

Article

Effect of Humidity on Metallizing on Polyphenylene Sulfide (PPS) with Atmospheric UV Treatment

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Abstract: Polyphenylene sulfide (PPS) is an engineering plastic; when reinforced with fillers, it exhibits high heat resistance, strength, and molding/dimensional stability. Plating on PPS without using harmful chemicals can meet the following requirements due to its excellent properties: low environmental load process, lightweight metal substitute materials, electromagnetic wave shielding materials, etc. This study focused on metallizing by atmospheric ultraviolet (UV) treatment of PPS. This process is generally used for the pretreatment of painting and adhesion, and it entails a small environmental load; however, the UV treatment of moist air produces various chemical species. Therefore, the humidity effect during metallizing via atmospheric UV treatment was investigated, revealing its influence on the adhesion strength of the resulting metal film. In a dry environment, a metal film with strong adhesion can be formed on PPS, and UV treatment under such conditions can maintain the structure of the PPS surface. In contrast, a weak layer was generated under wet conditions, reducing the adhesion strength between the metal film and PPS.



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1. Introduction

In recent years, plating on engineering plastics has been investigated using a construction method that excludes harmful chemicals [1–4]. There are three reasons for this. First, the allowed content of hazardous chemical substances in products is restricted by environmental regulations such as the Registration, Evaluation, Authorisation, and Restriction of Chemicals (REACH); therefore, plating methods must not use harmful chemicals. Second, to reduce automobile fuel consumption, metals need to be replaced with lighter materials [5]. Third, electromagnetic wave shielding materials are required for electric vehicle conversion [6,7]. Plating on engineering plastics without the use of harmful chemicals can meet all these requirements due to their excellent properties.

In this study, we focused on the metallizing of polyphenylene sulfide (PPS), an engineering plastic, by atmospheric Ultra-Violet (UV) pretreatment. PPS has excellent heat resistance and mechanical properties [8]; given its good fluidity and small molding shrinkage, it shows exceptional injection moldability and has been productized in various fields [9]. However, since PPS has high chemical stability, strong and harmful chemicals such as hydrofluoric acid must be used as a plating pretreatment on it [10]. Therefore, an environmentally friendly alternative pretreatment is required, and atmospheric UV treatment can meet this requirement. UV treatment can modify a resin surface and is often adopted as a pretreatment for adhesion and painting [11–14]. Nomura et al. used UV treatment to obtain a plating film with good adhesion on a PPS containing elastomer [3]. This method is therefore simple, useful, and carries a small environmental load. However, UV treatment under atmospheric conditions is affected by air humidity. Yoshihara et al. reported the generation

of hydrogen peroxide as nuclei of droplets by UV irradiation in a moist atmosphere [15]. Jin et al., irradiated the condensed phase of water with UV and observed the formation of hydroxyl radicals with high oxidizability [16]. We speculate that the UV-treatment effect changes with humidity because the above-mentioned species originate from the moisture on the PPS surface; therefore, the humidity influence during UV treatment in the atmosphere must be considered. Nevertheless, to the best of our knowledge, no study has investigated the relationship between humidity during UV treatment and plating adhesion. Thus, in this work, we tried to form a plating film on PPS through atmospheric UV treatment, and found that the adhesion strength of the resulting plating film was affected by humidity during the process. We also demonstrated the possibility to obtain a film with strong adhesion on elastomer-free PPS in a dry environment.

2. Materials and Methods

2.1. Experimental Procedure

As the test pieces, we used PPS resin “TORELINA™” A504X90B (40% fiberglass, Toray Industries, Inc., Tokyo, Japan) and prototype A, which is based on A504X90B with an added elastomer. A modified elastomer with increased reactivity was used and the amount was 3–7 wt%. The test piece was obtained by cutting a plate prepared by injection molding under the conditions shown in Table 1. The size of the test pieces was 80 mm × 15 mm × 3 mm. They were wiped with ethanol. Then the test pieces were UV-pretreated by using UV instrument (PL21–200 (s): SEN LIGHTS Corp., Toyonaka, Japan), a low-pressure mercury lamp which occupies >90% of the total radiant energy at the wavelengths of 254 and 185 nm. The test pieces were placed 20 mm away from the lamp and treated under atmospheric conditions, while the entire UV instrument was placed in a box where dry air was introduced. Dry air was obtained by removing moisture from the atmosphere with a Membrane Air Dryer (IDG60: SMC Corp.). Compressed air of 0.7 MPa was introduced into the filter to obtain dry air. For stabilization, UV treatment was performed after 30 min of introduction of dry air and continued during the treatment.

Table 1. Molding conditions.

		A504X90B	Prototype A
Cylinder temperature	°C	300	300
Mold temperature	°C	130	130
Filling time	s	15	15
Cooling time	s	15	15
Screw rotation speed	rpm	100	100
Injection speed	mm/s	100	100
Injection pressure	MPa	39	40
(Lower limit pressure)	MPa	29	30
Weighing position	mm	46	46
Injection end position	mm	8.88	7.21
Peak pressure	MPa	59.3	60.8

Table 2 summarizes the plating process. We performed the sensitizer and activator treatment twice to improve the deposition of Ni, for which a chemical Ni plating was used (SEP-LF Okuno chemical Industry Co., Ltd., Osaka, Japan).

2.2. Characterization

2.2.1. Adhesion Strength of the Plating Film

After annealing, a 30 µm thick Cu plating film was formed by electroplating, and notches with a width of 10 mm were made in it. The plating film was peeled off at 30 mm/min by a tensile tester (MCT-2150: A & D Co., Ltd., Tokyo, Japan) to have it uniform and perpendicular to the plastic substrate. We measured the adhesion strength (in N/cm) continuously for 10 mm while peeling and took the average value; for these measurements, we prepared five test pieces per treatment condition, and also recorded

the relative humidity and temperature every two minutes during the UV treatment, and calculated the average volume absolute humidity during the treatment.

Table 2. Plating process.

Ethanol Wiping	Ethanol Conc. 99.5%
Ultraviolet pretreatment	1 h
Alkaline aqueous solution treatment	KOH: 200 g/L, 65 °C, 10 min
•	
Cleaner/conditioner	45 °C, 2 min
•	
Sensitizer	SnCl ₂ aq.: Okuno Chemical Industry Co., Ltd. 30 °C, 1 min
•	
Activator	PdCl ₂ : 150 ppm, 30 °C, 1 min
•	
Sensitizer	SnCl ₂ aq.: Okuno Chemical Industry Co., Ltd. 30 °C, 1 min
•	
Activator	PdCl ₂ : 150 ppm, 30 °C, 1 min
•	
Electroless Ni plating	SEP-LF: Okuno Chemical Industry Co., Ltd.
•	
Annealing	120 °C, 1 h

•: water rinse.

2.2.2. Moisture Adsorption on the PPS Surface

We measured the water vapor adsorption isotherm to confirm the adsorption of water on the PPS surface by using a surface area and pore size analyzer (BELSORP-Max-N-VP-CM: Nippon Bell Co., Ltd., Osaka, Japan). The raw material powder of the molded pellet was the sample, which is a pure PPS powder. Measurements were performed after annealing at 50 °C for 4 h under vacuum. The temperature during the measurements is constant (298.15 K), the saturated vapor pressure was 3.17 kPa, and the saturated vapor pressure is 23.06 g/m³.

2.2.3. Observation of the Peeled Surface of the Plating Film and Surface Roughness before and after UV Treatment

After its formation, we peeled off the plating film. Its peeled surface was observed using a digital microscope (VHX-6000: KEYENCE Co., Ltd., Osaka, Japan).

Surface roughness before and after UV treatment was measured using a laser microscope (VK-X1100: KEYENCE Co., Ltd., Osaka, Japan).

2.2.4. Changes in the PPS Surface Chemical State after UV Pretreatment

We investigated the difference in the chemical state of the PPS surface modified by UV treatment under wet and dry conditions by using an X-ray photoelectron spectroscopy (XPS) system (JPS-9010TR: JEOL Ltd., Tokyo, Japan) equipped with a monochromatic Al K α source operated at 10 kV, 100 W. The C1s and S2p 3/2 spectra were measured.

2.2.5. Changes in the PPS Surface Physical State after UV Pretreatment

We conducted a hardness test on the surface of the test pieces before and after UV treatment by the nanoindentation method with a Nano Indenter (G200: KLC Co.). The load, surface approach speed, and peak retention time were 0.1 mN, 20 nm/s, and 10 s, respectively; the nanoindentation hardness was measured at 15 points. We tried to use the smallest possible load to make the measurement depth shallow. Young's modulus was calculated with the Poisson ratio of PPS as 0.4.

We also analyzed the structure of the outermost PPS surface through Grazing Incidence X-ray Diffraction (GIXRD) [17] by using a diffractometer (Smart Lab: Rigaku Corp., Tokyo,

Japan) equipped with a Cu K α source operated at 40 kV and 30 mA. The incidence angle was measured at $\omega = 0.08$.

3. Results and Discussion

3.1. Adhesion Strength of the Plating Film on PPS

Figure 1 illustrates the relationship between average volume absolute humidity and adhesion strength during UV pretreatment. A504X90B showed a maximum adhesion strength of 15.1 N/cm and a minimum adhesion strength of 2.8 N/cm, whereas their corresponding values for Prototype A were 22.6 N/cm and 11.4 N/cm. The difference in adhesion strength was probably due to the presence of the elastomer in Prototype A.

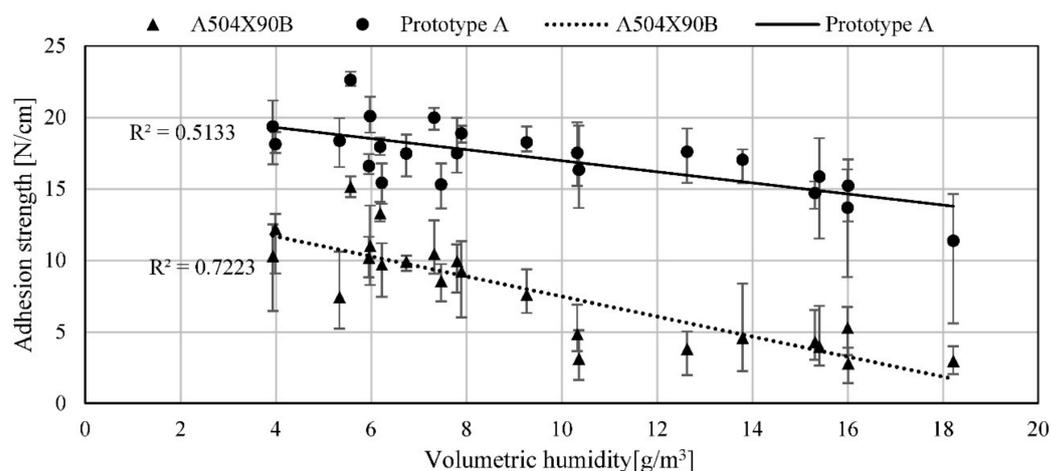


Figure 1. Relationship between volumetric humidity and adhesion strength.

The adhesion strength between the PPS resin and the plating film tended to increase as the volume of absolute humidity decreased. Therefore, we added an approximately straight line and calculated the coefficient of determination (R^2). For A504X90B, its value was 0.7223, and a slightly good approximation line was obtained. Similarly, the coefficient of determination for Prototype A was 0.5133, but with a slightly poor fit.

We performed regression analysis on these results by using Microsoft Excel 2016 (version 2201) (Table 3). For all the approximate lines, the p -value, which express “the probability that the null hypothesis will give a more extreme value than actually observed data under the correct assumption” [18], was $<1\%$. In this regression analysis, we set the null hypothesis that “there is no correlation between volume absolute humidity and adhesion strength of the plating film.” Therefore, since a p -value below 1% means that our null hypothesis is less likely than one in 100 times, these approximate straight lines are significant. It also indicates a negative correlation between volume absolute humidity and adhesion strength of the plating film.

Table 3. Regression analysis results.

Test Piece	Coefficient of Determination (R^2)	Regression Coefficient (Slope)	Standard Error	p -Value
A504X90B	0.7223	−0.70	0.09	2.86×10^{-7}
Prototype A	0.5133	−0.39	0.08	1.20×10^{-4}

3.2. Moisture Adsorption on the PPS Surface

Figure 2 displays the water vapor adsorption/desorption isotherms of the PPS powder before and after UV treatment. The amount of water adsorbed increased along with the relative humidity. Moreover, the UV-treated sample exhibited greater water adsorption than the untreated one. The shape of the isotherm shows the interaction between the gas and the surface, which is classified into several types. The low-pressure part has a small

adsorption amount and became greater as the high-pressure part increased. Both of these features showed type-III isotherms, indicating a small interaction between PPS surface and water molecules.

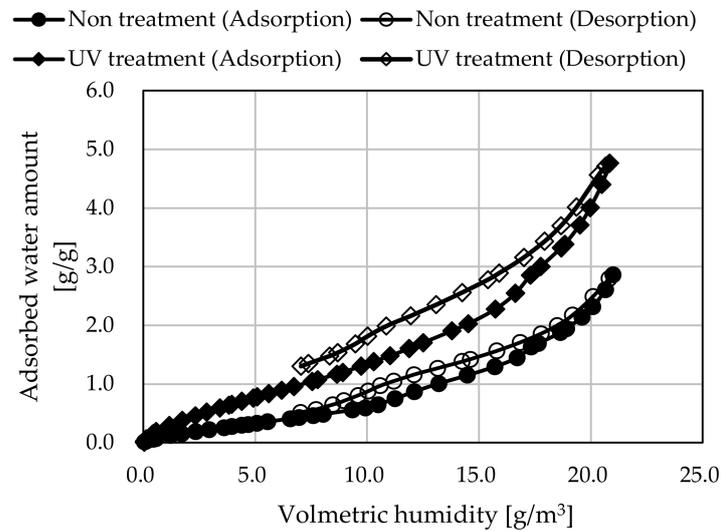


Figure 2. Water vapor adsorption isotherms of the untreated and UV-pretreated samples (Using pure PPS powder).

The specific surface area was derived from the nitrogen adsorption isotherms by using the Brunauer–Emmett–Teller (BET) adsorption theory. The measurement was performed twice to obtain the average. Its value for the untreated and UV-pretreated samples was 3.06 and the 3.36 m²/g, respectively, which suggests that the physical anchors responsible for the adhesion of the plating film are not formed by UV treatment.

3.3. Observation of the Peeled Surface of the Plating Film

Figure 3 displays magnified images of the surface of the plating film peeled off from UV-pretreated A504X90B under dry and wet conditions, showing the regions with and without broken PPS resin. For the dry conditions, the peeled plating film has a small region with PPS resin, which we speculate was mainly due to the peeling at the interface between the plating film and resin. However, for the test piece pretreated under wet conditions, the region with PPS resin was wide; most of the areas were broken and peeled off between the resin layers. This test piece also showed a lower adhesion strength between PPS resin and the plating film compared with that pretreated under dry conditions. We attribute this low adhesion strength to the deterioration of the PPS resin by UV treatment under wet conditions.

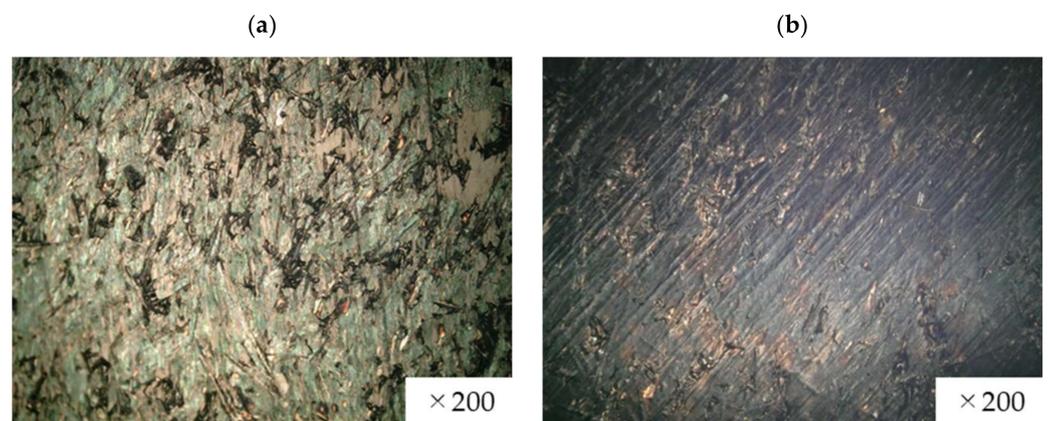


Figure 3. Cont.

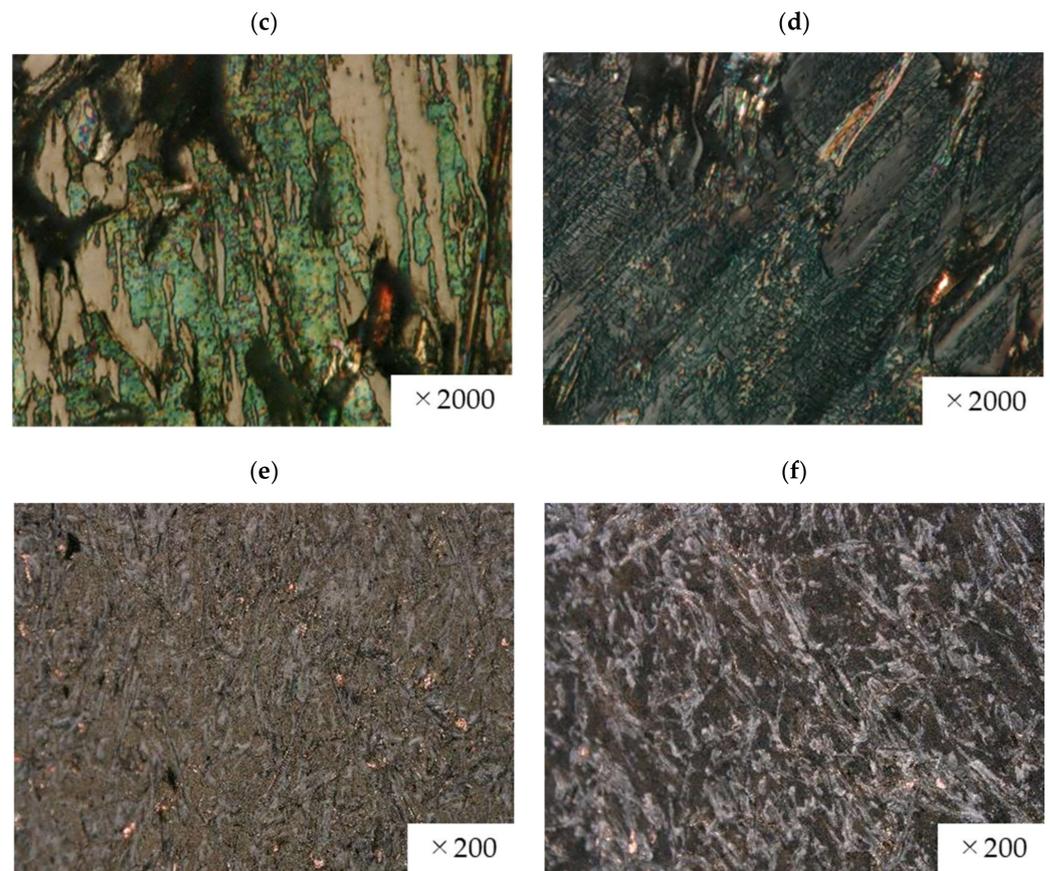


Figure 3. Images of the surface of plating film peeled off A504X90B (a–d) and Prototype A (e,f) test pieces that had been ultraviolet-pretreated under (a,c,e) dry (7.5 g/m^3) and (b,d,f) wet conditions (18.2 g/m^3): high (c,d) and low (a,b,e,f) magnification.

Table 4 compares the surface roughness. Sa represents the average of the absolute values of the height differences of each point with respect to the average surface of the surface. Sdr indicates how much the expanded area (surface area) of the definition area increases with respect to the area of the definition area. The difference between these values was small, and the surface roughness due to UV treatment did not change. It was clarified that there was no improvement in adhesion due to the increase in surface roughness.

Table 4. Surface roughness measurement result of each condition of the A504A90B test pieces.

	No Pretreatment	UV Treatment under Dry Conditions	UV Treatment under Wet Conditions
Measured surface image			
Sa [nm] (Arithmetic mean height)	304.1	255.2	271.6
Sdr (Expanded area ratio)	0.040	0.036	0.027

3.4. Changes in the PPS Surface Chemical State after UV Pretreatment

Figure 4 compares the XPS spectra of A504X90B before and after UV pretreatment under both humidity conditions. The C 1s spectra were normalized by the peaks of the C-H and C-C bonds, whereas the S 2p 3/2 ones were normalized by the S-C bond peak. The C 1s spectrum demonstrated that the UV pretreatment introduced some functional groups containing O. Regarding the C=O bond, test pieces pretreated under wet conditions showed the strongest peaks; this result indicates a relative decrease in the amount of C-H and C-C bonds, suggesting molecular chain scission. In the S2p 3/2 spectrum, a S-O bond peak was observed for the UV pretreated test pieces, and it was stronger for the wet conditions.

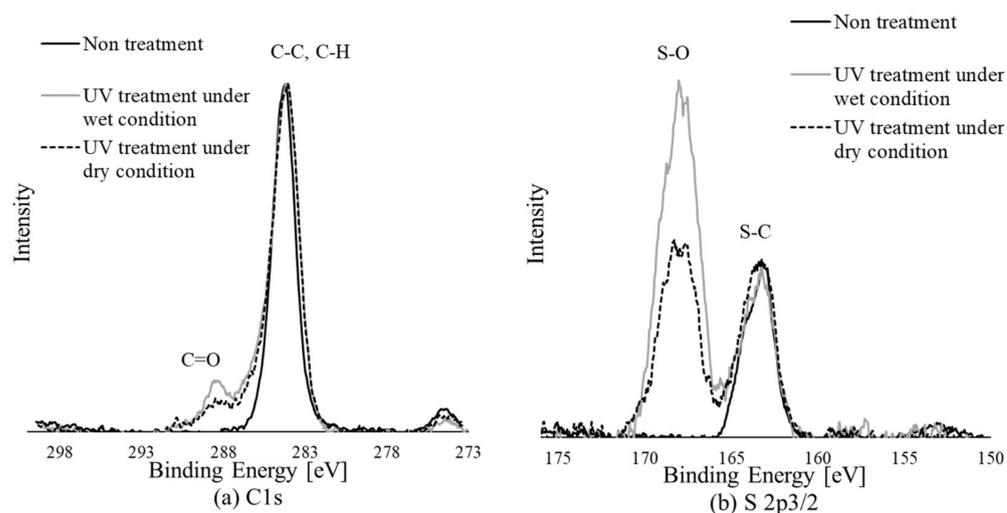


Figure 4. XPS spectra of each UV condition (A504X90B): (a) C1s spectra, (b) S2p3/2 spectra (wet conditions: 16.0 g/m³ and dry conditions: 7.8 g/m³).

It is assumed that the functional groups expressed by UV irradiation facilitate the adsorption of water on the surface and in the modified layer. As a result, some swelling may be considered, which may lead to weakness.

Table 5 lists the atomic concentrations on the surface of all the test pieces, derived from their XPS spectra. The UV pretreatment increased the relative proportion, which was the highest for the wet conditions. This indicates that the introduction of O-containing functional groups was further promoted by UV treatment under wet conditions.

Table 5. Atomic ratio after each treatment (dry conditions: 7.8 g/m³ and wet conditions: 16.0 g/m³).

Element	No Pretreatment	UV Treatment under Dry Conditions (2.6 g/m ³)	UV Treatment under Wet Conditions (6.6 g/m ³)
C	81.9	55.4	47.4
O	6.3	32.4	39.1
N	1.7	2.2	3.6
S	10.1	10.0	9.9

The adhesion between the plating film and resin can be explained by two mechanisms: physical adhesion and chemical adhesion [4,19]. In the physical mechanism, as typified by the anchor effect, a plating film is formed on the rough surface of the resin and gets caught on the rough surface. The adhesion is developed by being caught by the plating. In the chemical mechanism, adhesion results from the chemical interactions between the polar functional groups on the resin surface and the plating film. However, the XPS results indicate that the adhesion of the test piece UV-pretreated under wet conditions, which had more functional groups with polarity, was weaker compared to the one treated under dry conditions. Based on the observation of the peeled plating surfaces, we attribute this poor adhesion to the development of a weak layer by UV pretreatment under wet conditions. To confirm the formation of weak layers, changes in the physical state of the

PPS surface were investigated by testing its hardness via the nanoindentation method and GIXRD measurements.

3.5. Changes in the PPS Surface Physical State after UV Pretreatment

Figure 5 illustrates the relationship between the hardness obtained by the nanoindentation test and Young's modulus. The untreated test pieces and UV-pretreated ones under dry conditions exhibited similar values. The test pieces treated under wet conditions instead showed higher hardness and Young's modulus, suggesting changes in the structure of the outermost PPS surface.

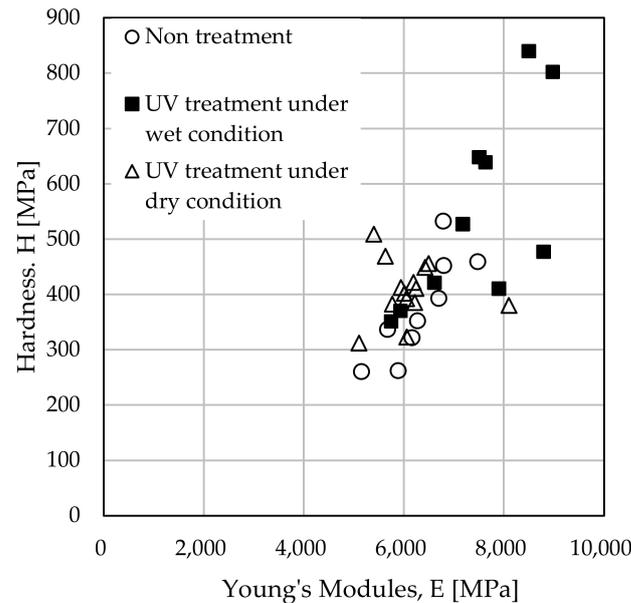


Figure 5. Young's modulus and hardness of the surface of each test piece. (A504X90B) wet conditions: 4.8 g/m^3 and dry conditions: 2.9 g/m^3 .

Figure 6 compares the GIXRD result of each test pieces. The untreated test piece and the one UV pretreated under dry conditions showed the same peak intensity, whereas that of the test piece treated under wet conditions was lower. This reduction in the peak intensity means that the crystal structure had collapsed and become amorphous. Reactions induced by UV treatment generally include functional group formation, recrystallization, cross-linking, and molecular chain scission [4,20–22]. This is consistent with the abovementioned XPS analysis, which revealed an increase in the amount of C-O and C=O bonds and a relative decrease in the C-H and C-C ones after UV pretreatment under wet conditions. In other words, in the UV treatment under wet conditions, functional group formation, molecular chain cleavage, and cross-linking proceeded at the same time; these reactions formed a weak layer on the PPS surface and decreased the adhesion strength. The UV-pretreated test pieces under dry conditions instead exhibited the same hardness and crystallinity as the untreated ones. The structure of their surface was maintained even after the UV treatment, resulting in high-adhesion strength.

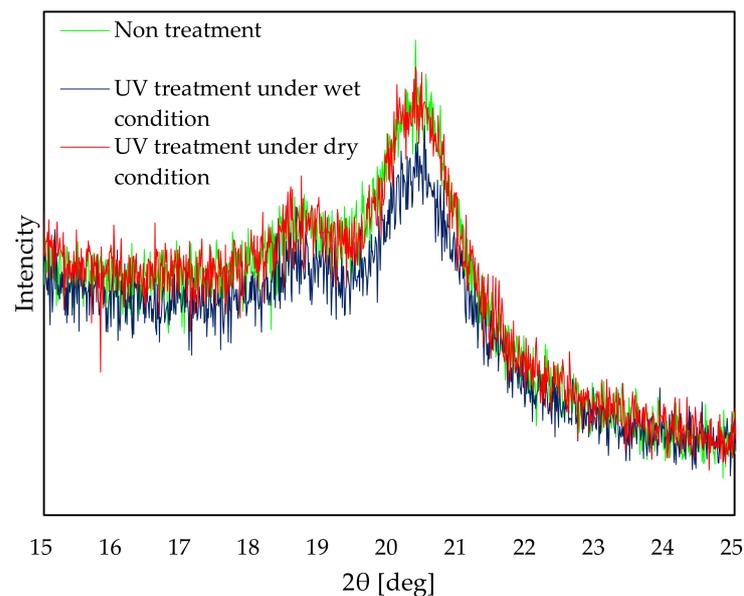


Figure 6. Diffraction pattern of each test piece obtained by the GIXRD method. (A504X90B) wet conditions: 6.6 g/m^3 and dry conditions: 2.6 g/m^3 .

4. Conclusions

We investigated the relationship between humidity during the UV pretreatment of PPS resin and adhesion of the plating film, obtaining the following results:

- The adhesion strength between the plating film and PPS resin was affected by humidity during the UV pretreatment. It decreased as the amount of atmospheric water vapor increased. Under wet conditions, A504X90B and Prototype A showed an adhesion strength of 2.8 and 11.4 N/cm, respectively; under dry conditions, the corresponding values were 15.1 and 22.6 N/cm, respectively.
- The reduction in the plating adhesion strength under wet conditions, which occurred despite the expression of functional groups was due to the formation of a weak layer. In contrast, the UV pretreatment under dry conditions could maintain the PPS surface structure, ensuring high adhesion strength.

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