

# **Report on Visit to Ruhr-University Bochum by International Training Program**

## **Nagoya University Shingo Kondo**

As a long-term placement program of ITP, I studied in the Prof. Dr. Keudell group in Ruhr University Bochum of Germany for 61 days from January 7, 2009 up to March 8, 2009. This is my report of the days.

My research theme of Ph.D. course is the elucidation of initial growth process of carbon nanowalls (CNWs). On the other hand, I studied in Ruhr University about the analysis of growth process of carbon films synthesized by plasma-enhanced chemical vapor deposition. Hence, both themes link strongly each other, and the study in Ruhr University will be helpful for my PhD thesis. CNWs are expected to be applied for next generation semiconductor devices, fuel cells, etc. due to their unique nanostructures and great electrical properties. However in present, we cannot control their morphology and characteristics accurately according as each application because the growth mechanism has not been clarified. That is the critical barrier against realizing the applications. My Ph.D. project focuses on the influence of ions and radicals in plasma on CNWs growth and atomic structures in CNWs, and then final approach is the elucidation of the growth process of CNWs. In Prof. Keudell group in Ruhr University, I tried to measure the film property by spectroscopic ellipsometry in real time.

Ellipsometry has polarized light, and it is one of the very sensitive measurement technologies for analysis of films, surfaces of films, and minute structures of materials, that can be obtained by the measurement of intensity changing and phase changing of polarized light before and after the reflection on the film. In some cases of films deposition and etching processes, film

characteristics, reactive speed, and reaction process can be changed during the processes, hence tools in real time are extremely useful. Also in the case of my researching material, CNWs, it was confirmed that the deposition rate and refractive index were changed in a few minutes after the beginning, which makes us more difficult to understand the growth mechanism of them.

The research and the life in the ITP in Germany are following. On the departing day, January 7 on Wednesday, I went to Central Japan International Airport near Nagoya in the early morning, and then I arrived at Dusseldorf Airport in the night on the same day via Narita International Airport and Frankfurt International Airport. Next, I took a rapid train from Dusseldorf Airport to Bochum for 40 min, which goes every 1 hour. Prof. Czarnetzki scheduled to pick me up at Bochum Hauptbahnhof (Bochum Main Station), and we could see each other without delay. During the week I arrived at Germany, a serious cold wave attacked Germany, which was the strongest for last 30 years. Even maximum temperatures were below 0 °C in that week. The lowest temperature in the arrival day was -12 °C, so I felt it was the real winter. In the heavy snow weather, we could get to the student dormitory by the special managements of Prof. Czarnetzki, and registered about the room. However, when I enter my room at the first time, I couldn't find a blanket that was the serious problem for me because of the cold weather. At last, Prof. Czarnetzki brought his sleeping bag from his house, and lent to me, so I could start sleeping at the first night.

In the first week, I tried to communicate with the student actively, and asked them to guide me to the

laboratory in order to get used to my life here. Fortunately, I could have my desk in the office, and a Ph.D. students and two master students were in the same office as me. I do not hate to communicate with people I met at the first time, hence it did not take so much time to become friends each other very much. The first impression for them was “honest” and “earnest”, because their mentions in English were very smart with good grammar, and they always concentrated their deskwork and experiments .

Students in Prof. Keudell's group and I went lunch to a university cafeteria called “MENZA” every day. We chose main dished and drinks, and then cashed, which was the same system as almost universities in Japan. Japonica rice does not exist in Germany, and Indica rice is the only rice we could eat. However I think Indica rice is not suitable for almost all of the Japanese people, so I often eat bread, potato or pasta instead of rice. Before staying, I imagined that German people usually eat sausage or meet, but in real, pasta and pizza are more popular.

In the second week, a post doctor taught me how to operate the spectroscopic ellipsometer and how to analyze carbon films by ellipsometry easily. I studied ellipsometry in order to master it. Ellipsometry analysis is very easy and it takes only a short time, however it springs just “ $\phi$ ” and “ $\Delta$ ” as law datum, which do not have the meanings of material properties. Ellipsometry requires to be constructed a fitting model on the basis of the characteristics according as materials in advance. Thickness, refractive index, etc. are calculated and estimated by the computer from measured “ $\phi$ ” and “ $\Delta$ ”. As a result, this method is very sensitive and comprehensive. However, human's know-how and skills in model construction and the fitting are also essential in this method. In this time, I learned weak points of a single-wavelength ellipsometer and the limitation of comprehension. In the same week, I taught a master student about the basic knowledge of optical emission spectroscopy and how

to use the software for it, and I helped measuring in a capacitively coupled plasma chamber for Silicon related material formation. The model of spectroscopy was the same as one I used when I was an undergraduate student, and I had know-how about the plasma cleaning in the chamber for carbon related material formation. So I can give him some suggestions. From this week I started to join weekly meetings in the group.

In the third week, I started my experiments. My apparatus had not used for a long time, so I spend all of time for reconstruction, for example gas cylinders, gas lines, preventing from the leak, mounting the single-wavelength ellipsometer, light angle adjustment, etc. In this week, I saw again a Ph.D. student who stayed my group in Nagoya University from April to May, 2008. We really enjoyed talking in that day.

In the fourth day, I could ignite the plasma. The reactive chamber was evacuated by an oil-sealed rotary pump and a turbo molecular pump, and then  $C_4F_8$  gas was introduced into the chamber. Capacitively coupled plasma was generated by applying 13.56 MHz power source to the upper electrode. During processes, single wavelength (632 nm) ellipsometry was carried out, and the thickness and refractive index of the films were *in-situ* analyzed. We confirmed the deposition of fluorocarbon film at the growth rate of a few hundred nm/min at a few ten Pa. As the gas pressure increased, the plasma was localized more around the upper electrode, as a result, the deposition rate of a fluorocarbon film on a Si substrate which was put on the lower electrode decreased. Samples I made were observed by an atomic force microscope by an undergraduate student, and I discussed about these results with her for the long time.

From fifth to seventh week, small amounts of Ar gas or  $H_2$  gas were diluted with  $C_4F_8$  gas. When the total gas flow rate was constant and the Ar/ $C_4F_8$  ratio was changed, the deposition rate had the maximum point at a certain ratio. This is because

the amount of carbon generated from  $C_4F_8$  gas and the electron density were changed as a function of the  $C_4F_8/Ar$  gas mixture ratio. On the other hand, when  $H_2$  gas was diluted, the maximum deposition rate was obtained at 1:1 of  $C_4F_8:H_2$ . I also performed optical emission spectroscopy (OES) in the plasma. It was confirmed that the intensity of H atom suddenly increased at the  $H_2$  gas flow ratio of more than 50%. As these results,  $CHF_2$  radical and CHF radical contributed to increasing growth rates as well as film formation.

Refractive index ( $n$ ) is also measured in all samples including Ar gas dilution and  $H_2$  gas dilution, and those  $n$  are all approximately 1.4.

Generally, it is difficult to analyze the morphology of materials which are composed of a large amount of void such as CNWs using ellipsometry technique. I tried to introduce a multi-layer model and learned available fitting methods in this term. These experiments and analysis will be also helpful for us to realize to make a model suitable for CNWs.

In the last week, I gave a presentation in the lecture room. It was about 30 minutes for talk and about 20 minutes for discussion. Not only students in Prof. Keudell group but also students and researchers I met in this stay joined my presentation. So that was bigger than I expected. Somehow I could succeed in my presentation. The contents were Japan's introduction and research project in my Ph.D. course.

My experiments in Ruhr University were done by me and an German undergraduate student who has never experienced in plasma science and technology. Hence, I have not only to learn but also to teach the beginner student especially focusing on our safety in English. In addition, I did not manage very well at the first, because the system and the environment in the laboratory there were absolutely deferent from my laboratory in Japan. However, those trainings got very good international opportunities for me and the student for our future works.

This was the first visit to Germany for me. I

learned deeply not only plasma science and technology but also German life, culture, and character of national people. German university students are extremely friendly for me. They bantered with me in English, and they explained for me about the menu in a school cafeteria every day. In addition, all group members with me sometimes watched movies which was Japanese one or was focused on the global environment, etc, and they also often held birth parties and farewell parties.

University students in German are interested in Japan very much, so they asked me everything about Japan. I also asked them a lot about Germany, as a result, this situation became an ideal international communication. A student in Germany who stayed in Nagoya University as same style as ITP started learning Japanese language just after he came back to Germany. He invited me to join Japanese language class. About 20 students from Germany and China attended and studied Japanese. They were strongly interested in Japanese culture, and their knowledge about Japanese anime and music might be higher than mine. The contents in the class were grammar, conversation, communication, words. It seemed to be difficult for them to master the conjugation in verb and adjective-verb. The teacher was Japanese who was the same age as me. She also plays as a bridge between young people in Germany and Japan. I hope young international communication will be more and more deeply in the future through this ITP program.

Especially, Ph.D. students are very active. When the students attended the interview for getting budget from the government in the next few years, they discuss about their researches, the future vision of plasma, the revolution of the education system, etc. with reviewers, which was very impressive for me. They also contrived the plasma lecture for people who have never studied, seen, or even heard "plasma". One of the contents was that they showed He plasma in the flask in a microwave oven, and all people seemed to be satisfied.

In Prof. Keudell group, nobody could speak Japanese, so I had to use English and a little Germany all of the day. I continued to use in ordinary conversation, questions and answer, discussion, etc. so I maybe improve my English very much. In the English education in Japan, reading and writing are focused on, and speaking and listening are shortage. That is why I could improve it.

As mentioned above, I am able to apply to my Ph.D. project in the future and Japan-German international communication through the research about carbon and the communication with the group members. Hence, the two-month stay in Germany got the invaluable treasure for me.

Finally, I deeply appropriate professors in Nagoya University and Ruhr University and staffs in Nagoya University who managed ITP.