Troubleshooting Customer Adhesion Issues

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Principal Investigator
DuPont Tedlar® Films

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Tedlar® is a registered trademark of DuPont
## Dozens of Proven Applications, Globally, For Over 50 Years

<table>
<thead>
<tr>
<th>Application</th>
<th>Description</th>
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</thead>
<tbody>
<tr>
<td><strong>Aerospace:</strong></td>
<td>Attractive, easy-to-clean, scuff-resistant surface protection; also release for manufacturing</td>
</tr>
<tr>
<td><strong>Photovoltaic (PV):</strong></td>
<td>30+ years of proven durability and reliability for solar modules</td>
</tr>
<tr>
<td><strong>Composite Release:</strong></td>
<td>High-performance release for various compounds including fiber-reinforced polymer (C/FRP);</td>
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<tr>
<td><strong>Wind:</strong></td>
<td>Resist weathering, moisture and UV rays, while offering low drag and effectively keeping surfaces clean; also for release in manufacturing</td>
</tr>
<tr>
<td><strong>Transportation:</strong></td>
<td>Keeps vehicles and marine vessels looking their best by providing an easy-to-clean surface that resists weathering, UV rays and harmful chemicals</td>
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<tr>
<td><strong>Gas Sample Bags:</strong></td>
<td>Resistance to gas permeation along with chemical inertness to maintain sample integrity</td>
</tr>
<tr>
<td><strong>Architecture and Construction:</strong></td>
<td>Significantly prolong the useful life and preserve the aesthetics for interior and exterior</td>
</tr>
<tr>
<td><strong>Graphics and Labels:</strong></td>
<td>Preserve desired aesthetics in the most abusive environments for many years; also graffiti and pollution resistant</td>
</tr>
<tr>
<td><strong>Electrical/Electronic:</strong></td>
<td>High quality release with optimal conformability, toughness and inertness to adhesives</td>
</tr>
<tr>
<td><strong>Transfer Printing:</strong></td>
<td>Excellent printability and release from rubber and metal</td>
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</table>
Agenda

Defining the Issue

Determination where the adhesion issue is seen

Verifying quality control data

ESCA and FTIR Analysis

Resolution
Laminate Adhesion

Dependency on Many Factors

- Raw Materials
  - Films
  - Adhesive
  - Substrate

- Process Parameters
  - Time
  - Temperature
  - Pressure
Defining the Issue

What is the adhesion issue?

Normal versus the issue

Where was it seen – Raw Materials
   Within a roll
   Random or continuous
   With a particular lot/set of materials

When was it seen – Process
   Use process time
   Use clock/calendar time
   Piece of equipment
Adhesion Tests

- Tape Pull Tests – ASTM D3359
  - Cross Hatch – X
  - Cross Hatch – squares

- Tensile Pull Test -- Modified ASTM F904
  - 90 Degree Peel
  - 180 Degree Peel

- On-line Test
  - Grab and Pull
Adhesion Tests – What is being measured

Tape Pull Tests – ASTM D3359

   Adhesive force of tape to top layer versus adhesive force of top layer to next layer

Tensile Pull Test – Modified ASTM F904

   Strength of top layer versus the adhesive force

Grab and Pull

   Strength of top layer versus the adhesive force

Criteria for Acceptance
Adhesion Quality Control Tests

For Tedlar®
Tests confirm film is treated and at proper level
  Heat seal test to standard adhesive
  ESCA sampling

For Customer
Proper adhesion is done via plant/laboratory trials
Once trials are completed, testing is done to confirm process and material performance
Electron Spectroscopy for Chemical Analysis (ESCA)

Ultra High Vacuum Surface Analytical Technique

Provides both quantitative and qualitative information about the top 20 to 100 angstroms of a surface

Effective method to determine where the surface issue originates
Advantages of Tedlar® Film To Detect Adhesion Issues Using ESCA

Fluorine atom is typically provided by film only

Treating Tedlar® film changes the amount of oxygen on the surface

Treated Tedlar® film will not change appreciably with time
Adhesion Problem 1

- Customer states that experimental film does not stick to substrate
- Customer tests by pulling film from substrate
- Customer pass criteria is that the film breaks irrespective of force necessary to pull film
- The film being tested is a new thicker film
Adhesion Problem 1 – The experimental film
Picture of Laminate

Inadequate Cohesive Adhesion

Adhesive
FTIR of Film and Laminate
Resolution

Improved contact between film and substrate during lamination

Film is stronger than adhesive

Changing the film treatment will not improve adhesion
Adhesion Problem 2

- Film is an experimental new formulation
- Customer states that film is peeling with too little force in laboratory tests
- See the issue with multiple lots of film
## ESCA Results

<table>
<thead>
<tr>
<th>Surface</th>
<th>C1s</th>
<th>O1s</th>
<th>F1s</th>
<th>Si2p</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tedlar® facing Epoxy</td>
<td>69.7</td>
<td>2.4</td>
<td>27.8</td>
<td></td>
</tr>
<tr>
<td>Epoxy facing Tedlar®</td>
<td>72.8</td>
<td>23.1</td>
<td>3.7</td>
<td>0.4</td>
</tr>
<tr>
<td>Tedlar® facing Epoxy</td>
<td>65.3</td>
<td>1.8</td>
<td>32.9</td>
<td></td>
</tr>
<tr>
<td>Epoxy facing Tedlar®</td>
<td>73.5</td>
<td>24.5</td>
<td>2.0</td>
<td></td>
</tr>
</tbody>
</table>

ESCA indicates that Tedlar® film surface has low oxygen content. Epoxy facing indicates virtually no PVF on surface.

## Retain Samples

<table>
<thead>
<tr>
<th>Sample</th>
<th>C1s</th>
<th>O1s</th>
<th>F1s</th>
<th>Si2p</th>
</tr>
</thead>
<tbody>
<tr>
<td>White Film Retain</td>
<td>75.7</td>
<td>11.6</td>
<td>12.8</td>
<td></td>
</tr>
<tr>
<td>White Film Retain Treated</td>
<td>69.9</td>
<td>15.7</td>
<td>14.4</td>
<td></td>
</tr>
</tbody>
</table>
Resolution

Modify formulation of experimental film
Adhesion Problem 3

- Customer states that the Tedlar® film adheres better with one manufacturer of resin
- Customer tests adhesion by pulling on loose film at edge of laminate
- A different manufacturer of resin is used on each side of laminate
- Same roll of film is used for the laminate
Laminate – Using Resin Manufacturer With Known Poor Adhesion
Good Adhesion – Resin Manufacturer With Known Good Adhesion

Sample Film
Cut at DuPont
Analytical Results

ESCA

<table>
<thead>
<tr>
<th>Sample Area</th>
<th>Interface</th>
<th>Surface</th>
<th>C1s</th>
<th>O1s</th>
<th>F1s</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>CC15BL3/Epoxy</td>
<td>CC15BL3</td>
<td>71.2</td>
<td>1.5</td>
<td>27.3</td>
</tr>
<tr>
<td>2</td>
<td>CC15BL3/Epoxy</td>
<td>CC15BL3</td>
<td>72.3</td>
<td>1.8</td>
<td>25.9</td>
</tr>
<tr>
<td>2</td>
<td>CC15BL3/Epoxy</td>
<td>Epoxy</td>
<td>71.8</td>
<td>3.5</td>
<td>24.7</td>
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Tedlar® film breaks in the thickness direction for both sides

FTIR

Shows phenolic resin on PVF

Shows no differences on surfaces of film and laminate for either side of laminate
Light Spots are phenolic resin

Dark spots are Tedlar® Film
Adhesion Problem 3 Resolution

Break points are within the resin and film

Both resin systems provide similar adhesion to film
Summary

- Understand adhesion is both raw material and process dependent

- Adhesion can be tested various ways with different criteria

- Analytical results of adhesion issue samples provide excellent quantitative and qualitative data

- Resolving the issue can be done via many different methods
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