Report on Visit to Sungkyunkwan University in Korea by International Training Program

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I report that I participated in International Training Program (ITP) and stayed at Center for Advanced Plasma Surface Technology (CAPST) of Sungkyunkwan University in Korea for 60 days from October 19, 2012 to December 17, 2012.

[About host institution]

Sungkyunkwan University has two campuses in Seoul city and Suwon city. I visited Center for Advanced Plasma Surface Technology (CAPST) in Suwon campus. In CAPST, some advanced studies in various fields such as the development and evaluation of new functionality film materials made with plasma and plasma diagnostics are performed. I stayed at the laboratory of Professor Han who is director of CAPST and researched.

The laboratory is mainly researching plasma enhanced chemical vapor deposition (PECVD) and magnetron sputtering and has so many experimental devices and film evaluation devices. So students are doing most-advanced research there.

[Research]

I discussed Professor Han, and we decided to research improvement of ultra-hydrophobic film by PECVD. I have studied generation of microcrystalline silicon by PECVD in Japan, so I have no knowledge of hydrophobic films. However, the theme looked interesting so I began to research.

Hydrophobic films are used everywhere. For example, glass of cars, liquid crystal display of smartphone and tablet PC, and so on.

Hydrophobicity is a phenomenon on solid surface among solid, liquid and gas phases. Young defined hydrophobicity with following expressions.

$$\gamma_{SV} = \gamma_{SL} + \gamma_{LV} \cos\theta \quad \cdot \quad \cdot \quad (1)$$

 γ_{SV} ...Tension between solid and gas

 γ_{SL} ...Tension between solid and liquid

 γ_{LV} ...Tension between liquid and gas

 θ ...Contact angle

Then, $\theta < 90^{\circ}$ is hydrophilicity, $90^{\circ} < \theta < 110^{\circ}$ is hydrophobicity, $110^{\circ} < \theta < 150^{\circ}$ is high hydrophobicity, $\theta > 150^{\circ}$ is ultra-hydrophobicity. Hydrophobicity mainly depends on surface energies and surface morphologies. More than 115 degrees contact angle cannot be obtained with smooth surface.



Fig.1 Definition of contact angle

Surface nanostructure is needed to get higher contact angle.

With nanostructure, contact angle is expressed following Cassie's expression.

 $\cos\theta_f = A_1 \cos\theta_1 + A_2 \cos\theta_2 \dots (2)$

 $\theta_1, \theta_2...$ Contact angle of substance 1,2 with smooth surface

 A_1, A_2 ...Fraction of substance 1,2

If substance 2 is air, $\theta_2 = 180^{\circ}$. If A_1 becomes smaller, θ_1 becomes larger, so contact angle becomes larger. The phenomenon that surface with nanostructure never get wet is

called lotus effect, and it is intensively studied to apply to industrial application. I experimented in hydrophobic films with Mr. Joon S. Lee who is a master course student. Wear resistance, transparence and mechanical property are required for good hydrophobic films. SiO_x thin films made by PECVD have a good adhesion, and film properties can be changed easily by changing plasma parameters. And lower cost substrates can be used due to low temperature process.

We used RF CCP CVD equipment for this experiment as shown Fig.2. Top and bottom circle electrodes were connected to RF power supply and matching box, respectively. Carrier gases and precursors were introduced to the chamber through shower head equipped with top electrode. Glass substrates were used for deposition at room temperature.

At first, glass substrate was set on the bottom electrode and done surface preparation by O_2 plasma made by bottom electrode. This surface preparation made better adhesion of SiO_x films. SiO_x films were deposited with OMCTS precursor. OMCTS was bubbled by O_2 carrier gas and introduced to the chamber, and plasma was produced by top and bottom electrodes. Then, Ar plasma was produced by bottom electrode. A lot of high energy ion hit the surface and made nanostructure. After that, HMDS was bubbled by H₂ and C-H bonds were attached to the SiO_x surface. Water contact angles of films were measured. Nanostructures of film surfaces were also investigated by AFM. Moreover, some durability tests were evaluated.



Fig.2 Experimental setup



Fig.3 RF power dependence of contact angle

Fig.3 shows RF power dependence of contact angle. Black dots show contact angles after deposition. Red dots show contact angles after rubbing test. With low RF power, contact angle became smaller after rubbing test. Surface nanostructure might be lost by rubbing because there was bad adhesion between SiO_x and hydrophobic film. Inner structure changed and film mechanical property improved during 60~80 W.



Fig.4 Ar treatment time dependence of contact angle





Fig.4 shows contact angles of Ar treatment time dependence. There were not much differences of contact angle after deposition. 5 minutes Ar treatment made a good durability, so good contact angle was obtained after rubbing test. Better nanostructure was made by 5 minutes treatment.

Fig.5 shows some durability tests. Experimental conditions are in Table.1.

Condition 1	Hydrophobic film
Condition 2	Hydrophobic film+SiO _x
Condition 3	Hydrophobic film+SiO _x +Ar treatment
Condition 4	Hydrophobic film $(C_2F_6$ precursor)
	+Ar treatment

Table.1 Experimental conditions

Contact angle of 160 degree was obtained by Condition 1. However film lost durability after durability tests. This is thought to result from poor hardness of hydrophobic film. Contact angle did not be so bad after durability test in Condition 3 because of good adhesion between SiO_x and hydrophobic film. Fig. 6 shows surface picture of the film of Condition 3. Nanostructure was observed on the surface. Height from valley to peak was about 100 nm.

[Laboratory life]

I usually went to the laboratory at 9:30 a.m. Some students were already in the laboratory, they normally did desk work. At first, I had no knowledge about hydrophobic films, so I studied to read some research papers Mr. Lee gave me.

We usually went to convenience store or ordered delivery service. Prices of lunch bags were under 250 yen, and we could eat lunch at dining hall for officials under 400 yen. We could use delivery service cheaply and enjoyed some Korean food very much.

I did experiment with Mr. Lee and Mr. Kim in the afternoon. When they taught me how to use experiment systems, I was surprise at their fluent English. I tried to ask them, but I didn't say what I wanted to ask well in English. Korean students are used to make slides in English, so I felt the difference between Japanese students and Korean students.

We often went to nabe restaurant. When I ate kimuchi-chige for the first time in Korea, it was too hot to stop my sweat. It was impressive that Mr. Kim ate same kimuchi-chige and said "This is normal revel, not too spicy." I usually went back home at 7~8 p.m. Some students were still in the laboratory at the time.

[Summary]

I had a lot of experience in Korea for 60 days. I couldn't have such experience in Japan. I couldn't read Korean

character and speak Korean language, so I had a lot of trouble in my life. However, every time I solve problems, I became stronger mentally. Foreign culture gave me a lot of interests, and I could think many things globally.

In the laboratory, all of students and staffs were very kind to me, so I could stay and experiment in comfort. Discussing in English was good experience to me. They could read and write something and in English easily, I was very surprised. I also want to speak English at will, so I will study English a lot. I got stimulated by their orientation s to their researches, I'd like to do my experiment more sincerely.

Finally, I really appreciate ITP officials giving me such a great opportunity. I really appreciate Professor Han and all the students and staffs supporting me the stay in Korea.