)

A glass has very slow crystal nucleation and growth kinetics that the liquid can be undercooled far below its melting point before a glass transition is reached, ‘freezing’ as a vitreous solid without crystallizing. The atoms retain an amorphous distribution (i.e. random packing with no long-range order). The production of a metallic glass by rapid cooling of the molten Au_{75}Si_{25} eutectic alloy in 1960 is generally considered to be the beginning of the era of metallic glasses [1]. Since then, a number of metallic glasses have been developed in Ni-, Fe-, Pd- and Al-based alloy systems etc. More importantly, metallic glasses exhibit unique properties that are superior to the conventional crystalline metals and alloys, such as high strength, high elastic strain limit, high hardness, good soft magnetic properties, and good corrosion resistance [2-4]. However, the early metallic glasses have mainly been fabricated in the form of thin ribbons, thin films and fine wires because of their very high cooling rate requirements of above $10^5$ Ks$^{-1}$, for glass formation. The limitations of size and shape as well as low thermal stability have restricted their fields of application.

Currently, with well designed compositions it is possible to cast liquid alloys into the glassy state at low critical cooling rates from 100 Ks$^{-1}$ to 1 Ks$^{-1}$ and in large critical sample sizes up to several centimeters. A recent significant advancement has been made in developing new types of BMGs and their composites with unique properties in our group, as exemplified for Ni-free Zr-Cu-Fe-Al BMGs with extremely high glass-forming ability, ductile Pd-Ni-Si-P BMGs and lighter Mg-Li-Cu-Y glassy composites etc. In addition, the fusion researches with other groups in WPI aim at using these high-performance materials to manufacture small devices for microelectromechanical systems (MEMS), or finding novel properties of metallic glasses as being catalysts, biomaterials and new energy materials. These progresses greatly raise the hope for possible applications of metallic glasses as advanced engineering materials.

References
Research Prospect
Universality among Diversely Different Glass-Forming Materials

Michio Tokuyama 1,3, Yuto Kimura 2, Hiroyuki Fujii 2, and Yayoi Terada 3

1. Introduction

Understanding of the glass transition is one of the pioneering problems in a wide variety of fields [1-3]. It is obviously important in technological applications, such as how to manufacture window glasses, optical fibers, and bulk metallic glasses (BMG) [4]. As a liquid is cooled from high temperatures, it may crystallize at a melting temperature \( T_m \), where the first-order phase transition usually occurs. As a liquid is further cooled to temperatures below \( T_m \) without crystallization, it is called a supercooled liquid. Cooling a supercooled liquid below the glass transition temperature \( T_g \) finally makes a glass. This scenario is essentially the same as that in any viscous liquids, such as fragile liquids (OTP, Toluene, and etc.), metallic melts (Pd\textsubscript{43}Ni\textsubscript{10}Cu\textsubscript{27}P\textsubscript{20}, Zr\textsubscript{41.2}Ti\textsubscript{13.8}Cu\textsubscript{12.5}Ni\textsubscript{10}Be\textsubscript{22.5}, and etc), and strong liquids (SiO\textsubscript{2}, GeO\textsubscript{2}, and etc). If the mechanism of the glass transition is clarified in a simple system, therefore, it may help us to understand it in more complex systems. Up to now, however, there is no satisfactory theory to elucidate the glass transition from a first principle. This is mainly because in a supercooled state the nonlinear fluctuations play an important role and cause long-lived spatial heterogeneities, leading to a non-exponential two-step slow relaxation, while in a liquid state the linear fluctuations play an important role and cause short-lived spatial heterogeneities, leading to an exponential decay [5]. In a supercooled state, therefore, one has to deal with long-time correlations caused by many-body interactions among particles analytically. This is still a hard work to do theoretically, although such slow relaxation processes in viscous liquids near the glass transition are extensively studied by experiments and computer simulations [6,7] with recent progress in science and technology. Instead of doing such a analytic work, in this paper we propose an existence of universality near the glass transition among different glass-forming materials and thus review two kinds of universalities among different glass-forming liquids near the glass transition obtained by analyzing the experimental data and the simulation results consistently from a unified point of view based on the mean-field theory (MFT) recently proposed by Tokuyama [8]. Thus, we show how these
Universality among Diversely Different Glass-Forming Materials

Michio Tokuyama\(^1,3\), Yuto Kimura\(^2\), Hiroyuki Fujii\(^2\), and Yayoi Terada\(^3\)

\(^1\)WPI Research center, Advanced Institute for Materials Research, Tohoku University, Sendai 980-8577, Japan
\(^2\)Department of Mechanical Engineering, Tohoku University, Sendai 980-8579, Japan
\(^3\)Institute of Fluid Science, Tohoku University, 980-8577, Japan

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universalities make the dynamics of complex materials such as BMG simple.

One of main purposes in our theoretical group is to find a basic equation to describe the dynamics of supercooled liquids in different systems. The most famous equation to describe the dynamics of liquids is a Navier-Stokes equation. The solution depends on a viscosity, time, and length. Hence it is invariant for different liquids if the dimensionless viscosity has the same value. Up to now, the basic equation for supercooled liquids has not been derived yet. From a unified point of view based on MFT [9], however, it was shown that the similar situation to the above occurs if the dimensionless long-time self-diffusion coefficient \( D_s^L(\lambda) \) has the same value. In fact, the following two types of universalities are proposed. The first is a universality for the dynamics of self-diffusion in different systems, which is described by a master curve at a fixed value of \( D_s^L(\lambda) \) [8-12], where \( \lambda \) is a control parameter, such as an inverse temperature, a volume fraction, a magnetic field, and so on. If \( D_s^L(\lambda) \) has the same value in different systems, the dynamics of a single particle, such as a mean-square displacement \( M_s(t) \) and a mean-fourth displacement \( M_4(t) \) (or a non-Gaussian parameter \( \alpha_2(t) \)), is completely identical to each other. Thus, one can easily show that the dynamics in a fragile glass former coincides with that in a strong glass former. This enables us to study complex systems by using simple systems. The second is universality for the long-time self-diffusion coefficient \( D_s^L(\lambda) \). For atom or colloid \( \alpha \), there exists a master curve for \( D_s^L(\lambda) \), even if \( \alpha \) belongs to a complex multi-component system \( A_B C_z \cdots \), where \( \alpha \in \{A,B,C\ldots\} \). By using this master curve, therefore, one can easily predict the whole \( \lambda \) dependence of \( D_s^L(\lambda) \) from a few data points [13].

From these universalities, we conclude that the value of \( D_s^L(\lambda) \) uniquely determines the state of the system, such as a liquid state [L], a supercooled state [S], and a glass state [G]. Hence it is convenient to introduce a universal parameter \( u \) by \( u = \log (\sigma v_0 / D_s^L) \), where \( \sigma \) is a particle diameter and \( v_0 \) an average particle velocity. Once the glass transition point \( u_g \) is found in one system, then, one can use it for other systems to determine the glass state [10]. As \( \lambda \) is increased from a liquid state [L], a supercooled state [S], and a glassy state [G] appear at \( \lambda_p \) (or \( u_p \)) and \( \lambda_g \) (or \( u_g \)), respectively, where \( \lambda_g > \lambda_p \) (\( u_g > u_p \)). In fact, detailed analyses of experimental data for suspensions of colloids [13] suggest that \( u_p \approx 2.6 \) and \( u_g \approx 5.1 \). Thus, these values must be universal numbers [9]. In Fig. 1, each state is classified by \( u \). The mean-field region in which MFT is valid depends on the system. For molecular systems it is valid for \( 0 < u \leq 3.04 \), while \( 0 < u < u_g \) for suspensions. Here we note that the solutions of the mode-coupling theory show \( \alpha_2(t) < 1 \) for any systems. Hence the mode-coupling theory (MCT) is also valid for the same mean-field region as that of suspensions.
2. Mean-Field Theory

We briefly summarize the mean-field theory of the glass transition (MFT) recently proposed by Tokuyama [8-10]. Since we are interested only in self-diffusion of particle $\alpha$, the relevant physical quantities to describe the dynamics of particle $\alpha$ are mainly given by the mean-square displacement $M_2^{(\alpha)}(t) = \langle |X^{(\alpha)}_i(t) - X^{(\alpha)}_i(0)|^2 \rangle$, and the mean-fourth displacement $M_4^{(\alpha)}(t) = \langle |\vec{X}^{(\alpha)}_i(t) - \vec{X}^{(\alpha)}_i(0)|^4 \rangle$, where $\vec{X}^{(\alpha)}_i(t)$ denotes the position vector of $i$th particle of $\alpha$ at time $t$. Then, the non-Gaussian parameter is given by $\chi_2^{(\alpha)}(t) = (3/5) M_4^{(\alpha)}(t) / M_2^{(\alpha)}(t)^2 - 1$. As shown in the previous paper [8], $M_2^{(\alpha)}(t)$ is described by the following two types of mean-field equations:

for suspensions of colloids (S)

$$\frac{d}{dt} M_2^{(\alpha)}(t) = 6D_0^x(\lambda) + 6[D_0^x(\phi) - D_0^x(\lambda)]^{M_2^{(\alpha)}(t)^{1/2}}, \quad (1)$$

and for molecular systems (M)

$$\frac{d}{dt} M_2^{(\alpha)}(t) = 6D_0^x(\lambda) + 6[D_0^x(\phi) - D_0^x(\lambda)]^{M_2^{(\alpha)}(t)^{1/2}}, \quad (2)$$

where $D_0^x(\phi)$ is the short-time self-diffusion coefficient [15] and $v_{in} = (k_B T / m_x)^{1/2}$, $m_x$ being mass of a particle $\alpha$. Here the mean-free path $\ell$ is a length in which a particle can...
move freely without any interactions between particles. Although it is originally related to the static structure factor $S(q)$ [9], it is determined by a fitting with data here. Equations (1) and (2) are easily solved to give formal solutions

$$M^{(o)}_{\perp}(t) = 6D^t_{\perp}t + 1^2 \ln \left( e^{6t/t_\perp} + \frac{D^t_{\perp}}{D^t_{\parallel}} \left[ 1 - e^{6t/t_\parallel} \right] \right)$$

(3)

$$M^{(o)}_{\parallel}(t) = 6D^t_{\parallel}t + 1^2 \ln \left( e^{6t/t_\parallel} + \frac{1}{6} \frac{t_\parallel}{t_\perp} \right) \left[ 1 - \left( 1 + \frac{6t}{t_\parallel} e^{6t/t_\parallel} \right) \right]$$

(4)

where $t_\parallel$ denotes a time for a particle to diffuse over a distance of order 1 with the diffusion coefficient $D^t_{\parallel}$ and is identical to the so-called $\beta$-relaxation time. Here $t_\perp$ is a mean-free time, within which each particle can move freely without any interactions between particles. As shown in the previous paper Ref. [10], the mean-free path $l$ is uniquely determined by $D^t_{\parallel}$. Hence the solutions (3) and (4) suggest that the dynamics is described by only one parameter $D^t_{\parallel}$.

Next we discuss the long-time self-diffusion coefficient $D^t_{\perp}$. As discussed in Ref. [11], it can be written in terms of a singular function of $\lambda$ as

$$D^t_{\perp}(\lambda) = \frac{D^t_{\parallel}(\phi)}{1 + \kappa_{\alpha\alpha} D^t_{\parallel}(\phi) \left( \frac{\lambda}{\lambda_c} \right)^{1 - \frac{3}{2}} \left( \frac{\lambda_c}{\lambda_c} \right)}$$

(5)

$$\frac{D^t_{\perp}(\lambda)}{\sigma_{\alpha\alpha} v_0} = \frac{1}{\kappa_{\alpha\alpha} \left( \frac{\lambda}{\lambda_c} \right)^{1 - \frac{3}{2}} \left( \frac{\lambda_c}{\lambda_c} \right)}$$

(6)

where $\lambda_c$ is a fictive singular point to be determined and $v_0 = (c_{\alpha\alpha}/m_\alpha)^{1/2}$, $c_{\alpha\alpha}$ being an energy. As shown in the previous paper [11], one can obtain the coefficient $\kappa_{\alpha\alpha}$ analytically in terms of potential parameters as

$$\kappa_{\alpha\alpha} = \frac{\sigma_{\alpha\alpha}}{c_{\alpha\alpha}} \left( \frac{c_{\alpha\alpha}}{c_{\alpha\alpha}} + \frac{U^{rep}(r)}{c_{\alpha\alpha}} \right) - \sigma_{\alpha\alpha},$$

(7)

where $U^{rep}(r)$ denotes a repulsive part of the potential $U_{\alpha\alpha}(r)$. The singular part of Eqs. (5) and (6) results from the long-time correlation effects due to the many-body interactions between particles and is in general difficult to calculate. In this paper, we treat it as a fitting parameter. For molecular systems (M), length, time, and temperature are scaled by $\sigma_{\alpha\alpha}$, $t_\perp$, and $c_{\alpha\alpha}/k_B$, respectively.

3. Universality for the dynamics of self-diffusion

We now discuss universality in the dynamics of self-diffusion among different glass-
analytically in terms of potential parameters as where it can be written in terms of a singular function of suggests that the dynamics is described by only one parameter Here Equations (1) and (2) are easily solved to give formal solutions to the static structure factor. Moving freely without any interactions between particles. Although it is originally related to the diffusion of hard spheres in the hard-gas system, as shown in the previous papers [9, 12], the dynamics of self-diffusion in any systems is described by a master curve if the value of \( u \) is the same as each other. We discuss the following three cases: We first discuss universality between self-diffusion of colloids with 6% size polydispersity [14] and that of A colloids in the Lennard-Jones binary colloids \( A_{80}B_{20} \) [16]. In Fig. 2, the mean-square displacement \( M_s(t) \) is compared at two different values of \( u \); in a liquid state [L] \( u \approx 1.347 \) where \( \phi=0.502 \) for the hard-sphere colloid and \( T=1.0 \) for the binary colloids and a liquid state [L] \( u \approx 2.273 \) where \( \phi=0.538 \) and \( T=0.6 \). At each value of \( u \) the experimental data and the simulation results are collapsed on the mean-field master curve given by Eq. (3). We next discuss universality between self-diffusion of hard spheres in the hard-sphere fluid with 6% size polydispersity and that of A particles in the Lennard-Jones binary mixtures \( A_{80}B_{20} \). In Fig. 3, the mean-square displacement \( M_s(t) \) is compared at three different values of \( u \); in a liquid state [L] \( u \approx 1.97 \) where \( \phi=0.525 \) for the hard-sphere fluid and \( T=1.0 \) for the binary mixtures, in a weak supercooled state \( [S_1] u \approx 2.77 \), where \( \phi=0.56 \) and \( T=0.625 \), and in a strong supercooled state \( [S_2] u \approx 4.44 \), where
Fig. 3. A log-log plot of $M_2(t)$ versus $t/t_{th}$ for hard-sphere fluids and Lennard-Jones binary mixtures. The filled circles indicate the simulation results for hard spheres with 6% size polydispersity at [L] $\phi=0.525$, [S$_1$] 0.56, and [S$_2$] 0.581 from Ref. [17] and the open squares for A particles at [L] $T=1.0$, [S$_1$] 0.625, and [S$_2$] 0.455 from Ref. [18]. The solid lines indicate the mean-field master curve given by Eq. (6), where $u \approx 1.97$ [L], 2.77 [S$_1$], and 4.44 [S$_2$].

Fig. 4. A log-log plot of $M_4(t)$ versus $t/t_{th}$ for hard-sphere fluids and Lennard-Jones binary mixtures. The solid lines are mean-field results. The details are the same as in Fig. 3.
Fig. 3. A log-log plot of $M_2(t)$ versus $t/t_0$ for hard-sphere fluids and Lennard-Jones binary mixtures. The filled circles indicate the simulation results for hard spheres with 6% size polydispersity at $L = 0.525$, $S_1 = 0.56$, and $S_2 = 0.581$ from Ref. [17] and the open squares for A particles at $L = 1.0$, $S_1 = 0.625$, and $S_2 = 0.455$ from Ref. [18]. The solid lines indicate the mean-field master curve given by Eq. (6), where $\sigma| = 1.97 [L]$, $2.77 [S_1]$, and $4.44 [S_2]$.

Fig. 4. A log-log plot of $M_4(t)$ versus $t/t_0$ for hard-sphere fluids and Lennard-Jones binary mixtures. The solid lines are mean-field results. The details are the same as in Fig. 3.

Fig. 5. A log-log plot of $M_2(t)$ versus $t/t_0$ for A in LJ binary mixture, Cu in Cu$_{60}$Ti$_{20}$Zr$_{20}$ melt, and O in SiO$_2$ melt. The filled circles indicate the simulation results for A in A$_{80}$B$_{20}$ at $L = 1.0$ and $S_2 = 0.588$ from Ref. [18], the open circles for Cu at $L = 1.0$ $T = 0.23$ from Ref. [20], and the open squares for O at $S_2 = 0.2165$ from Ref. [22]. The solid lines indicate the mean-field master curve given by Eq. (6), where $u \approx 1.95 [L]$ and 2.93 [S].

$\phi = 0.581$ and $T = 0.455$. At each value of $u$ the simulation results are collapsed on the mean-field master curve given by Eq. (6), except at a cage stage in $[S_2]$, where the simulation results seem not to reach a final equilibrium state yet. In Fig. 4, the mean-fourth displacement $M_4(t)$ is also shown under the same conditions as those in Fig. 3. At each value of $u$, the dynamics of $M_4(t)$ between different systems coincides with each other within error.

Finally, we discuss universality among self-diffusion of O in SiO$_2$, that of Cu in Cu$_{60}$Ti$_{20}$Zr$_{20}$ melt, and that of A particles in the Lennard-Jones binary mixtures A$_{80}$B$_{20}$ (LJ). In Fig. 5, $M_2(t)$ is compared at two different values of $u$; in a liquid state $[L]$ $u \approx 1.95$, where $T = 1.0$ for LJ and $T = 0.23$ for Cu, and in a supercooled liquid state $[S_2]$ $u \approx 2.93$, where $T = 0.588$ for LJ and $T = 0.2165$ for O. At the same value of $u$ all the simulation results on fragile and strong glass formers are collapsed on the mean-field master curve given by Eq. (4). As shown above, the dynamics of self-diffusion in different systems is identical to each other if $u$ has the same value. Thus, we conclude that at each value of $u$ there exists a master curve for $M_2(t)$ and $M_4(t)$. We next
4. Universality for long-time self-diffusion coefficient

Finally, we discuss universality for the long-time self-diffusion coefficient. For the same type of particles, the coefficient $\kappa_{\text{Nil}}$ is always the same. For example, $\kappa_{\text{A4}}=48$ for A particles in multi-component Lennard-Jones mixtures $A_xB_yC_z\ldots$ and $\kappa_{\text{CuCu}}=9$ for Cu in multi-component $\text{Cu}_x\text{Ti}_y\text{Zr}_z$ melt. The difference appears only in the singular point $\tilde{\lambda}_c$, which depends on the details of the systems. This suggests that there exists a master curve for $D^c_s(\lambda)$ of each component if $\lambda$ is scaled by $\tilde{\lambda}_c$. We first show this by using the experimental data for self-diffusion of Ni in different liquids, Ni [22], Ni$_8$P$_{20}$ and Pd$_{40}$Ni$_{40}$P$_{20}$ [23], Pd$_{43}$Ni$_{10}$Cu$_2$P$_{20}$ [24], and Pd$_{40}$Ni$_{10}$Cu$_{30}$P$_{20}$ [25]. Ni is well described by the Stillinger-Weber potential [26] whose repulsive part is given by $U_{\text{Nil}}^{\text{rep}}(r) = \varepsilon \sigma_{\text{Nil}}^2 / r^2$, where $\sigma_{\text{Nil}} = 2.2183\text{Å}$, $\varepsilon = 1.2879 \times 10^{-20}\text{J}$, and $m_{\text{Ni}} = 1.0284 \times 10^{-23}\text{Kg}$. Hence Eq. (7) leads to $\kappa_{\text{Nil}}=12$. By using Eq. (3), one can then find the singular temperature $T_c$ for each liquid, which is listed in Table 1. In Fig. 6, $D^c_s$ is plotted versus $T_c/T$ for different liquids. All the experimental data are collapsed onto a single master curve. The data can be described by the mean-field equation (6) with
Ni in liquids, a single master curve. The data can be described by the mean-field equation (6) with Pd using the experimental data for self-diffusion of Ni in different liquids, Ni [22], Ni [23], Pd [23], (Ref. [18], and the squares for A particles in A / 3.45 from Ref. [18], and the diamonds for A particles in A / 80 . The circles indicate the simulation results for A particles in A / 80 and the squares for A particles in A / 100 from Ref. [18], and the diamonds for A particles in A / 80 S from Ref. [27]. The dotted line indicates the mean-field result given by Eq. (6) with $\kappa_{A_d} = 48$ and the solid line a Vogel-Fulcher-Tamman fit from Ref. [13].

![Fig. 6. A log plot of $D_S^{L-c} / T$ versus $T_c / T$ for A particles in different systems, A_{100}, A_{80}B_{20}$, and A_{80}B_{20}S. The circles indicate the simulation results for A particles in A_{80}B_{20} and the squares for A particles in A_{100} from Ref. [18], and the diamonds for A particles in A_{80}B_{20}S from Ref. [27]. The dotted line indicates the mean-field result given by Eq. (6) with $\kappa_{A_d} = 48$ and the solid line a Vogel-Fulcher-Tamman fit from Ref. [13].](image)

Table 1. Singular temperatures for different systems [13].

<table>
<thead>
<tr>
<th>System</th>
<th>$1/T_c$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni</td>
<td>1.05</td>
</tr>
<tr>
<td>Ni_{80}P_{20}</td>
<td>1.11</td>
</tr>
<tr>
<td>Pd_{40}Ni_{10}Cu_{3}P_{20}</td>
<td>1.13</td>
</tr>
<tr>
<td>Pd_{40}Ni_{40}P_{20}</td>
<td>1.15</td>
</tr>
<tr>
<td>Pd_{40}Ni_{10}Cu_{3}P_{20}</td>
<td>1.16</td>
</tr>
<tr>
<td>A in A_{100}</td>
<td>3.45</td>
</tr>
<tr>
<td>A in A_{80}B_{20}S</td>
<td>3.41</td>
</tr>
<tr>
<td>A in A_{80}B_{20}S</td>
<td>2.06</td>
</tr>
<tr>
<td>O in SiO$_2$</td>
<td>5.92</td>
</tr>
</tbody>
</table>

$\kappa_{NiNi} = 12$ up to $u \approx 3.04$ well, while for $u > 3.04$ they start to deviate from it because the many-body correlation effects play important role. Since there is no theory in a strong supercooled liquid for $u > 3.04$, one of phenomenological theories, a Vogel-Fulcher-Tamman law, is shown in Fig. 6 for comparison [13]. The second example is Lennard-Jones liquids, A_{100} [18], A_{80}B_{20} [18], and A_{80}B_{20}S [27]. As is shown in Fig. 7, all simulation results are collapsed onto a single master curve, where $\kappa_{A_d} = 48$. Here we note that the LJ potential was originally used to model Ni. In fact, $D_S^{L-c}$ for A particle perfectly coincides with that for Ni if it is multiplied by 4.
5. Summary

In this paper, we have analyzed the simulation results and the experimental data on different glass-forming materials, including bulk metallic glass-forming Cu_{60}Ti_{20}Zr_{20} melt and network-forming SiO_{2} melt, from a unified viewpoint based on the mean-field theory. We have then shown two types of universalities among those systems. The first is an existence of a master curve for $M_2(t)$ and $M_4(t)$ at a fixed value of $u$. Hence the dynamics of self-diffusion in any systems is the same as each other at a given value of $u$ (see Figs. 2-5). The second is an existence of a master curve for $D_S^L$ on a given atom or colloid. The parameter $\kappa_{\alpha\nu}$ has the same values for a given particle $\alpha$, while the singular point $\lambda_c$ depends on the systems. Hence all the data for the same particle in different systems are collapsed on a single curve if $D_S^L$ is plotted versus $\lambda/\lambda_c$ (see Figs. 6 and 7). Thus, one can predict a whole data for $D_S^L$ by just measuring only one data for a liquid of interest. Once $D_S^L$ is given, the dynamics of that liquid is also predicted.

Acknowledgments

The authors are grateful to T. Furubayashi, P. Harrowell, Y.-H. Hwang, H. Löwen, M. Medina-Noyola, T. Narumi, K. L. Ngai, I. Oppenheim, G. Szamel, A. Takeuchi, and Th. Voigtmann for fruitful discussions. This work was supported by World Premier International Research Center Initiative, MEXT, Japan and also partially supported by Grants-in-aid for Science Research with No. 18540363 from Ministry of Education, Culture, Sports, Science and Technology of Japan.

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Summary

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Acknowledgments

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The 2009 WPI-AIMR Annual Workshop was held from March 1, Sunday through March 6, Friday, 2009, at the Miyagi-Zao Royal Hotel in Zao Town of Miyagi prefecture. One of the main purposes of the workshop was to stimulate immediate and/or future possible Fusion Researches among WPI-AIMR researchers and with outside researchers. The Program Committee has set up the plenary sessions, Mini workshops, and Poster sessions aiming for that task. The total participants of the workshop counted to 180 and the total presentation at the workshop was 164, which included 32 invited talks in the eleven plenary sessions and two Special sessions, 50 talks in eight Mini workshops, and 82 Poster presentations.

Here we present several papers of the proceedings of the Workshop whose authors kindly agreed to be included in the WPI-AIMR NEWS.

(T. Hashizume, program chair)
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(T. Hashizume, program chair)
Short-range order of Cu-Zr metallic glasses

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The atomic structure of Cu$_{40}$Zr$_{55}$, Cu$_{35}$Zr$_{60}$ and Cu$_{55}$Zr$_{30}$ metallic glasses was investigated by means of high-energy X-ray diffraction, neutron-diffraction and extended X-ray absorption fine structure spectroscopy. Three-dimensional structure models were developed by applying the Reverse Monte Carlo method. Total and partial atomic pair correlation functions were extracted from the model for the different compositions. The obtained structure models were analyzed in terms of the local atomic arrangements. The geometric short-range order of Cu-Zr glasses is characterized by a variety of polyhedra. The atomic sites of the nearest neighborhood around a Cu or Zr atom are randomly occupied by Cu or Zr atoms. The composition dependence of the short- and the medium-range order in the Cu-Zr glasses points to a solid solution-like replacement of Cu and Zr atoms in the whole composition range. No indications were observed for the existence of a special atomic arrangement at particular chemical compositions of bulk glass forming Cu-Zr alloys.

I. INTRODUCTION

In the last 15 years new bulk metallic glass (BMG) forming alloys were developed in Zr-, La-, Ti-, Ni-, Pd-, Mg-, Al-, Fe-, Cu- based systems [1,2]. These multi-component alloys can be obtained in a glassy state in mm or cm-dimension by copper mold casting due to high glass forming ability, or low critical cooling rates of $10^7$-$10^9$ K/s, respectively. This now enables to explore the exceptional mechanical properties of metallic glasses [3]. However, the information content of structural investigations is rather limited for glasses consisting of two or more different kinds of atoms.

Recently, BMGs could be prepared for some particular compositions in the binary Cu-Zr system [4-7]. Glassy samples of 1 - 2 mm thickness were reported for the alloys Cu$_{40}$Zr$_{55}$ [4], Cu$_{35}$Zr$_{60}$ [5], Cu$_{50}$Zr$_{50}$ [6], and Cu$_{55}$Zr$_{30}$ [7]. Furthermore, extraordinary plasticity was observed for some binary and ternary Cu-based BMGs [8-12]. These findings renewed the interest in structural investigations of the Cu-Zr system. It is well known from earlier work that Cu-Zr glasses can be prepared by rapid quenching from the melt in a wide composition range [13-16]. Partial atomic pair correlation functions of amorphous Cu-Zr alloys were determined by anomalous X-ray scattering and extended X-ray absorption fine structure (EXAFS) but the accuracy suffered from the weak energy dependence of the atomic form factors [17-19].

Molecular dynamic (MD) simulations of Cu-Zr glasses were done to correlate the atomic structure with mechanical properties and glass forming ability [20-23]. The atomic structure of metallic liquids and glasses is discussed by efficient packing of clusters [24]. For glassy alloys with low solute concentrations, the medium-range order is determined by packing of one dominant chemically defined polyhedron [25]. For more concentrated compositions the situation is much more complex, due to cluster overlap via solvent-sharing. For some liquid metals and metallic alloys the presence of an icosahedral atomic arrangement was concluded [26-28]. Icosahedral Cu$_{50}$Zr$_{30}$ clusters are discussed to be dominant in the Cu-Zr system from electronic arguments stabilizing the undercooled Cu-Zr liquid [29]. The presence of icosahedral clusters in bulk glass forming Cu$_{40}$Zr$_{55}$ was also concluded from Reverse Monte Carlo simulations (RMC) using XRD and EXAFS measurements [30] in contradiction to results reported on Cu$_{50}$Zr$_{50}$ glass [31]. From the analysis of composition dependent structural parameters no evidence for the existence of a special atomic arrangement in Cu-Zr glasses was found [16].

The aim of this work was, therefore, to get more insight into the atomic structure of Cu-Zr glasses from the experimental point of view. Experimental X-ray and neutron diffraction data were determined for different chemical compositions, which may act as critical tests for structure models. The partial atomic pair correlations and local structure information were extracted by applying the RMC method [32] which is meanwhile a well-established method for modelling the structure of glasses and disordered materials. Despite the fact, that RMC may give the most randomized configuration, the method has been proven to reproduce the general features and local atomic arrangements of coordination polyhedra present in the structure e.g. of covalently bonded glasses [33,34].

II. EXPERIMENTAL

Pre-alloyed ingots of nominal compositions Cu$_{40}$Zr$_{55}$, Cu$_{35}$Zr$_{60}$ and Cu$_{55}$Zr$_{30}$ were prepared by arc-melting elemental Cu and Zr with a purity of 99.9% or better in a Ti-gettered argon atmosphere. For reaching homogeneity, the samples were remelted several times. From these pre-alloys, 3 mm wide and 30 μm thick amorphous ribbons were prepared by means of rapid quenching from the melt using a single-roller melt-spinner under argon atmosphere. The chemical compositions of the ribbons were analyzed by the titration technique. The results of the analysis are in a good agreement with the nominal values which are used in the following (deviations are < 0.2 at%). The amorphous state of the as-quenched ribbons was confirmed for all compositions by the presence of only diffuse maxima in the XRD pattern, as well as by analyzing their crystallization behaviour. High
energy X-ray diffraction (XRD) experiments were conducted at the synchotron beam-line BW5 at HASYLAB, Hamburg. Intensities were recorded in transmission geometry by a Ge-point detector using a wavelength of $\lambda = 0.073$ nm. Neutron scattering experiments were carried out at the 7C2 diffractometer at the Laboratoire Léon Brillouin in Saclay. Samples were placed in vanadium containers (diameter 8 mm, wall thickness 0.1 mm) and measured using a wavelength of $\lambda= 0.073$ nm. The raw data were corrected for empty instrument background, scattering of the sample holder, absorption, incoherent and multiple scattering using standard procedures [35]. X-ray absorption spectra at the Cu- and Zr-K-edges were recorded at the beam-line C at HASYLAB, Hamburg [36]. From the recorded spectra the EXAFS-modulations were extracted by means of the usual algorithms implemented in the VIPER-programme [37]. The mass density was determined at room temperature by the Archimedes principle by weighing samples in air and in dodecane ($C_{12}H_{25}$).

### III. RESULTS AND DISCUSSION

For amorphous alloys a single diffraction experiment yields only the total structure factor $S(q) = 4 \pi \sin \theta / \lambda$ is the absolute value of the scattering vector), which is a weighted sum of the partial structure factors $S_{ij}(q)$ of the corresponding atom pairs $i,j$ [35]:

$$S(q) - 1 = \sum_i \sum_j w_{ij}(q) \cdot (S_{ij}(q) - 1).$$

The weights $w_{ij}(q)$ are determined by the sample composition $c_i$ and the scattering power of the atoms $i$. In the Faber-Ziman formalism [38] they can be expressed as

$$w_{ij}^X(q) = (2 - \delta_{ij}) c_i c_j f_i(q) f_j(q) \left( f(q) \right)_{ij}^2,$$

$$w_{ij}^N = (2 - \delta_{ij}) c_i c_j b_i b_j \left( b \right)_{ij}^2.$$  \hspace{1cm} (2)

Where $\delta_{ij}$ is the delta symbol of Kronecker, $f_i(q)$ is the $q$-dependent atomic form factor in case of X-ray scattering experiments (index X) and $b_i$ is the constant coherent neutron scattering length (index N). The corresponding weights $w_{ij}$ for the Cu-Zr glasses are given in table 1. From the structure factor $S(q)$ the total atomic pair correlation functions $g(r)$ are obtained by the Fourier transform of $S(q)$ according to [35]:

$$4\pi \cdot r \cdot \rho_0 \cdot (g(r) - 1) = \frac{2}{\pi} \cdot \int (S(q) - 1) \cdot q \cdot \sin(q \cdot r) \cdot dq,$$

\hspace{1cm} (3)

where $\rho_0$ is the mean atomic density ($\rho_0 = \sigma / N$, $\sigma$: mass density, $N$: average atomic mass, $\sigma$: Avogadro’s number). The total atomic pair correlation function $g(r)$ describes the weighted probability of finding an atom at a given distance $r$ from a central atom averaged over all atoms and normalized to the mean atomic density. Analogous to the total structure factor, the $g(r)$ curves represent in the $n$-component alloy the weighted sum of the $(n+1)/2$ partial atomic pair correlation functions $g_{ij}$, which describe the probability of finding an atom of kind $j$ around atom $i$:

$$g(r) = \sum_i \sum_j w_{ij} \cdot g_{ij}. $$

\hspace{1cm} (4)

Figure 1 shows the calculated total pair correlation functions $g(r)$ of the different Cu-Zr glasses obtained from the neutron and X-ray structure factors. For all $g(r)$ curves, two components of the first maximum are visible. The identification of the maxima in the total pair correlation function can be derived by the comparison with interatomic distances of crystalline Cu-Zr phases. However, this is not unambiguous. The first sub-maxima in the nearest neighborhood at $r = 0.272$ nm can be attributed to the non-resolved superposition of copper-copper ($r_{CuCu}^1$) and zirconium-copper ($r_{ZrCu}^1$) distances. The second sub-maxima at $r = 0.315$ nm corresponds to the nearest zirconium-zirconium ($r_{ZrZr}^1$) distance. This will be confirmed by the partial atomic pair correlation functions as shown later. With increasing Cu content the contributions of Cu-Cu and Cu-Zr pairs to the total atomic pair correlation functions $g(r)$ becomes more pronounced whereas the Zr-Zr contribution decreases in accordance with the composition dependence of the weights $w_{ij}$ (table 1). The variation of the weights is also responsible for the differences of the X-ray and neutron total atomic pair correlation functions.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>$\sigma$ (g/cm$^3$)</th>
<th>$\rho_0$ (nm$^3$)</th>
<th>$w_{ij}^X(q = 0 \text{ nm}^{-1})$</th>
<th>$w_{ij}^N$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu$<em>{52}$Zr$</em>{48}$</td>
<td>7.01</td>
<td>51.2</td>
<td>0.079</td>
<td>0.404</td>
</tr>
<tr>
<td>Cu$<em>{59}$Zr$</em>{41}$</td>
<td>7.33</td>
<td>57.1</td>
<td>0.177</td>
<td>0.487</td>
</tr>
<tr>
<td>Cu$<em>{66}$Zr$</em>{34}$</td>
<td>7.75</td>
<td>63.7</td>
<td>0.329</td>
<td>0.489</td>
</tr>
</tbody>
</table>
functions, i.e., the structural information, from the experimental data are obstructed by these modulations of the EXAFS signals. In the present study feff8.4 was used to obtain the backscattering factors [41].

Using the RMC method, the atomic partial pair correlation functions can be separated by simultaneously modelling X-ray and neutron diffraction measurements together with EXAFS data [39]. In this method atoms are moved around at random in a simulation box. Partial pair correlation functions are calculated from atomic positions after each move and are transformed to model the X-ray, neutron diffraction and EXAFS curves. The move is always accepted if the agreement between experimental data and model curves improves. To avoid getting trapped to local minima some moves leading to a worse agreement are also accepted. For details of the technique see [42,43].

Figure 2 shows for the Cu$_{35}$Zr$_{65}$ glass the comparison of the results of the RMC fit with the corresponding experimental data. For the other two glasses Cu$_{50}$Zr$_{50}$ and Cu$_{65}$Zr$_{35}$ similar good agreements between measured and calculated values were obtained. As a result of the RMC calculations, models of the three dimensional atomic structure of the Cu-Zr glasses were obtained. Figure 3 shows the atomic arrangements of the Cu-Zr glasses. From the model structures the partial pair correlation functions $g_{ij}(r)$ were determined, which are presented in Figs. 4. The positions of the first maximum represent the partial nearest neighbour distances $r_{ij}^1$. The integration over the first maximum gives the nearest neighbour number $N_{ij}^1$ of $j$-atoms around an $i$-atom in the interval between $r_1$ and $r_2$:

$$ N_{ij}^1 = \frac{4\pi^2}{\rho_0} \cdot c_j \cdot \rho_i \cdot \int_{r_1}^{r_2} g_{ij}(r) dr $$

The calculated values $r_{ij}^1$ and $N_{ij}^1$ are given in Table 2. The general behaviour of the corresponding partial pair correlation function is similar for the Cu-Zr glasses with different compositions. The nearest neighbour distances do not alter with the composition within the error limits of $\pm 0.002$ nm. Differences of the inter-atomic distances between the different Cu-Zr glasses occur at larger $r$-values. The maxima positions of the neighbour shells are shifted to higher values with increasing Zr-content which relates to the composition dependence of the atomic density, or the mean atomic volume respectively. The nearest neighbour numbers $N_{ij}^1$ are proportional to the concentration of the corresponding element in the glass. The monotonic behaviour of the coordination numbers with composition points to a continuous atomic substitution. This is also in agreement with the concentration dependences of the mean atomic volume and the total pair correlation functions of Cu-Zr glasses [16].
The analysis of the atomic structure of amorphous alloys requires the determination of the partial structure factors. The EXAFS signal corresponding to a single atom pair relates to the partial nearest neighbor distances. The partial pair correlation function is given by

\[ g_{ij}(r) = \frac{4\pi r \rho_i \rho_j}{\lambda^2} \int_{-\infty}^{\infty} S(q) \sin(qr) dq \]

where \( g_{ij}(r) \) is the partial pair correlation function of atom \( j \) around atom \( i \) in the interval \( r_i \) to \( r_j \), \( \rho_i \) and \( \rho_j \) are the atomic densities of \( i \) and \( j \) respectively, \( \lambda \) is the wavelength of the incident radiation, and \( S(q) \) is the structure factor. The integration is performed over the range of interatomic distances.

For binary alloys, the linear system of equations can be directly solved if three experimental datasets are available. The EXAFS signal at the Zr-edge can be used for Zr-glasses, while the Cu-edge gives information for Cu-glasses.

To obtain the backscattering factors, the EXAFS signals were calculated using the RMC method. The atomic partial pair correlations \( g_{ij}(r) \) were determined, which are presented in Table 2. The nearest neighbor distances \( r_{ij} \) for Cu-Zr glasses were obtained. Figure 3 shows the atomic structure of Cu-Zr glasses.

**Figure 2.** Comparison of experimental scattering data and RMC fit of Cu_{65}Zr_{35} glass.

**Figure 4.** Partial atomic pair correlation functions \( g_{ij}(r) \) of Cu-Zr glasses.

**Figure 3.** Atomic structure of Cu-Zr glasses: a) Cu_{65}Zr_{35}, b) Cu_{50}Zr_{50}, c) Cu_{35}Zr_{65} (Cu-atoms: red, Zr-atoms: blue).

**Table 2.** Nearest neighbor distances \( r_{ij} \), nearest neighbor numbers \( N_{ij} \) for Cu_{x}Zr_{100-x} glasses.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>( r_{CuCu} ) (nm)</th>
<th>( r_{ZrCu} ) (nm)</th>
<th>( r_{ZrZr} ) (nm)</th>
<th>( N_{CuCu}^1 )</th>
<th>( N_{ZrCu}^1 )</th>
<th>( N_{ZrZr}^1 )</th>
<th>( N_{CuCu}^2 )</th>
<th>( N_{ZrCu}^2 )</th>
<th>( N_{ZrZr}^2 )</th>
<th>( N_{CuCu}^3 )</th>
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<th>( N_{ZrZr}^3 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu_{65}Zr_{35}</td>
<td>0.261</td>
<td>0.275</td>
<td>0.314</td>
<td>4.6</td>
<td>6.9</td>
<td>3.7</td>
<td>9.4</td>
<td>11.5</td>
<td>13.1</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cu_{50}Zr_{50}</td>
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<td>0.279</td>
<td>0.313</td>
<td>5.9</td>
<td>6.0</td>
<td>6.0</td>
<td>8.0</td>
<td>11.9</td>
<td>14.0</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cu_{35}Zr_{65}</td>
<td>0.261</td>
<td>0.279</td>
<td>0.316</td>
<td>7.8</td>
<td>4.6</td>
<td>8.6</td>
<td>6.0</td>
<td>12.4</td>
<td>14.6</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
From the RMC structures models, further information can be extracted on the local structure in the Cu-Zr glasses. First, the nearest neighbourhood of Cu and Zr atoms were analyzed. For this, the kind and the number of nearest neighbours were counted around all atoms up to the first minimum of the corresponding atomic pair correlation functions. Figure 5 shows the frequency distribution of the numbers of nearest neighbours around the Cu atoms \( N_{Cu}^i \) ( \( N_{Cu}^i = N_{CuCu}^i + N_{CuZr}^i \) ), and around the Zr atoms \( N_{Zr}^i \) ( \( N_{Zr}^i = N_{ZrCu}^i + N_{ZrZr}^i \) ), respectively.

The values \( N_{Cu}^i \) are between 9 and 16 with a maximum for \( N_{Cu}^i = 12 \). The coordination number distribution around the Cu atoms does not essentially change with composition for the different Cu-Zr glasses (Tab. 2, Fig. 5). The number of nearest neighbours around Zr atoms ranges from 10 to 17 with the maximum \( N_{Zr}^i = 13 \) for the Cu_{65}Zr_{35} glass, and \( N_{Zr}^i = 14 \) for the Cu_{60}Zr_{40} glass respectively. This behaviour reflects the effect of the different atomic sizes. The larger Zr atoms can be surrounded by a higher number of the smaller Cu atoms. With increasing Cu content the number of Cu atoms around Zr statistically increases resulting in a higher mean coordination number (Tab. 2, Fig. 5).

The chemical composition of the first neighbourhood of an Cu (or Zr) atom is given by the ratio of the number atoms in the first coordination shell (around Cu: \( c_{Cu/Zr} = N_{CuCu}^1 / N_{CuZr}^1 \), and around Zr: \( c_{Cu/Zr} = N_{ZrCu}^1 / N_{ZrZr}^1 \) ). Figure 6 shows the frequency distributions \( c_{Cu} \) around the Cu atoms, and the Zr atoms respectively. The obtained frequency distributions follow probability distributions with a maximum corresponding to the average chemical compositions of the Cu-Zr glasses. There is also no obvious difference between the composition distributions around the Cu and the Zr atoms which should be the case if chemically ordered clusters would be dominant in the Cu-Zr glasses. The distributions of the kind of the first neighbour atoms around the Cu and Zr atoms point to a solid solution-like behaviour of the Cu-Zr glasses. Assuming that a Cu atom has 12 nearest neighbour sites around ( \( N_{CuCu}^1 + N_{CuZr}^1 = 12 \) ), which are randomly occupied by Cu and Zr atoms, the probability \( w(N_{CuCu}^1) \) of a certain number \( N_{CuCu}^i \) of Cu atoms ( \( N_{CuCu}^i = 0,1,...,12 \) ) is then given by:

\[
w(N_{CuCu}^i) = \left( \frac{12}{N_{CuCu}^i} \right) \times (x_{Cu})^{12-N_{CuCu}^i} \times (x_{Zr})^{N_{CuCu}^i},
\]

with \( x_{Cu} \), \( x_{Zr} \), as the average element concentrations of the Cu-Zr glass. Using eq. (9) the frequency distribution \( c_{Cu/Zr} \) can be calculated. Figure 6 shows the corresponding curves of the composition statistics for the random occupation of 12 nearest neighbours by the dashed lines. The calculations are in a qualitative agreement with the experimental results. The RMC results of the Cu-Zr glasses are therefore in accordance with the random site occupancy of the atoms in the first neighbourhood of Cu or Zr atoms. The differences occurring in detail are mainly caused by the simplification of 12 neighbours only and due to the neglect of the different atomic sizes.

To analyze the topology of the local atomic arrangements of the Cu-Zr glasses, the Voronoi tessellation method [44] was applied to the RMC data.
The Voronoi cell or the Voronoi polyhedra of an atom contains all points in space which are closer to the centre of the atom than to any other atom. The Voronoi tessellation thus produces convex polyhedral cells (polygons) which have planar faces and completely fill the space. The different coordination polyhedra surrounding a central atom can be characterized by the Voronoi index \( <n_1,n_2,n_3,... > \) where \( n_i \) denotes the number of \( i \)-edged faces of the polyhedron and \( \sum n_i \) the coordination number. Table 3 summarizes the relative frequencies of the most prominent polyhedra. A large amount of different polyhedra (> 1000) were identified in the structure of the Cu-Zr glasses. The ideal icosahedron \( <0,0,12,0> \) appeared with the highest frequency beside many other polyhedra with coordination number > 12 (Tab.3). With higher Cu content the fraction of icosahedra in the Cu-Zr glasses increases. The frequency distribution of the Voronoi polyhedra is in qualitative agreement with results of MD calculations [22,30] as well as with RMC analysis reported for Cu\(_{52}\)Zr\(_{48}\) [31] or Ni\(_{33}\)Zr\(_{67}\) [45] glasses. Several polyhedra having a high number of pentagon faces can be treated as a kind of distorted icosahedral arrangements. The frequency distribution of the two-dimensional Voronoi polygons is summarized in table 4. The pentagon is dominant (> 50%) and increases in number density with the Cu-content. A similar monotonic behaviour of the polygon distributions is reported for MD simulations of Cu-Zr glasses for a Cu content between 30 and 70 at% [22]. The variability of the local structures is probably the consequence of the substitution of Zr by Cu atoms with increasing Cu content.

### Table 3. Frequency distribution of Voronoi polyhedron of Cu-Zr glasses.

<table>
<thead>
<tr>
<th>Voronoi polyhedron</th>
<th>Cu(<em>{52})Zr(</em>{48})</th>
<th>Cu(<em>{30})Zr(</em>{50})</th>
<th>Cu(<em>{65})Zr(</em>{35})</th>
</tr>
</thead>
<tbody>
<tr>
<td>(&lt;0,2,8,1&gt;)</td>
<td>1.5</td>
<td>1.9</td>
<td>1.8</td>
</tr>
<tr>
<td>(&lt;0,2,8,2&gt;)</td>
<td>2.8</td>
<td>3.6</td>
<td>3.9</td>
</tr>
<tr>
<td>(&lt;0,3,6,3&gt;)</td>
<td>1.1</td>
<td>1.3</td>
<td>1.3</td>
</tr>
<tr>
<td>(&lt;0,0,12,0&gt;)</td>
<td>3.5</td>
<td>4.5</td>
<td>6.3</td>
</tr>
<tr>
<td>(&lt;0,10,2&gt;)</td>
<td>3.6</td>
<td>3.8</td>
<td>5.5</td>
</tr>
<tr>
<td>(&lt;0,2,8,3&gt;)</td>
<td>1.0</td>
<td>1.0</td>
<td>1.4</td>
</tr>
<tr>
<td>(&lt;0,3,6,4&gt;)</td>
<td>3.2</td>
<td>2.9</td>
<td>3.4</td>
</tr>
<tr>
<td>(&lt;0,10,3&gt;)</td>
<td>1.8</td>
<td>1.9</td>
<td>2.3</td>
</tr>
<tr>
<td>(&lt;0,2,8,4&gt;)</td>
<td>3.0</td>
<td>2.9</td>
<td>3.2</td>
</tr>
<tr>
<td>(&lt;0,3,8,3&gt;)</td>
<td>1.2</td>
<td>1.0</td>
<td>0.9</td>
</tr>
<tr>
<td>(&lt;0,3,6,5&gt;)</td>
<td>1.5</td>
<td>1.2</td>
<td>1.4</td>
</tr>
<tr>
<td>(&lt;0,10,4&gt;)</td>
<td>1.4</td>
<td>1.8</td>
<td>2.5</td>
</tr>
<tr>
<td>(&lt;0,2,8,5&gt;)</td>
<td>1.6</td>
<td>1.8</td>
<td>2.2</td>
</tr>
</tbody>
</table>

### Table 4. Frequency distribution of Voronoi polygons of Cu-Zr glasses.

<table>
<thead>
<tr>
<th></th>
<th>Cu(<em>{52})Zr(</em>{48})</th>
<th>Cu(<em>{30})Zr(</em>{50})</th>
<th>Cu(<em>{65})Zr(</em>{35})</th>
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<tbody>
<tr>
<td>Triangle</td>
<td>3.6</td>
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<tr>
<td>Tetragon</td>
<td>18.0</td>
<td>16.9</td>
<td>15.8</td>
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<tr>
<td>Pentagon</td>
<td>52.1</td>
<td>54.2</td>
<td>57.1</td>
</tr>
<tr>
<td>Hexagon</td>
<td>23.3</td>
<td>22.6</td>
<td>22.2</td>
</tr>
<tr>
<td>Heptagon</td>
<td>2.7</td>
<td>2.6</td>
<td>2.0</td>
</tr>
<tr>
<td>Octagon</td>
<td>0.2</td>
<td>0.2</td>
<td>0.1</td>
</tr>
</tbody>
</table>

### VI. CONCLUSIONS

The atomic structure of Cu-Zr glasses was analyzed by means of RMC method using XRD neutron and EXAFS. The experimental results on the composition dependence of the atomic structure of Cu-Zr glasses point to a solid solution-like replacement of Cu and Zr atoms in the whole composition range. The local structures show a large variety of polyhedra arrangements. No indications are observed that would support the existence of a dominant special structure or of distinct cluster in the atomic structure. Hence, from the structural point of view there is no obvious reason that relates to the high glass forming ability or plasticity found for particular Cu-Zr compositions.

### ACKNOWLEDGEMENTS

HASYLAB is gratefully acknowledged for the support during synchrotron beam time. The neutron diffraction measurements at Saclay (France) were supported by the European Commission under the 6th Framework Programme through the Key Action: Strengthening the European Research Area, Research Infrastructures. Contract No: HI23-CT-2003-505925. The authors are grateful to P. Jovari for RMC calculations, I. Kaban and S. Gruner for experimental support, and H. Hermann for valuable discussions.

[38] T.E. Faber, J.M. Ziman, Phil. Mag. 11, 153 (1965).
Microscopic deformation behavior and microstructural evolution in Ti-Nb-Ta-M (M=In, Ag, or Cr) β alloys

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The microstructural evolution upon compressive loading was identified for metastable Ti-Nb-Ta-M (M=In, Ag, or Cr) β alloys with different phase stability against β→α′ martensitic transformation. With increasing degree of deformation, martensitic transformation and deformation twinning are initially operative followed by slip of dislocations for the less stabilized In- and Ag-containing alloys, whereas slip is initially operative followed by fine deformation twinning for the relatively more stable Cr-containing alloy. As a result of the cumulative interactions between slip dislocations, α′ martensite and deformation twins, a pronounced grain refinement of the β grains to the nanoscale (50–100 nm) occurs in the In- and Ag-containing alloys with low stacking fault energy. A plausible mechanism is proposed for the easy deformation-induced nanostructuring.

1. INTRODUCTION

Biocompatible β-titanium alloys containing non-toxic β-stabilizing alloying elements have attracted a lot of attention for biomedical implant applications due to the advantages of lower Young’s modulus and better formability over α and α+β titanium alloys [1-8]. In terms of mechanical properties, low Young’s modulus and high yield strength, which strongly depend on alloy design and materials processing [1, 9], are desirable for β-titanium alloys for use as biomedical implants [10, 11]. For example, a solution-treated and aged Ti-29Nb-13Ta-4.6Zr (wt.%) β alloy showed a Young’s modulus of 80 GPa as well as a yield strength of 864 MPa [9]. On the other hand, a solution-treated Ti-35Nb-5Ta-7Zr (wt.%) β alloy exhibited a lower Young’s modulus of 55 GPa and a lower yield strength of 530 MPa [1]. For metastable β-titanium alloys, phase transformations play a crucial role in order to control the microstructure and the resulting mechanical properties. Depending on the stability, metastable β-titanium alloys may undergo martensitic transformations (β→α, β→α′, or β→α″) upon quenching and/or deformation [12], leading to a distinct deformation behavior. Many of the metastable β-titanium alloys favoring stress-induced martensitic transformation exhibit a shape memory effect and pseudoelastic deformation behavior [5, 6, 13]. In a hot-rolled Ti-24Nb-4Zr-7.9Sn (wt.%) β alloy composed of α′ martensite, a recoverable strain of 3.3% was achieved upon unloading at an imposed strain of 4% [6]. On the other hand, the origin of pseudoelastic behavior in Ti-xMo-4Nb-2V-3Al (x>9%, wt.%) β alloys was attributed to the formation of a new reversible orthorhombic α″ martensite other than α″ [5]. Upon unloading, a recovery of 80% of the imposed strain was reported for a Ti-10Mo-4Nb-2V-3Al β alloy [5]. However, the double yielding effect found for some β-titanium alloys favoring α″ martensitic transformation [13, 14], was not observed in Ti-24Nb-4Zr-7.9Sn [6] Ti-10Mo-4Nb-2V-3Al [5].

Usually, the differences in macroscopic deformation behavior can be elucidated by the microscopic deformation mechanisms that are operative to accommodate the deformation strain. With decreasing phase stability, the microscopic deformation mechanism of metastable β-titanium alloys varies from slip of dislocations to deformation twinning and/or martensitic transformation [7, 8, 15, 16]. As a result, the less-stabilized β alloys exhibit a low yield strength as well as large strain hardening, whereas the relatively stabilized β alloys showed high yield strength and limited strain hardening [7, 13]. The differences in mechanical properties are viewed as a reflection of whether a martensitic transformation happens. However, the microstructural evolution with increasing degree of deformation and its correlation with the deformation behavior are still not well understood and established due to the complex interaction between slip dislocations, twins and martensite. In the present study, the microstructural evolution upon compressive loading was identified for metastable Ti-Nb-Ta-M (M=In, Ag, or Cr) β alloys with different β phase stability in order to gain more insight into the correlation between the microscopic deformation mechanism, the microstructural evolution and the overall deformation behavior.

2. EXPERIMENTAL

Ti-Nb-Ta-M (M=In, Ag, or Cr) master alloys with a nominal composition of Ti-29Nb-13Ta-5M (wt.%) were prepared by arc-melting a mixture of pure elements (99.99%) under a Ti-gettered argon atmosphere. The master alloys were remelted at least four times by reversing the alloy buttons in order to avoid
inhomogeneity. Rods of 4 and 6 mm diameter were prepared by suction casting the arc-remelted alloys into cylindrical cavities of a water-cooled copper mold. Structural investigations were carried out by X-ray diffraction analysis (XRD, Cu-Kα), optical microscopy (OM), scanning electron microscopy (SEM, 30 kV) with an energy dispersive X-ray analyzer (EDX) and transmission electron microscopy (TEM, 200 kV). Alloy composition measured by SEM/EDX for the as-cast Ti-Nb-Ta-M alloys is summarized in Table 1. The content of In or Ag in the prepared master alloys deviated from the nominal composition to a certain extent due to the evaporation loss during arc melting (the boiling point of In or Ag is much lower than Ti, Nb and Ta). The samples for XRD analysis were prepared by fine polishing in order to remove the damage layer during cutting and grinding so as to avoid the formation of stress-induced martensite due to mechanical processing. Transverse sections of the resulting samples for the OM and SEM studies were prepared by conventional mechanical grinding, polishing and chemical etching in Kroll’s reagent. For TEM studies, thin slices were prepared by mechanical grinding and polishing followed by argon ion milling. The room temperature mechanical properties were characterized under uniaxial compression loading using an Instron 8562 testing machine at a strain rate of $1.2 \times 10^{-4}$ s$^{-1}$. The oxygen content analyzed using a carrier gas hot extraction (O/N analyzer TC-436/Leco) was less than 600 p.p.m.

3. RESULTS

3.1. As-cast microstructures of Ti-Nb-Ta-M β alloys

Fig. 1 shows typical microstructures for the as-cast Ti-Nb-Ta-M β alloys, which are mainly composed of dendritic β phase (Microstructures at a lower magnification were given in Ref. 7). Large-angle grain boundaries (marked by black arrows in Fig. 1) were revealed and the grain size was estimated to be about 80 μm. In the as-cast In-containing alloy (Fig. 1 (a)), the athermal $\alpha''$ martensite [14] was clearly observed to form at the interdendritic zones, as marked by the white arrow in Fig. 1 (a). Similarly, $\alpha''$ martensite was also identified at the interdendritic zones in the as-cast Ag-containing alloy (Fig. 1 (b)). However, the $\alpha''$ martensite plates formed in the as-cast Ag-containing alloy were not clearly visible. Detailed investigations (inset of Fig. 1 (b)) indicated that the β phase was composed of tiny domains of about 100 nm. Compared with the as-cast In- and Ag-containing alloys, the as-cast Cr-containing alloy exhibited a microstructure consisting only of β phase without the

![Fig. 1. SEM micrographs of the as-cast In-, Ag- and Cr-containing β-titanium alloys. (a) Ti-Nb-Ta-In; (b) Ti-Nb-Ta-Ag; (c) Ti-Nb-Ta-Cr. The inset in (b) indicates the formation of tiny domains in the β dendrites of the Ti-Nb-Ta-Ag alloy. GB: grain boundary.](image)

<table>
<thead>
<tr>
<th>Alloys</th>
<th>Area</th>
<th>Alloy composition (wt. %)</th>
<th>Ti</th>
<th>Nb</th>
<th>Ta</th>
<th>In, Ag or Cr</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti-Nb-Ta-In</td>
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</tr>
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<td>D</td>
<td></td>
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<td>36.4</td>
<td>23.2</td>
<td>1.5</td>
</tr>
<tr>
<td></td>
<td>I</td>
<td></td>
<td>54.9</td>
<td>31.1</td>
<td>11.4</td>
<td>2.6</td>
</tr>
<tr>
<td>Ti-Nb-Ta-Ag</td>
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<td>36.0</td>
<td>23.4</td>
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</tr>
<tr>
<td></td>
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<td>59.5</td>
<td>28.6</td>
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<td>5.9</td>
</tr>
</tbody>
</table>

O: overall; D: dendritic; I: interdendritic zone

Table 1. Measured alloy composition for Ti-Nb-Ta-M (M=In, Ag, or Cr) β alloys.
formation of athermal $\alpha''$ martensite (Fig. 1 (c)).

Fig. 2 shows the XRD patterns of the as-cast Ti-Nb-Ta-M $\beta$ alloys. The bcc $\beta$ phase was identified as dominant phase in all alloys, which is in agreement with the microstructure identification. The formation of the orthorhombic $\alpha''$ martensite in the as-cast In- and Ag-containing alloys was also evidenced, especially in the XRD patterns in the range of $33^\circ \leq 2\theta \leq 45^\circ$ at a slow scan rate (Fig. 2 (b)). Obviously, no $\alpha''$ martensite was detected in the as-cast Cr-containing alloy.

Fig. 3 shows TEM micrographs of the as-cast In- and Ag-containing alloys as well as the corresponding selected-area diffraction (SAD) pattern. Fig. 3 (a) is a bright-field image revealing $\alpha''$ martensitic plates in the as-cast In-containing alloy. The SAD pattern for Fig. 3 (a) indicated the presence of both $\beta$ and $\alpha''$ phases (Fig. 3 (b)). The orientation relationship between the $\beta$ and $\alpha''$ phases was identified as $\{\overline{T15}\}\beta//\{\overline{T1}\}\alpha''$. In addition, although no $\omega$ phase was detectable from the XRD patterns, the athermal $\omega$ phase ($\omega_{\alpha''}$) can be identified from the SAD pattern due to the presence of faint peaks surrounding the peaks of the $\alpha''$ phase [14, 17-19], as indicated by the white arrows in Fig. 3 (b). For the as-cast Ag-containing alloy, bright-field images (Fig. 3 (c)) revealed that the $\beta$ phase was fully composed of fine twins with a spacing of 150~200 nm. Ultrafine and strained $\beta$ domains of 80~150 nm (marked by the white arrow in Fig. 3 (c)) were formed in the as-cast Ag-containing alloy, which is probably induced by the interaction of twins.

3.2. Deformation behavior of Ti-Nb-Ta-M $\beta$ alloys

It is obvious that the In- and Ag-containing $\beta$ alloys have lower stability against $\alpha''$ martensitic transformation upon solidification in comparison with the Cr-containing alloy. The deformation behavior was found to strongly depend on the stability of the $\beta$ phase. Fig. 4 shows the room-temperature compressive true stress-strain curves for the Ti-Nb-Ta-M $\beta$ alloys. All alloys displayed a similar elastic strain limit of about 0.8~1.0 % (the inset of Fig. 4). The In- and Ag-containing alloys exhibited a similar deformation behavior with low yield strength (373 and 414 MPa, respectively) and remarkable strain hardening. On the other hand, the Cr-containing alloy showed a different deformation behavior with higher yield strength of 612 MPa and less pronounced strain hardening. As a result, the In- and Ag-containing alloys have lower Young’s moduli of 55 and 60 GPa,
respectively, in comparison with 75 GPa for the Cr-containing alloy.

3.3. Microstructural evolution during deformation

In the uniaxially compressed sample, the degree of deformation is inhomogeneous and increases from the sample surface towards the center. Depending on degree of deformation, the microstructure can be classified into three types occurring in different zones throughout the specimen, including macroscopically deformed β dendrites, partially deformed and elongated β dendrites, and severely deformed β phase in the form of filaments, which are presented in the following sections in terms of phase stability of β alloys.

3.3.1. Deformation microstructures in the less stabilized In- and Ag-containing β alloys

Fig. 5 shows the SEM micrographs illustrating the typical deformation microstructures corresponding to the three deformed zones in the less stabilized In- and Ag-containing alloys. Close to the sample surface of the deformed In-containing alloy, deformation twins formed and an increase of the volume fraction of α" martensite was observed in comparison with the as-cast sample (Fig. 5 (a)). Some of the β dendrites fully transformed into α" martensite to accommodate the applied strain. Similar to the In-containing alloy, the Ag-containing alloy also exhibited deformation twinning at the less-deformed zone (Fig. 5 (b)). However, α" martensite was visible only at a higher magnification in certain areas (the inset of Fig. 5 (b)) and its volume fraction was much lower than in the In-containing alloy. This indicates that the Ag-containing alloy has a relatively higher stability than the In-containing alloy. With increasing degree of deformation, the equiaxed β dendrites were partially deformed and elongated along the direction perpendicular to the loading axis (Fig. 5 (c)). Although deformation twins and α" martensite were still visible, shear banding started to occur and partially contributed to the strain accommodation, as indicated by the arrow in Fig. 5 (c). With further increasing degree of deformation at the sample center, the β dendrites were severely deformed into filaments (Fig. 5 (d)). At this stage, deformation twins and α" martensite could not be easily identified due to the filamentary structure. However, remarkable shear banding was revealed, which mainly contributed to the strain accommodation.

In order to gain more insight into microstructural changes during deformation, detailed TEM investigations were carried out on the deformed In- and Ag-containing samples, which are shown in Figs. 6 and 7. Fig. 6 exhibits bright-field TEM images and the SAD patterns taken at the less deformed zone of the In-containing alloy. In Fig. 6 (a), the bright plate-like phase with a lamellar spacing of 150 nm was identified as α" martensite whereas the dark phase in between was the β parent phase. The SAD pattern taken along the [113]_β zone axis (the inset of Fig. 6 (a)) confirmed the presence of both β and α" phases with [113]_β∥[002]_α" orientation relationship [7]. In addition, from the SAD pattern a {112}<111> twinned structure was identified within the β phase. Furthermore, the HRTEM examination on the β phase (Fig. 6 (b) for the dashed-circle region in Fig. 6 (a)) and the corresponding SAD pattern along the [112]_β zone axis (inset of Fig. 6 (b)) implied that the athermal ω phase was present inside the parent β phase, as revealed from the broadening of the diffraction spots as well as the formation of diffuse streaks. Due to its athermal characteristics, the ω phase has no clear boundary with
the β phase [20], but it can be inferred from the stained contrast in the HRTEM micrograph.

Fig. 7 exhibits bright- and dark-field TEM micrographs and the SAD patterns taken at the severely deformed zone close to the sample centers of the In- and Ag-containing alloys. Fig. 7 (a) shows a bright-field image of the filamentary structure formed at the severely deformed zone in the In-containing alloy, in which the filamentary boundaries are indicated by the arrows. From the SAD pattern (not shown here), no α” martensite and athermal α phase can be identified other than the β phase for the filamentary structure. Fine deformation twins with a twin boundary spacing of less than 100 nm were found to form inside some of the β filaments. The deformation twins formed in each filament were confined by the filamentary boundary, showing a clear orientation relationship for twins inside two neighboring filaments. In addition to the formation of fine deformation twins, a pronounced grain refinement was observed in some of the β filaments to produce nanostructured β grains in the range of 50–100 nm, as evidenced from the bright- and dark-field images (Figs. 7 (b) and (c), respectively), and the SAD ring pattern (the right upper inset of Fig. 7 (b)). The nanostructured β grains exhibited a complicated moiré fringe pattern (the lower left inset of Fig. 7 (b)), which is similar to that of a nanostructured NiTi alloy processed by severe plastic deformation (SPD) [21]. Fig. 7 (d) shows the filamentary structure formed at the severely deformed zone in the Ag-containing alloy. In contrast to the In-containing alloy (Fig. 7 (a)), no fine deformation twins were visible inside the filaments. In addition, a remarkable grain refinement (50–100 nm) also occurred in some of the β grains at the severely deformed zone of the Ag-containing alloy, as indicated from the bright- and dark-field images (Figs. 7 (e) and (f), respectively) and the SAD pattern (the inset of Fig. 7 (e)). Upon severe deformation, the extent of grain refinement in the Ag-containing alloy was less pronounced than for the In-containing alloy, as evidenced from the SAD pattern composed of rings and diffraction spots. The grain refinement in the In-containing alloy led to the formation of nearly equiaxed nanoscale β grains (Fig. 7 (c)), whereas insufficient grain refinement in the Ag-containing alloy resulted in the coexistence of both plate-like (marked by the white arrow in Fig. 7 (f)) and nearly
equiaxed β grains, as indicated in Figs. 7 (e) and (f). Furthermore, it should be noted that the boundaries of the refined β grains were remarkably distorted, especially for the Ag-containing alloy.

3.3.2. Deformation microstructures in the stabilized Cr-containing β alloy

Compared with the less stabilized In- and Ag-containing alloys, the relatively stabilized Cr-containing alloy exhibited different deformation microstructures (Fig. 8). At the less deformed zone, no α″ martensite and deformation twins were detectable. Instead, slip bands (marked by the black arrow in Fig. 8 (a)) which could not be easily identified in the In- and Ag-containing alloys, formed in some β grains to accommodate the further imposed strain. With increasing degree of deformation, the lack of martensitic transformation and deformation twinning made plastic deformation of the β dendrites more difficult. Slip is not capable to accommodate the increasing strain, leading to the occurrence of strain localization and the appearance of shear bands even in the β dendrites without significant shape changes (Fig. 8 (b)). With further increasing degree of deformation, a large number of shear bands formed in the severely deformed zones, cutting off the filamentary structure in order to release the increasing strain (Fig. 8 (c)).

Fig. 9 shows bright-field TEM micrographs, and the corresponding SAD patterns at the severely deformed zone of the Cr-containing alloy. The filamentary structure formed in the Cr-containing alloy is illustrated in Fig. 9 (a). The SAD pattern (inset of Fig. 9 (a)) indicated the presence of both β and athermal α phases [14, 17-20], in which main diffraction spots and weak diffusing streaks (indicated by the black arrow) correspond to β and α phases, respectively. No nanostructuring of the β phase was found for the Cr-containing alloy after severe deformation. Instead, in some of the filaments both submicron and nanoscale deformation twins were clearly visible, as revealed by the bright-field TEM image shown in Fig. 9 (b). The presence of deformation twins can be deduced from the higher order diffraction spots (marked by the white arrows in the inset of Fig. 9 (b)). Fig. 9 (c) shows a high-resolution TEM image of the nanoscale twins (the dashed-circle region in Fig. 9 (b)). Twin faults...
(marked by white arrows in Fig. 9 (c)) were present at the twin boundaries. Compared with the stacking faults formed in the In-containing alloy, the twin faults formed in the Cr-containing alloy exhibited a smaller width [8].

4. DISCUSSION

4.1. Microstructural evolution during deformation and its correlation with the deformation behavior of β-titanium alloys

The control of β phase stability is important for designing metastable β-titanium alloys since it strongly affects the phases formed upon quenching or deformation. In metastable β-titanium alloys with a large amount of β-stabilizers, the phase transformations usually involve β→α” and/or β→α martensitic transformations, which to a great extent determine the resultant deformation behavior. The examination of the microstructural evolution is, therefore, an effective way to establish a correlation between phase stability and deformation behavior. In the present study, the three metastable β-titanium alloys show different phase stability. Table 2 summarizes the phases formed in both the as-cast and deformed states for the Ti-Nb-Ta-M β alloys. Among these alloys, the In-containing alloy has the lowest stability, leading to the formation of both α” martensite and athermal α phase upon quenching. On the other hand, the Cr-containing alloy has the highest stability, resulting in the formation of only athermal α phase.

As indicated from the compressive true stress-strain curves (Fig. 4), the β-titanium alloys with low stability exhibit lower yield strength, remarkable strain hardening and higher ductility in comparison with the stable β-titanium alloys. For the convenience of establishing the correlation between microstructural evolution and deformation behavior, it is reasonable to divide the true stress-strain curves into three stages corresponding to elastic deformation, strain hardening and easy glide. It is noted that not only one unique deformation mechanism is operative to result in such three-stage deformation curves exhibiting an enhanced ductility. The two stages related to plastic deformation are discussed as follows.

Depending on the phase stability, martensitic transformation, deformation twinning and slip are viewed as the main deformation mechanisms for metastable β-titanium alloys [13, 15, 16]. For the In- and Ag-containing alloys with lower β phase stability, a stress-induced β+αm→α” martensitic transformation is easily triggered after the initial linear elastic deformation rather than slip [14], leading to low yield strength. Based on the preexistent martensitic plates formed upon quenching, new α” martensite nucleates and freely grows into the β dendrites to release the increasing deformation strain. As reported in a previous study [7], the stability increases from the interdendritic zone to the center of the β dendrites due to solute redistribution. When deformation proceeds, nucleation of additional α” martensitic plates becomes more difficult towards the central region of the partially consumed β dendrites. As a result, only some but not all of the β dendrites can fully transform into α” martensite. In the meantime, deformation twinning {112}<111> is also operative to accommodate the increasing strain. However, owing to their characteristic, martensitic transformation and deformation twinning cannot result in a large shape change of the dendrites. With increasing degree of deformation, it is believed that slip is activated in the unconsumed dendrites since it is difficult to further produce more α” martensite and deformation twins. Subsequently, slip dislocations may interact with α” martensite, remaining athermal α phase and deformation twins, thus influencing the deformation behavior to a great extent. Especially, the orthorhombic α” martensitic phase with less independent slip systems [22] plays a role as a barrier for slip of dislocations, resulting in dislocation pile-ups that might be responsible for the more pronounced strain hardening observed for the In- and Ag-containing alloys. With further increasing deformation above a certain strain level, the pronounced strain hardening proceeds until the occurrence of the reverse α”→β transformation. This reverse transformation may happen if the temperature rises

<table>
<thead>
<tr>
<th>Alloy</th>
<th>As-cast</th>
<th>Less deformed</th>
<th>Severely deformed</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti-Nb-Ta-In</td>
<td>β+α”+αm, twinning, increase in volume fraction of α”</td>
<td>α”+αm, twinning, Pronounced grain refinement to nanoscale</td>
<td></td>
</tr>
<tr>
<td>Ti-Nb-Ta-Ag</td>
<td>β+α”+αm, twinning, increase in volume fraction of α”</td>
<td>β+αm, twinning, Pronounced twinning, No visible grain refinement to nanoscale</td>
<td></td>
</tr>
<tr>
<td>Ti-Nb-Ta-Cr</td>
<td>β+αm</td>
<td></td>
<td></td>
</tr>
</tbody>
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locally and exceeds the austenitic start temperature $A_s$ [14] as a result of plastic heat generation upon deformation [23]. This might be the reason that the $\alpha''$ martensitic phase could not be identified at the severely deformed zones. Most important, the structure change from orthorhombic to bcc removes the barrier for slip, rendering some part of the piled-up dislocations to become mobile. Subsequently, the stage of easy glide with little strain hardening occurs, during which the dislocations are able to move over relatively large distances without encountering barriers (but the motion of dislocations is confined within the $\beta$ filaments). This might be one of the contributions to the ductility enhancement. In addition, the occurrence of nanostructuring upon severe plastic deformation might further contribute to the ductility, which will be discussed in Section 4.2.

For the Cr-containing alloy with high $\beta$ phase stability, no $\alpha''$ martensitic transformation and deformation twinning following the initial linear elastic regime could be identified, indicating that the operative deformation mechanism is only slip. As a result, the Cr-containing alloy exhibits high yield strength since the trigger stress for slip is much higher than that inducing martensitic transformation [13]. With increasing degree of deformation, the only barrier encountered by slip of dislocations is the athermal $\alpha$ phase, which results in a limited strain hardening in comparison with the In- and Ag-containing alloys. During this stage of plastic deformation, dense dislocation walls and dislocation tangles begin to develop, making further motion of dislocations difficult. Fine deformation twinning is becoming the favorable deformation mode for further strain accommodation. The formation of a large number of nanoscale twins with a width of less than 10 nm (Fig. 9 (b)), is believed to contribute to the ductility to a certain extent, since the lattice orientation changes by twinning may enable additional slip to occur [24].

### 4.2. Deformation-induced nanostructuring in $\beta$-titanium alloys

Deformation-induced grain refinement to the nanoscale usually occurs when an externally repeated force is imposed on bulk polycrystalline metallic materials or their surfaces such through as ball milling [25], severe plastic deformation [26] and surface mechanical attrition treatment [27]. The lattice structure and the stacking fault energy of alloys significantly influence the plastic deformation in metallic materials, which inherently govern the deformation-induced grain refinement [27, 28]. Recently, deformation-induced grain refinement mechanisms were investigated in terms of stacking fault energy for several typical metallic materials [27]. It was found that deformation twinning and martensitic transformation play a crucial role for grain refinement in alloys with low stacking fault energy [27]. For $\beta$-titanium alloys, a remarkable grain refinement to the nanoscale (less than 50 nm) was achieved in a cold-rolled Ti-24Nb-4Zr-7.9Sn (wt.%) $\beta$ alloy after 90% reduction in thickness [6]. In addition, a less pronounced grain refinement was reported for a Ti-24Nb-4Zr-7.6Sn (wt.%) alloy favoring martensitic transformation [6]. However, our recent investigation on Ti-Nb-Ta-(In, Cr) $\beta$ alloys implied that upon severe plastic deformation nanostructuring occurs more easily in the less-stabilized $\beta$-titanium alloy with low stacking fault energy, especially at martensitic transformed zones [8]. According to Wang et al. [29], grain refinement can be more easily promoted if a complex stress state and complicated straining patterns are present. Based on the deformation behavior (Fig. 4) and the phase formation/microstructural evolution (Figs. 5-9 and Table 2), a plausible grain refinement mechanism can be proposed for the In- and Ag-containing alloys. Easy nanostructuring might stem from the complex interaction among slip dislocations, martensite and deformation twins, which is qualitatively elucidated as follows.

As indicated from Fig. 1 and Table 2, prior to deformation $\alpha''$ martensitic plates are already present at the interdendritic zones in the as-cast In- and Ag-containing alloys, resulting in an inhomogeneous microstructure, i.e. martensitic transformed and untransformed zones. In the martensitic transformed zones, the martensitic plates have at a spacing of 150 nm, dividing the $\beta$ phase into a sandwiched structure with a length-scale as fine as 100 nm. Upon compressive loading above a trigger stress, stress-assisted $\beta + \alpha'' \rightarrow \alpha''$ martensitic transformation [14] and mechanical twinning are mainly operative to accommodate the deformation strain at the initial stage of plastic deformation [7]. The formation of such a fine martensite can be viewed as a crucial step for the subsequent easy nanostructuring. With increasing degree of deformation, instead of martensitic transformation and mechanical twinning, slip becomes dominant and mainly contributes to accommodate the increasing strain. At this stage the interaction among slip dislocations, pre-existing $\alpha''$ martensite and twins is quite pronounced [8]. The low-symmetry orthorhombic $\alpha''$ martensite lacks sufficient independent slip systems [22], making slip dislocations difficult to pass through it. Instead, slip dislocations strongly interact with the partial dislocations of the $\beta/\alpha''$ interface and pile-up at the interface to subdivide the sandwiched $\beta$ phase into refined blocks, establishing the prerequisite for subsequent nanostructuring. The difficulty for the motion of slip dislocations is believed to remarkably contribute to the strain hardening. With further increasing deformation above a certain strain level (severely plastically deformed), the temperature increase locally exceeds the austenitic start temperature (as low as 166 °C for Ti-10V-2Fe-3Al [14]), which might lead to the reverse $\alpha'' \rightarrow \beta$ transformation [14] at some localized zones. The structure change from orthorhombic back to bcc provides more slip systems, activating part of the piled-up dislocations at the $\beta/\alpha''$ interface. These reactivated dislocations experience an easy glide stage.
within a β filament and pile-up again at the filametary boundary, forming subboundaries and further subdividing the β filaments into refined blocks. The refining process will proceed within the filaments with increasing degree of deformation. Therefore, upon severe deformation easy nanostructuring occurs rather at the martensitic transformed zones than at the untransformed zones, leading to an inhomogeneous microstructure, i.e. nanoscale β grains (50–100 nm) together with a submicron β filametary structure (200–800 nm). It is believed that to a great extent the nanostructuring process and the inhomogeneous microstructure are beneficial to the enhancement of the ductility. Grain refinement to nanoscale grains creates more free volume/defects (grain or sub-grain boundaries) that significantly contribute to the ductility. On the other hand, the inhomogeneous microstructure consisting of nanoscale equiaxed grains and submicron β filaments is conceptually similar to the bimodal microstructure observed in a ductile nanostructured Cu “composite” [29], which is believed to greatly contribute to ductility as deformation proceeds.

In contrary, upon the applied compressive loading easy nanostructuring was not observed in the Cr-containing alloy with high stacking fault energy although a very fine filamentary structure was formed at the severely deformed zone [8]. However, the formation of submicron and nanoscale deformation twins above a certain strain level implies that it is feasible to utilize severe plastic deformation techniques to achieve a grain refinement to nanoscale in β-titanium alloys with high stacking fault energy, which is currently under investigation.

5. CONCLUSIONS

The microscopic deformation behavior and the microstructural evolution have been identified for metastable Ti-Nb-Ta-M (M=In, Ag, or Cr) β alloys with different phase stability against β→α” martensitic transformation. Among these alloys, the In-containing alloy exhibits the lowest stability, leading to the formation of both α” martensite and athermal α phase upon quenching. On the other hand, the Cr-containing alloy shows the highest stability, resulting in the formation of only athermal α phase. With increasing degree of deformation, martensitic transformation and deformation twinning are initially operative followed by slip of dislocations for the In- and Ag-containing alloys, whereas slip is initially operative followed by fine deformation twinning for the Cr-containing alloy. Especially, a pronounced grain refinement of β grains to the nanoscale (50–100 nm) easily occurs in the In- and Ag-containing alloys with low stacking fault energy, which is attributed to the reversible β→α” martensitic transformation and the complicated interactions among slip dislocations, α” martensite and deformation twins.

Based on the present results, a preliminary correlation has been established between microscopic deformation mechanism, microstructural evolution and deformation behavior. For the In- and Ag-containing alloys, the orthorhombic α” martensitic phase with less independent slip systems plays a role as a barrier for slip of dislocations, resulting in dislocation pile-ups that might be responsible for the more pronounced strain hardening. In terms of ductility enhancement, the reverse transformation from orthorhombic α” martensite back to the bcc β phase removes the barrier for slip, making part of the piled-up dislocations become mobile for easy glide and subsequently contributing to ductility. In addition, the nanostructuring process and the formation of an inhomogeneous deformation microstructure are beneficial for ductility enhancement due to the creation of more free volume/defects and the composite-like bimodal microstructure, respectively. On the other hand, for the Cr-containing alloy the lack of interactions among slip dislocations, α” martensite and deformation twins produces less barriers for slip, leads to limited strain hardening and makes grain refinement more difficult.

ACKNOWLEDGEMENTS

The authors thank U. Kühn, W. Löser, S. Pauly and B. Rellinghaus and for valuable discussions, and M. Branzei, M. Frey and H.-J. Klaub for technical assistance. Funding from the EU within the framework of the RTN-network on “Ductile BMG composites” (MRTN-CT-2003-504692), the Australian Research Council with the ARC Centre of Excellence for Design in Light Metals, the Alexander-von-Humboldt Foundation, and the German Science Foundation is also acknowledged.

Globally and Locally Strained Si Nanomembranes Fabricated by Elastic Strain Sharing

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Technology has evolved towards miniaturization of device features with the ultimate goal of integrating many components onto a compact chip, producing integrated circuits with low cost and high degrees of functionality. A recently developed approach is based on the use of nm-thick membranes as an alternative to bulk substrates in various applications. These membranes can be completely freestanding or tethered to a substrate, and they can be flat or shaped in three-dimensional (3D) structures.

Nanomembranes (NMs) are distinguished from bulk materials most significantly by thinness, flexibility, and nearness of two surfaces or interfaces. Excellent structural, electronic, and optical properties have been measured, both for flat and curled films. Furthermore, the ability to transfer released NMs to a large variety of hosts has been successfully used for the fabrication of hybrid or highly mismatched single-crystal multilayer stacks, and for the development of bendable and stretchable electronics.

In recent work we have taken advantage of the mechanical compliance of freestanding NMs to establish a uniform or spatially varying strain field in the ultra-thin layer. Elastic strain sharing between a SiGe and a Si film completely unsupported by a solid allows the fabrication of tensilely strained SiNMs. Similarly, a spatially varying strain field has been established in Si nanoribbons by growing "local stressors" (e.g., Ge quantum dots (QDs)) rather than a "uniform stressor" (e.g., a SiGe strained layer). Strain engineering in SiNMs in turn offers the possibility of tuning materials properties, such as band gap and carrier mobility, both on a global and a local scale.

Among the most relevant results achieved in our group in the field of NM technology are: 1) the fabrication of dislocation-free (110) Si as thin as 10 nm, isotropically and biaxially strained with an in-plane strain as high as 0.6%; 2) the fabrication of hybrid-orientation templates alternating (110) and (001) Si regions; and 3) the fabrication of a mechno-electronic superlattice in a single material, namely a SiNM. We briefly describe these three topics.

We have used elastic strain sharing in Si/SiGe/Si multilayers to fabricate dislocation-free, tensilely strained (110) oriented SiNMs, which has indeed emerged as an attractive material because of its higher hole mobility compared to (001) Si. Good crystal quality of the layers is revealed by X-ray diffraction (XRD). Surface morphology measurements, performed by atomic force microscopy (AFM), confirms the absence of any mechanism of plastic relaxation, i.e., nucleation of line and plane defects, and roughening. The first step is the growth of a compressively strained SiGe layer (below its kinetic critical thickness for plastic relaxation) on the Si template layer of a (001) or (110) silicon-on-insulator (SOI) substrate, followed by a top Si capping layer. The SiGe alloy and top Si layers were pseudomorphically grown by chemical vapor deposition (CVD) in a cold-wall ultra-high vacuum (UHV) CVD reactor or by solid-source molecular beam epitaxy (MBE). In the as-grown trilayer the SiGe is compressively strained and under tetragonal distortion, whereas the Si is unstrained, at the substrate lattice constant. Membrane formation is then achieved by removal of the underlying SiO₂, i.e., the buried oxide (BOX). The release of the membrane from the SiO₂ results in its overall lateral expansion driven from the elastic strain relaxation of the SiGe layer. During this process the compressive strain in the SiGe membrane partially transfers to tensile strain in the Si layers. Equal or nearly equal thicknesses of top and bottom Si layers are mandated to produce flat strain-balanced SiNMs after the release step.

Membranes were then harvested using a wet-transfer technique that has so far been used to transfer membranes in sizes ranging from 5x5 mm² to 1x1 cm² to a variety of substrates. The procedure is scalable to both significantly larger and smaller dimensions and to almost any host substrate that is not rapidly dissolved in water. A dry printing technique for transferring the membranes to any kind of new host has also been used successfully. Similar methods work for Ge NMs or those of other materials.

Comparison of XRD 0–2θ line scans on as-grown and released trilayers with various thicknesses and compositions demonstrate that tensile strain in (110) SiNMs can be tuned by changing the Ge content in the alloy layer and the alloy layer thickness relative to the thickness of the Si layers in the trilayer. A maximum tensile strain of 0.65±0.01% in (110) SiNMs is calculated from XRD scans and confirmed by continuum elasticity theory.

The transfer of (110) SiNMs (even unstrained) to a (001) oriented Si template and subsequent overgrowth has been used to fabricate mixed-crystal-orientation templates. This architecture may allow the manufacturing, in close proximity to each other, of p- and n-channel devices on (110) Si (high hole mobility) and (001) Si (high electron mobility) regions, respectively, with the benefit of reducing the current drive imbalance between PMOS and NMOS devices. The ability to transfer the membranes to new hosts opens the possibility for integrating high-electron mobility (001) regions with dislocation-free...
strained (110) Si, which promises hole mobility enhancements of up to 75% compared to the (001) universal mobility. Furthermore, rotating the strained (110) membrane relative to its (001) host during transfer offers a concrete possibility of optimizing channel direction in n- and p-type devices. We have fabricated mixed-crystal-orientation material in flexible form using SiNM transfer and overgrowth. The SiNM fabrication begins with a (110) SOI substrate. The (110) Si template layer is patterned with an array of holes using standard photolithography and RIE, and removed from its handle substrate via selective etching of the BOX layer. This procedure creates a temporarily freestanding (110) SiNM, which is then bonded to a (001) Si substrate. The holes define the regions where the (001) Si crystal planes are exposed through the membrane. Deposition of Si over the structure using CVD with SiH$_4$ precursor gas, at a substrate temperature of 580°C, fills the holes because the Si growth rate is about 10 times faster on the (001) orientation than on the (110) orientation at 580°C. Hence fabrication of a flat mesh of (001) Si and (110) Si regions is achieved.

As the third example, strain sharing between Ge nanostressor islands (quantum dots, QDs) and a compliant SiNM can be used to create very small regions of high local strain in the membrane. As a result the SiNM undergoes distortion, forming into a slightly wavy sheet. The local strain provides a feedback for self-organization of the QDs. We have reported ordered growth of nanostressors on freestanding ribbons with a 50 to 250 nm width, fabricated from SOI with a 5 to 50 nm-thick Si templates. Ge or a Ge-rich SiGe alloy is deposited simultaneously on both sides of these free-standing nanoribbons using CVD. Because CVD involves the vapor phase transport of precursor molecules, deposition is possible at all locations accessible to the growth gases. The feedback mechanism leads to a spatially periodic arrangement of pyramid shaped QDs, alternating on the top and on the bottom surface of the SiNMs. Consequently a periodically varying strain is established along a thin single-crystal Si ribbon. The strain lattice in the Si membrane in turn produces a modulation in the electronic band structure that extends through the thickness of the membrane, and thus creates an electronic superlattice without a need for compositional modulation, the conventional method for forming electronic heterostructures.

We calculate the periodic change induced in the Si band gap by the strain field as a function of position along the ribbon. For this purpose the local strain was modelled with a 2D finite-element analysis of the elastic deformation and elastic energy resulting from two opposite-side QDs in one dimension corresponding to the ribbon geometry with one line of dots on each side. Our calculations predict that the maximum tensile strain beneath a Ge QD with a height of 8 nm and a base width of 80 nm, epitaxially grown on a Si membrane, is ~1.5% for a 10 nm thick ribbon. The resulting reduction of the band gap can be up to 250 meV, more than 20% of the bulk value of the band gap for the thinnest ribbons. The calculated band gap modulation was confirmed qualitatively by combining photoelectron emission microscopy (PEEM) (as the detection tool) with high-resolution X-ray absorption spectroscopy (XAS). The calculated bandgap modulation in the SiNM due to the growth of nanostressors is similar to the one observed in a 1D heterostructure with essentially all the band offset occurring in the conduction band. We solved the variable effective-mass Schrödinger equation to obtain the miniband structure in a 1D periodic potential. The results show that minibands with very small separations (i.e., minigaps) form within the potential well. However, the widths of minigaps range from 2 up to 8 meV, which is much smaller than the thermal energy kT at 300 K (26 meV). Therefore these minibands can be essentially considered as continuous states at room temperature (in the classical limit). The Ge/Si nanoribbon structure has to be cooled to below 77 K to minimize the thermal smearing sufficiently to make discrete minibands observable.

We predict that it is possible to observe discrete minibands in the potential well of the 1D strain superlattice at room temperature if one uses InAs nanostressors QDs, which form on Si with strain as high as 11.5%. We calculate that the maximum tensile strain beneath a pure-InAs pyramidal QD with a height of 6 nm and a base width of 11 nm pseudomorphically grown on a Si membrane is ~2.7% for a 10 nm thick ribbon. The resulting depression of the Si band gap is expected to be up to 350 meV, more than 30% of the bulk value of the band gap for the thinnest ribbons. We predicted the formation of well-separated minibands in the potential well already for a 25 nm thick membrane (potential well depth 200 meV), with minigaps ranging from 15 to 85 meV, and with only the first minigap being smaller than the thermal energy of ~26 meV at 300 K. It should therefore be possible to observe discrete minibands in the potential near room temperature.

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Research supported by USDOE, USAFOSR, and USNSF/MRSEC
Synthesis of Advanced Materials by Mechanical Alloying

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Mechanical alloying (MA) is a powder metallurgy processing technique that involves repeated cold welding, fracturing and rewelding of powder particles in a high-energy ball mill. Started in 1960 to produce oxide dispersion strengthened (ODS) nickel-based superalloys to obtain high strength both at room and elevated temperatures, this technique is now widely used to synthesize a variety of non-equilibrium alloys including solid solutions, intermetallics, and glassy alloys. Apart from the ODS alloys, the products of MA are now finding applications in newer and innovative areas. The present article briefly summarizes the recent contributions of the author and his colleagues and collaborators in the field of MA to synthesize commercially useful and advanced materials. Four specific topics have been covered – nanocomposites, Fe-based amorphous alloys, photovoltaic materials, and use of MA powder particles in metal combustion. The article concludes with an indication of the topics that need special attention in the near future.

I. INTRODUCTION

Advanced materials are defined as those where first consideration is given to the systematic synthesis and control of the crystal structure and microstructure of materials to provide precisely tailored properties for demanding applications. With the rapid advances taking place in the technology sectors, there has been a great demand for materials that are stronger, stiffer, lighter, useful at higher temperatures, and less expensive than the existing and available materials. Thus, the high-tech industries have given fillip to efforts in developing novel materials that will perform better for demanding applications. Some of the advanced materials developed during the last few decades include metallic glasses, quasicrystals, superconductors, superhard carbo-nitrides, thin-film diamond synthesis, and nanostructured materials. A number of new techniques for processing and characterization of materials have also been developed towards achieving this goal. But, a common underlying principle in obtaining advanced materials has been to process the materials under far-from-equilibrium conditions, i.e., the materials developed are mostly in a metastable condition. Turnbull has coined the phrase “energize and quench” to describe these processes. It is through such processes that the constitution and microstructure of the materials can be drastically altered leading to achieving the desired properties. Some of these “non-equilibrium processing” techniques include rapid solidification from the melt, mechanical alloying, laser processing, plasma processing, spray forming, physical and chemical vapor deposition techniques, ion mixing, among others. The relative advantages and disadvantages of these techniques have been discussed in a recent publication. In the following paragraphs, we will describe some of the advanced materials that have been recently developed using the technique of mechanical alloying.

II. THE PROCESS OF MECHANICAL ALLOYING

Mechanical Alloying (MA) is a process that was developed by John Benjamin of INCO International in the late 1960’s to produce oxide dispersion-strengthened materials. The process can be described as a high-energy milling process in which powder particles are subjected to repeated cold welding, fracturing, and rewelding. The generally accepted explanation for alloying to occur from blended elemental powders and formation of different types of phases is that a very fine and intimate mixture (often lamellar if the constituent elements are sufficiently ductile) of the components is formed after milling, if not the final product. The crystalline defects introduced into the material (grain boundaries, interfaces, dislocations, stacking faults, vacancies, and others), which act as fast diffusion paths, and a slight rise in the powder temperature during milling as a result of frictional forces and impact of the grinding balls against other balls and the surfaces of the container, were found to facilitate alloy formation. If the final desired phase had not formed directly by MA, then a short annealing treatment at an elevated temperature was found to promote diffusion and consequently alloy formation. Accordingly, it was shown that by a proper choice of the process parameters during MA and choosing an appropriate alloy composition, it is possible to produce solid solutions (both equilibrium and supersaturated), intermediate phases (crystalline and quasicrystalline) and amorphous alloys. The thermodynamic criterion that the phase with the lowest free energy, under the continuous deformation conditions, would be the most stable phase has been found to be valid. A significant attribute of MA has been the ability to even alloy metals with positive heats of mixing that are difficult to alloy otherwise.

The effects of MA could be categorized under two groups:
1. Constitutional changes: Milling of the blended elemental powders could lead to the formation of solid solutions (both equilibrium and supersaturated), intermetallic phases (equilibrium, metastable and quasicrystalline), and amorphous phases in alloy systems. The actual type of phase formed could be different depending on the alloy system and the milling conditions employed.

2. Microstructural changes: As a result of MA, the processed materials could develop ultrafine-grained and nanostructured phases. In addition, uniform dispersion of a large volume fraction of very fine oxide or other ceramic particles can be achieved in different matrices; not possible or easy by other processes.

In many cases, chemical reactions have also been found to take place leading to the formation of novel materials and microstructures. Such a process has been referred to as mechanochemical processing. All these effects have been well documented in the literature. As has been frequently pointed out in the literature, the technique of MA was developed out of an industrial necessity in 1966 to produce nickel-based superalloys that combined the room temperature strength obtained by precipitation hardening with the high-temperature strength achieved through dispersion hardening. The historical development of MA during the last 40 years or so can be divided into three major periods. The first period, covering from 1966 to 1985, was mostly concerned with the development and production of oxide dispersion strengthened (ODS) superalloys for applications in the aerospace industry. Several alloys, with improved properties, based on Ni and Fe were developed that found useful applications. These included the MA754, MA760, MA956, MA957, MA6000, and others. The second period, covering from 1986 up to about 2000, witnessed lot of advances in the fundamental understanding of the processes that take place during MA. Along with this, a large number of dedicated conferences were held and there was a burgeoning of publication activity. Comparisons have also been frequently made between MA and other non-equilibrium processing techniques, notably rapid solidification processing (RSP). It was shown that the metastable effects achieved by these two non-equilibrium processing techniques are similar. Revival of the mechanochemical processing (MCP) took place and a variety of novel substances were synthesized. Several modeling studies were also conducted to enable prediction of the phases produced or microstructures obtained, although with limited success. The third period, starting from about 2001 saw a reemergence of the quest for new applications of the MA materials, not only as structural materials, but also for chemical applications such as catalysis and hydrogen storage, with the realization that contamination of the milled powders is the limiting factor in the widespread applications of the MA materials. Innovative techniques to consolidate the MA powders to full density while retaining the metastable phases (including glassy phases and/or nanostructures) in them were also developed. All these are continuing with the ongoing investigations to enhance the scientific understanding of the MA process.

As mentioned earlier, in recent years, there has been a resurgence of search for novel applications of MA products. Apart from this, investigations are also continuing on developing new and “better” materials through MA and also to understand the science behind the formation of these novel materials. In this paper, we will summarize our recent activities in the general area of MA in developing advanced materials. As typical examples, we would like to discuss four specific topics:

1. Development of homogeneously-dispersed nano-composites,
2. Formation of metallic glasses,
3. Synthesis of photo-voltaic materials, and
4. Use of energetic metal particles in combustion.

All the experiments were carried out either in a SPEX CertiPrep high-energy ball mill or a Fritsch P-5 planetary ball mill or an attritor under controlled conditions. Loading and unloading of the powders was carried out in a glove box filled with argon gas. Normally a ball-to-powder weight ratio of 10:1 was maintained. Characterization of the milled powders was conducted through X-ray diffraction, scanning electron microscopy, and transmission electron microscopy techniques and the results were analyzed using standard procedures.

III. NANO COMPOSITES

Achievement of a uniform distribution of the reinforcement in a matrix is essential to achieving good mechanical properties of the composites. Further, the mechanical properties of the composite tend to improve with increasing volume fraction and decreasing particle size of the reinforcement. Traditionally, a reasonably large volume fraction of the reinforcement could be added, if the size of the reinforcement is large (on a micrometer scale). But, if the reinforcement size is very fine (of nanometer dimensions), then the volume fraction added is restricted to about 2 to 4%. This is because the fine powders tend to float to the top of the melt during processing of the composites through solidification processing. However, if we are able to introduce a large volume fraction of nanometer-sized reinforcement, the mechanical properties of the composite are likely to be vastly improved. In our investigations, during the last few years, we have successfully achieved a very uniform distribution of the reinforcement phases in different types of matrices through the solid-state powder processing technique of MA. These include homogeneous dispersion of graphite in an Al-6061 alloy matrix, effect of clustering of the reinforcement on the mechanical properties of the composites, dispersion of a high volume fraction of Al2O3 in Al, and of TiSi3 in γ-TiAl, synthesis of MoSi2+Si3N4 composites for high-temperature applications, and synthesis of amorphous+intermetallic composites in Al-Mg alloys. However, in this chapter, we will describe only two typical examples.
III. 1. \textit{Al-Al}_2\textit{O}_3 Composites

Aluminum-based metal matrix composites are ideal materials for structural applications in the aircraft and automotive industries due to their lightweight and high strength-to-weight ratio. Reinforcement of the ductile aluminum matrix with stronger and stiffer second-phase reinforcements like oxides, carbides, borides, and nitrides provides a combination of properties of both the metallic matrix and the ceramic reinforcement. Uniform dispersion of the fine reinforcements and a fine grain size of the matrix contribute to improving the mechanical properties of the composite.

Most of the work carried out to-date on discontinuously reinforced aluminum was primarily concerned with dispersing reinforcements on the micrometer scale (a few to a couple of hundred micrometers), as represented by region \#1 in Fig. 1. Even though studies were carried out on nanometer-sized reinforcements, the volume fraction of the reinforcements was very small, typically about 2-4\% by volume, indicated by region \# 2 in Fig. 1. Thus, as shown by region \# 3 in Fig. 1, there is a gap in understanding the structure and properties of high volume fraction composites containing nanometer-sized reinforcements. Therefore, we have conducted investigations to synthesize and characterize \textit{Al-Al}_2\textit{O}_3 composites with the \textit{Al}_2\textit{O}_3 reinforcement varying in size from 50 nm to 5 \textmu m, and the volume fraction from 5 to 50\% by volume. The reasons behind the choice of this combination of particle sizes and volume fractions were to check (i) whether there is a maximum volume fraction of \textit{Al}_2\textit{O}_3 beyond which it will be difficult to achieve a uniform distribution and also (ii) to see if there is a minimum particle size, below which again it will be difficult to achieve a uniform distribution of the two components in the composite.

Table 1 lists the powder sizes and volume fractions of the \textit{Al}_2\textit{O}_3 reinforcements used in this study. Mechanical alloying of the powder blends containing different amounts and size fractions of \textit{Al}_2\textit{O}_3 was found to reach a stable and uniform distribution of the reinforcement. A milling time of 20 h was found to be sufficient in all the combinations of the composites. Figure 2 shows that a very uniform dispersion of the fine 50-nm \textit{Al}_2\textit{O}_3 particles was achieved even when the volume fraction of the reinforcement was 50\%. This corresponds to the finest particle size and largest volume fraction, the maximum value achieved so far in any nanocomposite. Similar uniform distributions were obtained in the larger size and smaller volume fractions of the reinforcement also. The uniform distribution of the reinforcement phase was also confirmed through the X-ray elemental mapping technique (Fig. 3). An interesting observation made during this study was that the stable \gamma-\textit{Al}_2\textit{O}_3 transformed to \alpha-\textit{Al}_2\textit{O}_3 on milling, when the \textit{Al}_2\textit{O}_3 powder particle size was small, e.g., 50 and 150 nm.\cite{29} However, no such transformation occurred when the \textit{Al}_2\textit{O}_3 particle size was large, e.g., 5 \textmu m.

These composites with a large volume fraction of the reinforcement of the \textit{Al}_2\textit{O}_3 powders were very hard and strong and consequently it was not easy to consolidate them to full density by any of the single different techniques that are presently available. Therefore, to determine the effect of reinforcement particle size and

![FIG. 1. Schematic representation of the relationship between interparticle spacing, \(\lambda\), particle size, \(d\), and volume fraction, \(f_v\), of the reinforcement phase. Note that at volume fractions greater than 0.125, \(\lambda\) becomes smaller than the particle size. Regions \#1, \#2, and \#3 correspond to reinforcements with large particle size and large volume fractions, small particle size and small volume fractions, and small particle size and large volume fractions, respectively.](image)

<table>
<thead>
<tr>
<th>\textit{Al}_2\textit{O}_3 content (vol.%)</th>
<th>Particle Size</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>50 nm</td>
</tr>
<tr>
<td>5</td>
<td>✓</td>
</tr>
<tr>
<td>10</td>
<td>✓</td>
</tr>
<tr>
<td>20</td>
<td>✓</td>
</tr>
<tr>
<td>30</td>
<td>✓</td>
</tr>
<tr>
<td>50</td>
<td>✓</td>
</tr>
</tbody>
</table>
volume fraction, nanocomposites with 50 nm and 150 nm particle sizes and 5 and 10 vol.% were consolidated to full density. Even at these small volume fractions, full (close to 100%) density was achieved only by a combination of vacuum hot pressing followed by hot isostatic pressing (Fig. 4). Compression testing was done on the fully dense samples, and the strength properties and modulus values were determined. Table II lists the mechanical properties of these composites. Figure 5 shows the variation of the modulus of elasticity as a function of the $\text{Al}_2\text{O}_3$ content. From this figure, it may be noted that the strength and modulus increased with (i) increasing volume fraction and (ii) decreasing size of the reinforcement. Comparison of these modulus values with iso-strain condition, while composites with the larger reinforcement size (150 nm) behaved closer to the iso-stress condition.\textsuperscript{30,31} This observation clearly suggests that it is possible to tailor the modulus (and strength) of the nanocomposite by choosing the appropriate reinforcement size and volume fraction. Such a processing methodology should be equally applicable to other systems, even though the details of processing and consolidation would be different.

### III. 2. $\gamma$-TiAl-$\gamma'$Ti$_3$Si$_3$ Composites

Lightweight intermetallic alloys based on $\gamma$-TiAl are promising materials for high-temperature structural applications, e.g., in aircraft engines or stationary turbines.\textsuperscript{32,33} Even though they have many desirable properties such as high specific strength and modulus both at room and elevated temperatures, and good corrosion and oxidation resistance, they suffer from
TABLE II. Mechanical properties of Al-Al₂O₃ nanocomposites obtained by milling and subsequent consolidation by vacuum hot pressing and hot isostatic pressing.

<table>
<thead>
<tr>
<th>Al₂O₃</th>
<th>Volume fraction</th>
<th>Yield strength (MPa)</th>
<th>Compressive strength (MPa)</th>
<th>Elastic Modulus (GPa)</th>
<th>Elastic Modulus calculated by the rule of mixtures* (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>50 nm</td>
<td>5%</td>
<td>488</td>
<td>605</td>
<td>78</td>
<td>83</td>
</tr>
<tr>
<td>50 nm</td>
<td>10%</td>
<td>515</td>
<td>628</td>
<td>90</td>
<td>95</td>
</tr>
<tr>
<td>150 nm</td>
<td>5%</td>
<td>409</td>
<td>544</td>
<td>75</td>
<td>83</td>
</tr>
<tr>
<td>150 nm</td>
<td>10%</td>
<td>461</td>
<td>600</td>
<td>77</td>
<td>95</td>
</tr>
</tbody>
</table>

*Eᵥ = VᵥEm + VₑEr, where E and V represent the elastic modulus and volume fraction, respectively and the subscripts c, m, and r represent the composite, matrix, and reinforcement, respectively.

FIG. 5. Variation of modulus of elasticity with volume fraction of Al₂O₃ for two different particle sizes (50 and 150 nm). The solid lines were drawn on the basis of iso-stress or iso-strain conditions using the rule of mixtures. Note that the variation is close to the iso-strain condition for 50 nm-size Al₂O₃ particles and close to the iso-stress condition for the 150 nm-size Al₂O₃ particles.

inadequate room temperature ductility and insufficient creep resistance at elevated temperatures, especially between 800 and 850 °C, an important requirement for elevated temperature applications of these materials. Therefore, current research programs have been addressing the development of high-temperature materials with adequate room temperature ductility for easy formability and ability to increase the high-temperature strength by a suitable heat treatment or alloying additions to obtain sufficient creep resistance.

It has been shown that the compressive strength of binary γ-TiAl alloys with nanometer-sized grains is about 2600 MPa at room temperature and that, at temperatures higher than about 500 °C, the strength drops very rapidly to low values.34,35 In fact, the strength was found to decrease at a faster rate for ultrafine-grained materials than for the coarse-grained counterparts. That is, the smaller the grain size of the specimen, the stronger and sharper is the drop in yield strength on increasing the temperature. This observation suggests that monolithic nanostructured materials may not be suitable for achieving the desired creep resistance.

The Ti-Al-Si alloy system was chosen because it is a model system to study the influence of phase distribution and microstructure on the high-temperature deformation behavior of ceramic-intermetallic composites. Earlier work has demonstrated that favorable deformation behavior could be obtained in nanostructured/submicron-sized TiAl-based intermetallics. Thus, it was decided to check whether the microstructure has a similar influence on the mechanical behavior of ceramic-based ceramic-intermetallic composites which could then lead to easy deformability and eventually the possibility of achieving superplasticity. It is possible that the creep properties of these composites will be poor at low temperatures, but, similar to that in TiAl-based alloys, it could be improved through grain coarsening after deformation at elevated temperatures.

Composites of γ-TiAl and ξ-Ti₅Si₃ phase, with the volume fractions of the ξ-Ti₅Si₃ phase varying from 0 to 60 vol.%, were produced by MA of the blended elemental powders. Fully dense and porosity-free compacts were produced by hot isostatic pressing, with the resulting grain size of each of the phases being about 400 nm. Figure 6 shows a scanning electron micrograph of the γ-TiAl + 60 vol.% ξ-Ti₅Si₃ composite showing that the two phases are very uniformly distributed throughout the microstructure. Such a microstructure is expected to be conducive to superplastic deformation behavior. To test this hypothesis, both compression and tensile testing of these composite specimens were conducted at different temperatures and strain rates. From the tensile stress-strain plots of the γ-TiAl + 60 vol.% ξ-Ti₅Si₃ composite specimen shown in Fig. 7(a), we can draw the following conclusions.

Firstly, the strength decreased with increasing temperature and decreasing strain rate, both expected from normal mechanical behavior of metallic materials. Secondly, the specimens tested at 950 °C and a strain rate of 4×10⁻⁵ s⁻¹ and 1000 °C and a strain rate of 4×10⁻⁴ s⁻¹ exhibited large ductilities of nearly 150 and 100%, respectively. Considering that this composite is based on a ceramic material (Ti₅Si₃) this is a very high amount of
IV. GLASS FORMATION IN Fe-BASED SYSTEMS

Metallic glasses have an interesting combination of mechanical, physical, and chemical properties. They have been produced in several alloy systems by many different techniques. But, the most commonly used technique has been rapid solidification processing (RSP). As is well known, the critical cooling rate for glass formation needs to be exceeded so that formation of the crystalline nuclei could be avoided for the glass to form. However, since this is an experimentally determined value (and is time-consuming and also difficult to measure), several other empirical criteria have been proposed to explain glass formation in alloy systems. Two most commonly used criteria have been (a) a high reduced glass transition temperature, \( T_g = T_g/T_i \), where \( T_g \) is the glass transition temperature and \( T_i \) is the liquidus temperature of the alloy, and (b) presence of deep eutectics in the phase diagrams. The development of bulk metallic glasses in the late 1980’s [Ref. 40] has attracted the attention of materials scientists all over the world. Three criteria were proposed for the formation of bulk metallic glasses, viz., (a) presence of at least three components in the alloy system, (b) atomic radius difference of >12% between the elements, and (c) negative heat of mixing among the constituent elements. A very large number of bulk metallic glasses were produced using this set of criteria. But, since 2003 a number of new criteria based on the thermal properties of the alloys and physical characteristics of the component atoms were developed. These include the so-called \( \alpha \), \( \beta \), \( \gamma \), \( \gamma_{\text{iso}} \), \( \delta \), \( \sigma \), etc. parameters. But, it is important to remember that all these criteria have been developed for metallic glasses processed via the solidification route. As mentioned earlier, mechanical alloying (MA), a completely solid-state powder processing technique, is another important technique to produce amorphous alloys. Therefore, we wished to determine if these same criteria would be applicable to the solid-state processed amorphous alloys or other criteria need to be formulated to predict glass formation in alloy systems processed by MA. For this purpose, we had undertaken a comprehensive investigation on the glass formation behavior and stability of several Fe-based glassy alloys synthesized by MA.

IV.1. Number of Intermetallics in the Phase Diagram

We have chosen the general alloy composition as \( \text{Fe}_{28}X_{32}Zr_{10}B_{20} \) with \( X = \text{Al}, \text{Co}, \text{Ge}, \text{Mn}, \text{Ni}, \text{or Sn} \). The subscripts represent the composition of the alloy in
atomic percent. The element X was selected based on the number of intermetallics it forms with Zr under equilibrium conditions at room temperature. This number increases from 1 with Mn to 8 with Al, which provides a basis to analyze the results systematically. Further, the negative heat of formation of the intermetallics with Zr is much higher than that with either Fe or B. Thus, the probability of forming an intermetallic with Zr during milling is higher than with Fe or B. Blended elemental (BE) powders of the different constituents were weighed out in the correct proportion and milled in a high-energy SPEX mill for different times.

The sequence of phase formation on milling the powders was different depending on the alloy system. An amorphous phase had formed only in some alloy systems and not in all. The structural evolution in the Fe<sub>28</sub>Al<sub>20</sub>Zr<sub>10</sub>B<sub>20</sub> and Fe<sub>28</sub>Co<sub>20</sub>Zr<sub>10</sub>B<sub>20</sub> alloy systems as a function of milling time is shown in Figure 8. It may be noted that while an amorphous phase had formed on milling the Fe<sub>28</sub>Al<sub>20</sub>Zr<sub>10</sub>B<sub>20</sub> powder blend for 20 h, amorphization did not occur in the Fe<sub>28</sub>Co<sub>20</sub>Zr<sub>10</sub>B<sub>20</sub> powder blend. The phase formation sequence in all the six powder blends can be summarized into three groups as follows:

1. BE powder → Intermetallics → Solid solution
   - Examples: Mn- and Sn-containing systems,
2. BE powder → Solid solution
   - Example: Co-containing system,
3. BE powder → Intermetallics → Amorphous phase
   - (Mechanical) Crystallization
   - Examples: Al-, Ge-, and Ni-containing systems.

The time required for amorphization, which can be considered a measure of the glass-forming ability (GFA) of the alloy, is also different for different powder blends. Table III summarizes the results obtained, including the equilibrium number of intermetallic phases present between X and the constituent elements (Zr, Fe, or B) in the powder blend. A close examination of Table III clearly reveals that the ease of amorphization (i.e., GFA) increased with the number of intermetallics present in the constituent Zr-X binary phase diagrams. This is apparent from the powder blends containing Al or Ni, which amorphized in 10 and 20 h, respectively. While the quaternary Fe-Zr-Al-B contains eight intermetallic phases in the binary system between Zr and Al, the Fe-Zr-Ni-B system contains seven intermetallic phases in the binary system between Zr and Ni. Similarly, the Ge-containing system which also amorphized in 10 h, contains five intermetallic phases between Zr and Ge. The Zr-Co, Zr,Mn, and Zr-Sn systems which did not show

<table>
<thead>
<tr>
<th>Element X</th>
<th>Number of intermetallics between X and Zr</th>
<th>Number of intermetallics between X and Fe</th>
<th>Number of intermetallics between X and B</th>
<th>Total number of intermetallics</th>
<th>Milling time required for amorphization (h)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>8</td>
<td>5</td>
<td>2</td>
<td>15</td>
<td>10</td>
</tr>
<tr>
<td>Co</td>
<td>5</td>
<td>Nil</td>
<td>3</td>
<td>8</td>
<td>No amorphization</td>
</tr>
<tr>
<td>Ge</td>
<td>5</td>
<td>5</td>
<td>Nil</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>Mn</td>
<td>1</td>
<td>Nil</td>
<td>5</td>
<td>6</td>
<td>No amorphization</td>
</tr>
<tr>
<td>Ni</td>
<td>7</td>
<td>1</td>
<td>4</td>
<td>12</td>
<td>20</td>
</tr>
<tr>
<td>Sn</td>
<td>3</td>
<td>2</td>
<td>Nil</td>
<td>5</td>
<td>No amorphization</td>
</tr>
</tbody>
</table>

*TABLE III. Time required for glass formation in Fe<sub>28</sub>X(Zr)<sub>10</sub>B<sub>20</sub> alloys and the number of intermetallics in the respective binary alloy systems.*
amorphization on milling contain five, one, and three intermetallic phases, respectively. However, when we consider the total number of intermetallic phases, it can be clearly seen that the systems which amorphized on milling contain $\geq 10$ intermetallic phases in all the constituent binary phase diagrams. If the alloy system contained $<10$ intermetallic phases, then amorphization was not observed.

From an analysis of the phase diagrams of the alloy systems, it appears that the presence of a large number of intermetallic phases in the phase diagrams is conducive to formation of the amorphous phase. Again from the binary phase diagrams between these elements and Fe, it is apparent that those alloy systems that formed the amorphous phase contained a number of intermetallic phases, whereas those that did not form the amorphous phase easily had phase diagrams which featured a solid solution at room temperature. Thus, it is clear that the presence of a large number of intermetallic phases in the system helps in promoting the glassy phase formation.

The presence of intermetallics in an alloy system easily contributes to amorphization. This is due to two important effects. Firstly, disordering of intermetallics contributes an energy of about 15 kJ/mole of atoms to the system. For example, in strongly ordered intermetallics such as NiAl and γ-TiAl that continue to be in the ordered state till melting, the disordering energy has been estimated to be about 17.5 kJ/mol. Secondly, a slight change in the stoichiometry of the intermetallic increases the free energy of the system drastically. Additionally, grain size reduction contributes about 5 kJ/mole. Since MA reduces the grain size to nanometer levels and also disorders the usually ordered intermetallics, the energy of the milled powders is significantly raised. In fact, it is raised to a level above that of the hypothetical amorphous phase. This condition leads to a situation where the preferential formation of the amorphous phase is favored over the crystalline phase.

On the other hand, it is noted that in alloy systems which did not amorphize on milling, a solid solution phase only had formed. Since the only way a solid solution could contribute to an increase in the energy of the system is by grain size refinement, the total increase in the energy is not sufficiently high to amorphize the system. Thus, these observations provide a simple and visual means of identifying alloy systems which are likely to become amorphous on subjecting them to milling. If the equilibrium phase diagrams show the presence of a room temperature solid solution phase over a wide composition range, then amorphization is not easy. On the other hand, if the phase diagram contains a large number of intermetallic phases, then amorphization is a possibility.

**IV.2. Mechanical Crystallization**

We have also noted other interesting observations during MA of metallic powders. For example, it was noted that continued milling of the powder containing the amorphous phase led to crystallization, termed mechanical crystallization (Fig. 9). The main reason for the mechanical crystallization to occur during mechanical milling is that the relative stabilities of the different phases change due to the incorporation of a variety of defects. For example, we can discuss the relative stabilities of the competing phases in the Fe$_{28}$Ni$_{32}$Zr$_{10}$C$_{6}$B$_{10}$ system with respect to the free energy vs. composition diagram shown in Fig. 10.

The different phases that could exist in these alloys are the blended elemental powder mixture, α-Fe phase (the solid solution of Fe containing the different alloying elements), intermetallic phases and the amorphous phase. For our discussion, we have grouped all the possible intermetallics together and labeled them as “intermetallics” in the diagram.

In alloy systems with negative heat of mixing, the blended elemental powder mixture has obviously a high free energy (point “1”). Therefore, on MA, the blended powder mixture is expected to change into a more stable configuration to reduce its free energy. Thus, the α-Fe" solid solution phase (point “2”) containing all the solute elements in Fe is seen to be forming on milling. Since MA introduces many crystal defects including vacancies, dislocations, grain boundaries, surfaces, and others, the milled powders will have excess energy. It was clearly shown by us earlier that intermetallics form in the early stages of milling in many of the Fe-based powder mixtures. And disordering of such intermetallics can also substantially increase the free energy of the system. Hence, on continued milling, the free energy of the crystalline phase increases to a level above that of the

![Fig. 9](image.png)

**FIG. 9.** X-ray diffraction patterns of the Fe$_{28}$Ni$_{32}$Zr$_{10}$C$_{6}$B$_{10}$ powder blend milled for 8 and 30 h. Note that the powder milled for 8 h shows the presence of an amorphous phase. On continued milling of this powder containing the amorphous phase to 30 h, mechanical crystallization had occurred resulting in the formation of several crystalline phases.
amorphous phase which results in the destabilization of the crystalline phase and the "amorphous" phase (point "3") begins to be stabilized. But, further milling leads to mechanical crystallization resulting in the formation of a solid solution or intermetallics co-existing with the remaining amorphous phase. Since primary crystallization had occurred, the Fe-solid solution will now have a composition different from the original α-Fe sol. solid solution; let us call this α-Fe'. This solid solution will now coexist with the amorphous phase which also has a different composition because of the precipitation of α-Fe' from the original amorphous phase (amorphous'). Point "4" now represents the free energy of the mixture of amorphous' and α-Fe'. The lowest free energy for the system will, however, correspond to a condition when a mixture of the equilibrium solid solution (α-Fe) and the intermetallics coexist. This situation is indicated by point "5" in the figure.

The influence of additional alloying elements with different atomic sizes and heats of mixing were also investigated with useful attributes for consolidating the milled powders to full density.\textsuperscript{46,47}

V. SYNTHESIS OF PHOTOVOLTAIC MATERIALS

Copper indium diselenide (CulnSe\textsubscript{2})-based photovoltaic thin film solar cells continue to receive attention because of their high conversion efficiency.\textsuperscript{48} It has been shown that the band gap of CulnSe\textsubscript{2} can be modified continuously by substituting Ga for In; the band gap increases with increasing Ga content. For achieving the maximum efficiency of the cell, a Ga/(Ga+In) ratio of 0.3 and a slight deficiency in the copper content have been found to be beneficial.

The CulnSe\textsubscript{2}-based alloy films are usually obtained either by multisorce thermal evaporation and co-deposition or reactive annealing of precursor films. These processes are very involved, contain a number of deposition and annealing steps, pose difficulties in maintaining the desired film composition, and can be expensive. Therefore, we decided to perform band gap engineering through the technique of MA. The two simple steps involved will be synthesis of stoichiometric powder of the Culn\textsubscript{0.7}Ga\textsubscript{0.3}Se\textsubscript{2} (CIGS) alloy by MA and obtain the bulk target by hot isostatic pressing of the milled powder. Milling of the blended elemental powder was conducted in a Fritsch Pulverisette P5 mill using a copper container and stainless grinding balls at two different speeds and at two different ball-to-powder weight ratios (BPR).

Milling of the powders even for a short time resulted in the formation of the desired Culn\textsubscript{0.7}Ga\textsubscript{0.3}Se\textsubscript{2} phase. For example, by milling at a BPR of 20:1 and a speed of 300 rpm, the tetragonal Culn\textsubscript{0.7}Ga\textsubscript{0.3}Se\textsubscript{2} phase had formed. But, small amounts of other phases were also present at this stage. However, on continued milling of this powder for 20 min, only the tetragonal Culn\textsubscript{0.7}Ga\textsubscript{0.3}Se\textsubscript{2} phase had formed homogeneously (Fig. 11). This phase could be indexed on the basis of a tetragonal structure with the lattice parameters $a = 0.5736$ nm, $c = 1.1448$, and $c/a = 1.9958$ having the space group 142d. Even though the quaternary Cu-In-Ga-Se tetragonal phase exists over a

![FIG. 10. Hypothetical free energy vs. composition diagram to explain the mechanism of mechanical crystallization in the Fe$_2$Ni$_3$Zr$_{10}$Cu$_3$B$_9$ system. Note that point "1" represents the free energy of the blended elemental powders. Similarly, point "2" represents formation of the α-Fe solid solution of all the alloying elements in Fe point "3" formation of the homogeneous amorphous phase, point "4" a mixture of the amorphous phase with a different solute content (amorphous') than at "3" and the solid solution α-Fe', and point "5" is for the equilibrium situation when the solid solution and intermetallics coexist.](image)

![FIG. 11. X-ray diffraction patterns of the CIGS blended elemental powder mixture milled at a BPR of 20:1 and a speed of 300 rpm for 5 and 20 min. The powder milled for 5 min contained the tetragonal CuIn$_{0.7}$Ga$_{0.3}$Se$_2$ phase and small amounts of elemental Cu and In. When the powder was milled for 20 min, it contained only the tetragonal CuIn$_{0.7}$Ga$_{0.3}$Se$_2$ phase.](image)
that the compact was fully dense and did not contain any porosity. Figure 12(a) shows the XRD pattern of the hot isostatically pressed alloy suggesting that the CuIn0.7Ga0.3Se2 phase is retained with the exact stoichiometry and that the peaks are sharper than what they were in the milled powder.51 This is mainly due to grain growth, as confirmed by TEM investigations, Fig. 12(b). The TEM micrograph shows that the grain size now is about 50-60 nm.

This investigation clearly proves that it is possible to obtain the CIGS targets of the required stoichiometry through a combination of MA and hot isostatic pressing or vacuum hot pressing. Further, it is possible to control the stoichiometry by optimizing the MA process parameters. Such an approach should be equally applicable to synthesize other target materials.

VI. COMBUSTION STUDIES OF METAL PARTICLES

One of the potential applications for materials with fine grain sizes is in improving the combustion characteristics of propellants.52 It is known that Mg can be easily combusted, but it has a low heat content (14.9 MJ/kg). On the other hand, Al has a higher heat content (32.9 MJ/Kg), but is more difficult to combust. Therefore, it was decided to see if Al and Mg can be alloyed together to increase the net heat content and also be able to combust relatively easily. Accordingly, a series of Al-Mg powder blends of different compositions were mechanically alloyed in a SPEX mill. Since the milled powder normally contains a range of particle sizes, the chemical composition of the milled powder under different milling conditions is shown in Table IV. From the results it is clear that the composition of the milled powder is reasonably close to the targeted composition. But, because some copper has been picked up from the container and/or the grinding balls during milling, the copper content appears to be higher than what the stoichiometry requires. But, the powder milled at a BPR of 10:1 and a speed of 150 rpm for 40 min did not pick up much copper. Consequently, the chemical composition of the powder milled under these conditions is quite close to the starting powder mixture. By further optimizing the powder processing conditions (BPR, speed of rotation, and time of milling), it is possible to control the composition of the final powder to the exact desired value.

The milled powder containing the homogeneous CuIn0.7Ga0.3Se2 phase was consolidated by hot isostatic pressing to full density at 750 °C and 100 MPa for 2 h. Examination in a scanning electron microscope indicated that the compact was fully dense and did not contain any porosity. The Scherrer analysis of the peak widths50 confirmed that the crystallite size reached a saturation value of about 8 nm. The phase identification and crystallite size determination were confirmed by transmission electron microscopy (TEM) studies.50

The chemical analysis of the milled powder under different milling conditions is shown in Table IV. From the results it is clear that the composition of the milled powder is reasonably close to the targeted composition. But, because some copper has been picked up from the container and/or the grinding balls during milling, the copper content appears to be higher than what the stoichiometry requires. But, the powder milled at a BPR of 10:1 and a speed of 150 rpm for 40 min did not pick up much copper. Consequently, the chemical composition of the powder milled under these conditions is quite close to the starting powder mixture. By further optimizing the powder processing conditions (BPR, speed of rotation, and time of milling), it is possible to control the composition of the final powder to the exact desired value.

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<table>
<thead>
<tr>
<th>BPR</th>
<th>Speed (rpm)</th>
<th>Time (min)</th>
<th>Cu</th>
<th>In</th>
<th>Ga</th>
<th>Se</th>
</tr>
</thead>
<tbody>
<tr>
<td>20:1</td>
<td>300</td>
<td>120</td>
<td>32.61</td>
<td>15.23</td>
<td>6.53</td>
<td>45.62</td>
</tr>
<tr>
<td>10:1</td>
<td>300</td>
<td>240</td>
<td>31.73</td>
<td>15.63</td>
<td>6.71</td>
<td>45.93</td>
</tr>
<tr>
<td>10:1</td>
<td>150</td>
<td>150</td>
<td>25.98</td>
<td>16.98</td>
<td>7.25</td>
<td>49.78</td>
</tr>
<tr>
<td>Targeted (Cu0.97In0.03Ga0.3Se2)</td>
<td></td>
<td></td>
<td>24.43</td>
<td>17.63</td>
<td>7.56</td>
<td>50.38</td>
</tr>
</tbody>
</table>

TABLE IV. Chemical analysis (at.%) of the Cu-In-Ga-Se powder mechanically alloyed under different processing conditions.50

FIG. 12. (a) X-ray diffraction pattern and (b) transmission electron micrograph of the CIGS powder mechanically milled and consolidated to full density at 750 °C, 100 MPa and 2 h. The XRD pattern shows that the CuIn0.7Ga0.3Se2 phase is fully recrystallized as revealed from the sharp peaks and the TEM micrograph confirms this observation. The grain size of the recrystallized material is now about 50 nm.
milled powder was sieved to well-defined particle sizes in the ranges of 20-25 μm, 45-53 μm, and 90-106 μm (Fig. 13). These powder particles were then burned in a methane-air premixed flame on a Meeker burner, at a temperature of approximately 1,800 K. Burning of the powder particles was recorded using chopped images by long-exposure digital photography. Knowing the rotational speed of the chopper and the number of exposures per revolution, the burning time, τ_b and the ignition time, τ_i could be determined. Figure 14 shows the plot of variation of τ_i and τ_b with particle size for two different alloy compositions (Al-20 at.% Mg and Al-90 at.% Mg).\textsuperscript{33} It is clear from this plot that both the ignition and burning times decrease with (a) a decrease in particle size and (b) an increase in Mg content. Thus, by a judicious choice of particle size and alloy composition, it should be possible to control the ignition and burning times.

VII. CONCLUSIONS

From the above description of the type of activities, it becomes amply clear that the technique of MA is very efficient in synthesizing a variety of equilibrium and non-equilibrium advanced materials. Some of the present issues that need immediate attention are that we need homogeneous, pure, macro-defect (i.e., porosity and cracks)-free and fully dense materials for obtaining reproducible results. Further, large-scale applications require materials that could be produced in tonnage quantities, and materials that have been well characterized and exhibit reproducible properties. But, the bane of MA has been the three C’s – Cost, Consolidation, and Contamination. Powder processing is expensive and therefore, unless one is able to identify niche markets for the MA products and the powders produced in large volumes, cost is likely to continue to be high. However, in some cases, e.g., hydrogen storage materials or magnetic alloys, the high cost may be acceptable in view of the improved properties and enhanced performance.

Consolidation of the milled powder continues to be a serious problem. There have not been many investigations to report successful consolidation of MA powders to achieve full density and simultaneously retain the metastable effects. Newer and improved methods or modification/combination of the existing methods may be necessary.

The last point is that contamination of the MA powder has been a serious issue in many cases.\textsuperscript{13,14} Some solutions have also been suggested to minimize/avoid contamination completely. These include enclosing the mills inside chambers that have been evacuated and/or filled with an inert gas, or using high-purity gaseous atmosphere in which the powders could be milled. Both options could be expensive and/or impractical.

The MA powders could also be used in the as-produced powder condition without consolidation. Examples for such applications include use as catalysts, pigments, solder, hydrogen storage materials, materials for printed electronics, (CIGS powders are already in use for printed solar cells, Nanosolar, etc.). In that case, some of the above problems mentioned above may not be relevant. Further, even if a bulk component made of MA powder needs to be used, then if that material could tolerate high gaseous impurities, then also there is no problem. With the intense research activity going on all over the world, there is great potential for MA products and bright future for research in this area.

ACKNOWLEDGEMENTS

The work reported here is supported by the US National Science Foundation under grants DMR-0314212 and DMR-0334544 and by the Office of Naval Research. The authors also acknowledge fruitful collaboration and useful discussions with Drs. Raj Vaidyanathan, Linan An, and Ruey-Hung Chen of the University of Central Florida, Orlando, USA, Professor Rüdiger Bormann of the Hamburg-Harburg Technical University, Hamburg, Germany, and Dr. S.J. Hong of Kongju National University, Kongju, South Korea. C.S. is also thankful to the experimental input from his graduate students – Pushkar Katiyar, Balaji Prabhu, Satyajeet Sharma, and Uma Maheswara Rao Seelam, all from the University of Central Florida.
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Root of tunnel magnetoresistance and its development

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Nowadays usually we use the term, tunnel magnetoresistance, but it required a long time to combine both words tunnel and magnetoresistance. The study of tunnel junction may originate from p-n junction studied first around 1950. On the other hand, magnetoresistance effect was reported first in 1857 which was about 100 years earlier than the start of tunnel junction study. The research of tunnel magnetoresistance has been mainly developed first for Al-oxide tunnel barrier junctions and made a remarkable progress by the appearance of MgO barrier junctions for both basic research and applications. Most recently, Heusler electrode tunnel junction exhibits a large TMR ratio up to about 750%. We will describe first the history of the study of tunnel junction and magnetoresistance effect. Then, we will focus on the Heusler electrode junctions and also application of tunnel magnetoresistance junctions.

1. Introduction

The discovery of giant magnetoresistance effect1-2 (GMR) of a metallic artificial lattice and tunnel magnetoresistance effect (TMR)3,4 in the ferromagnetic tunnel junction became a trigger for the field called “spin-electronics” or “spintronics” in the past about 20 years, which has developed steadily. The point that this research is excellent is that not only the object of a basic research but also application. It has greatly contributed to the development of industry, as the magnetoresistive reading heads and nonvolatile random access memory (MRAM). First of all, the authors want to examine the root of tunnel magnetoresistance here. Next, the development of the TMR effect using the Al2O3 barrier is described. Using the MgO barrier instead of the Al2O3 barrier, the TMR ratio increased rapidly. Also the TMR ratio increased, when the Heusler alloy is used for the electrodes. Finally, the situations of spin-RAM development and a few associated research are being done now are introduced.

2. Root of TMR effect

(a) Root of tunnel effect

The term TMR consists of the two words of the magnetoresistance and the tunnel. Why does the physics of the tunnel phenomenon become to be interested in? As you know, the tunnel effect is a quantum-mechanical phenomenon which is impossible on basis of classical mechanics. G. Gamow explained the α particle (He-nuclei) - decay on the basis of the tunnel effect in 1928. It may be said that the term tunnel effect was used, for the first time, by him. However, it was an electronic tunnel phenomenon in solid that in around 1960 after about 30 years it was popularly known. Especially, the research of the diode p-n junction was developed into the device by Esaki.5 in 1958. Giaever et al.6, early in 1960’s, began the research of the energy gap of the superconductor, using the junction consisting of superconductor/insulator/superconductor. The conduction electron can pass through the insulating layer by the tunnel effect even if the metal or the semiconductor is separated by a thin insulator or a potential barrier. Giaever et al.6 paid attention to this respect, measured the energy gap of the superconductor for the |J|V' characteristics of various junctions, and clarified the density of state of superconductor. On the other hand, Tedrow et al.7, obtained the various ferromagnetic polarization including Ni by replacing the other superconductor of the junction by ferromagnetic material, and analyzing the |J|V' characteristics. These data of spin polarization were fine and very useful in order to investigate a high TMR junction. Julliere8 (1975) reported on the applied voltage dependence of conductance of tunnel junction of which both electrodes consist of ferromagnetic substance. Moreover, they discussed the change of conductance with the change of magnetization direction in both magnetic layers using spin polarization. The term “magnetoresistance” is not used, though this paper is the first one of the TMR effect. The term magnetoresistance was not used though the researches using a ferromagnetic/insulator/ferromagnetic/ junction by 1990 were often reported. One of double terms, “tunnel” was often used in the papers. An interesting respect is a point to approach to the terms magnetoresistance. The term of tunneling resistance by Maekawa et al.9 or magnetic valve by Slonczewski10,11, was used in each of their papers. The typical junctions mentioned above are shown in Table 1, together with the fields of the research. By the way, tunneling junction with the vacuum instead of the insulator was reported by Binnig et al.12 in 1982. It has been practically used as STM (Scanning Tunneling Microscope), which gives much contribution to the research on the physics of surface phenomena.

(b) Root of magnetoresistance effect

The first report of magnetoresistance effect in Ni and Fe was published by Thomson13 in 1857. The term of the so-called magnetoresistance was not used at that time even though the paper was published on the effect of an anisotropic resistance. By the way, the title is “Effects of magnetization on the electric conductivity of Nickel and of Iron.” He was later elevated to Lord Kelvin by his
contribution to the fundamental science and engineering. However, the one century is required for the contribution to engineering in work practically, since at that time it did not attract much practical attention. The term magnetoresistance was used in the paper of McKeehan in 1930 though the first paper using magnetoresistance is not made clear. An overview of anisotropic magnetoresistance up to about 1975, was given by Meguire and Potter. Moreover, the review of the applied researches that came to be done extensively around 1960-1970 was given by Thomson. The first attempt to construct the theory of anisotropic magnetoresistance was made mainly in Europe in the 1950-1970’s. Especially, Smit took up this problem at early time. Moreover, both the theory and the experiment have been researched by Fert and Campbell for a long time. These theoretical considerations were based on the two current model by Mott. Research of magnetoresistance is roughly summarized as shown in Table 2. Apparently, the term “tunnel magnetoresistance” was not used in the 1980’s. Later in 1990’s several reports of magnetoresistance of the tunnel junction became to be published besides GMR. It may be said, from the other point of view, that the research intended for application could link magnetoresistance with tunnel. The titles of the papers of tunnel junction quoted often until 1995 are shown in Table 3. The term tunnel magnetoresistance (TMR) is not used, though the two terms of tunnel (tunneling) and magnetoresistance were used in the title. This kind of research has been increasing gradually after 1995 and the term TMR came to be used. The author’s group used TMR, for the first time in the latter half of 1990’s. The root in the effect of TMR will be maintained in the following. Moreover, the author’s group observed a large TMR ratio of about 18 % at room temperature for the first time with the corresponding hysteresis curve as shown in Fig. 2.

### 3. Giant tunnel magnetoresistance effect

#### 3.1 Tunnel junction with MgO barrier

During early ten years of the start of TMR research, the effect of TMR with tunnel barrier layer of an amorphous aluminum oxide (Al-oxide) was a main current of research. After 2004, however, much more attention has been paid to the research of junction of which barrier layer is the crystalline magnesium oxide (MgO). When the electron tunnels through the

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<table>
<thead>
<tr>
<th>Table 1. Historical details of research of junctions.</th>
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<tr>
<td>Junction</td>
</tr>
<tr>
<td>---</td>
</tr>
<tr>
<td>1.</td>
</tr>
<tr>
<td>2.</td>
</tr>
<tr>
<td>3.</td>
</tr>
<tr>
<td>4.</td>
</tr>
<tr>
<td>5.</td>
</tr>
<tr>
<td>6.</td>
</tr>
</tbody>
</table>


<table>
<thead>
<tr>
<th>Table 2. History of research of magnetoresistance effect.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1930-60</td>
</tr>
<tr>
<td>1960-70</td>
</tr>
<tr>
<td>1975</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Table 3. Early research of tunnel magnetoresistance effect.</th>
</tr>
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<tbody>
<tr>
<td>1975</td>
</tr>
<tr>
<td>1982</td>
</tr>
<tr>
<td>1987</td>
</tr>
</tbody>
</table>

1988 GMR reports

| 1989 | Sionczewski: Conductance and exchange coupling of two ferromagnets separated by tunneling barrier |
| 1991 | Miyazaki et al.: Large magnetoresistance effect in BiFeCo/AlO3 tunneling junction |
| 1992 | Nakaizumi and Kitada: Changes in the electrical resistivity of Fe-AlO3/Fe-Ru multilayered films due to a magnetic field |
| 1992 | Novak and Raulowski: Spin dependent electron tunneling between ferromagnetic films |
| 1995 | Guo and Gao: Spin-polarized electrons and magnetoresistance in ferromagnetic tunnel junctions and multilayers |

1995 Miyazaki and Tanaka: Giant magnetic tunneling effect in FeAlO3/Fe junction |

1995 Mochida et al.: Large magnetoresistance at room temperature in ferromagnetic thin film tunnel junctions |
amorphous Al-oxide barrier layer, scattering is received and information on the wavenumber vector is lost (incoherent tunnel). On the other hand, the electron can tunnel coherently through the tunnel junctions on epitaxially developed layer such as Fe (001)/MgO (001)/Fe (001) junction. Butler and Mathon showed that it is possible to obtain a giant TMR ratio of as large as over 1000% by the theoretical calculation for Fe/MgO/Fe tunnel junction in 2001. In the tunnel junction with the MgO barrier, Fe/MgO/Fe made by the MBE method, Yuasa et al. observed as large as 88 % TMR ratio experimentally at room temperature. Having exceeded the maximum of TMR ratio of the tunnel junction with the Al-oxide barrier layer at that time is of meaningful use, though it was relatively smaller than the theoretical estimation. Afterwards, Yuasa et al. (Fig. 3) and Parkin et al. succeeded in the observation of as large as 200% TMR ratio at room temperature in 2004. In addition, the researchers at AIST and Canon-Anelba jointly showed that the huge TMR ratio can be achieved by combining the amorphous ferromagnetic material and the MgO barrier layer named CoFeB by the sputtering method appropriate for the productive process. Afterwards, this technology continues developing, and the TMR ratio up to 600% at room temperature is observed now. The MgO barrier tunnel junction is used now for the read head of the latest hard disk.

The TMR ratio of typical MgO barrier tunnel junctions is summarized in Table 4. Moreover, the growth of the TMR ratio at room temperature, using the MgO and Al2O3 barrier for years is plotted in Fig. 4.

Fig. 1. TMR research combined with tunnel and magnetoresistance through GMR.

Fig. 2. (a) magnetoresistance curve of Fe/Al2O3/Fe junction at RT. (b) the corresponding magnetization curve.

Fig. 3. Magnetoresistance curve of Fe/MgO/Fe junction.

Table 4. TMR ratio of typical MgO barrier tunnel junction.

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe/MgO/Fe</td>
<td>MBE</td>
<td>88%</td>
<td>[30]</td>
</tr>
<tr>
<td>Fe/MgO/Fe</td>
<td>MBE</td>
<td>180%</td>
<td>[31]</td>
</tr>
<tr>
<td>CoFe/MgO/CoFe</td>
<td>Sputtering</td>
<td>220%</td>
<td>[32]</td>
</tr>
<tr>
<td>CoFeB/MgO/CoFeB</td>
<td>Sputtering</td>
<td>230%</td>
<td>[33]</td>
</tr>
<tr>
<td>CoFeB/MgO/CoFeB</td>
<td>Sputtering</td>
<td>604%</td>
<td>[34]</td>
</tr>
</tbody>
</table>
The attempt to improve the TMR ratio by using ferromagnetic electrode material with the large spin polarization has been made as an approach besides the MgO barrier layer tunnel junction.

The so-called half metal is expected for an ideal ferromagnetic material to improve the effect of TMR. It is a material that completely polarizes spin on Fermi surface (only either one type of electronic spin of the two types of electrons i.e., electron with spin up, or electron with spin down), of which spin polarization is ideally 100 % (Fig.5). The TMR ratio could be expected to become infinity theoretically in the tunnel junction using the half metal for the electrodes, based on the Julliere’s model. Half metal material is classified roughly into the oxide system and the metal system. First of all, the related physical quantities such as Curie temperature, energy gap, magnetic moment for each unit molecule (theoretical value), crystal structure, and the TMR ratio of the tunnel junction with these electrodes are collectively shown for typical oxide system half metal reported up to now in Table 5. The band gap of the oxide system half metal is as large as 1eV, which is advantageous for large TMR ratio in the high bias voltage. On the other hand, the disadvantage is a point that the Curie temperatures of them are rather low. Each of the Curie temperatures of LSMO, LCMO, and CrO₂ are level with room temperature, therefore it is difficult to observe a large TMR ratio at room temperature. As understood from Table 5, TMR ratios at room temperature of tunnel junction using the oxide system half metal are as small as 10% in the reports of the past, in contrast to the huge TMR ratios observed at low temperatures. Moreover, another feature of oxide system is for the effect of inverse-TMR to be observed. In contrast to a usual effect of TMR, inverse-TMR effect has a feature that the parallel resistance is large, but the anti-parallel resistance is small in respect of orientation of magnetization. It is, physically, a very interesting phenomenon though a huge effect of inverse-TMR is not observed at present. The authors explain, here, historical details of the Heusler alloy that is a half metallic material: it was discovered by F. Heusler first in 1903, which had been named commemorating the discoverer. It is notable that the material exhibits strong ferromagnetism though an alloy named Cu₃MnAl is composed only of the nonmagnetic metals. Afterwards, it has become attracting the attention as a half metal material after 1983, though a basic research on the evaluation of physical properties had been studied for about 80 years. Groot et al. in 1983 carried out the band calculation of the Heusler alloy of composition named NiMnSb and concluded that this material has the half metal electronic state. In 1995 afterwards, the calculated result that the compounds such as Co₃MnSi and Co₃MnGe could be the half metal was reported by Ishida et al. It came to be paid attention again in the Spintronics field, since the high Curie temperature of the Heusler alloy is very advantageous for application. Physical quantities of typical Heusler alloys such as the Curie temperature, the magnetic moment, and the TMR ratio are level in the reports of the past, in contrast to the huge TMR ratios observed at low temperatures. Moreover, another feature of oxide system is for the effect of inverse-TMR to be observed. In contrast to a usual effect of TMR, inverse-TMR effect has a feature that the parallel resistance is large, but the anti-parallel resistance is small in respect of orientation of magnetization. It is, physically, a very interesting phenomenon though a huge effect of inverse-TMR is not observed at present. The authors explain, here, historical details of the Heusler alloy that is a half metallic material: it was discovered by F. Heusler first in 1903, which had been named commemorating the discoverer. It is notable that the material exhibits strong ferromagnetism though an alloy named Cu₃MnAl is composed only of the nonmagnetic metals. Afterwards, it has become attracting the attention as a half metal material after 1983, though a basic research on the evaluation of physical properties had been studied for about 80 years. Groot et al. in 1983 carried out the band calculation of the Heusler alloy of composition named NiMnSb and concluded that this material has the half metal electronic state. In 1995 afterwards, the calculated result that the compounds such as Co₃MnSi and Co₃MnGe could be the half metal was reported by Ishida et al. It came to be paid attention again in the Spintronics field, since the high Curie temperature of the Heusler alloy is very advantageous for application. Physical quantities of typical Heusler alloys such as the Curie temperature, the magnetic moment, and

Table 5. Magnetic properties and TMR ratio of oxide half metal.

<table>
<thead>
<tr>
<th>Materials</th>
<th>Curie Temp. (K)</th>
<th>Band gap (eV)</th>
<th>Moment (μB/μJ)</th>
<th>Crystal structure</th>
<th>TMR ratio</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>LaCoMnO₅ (LCO)</td>
<td>89 ± 1.5</td>
<td>0.8 ± 1.5</td>
<td>Perovskite</td>
<td>LSMO/LaCoO₃/LMO</td>
<td>60% (70%), 60% (19%)</td>
<td>[35,36]</td>
</tr>
<tr>
<td>LaMnMoO₃ (LMMO)</td>
<td>370 ± 1.4</td>
<td>1.4 ± 0.4</td>
<td>Perovskite</td>
<td>LSMO/LaMnO₃/LMO</td>
<td>100% (4.2K)</td>
<td>[37,38,39]</td>
</tr>
<tr>
<td>CrO₂</td>
<td>308 ± 1.3</td>
<td>1.5 ± 0.3</td>
<td>Di-octahedral</td>
<td>G0/2O₂</td>
<td>-5% (100%)</td>
<td>[40,41]</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>505 ± 1.3</td>
<td>1.3 ± 0.1</td>
<td>Sphalerite</td>
<td>Fe₂O₃/AI oxide/Co</td>
<td>40% (4.2K)</td>
<td>[42,43]</td>
</tr>
</tbody>
</table>
the energy gap are summarized in Table 6. The Heusler alloys are classified into a half Heusler alloy such as NiMnSb and a full Heusler alloy such as Co₅MnSi. Here, the full Heusler alloy with a large TMR ratio will be simply described the Heusler alloy, and the authors explain it a little more in detail. The Heusler alloy basically has the X₂YZ composition, and takes three units lattice structures according to the rule of the atomic ordered arrangement as shown in Fig. 6. The elements that enter each of sites are as follows.

X: Fe, Co, Ni, Ru, Rh, Pd, Ir, Pt, Cu, Zn, Ag, Cd, and Au/chiefly VIII, IB, IIB family
Y: Ti, V, Cr, Mn, Y, Zr, Nb, Hf, Ta, Gd, Tb, Dy, Ho, Er, Tm, Yb/chiefly III, IV, V, VI, VII family
RE Z: Al, Si, Ga, Ge, As, In, Sn, Sb, Ti, Pb, and Bi/chiefly, III B, IVB family.

L₂₁ structure: the atoms are completely and regularly arranged.
B₂ structure: the Y-Z atoms are irregularly arranged.
A₂ structure: the X-Y-Z atoms are completely irregularly arranged.

The property as half metal, an electric conduction characteristic, and magnetic characteristic etc. of Heusler alloy may be influenced according to the degree of the order. Miura et al. calculated the irregularity dependence of spin polarization and magnetic moment in the Co₅Cr₅Al Heusler alloy. As a result, an ideal state of half metal is achieved in the state of a complete order (L₂₁ structure), but the spin polarization decreases rapidly with an irregularity between Co-Cr (A₂ structure). Therefore, the evaluation of the structure factor is important for developing the Heusler alloy. The evaluation of the most popular atomic structure factors can be deduced by the X-ray diffraction experiment. Briefly, it can be estimated from measuring the strength ratio of the basic lattice line to the super-lattice line. The TMR ratios of tunnel junction that combine the Heusler alloy electrode with the amorphous Al-oxide barrier layer are collectively shown in Tab. 7. In a pioneering research, the composition NiMnSb of the Heusler alloys was used for an electrode. But the TMR ratio is as small as about 10% even at low temperature, which is not enough to prove the characteristics of half metal. Afterwards, in around 2003 the research using a full Heusler with the structures steadier than the half Heusler ones was started and the tens % TMR ratio came to be observed. The breakthrough in the tunnel junction using Heusler alloy began with making the interfaces of the epitaxially developed Heusler alloy between insulators smoothly and cleanly. Sakuraba et al. obtained the ratio of 159% at low temperature for the Co₅MnSb/Al-oxide/CoFe tunnel junction with the MgO single crystaline substrate. The spin polarization of Co₅MnSb estimated on the basis of Julliere’s model from this TMR ratio is as large as 89% (spin polarization of CoFe is assumed to be 50%). It was shown for the first time that the spin polarization of Co₅MnSb is very high. Afterwards, 570% TMR ratio was observed in Co₅MnSb/Al-oxide/Co₅MnSb junction of which composition of both electrodes is Co₅MnSb.

Table 7. TMR ratio of typical Heusler alloy electrodes and Al-oxide barrier tunnel junctions.

<table>
<thead>
<tr>
<th>Materials</th>
<th>Crystal structure</th>
<th>TMR ratio (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>NiMnSb</td>
<td>NiMnSb/Al-oxide/NiFe</td>
<td>5% (RT), 19% (4.2K)</td>
</tr>
<tr>
<td></td>
<td>NiMnSb/Al-oxide/NiFe</td>
<td>2.4% (RT), 8% (77K)</td>
</tr>
<tr>
<td>Co₅MnSb</td>
<td>Co₅MnSb/Al-oxide/CoFe</td>
<td>33% (RT), 84% (10K)</td>
</tr>
<tr>
<td></td>
<td>Co₅MnSb/Al-oxide/CoMnSb</td>
<td>70% (RT), 159% (2K)</td>
</tr>
<tr>
<td>Co₅MnAl</td>
<td>Co₅MnAl/Al-oxide/CoFe</td>
<td>87% (RT), 870% (2K)</td>
</tr>
<tr>
<td>Co₅MnGe</td>
<td>Co₅MnGe/Al-oxide/CoFe</td>
<td>6% (RT), 10% (10K)</td>
</tr>
<tr>
<td></td>
<td>Co₅MnGe/Al-oxide/CoMnSb</td>
<td>19% (RT), 26% (2K)</td>
</tr>
<tr>
<td>Co₅MnAl</td>
<td>Co₅MnAl/Al-oxide/CoFe</td>
<td>40% (RT), 60% (10K)</td>
</tr>
</tbody>
</table>

Fig. 6. Crystal structure of Heusler alloy.

Fig. 7. Temperature dependence of TMR ratio of Heusler electrode with Al-oxide barrier tunnel junction.
However, the TMR ratio was as small as 100% or less at room temperature in the junction using the AI-oxide barrier layer. The analysis of the bias dependence of conductance shows clearly the band gap of the Co$_2$MnSi electrodes, but the Fermi level in the gap is very close to the bottom conduction band (Fig.8). This is the reason for the strong temperature dependence of TMR. Recently, the development of the tunnel junction that combines the Heusler alloy with the MgO barrier layer has rapidly advanced. Table 8 shows typical results. The lattice adjustment of the Heusler alloy and MgO has an advantage of full possible epitaxial growth up to the above part of ferromagnetic electrode. In addition, the possibility to generate a large TMR ratio was theoretically shown by the synergy effect of the half metal: the coherent tunneling phenomenon and the Heusler alloy separated by the MgO barrier layer. Experimentally, a huge TMR ratio of 200% or more was obtained, at room temperature for the tunnel junction that combines the Heusler alloy with MgO. High expectations have been paid for the progress of a further research and development in a near future. The growth of TMR ratio of the tunnel junction with Heusler electrode vs. year is presented in Fig. 9.

4. Spin transfer torques

In the effect of the tunnel magnetoresistance (same with the effect of GMR), the electric current can be controlled by the direction of magnetization. In contrast, the direction of magnetization may be altered, depending on the current in a spin transfer torque. The current with polarized spin is altered by a relative angle of magnetization for the effect of TMR. Thus the spin polarized current is needed, to cause the spin transfer torque.

![Fig. 8. Bias dependence of tunnel conductance of Co$_2$MnSi/Al-O/CoFe junction.](image)

![Table 8. TMR ratio of typical Heusler alloy electrodes and MgO barrier tunnel junctions.](image)

For an intuitive image of this physics, it is only thought that the magnetoresistance effect and spin transfer effect (torque) could analogously correspond to “law of action and reaction” of the Newton mechanics (Fig.10).

We will explain a little more detail of spin transfer torque: when both ends of ferromagnetic (F1)/nonmagnetic (N)/ferromagnetic (F2) junction could be applied by voltage, current flows from right to left, and electron, vice versa, as shown in Fig.11. Each of spin angular momentum of electron in electrodes is denoted as $S_1$ and $S_2$, respectively. The (unpolarized) spin $s$ of the conduction electron which enters in the F1 layer from a left electrode can be changed to spin-polarized ones parallel to $S_2$. When this polarized spin $s$ pass through F2 layer, the momentum of $s$ can be altered by interaction of $s$ and $S_2$ and the difference of angular momentum moved to $S_2$. As a result, $S_2$ may receive torque. The direction of the torque received by $S_2$ is expressed in terms of $S_2$ and the unit vector of spin $s$ of conduction electron. It is noted that depending on the direction of the current,
The reversal spin condition is given by the spin polarized electron and was given by Slonczewski respectively, \( g \) is also an important subject. The magnetic memory MRAM (Magnetoresistive Random Access Memory) development has been studied using this physics. In the magnetic memory (especially, spin-RAM in distinction from the conventional type) has come to be paid to attention. The spin memory can be 4 kbit (Sony) in 2005, and 2 Mbit in 2007 (Hitachi-Tohoku University). In addition, using the cell of perpendicular magnetization to plane than in plane, an excellent spin-RAM technology has been made in the research of the most recently by Toshiba. The research for the target has been studied intensely all over the world. The current trend from MRAM to spin-RAM is schematically shown in Fig. 12. Then, why is the cell geometry of vertical magnetization advantageous compared with the case in plane? The critical current density \( J_{c0} \) necessary to reverse the magnetization according to the spin transfer torque is given by Slonczewski:

\[
J_{c0} = \frac{2\alpha}{g} dM_s H_{eff} \tag{1}
\]

Here, \( d \) is the film thickness, on the other hand, the meaning of the other signs has been mentioned already in the foregoing section. The value of 0.5 MA/cm² is expected as \( J_{c0} \) practicably, though details are skipped here. On the other hand, the direction of magnetization in cell tends to turn around easily by thermal energy \( k_B T \) with decreasing the size of cell (the signal disappears).

The product of magnetic anisotropy constant and the volume of magnetic substance should be large to prevent this loss of signal. The ratio of both of them \( \Delta \) is given by

\[
\Delta = \frac{K_u V}{k_B T} \tag{2}
\]

5. Development of MRAM using spin transfer torque

Magnetic memory MRAM (Magnetoresistive Random Access Memory) development has been increased every year, i.e., 1 kbit in 2000 (IBM), and 2 Mbit in 2004 (Motolola) and 16 Mbit in 2006 (Toshiba-NEC). However, power consumption is as large as about 4 mA per cell in switching of the magnetization due to the magnetic field by the conventional wiring current method. The achievement of the large capacity MRAM was considerably difficult by the cell structure. However, the reversal of magnetization was proven to be possible by spin transfer torque as mentioned above, and the development of magnetic memory (especially, spin-RAM in distinction from the conventional type) has come to be paid to attention. The spin memory can be 4 kbit (Sony) in 2005, and 2 Mbit in 2007 (Hitachi-Tohoku University). In addition, using the cell of perpendicular magnetization to plane than in plane, an excellent spin-RAM technology has been made in the research of the most recently by Toshiba. The research for the target has been studied intensely all over the world. The current trend from MRAM to spin-RAM is schematically shown in Fig. 12. Then, why is the cell geometry of vertical magnetization advantageous compared with the case in plane? The critical current density \( J_{c0} \) necessary to reverse the magnetization according to the spin transfer torque is given by Slonczewski:

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The product of magnetic anisotropy constant and the volume of magnetic substance should be large to prevent this loss of signal. The ratio of both of them \( \Delta \) is given by

\[
\Delta = \frac{K_u V}{k_B T} \tag{2}
\]
A necessary condition is $\Delta>60$ for commercial product. After all, the current density and the heat stability shown by eqs. 1 and 2 will mean which to be satisfied easily with a cell of the magnetization in a plane or out of plane. The effective magnetic fields $H_{\text{eff}}$ in eq.(1) are denoted as $H_{k_{//}}=2\pi M_s$ and $H_{k_{\perp}}=4\pi M_s$, respectively, depending on the magnetization in the plane or out of plane. $K_e$ in eq.(2) is given by $K_e=H_{k_{//}}/2$. For various $M_s$, both of $J_{c0}$ and $\Delta$ may be calculated by eqs. 1 and 2, as shown in Fig. 13. Here, it is assumed that in any case of in-plane or out of plane, the magnitudes of the quantities $g$, $d$ are the same, and 0.01 0.5, and 2 nm, respectively. The fine lines in the figure are the case of the magnetization in the plane, while the solid line is the one out of plane. Moreover, $J_{c0}$ and $\Delta$ are plotted for a few anisotropic magnetic fields. The out of plane magnetization shown by the solid line can be understood to take the margin overwhelmingly to satisfy the conditions of both $J_{c0}<0.5MA/cm^2$ and $\Delta>60$ in the range of $M_s$ between 100 and 400 emu/cm$^3$. Therefore, the development of the thin film material with 400 emu/cm$^3$ or less and perpendicular magnetic anisotropy will be important. For an example, the authors exhibit the hysteresis loop of the new perpendicular anisotropy material Mn$_2$Ga which they are studying now (fig.14) To be interesting, it has a large perpendicular anisotropy of the order of $10^5$ erg/cm$^3$, even though $M_s$ is as small as 300 emu/cm$^3$. Taking account to eq.(1) that $J_{c0}$ is proportional to $\alpha$, correspondingly, the study to lower the damping constant $\alpha$ is also an important subject.

Figure 15 shows the $M_s$ dependence of $\alpha$. Certainly, $\alpha$ tends to be increased with a decrease of $M_s$ which will be studied further.

6. Spin transfer resonance and spin torque diode effect

The magnetic field exerts a torque on magnetization, which gives rise to the magnetic moment to precess. The authors explained that the change in electronic angular momentum of $S_e$ was given by the spin polarized electron in Fig.11 of the foregoing section. It should be noted that

![Figure 14. Magnetization curve of Mn$_2$Ga single crystalline thin film.](image)

![Figure 15. Relation between damping constant $\alpha$ and saturation magnetization.](image)
each of an angular momentum or a magnetic moment is the torque operating to magnetic moment while $\gamma$ is fixed at constant, and $H$ is swept $\gamma\frac{m_0 e}{2m_0} g$, where $m_0$ is permeability of free space, $g$ is $g$ -factor (2 in spin). Under assumption that $H = 10^6$ A/m, we have $\omega = 2.2 \times 10^{11}$/sec, or about 35 GHz for frequency $\omega$. In the figure, (1) shows the torque exerted on a magnetic moment while (2) the damping of precession. In ferromagnetic resonance (FMR), the external high frequency $h = h_0 \sin \omega_0 t$ magnetic field is exerted vertically on spin, which make the precession to be continued. The resonance condition depends on both of $f$ and $H$. In general, $f$ is fixed at constant, and $H$ is swept for the resonance condition, which is unusually in case of opposite. Kieselev.et al succeeded the first time in a spectrum measurement of the high frequency output of Co/Cu/Co junction. Spin-transfer torque shown in (1) of figure takes place in this case, and the precession of spin is continued. On the other hand, the adjustment of both of the spin polarization current from F1 to F2 and the external magnetic field is necessary to look for the resonance condition in spin-transfer oscillation (STO). The application of a small radio-frequency alternating current to a magnetic tunnel junction can give rise to a direct-current (d.c.) voltage across the device when the frequency is resonant with the spin oscillations that arise from the spin-torque effect, which can be tuned by an external magnetic field. This behavior is remarkably different from that of a conventional semiconductor diode. As an intuitive understanding why the direct current voltage is caused, it only has to be thought that the resistance of the element may change by the magnetoresistance effect with the same phase of frequency of an applied current, since the voltage is a product of the current and resistance. Refer to details described in topics and commentary of the Magnetics Japan.

7. Conclusions

The contents described in secs. 4 and 5 are well-known among researchers of spintronics field with keen interest recently. There are many researches such as a basic research on the magnetic domain wall movement using spin transfer torques and the application research such as race-track memory etc. In addition, a similar research on spintronics of magnetic semiconductor instead of metals is being advanced. Furthermore for an interesting phenomenon in spintronics, the coherence length of spin (several ten Å) , amount of the spin polarization of material and etc. are important physical quantities. It is necessary to process the material by the accuracy of this level or more to catch the phenomenon adequately if we would pay attention to the physical quantity of the former. It came to be able to make an artificial material compared with the past, especially the control of the film thickness by remarkable accuracy with the formation of spintronics. However, the authors think that there are not a few studies that should be still done for microfabrication processing of the magnetic substance.

Are the thin film making technology and the microfabrication technology that raise the level by another step indispensable for an ultimate spin control? Moreover, such a technology might be requested corresponding to each interesting material. New development may be opened by its progress.

Acknowledgements

A part of this work was supported by the IT Program of the Research Revolution 2002(RR2002) by a Grant-in Aid for Scientific Research from Ministry of Education, Culture, Sports, Science and Technology of Japan and by Strategic Information and Communications R&D Promotion Programme (SCOPE) by Ministry of Internal Affairs and Communications and NEDO Spintronics nonvolatile devices project.

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spectrum measurement of the high frequency output of

Moreover, ( ) is the direction shown in figure. Using

the F2 layer is put on the magnetic field z axially. In this

the opposite direction. Fig. 16 shows the schematic of the
each of an angular momentum or a magnetic moment is

of free space,

assumption that

and

vertically on spin, which make the precession to be

resonance condition in spin-transfer oscillation (STO).

moment, the angular velocity of the precession is

frequency

from the spin-torque effect, which can be tuned by an

r torque (Schematic)

Fig.

2) M. N. Baibich, J. M. Broto, A. Fert, F. Nguyen Van

Affairs and Communications and NEDO Spintronics

Culture, Sports, Science and Technology of Japan and by

Aid for Scientific Research from Ministry of Education,

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phenomenon in spintronics, the coherence length of spin

spintronics of magnetic semiconductor instead of metals

Dau, F. Petroff, P. Eitenne, G. Creuzet, A. Friederich


71) F. Wu et al., (to be submitted)
New Research Organization
We modified our research organization as of April 1, 2010 as follows:

**Thrust 1: Bulk Metallic Glasses**
- Advanced Functional Materials Laboratory
  - Chen group, Louzguine group, Greer group, Yavari group, Hemker group
- Theory Laboratory
  - Tokuyama group

**Thrust 2: Materials Physics**
- Advanced Spectroscopy for Materials Physics Laboratory
  - Yamada group, Takahashi group, Xue group
- Interface PhysChem Laboratory
  - Kawasaki group, Ikuhara group, Lagally group, Weiss group, Teizer group, Hitosugi group
- Electronic Materials Laboratory
  - Tanigaki group
- Theory Laboratory
  - Tsukada group, Shluger group

**Thrust 3: Soft Materials**
- Interface PhysChem Laboratory
  - Itaya group, Kurihara group, Wan group
- Organosoft and Hybrid Materials Laboratory
  - Yamamoto group, Yamaguchi group, Nishi group, Adschiri group, Russell group

**Thrust 4: Device/System Construction**
- Spintronics Materials Laboratory
  - Miyazaki group
- Electronic Materials Laboratory
  - Ohmi group
- MEMS Materials Laboratory
  - Esashi group, Gessner group
- Bio Device Laboratory
  - Shimomura group, Khademhosseini group, Wu group
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- **MEMS Materials Laboratory**
  - Esashi group, Gessner group
- **Bio Device Laboratory**
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1998-2001  Research Fellow (Alexander von Humboldt Foundation), Department of Materials Science, California Institute of Technology, Pasadena, USA
2001  Senior Research Fellow, California Institute of Technology, Pasadena, USA
2001-2003  Assistant Professor (Tenure-track), Department of Materials Science, University of California, Davis, USA
2003-2007  Assistant Professor (Tenure-track), Department of Materials, ETH Zurich, Switzerland
2007-present  Full Professor and Chair of Metal Physics and Technology, Department of Materials, ETH Zurich, Switzerland
2010-present  Head of Department of Materials, ETH Zurich, Switzerland

Recognition:
✦ ETH Medal and Award for Outstanding Ph.D. Thesis, ETH Zurich, Switzerland (1998)
✦ Alexander von Humboldt Fellow (Feodor-Lynen Program), Germany (1998-2001)
✦ Visiting Faculty Position, California Institute of Technology, Pasadena, USA (2001)
✦ Masing Memorial Prize of the German Society for Materials (2005)

Current Research:
- Bulk Metallic Glasses
- Metals for Medical Applications
- Magnetism on Mesoscopic Length Scales
- Nano-Optics
- Microstructure Analysis and Modeling
Curriculum Vitae of Adjunct Professor

Taigang Nieh

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**Academic:**
B.S., Physics, National Cheng–Kung University, Taiwan, 1973
M.S., Physics, University of Washington, Seattle, Washington, 1976
Ph.D., Materials Science and Engineering, Stanford University, 1980

**Professional Experience:**
1980-1980 Postdoctoral Fellow, Stanford University
1992-2004 Senior Fellow, Lawrence Livermore National Laboratory
2004-present Professor, University of Tennessee

**Recognition:**
✦ Fellow, ASM International, 1992
✦ ISI Highly-cited Materials Scientist 2003
✦ Fellow, The Minerals, Metals & Materials Society (TMS), 2004

**Current Research:**
Bulk metallic glasses, nanocrystalline materials, high temperature alloys, superplasticity and superplastic forming, lightweight aluminum and magnesium alloys, metal-matrix composites, intermetallics, refractory metals, bioceramics, and multilayers.
Curriculum Vitae of Adjunct Associate Professor

Yuzuru TAKAMURA

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Fax: +81-761-51-1665
E-mail: takamura@jaist.ac.jp

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1990  B.S. in Metallurgy and Materials Science, The University of Tokyo, Japan
1992  M.S. in Metallurgy and Materials Science, The University of Tokyo, Japan
1995  Ph.D. in Metallurgy, The University of Tokyo, Japan

PROFESSIONAL EXPERIENCE:
1995-1996  Japan Society for the Promotion of Science Research Fellow, The University of Tokyo
1996-1999  Research Associate, The Institute of Space and Astronautical Science
1999-2003  Research Associate, Department of Materials Engineering, The University of Tokyo
2003-present  Associate Professor, Department of Materials Science,
Japan Advanced Institute of Science and Technology (JAIST)
2003-2007  PRESTO Researcher of Japan Science and Technology Agency

RECOGNITION:
✦ Small and Medium Enterprise Excellent New Technology New Product Award,
Resona Foundation and The Nikkan Kogyo Shinbun (2009)
✦ Incentive Award, Society for Chemistry and Micro-Nano Systems (2009)

CURRENT RESEARCH:
Creation of highly integrated functional biochip employing nanofabrication technique
Study on high sensitive biosensors using carbon nano tube and nano particles
Development of micro elemental analyzer using liquid electrode plasma in micro channel
Curriculum Vitae of Adjunct Associate Professor

Mitsuo UMETSU

PRIMARY AFFILIATION:
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ACADEMIC:
1995 B.S. in Engineering, Tohoku University
1997 M.S. in Engineering, Tohoku University
2000 Dr. of Engineering, Tohoku University

PROFESSIONAL EXPERIENCE:
2000-2001 JSPS Postdoctoral Fellow for Research Abroad (Leiden University, The Netherlands)
2001-2002 Research associate, Department of Biomolecular Engineering, Tohoku University
2002-2006 Research associate, Institute of Multidisciplinary Research for Advanced Materials, Tohoku University
2006-present Associate professor, Department of Biomolecular Engineering, Tohoku University
2008-present Researcher, Precursory Research for Embryonic Science and Technology (PRESTO), Japan Science and Technology Corporation (JST)
2008-present Senior Researcher, Bio Electromechanical Autonomous Nano Systems (BEANS) Laboratory

RECOGNITION:
✦ Incentive Award, Aoba Foundation for the Promotion of Engineering (2003)
✦ YABEC Award, Young Asian Biochemical Engineers’ Community (2004)
✦ Incentive Award, Intelligent Cosmos Foundation (2005)
✦ Incentive Award (Naito-Masayoshi Prize), Society of Chemical Engineers, Japan (2005)
✦ Best Talk Award, Division of Biofunctional Chemistry, The Chemical Society of Japan (2006)
✦ Best Poster Award, 2008 MRS Spring Meeting (2008)

CURRENT RESEARCH:
✦ Protein engineering design for generating organic-inorganic-biomolecule hybrid nanodevices
Announcement
Junior Faculty/Post-doctoral Positions

Tohoku University
WPI-AIMR

Effective October 1, 2007, Tohoku University created a new Research Institute, the Advanced Institute for Materials Research (AIMR), based on an initiative of the Japanese Department of Education (MEXT) for World Premier International Research Center Initiative (WPI) to bring together scientists involved in research on nano-science and technology.

In the 21st century, material science, broadly defined as the study of how complex/novel properties arise in matters/materials from the interactions of individual components, will comprise of inter-discipline collaboration. (http://www.wpi-aimr.tohoku.ac.jp)

Over the next few years, as many as one hundred new appointments at the levels of post-doctoral fellows and junior faculty will be available. All innovative researchers are welcome as active promoters of basic/applied sciences in the fields of physical metallurgy, physics, chemistry, precision mechanical engineering and electronic/informational engineering.

We are continuously looking for excellent applicants throughout the year. Please submit 1) a curriculum vitae, 2) research proposal (<3,000 words), 3) summary of previous research accomplishments (<2,000 words), 4) copies of 5 significant publications, and 5) 2 letters of recommendation by email to: wpi-office@bureau.tohoku.ac.jp

All files must be submitted electronically in pdf or Word format.

Applications from, or nominations of, women and minority candidates are encouraged. Tohoku University WPI-AIMR is an affirmative action / equal opportunity employer.
Junior Faculty/Post-doctoral Positions

Tohoku University
WPI-AIMR

Effective October 1, 2007, Tohoku University created a new Research Institute, the Advanced Institute for Materials Research (AIMR), based on an initiative of the Japanese Department of Education (MEXT) for World Premier International Research Center Initiative (WPI) to bring together scientists involved in research on nano-science and technology.

In the 21st century, material science, broadly defined as the study of how complex/novel properties arise in matters/materials from the interactions of individual components, will comprise of inter-discipline collaboration.

Over the next few years, as many as one hundred new appointments at the levels of post-doctoral fellows and junior faculty will be available. All innovative researchers are welcome as active promoters of basic/applied sciences in the fields of physical metallurgy, physics, chemistry, precision mechanical engineering and electronic/informational engineering.

We are continuously looking for excellent applicants throughout the year. Please submit
1) a curriculum vitae,
2) research proposal (<3,000 words),
3) summary of previous research accomplishments (<2,000 words),
4) copies of 5 significant publications, and
5) 2 letters of recommendation
by email to:

wpi-office@bureau.tohoku.ac.jp

All files must be submitted electronically in pdf or Word format.

Applications from, or nominations of, women and minority candidates are encouraged. Tohoku University WPI-AIMR is an affirmative action/equal opportunity employer.
Graduate Student Scholarship in Materials Science/Engineering

WPI-AIMR
Graduate Student Scholarship

Effective October 1, 2007, Tohoku University created a new Research Institute, the Advanced Institute for Materials Research (AIMR), based on an initiative of the Japanese Department of Education (MEXT) for World Premier International Research Center Initiative (WPI) to bring together scientists involved in research on nano-science and technology.

In the 21st century, material science, broadly defined as the study of how complex/novel properties arise in matters/materials from the interactions of individual components, will become an essential and most important research topic. TU WPI-AIMR is now looking for young motivated Ph.D. graduate student candidates in the fields of physical metallurgy, physics, chemistry, mechanical engineering and electronic/informational technology. All innovative M.S. students are welcome as active promoters of basic/applied sciences in these fields.

Applications are continuously screened throughout the year. Please submit
1) a curriculum vitae,
2) research proposal (<1,000 words),
3) 2 letters of recommendation,
4) by email to:
   wpi-office@bureau.tohoku.ac.jp

All files must be submitted electronically in pdf or Word format.
WPI-AIMR
Workshop Guideline

Tohoku University’s new Research Institute, the Advanced Institute for Materials Research (WPI-AIMR) solicits several applications per year for International Workshops in the field of “broadly defined Materials Science.”

Guidelines:
1) Organizers
Qualified research staff of academic institutions and public or private research establishments can submit the application for an international workshop to be held at WPI-AIMR or its Satellite branches, jointly with the WPI-AIMR principal investigator(s) whose research interest overlaps with the scope of the workshop.

2) Financial support
Under normal circumstances, WPI-IMR supports up to 2/3 of the workshop budget, while the organizer is expected to cover the rest.

3) deadline
The application must be received at least four months in advance to:

wpi-office@bureau.tohoku.ac.jp

All files must be submitted electronically in pdf or Word format.
Appendix
Laboratories Visit
Ikuhara Group (Materials Physics)
Itaya Group (Soft Materials)

Scanning Tunneling Microscopy and Non-Contact Atomic Force Microscopy under Ultra High Vacuum
Low Energy Electron Diffraction System

Group Photo
Khademhosseini Group (Device/System)
Inverted Zeiss Axio Observer.Z1 Microscope

Group Photo
Kurihara Group (Soft Materials)

Surface Forces Apparatus and Resonance Shear Measurement System

Langmuir-Blodgett Trough

Surface Forces Apparatus
Group Photo
Miyazaki Group (Device/System)

Time-Resolved Magneto-Optical Kerr Effect Measurement System for Spin Dynamics Investigation

Ultra-High Vacuum Deposition System for Magneto-Organic Hybrid Junctions

X-ray Diffraction System for Ultrathin Films

Magnetic Tunnel Junctions Deposition System for Gigabits Class Spin Memory
Cryogenic Hall Effect Measurement System under High Magnetic Field

Group Photo

X - 10
Nishi Group (Soft Materials)

Atomic Force Microscope
Tsukada Group (Device/System)

High Performance Computing Systems installed in the Server Room of Tsukada Group
Wu Group (Device/System)

Inverted Zeiss Axio Observer.Z1 Microscope

Group Photo

X - 15
Yamada Group (Materials Physics)

Two Mirrors Floating Zone Furnace

Vacuum Electric Furnace

Laser Heated Floating Zone Furnace

Four Mirrors Floating Zone Furnace
Group Photo

HERMES: Neutron Powder Diffractometer for High Efficiency and High Resolution Measurements (Japan Atomic Energy Agency (JAEA) in Ibaraki prefecture)

AKANE: Triple-Axis Neutron Spectrometer (JAEA)
## DIRECTORY

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Cover: Snapshot of long-lived spatial heterogeneities, projected onto a x-y plane, obtained by numerically solving the Tokuyama nonlinear stochastic equation for density fluctuations in a colloidal suspension at a liquid state with volume fraction $f=0.466$ and a supercooled-liquid state with $f=0.566$, where the relaxation time is of order $10^3$ and $6\times10^4$, respectively. (Michio Tokuyama, WPI-AIMR)
Dynamics of spatial heterogeneities in a suspension of colloids

$t = 2.0 \times 10^8 t_e$

$t = 2.01 \times 10^8 t_e$

$t = 2.6 \times 10^7 t_e$