Research Article

Photocatalytic Surface Modification of PI Film for Electroless Copper Plating

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This study investigated the surface modification of polyimide (PI) film through TiO₂ photocatalytic treatment. The effects of TiO₂ content, treatment duration, and UV power on the surface topography, surface contact angle, and adhesion strength of the surface-modified PI films were investigated. The results indicated that, after surface modification under the optimal photocatalytic conditions, the surface contact angle of the PI film decreased from 84.4° to 38.8°, and the adhesion strength between the PI film and the electroless copper film reached 0.78 kN/m. X-ray photoelectron spectroscopy analysis further demonstrated that carboxyl groups formed on the surface of the PI film after photocatalytic treatment. The surface hydrophilicity and adhesion strength of the surface-modified PI film were enhanced due to the numerous carboxyl groups formed on its surface. Therefore, the photocatalytic treatment is an environmentally friendly and effective method for the surface modification of PI films.

1. Introduction

Polyimides (PIs) possess many desirable traits, such as low dielectric constant, high breakdown voltage; good planarization, wear resistance, radiation resistance, inertness to solvents, and hydrolytic stability; low thermal expansion; long-term stability; and excellent mechanical properties. Given these attributes, PIs have been extensively used as substrates in the production of ultra-large-scale integrations, electronic packages, and circuit boards [1–3]. In several of these applications, the deposition of a metal layer on PI substrates is necessary to allow electrical conduction via interconnections [4]. However, poor adhesion strength between the copper film and the PI substrate is a serious problem in these applications.

Over the years, many studies have investigated surface modification methods for improving the adhesion of metals to PI film. The surfaces of PI films are commonly modified through plasma treatment [5–10], ion implantation [11, 12], chemical treatment [13–18], and UV/ozone treatment [19–21]. However, physical processes, such as plasma and ion implantation, require expensive equipment and are thus

associated with high investment costs. Chemical treatments, such as oxidation, polymer grafting, and etching, require expensive waste disposal. The surfaces of polymers can be modified with photoirradiation, which occurs through the formation of an electron-hole pair in the semiconducting material when the photon energy exceeds the band gap [22]. Photocatalysis has been receiving increasing attention as an advanced oxidation process due to its low cost and environmental friendliness. In addition, the high chemical stability of TiO₂ and the potential use of sunlight as the irradiation source make photocatalysis an attractive method for the surface modification of PI films.

Thomas reported that base hydrolysis and acidification resulted in the formation of poly(amic acid) on the surface of PI film [23]. In this study, we introduced a method that combines the surface formation of hydrophilic species and the cleavage of imide rings to enhance the adhesion of copper to PI film. The effects of TiO₂ content (0.5, 1.0, 2.5, and 5.0 g/L), UV light power (100 and 300 W), and treatment duration (10, 20, 30, and 40 min) on the surface topography, surface roughness, adhesion strength, and surface chemistry of the

TABLE 1: The compositions of the electroless copper plating bath.

Composition	Content
Copper sulfate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$)	10 g/L
Ethylenediaminetetraacetic acid disodium salt (EDTA-2Na)	30 g/L
Formaldehyde (HCHO , 37%)	3 ml/L
2,2'-Dipyridyl	15 mg/L
Poly(ethylene glycol) (PEG 1000)	0.5 g/L

PI films were investigated. The surface topography and chemistry of the PI films were evaluated through scanning electron microscopy (SEM), atomic force microscopy (AFM), surface contact angle measurements, and X-ray photoelectron spectroscopy (XPS).

2. Experimental Section

PI films with a density of 1.42 g/cm^3 were procured from DuPont Chemical Co., Ltd., as Kapton HN in rolls that were 40 mm wide and $75 \mu\text{m}$ thick. The rolls were then cut into $40 \text{ mm} \times 25 \text{ mm}$ samples for all experiments. Before photocatalytic treatment, the surfaces of the PI films were cleaned with acetone in an ultrasonic water bath for 30 min at room temperature and washed with deionized (DI) water. The photocatalysis experiments were conducted with a TiO_2 suspension over the PI surface. Then, 0.5–5.0 g of TiO_2 powder (JR05, Xuancheng Jingrui New Materials Co., Ltd., 5 nm), which was primarily in anatase phase, was dispersed in 1 L distilled water, respectively. Photocatalytic irradiation was conducted under a high-pressure mercury vapor lamp with a maximum wavelength of 365 nm. The distance between the PI film and the mercury lamp was fixed at 5 cm. After photocatalytic treatment, PI films were rinsed thrice with DI water.

The surface activation of the PI film was carried out in an activation solution (CATAPOSIT 44, purchased from Rohm and Haas Company) at 45°C for 5 min. Subsequently, the surface sensitization of the substrates was conducted by immersing the samples in 10% hydrochloric acid aqueous solution at 30°C for 1 min. The PI films were washed with distilled water after each step. The chemical composition of the electroless copper plating solution was the same as that in the literature [18], and the compositions are shown in Table 1. The pH of the solution was adjusted to 11.5 using NaOH, and the bath temperature was 70°C . After electroless plating for 40 min, the PIs were electroplated with copper at room temperature with a current density of 0.03 A/cm^2 for 1 h. The copper layer was deposited at a thickness of $20 \mu\text{m}$. After annealing at 100°C for 120 min in an oven, the copper-coated PI films were cut into $10 \text{ mm} \times 40 \text{ mm}$ strips. The adhesion strength of the PI films was measured through a 90° peel test at a peel rate of 25 mm/min [24]. The peel test was repeated four times, and the adhesion strength was reported as the average value of four measurements.

The surface topographies of the PI films were observed through SEM (Philips-FEI Quanta 200 electron microscope).

FIGURE 1: Dependence of the surface contact angle upon the TiO_2 content and the treatment time under 300 W UV light.

Moreover, the surface roughness of the PI films was measured through AFM (WETSPM-9500-J3, Shimadzu Co., Ltd.). Surface roughness was reported as average roughness (R_a) and root-mean square roughness (R_{ms}). The surface contact angles of the PI films were determined as quickly as possible after different time photocatalytic treatment with a video-based contact angle measurement instrument. The volume of the water drop used in the measurements was $2 \mu\text{L}$, and measurement values were taken at least five positions on the surface of the samples and averaged. The surface composition and chemistry groups of the PI films were determined by XPS measurements taken with a JPS90-MXV spectrometer from JEOL with a nonmonochromatized Mg Ka X-ray source. All binding energies were corrected on the basis of the Cls binding energy at 285 eV [25].

3. Results and Discussions

3.1. Effects of UV Power and Treatment Time on the Surface Contact Angle of PI Films. The wettability of the PI films was evaluated by measuring the surface contact angles of the film at room temperature. When the UV power was 300 W, effects of the TiO_2 content and the treatment duration upon the surface contact angle were measured, and the results are shown in Figure 1. As seen in the figure, the surface without photocatalytic treatment PI film was hydrophobic. The contact angle between distilled water and the untreated PI film was 84.8° . The surface contact angle of the PI film continuously decreased with increasing treatment duration when the PI films were treated with 0.5 g/L TiO_2 suspension under 300 W of UV light, suggesting that 0.5 g/L TiO_2 content

FIGURE 2: Dependence of the surface contact angle upon UV power and the treatment time at the TiO_2 content of 1.0 g/L.

was insufficient for inducing hydrophilicity. The surface contact angle of the PI film treated with 1.0 g/L TiO_2 suspension decreased with prolonged treatment duration and reached the minimum value with an irradiation time of 30 min; after that, the surface contact angle slowly increased with the further increase in the irradiation time. This result indicated that photocatalytic treatment induced the hydrophilicity of the PI film surface. Meanwhile, the surface contact angle of PI film treated with 1.0 g/L TiO_2 suspension was considerably lower than that of the PI film treated with 5.0 g/L TiO_2 suspension for the same treatment duration. This result was attributed to the agglomeration of TiO_2 particles in the saturated TiO_2 solution, because agglomeration decreases the oxidative capacity of generated $\cdot\text{OH}$ radicals. Under 100 W of UV light, the change trend in the surface contact angle in response to TiO_2 content and treatment duration was similar to that under 300 W of UV light. The results indicated that 1.0 g/L TiO_2 suspension and 30 min of photocatalytic treatment are appropriate process parameters for the surface modification of PI film.

The effects of UV light power and treatment duration on the surface contact angle of PI films treated with 1.0 g/L TiO_2 solution are shown in Figure 2. Under 100 and 300 W of UV light, the surface contact angle of the PI film first decreased with prolonged treatment time and reached the minimum value after 30 min of treatment, and then, the surface contact angle increased when treatment time was continuously prolonged. Furthermore, the surface contact angle decreased with increased UV power at the same treatment time and fixed TiO_2 content. One potential reason for this result could be that the oxidation capacity of the photocatalytic system was strengthened by the increased intensity of UV light.

The roughness and hydrophilicity of the film surface affect the surface contact angle of the film. Specifically, the surface contact angle decreases with enhanced surface hydrophilicity and decreases with increasing surface roughness. Therefore, surface morphology and surface roughness observations were performed through SEM and AFM.

3.2. Effects of Surface Modification on the Surface Morphology and the Surface Roughness of PI Substrates. Figure 3 shows the changes in surface morphology before and after surface modification with 1.0 g/L TiO_2 suspension under 100 or 300 W UV light. The wet-chemical pretreatment may induce changes in morphology and the removal of weakly cohesive surface material. Thus, the surface morphology of the film is very rough and uneven after wet-chemical pretreatment [26, 27]. However, as shown in Figure 3, the surface morphology of the PI film treated with 1.0 g/L TiO_2 solution showed almost no change compared with that before photocatalytic treatment. Thus, the electroless copper film was more uniformly deposited through the photocatalytic reaction than through the wet-chemical pretreatment.

The AFM observations of the PI film morphology before and after pretreatment are shown in Figure 4. Before photocatalytic treatment, the PI film surface was flat and clean with R_a and R_{ms} of 13 and 17 nm, respectively. R_a and R_{ms} of the PI film modified under 100 W for 30 min were 14 and 19 nm, respectively. In addition, R_a and R_{ms} of the PI film modified under 300 W for 30 min were 15 and 21 nm, respectively. The results indicated that increasing UV power does not substantially change the surface topography of the PI films.

3.3. Effects of Surface Modification on the Adhesion Strength.

The dependence of the adhesion strength on surface modification was investigated, and the results are shown in Figures 5 and 6. The adhesion strength between the electroless copper film and untreated PI film was zero. After photocatalytic treatment for 10 or 40 min, the adhesion strength between the electroless copper film and PI film increased with the increasing of TiO_2 content when the treatment time was fixed. And the adhesion strength between the electroless copper film and PI film reached the highest value after the PI film was treated with 1.0 g/L TiO_2 suspension. After that, the adhesion strength between the electroless copper film and PI film decreased when TiO_2 content was further increased. It can be seen from Figure 6, when PI film was treated with 1.0 g/L TiO_2 suspension, the adhesion strength between the electroless copper film and PI film increased with the increasing of UV power. Adhesion strength reached the maximum value when the PI film was treated for 30 min. This result was consistent with the changes in the surface contact angle of the PI film in response to TiO_2 content, treatment duration, and UV power. Given that hydrophilicity can improve the wettability of the film surface, the overall increased wettability of the PI film improved the intimate contact between the PI substrate and the electroless deposited copper film.

Adhesion strength is dependent not only on the surface roughness but also on surface hydrophilicity or density of

FIGURE 3: SEM images in surface morphology of PI films before and after surface modification with 1.0 g/L TiO_2 dispersed solution for 30 min, UV power: (a) 0 W, (b) 100 W, and (c) 300 W.

polar groups. Therefore, the effects of surface modification on the surface chemistry of PI films were investigated.

3.4. Surface Chemistry of the PI Film before and after TiO_2 Photocatalytic Treatment. The surface chemical properties of the PI films before and after photocatalytic treatment were characterized by analyzing the XPS spectra of the PI films. The surface element contents of the PI films were determined through XPS, and the results are shown in Table 2. It can be seen that the carbon content decreased with the increasing UV power and the oxygen content increased with the increasing UV power. The surface carbon and oxygen contents of the untreated PI film were 78.2% and 15.8%, respectively. When the UV power was 300 W, after 30 min of photocatalytic treatment, the carbon content of the PI film decreased from 78.2% to 75.4%, and the oxygen content of the PI film increased from 15.8% to 19.3%. This result was attributed to the increased density of hydrophilic groups under increased UV power. The trend in the variation of the aforementioned elemental contents corresponded with that of the surface contact angle.

TABLE 2: The element contents of PI surface before and after surface modification with 1.0 g/L TiO_2 dispersed solution under different UV power.

Treatment condition	C (%)	O (%)	N (%)
Before treatment	78.2	15.8	6.0
100 W, 30 min	77.0	17.5	5.5
300 W, 30 min	75.4	19.3	5.3

The C1s XPS spectra of PI films before and after treated with 1.0 g/L TiO_2 and UV power of 300 W were obtained and the results are shown in Figure 7. Four peaks were observed in the spectrum of the PI film before photocatalytic treatment. The peaks at 285.0, 286.2, 288.6, and 291 eV were attributed to C-H/C-C/C=C, C-O/C-N, $\text{N}(\text{C}=\text{O})_2$, and $\pi-\pi^*$, respectively. A peak at 289.0 eV appeared in the spectrum of the PI film treated with 1.0 g/L TiO_2 for 30 min under 300 W UV light. This peak could be attributed to the -COOH group. The same phenomenon was observed when the PI film was treated with 1.0 g/L TiO_2 for 30 min under 300 W UV light. The

FIGURE 4: AFM images in surface morphology of PI films before and after surface modification with 1.0 g/L TiO_2 dispersed solution for 30 min, UV power: (a) 0 W, (b) 100 W, and (c) 300 W.

FIGURE 5: Dependence of the adhesion strength upon the TiO_2 content and the treatment time under 300 W UV light.

FIGURE 6: Dependence of the adhesion strength upon the UV power and the treatment time at the TiO_2 content of 1.0 g/L.

FIGURE 7: The Cls XPS spectra of the PI films before and after surface modification with 1.0 g/L TiO_2 dispersed solution for 30 min under 300 W UV light. (a) The Cls XPS spectra of the PI films before surface modification. (b) The Cls XPS spectra of the PI films after surface modification.

carbon group contents of the PI films before and after being treated for 30 min under different UV powers and 1.0 g/L TiO_2 content are shown in Table 3.

When the UV power increased from 0 W to 300 W, the content of hydrophobic groups (C-H/C-C/C=C) decreased from 53.7% to 46.5%, that of the $\text{N}(\text{C}=\text{O})_2$ group decreased from 14.8% to 6.8%, and that of C-O/C-N and π - π^* groups showed almost no change. However, the content of

hydrophilic groups (-COOH) increased from 0% to 6.1%, indicating that partial bonds of the PI film were cleaved and that amide and carboxyl groups formed on the surface of PI films after treatment with TiO_2 . Given that carboxyl and amide groups can coordinate with the copper atom, their quantities can affect the adhesion strength of copper film to the PI film [28]. As a result, the high contents of -COOH groups enhanced the surface hydrophilicity of the PI film and

TABLE 3: The contents of carbon groups on the PI film surface before and after photocatalytic treatment (treatment time: 30 min, C_{TiO_2} : 1.0 g/L).

UV power	C-C, C-H, C=C (%)	C-O, C-N (%)	N(C=O) ₂ (%)	$\pi-\pi^*$ (%)	-COOH (%)
0	53.7	38.7	14.8	2.8	0
100 W	48.0	37.6	7.3	2.2	4.9
300 W	46.5	38.3	6.8	2.3	6.1

thus improved the adhesion strength between the electroless copper film and the PI film.

4. Conclusion

An environmentally friendly and effective photocatalytic treatment method was used to modify PI films. The effects of TiO_2 content, treatment duration, and UV power on the surface hydrophilicity and adhesion strength of the modified PI film were investigated through surface contact angle measurements, SEM observation, AFM, and XPS. The surface hydrophilicity and the adhesion strength of the photocatalytically treated PI film increased with increasing UV power and prolonged treatment duration. Surface modification with 1.0 g/L TiO_2 suspension was more effective than with other TiO_2 contents under 300 W of UV power. High TiO_2 content facilitated the aggregation of TiO_2 particles, thus decreasing the oxidative capacity of the hydroxyl radicals generated by the TiO_2 suspension. Under the optimal conditions of photocatalytic treatment, the adhesion strength between the electroless copper film and the PI film reached 0.78 kN/m, and the surface topography and the surface roughness of the PI film slightly changed. XPS results indicated that surface oxygen content increased with prolonged treatment duration and increased UV power. Meanwhile, the surface carbon content decreased with prolonged treatment duration because the content of -COOH polar group increased. Photocatalytic treatment enhanced the surface hydrophilicity of PI film, thus resulting in high adhesion strength between the PI film and electroless copper film.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this article.

Authors' Contributions

Wenxia Zhao and Zenglin Wang contributed equally to this paper.

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