

# Properties of EB Top-Coated Metallized Films Prepared in Vacuum

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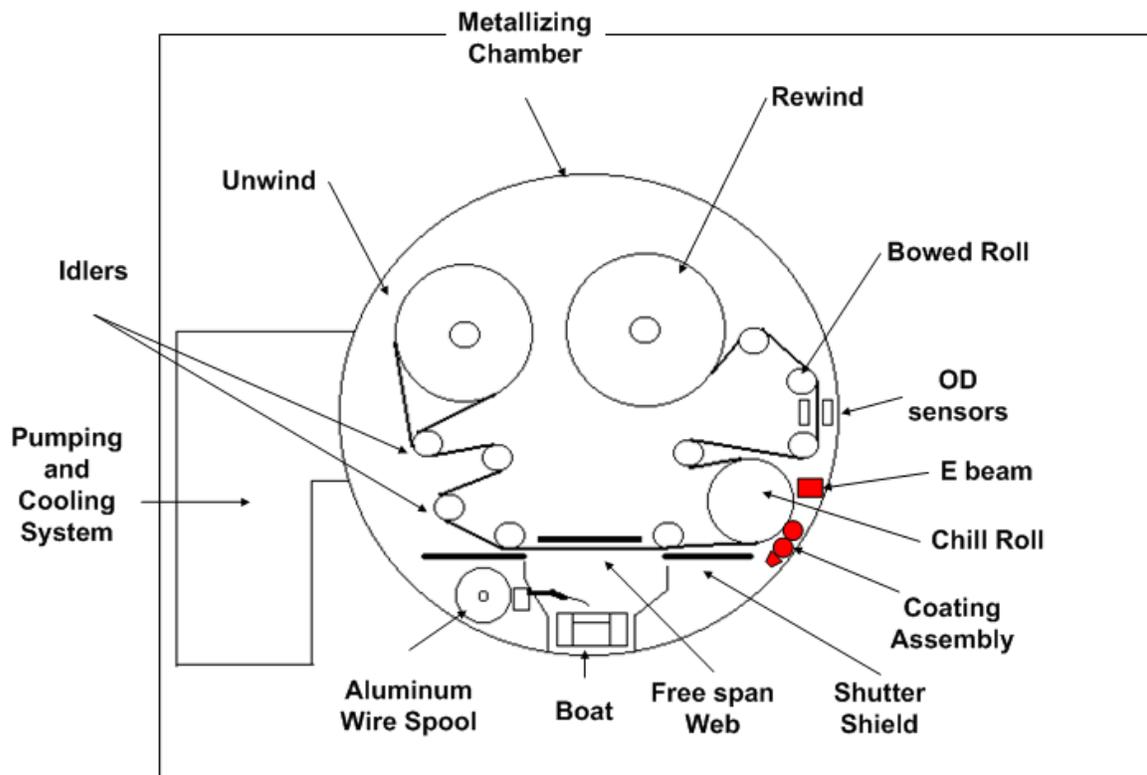
## The Single-Pass Metallizing & Coating Process

The traditional method of preparing a top-coated metallized film using roll-to-roll processes is to carry out two separate, independent steps:

- ◇ Unwind the film under vacuum, metallize the film surface, rewind the film under vacuum, vent the chamber to atmosphere.
- ◇ Unwind the film in atmosphere, top-coat the metallized surface, rewind the film in atmosphere.

This two-step process creates certain inherent disadvantages, which we attempted to overcome by retrofitting a traditional single zone free-span metallizing chamber with an in-line top-coating process directly after the metallizing area. Figure 1 shows a schematic of the one-step process, while Table 1 provides a comparative overview between the processes.

Figure 1: Schematic of free span metallizer with coating assembly and E-beam



**Table 1: Comparison Between 1-Pass and 2-Pass Processes**

	<b>Two-step process: Metallize first, then top-coat</b>	<b>One-step process: Metallize &amp; top-coat in one pass</b>
<b>Capital investment</b>	<ul style="list-style-type: none"> <li>• Need to purchase two separate pieces of equipment</li> <li>• Require two production areas</li> <li>• Higher fixed costs</li> </ul>	<ul style="list-style-type: none"> <li>• Need to purchase or retrofit one piece of equipment</li> <li>• Requires one production area</li> <li>• Lower fixed costs</li> </ul>
<b>Operating costs</b>	<ul style="list-style-type: none"> <li>• Requires two sets of operators</li> <li>• Requires two sets of drives &amp; web handling/winding controls</li> <li>• Need metallized WIP inventory</li> <li>• Film yield losses occur in both steps, can total 10% or greater</li> <li>• If using water-based coating, require drying oven</li> <li>• If using solvent-based coating, require oven &amp; oxidizer</li> <li>• Higher operating costs</li> </ul>	<ul style="list-style-type: none"> <li>• Requires one set of operators</li> <li>• Requires one set of drives &amp; web handling controls</li> <li>• No WIP</li> <li>• Film yield losses around 5%, similar to metallizing alone</li> <li>• No ovens or oxidizers required</li> <li>• Need to degas liquid prior to feeding into vacuum chamber</li> <li>• Lower operating costs</li> </ul>
<b>Metallized film properties</b>	<ul style="list-style-type: none"> <li>• Aluminum surface is oxidized in air prior to coating</li> <li>• Pinholes in aluminum layer created by flaking off of surface impurities from film during web handling &amp; winding</li> </ul>	<ul style="list-style-type: none"> <li>• Coating is applied to pure, unoxidized aluminum surface</li> <li>• No pinholes in aluminum from flaking off of surface impurities, since these are trapped by coating immediately after metallizing</li> </ul>
<b>Coating properties</b>	<ul style="list-style-type: none"> <li>• Broad range of chemistries &amp; viscosities available, including water-based, solvent-based, and energy curable</li> <li>• No limitations as to layer thickness, surface dyne level, surface COF</li> </ul>	<ul style="list-style-type: none"> <li>• Require EB coatings with a specific chemistry, that do not auto-polymerize in vacuum</li> <li>• Tends to be more expensive raw material cost per dry pound</li> <li>• Consistent coat weight down to 0.2 micron (0.2 gsm)</li> <li>• Limits to surface properties</li> </ul>

The EB curing process was chosen over UV curing due to the more ready acceptance of EB coatings for direct & indirect food contact purposes. The photo-initiators inherent in UV curable compounds add an extra level of complexity to any food packaging approval process. EB curable coatings & adhesives are currently in commercial use for many food packaging applications. There are two key factors required to meet the needs of a food contact application: a robust process with fail-safes in place to ensure uncured or under-cured coatings cannot be produced, and that fully cured coatings pass all extractables testing (detectable limit of <50 ppb for most EB curable substances).

## Metal Adhesion

As a starting point, suitable bond strength must be achieved between the metal layer and the coating. This can be tested many different ways, but the industry accepted method is to use Scotch 610 or 810 tape, pulled back at a 180° angle from the specimen surface. For the purposes of most applications of interest using this chemistry, a pick-off level of < 1% was desired. This bond level has not always been easy to achieve using traditional atmospheric EB coatings. One possible reason is that the oxidized aluminum surface, although it has a high surface energy, requires aggressive acidic or basic chemistries to achieve good bonding with the coating.

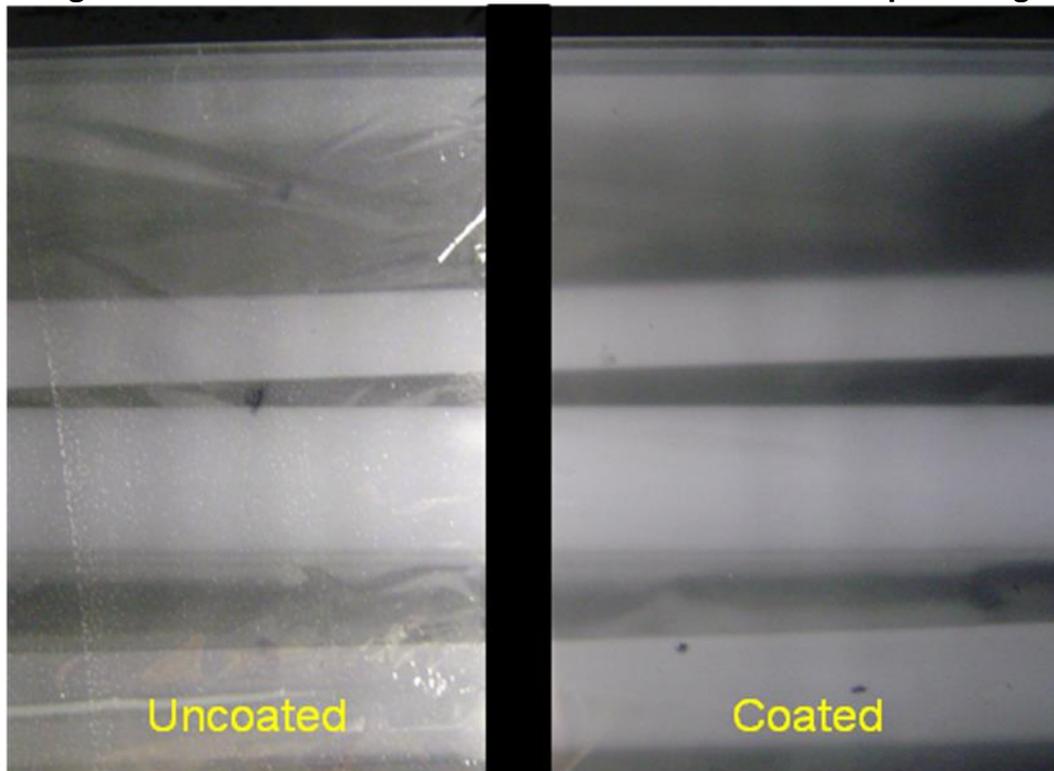
With the different EB chemistries evaluated, it was found that acceptable bonds between the coating & metal could be achieved, with tape test values of <1% pick-off.

## Barrier Properties

For food packaging, barrier properties are critical to determining the shelf life of a product. For a metallized film, barrier is typically a function of the quality of the metallized layer, and to a lesser extent the substrate. In particular, pinhole theory dictates that the transmission rate of gases is most dependent on the number and size of defects in the aluminum layer.

When samples are placed on a light box, as in Figure 2, it is apparent that the metallized layer with a top-coating applied in a single pass shows virtually no pinholing. Meanwhile, the same metallized layer prepared on the same metallizer without a top-coating shows typical pinholing in the metal layer.

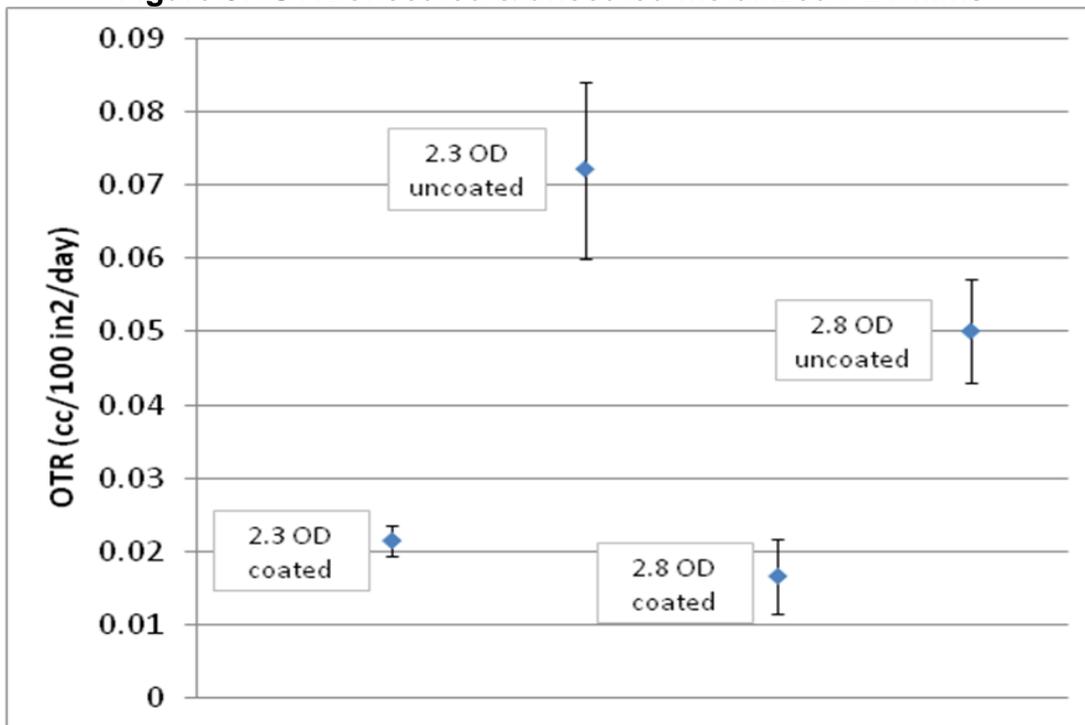
**Figure 2: Back-lit metallized PET films with & without top-coating**



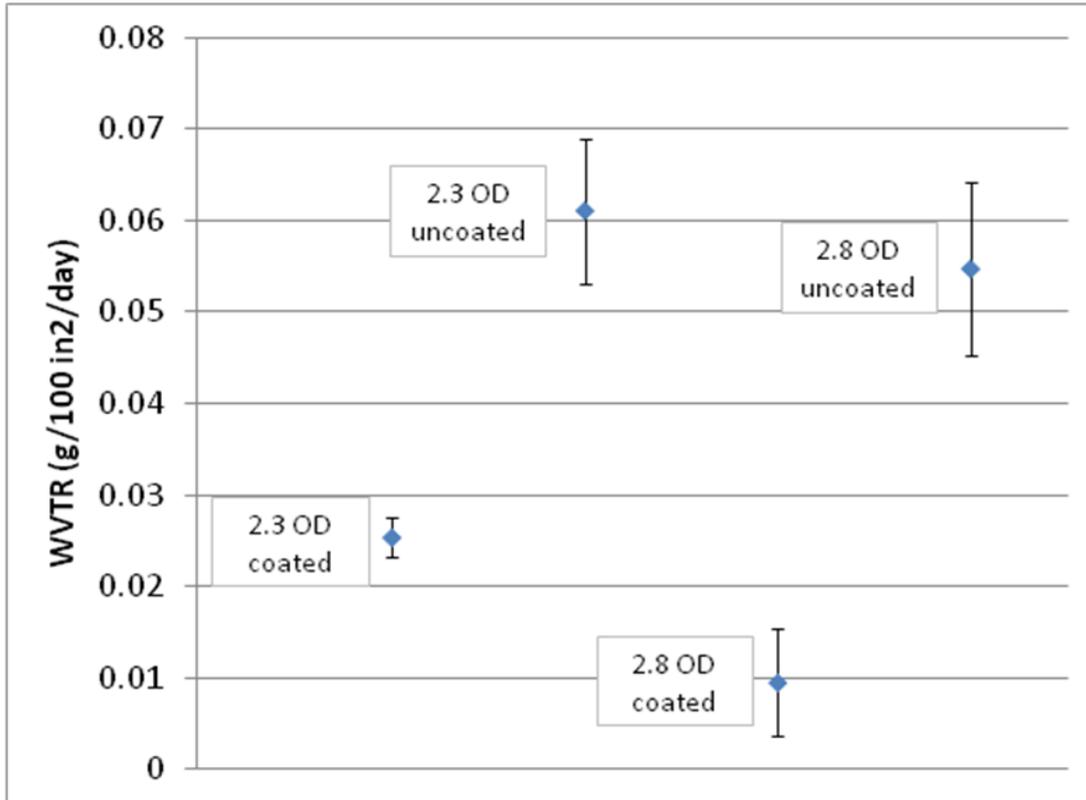
One reason commonly cited for the appearance of pinholing in the metallized layer on a PET film is that surface impurities (oligomers, debris, etc.) on the film surface can flake off when that surface comes into contact with rollers in the metallizer downstream of the metallizing zone, and particularly when that surface is wound against the backside of the PET film in the finished roll. It appears that by top-coating the freshly deposited aluminum surface directly after metallizing, these surface impurities do not get a chance to come off, and the top-coat traps the metal layer in place on top of said impurities.

This suggests that barrier properties of a top-coated film might be superior to the same film metallized without top-coating. This indeed is the case with the metallized PET films that have been evaluated to date. Figures 3 & 4 compare the OTR & WVTR, respectively, of two different sets of metallized PET films run to different optical densities under the same metallizing conditions, on the same metallizer. In each case, the film was evaluated with & without top-coating. The data points are average measurements, with error bars representing 95% confidence intervals based on a minimum of 3 test results per sample.

**Figure 3: OTR of coated & uncoated metallized PET films**



**Figure 4: WVTR of coated & uncoated metallized PET films**

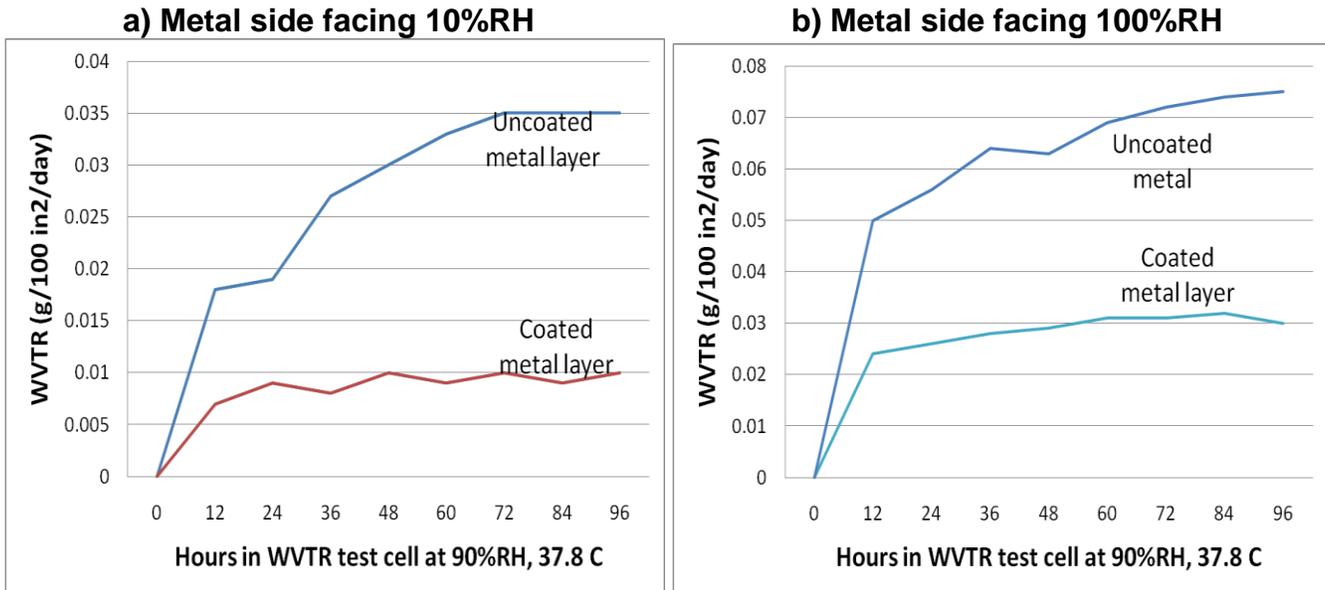


Some applications require the converter to not only look at standard OTR or WVTR test results, which typically take 24 hours, but also to look at long-term ageing. In particular, it has been shown that WVTR measurements can change over time in “jungle room” type test conditions of 37.8°C and 90%RH<sup>1</sup>, as the water molecules interact with the metallized surface, even when the metallized surface is buried in a lamination. Figures 5a & b show the evolution of the WVTR over a four day period with two sets of samples.

An uncoated & coated sample, both metallized on the same substrate and to the same 2.8 OD under the same metallizing conditions, were placed in a Lyssy L-80 WVTR test unit. The L-80 model performs according to ASTM E-398, with 100%RH on one side of the test cell and nominally 10%RH on the other side. In Figure 5a, the metallized surface of each sample was facing away from the high humidity side of the chamber, as would be the typical orientation for a standard WVTR measurement. As we can see, the uncoated metal layer deteriorates slowly over time, giving a final steady state WVTR value of 0.035 g/100 in<sup>2</sup>/day after 3 days. The coated metal layer appears to come to equilibrium more quickly, achieving a steady state value of 0.010 g/100 in<sup>2</sup>/day in about a day.

In Figure 5b, the uncoated metal layer is interacting to an even greater degree with the moisture on the 100%RH side of the test cell, climbing more rapidly than in Figure 5a, and does not even reach an equilibrium value within 4 days. The coated metal layer interacts with the moisture as well, but to a much lower degree, and appears to equilibrate within 2 – 3 days.

**Figure 5: WVTR of coated & uncoated metallized PET films**

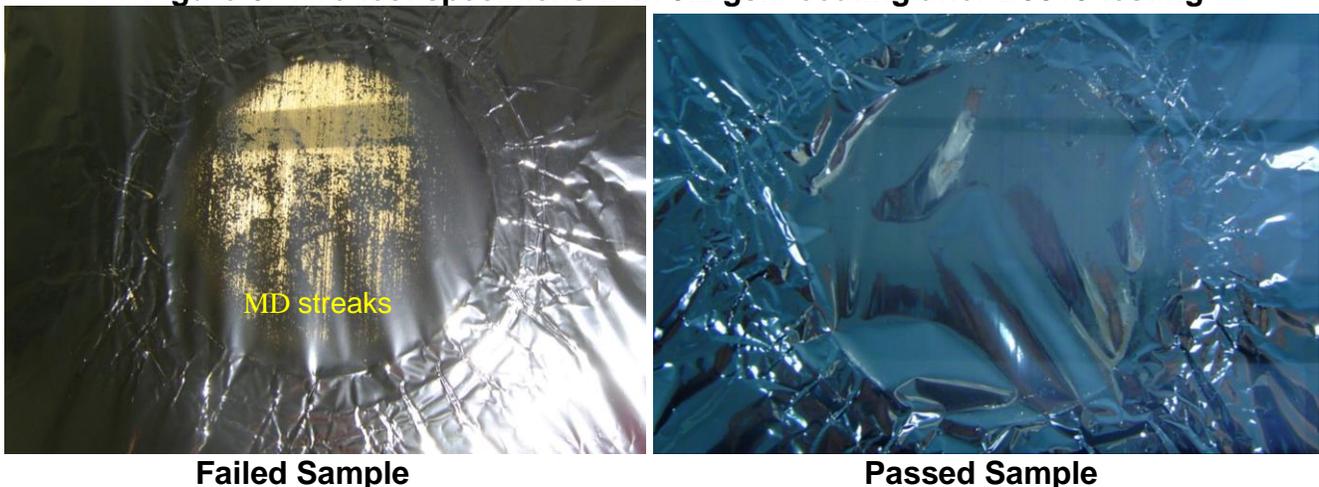


## Corrosion Resistance

For certain industrial markets, such as reflective insulation, good corrosion resistance is essential. This is measured by directly exposing the coated metallized surface to elevated heat and humidity (71°C, 100%RH) for seven consecutive days, then carrying out a subjective evaluation of the level of corrosion in the metal surface, in accordance with ASTM D3310.

The right EB chemistry is important to achieve good moisture resistance. Equally important, however, is a consistent, even coating applied across the web. Any coating defects, such as streaks or skips, will show up during the ASTM D3310 testing. Achieving a consistent, thin, even coating with a 100% solids EB coating has been one of the biggest technical challenges to overcome. However, we have found it is possible to achieve consistent, even coatings that provide suitable corrosion resistance across the web.

**Figure 6: Two test specimens with 0.2 gsm coating after D3310 testing**



## Emissivity

High radiant heat reflection and low radiant heat emission is essential for radiant barriers in building insulation. The best way to measure a material's radiant heat insulation capability is by measuring its emissivity. By definition<sup>2</sup>, radiant barriers require an emissivity of  $< 0.1$ , although practically speaking the market demands emissivity values closer to 0.05. Emissivity is most commonly measured according to ASTM C1371, using a Devices & Services RD1 model emissometer.

One study<sup>3</sup> suggests the emissivity of an uncoated metallized film surface is dependent on the thickness of aluminum deposited as well as the crystallization characteristics of the PET film. Therefore, all tests were carried out with a similar 12 micron PET base film, and metallizing was carried out to similar optical densities.

It was found that the emissivity of a PET film that has been metallized to a 2.5 OD and then coated in a second pass using the traditional atmospheric EB coating process gives emissivity values in the 0.05 – 0.07 range. The emissivity of the same PET film metallized to 2.5 OD and then top-coated in a single pass gives emissivity values in the 0.04 – 0.05 range, even with the same coating thickness. This finding has been reproduced several times. It is uncertain at this time why this difference exists, but further study will be undertaken in this area.

## Scuff Resistance

One additional requirement for many applications, including both food packaging and insulation applications, is good scuff resistance. This is measured using a Sutherland 2000 Rub Tester, using a 4 lb weight and 25 rubs, testing coated side to coated side. The rubbed samples are then compared with standards to determine, using a subjective rating system of 1 to 5, how scuffed the surfaces are. Two samples from top-coating trials with different chemistries are shown in Figure 7.

**Figure 7: Rub test results from two different top-coated metallized PET films**



It was found that scuff resistance is primarily a function of the coating chemistry, as well as the EB dosage applied to the coating. A more reactive chemistry and a higher EB dosage are correlated with better scuff resistance, as increased levels of cross-linking tend to improve the abrasion resistance of the coating. Coatings with a lower inherent surface energy also lead to improved scuff resistance.

## **Conclusion**

The original objective of developing a single pass metallizing & top-coating process was purely one of economics, as it was determined it would be much more economical to commercially produce these products in a single pass vs. a double pass. However, we have found many potential technical advantages to metallizing & top-coating in a single pass, including improved barrier and lower emissivity. These phenomena warrant further investigation, both on PET film and other substrates.

## **References**

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