The 50th William Blum Lecture
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Alloy Plating by Heating Stacked Single Layers and the Possibility of its Application in the Future

by
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Recipient of the 2012 William Blum
NASF Scientific Achievement Award

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ABSTRACT

In recent years, the plating industry has had the serious challenge of severe environmental regulations. Various "hazardous" metals, e.g., lead, cadmium, etc., have been the target for regulations one by one. As a result, a pessimistic outlook that any metal could be classified into a regulated one by laws has appeared. However, any element, or materials can present both hazardous and benign situations. In many cases, the so called "hazardous" metals would be toxic, when they exist as an ion, but not as a solid metal. Therefore, alloy plating can be one of the best solutions to avoid disuse by the environmental regulations with minor changes for production. Alloy Plating has been conventionally carried out by coelectrodeposition in solutions. On the other hand, the author has proposed a unique method of alloy coating through the combination of heat treatment and multiple plating. In this lecture, the author shows the results and the possibilities for application in the future.

Introduction

Alloy plating continues to have potential for practical application. Conventionally, it can produce unique properties such as corrosion characteristics, wear resistance, color tone, etc., that cannot otherwise be realized with any single element film. However, recent trends need more innovative developments to solve the practical problems through the utilization of alloy films.

For example, environmental problems seem to hamper the usage of some useful metals (from the functional standpoint at least) at this point. Actually, the use of alloy films can alleviate the environmental burden to some extent. In addition, many emerging needs, such as electromagnetic or optoelectronic properties can appear on the material surface by alloy film formation.

Usually, the alloy film is produced by coelectrodeposition from aqueous solution. However, the author has proposed the combination of surface treatments and heat treatments to produce alloy films in numerous studies.[1-25] In this article, I will describe the alloy film formation technique with which my colleagues and I have tried to produce unique materials and describe the characteristics, meaning and the future scope of this technology. Hopefully, this lecture will allow this unique method and technology to produce alloy films and empower many electroplaters and surface finishers to launch new applications of alloy films.

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Principles and characteristics of the HSSL process

The proposed alloy film formation process is usually called the Heating Stacked Single Layers Process (HSSL process). As shown in Fig. 1, alloy deposits have been produced to date through coelectrodeposition from aqueous solutions. In this process, the alloying phenomenon occurs during the deposition process. From the standpoint of procedure, the conventional process is composed of a single step.

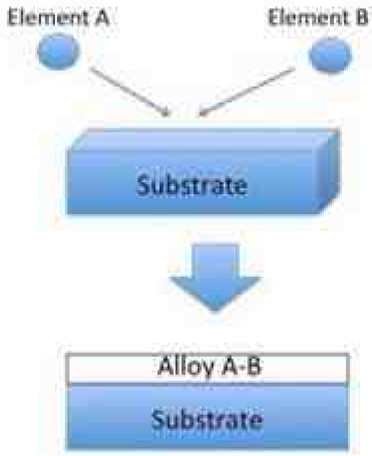

Figure 1 - Schematic illustration for conventional alloy plating.

On the other hand, HSSL process can be divided into two steps. During the first step, the single layers are stacked onto the substrate surface layer by layer. The surface coating method can be chosen freely by the producer in the wide range from plating to cladding or painting. Heat is then added to alloy the stacked layers by mutual interdiffusion on the substrate.

Figure 2 shows the schematic illustration for the HSSL process. Although the process is composed of multiple steps, the process as a whole is not complicated and alloy formation is controlled by the heat treatment process which can be operated more easily than the electrodeposition process.

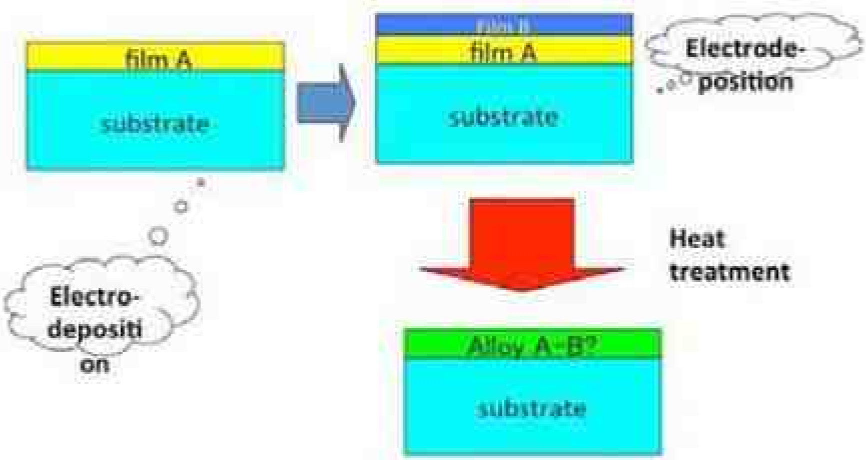

Figure 2 - Schematic illustration for the HSSL process.

Table 1 compares the merits and demerits of conventional alloy plating versus the HSSL process. The most positive characteristic for the HSSL process is its wide selectability in choosing the appropriate method of film formation. This means that one can avoid environmental regulatory issues. Such a state of affairs is desirable for today's plating industries, which have to struggle with continually emerging environmental regulations. In addition, the multi-step process might not be as complicated, as most plating plants already have thermal treatment facilities to produce stacked single layers. For a number of reasons, many plating processes require other underlayers. From this standpoint, it is highly likely that electroplaters can carry out the HSSL process without significant modification of their facilities.

Table 1 - Advantages and disadvantages of the HSSL process.

Advantages	Disadvantages
Environmental friendliness Easy to operate	Multistep treatments

Representative examples of HSSL processes and their applicability

Tin-nickel and tin-zinc alloy plating

Although chromium plating has been effective and highly functional for many purposes, the environmentally harmful hexavalent chromium ion, one of the ionic states for the element, has encouraged the need for substitutes. To that end, a number of substitutes have already reached the market, including tin-nickel and tin-cobalt plating, among others. Regarding the former, the author has investigated the alloying phenomenon in detail. Figure 3 shows a schematic illustration of the process.

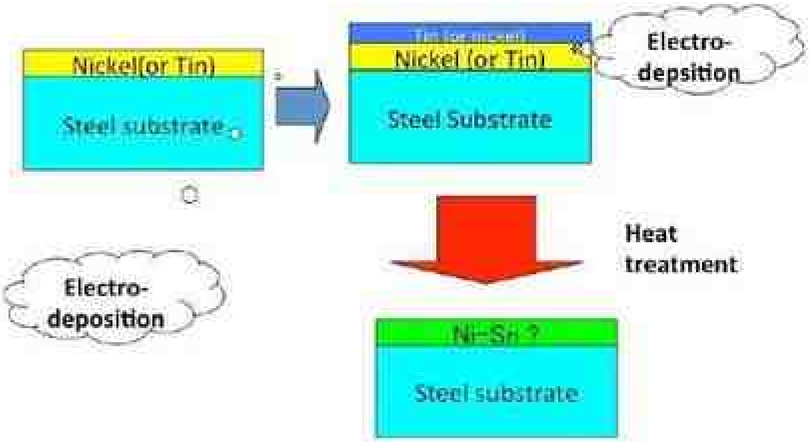

Figure 3 - Schematic explanation of an HSSL process used to produce a Ni-Sn alloy film.

There are two ways to produce stacked single layers of tin and nickel. The first is to produce a tin layer followed by a nickel layer and the second is to produce a nickel layer followed by a tin layer. When a fluoboric acid bath was used for tin and a Watts bath was used for nickel, tin could be dissolved during the subsequent electrodeposition of nickel from the Watts bath. As a result, the appropriate tin and nickel stacked layers could not form. If those inappropriately stacked layers had been heat treated, the intermetallic compound layers would not have been produced. Therefore, nickel was deposited first and then tin could be deposited on the nickel.

Since the melting point of tin layer is about 230°C, the subsequent heat treatment was carried out just above or below the melting point of tin. In that temperature range, the tin layer, the lower melting point metal phase, was melted, while the nickel layer, the higher melting point metal phase, remained in the solid state. Since the melting point of nickel is much higher (1455°C) and would require relatively elaborate furnaces for the heat treatment, the relatively low temperature heat treatment would be desirable from a practical standpoint. In such a case, there are two possibilities - the heat treatment temperature held above the melting point of tin or below it. In the former case, the tin layer would be melted, while nickel layer would be solid. In the latter case, both layers would be solid. The two cases have their own different mechanisms to form an alloy film. In both cases, the mutual diffusion between the two stacked phases on the material surface is the key mechanism. However, the solid-liquid interaction would occur in the former, while the solid-solid one would occur in the latter. In the former, the diffusion rate would be very high, since the solid nickel would diffuse very easily into the liquid tin phase or vice versa. As a result, the former case would take several minutes for alloy formation to result, while the latter case would take several days or weeks. In any case, some intermetallic compound layers of tin and nickel would form through mutual diffusion.

One may ask if the liquid tin phase would be lost during the heat treatment process. However, we confirmed that such a phenomenon never occurred, meaning that the mutual diffusion that formed the intermetallic compound layers occurred at very high rate. Figure 4 shows the schematic illustration for the alloy film formation in that case.

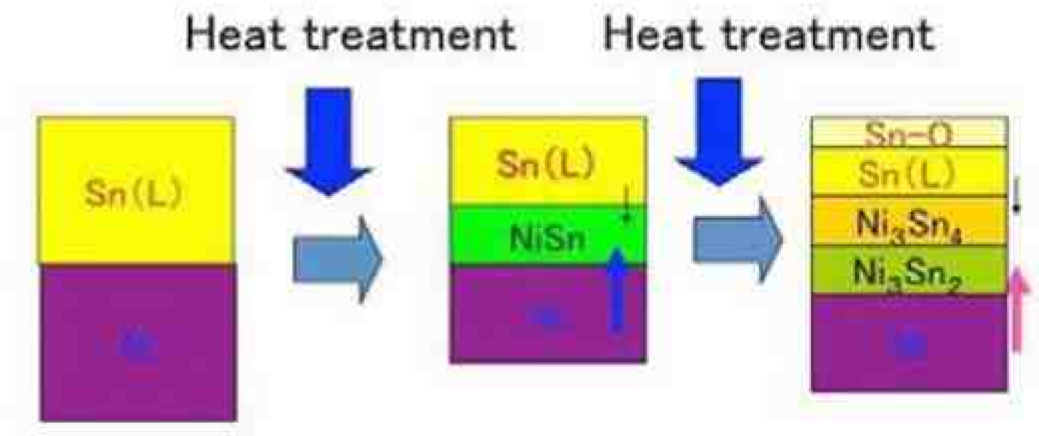

Figure 4 - Schematic illustration for the formation of intermetallic compounds between tin and nickel.

The film of intermetallic compounds produced between the tin and nickel layers has high corrosion resistance and relatively high hardness. The polarization curve of the alloy film specimen measured in 3% NaCl solution showed that no corrosive dissolution occurred in the usual potential range for a steel substrate and that it had very high corrosion resistance. On the other hand, Fig. 5 shows the Vickers hardness on the surface of specimens measured by a Micro Vickers hardness testing machine. The hardness depended on the heat treatment temperature. When the specimen was heated at 550°C, the maximum hardness was obtained at the vicinity of surface of the specimen (750_{VHN}). The color tone also depended on the heat treatment temperature and usually it generally was a dark gold or yellow ochre. It was noteworthy that the color tone was different from that obtained by the conventional alloy electrodeposition process. In that case, it was pink tinted.

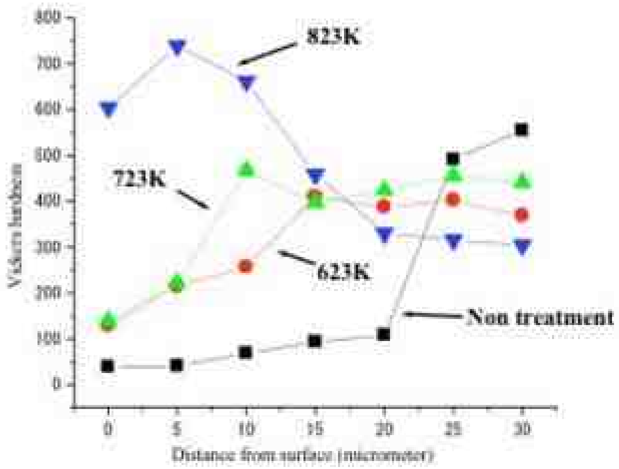

Figure 5 - Vickers hardness for a tin-nickel alloy film produced by the HSSL process

A tin-zinc alloy film could be formed in the same way. The alloy film could be applied as a substitute for cadmium plating, which has been used for aircraft parts. During World War II, cadmium was in short supply for a number of reasons. Around that time, tin-zinc alloys were used as a substitute. However, today cadmium substitutes are considered from an environmental standpoint. The phase diagram of the tin-zinc system clearly shows a eutectic. To produce high corrosion resistance films, a zinc-rich alloy film would be desirable (more than 90% zinc, conventionally). Also in this system, the order of stacked single layers are arbitrary (zinc as an underlayer and tin as an overlayer, and vice versa). As a concrete example, we produced stacked single layers composed of tin films from an acid tetrafluoboric tin bath and zinc films from a zinc sulfate bath and heat-treated them at 350-400°C. X-ray diffraction analysis showed that the alloy phases were mixed crystals of zinc and tin. For this process, versatile combinations of heat treatments and plating processes would be possible. For example, the author used a laser treatment process with some plating methods to shorten the process time.

Antibacterial alloy films.

Using the principles for the combination of alternating plating for the formation of single layers and the subsequent heat treatment, many alloy films are possible to produce. My colleagues and I have investigated many elements and combinations thereof. However, it is essential that the application be considered from the practical viewpoint. For the studies of the HSSL process, we began with tin alloy films, as described above.

Tin plating has been used in the food processing industry. Considering this, we found a second focus on the antibacterial effects and worked to produce an antibacterial effect in tin plating by adding other antibacterial elements through the HSSL process. Generally speaking, tin shows little antibacterial effects. Table 2 shows the test results of the antibacterial effect for tin plated steels.

The test method, commonly called the Film Covering Method (ISO 22196), provides useful information needed for practical applications. The specimens are put in a plastic Petri dish, while the bacterial solution is prepared as follows. The bacteria are incubated in 10 mL of a nutrient broth for 24 hr at 35°C, then diluted two-thousand fold with sterilized water and established as a bacterial solution. The diluted bacterial solution is applied to the specimen (16 µL/cm) and a polymer film was then laid over the solution. The sample was kept in an incubator for 24 hr at 35°C.

After the incubation, a solution of 10 mL of sterilized water containing 200 µL of Tween® 80 (a nonionic surfactant and emulsifier) was introduced into the plastic Petri dish and the bacteria attached to the specimen and polyethylene film were washed into the aqueous solution. To determine the number of viable cells, serial decimal dilutions of the cell suspension were made, a 0.1 mL portion of which was uniformly spread on an agar medium. The plate was incubated at 35°C for 24 hr and the colonies formed were counted. The viable cell count was represented as colony forming unit per milliliter (CFU/mL). The final colony formation number was measured to evaluate the antibacterial properties.[27] As shown in Table 2, the number of bacteria was not reduced after 24 hr had passed. Rather, it increased slightly. At minimum, a decrease of bacterial numbers on the order of 10 to the second power would be required to confirm the antibacterial effect.

Table 2 - Effect of the presence of tin-plated steel on bacterial count.[26]

Time, hr	Specimens	E-coli (*Eschericia coli* ATCC25032)
0	Control	9.5×10^4/plate
24	Control	2.04×10^6/plate
	Tin-plated steel	7.51×10^6/plate

Figure 6 shows a schematic illustration for making Sn-Cu alloy films. In the example, the copper film was prepared by electroplating. The tin film was produced by a magnetron sputtering process. However, any combination (*e.g.*, even if all films were produced by electroplating) would lead to the same result. The heat treatment temperature was set to 300°C, just above the melting temperature of tin.

The tin layer was melted after the specimens were heated to the temperature. The mutual diffusion rate then increased significantly to form intermetallic compounds of tin and copper. When the film thickness of the tin layer was relatively small (5 μm tin and 10 μm copper), the main intermetallic compound layer was Cu_6Sn_5, while the thicker film specimen contained Cu_3Sn. Table 3 shows the antibacterial effects for various tin-copper plated films by the film covering method. As Table 3 shows, tin-copper alloy films clearly showed strong antibacterial effects.

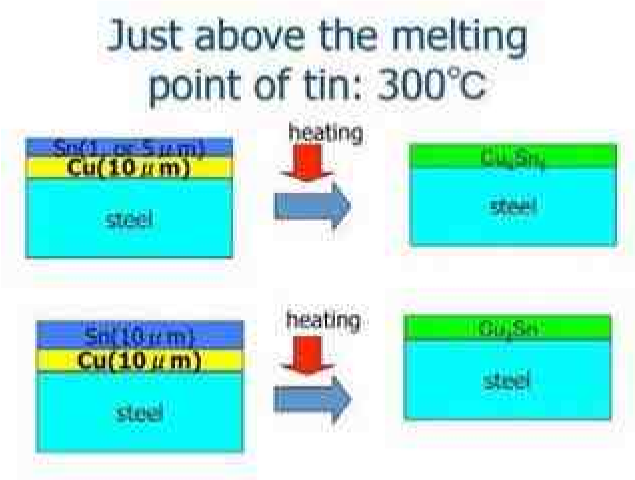

Figure 6 - Schematic illustration for producing a tin-copper alloy film by HSSL.[26]

Table 3 - Antibacterial effects of tin-copper plated films by the Film Covering Method.[26]

Time, hr	Specimens	*Escherichia coli*	*Klebsiella pneumonia ST101*	*Staphylococcus aureus 209P*
0	Control	9.5×10^4/plate	1.71×10^5/plate	1.09×10^5/plate
24	Control	2.04×10^6/plate	6.76×10^6/plate	8.30×10^5/plate
	Sn-Cu Plating	0	0	0

My colleagues and I confirmed that the antibacterial effect of intermetallic compounds for tin-based films was also observed by adding silver[17,28-30] and palladium[31,32] to tin films. However, from a practical viewpoint, silver seems to be the most important in addition to copper. Actually, most of the patents in Japan have been concentrated on silver and copper compounds, while titanium oxides were another option due to their photocatalytic effect. Many other elements may work equally well. This might be a good future research topic, since surface finishing for antibacterial effects will be very important for aging societies in advanced countries.

Applicability and limitations

When one wants to use HSSL process, are any alloy systems available, so long as the stacked single layers can be formed? The answer is obviously negative. However, we have to know in advance how we could tell the possible cases from impossible ones.

One of the ways to wean out the practical possibilities lies in the use of phase diagrams. For example, the nickel-tin phase diagram clearly shows the intermetallic compound phases, as shown in Fig. 7. On the other hand, the silver-nickel system would not lead to the formation of alloy films, based on the phase diagram for the two elements shown in Fig. 8. When the phase diagram shows some intermetallic compounds, alloying is possible. Therefore, the phase diagram is a guideline for the HSSL process.

On the other hand, the simulation software that Dr. Yoshitake at NIMS developed on the NIMS website may work well to predict the effectiveness of alloying for practical purposes.[33] The software was originally developed to predict the experimental results in advance, *i.e.*, whether a substrate element A were to diffuse into the film element B to the top of the film or not.[34-36] Figure 9 shows the possible cases for the calculation. The software could well solve the problems from the practical viewpoint. For example, Fig. 10 shows the case for the stacked single layers of nickel and silver. In this case, nickel exists as the upper layer and silver is the lower one. Vacuum or inert gas was chosen as the atmosphere. The figure shows the screen shot for the virtual investigation and calculation and suggests that silver under nickel could diffuse into the upper nickel layer and appear on top of it on the top surface by appropriate heating. We have not yet investigated this case under actual conditions. However, the stacked single layers and heating might make it possible to impart an antibacterial effect to the surface of nickel plating in the future.[37]

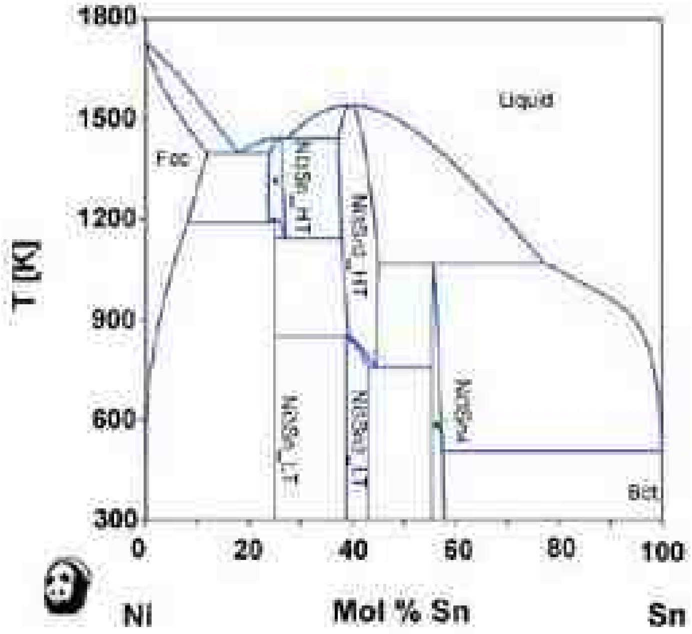

Figure 7 - Phase diagram of nickel and tin (Provided by the National Institute for Materials Science (NIMS)).

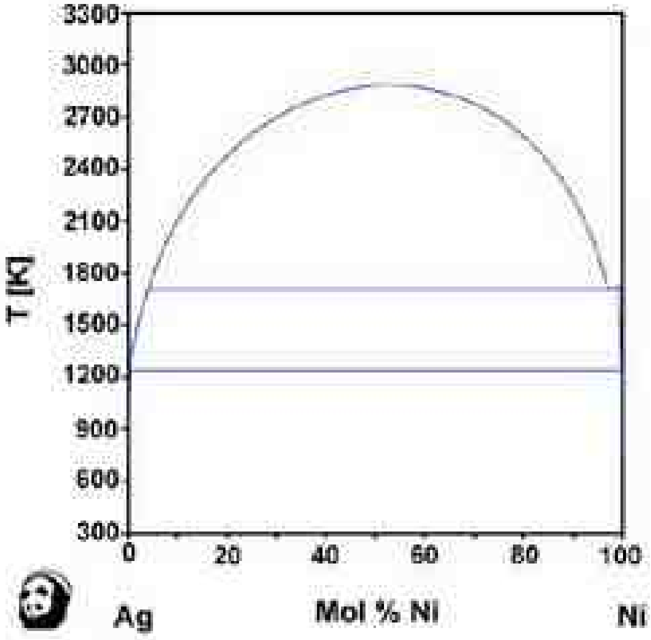

Figure 8 - Phase diagram of nickel and silver (Provided by the National Institute for Materials Science (NIMS)).

Figure 9 - Decision branches for segregation simulation on the web page of Surfseg provided by the National Institute of Materials Science (NIMS); http://surfseg.nims.go.jp/SurfSeg/select.html.

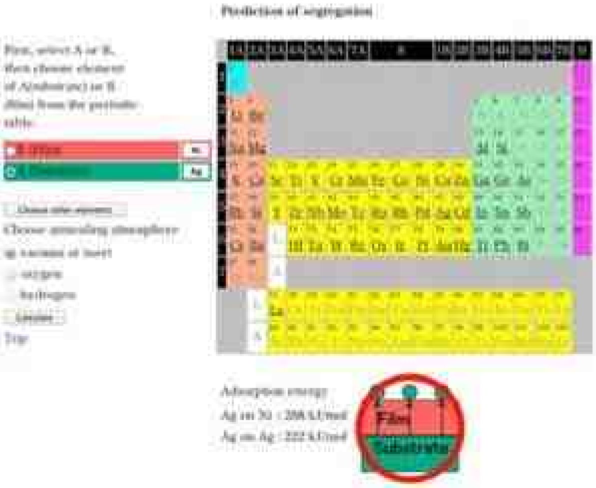

Figure 10 - A calculation result for a nickel-silver alloy film by heating by Surfseg (NIMS).

Future scope – from antibacterial effects to biofilm control

Bacteria and fouling caused by them pose significant concerns in the food processing, sanitary and medical industries. However, we human beings know that bacteria do not exist alone, but rather they live in colonies in biofilms on materials.

A biofilm can be defined as a structured community of bacterial cells enclosed in a self-produced polymeric matrix and adherent to an inert or living surface.[38] Figure 11 shows the mechanism of biofilm formation. Bacteria which flow in oligotrophic environments, *e.g.*, air, various aqueous solutions like sea, river, clean water, etc., are generally called planktonic bacteria. They are always seeking nutrients to survive. At the same time, they need to hide themselves from their natural predators and shelter from the flowing media in which they find themselves, which may sweep them away. For those reasons of survival, bacteria attach themselves to material surfaces where organic compounds are absorbed as nutrients. The bacteria thus migrate to the nutrient-enriched surface and attach there.

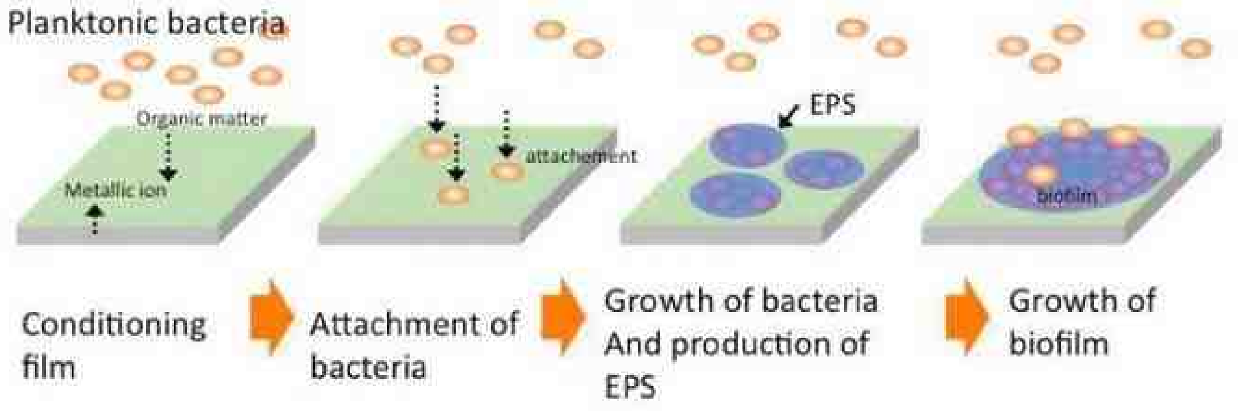

Figure 11 - Schematic illustration of biofilm formation.[39]

For a while, they repeat the attachment and detachment process, and finally the number of attached bacteria becomes to dominate the number of detached ones at a certain time. This process goes on, with bacteria repeatedly attaching and detaching themselves, but ultimately increasing in number attached to the surface. When the number of bacteria attached to the surface exceeds a threshold value, they simultaneously secrete polysaccharides and surround themselves with it. At this point, a biofilm forms and it grows, increasing the number of bacteria, polysaccharides, DNA, etc. (referred to as exopolymeric substances, or EPS) and incorporating various elements such as silicon, calcium, magnesium, zinc, etc.[39]

Such biofilms can affect many industrial problems such as corrosion, sanitary/food processing industry, infection of biomaterials, shipment problems, etc., as shown in Fig. 12. Of particular interest to this laboratory is the problem of fogging of glasses by water staining and other processes, which can lead to a serious setback for the application of glasses to heliostats.

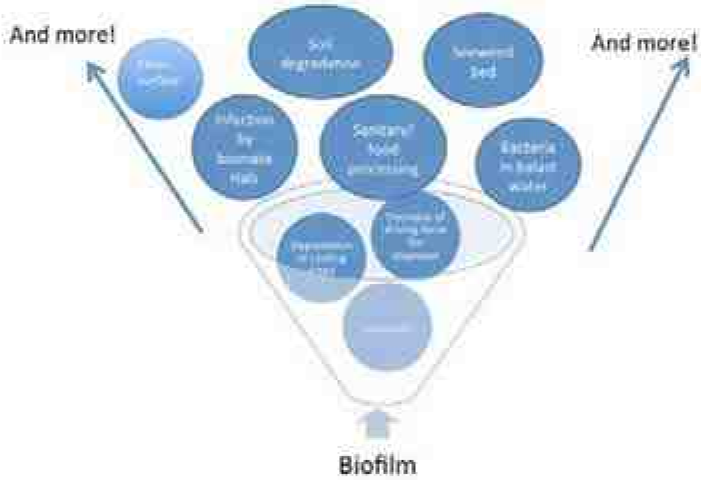

Figure 12 - Industrial problems caused by biofilms.[40]

The heliostat is one of a number of apparatuses related to photovoltaic power generation. It reflects the sunlight in the polar direction with a flat mirror. The polar axis supporting the flat mirror makes a 360° roll each day, chasing the sunlight and reflecting it to the measuring device. Its efficiency depends on the mechanical accuracy of the fine driving apparatus. However, the decreasing reflectivity of glass being fogged causes serious deterioration of the power generation efficiency. The fogging of glass itself is never caused by biofilms, but rather simple water stains. However, once the biofilm forms on a glass surface, the sticky EPS produced by bacterial activities can firmly fix the stain and fouling on the glass surfaces and the performance and transparency significantly decrease as a result.[41,42]

As countermeasures, certain special surface treatments such as the application of a water-shedding coating have been already devised. However, those have been insufficient to solve the problem completely. Antibacterial coatings could well offer an improved solution. The HSSL process may be applied to solving the problem in the future. As shown in Fig. 13, such a novel countermeasure may also address the fogging problem on touch panel sensors, whose market will be very big in the near future.

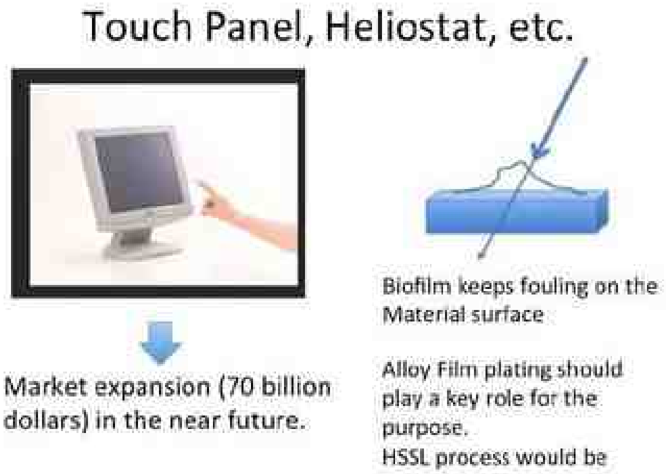

Figure 13 - The applicability of the HSSL process and anti-bacterial coatings to biofilm control.

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About Dr. Hideyuki Kanematsu

Dr. Hideyuki Kanematsu, FIMF (a Fellow of IMF) is a full professor and the dean in the Department of Materials Science and Engineering, at Suzuka National College of Technology, Suzuka, Mie, Japan. There, he is also a researcher in Environment Materials Engineering. He is interested particularly in the interfacial phenomena between the metallic surface and organisms from an environmental science viewpoint. He holds a B. Eng. (1981), a M. Eng. (1983) and a Ph.D. in Materials Science and Engineering (1989), all from Nagoya University. He is active as a member, board member, and editorial member of ASM International, the National Association for Surface Finishing, USA (NASF), the Institute of Metal Finishing, UK (IMF), as well as the Minerals, Metals & Materials Society, USA (TMS), the American Chemical Society (ACS), the Japan Institute of Metals (JIM) and the Iron & Steel Institute of Japan (ISIJ).

He was the NASF Scientific Achievement Award Winner for 2012. On receipt of the award, he noted, "Since 1998, I have joined the NASF SUR/FIN (formerly the AESF Annual Technical Conference) as regular contributor. Fortunately, I was awarded the Scientific Achievement Award in 2012. Humbly, I accepted this great award, first given in 1959, with great honor and pleasure. When I come to think about the SUR/FIN over these 15 years, my award could be attributed to my talks most of which have been related to the unique way of alloy film formation on materials surfaces."

Dr. Kanematsu Displays his William Blum Award Plaque with NASF Past President Tony Revier (Front, 2nd from Right) and Staff Members at Suzuka National College of Technology.