Determination of Metal Adhesion Strength of Metallized Films by Peel Tests

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Outline

- Introduction
- Peel test procedures for metal adhesion
- Experimental results and problems
- Alternative peel test approaches
- Summary and Conclusions
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Metallized films in Packaging Industry

**Film Producer:**
Substrate film production

**Metallizer:**
- Pre-treatment
- Vacuum web coating

**Converter:**
Lamination and printing

SUBSTRATE FILM (e.g. BOPP, PET)

METALLIZED FILM

METALLIZED LAMINATES

- Sealable top layer (e.g. PE)
- PET substrate film

END-PRODUCT
Metallized Films in Packaging Industry

- Metallized Films
  - Metallized film production worldwide: $2.4 \times 10^{10}$ m² / year (source AWA 2011)
  - About 85% of metallized films are used in flexible packaging
  - Worldwide flexible packaging market: € 50 billion

- Why metallisation?
  - Barrier improvement up to factor 100, longer shelf life of packed products
  - Barrier to light
  - Decorative
  - Higher productivity
  - Low-cost production

- Important quality parameter
  - Adhesion strength between thin metallised layer and the substrate
Metallised Films in Packaging Industry

PE / Adhesive / PET 12 µm / MET / Adhesive / Printing / PET 12 µm

- Metal adhesion problem:
  - Unexpected de-lamination between PET 12 µm substrate film and the metallised layer
  - ~ 100 % metal transfer to the adhesive laminated top layer
Metallised Films in Packaging Industry

- Products sensitive to oxygen and water / water losses
- (BO)PET substrates for high oxygen barrier
- (BO)PP substrates for general purposes

Substrate film

Barrier layer

Adhesive

Heat sealing layer

Laminate bond strength

Metal adhesion

adhesive

Al layer

800 nm
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Peel Test Procedures

Metal adhesion: EAA-Peel test

Laminate bond strength, sealed-seam strength: $90^\circ$ peel test

- Laminated-film: EAA (used in peel tests)
- Metal layer
- Substrate film (e.g. PET)
- Double-sided adhesive tape
- Al-plate (1 mm thick)
- Peel direction
- Peel-off angle: $180^\circ$
- Peel distance
- Fixed in the bottom clamp
What do you really measure in a peel test?

Measured work consists of:
- Delamination
- Elastic deformation
- Plastic deformation

Just a fraction of the work goes into delamination

A.J. Kinloch et. al., Int. J. of Fracture 66 (1994) 45 - 70

Force-Displacement Curve in an Ideal Peel Test
Properties of Extruded EAA-film

<table>
<thead>
<tr>
<th>Ethylene (E)</th>
<th>Acrylic Acid (AA)</th>
<th>Peel Test Parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water &amp; moisture resistance</td>
<td>Polarity</td>
<td>Procedure</td>
</tr>
<tr>
<td>Themoplasticity</td>
<td>Softening</td>
<td>EMA</td>
</tr>
<tr>
<td>Flexibility</td>
<td>Adhesion</td>
<td>AIMCAL</td>
</tr>
<tr>
<td>Crystallinity</td>
<td>Low heat seal temperature</td>
<td></td>
</tr>
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Metallized Films – Thermal Evaporation

Vacuum Metallization Process:

• Typical width: 2.4 up to 4.5 m
• Max. web speed: 18 m/s
• Annual output: Some hundreds of millions of m²

Three PET (12 µm) films with different surface properties were selected:

• Sample - A
• Sample - B
• Sample - C

1 Unwinder
2 Spread roll
3 Plasma treatment
4 Evaporation source
5 Process roll
6 In-line optical monitor
7 Tension roll
8 Rewinder

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EAA-Peel Test: Sample-A (Metallized PET)

PET-A 12µm / met // EAA

After EAA - peel test

Successful EAA-peel test!

Peel distance: ≈ 41 mm

EAA-Peel Test: Sample-B (Metallized PET)

PET-B 12µm / met // EAA

Improved Adhesion (B)

Problematic EAA-peel measurement!
Partial delamination of Aluminum!


© Fraunhofer
EAA-Peel Test: Sample-C (Metallized PET)

PET-C 12µm / met // EAA

Further Improved Adhesion (C)

Problematic EAA-peel measurement!
No delamination of Al, EAA film stretching

Representative test specimens

Peel distance: ≈ 30 mm

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Mechanical Properties of Extruded, Heat - Sealable Films

- EAA
- Amorphous Polyamide (aPA)
- Amorphous poly(ethylene terephthalate) (aPET)
Alternative Approaches – aPET Peel Test

PET 12µm / met // aPET

Full delamination of Al

Correct sequence (A < B < C)
Alternative Approaches: EAA, aPA, aPET Peel Test Comparisons

PET 12µm / met // EAA
PET 12µm / met // aPA
PET 12µm / met // aPET

- Peeled laminates are no chains that break at the weakest link
- If you like reliable results, avoid the use of yielding polymers in peel tests!
- Relative rankings are only possible with identical polymer films
- **But:** If you just like high figures for your customers, use yielding polymers, also in laminates

<table>
<thead>
<tr>
<th>Peel tests</th>
<th>EAA (25 µm)</th>
<th>aPA (20 µm)</th>
<th>aPET (100 µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(A)</td>
<td>0.67</td>
<td>0.2</td>
<td>0.4</td>
</tr>
<tr>
<td>(B)</td>
<td>• Al layer partially peeled off 0.3 0.7</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>• Stretching of EAA-film</td>
<td></td>
<td></td>
</tr>
<tr>
<td>(C)</td>
<td>• Al layer not peeled off at all 0.8 1.8</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>• EAA-film stretches</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>• Measured Force: ≥ 3.4 N/15 mm</td>
<td></td>
<td></td>
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</table>
Alternative Approaches: PE-LD Peel Test

**PET 12µm / met / Adhesive / PE-LD**

Lamination of low density polyethylene (PE-LD) film (30 µm) by polyurethane based adhesive

Bench - scale lamination: K-bar

Pilot - scale lamination
Alternative Approaches: PE-LD Peel Test

Bench - scale lamination

Sample-B

Sample-A

Pilot - scale lamination

Sample-B

Sample-A

Average

Typical range
## Alternative Approaches: PE-LD Peel Test

<table>
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<th>Peel tests</th>
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<th>PE-LD (30 µm)</th>
</tr>
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<tbody>
<tr>
<td></td>
<td>(Bench)</td>
<td>(Pilot)</td>
</tr>
<tr>
<td>(A)</td>
<td>0.7</td>
<td>0.6</td>
</tr>
</tbody>
</table>
| (B)        | • Al partially peeled off  
• Stretching of EAA-film | 0.8 | 0.8 |
| (C)        | • Al not peeled off at all  
• EAA-film stretches  
• ≥ 3.4 N/15 mm | ≥ 2.4 | ≥ 2.8 N/15 |

### Average Peel Force (N/15 mm)

<table>
<thead>
<tr>
<th>Sample-A</th>
<th>Sample-B</th>
<th>Sample-C</th>
</tr>
</thead>
<tbody>
<tr>
<td>„Bench“</td>
<td>„Pilot“</td>
<td>„Bench“</td>
</tr>
<tr>
<td>„Pilot“</td>
<td>„Bench“</td>
<td>„Pilot“</td>
</tr>
</tbody>
</table>
Current standards, e.g. EN ISO 11339 or EN 1895 (similar)

Required results:

EN ISO 11339: max., average and min. force

EN 1895: average forces
Current standards, e.g. DIN 55529

Required results:
• Initial force
• Average separation force
• Maximum force
• Fracture images (see examples)

Where are e.g.
• delamination distances?
• elastic or plastic deformation?
Summary and Conclusions

- There is a lack of test methodologies for the measurement of metal adhesion on polymeric substrates
- Current measurement standards are largely incomplete
- The available EAA-peel tests are not always applicable by the metallizing companies due to the overstretching of the EAA-film during the peel test
- Mechanically stronger heat sealable films may be more promising
- Peel test results of laminates are influenced by several factors, which are usually not recorded during the measurements such as peel distance, peel-off angle, and thickness, elastic, viscoelastic properties of the peeled material,
- Relations to practical load conditions are still not known
- There is still a considerable amount of work to be done about development of adhesion test methods relevant to packaging and packaging materials!
Published in the Special Issue of Journal of Adhesion Science and Technology „Adhesion Aspects in Packaging Industry“:

Evaluation of adhesion strength between thin aluminum layer and poly(ethylene terephthalate) substrate by peel tests – A practical approach for the packaging industry

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