Modifying Nylon and Polypropylene Fabrics with Atmospheric Pressure Plasmas

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ABSTRACT

Polypropylene and nylon 66 fabrics are subjected to atmospheric pressure He and He-O2 plasmas for selected exposure time intervals. Scanning electron microscopy analysis of the fabrics shows no apparent changes in the plasma-treated nylon fiber surfaces, but significant surface morphological changes for the polypropylene. Surface analyses of the nylon filaments reveal small differences in the surface carbon and oxygen contents between the treated and control groups. The surface oxygen and nitrogen content of the polypropylene fabric increases significantly after treatment in both He and He-O2 plasmas. There is a slight decrease in nylon fabric tensile strength after treatment in He plasma for 3 minutes, while there is no significant change in tensile strength of the nylon fabric treated with He-O2 after exposure times of up to 8 minutes.

Plasma treatment of textile fabrics and yarns is being investigated as an alternative to wet-chemical fabric treatment and pretreatment processes, e.g., shrink resistant (wool) or water-repellent finishing, which tend to alter fabric mechanical properties and are environmentally hazardous. Plasma treatment of textiles may result in desirable surface modifications, including but not limited to surface etching, surface activation, crosslinking, chain scission, decrystallization, and oxidation. Treatment depends on the choice of working gas and plasma density and energy [1, 3, 6, 14, 17, 19]. The effects of low-pressure (vacuum) plasma treatments of polyester, high performance polymers, and wool are well documented [3, 11, 17, 19], while relatively little has been published about atmospheric pressure plasma treatments [4, 14, 17].

Despite the effectiveness of plasma treatments on textiles, the inability to successfully incorporate low-pressure plasma treatment equipment into a continuous textile finishing operation has seriously limited the commercial viability of the technique [11]. Previously, plasmas generated at atmospheric pressure were believed to be neither uniform nor stable; however, recent research on atmospheric plasmas has shown their uniformity, stability, and applicability to processing and treatment of textiles, as well as many other materials [2]. In addition, atmospheric pressure plasma treatment devices can be integrated with continuous on-line processing of textile materials.

While plasma treatment may result in many favorable or desirable surface modifications of textile materials, it may also cause undesirable physical degradation of the polymer surface [5, 10, 11]. Treatment parameters must be optimized in order to achieve desirable surface modifications without changing bulk properties.

This paper presents results on polypropylene and nylon 66 fabrics treated with atmospheric plasmas of helium (He) and a mixture of helium and oxygen (He-O2). Helium is typically used to initiate and generate the plasma at atmospheric pressure before another gas is introduced to the system. If there are no measurable changes in surface or mechanical properties as a result of exposure to the He plasma, then all changes in the treated material can be attributed to the second gas introduced to the system, e.g., O2, or any other reactive gas. Oxygen is used frequently to control, optimize, and modify treatments with other gases during substrate exposure to plasma, and therefore, interactions of O2 plasmas with textile materials must be well characterized.
Experimental

The nylon 66 was a plain-weave unfinished fabric with 16.8 picks/cm (42 picks/in.), 16.1 ends/cm (41 ends/in.), and a yarn linear density of 72.4 tex. A residual spin finish accounted for 0.73% of total fabric weight. All samples were cut to a size of 15.24 × 15.24 cm (6 × 6 inches).

The polypropylene fabric was an unfinished nonwoven spunbond-meltspun-spunbond (SMS) fabric, which is extremely hydrophobic and unwettable. The basis weight was approximately 75 g/m².

Plasma Treatment

Atmospheric plasma treatments were performed in a chamber designed and fabricated by the Nuclear Engineering Department at North Carolina State University [7, 8, 9]. This capacitively-coupled plasma device (Figure 1) operates at low frequencies (audible) between 1 and 12 kHz. The voltage across the plates can be up to 7.8kV_{rms}, and the electrode gap spacing is adjustable. Plasma stability was achieved during operation in the frequency range of 6-9 kHz. The circuit diagram of the device is shown in Figure 2, where the plasma is shown by its equivalent lumped circuit elements. The plasma is represented by its resistance $R_p$, capacitance $C_p$, and inductance $L_p$, while the capacitances of the sheaths and the dielectric are shown as $C_s$ and $C_d$, respectively. This equivalent circuit is more common in modeling capacitively-coupled plasma devices, but some other models may incorporate all capacitors in one equivalent capacitance. Such plasma models may be more complex and are beyond the scope of this paper.

Treatments were conducted with plasmas generated from both He and O₂ gases; however, with the device operating at atmospheric pressure, some air remained inside the plasma chamber. While air is a small but significant fraction, He or O₂ gas dominates the plasma, respectively. For the O₂ treatments, a plasma was initially generated using He gas (it requires less potential for gas breakdown and has self-sustained discharge conditions), then O₂ was flushed into the system. Three different O₂ flow rates were used in the experiment (30, 60, and 90 sccm). The plasma stabilized at a RF frequency of 7.5 kHz with the He pressure at 34.5 kPa (5 psi). These values were held constant throughout the experiment, and only the sample exposure time varied between 0.5 and 3 minutes at 30-second intervals. Power levels were 11.3 kVrms (low power) and 13.3 kVrms (high power).

Additional nylon samples were treated for periods of 1, 2, 4, and 8 minutes in an O₂ plasma at a flow rate of 90 sccm, at low and high power. This set of experiments was evaluated with tensile testing only.

Polypropylene nonwoven fabrics were treated with He and He-O₂ gas plasmas with the same treatment parameters used for the nylon fabrics, at high power. In order to compare them to nylon fabrics, the experiments were set to evaluate the effect of the flow rate of O₂ gas (30, 60, and 90 sccm). Polypropylene samples were exposed for 0.5 and 4 minutes.

Microscopy and Spectroscopy

Nylon samples exposed to He and He-O₂ plasma were analyzed with a Hitachi scanning electron microscope (SEM), model S-3200 (variable pressure SEM). The fabrics were inspected at magnifications of up to 8000× at 10.0kV to see if there was any evident change of fiber surface morphology due to plasma treatments.

For the x-ray photoelectron spectroscopy, nylon 66 and polypropylene samples exposed to He-O₂ plasma were analyzed with a Riber XPS system with Mg K-alpha x-ray (1,253.6 eV, non-monochromated) from a Mg-Al dual source and a Mac 2 cylindrical mirror analyzer. Survey scans from 0 to 1,200 eV were acquired with a step size of 1.0 eV and a dwell time of 0.1 second. Narrow scans of individual photopeaks were acquired with a step size of 0.1 eV and a dwell time of 0.2 second.
Fabric Tensile Test and Analysis

The tensile test specimens were prepared according to ASTM method D-5035, Standard Test Method for Breaking Force and Elongation of Textile Fabrics (strip force). Only nylon fabrics were tested for tensile properties. The fabrics were cut to 15.24 x 3.81 cm (6 x 1.5 in.), and 6.35 mm (0.25 in.) of warp or filling yarn (depending on direction) was unraveled off the width on either side. The tensile test was conducted with a Sintech universal mechanical test machine, model I/S, with a 4449 N (1000 lb) load cell at a constant strain rate of 30.48 cm/min (12 in./min).

The tensile test results were analyzed with a one-way analysis of variance (ANOVA) and Fisher pairwise comparisons [13]. A level of 0.05 was adopted to test the statistical significance in all analyses of fabric tensile strength.

Results and Discussion

Scanning Electron Microscopy

The effect of plasma treatment morphological changes on untreated and treated samples is shown in Figures 3-12. Untreated nylon filament (Figure 3) showed a relatively smooth surface with stripes (aligned grooves, presumably an artifact of the fiber production process). Our examination of the He treated nylon revealed filament surfaces that appeared grainy, with wearing of the grooves and significant accumulation of particulate matter (Figure 4). The presence of particulate matter on the surfaces of filaments may be attributed to the residual spin finish. There were similar changes in the surface appearance after He-O₂ plasma treatments (Figures 5-7).

FIGURE 4. He treated nylon 66, 2 minutes and medium power.

All polypropylene samples treated with either He or He-O₂ gas plasmas exhibited significant surface morphological changes. Untreated filaments (Figure 8) had a smooth surface, while He gas treatment resulted in a slight increase in surface rippling and waviness (Figure 9). With He-O₂ gas treatment, the surface of samples changed dramatically (Figures 10-12). The surface texture became somewhat gossamer in appearance, as if it were a separate thin layer superimposed on the filament. This layer was highly textured, with intense rippling and roughening. Our results were consistent with those of Tsai et al. [14]. Surface changes appeared more pronounced with increased flow rates and exposure times (Figures 11 and 12).
Figure 5. He-O$_2$ treated nylon 66, 60 scem, 0.5 minute, and medium power.

Figure 6. He treated nylon 66, 0.5 minute, and high power.

Figure 7. He-O$_2$ treated nylon 66, 60 scem, 0.5 minute, and high power.

Figure 8. Untreated polypropylene.
Figure 9. He treated polypropylene, 0.5 minute, and high power.

Figure 10. He-O₂ treated polypropylene, 30 sccm, 0.5 minute, and high power.

Figure 11. He-O₂ treated polypropylene, 60 sccm, 0.5 minute, and high power.

Figure 12. He-O₂ treated polypropylene, 30 sccm, 4.0 minutes, and high power.
ANALYSIS OF SURFACE ELEMENTAL COMPOSITION

Alterations of polymer surface chemistry have been reported as a result of treatment in air and other etchant gases, including oxygen [14]. The results of surface elemental analyses of the nylon and PP fabric surfaces are shown in Tables I and II.

Padhye et al. achieved etching of nylon 66 film with low pressure oxygen gas plasma [10]. Following our atmospheric He-02 gas plasma treatment, there was a very slight difference in the surface elemental composition of the nylon treated and control groups. Table I shows that the untreated nylon 66 surface appears to contain no nitrogen groups. This result suggests that the presence of a residual spin finish on the nylon filaments may be a factor in the effectiveness of plasma surface treatment of unfinished fabrics. For the nylon samples, O/C ratios did not appear to be related to power level or flow rate of He-O2 gas. This result is consistent with reports of the surface composition of nylon exposed to low-pressure (vacuum) plasma treatments [16, 18]. The nature of the plasma-polymer interaction has not been definitively characterized. Covalent bonds may be broken as a result of exposure to UV radiation, photons, charged particles, ions, and free radicals [14]. The remaining chain ends may then become crosslinked through formation of C-C or other covalent bonds [18].

PP showed remarkable increases in surface oxygen and nitrogen content. The O/C ratio of He-O2 gas treated samples increased over seven times. The methyl group on the PP polymer appears to be highly susceptible to bond cleavage, and may be replaced by other functional groups including carboxyl, carbonyl, and hydroxyl groups [14].

Untreated nylon samples had higher O/C ratios than untreated PP, which is consistent with the constitution of the two polymers and the presence of residual spin finish on the nylon 66 fabrics. After He-O2 plasma treatment, the O/C and (O+N)/C ratios of PP were higher than those for the nylon samples.

He gas treatment at high power increased the O/C and N/C ratios on both nylon 66 and PP. This is most likely a result of the small but significant fraction of air remaining in the chamber during plasma treatment. This increase of O/C ratio following exposure to He gas plasma also appeared in PP samples.

For the PP samples, the O/C ratio appeared to increase at low flow rates and exposure times, then decrease somewhat for higher flow rates and exposure times. The increased flow rate may lead to flushing of the plasma from the chamber, thus reducing the density of activated species. It is well known that ablation and chemical reaction occur simultaneously in the plasma state [3, 6, 14, 19]. At higher exposure times, ablation may dominate surface treatment. The severity of surface roughening greatly increases at higher exposure times (Figure 12). Similar effects have been demonstrated for atmospheric plasma treatment of PP films [4].

### Table I. XPS results for nylon 66 treated in He-O2 plasma.

<table>
<thead>
<tr>
<th>Power level</th>
<th>Gas</th>
<th>Time, min.</th>
<th>Flow rate, sccm</th>
<th>C15 C, %</th>
<th>O15 O, %</th>
<th>N15 N, %</th>
<th>O/C</th>
<th>N/C</th>
<th>(O+N)/C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Untreated</td>
<td>He</td>
<td>0.5</td>
<td>30</td>
<td>89.0%</td>
<td>11.0%</td>
<td>0.0%</td>
<td>0.121</td>
<td>0.000</td>
<td>0.121</td>
</tr>
<tr>
<td>High</td>
<td>He</td>
<td>0.5</td>
<td>60</td>
<td>86.0%</td>
<td>13.0%</td>
<td>2.0%</td>
<td>0.146</td>
<td>0.019</td>
<td>0.165</td>
</tr>
<tr>
<td>Low</td>
<td>He-O2</td>
<td>0.5</td>
<td>90</td>
<td>88.0%</td>
<td>12.0%</td>
<td>0.0%</td>
<td>0.135</td>
<td>0.000</td>
<td>0.135</td>
</tr>
<tr>
<td>Low</td>
<td>He-O2</td>
<td>0.5</td>
<td>30</td>
<td>85.7%</td>
<td>14.3%</td>
<td>0.0%</td>
<td>0.167</td>
<td>0.000</td>
<td>0.167</td>
</tr>
<tr>
<td>High</td>
<td>He-O2</td>
<td>0.5</td>
<td>60</td>
<td>86.1%</td>
<td>13.9%</td>
<td>0.0%</td>
<td>0.162</td>
<td>0.000</td>
<td>0.162</td>
</tr>
<tr>
<td>High</td>
<td>He-O2</td>
<td>0.5</td>
<td>90</td>
<td>85.5%</td>
<td>14.5%</td>
<td>0.0%</td>
<td>0.169</td>
<td>0.000</td>
<td>0.169</td>
</tr>
</tbody>
</table>

### Table II. XPS results for polypropylene treated in He-O2 plasma.

<table>
<thead>
<tr>
<th>Power level</th>
<th>Gas</th>
<th>Time, min.</th>
<th>Flow rate, sccm</th>
<th>C15 C, %</th>
<th>O15 O, %</th>
<th>N15 N, %</th>
<th>O/C</th>
<th>N/C</th>
<th>(O+N)/C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Untreated</td>
<td>He</td>
<td>0.5</td>
<td>0</td>
<td>95.7%</td>
<td>4.3%</td>
<td>0.0%</td>
<td>0.045</td>
<td>0.000</td>
<td>0.045</td>
</tr>
<tr>
<td>High</td>
<td>He</td>
<td>0.5</td>
<td>30</td>
<td>88.6%</td>
<td>10.8%</td>
<td>0.6%</td>
<td>0.122</td>
<td>0.007</td>
<td>0.128</td>
</tr>
<tr>
<td>High</td>
<td>He-O2</td>
<td>0.5</td>
<td>60</td>
<td>81.5%</td>
<td>18.4%</td>
<td>1.5%</td>
<td>0.226</td>
<td>0.001</td>
<td>0.228</td>
</tr>
<tr>
<td>High</td>
<td>He-O2</td>
<td>0.5</td>
<td>90</td>
<td>76.1%</td>
<td>22.0%</td>
<td>1.9%</td>
<td>0.289</td>
<td>0.025</td>
<td>0.314</td>
</tr>
<tr>
<td>High</td>
<td>He-O2</td>
<td>4.0</td>
<td>30</td>
<td>79.3%</td>
<td>20.0%</td>
<td>0.7%</td>
<td>0.252</td>
<td>0.009</td>
<td>0.261</td>
</tr>
<tr>
<td>High</td>
<td>He-O2</td>
<td>4.0</td>
<td>90</td>
<td>84.4%</td>
<td>15.1%</td>
<td>0.5%</td>
<td>0.179</td>
<td>0.006</td>
<td>0.185</td>
</tr>
<tr>
<td>High</td>
<td>He-O2</td>
<td>4.0</td>
<td>90</td>
<td>83.4%</td>
<td>14.7%</td>
<td>1.9%</td>
<td>0.177</td>
<td>0.022</td>
<td>0.199</td>
</tr>
</tbody>
</table>
FABRIC STRENGTH MEASUREMENTS

Tensile strength data for nylon fabrics treated at various exposure times to the He and He-O_2 plasmas are presented in Tables III-V. The zero exposure time on the tables represents untreated samples. There was no significant decrease in strength for any of the treated samples, except in the case of the He treated fabric that was exposed for 3 minutes. However, there appeared to be slight increases in strength at short treatment times. This may be the result of chemical reactions with the oxygen gas, or a crosslinking reaction between polyamide molecules. Etching reactions are possible even in the He plasma, due to the presence of a small amount of air.

We also noticed a similar trend for samples treated at different power levels (Table V). At low treatment times, there was a slight increase in fabric tensile strength. Overall, there was no significant difference in tensile strength between treated and control groups.

| TABLE III. Tensile strength of plasma-treated nylon 66 fabrics. The letters represent the ranks of data in multiple comparison. Mean values marked with different letters are significantly different (P < 0.05). |
|---|---|
| Helium | Helium-oxygen - 30 sccm |
| Tensile strength, cN/tx | Tensile strength, cN/tx |
| Exposure time, min. | Sample size | Mean | Standard deviation | Sample size | Mean | Standard deviation |
| 0 | 10 | 72.681^a | 1.743 | 8 | 72.544^a | 1.93 |
| 0.5 | 10 | 71.117^ab | 4.175 | 8 | 75.187^a | 1.078 |
| 1.0 | 10 | 73.729^ab | 2.095 | 8 | 74.224^ab | 1.402 |
| 1.5 | 10 | 72.481^ab | 2.951 | 8 | 73.831^ab | 2.277 |
| 2.0 | 10 | 71.568^ab | 3.244 | 8 | 74.838^ab | 1.815 |
| 2.5 | 10 | 70.223^ab | 3.529 | 8 | 76.590^a | 1.636 |
| 3.0 | 10 | 69.177^ab | 3.887 | 8 | 74.109^ab | 4.511 |

| TABLE IV. Tensile strength of plasma-treated nylon 66 fabrics. The letters represent the ranks of data in multiple comparison. Mean values marked with different letters are significantly different (P < 0.05). |
|---|---|
| Helium-oxygen - 60 sccm | Helium-oxygen - 90 sccm |
| Tensile strength, cN/tx | Tensile strength, cN/tx |
| Exposure time, min. | Sample size | Mean | Standard deviation | Sample size | Mean | Standard deviation |
| 0 | 8 | 73.875^ab | 3.193 | 16 | 72.564^ab | 2.767 |
| 0.5 | 8 | 75.655^a | 2.083 | 16 | 74.850^ab | 1.776 |
| 1.0 | 8 | 74.854^ab | 2.278 | 16 | 75.008^ab | 2.491 |
| 1.5 | 8 | 74.307^ab | 1.888 | 16 | 74.450^ab | 2.568 |
| 2.0 | 8 | 74.363^ab | 1.739 | 16 | 74.612^ab | 2.088 |
| 2.5 | 8 | 75.092^a | 0.884 | 16 | 73.919^ab | 2.661 |
| 3.0 | 8 | 75.433^a | 1.746 | 16 | 73.966^ab | 3.083 |

| TABLE V. Tensile strength of plasma-treated nylon 66 fabrics. The letters represent the ranks of data in multiple comparison. Mean values marked with different letters are significantly different (P < 0.05). |
|---|---|---|
| Helium-oxygen - 90 sccm, low power | Helium-oxygen - 90 sccm, high power |
| Tensile strength, cN/tx | Tensile strength, cN/tx |
| Exposure time, min. | Sample size | Mean | Standard deviation | Sample size | Mean | Standard deviation |
| 0 | 4 | 72.536^a | 1.748 | 4 | 72.536^a | 1.748 |
| 1.0 | 4 | 75.247^ab | 1.768 | 4 | 76.752^a | 0.683 |
| 2.0 | 4 | 76.578^a | 0.474 | 4 | 78.339^a | 1.655 |
| 4.0 | 4 | 76.451^a | 1.358 | 4 | 75.342^ab | 2.763 |
| 8.0 | 4 | 73.930^ab | 1.589 | 4 | 75.722^ab | 0.684 |

Conclusions

The surface of the nylon 66 filament appears unchanged by exposure to atmospheric He-O_2 plasma treatment. He gas plasma treatment results in the appearance of wear on surface grooves and deposition of particulate matter. Conversely, PP surfaces are only slightly affected by exposure to He gas plasma, but the surface morphology is rough and rippled after treatment in He-O_2 plasma.

Surface elemental analyses indicate a slight increase in surface oxygen content in both He and He-O_2 treated nylon. Effects of the same treatment on the surface elemental composition of PP are profound. He-O_2 treatment of PP results in an increase of relative surface oxygen content (O/C) from close to zero to up to approximately 29%. These differences in treatment effects on nylon 66 and PP samples are due to their molecular structures and properties, chemical reactions occurring in atmospheric plasma, and plasma conditions. Additionally, the presence of residual spin finish on the nylon filaments may have an effect on the efficacy of the treatment.

Bulk properties of nylon are relatively unchanged as a result of plasma treatment. There is a small but significant decrease in nylon fabric strength for He plasma treatments at 3 minutes, but no other significant differences in fabric tensile strength as a result of any treatment. Since He is not a reactive gas, we believe this trend, even in He plasma, could stem from the component of air that is in the system at the time of treatment.

We have found that the He-O_2 plasma causes the tensile strength of nylon to increase slightly within the first 30 seconds of exposure time.

There is a great need for further investigation into plasma operational conditions at atmospheric pressure. Improved understanding of atmospheric plasma properties, as well as plasma-matter interactions, will lead to optimization and modeling of atmospheric plasma pa-
rameters for treatments on textiles (or any other sub-
strate).

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