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## PERMANENT HYDROPHILIC SURFACE FORMATION BY ION ASSISTED REACTION

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### ABSTRACT

Since totally wettable hydrophilic polymer surfaces from hydrophobic polymers (PMMA, PTFE, PET and PC) have been demonstrated for the first time at Materials Research Society meeting, 1995 Fall meeting, Boston, the application of ion assisted reaction (IAR), in which energetic ions (0.5~1.5 keV) are irradiated on materials with blowing reactive gases near the irradiating surfaces, has been extended to various polymer, ceramic and metal for creating permanent hydrophilic surfaces. The surface energy was measured by Young's equation and the highest energy,  $E_s$ , of 60~70 mN/m, which is similar to surface energy of water ( $E_s$  of H<sub>2</sub>O : 72 mN/m) is obtained by controlling ion dose, energy, and amount of blown gas. The higher surface energy of materials possesses the more wettable surface, and relation between wettability and adhesion has been discussed. The remarkable result is the strong adhesion of inert nonattachable material such as Pt on the modified surface. The improvements of adhesions, wettability and surface energy are mainly due to polar force and hydrophilic functional groups such as C=O, (C=O)-O, C-O, etc. on the modified surface without surface damage by surface analyses. Advantages of the method are (1) high reproducibility, (2) simplicity, (3) changing ability of wettability degree, and (4) easy connection to conventional semiconductor process line. Improvements of efficiencies by changing heat transfer coefficients have been presented for the compact heat exchanger system.

### INTRODUCTION

Polymer is the most widely used material in many applications including electronics, bio-medical, displays, sensors, and so on. In general, the surface characteristics of most polymers are hydrophobic. Such hydrophobic characteristic of polymer surface gives many limitations to attempts to extend the application field of polymer and improve the property such as adhesion. Hydrophobic polymer surface gives rise to severe problems in applications especially requiring the wettable surface property and good adhesion between polymer and dissimilar materials. Therefore, up to date, many extensive studies have been performed to improve the wettability and the adhesion of metal/polymer and adhesive/polymer structure. There are major two approaches to enhance the wettability of polymer surface. Firstly, it is to create the functionality on polymer surface by using dry processes such as corona

discharge, plasma discharge, and other techniques. The second method as wet process is to coat the surfactant with hydrophilic property on polymer surface. However, in above-mentioned methods, there are problems as followings; (1) surface damage such as severe bond scission, carbonization, cross-linking etc. in the case of first methods, and (2) degradation of hydrophilic property due to the solution of surfactant layer in water environment. Therefore, a damage free and permanent surface modification technique came to be required in order to overcome those disadvantages.

A new surface modification technique of ion assisted reaction (IAR) was developed for modifying the polymer surface into hydrophilic by Koh and his co-researchers in 1995. The process of IAR is that ion beam with energy of around kiloelectron volt is irradiated on the surface of polymer in a reactive gases environment, resulting in creating the hydrophilic functional groups on the polymer surface. The merits of IAR technique include (1) excellent hydrophilicity, (2) long term durability, (3) damage free treatment, (4) high reproducibility, (5) simple process, (6) improvement of adhesion between polymer and other material, and so on.

As well as polymer, metal surface have been required to modify into hydrophilic characteristics in many applications such heat exchanger, coloring, composite material, etc. Optimum plasma zone (OPZ) technique for modifying the metal into hydrophilic was developed successfully by Koh and his colleagues. OPZ process is to use plasma of specific gases resulting in the formation hydrophilic layer on the metal surface. Layer created by OPZ has characteristics such as hydrophilicity, good adhesion to metal, and long term durability, etc.

In this study, the results of improvements in hydrophilicity and surface energy of various polymers by IAR process are represented. X-ray photoelectron spectroscopy was carried out to identify the formation of hydrophilic groups as a result of chemical reactions between modified polymer chains and oxygen gas. We report the durability of wettable polymer surface as well as adhesion improvements by IAR. Effects of hydrophilic layer by OPZ on the heat transfer efficiency were investigated. In addition, examples on industrialization of IAR and OPZ would be introduced.

## EXPERIMENTAL

### Ion Assisted Reaction Process

Fig. 1 shows the schematic diagram of ion assisted reaction (IAR) system. The IAR system consists of ion source, environmental gas supplier, specimen holder, and pumping system. The Ar ion beam was generated by a cold hollow cathode-type ion source. Ion beam energy was controlled from 0.5 to 1.5 keV. Ion doses were changed from  $5 \times 10^{14}$  to  $1 \times 10^{17}$  ions/cm<sup>2</sup> by adjusting exposure time and ion beam current density. Reactive gas was blown on the polymer specimens and its flow rate was changed from 0 to 8 ml/min during the energetic ion beam irradiation. Each polymer specimens were cleaned by conventional method of ultrasonic cleaning with proper solution for the samples.

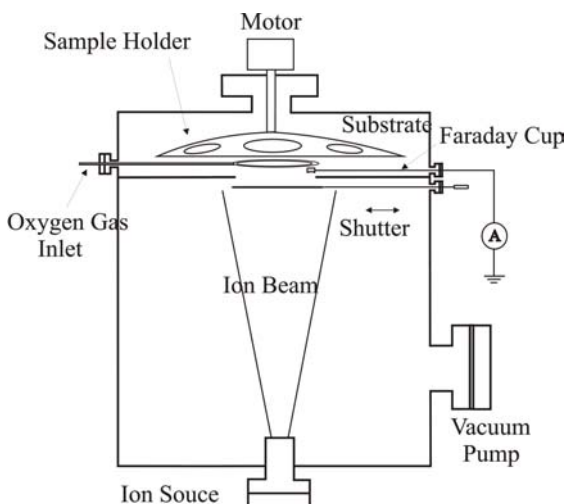


Fig. 1 Schematic diagram of ion assisted reaction system

### Optimum Plasma Zone process

In order to modify the metal surface, optimum plasma zone (OPZ) process was utilized. The schematic diagram of the OPZ system is shown in the Fig. 2.

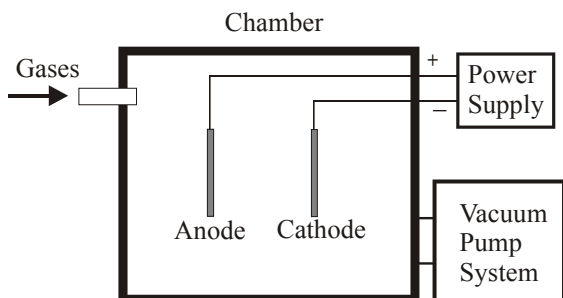


Fig. 2 Schematic diagram of optimum plasma zone system. The OPZ system mainly consists of three parts; (a) DC power applying part, (b) gas feeding part, and (c) pumping unit. Samples were positioned at anode electrode place. The vacuum chamber was evacuated by a rotary pump, a molecular booster pump and a diffusion pump. The mixture gases for polymerization were introduced up to the pressure of a predetermined value. Plasma was generated under appropriate vacuum condition by applying DC power. Applied bias was changed from 0.6 to 1 kV.

### Characterizations

The hydrophilicity of polymer surface was investigated with the water contact angle. The static contact angle measurement was conducted using contact angle meter (Tantec Co.; CAM-micro) to investigate the hydrophilicity of metal surface treated by OPZ process. Contact angles of twenty points per each specimen were acquired and the average contact angle was calculated. The deviation of contact angles was about 2 ~ 3°. In order to analyze the chemical state of polymeric surface on metal deposited by OPZ, X-ray photoelectron spectroscopy (XPS) was performed using a Surface Science Instrument 2803-S spectrometer which had a base pressure of  $2 \times 10^{-10}$  Torr. Changes in surface roughness of polymer surface after IAR treatment were observed with atomic force microscopy (AFM) (Park Scientific Instrument STM-SU-210). The root mean square value,  $\sigma_{\text{rms}}$ , of surface roughness was averaged from scanning 10 times over  $1 \mu\text{m}^2$  at different places.

## RESULTS AND DISCUSSION

### Hydrophilicity and Surface Energy

Fig. 3 represents the contact angles of polyimide (PI), polypropylene (PP), and polycarbonate (PC) with ion dose. In the case of ion irradiation without oxygen environment, the contact angles of water to PI, PP, and PC are reduced from 67° to 40°, 83° to 40°, and 78° to 51°, respectively. This reduction of contact angles is similar to values reported by other researchers. These small decreases of contact angles may be attributed to the change of surface morphology and surface cleaning. The contact angles of water to PI, PP and PC modified by ion irradiation in oxygen environment significantly decrease from 67° to 7°, 83° to 8°, and 78° to 12°, respectively. The hydrophilicity of polymer surface treated by ion irradiation with oxygen environment, that is, IAR process was remarkably improved. The improvement in wettability of IAR treated polymer might mainly be contributed to by the hydrophilic functional groups created newly on its surface. The

existence of hydrophilic functional groups formed by IAR will be discussed in results of XPS analyses.

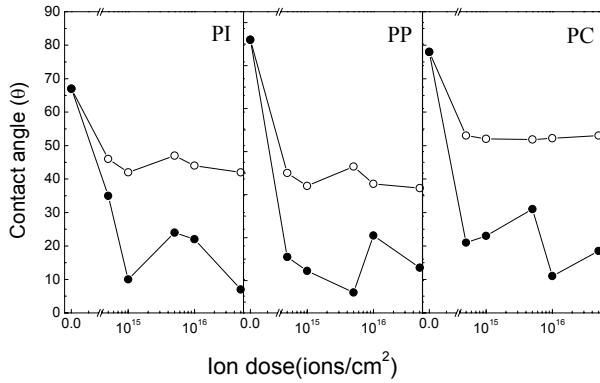


Fig. 3 Contact angles of PC, PP, and PI with ion dose ; ion irradiation only(open circle), and ion irradiation in oxygen environment(closed circle).

Changes of surface energy of polymer through IAR treatment were measured and represented in Fig. 4.

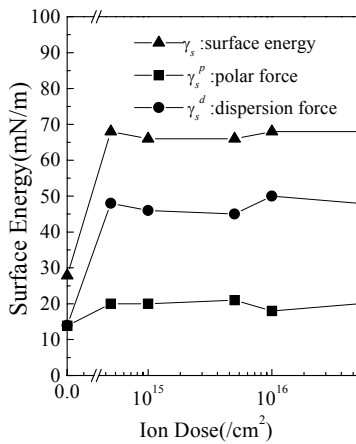


Fig. 4 Changes of surface energy for polycarbonate(PC) modified by IAR.

The surface energy of pristine PC is 28 mN/m and those of PC surfaces modified by IAR significantly increase up to 70 mN/m, which is comparable to that of water (72 mN/m). The surface energy consists of the dispersion force and the polar force. From Fig. 4, it is known that it is the polar force that mainly contributes to increase the surface energy of PC after IAR modification. Since the wettability depends on the surface energy, the improvement in hydrophilicity of polymer surface by IAR treatment could result from the dominant increase of the polar force. Therefore, it could be

assumed that the hydrophilic functional groups affecting the polar force could be highly created by IAR treatment.

**Surface Roughness and Chemical State**

The effect of ion irradiation on the surface roughness of polymer during IAR process was examined by using AFM analyses. Fig. 5 represents the AFM images of polyethylene (PE) surfaces with ion doses at IAR treatment. In Fig. 5, a noticeable change in surface morphology is not found on the PE surface after ion irradiation. Root mean square roughness ( $\sigma_{rms}$ ) of pristine PE was 56 Å. The  $\sigma_{rms}$  of PE surface irradiated at ion dose of  $1 \times 10^{15}$ ,  $1 \times 10^{16}$ , and  $1 \times 10^{17}$  ions/cm<sup>2</sup> slightly increase to 76, 70, and 71 Å, respectively. This results from ion erosion phenomenon accompanied by ion irradiation. However, this slight increase of roughness cannot sufficiently explain the remarkable drop of contact angles after IAR treatment.

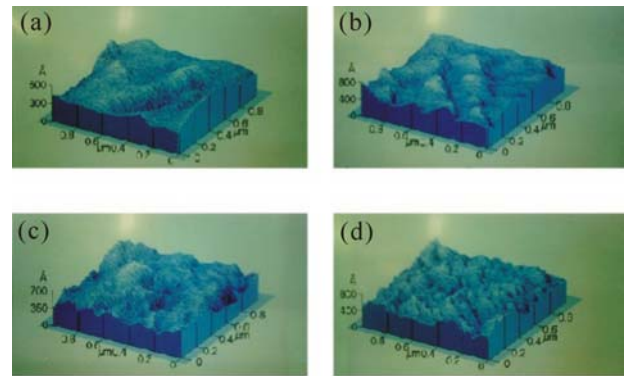


Fig. 5 AFM images of PE surface irradiated by IAR with ion dose; (a) pristine, (b)  $1 \times 10^{15}$ , (c)  $1 \times 10^{16}$ , and (d)  $1 \times 10^{17}$  ions/cm<sup>2</sup>.

In order to identify the change of chemical states for surface modified by IAR surface XPS analysis was carried out. Fig. 6 shows the C1s core level spectra of pristine polystyrene (PS) and PS irradiated at Ar ion dose of  $5 \times 10^{16}$  ions/cm<sup>2</sup> with oxygen environment. In the case of untreated PS, the C-C peak and/or the C-H peak located at 284.6 eV appeared, and other peak due to  $\pi \rightarrow \pi^*$  transition are observed at 291.0 eV. For PS treated by IAR, the intensity of C-C peak (284.6 eV) decreases and that of  $-(C-O)-$  peak (286.0 eV),  $-(C=O)-$  peak (287.4 eV),  $-(C=O)-O-$  peak (288.8 eV) increases, respectively. From this result, it could be explained that the C-C or C-H chains in polymer are broken by energetic ion bombardment and the induced unstable chains react with oxygen. It is confirmed by XPS analyses that the hydrophilic functional groups such as  $-(C-O)-$ ,  $-(C=O)-$ , and  $-(C=O)-O-$  are created on the polymer surface by IAR process. The mechanism as to the formation of hydrophilic functional groups by IAR could be explained

by the two step process which includes; (1) a creation of an unstable polymer chain by ion irradiation and (2) a chemical reaction between highly unstable reactive radicals and introduced reactive oxygen gas. In consequence, the newly formed functional groups could give an effect to the improvement of hydrophilicity. XPS results of other polymers such as PE, PC, PMMA, PI, etc. were similar to that of PS.

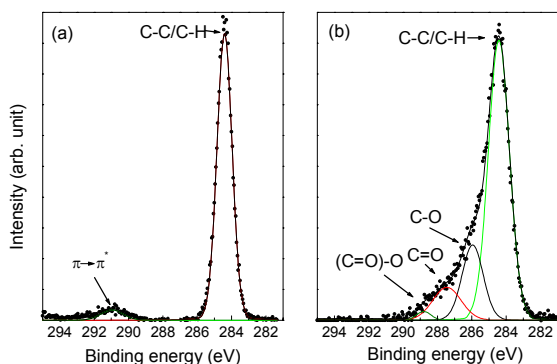


Fig. 6 Cls core level spectra of (a) the untreated PS and (b) IAR treated PS.

There are four main methods to improve the wettability of material surface. These include surface roughening, surface cleaning, hydrophilic layer coating and the formation of hydrophilic group. Schematic of methods to improve the hydrophilicity is shown in Fig. 7. Firstly, when the surface becomes rougher, the wettability of material can slightly increase. This phenomenon is attributed to the increase of surface area due to roughening. Secondly, the wettability of material can be also improved by removing the contaminants on the surface. Contaminants such as oil vapor or grease on the surface prevent the water from spreading. However, surface roughening and surface cleaning have inevitable limitations to increase wettability, since the surface energy of material at these methods cannot help depending its bulk property. That is, the surface energy higher than intrinsic value of material cannot be obtained by these two methods. However, in the cases of the hydrophilic layer coating and the formation of hydrophilic group, the material can be modified to have the surface energy higher than its own value, resulting in getting more wettable than pristine state. The degree of improvement on wettability through layer coating depends on the hydrophilicity of coating layer. The formation of hydrophilic groups as the fourth method can modify just surface by creating new chemical bonds on. The wettability of surface by the formation of hydrophilic group is affected by the density and stability of hydrophilic groups. IAR treatment used in

this study belongs to method of the formation of hydrophilic groups on surface.

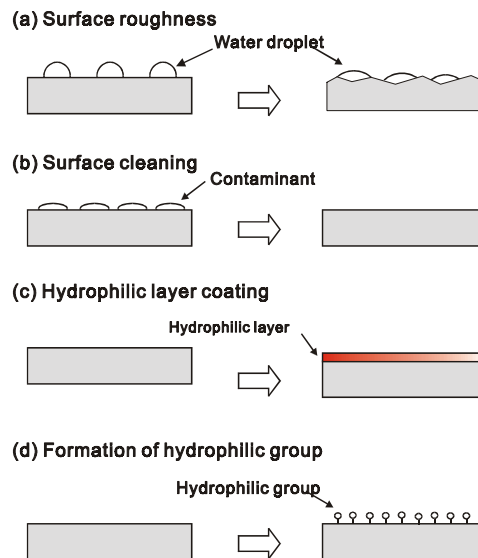


Fig. 7 Schematic of methods for improvements of wettability.

#### Durability of Hydrophilicity

Fig. 8 shows the changes of water contact angles of PE surfaces kept in air and distilled water with time.

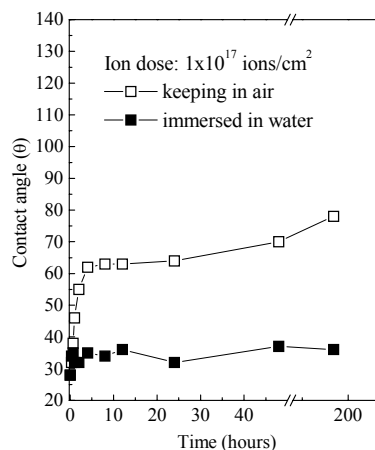


Fig. 8 Contact angle of water on IAR treated PE surface kept in air and water environments with time.

In case of PE kept in air, the contact angle of PE increased up to  $80^\circ$  after 192 hr. This result seems for hydrophilic functional groups to be removed with time. However, this phenomenon is not removal of functional groups, but the reorientation of those. Since the surface energy goes toward

lowering in non-polar environments such as air, the reorientation of polar groups naturally occurs in polymer surface. The reorientation of functional groups on IAR treated polymer surface can be easily identified by the fact that the contact angle of polymer kept in air recovers to low contact angle after immersion of that in water. The interesting result in Fig. 8 is that the contact angle of PE treated by IAR remains to be initial value in water environments with time. If the hydrophilic groups are unstable or soluble, the wettability of polymer cannot maintain when keeping in water. The loss of hydrophilicity is generally shown in the cases of plasma discharge treatment and surfactant coating. However, the polar groups on polymer surface modified by IAR could be known to be very stable and durable from the retention of hydrophilicity in water.

Durability of hydrophilic groups on polymer surface modified by IAR can be more precisely identified by XPS analyses of IAR-treated polymer surface before and after washing, and the results of PP are shown in Fig. 9.

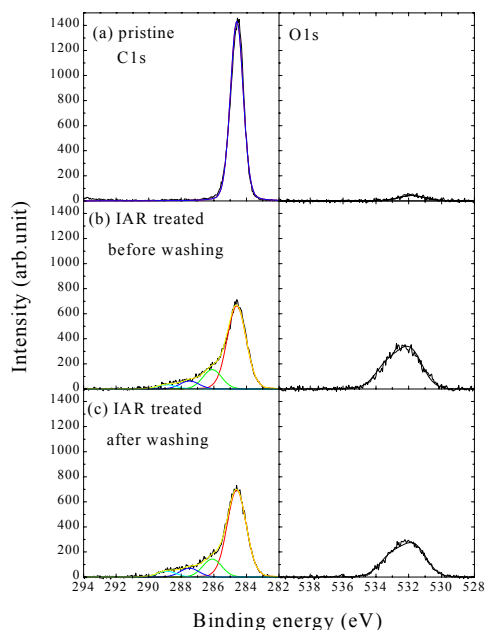


Fig. 9 Cls core level spectra of (a) pristine PP, (b) IAR treated PP before washing and (c) IAR treated PP after washing.

In Fig. 9 (b), it is shown that the hydrophilic functional groups were highly created on the PP surface modified by IAR. After washing this PP treated by IAR, the intensities of peaks related to hydrophilic groups in Cls and O1s remained to be the same level as that of PP before washing, indicating that the loss of hydrophilic groups scarcely occurred. In the contrary to results of PP treated by IAR, it was reported by Muhlhan *et al.* that the O1s peak showed a

remarkable loss in its intensity after washing in PP modified by low-pressure plasma. This means that a large number of oxygen functional groups could be removed by washing, and the functional groups created by plasma treatment would be unstable due to their weak bonding to the substrate and damaged surface. From these results, it is considered that the hydrophilic functional groups formed on polymer surface by IAR would be very stable and not washed in water environment, leading to long-term durability of hydrophilicity.

### Adhesion Improvement

Fig. 10 shows the optical microscope photographs of Pt thin films on the pristine and IAR-modified PVDF after boiling test, in which the PT/PVDF specimens were kept in hot boiling water for 4 hours.

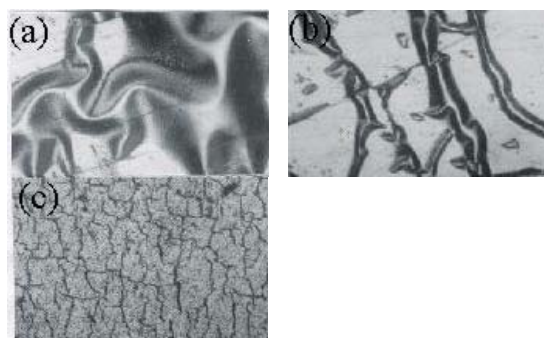


Fig. 10 Optical microscope photographs of Pt films on PVDF after boiling test during 4 hours: (a) pristine, (b)  $5 \times 10^{14}$  ions/cm<sup>2</sup>, and (c)  $1 \times 10^{15}$  ions/cm<sup>2</sup>.

From Fig. 10 (a), it is shown that the large amounts of Pt thin films in Pt/pristine PVDF specimen buckled after boiling test. The stress at the interface of Pt/PVDF was applied due to the difference of thermal expansion between metal and polymer during boiling test, and the residual stress applied to the Pt layer deformed the metal films, resulting in the detachment of metal films from polymer surface in the form of buckling. The failure of the Pt layer on the PVDF modified at  $5 \times 10^{14}$  ion/cm<sup>2</sup> is represented in Fig. 10 (b), and the buckled area of Pt layer was significantly reduced. The interesting result is that the only cracks on Pt layer were formed without buckling in the specimen of Pt/PVDF irradiated at  $1 \times 10^{15}$  ions/cm<sup>2</sup>. The stress-release mode was converted from the buckling of the Pt layer to crack propagation within the Pt layer with IAR modification. It is considered that since the adhesion strength between Pt layer and IAR-treated PVDF increase highly enough not to detach the Pt layer, the stress applied to Pt layer is released by a formation of crack and propagation. From these results, it is known that IAR

modification of PVDF can remarkably enhance the adhesion of metal layer. The adhesion improvement of Pt/PVDF irradiated by IAR should be attributed to the interaction between the electron acceptor from electronegative oxygen in the hydrophilic groups and the electron donor from electropositive metal. It is known that Pt as inert metal is difficult to obtain good adhesion on polymer substrate. Despite this weakness of Pt, Pt thin films on the IAR-treated PVDF represent good adhesion due to the increased surface energy and the formation of charge transfer complex. As well as Pt/PVDF structure, it was observed that the adhesions of Cu/PTFE, Cu/PI, Pt/PI, etc. were highly improved by IAR modification.

### Optimum Plasma Zone Process

**Hydrophilicity.** The contact angles of Al surfaces treated by OPZ ranges from  $10^\circ$  to  $20^\circ$ , while the bare contact angle of bare Al surface is around  $90^\circ$ . Fig. 11 shows the photograph of bare Al surface and OPZ treated Al surface after water spraying. In Fig. 11, only parts of symbol of P&I and square line were modified by optimum plasma zone (OPZ), and other parts of Al were untreated. While the water on bare Al surface appeared to be in the formation of droplet, the sprayed water on the modified Al surface formed the water film, indicating that the modified surface has excellent hydrophilic. Also, the drainage of water on the OPZ-treated Al is excellent due to water of film type.



Fig. 11 Photograph of bare Al surface and OPZ treated Al surface after water spraying (symbol of P&I: OPZ treated surface, and other place; bare surface)

In order to examine the long term durability of hydrophilic property of OPZ treated Al surface, the contact angles of OPZ treated Al were measured with storage time in water environment, and its results are shown in Fig. 12. In the case of surfactant coating in water environment, the hydrophilicity of layer is gradually lost with time due to the dissolution of surfactant in water. The surfactant coated Al

reveals the good wettability at initial stage, but the surface of Al recovers to the hydrophobic surface of bare Al in certain period of operation. However, it can be known from Fig. 12 that the hydrophilicity of OPZ treated Al surface maintains even after the storage time of 540 days in water environment, meaning that the hydrophilic groups on OPZ layer surface would be very stable, and could not be solved in water. So, OPZ treatment can overcome the problem in durability of hydrophilicity that occurs in the case of surfactant coating.

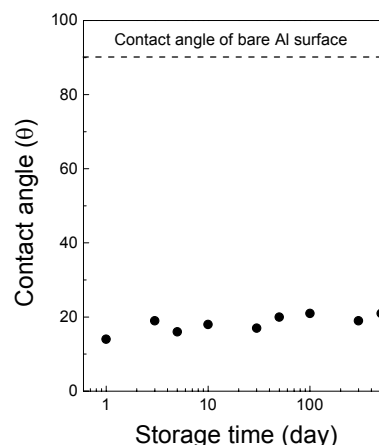


Fig. 12 Long-term durability of hydrophilicity of OPZ treated Al.

**Improvements of heat transfer efficiency.** Plain, and low-finned tubes were treated by OPZ to investigate the effects of hydrophilic surface treatment on evaporation heat transfer at the outside wall of copper tubes in an absorption refrigerator system. Experimental setup and procedure of this study were described in detail in paper recently reported by Kim, H. Y. et al. The effects of the evaporation pressure in the test chamber on the heat transfer rate are shown in Fig. 13(a). In this study, the inlet temperature of the nozzles and that of water entering the evaporator were maintained at 20 and 50 °C, respectively. The pressure in the test chamber was changed as 2.34, 4.25, and 7.38 kPa, and the corresponding evaporation temperature was 20, 30 and 40 °C, respectively. It can be known from Fig. 13(a) that as the evaporation pressure increases, the heat transfer rate,  $q$ , slightly decreases, resulting from the fact that the difference between the evaporator inlet temperature and the evaporation temperature decreases with the increase of chamber pressure. All heat transfer rates obtained at several pressures for plain tube and low-finned tube treated by OPZ much increased compared to those of bare copper tubes. And in Fig. 13(b) showing the effects of the OPZ treatment on the product, UA, of the total heat transfer coefficient, U, and the heat transfer area, A, the OPZ modification could

enhance the UA values by approximately 40% for a plain tube, and 19~26% for a low-finned tube. While the liquid on hydrophobic surface such as untreated tube forms discrete sessile drops, the liquid on the hydrophilic surface treated by OPZ drains as a film type, as shown in Fig. 11. Liquid of film type is relatively very thin and the resultant thermal resistance to heat transfer is quite low. Therefore, enhancement of heat transfer efficiency through OPZ treatment could be explained by the fact that the thermal boundary-layer thickness has been reduced by hydrophilic surface.

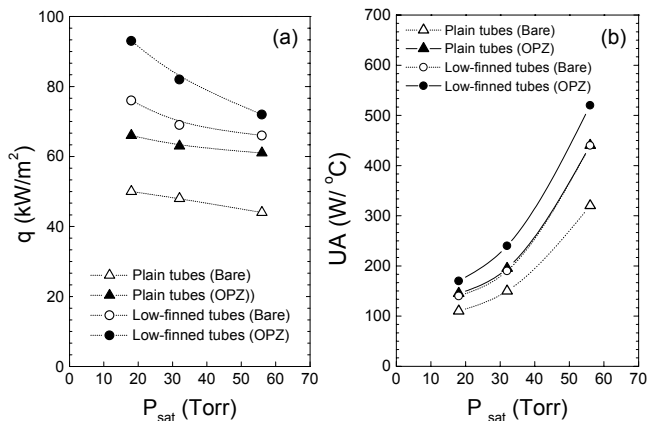


Fig. 13 Experimental results of the evaporation heat transfer with the saturation pressure. (a) heat transfer rate per unit surface area, and (b) the product of total heat transfer coefficient, U, and heat transfer area, A.

### Industrialization of IAR and OPZ Processes

Modification of polymer by IAR covers a variety of applications including electronics, bio-medical, sensor, household appliance, optical device and so on. Up to date, industries adopting IAR technology are following as; PTFE tape, Petri-dish for cell culture, Sensor using piezoelectric polymer, thermo-conductive silicon rubber for plasma display panel (PDP), hard coating on plastic case, etc. In order to adopt IAR technology to industry, mass production scale system is essential. Types of IAR mass production scale system can be divided into two; (a) batch type and (b) roll-to-roll type. Batch type is suitable for modifying 3-dimensional specimen or non-flexible material. In case of roll-to-roll type, the polymer films could be continuously treated without vacuum breaking as long as required. The two types of real IAR system for mass production are shown in Fig. 14. Batch type shown in Fig. 14 (a) adopt cassette feed-in for supplying sample to be modified. This batch type system is to treat Petri dishes for cell culture, and is capable of modifying about 2500 dishes per hour. Roll to roll system shown in Fig. 14 (b) consists of film winding

system, IAR processing system, and unwinding system. In roll-to-roll system, the productivity of system is dependent on the control of line speed, ion dose, and ion beam energy for optimum modification. Roll to roll system covers application fields including PTFE tape, Cu/PI for flexible circuit copper clad, PC film for flexible display, PET film for package, and so on.

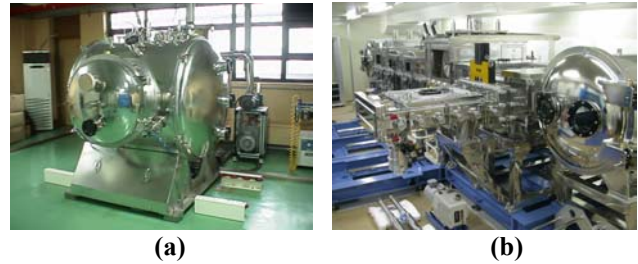


Fig. 14 Photographs of IAR mass production systems: (a) batch type and (b) roll to roll type.

As for OPZ process, LG electronics adopted this technique to improve the performance of heat exchanger installing in air-conditioner. LG electronics has already established mass production lines using this technology, and sells the products with heat exchanger treated by OPZ, worldwide. There is modification of stainless steel sheet as another application of OPZ process. OPZ treated stainless steel surface can be utilized in the fields of coloring stainless steel sheets and SUS sheets attaching other material due to good wettability and adhesion. SPTEch incorporation is developing several stainless steel sheet products using OPZ technology. Nowadays, the researches on improving the performances of IAR and OPZ processes are actively going on. In near future, more industries of various application fields are expected to adopt our technologies.

### CONCLUSIONS

1. The contact angles of hydrophobic polymer surfaces were remarkably reduced up to around 10° by ion assisted reaction (IAR) technique, and the surface energies of polymers modified by IAR increased to about 70 mN/m, which is comparable to that of water (72 mN/m).
2. Excellent hydrophilicity of IAR-treated polymer surface is attributed to the newly formed functional groups such as -C-O-, -(C=O)-, and -(C=O)-O-, confirmed by XPS analysis.
3. Adhesion between polymer and metal can be highly enhanced by IAR surface modification, due to the high surface energy.



4. Layers on metal surface created by optimum plasma zone (OPZ) show the good wettability, and the heat transfer system using OPZ-treated metal exhibits higher heat transfer efficiency, resulting from the hydrophilicity of surface.

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