

# Improved Surface Characteristics and the Conductivity of Polyaniline–Nylon 6 Fabrics by Plasma Treatment

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**ABSTRACT:** The effect of plasma treatment on the surface characteristics and conductivity of polyaniline–nylon 6 composite fabrics was investigated. Plasma surface modifications with oxygen, ammonia, and argon were performed on the nylon 6 fabrics to improve the adhesion and rate of polymerization. The surface morphology of the fiber was observed with scanning electron microscopy, and functional groups introduced onto the surface of nylon 6 fibers by various plasma treatments were characterized by X-ray photoelectron spectroscopy. With oxygen plasma treatment, the fiber surface was effectively etched; polar groups such as —OH and —OOH were introduced onto the surface of nylon 6 fiber, and they increased surface activity, promoted oxidation polymerization, and resulted in higher add-on and electrical conductivity. However, the introduced amine and amide groups with ammonia treatment caused a reduction in conductivity. Argon did not significantly alter the surface characteristics of the nylon 6 fibers. In addition, to control fabric conductivity and cover as wide a range of conductivity as possible, we observed the effects of the monomer concentration and number of deposits on the fabric conductivity. The results showed that fabric conductivity increased as the monomer concentration increased up to 0.5M and then leveled off, and further increases were achieved with an increase in the number of multiple deposits.

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**Key words:** plasma treatment; surface modification; conductive textiles; polyaniline; nylon 6

## INTRODUCTION

Textile materials as substrates and reinforcing materials for many polymers have wide industrial applications. With the rapid development

of the electronics industry, the demand for electrically conductive textiles, such as electromagnetic wave interference (EMI) shielding materials for personal computers and home electronics devices, flooring and ceiling materials, deelectrifying cloths,<sup>1,2</sup> and radar crosssection reducing protective fabrics for stealth technology, has increased. Among the manufacturing processes used to produce conductive materials, there have been great expectations for  $\pi$ -electron conjugate polymers such as polythiophene, poly-

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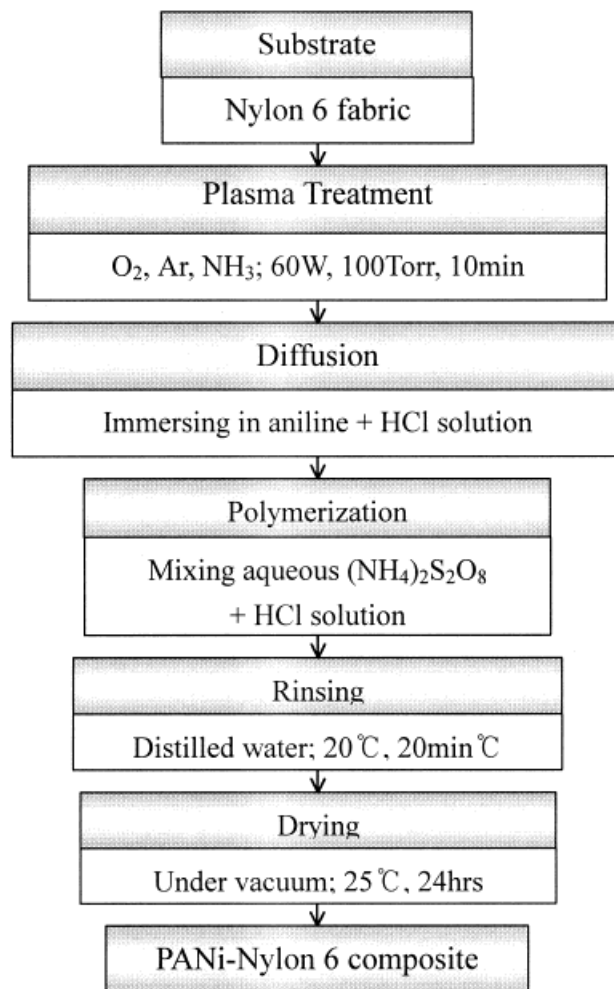
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niline (PAn),<sup>3,4</sup> and polypyrrole.<sup>5,6</sup> Within the class of conducting polymers, a wide range of associated electrical and optical properties, coupled with good stability, make PAn attractive as an electronic material for potential use in a variety of applications.<sup>3,7,8</sup>

Aniline monomer can be polymerized on fabric from an aqueous solution.<sup>9-11</sup> Aniline is soluble in water to some extent. This solubility drastically decreases for the dimer and oligomers and causes precipitation of the polymerizing species. When the polymerization is conducted in the presence of a fabric with a high surface area, the entire polymerization occurs on the surface of the fabric in a high yield. In our previous work,<sup>11,12</sup> we found that highly conductive nylon 6 fabrics could easily be produced by the immersion of the fabric in an aqueous hydrochloride solution of aniline and the initiation of polymerization with a mixture of a prepared oxidant and dopant solution with a dilute aniline solution. The conductivity of PAn-nylon 6 fabric depends on a deposition of PAn onto the surface or in the interstices of the fabric.<sup>4,10</sup> As investigated by Gregory et al.,<sup>4</sup> it is continuing physical adsorption followed by polymerization in the plane of the liquid-solid interface. Monomer and oxidant are adsorbed on the surface of the fiber instead of diffusing into the fiber. Therefore, it was expected that the fabric conductivity and adhesion of the polymer on the fiber surface could be improved with an increase in the surface energy and surface area.

In this research, a plasma surface treatment was applied to nylon 6 fabric to improve surface adhesion and increase the amount of PAn deposition, thereby increasing electrical conductivity and durability.<sup>13</sup> Many authors have shown that low-temperature reactive-gas plasma treatments are highly efficient for increasing surface energy and reactivity without altering substrate bulk properties.<sup>14-16</sup> Among various gases, oxygen, argon, and ammonia were applied in this study to nylon 6 fabrics to improve the adhesion and rate of polymerization. Surface characteristics of the plasma-treated nylon fabrics were analyzed with X-ray photoelectron spectroscopy (XPS). Furthermore, multiple aniline polymerization and dedoping-redoping were performed to enhance the electrical conductivity and durability of the PAn-nylon 6 fabrics.



**Figure 1** Schematic diagram of the PANi-nylon 6 composite fabric preparation process.

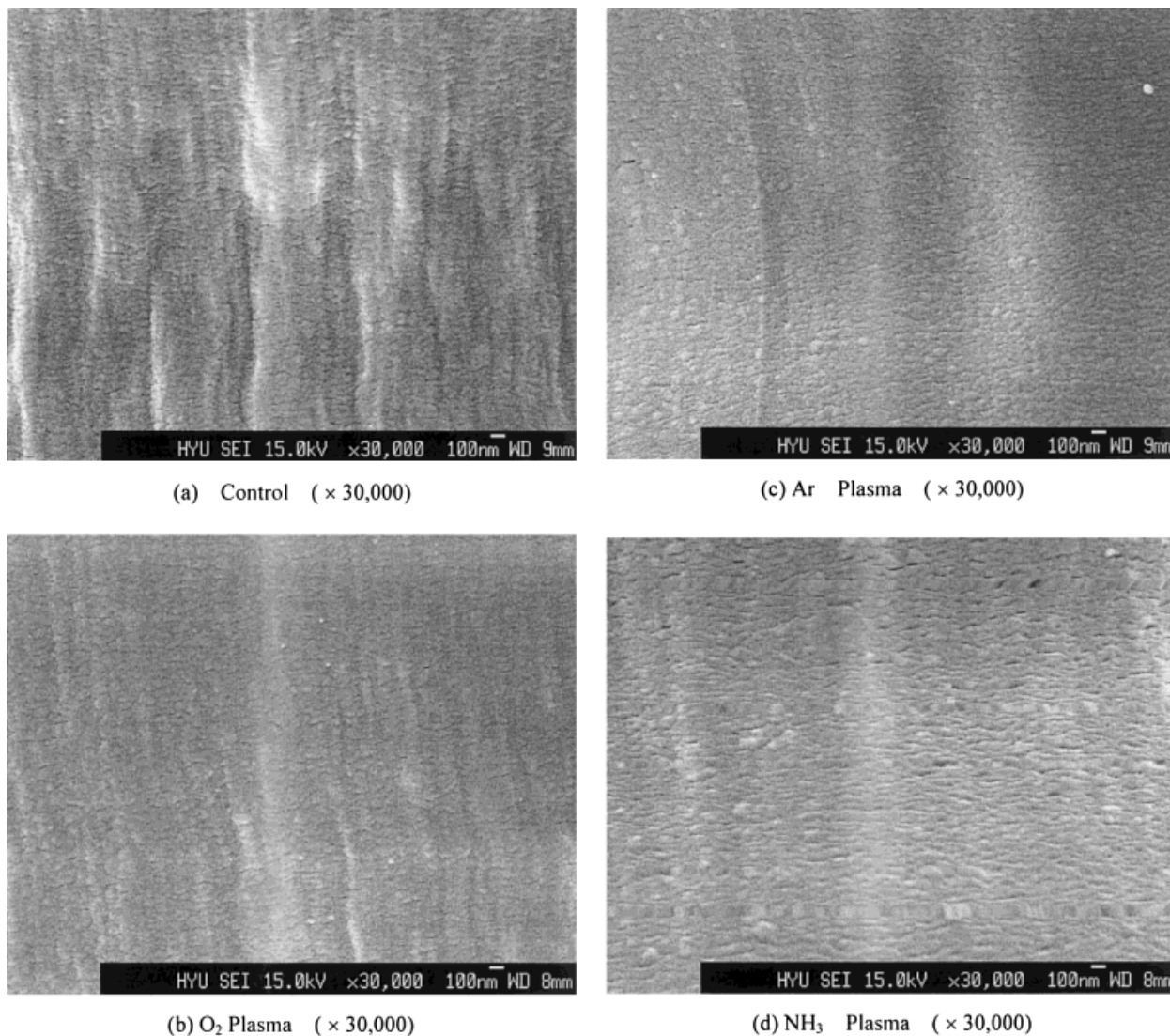
## EXPERIMENTAL

### Materials

Scoured nylon 6, plain-weave fabrics (70 d × 70 d, 43 × 30 cm<sup>-1</sup>) obtained from the Korea Apparel Testing Research Institute (KATRI) (Korea) were washed with distilled water. Aniline (Shiny Pure Chemicals Co., Ltd.) was distilled under vacuum prior to use. Ammonium peroxydisulfate (Junsei Chemical Co., Ltd.) was selected as an oxidant, and hydrochloric acid (HCl; Duksan Pure Chemical Co., Ltd.) was used as a dopant.

### Fabric Treatment

Conductive PANi-nylon 6 composite fabrics were prepared by the following process. Oxygen, ammonia, and argon gases were used for the plasma



**Figure 2** Longitudinal view of FESEM surface photographs of plasma-treated nylon 6 fiber at 30,000 $\times$  magnification: (a) control, (b) oxygen, (c) ammonia, and (d) argon.

treatment of nylon fabric. Plasma treatment was performed *in vacuo* at 100 Torr for 10 min with radio frequency (RF) power at 60 W.

Freshly distilled aniline was dissolved in a 0.35M HCl solution for diffusion. Fabrics were immersed in an aqueous hydrochloride solution of

**Table I** Atomic Composition Ratio of Nylon 6 and Plasma-Treated Nylon 6 Surfaces Measured by XPS

Core Level	Sample			
	Control	O <sub>2</sub> Plasma Treatment	NH <sub>3</sub> Plasma Treatment	Ar Plasma Treatment
N <sub>1s</sub> /C <sub>1s</sub>	0.075	0.051	0.094	0.087
O <sub>1s</sub> /C <sub>1s</sub>	0.301	0.364	0.349	0.378

**Table II** Deconvoluted Peak Assignment of the XPS Spectra of Plasma-Treated Nylon 6 Fabrics<sup>27,28</sup>

Functional Group	Binding Energy (eV)		
	C <sub>1s</sub>	N <sub>1s</sub>	O <sub>1s</sub>
C—C	284.6	—	—
C—N	285.4	—	—
C—O, C—OH	286.1	—	531.6
CONH	287.9	399.4	532.3
C=O	—	—	531.1

aniline at 40°C for 2 h. Successive polymerization was then initiated with a mixture of the same volume of the oxidant/dopant solution containing ammonium peroxydisulfate and HCl in the diffusion bath. The polymerization bath contained a 1/0.7/1 molar ratio of aniline, ammonium peroxydisulfate, and HCl, respectively. Polymerization was carried out at 5°C for 30 min, and the fabrics were then vacuum-dried.<sup>2</sup> The schematic procedure used to prepare the PAN-nylon 6 composite fabric is shown in Figure 1.

### Evaluation

The sorption property of aniline was measured by the weighing method on the basis of the weight changes of the fabric before and after treatment. The conductivity of the composite fabric was measured by the four-line probe method<sup>17,18</sup> with Keithley 238 source-measure units, which are fully programmable instruments capable of sourcing and measuring voltage or current simultaneously with  $\pm[0.028\% + 450 \mu\text{V} + (\text{input current/full-scale current}) \times 600 \mu\text{V}]$  accuracy. Four equally spaced line probes were used. A constant current source was used to pass a steady current through the two outermost probes, and the voltage drop across the inner two was measured. The conductivity of the sample was given by the following formula:

$$\sigma \text{ (S/cm)} = \frac{1}{dW} \cdot \frac{I}{V}$$

where,  $\sigma$  is the electric conductivity,  $l$  is the spacing between probes (0.3 cm),  $d$  is the thickness of the sample (i.e., the fabric),  $W$  is the length of the line probe (1 cm),  $I$  is the current passed through

outer probes, and  $V$  is the voltage drop across inner probes.

The XPS measurements were performed with an SSI 2803 spectrophotometer from Surface Science Instruments. This spectrometer, from VG Microtech, Ltd., was equipped with an Al K $\alpha$  X-ray source. The X-ray source was 12 kV and 20 mA. All core-level spectra were obtained at a photoelectron takeoff angle of 35° with respect to the sample surface. To compensate for the surface-changing effect, all binding energies were referenced to a C<sub>1s</sub> neutral carbon peak at 284.6 eV. XPSPEAK 95 version 3.1 (*XPS Peak Fitting Program for WIN95* produced by Raymond W. M. Kwok, Chinese University of Hong Kong) was used for the XPS peak deconvolution. Two parameters were needed for this program, such as the peak position assignment and the selection of distribution statistics, and the Gaussian distribution was used for the calculation of each specific peak. The full-width half-maximum for the optimal deconvolution and the area of the subpeaks were calculated with the XPSPEAK 95 deconvolution software. The main algorithm used in this software is Newton's method for optimization, and Shirley and Linear background was used. Only the peak position was assigned by an operator, and the same values for all other parameters were used for every XPS chart to investigate the consistent intensity of the functional groups. Differential scanning calorimetry (DSC) studies were performed on a DuPont DSC 2100V 4.1C system under a nitrogen atmosphere with a programmed heating of 10°C/min. JEOL JSM-6340F field emission scanning electron microscopy (FESEM) was used for high-magnification observation, and a JEOL JSM-6300 was employed for study by scanning electron microscopy (SEM).

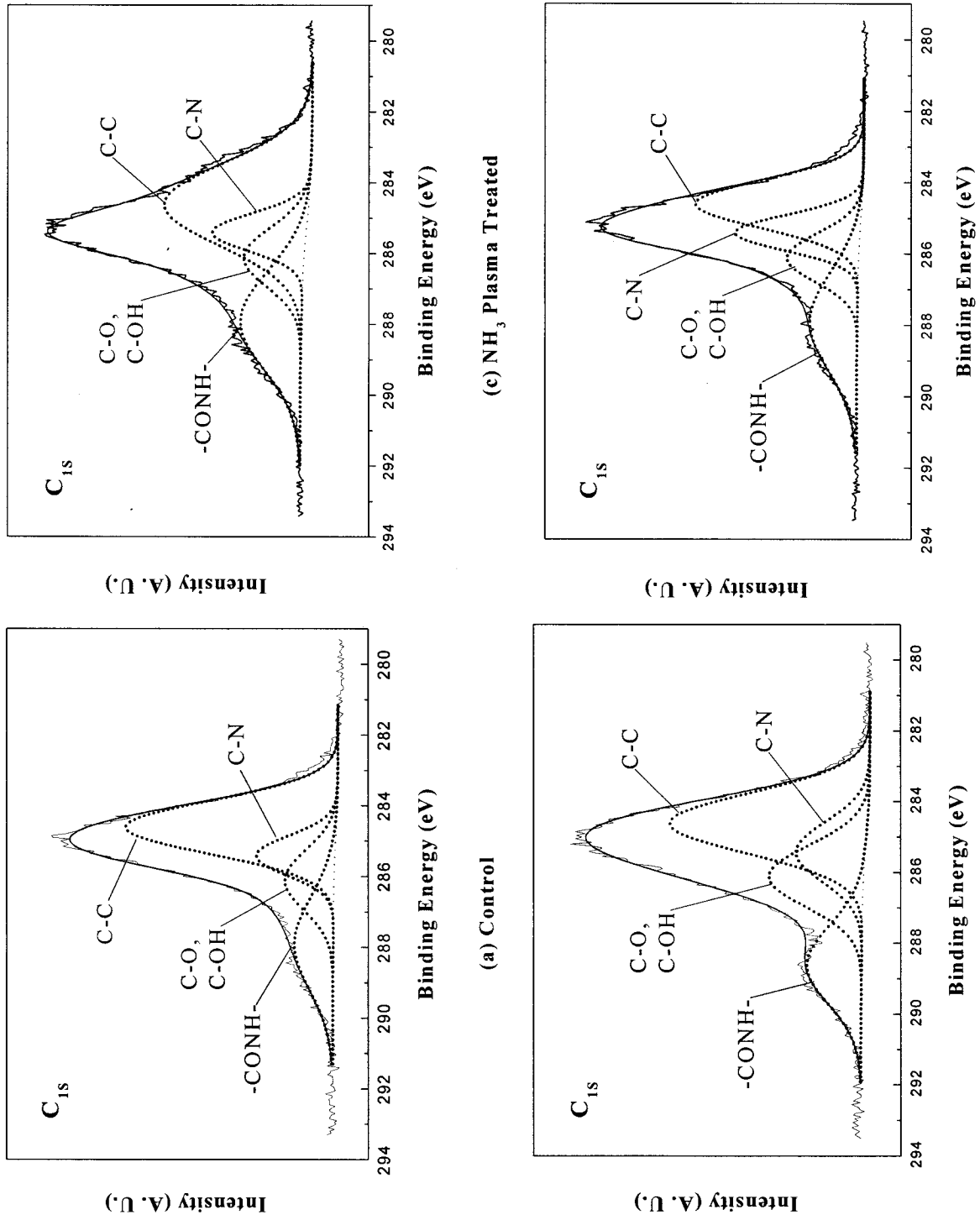
The serviceability of the conductive fabrics as a clothing material was also evaluated in terms of the conductivity stability against abrasion (AATCC 8-1988) and washing (AATCC 16-1986).

## RESULTS AND DISCUSSION

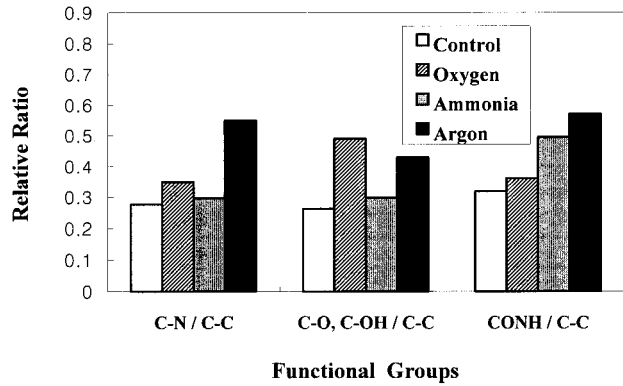
### Surface Characteristics

Plasma surface treatment can be an effective method to increase aniline deposition and polymerization on nylon 6 fabric because the surface properties of plasma-treated nylon are highly dependent on the selection of treatment gases.<sup>13</sup> After the careful selection of the type of treatment





**Figure 3** XPS C<sub>1s</sub> core-level spectra of plasma-treated nylon 6 surfaces at a takeoff angle of 35°: (a) control, (b) oxygen, (c) ammonia, and (d) argon.

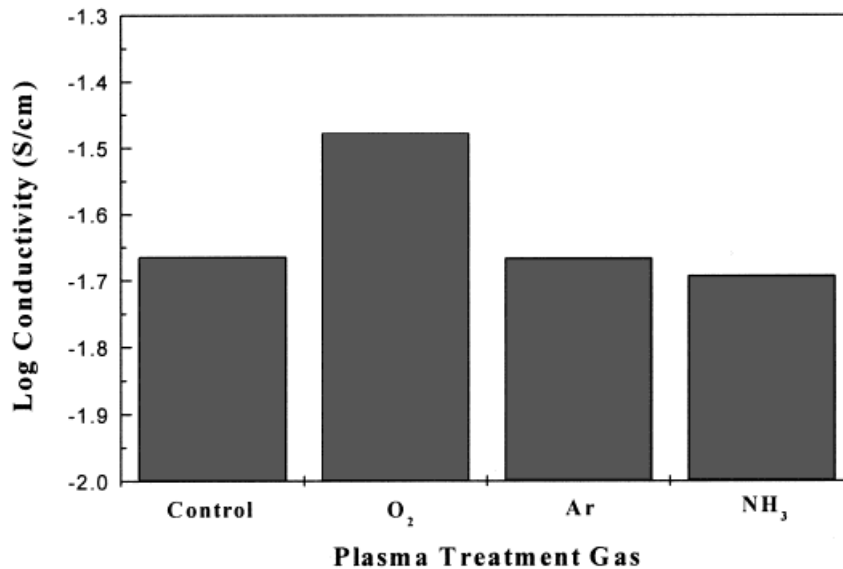


**Figure 4** Relative composition changes of functional groups with various plasma treatments ( $C_{1s}$ ).

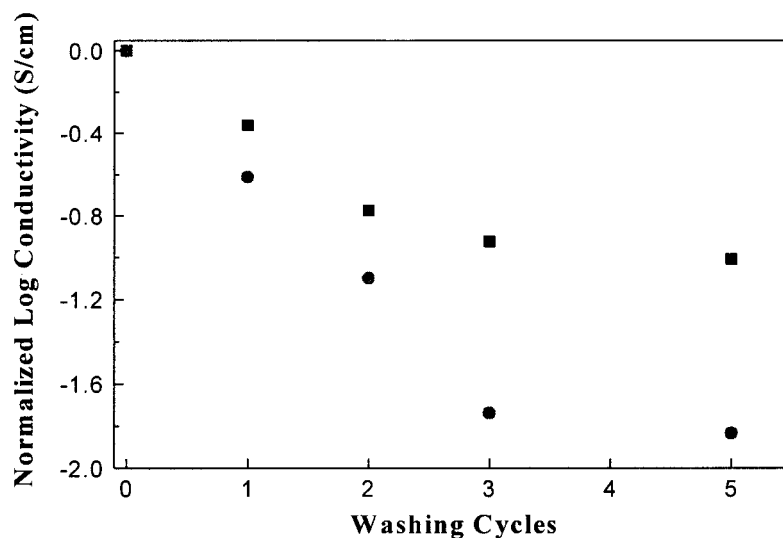
gas, oxygen, ammonia, and argon plasma surface treatments were performed on the nylon 6 fabrics. The morphology changes in the nylon 6 fiber surface with various gases were observed with FESEM, as shown in Figure 2. Clean and even surfaces were observed at  $30,000\times$  FESEM photos of the plasma treatments. The control sample had an irregular surface originally caused by the fiber spinning process shown in Figure 2(a). Atomic composition ratios of nylon 6 and plasma-treated nylon 6 surfaces were calculated from the data obtained by XPS, as shown in Table I. Plasma treatment produced nitrogen and oxygen atoms on the nylon 6 fabrics.  $N_{1s}$  and  $O_{1s}$  core levels were divided by the  $C_{1s}$  core-level intensity

to compare atomic species produced on the nylon 6 surfaces by each plasma treatment. The relative  $O_{1s}/C_{1s}$  core-level intensity of the control was 0.301, and that of oxygen-plasma-treated nylon was 0.364. More oxygen species were produced by oxygen plasma treatment, which might have enhanced aniline polymerization on the nylon 6 fiber surface. The fiber surface treated by ammonia plasma had more nitrogen species, whose relative  $N_{1s}/C_{1s}$  core-level intensity was 0.094, which was higher than that of the control and the oxygen-plasma-treated nylon. Like ammonia-plasma-treated nylon, the  $O_{1s}/C_{1s}$  and  $N_{1s}/C_{1s}$  relative core-level intensities of nylon were increased by argon plasma treatment.<sup>19,20</sup> The argon plasma treatment was an inert treatment and played the role of surface cleaning and etching.<sup>21,22</sup> With argon plasma treatment,  $N_{1s}/C_{1s}$  increased to 0.087 and  $O_{1s}/C_{1s}$  increased to 0.378 with respect to those of the control. Exposure to argon plasma was known to cause a substantial amount of oxygen incorporation into the polymer surface,<sup>13</sup> and this could apply to the graft polymerization of PAn.<sup>23–26</sup>

$C_{1s}$  core-level spectra of nylon 6 and plasma-treated nylon 6 were deconvoluted with four components, and the deconvoluted binding energy assignment is shown in Table II. The XPS spectra were also deconvoluted for the specific functional groups, as shown in Figure 3. For oxygen-plasma-treated nylon 6 fabric, the intensity of C—O and



**Figure 5** Effect of plasma treatment on the electrical conductivity of PAN-nylon 6 fabrics.



**Figure 6** Variation of the electrical conductivity of PAn-nylon 6 fabrics with washing cycles with oxygen plasma treatment (■) and without plasma treatment (●).

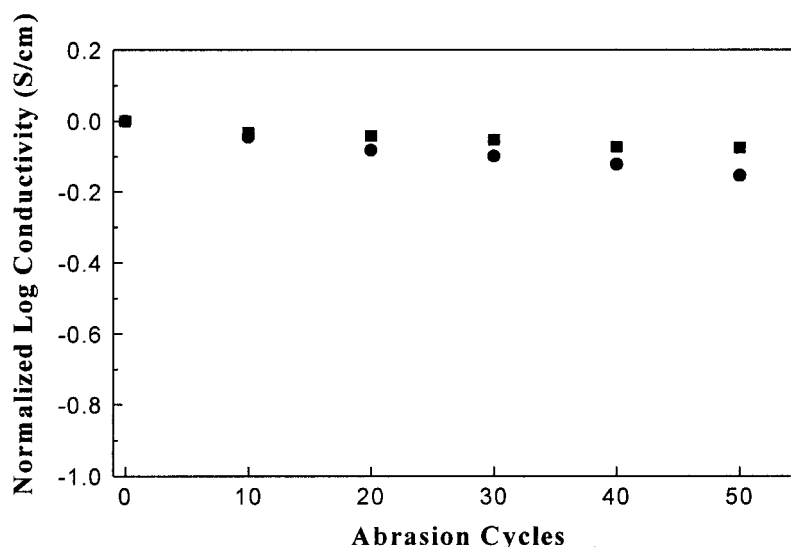
C—OH was higher than those of the control, whereas the intensities of C—N for ammonia and argon plasma treatments were greater than for the control and oxygen-plasma-treated nylon 6 fabrics.

A ratio of the intensity of specific functional groups to the intensity of carbon-to-carbon (C—C) bonding was calculated for the control and the plasma-treated samples, as shown in Figure 4. Relative ratios of C—O and C—OH groups to C—C groups for the oxygen-plasma-treated and argon-plasma-treated nylon largely increased

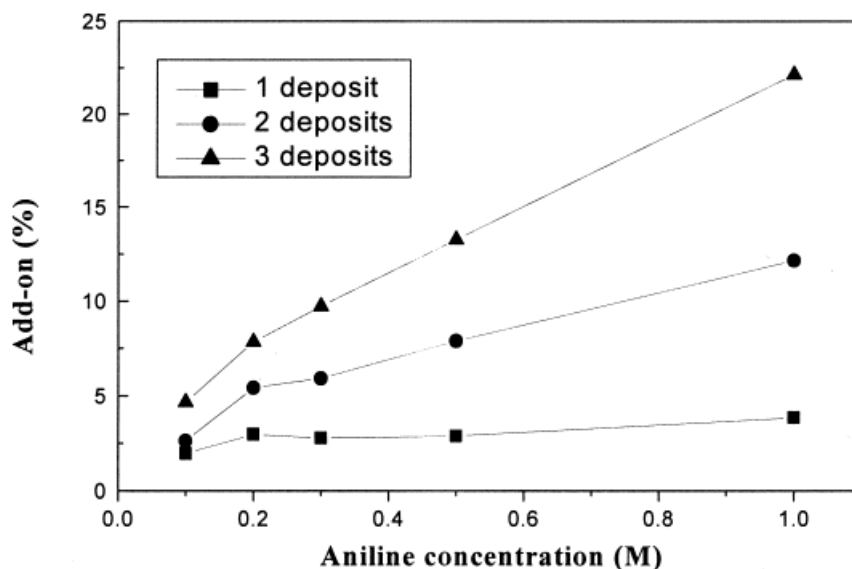
with respect to those of the control and ammonia-plasma-treated fabrics. The relative ratio of CONH groups to C—C groups increased with ammonia plasma treatment.

#### Fabric Conductivity and Its Durability

The electric conductivity of plasma-treated and PAn-deposited nylon 6 fabrics was measured with the four-probe method.<sup>17</sup> Oxygen, ammonia, and argon plasma treatments were performed on ny-



**Figure 7** Variation of the electrical conductivity of PAn-nylon 6 fabrics with abrasion cycles with oxygen plasma treatment (■) and without plasma treatment (●).

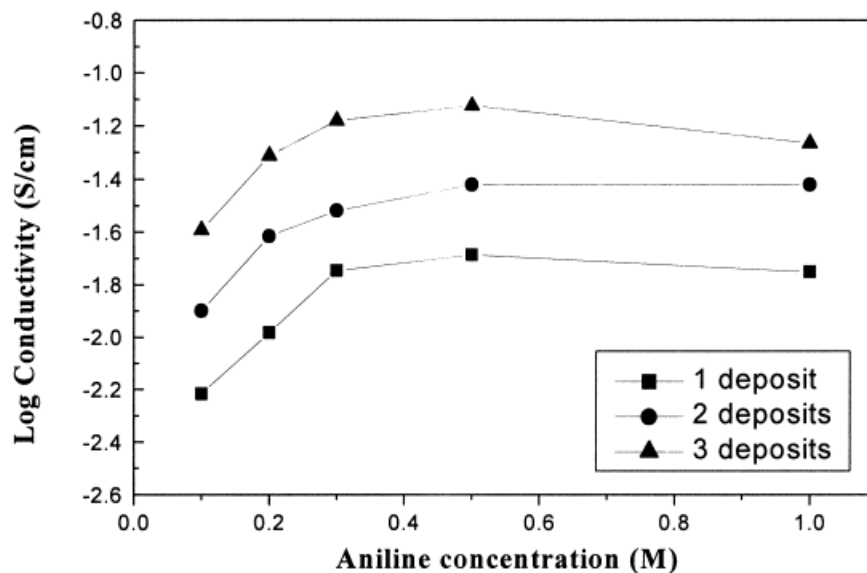


**Figure 8** Percentage add-on of PAN-nylon 6 fabrics as a function of multiple deposits and monomer concentration.

lon 6 fabric, and then aniline was polymerized on the plasma-treated fabric. Oxygen-plasma-treated and PAN-deposited nylon 6 fabric showed the highest conductivity, as shown in Figure 5. It seems that acidic functional groups, such as C—OH, C=O, and C—O, produced by oxygen plasma treatment enhanced the degree of polymerization of PAN on the nylon.

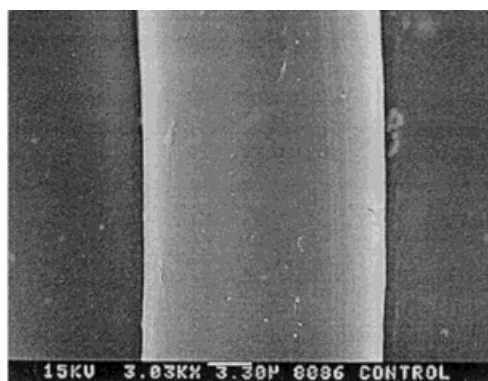
Even though lots of microgrooves were formed by ammonia plasma treatment, fabric conductiv-

ity was not increased and was rather slightly decreased in comparison with the control fabric. We assumed that basic amide and amine groups produced by ammonia plasma treatment reduced the acid doping level of the PAN on the fiber surface. Consequently, the fabric conductivity decreased because the conductivity depended strongly on the acid doping level of the polymer.<sup>11</sup> Protonic acid doping for an emeraldine base with amine and imine groups to conductive emeraldine

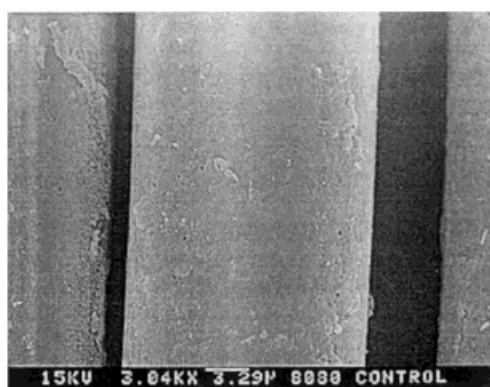


**Figure 9** Variation of the electrical conductivity of PAN-nylon 6 fabrics with multiple deposits and various monomer concentrations.

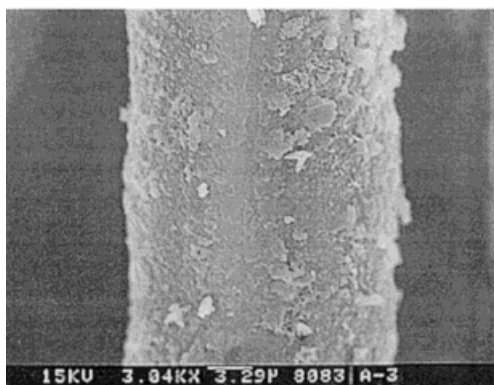




(a) control



(b) 1 deposit



(c) 3 deposits

**Figure 10** Longitudinal view of SEM surface photographs of nylon fiber with 0.5M aniline: (a) control, (b) one deposit, and (c) three deposits.

hydrochloride needed acidity. However, the ammonia plasma treatment produced basic amide and amine groups. Therefore, the basic surface by ammonia plasma treatment was not helpful for increasing the PAn doping level and conductivity.

The stability of the conductivity of PAN–nylon 6 composite fabric against multiple washing and abrasion was investigated. Aniline (0.5M) was polymerized on oxygen-plasma-treated and untreated nylon 6 fabric. Degrees of fabric conductivity depression were observed under normal home-laundry conditions when a 0.1% neutral detergent solution was used as the washing medium. As shown in Figure 6, the overall fabric conductivity gradually decreased with each washing cycle. From the previous study,<sup>11</sup> we assumed that the degradation of fabric conductivity by multiple washings depended on the degree of anion dedoping and the removal of the PAn deposit from the fabric surface. Because samples were washed under the same conditions, we expected that the difference in the change of fabric conductivity between plasma-treated and untreated samples was mainly caused by the adhesion of PAn over the fabric rather than anion dedoping. Therefore, the fact that the degree of the conductivity depression for the fabric treated with oxygen plasma was less than that of the untreated fabric indicated that good adhesion of the PAn layer on nylon 6 was formed with oxygen plasma treatment. Furthermore, as shown in Figure 7, no significant changes in the conductivity were observed after the composite fabrics were abraded over 50 cycles. It also confirmed that good adhesion of the PAn layer on nylon 6 formed.

### Multiple Deposits

Because oxygen was revealed to be a good plasma medium, the following experiment was carried out after the fabric was treated with oxygen plasma. To control fabric conductivity and cover as wide a range of conductivity as possible, we studied the effect of the monomer concentration and number of deposits on the fabric conductivity. The molar ratio of the concentrations of monomer, dopant, and oxidant was fixed as 1/0.7/1, respectively; this was revealed to be an optimum condition in a previous study.<sup>11</sup> The monomer concentration varied from 0.1 to 1M, and the number of deposits varied from one to three. After each deposit process, fabric was dedoped with 0.1M NH<sub>4</sub>OH and redoped with 0.35M HCl to remove irregular deposits of oligomer or monomer that might cause a depression of conductivity. As shown in Figure 8, the percentage add-on increased as the concentration and number of deposits increased. We derived Figure 9 by taking the conductivity reached for one, two, and three

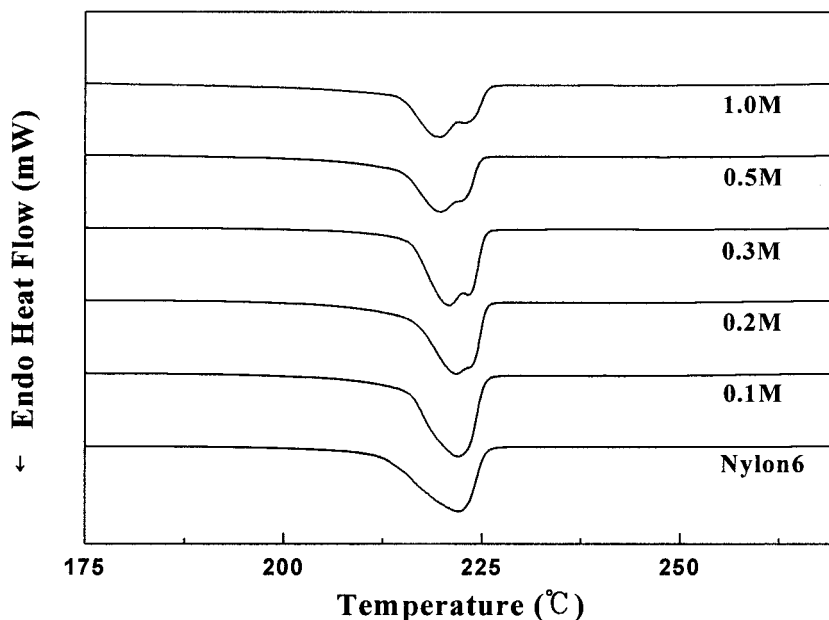


Figure 11 DSC thermograms of nylon 6 and PAN-nylon 6 fabrics.

deposits with various aniline concentrations. This allowed the determination of the reactant solution concentrations and the number of deposits used to achieve a selected conductivity. Fabric conductivity increased as the monomer concentration increased up to  $0.5M$  and then leveled off, and further increases were achieved with an increase in the number of multiple deposits. Fabric log conductivity in the range of  $-2.2$  to  $-1.6$  S/cm was reached with a single deposit,  $-1.9$  to  $-1.4$  S/cm was reached with double deposits, and  $-1.6$  to  $-1.1$  S/cm was reached with triple deposits. Overall fabric conductivity higher than  $-1.6$  S/cm could be obtained with an increase in the number of deposits.

Figure 10 shows the surface morphology of the fiber coated with single and triple deposits of PAN. It reveals that the thickness of the PAN

coating increases and the fiber surface becomes less uniform as the number of PAN deposits increases. The surface of the PAN coating appears coherent. The diameter difference between the control nylon fiber and single-coated and triple-coated fibers with PAN were  $0.66$  and  $2.43$   $\mu\text{m}$ , respectively, showing that the conductive PAN coatings on the PAN-nylon 6 composite fiber were approximately  $0.33$  and  $1.22$   $\mu\text{m}$  thick.

#### Thermal Studies

The thermal properties of the PAN-nylon 6 composite fabric prepared by three deposits of aniline of various concentrations after oxygen plasma treatment were analyzed; they are compared in Table III and Figure 11. The heat of fusion and degree of crystallinity of PAN-nylon 6 fabrics

Table III Variation of Heat of Fusion, Melting Temperature, and Crystallinity of PAN-Nylon 6 Composite Fabrics (Three Deposits)

Aniline Concentration ( $M$ )	Heat of Fusion: $\Delta H_f$ (J/g)	Melting Temperature: $T_m$ ( $^{\circ}\text{C}$ )	Degree of Crystallinity $X_c$ (%)
Nylon 6	85.6	222.0	33.7
0.1	79.2	222.0	33.3
0.2	72.5	221.8	31.0
0.3	70.4	220.8	30.7
0.5	64.7	219.8	30.2
1.0	59.7	219.7	27.6

slightly decreased as the aniline concentration increased. The melting temperature of the composite fabric also tended to shift to a slightly lower temperature as the aniline concentration increased. Diffusion of aniline into the nylon 6 fabric, to some extent, might disturb the crystalline region and decrease its thermal properties. At the high aniline concentrations over 0.2M, the endothermic peaks had a shoulder. This was supposed to be caused by hydrogen bonding between nylon 6 and PAn. PAn powder was measured in DSC; thermal transition peaks were not shown from 200 to 235°C. Therefore, the double endothermic peaks were not from PAn.

## CONCLUSIONS

The effect of plasma treatment gas on the electrical conductivity and performance of PAn-nylon 6 fabrics was studied. Oxygen-plasma-treated nylon fabric retained the highest conductivity and PAn deposit. The surface of nylon 6 fibers was strongly etched by oxygen plasma treatment, and various functional groups such as COOH and C=O could be produced on the nylon 6 fiber surface, which was confirmed by XPS. Oxygen was revealed as the most aggressive and effective plasma gas compared with ammonia and argon plasma in this system. The conductivity of oxygen-plasma-treated PAn-nylon 6 fabric was more stable with repeated washing and abrasion cycles than PAn-nylon 6 fabric without plasma pretreatment.

The effects of the monomer concentration and multiple deposits of PAn on the fabric morphology, conductivity, and durability were also investigated. Fabric conductivity increased as the monomer concentration increased up to 0.5M and then leveled off, and a further increase could be achieved with an increase in the number of multiple deposits. Fabric log conductivity in a range of -2.2 to -1.6 S/cm could be reached in a single deposit, -1.9 to -1.4 S/cm could be reached in two deposits, and -1.6 to -1.1 S/cm could be reached in three deposits.

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