Troubleshooting Customer Adhesion Issues

Tedlar® polyvinyl fluoride films have been used for decades in many different applications as release film (used during manufacturing of parts) and also as a protective layer (staying with a part for its lifetime). The fact that the film is used in such different and contrasting applications has created a few situations where delamination investigations have been conducted. These investigations have led to insights into understanding effective means to determine the root cause of adhesion issues.

When considering film adhesion within a laminate, materials and process impact the results. Typically, materials will include film, adhesive, and substrate while process will include equipment, time, pressure, and temperature. When exploring adhesion issues, questions about changes in materials and process are asked. Also, questions about when successful laminations are created will help to narrow down the potential root causes of inadequate adhesion.

The film adhesion tests run on laminates are typically either a tape pull test or a tensile pull test. ASTM D3359 is the standard method used for a tape pull test that uses a cross hatch or a series of squares. Some customers have modified the procedure in an effort to better discern good adhesion. ASTM F904 provides the framework to determine the adhesive force between a film and adhesive. Many customers have modified the procedure to develop a tab to start pulling the film from the adhesive. Some tests are run with a 180-degree peel while others use 90-degree peels. The different angles can create different forces on both the adhesive and the film.

The tape pull test is testing the adhesive force of the film to the adhesive versus the force holding the tape to the film. For some cases, changes in the adhesion strength between tape and film can create a change in the test results despite the adhesive strength between the film and the adhesive remaining constant. The tensile pull test is comparing the strength of the film to the strength of the adhesive bond. The criteria for acceptable results in this test can either be a film breaking bond or a minimum adhesive force to separate the layers.

The criteria for acceptance is important to understand system changes. For many customers, a modification of the tensile pull test is run on-line. In this case, the check-out sample has a tab to start a peel and the customer pulls the top layer until it breaks.

Adhesion quality control tests are typically run to ensure that the process is under control and performing similarly to previous runs. For Tedlar® films, two tests are run:

- An adhesion test to a known substrate and adhesive; and
- An ESCA (Electron Scanning Chemical Analysis) test

Together the tests provide independent data that the treatment level is in control and similar to previous production.
For laminators, typically trials are run to determine process parameters necessary for good adhesion as well as the raw materials necessary. Once the trials are complete, a less extensive set of tests are run on standard production to ensure performance seen in trials.

ESCA is an extremely useful analytical test for Tedlar® film. ESCA examines the top 20 to 100 angstroms of a surface and provides qualitative and quantitative data. This data is especially useful for polyvinyl fluoride (PVF) films because

- The fluorine atom is typically unique to Tedlar® films
- Treated PVF film will have increased oxygen content
- Treated PVF film does not change treatment level over time.

Testing both sides of an adhesion issue provides valuable information on break point interface. When more material is left on the interface, FTIR measurement can provide similar data as ESCA. One caution with ESCA measurement is that finger contamination can be detected in this method. To avoid contamination, laminate samples exhibiting the adhesion issue but fully pulled apart are extremely useful; these samples can be pulled further apart prior to test to yield a contamination-free surface.

Three examples of customer adhesion problems are given using experimental (not commercial) Tedlar® samples. The first example entailed a laminate where the customer expected a film breaking bond when the film was pulled from the substrate. The customer was testing a new thicker film. Examination of the film and the substrate showed both an adhesion issue where the adhesive was not in contact with the film at lamination temperatures as well as inadequate cohesive adhesion. The cause was that the thicker film was stronger than the previous film causing the cohesive adhesion issue. The inadequate adhesion was due to the film not being pressed to the substrate at activation temperatures.

In the second example, the customer stated that the new experimental film did not have sufficient adhesion to the substrate – it did not reach a minimum peel force value. The defect was observed with multiple lots of film. ESCA results of the laminate shows that the film appears to be non-treated based on oxygen and fluorine content. The epoxy side of the laminate shows virtually no fluorine. The retain sample of the non-treated sample shows a depressed fluorine content; the treated retain sample shows increased oxygen content but still a low fluorine content. Based on the retain and laminate data, the conclusion drawn was that an additive in the PVF film bloomed to the surface creating a weak boundary layer. The resolution was reformulation of the experimental film to eliminate the excess additive.

The third example presented is where the customer stated that the film did not stick to one supplier’s resin system but did stick to a second system. The chemistry of the resins was the same. The adhesion was one where the laminate producer pulled the film from the edge of the laminate; the criteria for acceptance is a film-breaking bond. ESCA results indicated that the both surfaces of the interface were PVF film. FTIR results shows phenolic resin on the PVF film side and PVF film on the laminate side. The results were similar for both sides of the laminate once a tab was created on the “good” adhesion side.
The conclusion is that the resins are performing the same, and that the interface shows a mixed breaking mode within the film and resin.

Tedlar® is a registered trademark of DuPont for its polyvinyl fluoride films.

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