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EXTRUDED FILMS FROM MODIFIED POLYPROPYLENE RESIN: DIELECTRIC AND BREAKDOWN STUDIES

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EXTRUDED FILMS FROM MODIFIED POLYPROPYLENE RESIN:
DIELECTRIC AND BREAKDOWN STUDIES

Robert J. Mamrone, William L. Wade, Jr. and Michael Pinder

Thin films (approximately 25 microns) formed by melt-extruding polypropylene resin after it had been briefly exposed to a low-pressure, low-temperature, CF<sub>4</sub>/O<sub>2</sub> gas plasma had significantly increased dielectric breakdown strengths with very little accompanying changes in dielectric properties.
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INTRODUCTION

High reliability, spirally wound, film capacitors for industrial applications require high quality dielectric films. Film limitations are presently due to poor insulation resistance and/or low dielectric breakdown strengths. Dielectric breakdown strengths, $V_b$, expressed in V/micron or kV/mil of thin polymer films play a key role in determining ultimate attainable energy densities when these films are used as dielectrics in capacitor applications. This is because energy densities of film capacitors increase as the square of the voltage across the capacitor. If $V_b$ of polymer films can be increased, capacitors can be operated at higher voltages which translates into higher electrostatic energy densities.

Since the capacitor industry is cost and performance driven, constantly increasing demands are being made to lower cost, and improve reliability and performance of materials. One rapid and inexpensive way to increase $V_b$ of polymer films is to briefly expose polymer films to low pressure, low temperature gas plasmas. However, much less information is available on effects of gas plasmas on finely divided powders where morphological changes, as well as modification of specific surface areas, surface functionalities and charge densities, may occur. Such alterations in properties for powders with extremely high surface-to-volume ratios could subsequently affect wetting, adhesion and stability. Surface effects of treated resins may also manifest themselves as changes in bulk dielectric properties when they are subsequently melt extruded into films. In the present study, we measured $V_b$ and other dielectric properties on melt extruded polypropylene, PP, films where the powdered resin had been briefly exposed (prior to melt extrusion) to CF$_4$/O$_2$ gas plasmas.

EXPERIMENTAL

Pellets of PP resin (Himont 6823) were milled in a Thomas-Wiley mill and exposed to 96% CF$_4$/4% O$_2$ gas plasma by evenly distributing a thin layer of ground-up resin on aluminum foil in a Branson/IPC (Fort Washington, PA) Model 4150 barrel plasma etcher at power levels of approximately 0.006 W/cm$^2$ for 4 minutes. Treated and untreated PP resins were sieved, and portions of powder captured by 30 or 40 mesh screens were extruded on a screw-type, Randcastle Microextruder under the following conditions: screw RPM, 50; die temperature, 450°F; barrel zone temperatures were 177°C for zone 1, 204°C for zone 2, and 232°C for zone 3. Translucent PP films, approximately 25 microns thick and 40 mm wide, were made from both untreated PP resin and PP resin that had been exposed to 96% CF$_4$/4% O$_2$ plasma.
Breakdown voltages of these PP films were measured in air at room temperature by ramping the voltage from zero volts at 500 volts per second until breakdown occurred and the film could not hold off additional voltage.

RESULTS AND DISCUSSION

Table 1 lists dielectric properties of two kinds of PP film: PP film extruded from unexposed PP resin and PP film extruded from PP resin that had been briefly exposed to CF$_4$/O$_2$ plasma. The data clearly show that exposure of PP resin (prior to melt extrusion) to CF$_4$/O$_2$ plasma increased the subsequent breakdown voltages of formed films by about 75% without significantly affecting either the dielectric constant or dielectric loss. This dramatic increase in breakdown voltage may be simply due to removal of surface contaminants and/or inhibitors or antioxidants which would be normally adsorbed on powdered resin surfaces. These species would have become trapped within the PP film during melt extrusion, thereby lowering breakdown voltages. Exposure of powdered resins to CF$_4$/O$_2$ plasma may be removing these impurities, and the resulting purer resins yield films having higher breakdown voltages. Another possible explanation is that reaction of powdered PP resin surfaces with CF$_4$/O$_2$ plasma forms thin, crosslinked or chemically modified (perhaps fluorinated) surface layers. When this resin, with high surface-to-volume ratios, is then melt extruded, the chemically modified species, formerly on the surface, now blend into the bulk to form films having higher breakdown voltages than films formed from pure PP resin. This explanation, however, does not explain why these films, formed from plasma treated resins, show no substantial difference in bulk dielectric properties over films formed from untreated resins.

CONCLUSIONS

Breakdown voltages of thin, melt extruded PP films can be substantially increased by briefly exposing ground-up PP resin (prior to melt extrusion) to low pressure, low temperature, CF$_4$/O$_2$ gas plasma.

ACKNOWLEDGEMENTS

We thank Dr. Sol Gilman for his thoughtful guidance, and encouragement, as well as participation in extensive technical discussions.
TABLE 1. Comparison of dielectric properties (dielectric constant, dielectric loss and breakdown voltage) for PP films (24-28 microns thick) melt extruded from 30 mesh PP resin which had been briefly exposed to 96% CF$_4$/4% O$_2$ gas plasma.

<table>
<thead>
<tr>
<th>Property</th>
<th>Baseline</th>
<th>Exposed to CF$_4$/O$_2$</th>
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</thead>
<tbody>
<tr>
<td>Dielectric constant</td>
<td></td>
<td></td>
</tr>
<tr>
<td>@1000 Hz</td>
<td>2.19</td>
<td>2.25</td>
</tr>
<tr>
<td>@10,000 Hz</td>
<td>2.19</td>
<td>2.25</td>
</tr>
<tr>
<td>Dielectric loss</td>
<td></td>
<td></td>
</tr>
<tr>
<td>@1000 Hz</td>
<td>6.6 $\times$ 10^{-4}</td>
<td>7.26 $\times$ 10^{-4}</td>
</tr>
<tr>
<td>@10,000 Hz</td>
<td>5.5 $\times$ 10^{-4}</td>
<td>6.35 $\times$ 10^{-4}</td>
</tr>
<tr>
<td>Breakdown Voltage</td>
<td></td>
<td></td>
</tr>
<tr>
<td>kV/mil</td>
<td>5.2</td>
<td>9.1</td>
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<tr>
<td>V/micron</td>
<td>205</td>
<td>358</td>
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