Transparent Barrier Coatings for Environmental-Friendly Packaging Applications

Roland Trassl
Applied Materials

AIMCAL Web Coating & Handling Conference
Oct 22\textsuperscript{nd} 2012, Myrtle Beach, SC
Outline

- Requirements and challenges for transparent barriers

- TopMet CLEAR
  - Transparent Barriers by Reactive Evaporation
  - Transparent Barriers by Plasma-Assisted Reactive Evaporation
  - Barrier Improvement and Protection by TopCoat
  - High-Barrier Layers by PECVD

- Summary
Challenges for Barrier Coatings at High Productivity

- **Barrier**
  - OTR, WVTR dependent on substrate material
  - wide variety of materials and qualities

- **Layer Properties**
  - high uniformity over large substrates and long coating length

- **Adhesion of the Coating to the Substrate**
  - optimization on downstream processes

- **Minimum Defects**
  - Roll edge, wrinkles, tramlines, splashes, substrate overheating, electrostatic charging, blocking, ……

- **Cost**
Machine Quality

Machine must ensure stable product quality for 24/7 mass production

- **Barrier**
  - Closed-loop inline coating control, stable and homogeneous vacuum conditions, data logging of all relevant machine parameters

- **Layer Properties**
  - Closed-loop inline process control

- **Adhesion of the Coating to the Substrate**
  - Substrate Pretreatment

- **Minimum Defects**
  - High quality winding system, general machine lay-out
TopMet® - the Machine ...  

Barrier and Decorative Coating

- Improved freshness & shelf life
- Decorative coating

• Wide range of substrates from thin plastic films; PET, OPP, BOPP & more, to pre-coated papers
• Extremely thin, highly uniform layer of Aluminum or AlOx as protective barrier to improve freshness and longer shelf life and/or decorative coating for packaging materials.

... to metallize the materials for sophisticated packaging systems.
Industry Proven Since Early 1990s

- Recognized as industry standard for:
  - Flexibility
  - Productivity
  - Reliability
  - Quality

- Over 50 years of flexible coating technology experience

- ~200 TopMet® coaters; ~700 overall coating systems installed worldwide

TopMet® 4450

World’s largest & fastest evaporation metallizers for packaging (up to 4.5 m wide, 20m/sec coating speed)
Barrier Landscape

Targeted range for TOPMET barrier

Source: Alcan Packaging
Process Options for TOPMET CLEAR™

- Metallizing with base system
- All other Processes optional
- Also available for Field Retrofit

**Standard AI Metallizing**

- High Barrier AlO₅ₓ on PET
- AlO₅x-ECON: (No plasma)
- High barrier AlO₅ₓ on PET, OPP, PLA, CPP
- AlO₅x-HAD: (HAD-plasma assisted process)

- Freshure® single coat, directly on polymer substrate
- or Freshure® topcoat on Al / AlO₅ₓ

Source: DSM
Reactive $\text{AlO}_x$ - Evaporation

Evaporation of $\text{Al} + \text{Oxygen Inlet} = \text{AlO}_x$ on Substrate
HiRES™ TopMet® High Rate Evaporator

- 25% higher coating speed
- Better layer uniformity
- 10%+ lower costs/m² or costs/kg
- 5% higher Al efficiency
- System down time less due to bigger roll diameters (1250mm)

The solution is..

HiRES™ = HIGH RATE EVAPORATOR SYSTEM with patented staggered-boat design
eCharge: Advanced Film Cooling
Innovative System Increases Web Speed & Quality

Benefits
- Better cooling allows higher speeds for heat sensitive films
- Example: eCharge boosts CPP film coating speed from 8 to 13 m/s
- Resulting in 62% boost in speed and 26% reduction in CoO

Problem
- Normal winding tension leaves small gaps between film and coating drum
- Low contact force = poor heat transfer

Solution
- Apply electrostatic charge to film to increase contact force and improve heat transfer

Patented eCharge enables faster coating speeds for heat-sensitive substrates
Effect of Electron Charging on Coating Productivity

<table>
<thead>
<tr>
<th>SUBSTRATE</th>
<th>NO E-CHARGE</th>
<th>WITH E-CHARGE</th>
</tr>
</thead>
<tbody>
<tr>
<td>CPP 20µm</td>
<td>web speed: 10 m/s, tram lines: Increased Potential</td>
<td>web speed: 13 m/s, tram lines: none</td>
</tr>
<tr>
<td>PLA 25µm</td>
<td>web speed: 6 m/s, tram lines: Increased Potential</td>
<td>web speed: 10 m/s, tram lines: none</td>
</tr>
</tbody>
</table>

CPP film 20 micron thickness
Basic System Layout – AlOx

- Oxygen inlet
- Evaporation boats
Reactive AlO$_x$ - Evaporation: Optical Inline Coating Control

Evaporation of Al in oxygen:
- increase in optical transparency
- decrease in barrier performance

Due to the narrow process window optical inline coating control is essential
In-Line Process Control
**AlOx ECON – Performance**

coating width: 1250 mm
process speed: 12 m/s

**AlOx uniformity over complete foil ~ ± 3% (2-sigma)**
Barrier Performance of AlOx Coated Substrates

reactive evaporation

<table>
<thead>
<tr>
<th>substrate</th>
<th>OTR [cc/m²day]</th>
<th>WVTR [g/m²day]</th>
</tr>
</thead>
<tbody>
<tr>
<td>PET</td>
<td>0.8 – 1.2</td>
<td>0.3 – 0.7</td>
</tr>
</tbody>
</table>

measurement conditions:
OTR @ 23°C, 50% RH
WVTR @ 38°C, 90% RH
Plasma-Assisted AlO$_x$ - Evaporation

New advanced process developed by and up scaled with the Fraunhofer FEP in Dresden
Basic System Layout – AlOx – HAD

- plasma source
- evaporation boats
Plasma Assisted Evaporation in TopMet CLEAR™

AlO$_x$ process proven for web speed $\leq$ 8m/s and coating length 50,000m
Process Window for Reactive Evaporation with Plasma Assistance

With plasma increased process window

Optical transmission vs. WVTR, OTR

Process window without plasma vs. process window with plasma

Oxygen flow, process pressure

With plasma increased process window
Effect of Plasma Activated Evaporation: \( \text{Al}_2\text{O}_3 \) on PET

\( \text{Al}_2\text{O}_3 \) by Reactive Evaporation

Plasma assistance creates denser and more uniform coating structure
Barrier Properties of Plasma-Assisted AlOₓ Process

- Substrate: RHBY 12µm
- Optical transmission ≥98%

Optimal barrier properties at low layer thickness
Mechanical Barrier Stability AlO$_x$ with Plasma

- Improved mechanical stability due to plasma assisted process.
- Better downstream robustness
## Barrier Performance of AlOx Coated Substrates

### reactive evaporation

<table>
<thead>
<tr>
<th>substrate</th>
<th>OTR [cc/m²day]</th>
<th>WVTR [g/m²day]</th>
</tr>
</thead>
<tbody>
<tr>
<td>PET</td>
<td>0.8 – 1.2</td>
<td>0.3 – 0.7</td>
</tr>
</tbody>
</table>

### plasma-assisted evaporation

<table>
<thead>
<tr>
<th>substrate</th>
<th>OTR [cc/m²day]</th>
<th>WVTR [g/m²day]</th>
</tr>
</thead>
<tbody>
<tr>
<td>PET</td>
<td>0.5 – 1.0</td>
<td>0.3 – 0.5</td>
</tr>
<tr>
<td>OPP</td>
<td>25 – 40</td>
<td>0.15 – 0.4</td>
</tr>
<tr>
<td>PLA</td>
<td>25 – 70</td>
<td>&gt;0.12</td>
</tr>
</tbody>
</table>

**measurement conditions:**
- OTR @ 23°C, 50% RH
- WVTR @ 38°C, 90% RH
Basic System Layout – Al/AloX + Top Coat

top coating

oxygen inlet

evaporation boats
# Freshure® Top Coat

| Specification of process step | OTR [cc/m²/day @23°C; 0% RH] | | | |
|-----------------------------|-----------------------------|-----------------------------| | |
|                            | 12µmPET-AlOx without Freshure® Top Coat | 12µmPET-AlOx with Freshure® Top Coat | | |
| After Coating               | 1.2                         | 0.6                         |  | |
| After printing (rotogravure)| 6.0                         | 1.3                         |  | |
| After retort*               | 5.8                         | 1.7                         |  | |

* Laminated against OPA/CPP. Retort condition: 30 min at 121°C

## Better Barrier:
Freshure® Top Coat provides better barrier and protects the film during downstream processing.

## Better Printability:
Freshure® Top Coat provides good ink adhesion without loss of glossy appearance of metallized film.

Source: DSM
SEM: Freshure® Layer on Al-Coated OPP

Signal A = InLens

Mag = 100.00 K X
EHT = 5.00 kV
Date :26 Mar 2012
Time :13:56:24

Cursor Width = 276.9 nm
**Freshure® Top Coat**

- **Freshure parameters:**
  - 50 nm @ 8m/s
  - NU ~ ± 5%

- Oxygen barrier improvement on PET by Freshure Top Coat
  - AIOx + Freshure

Top coat acts as protection for barrier layer possibility to eliminate downstream processes
temperature ramp for Freshure evaporation leads to constant deposition rate over coating length
Impact of Substrate Material

- 3 different PET foils in a composite roll (1000m each)
- identical process for all substrates

**AlOx + Freshure @ different PET Types**

<table>
<thead>
<tr>
<th>PET Film type</th>
<th>OTR Barrier</th>
<th>WVTR Barrier</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.41</td>
<td>2.22</td>
</tr>
<tr>
<td>2</td>
<td>0.25</td>
<td>0.60</td>
</tr>
<tr>
<td>3</td>
<td>0.49</td>
<td>1.26</td>
</tr>
</tbody>
</table>

**strong impact of substrate material on barrier performance**
Freshure® Top Coat

Improvement of adhesion through post treatment → printability of barrier-coated substrates
## Barrier Performance of Freshure® Coated Substrates

<table>
<thead>
<tr>
<th>Substrate</th>
<th>OTR [cc/m²day]</th>
</tr>
</thead>
<tbody>
<tr>
<td>PET</td>
<td>~3</td>
</tr>
<tr>
<td>OPP</td>
<td>40</td>
</tr>
<tr>
<td>PLA</td>
<td>20</td>
</tr>
<tr>
<td>OPA</td>
<td>1</td>
</tr>
</tbody>
</table>

**Measurement conditions:**
- OTR @ 23°C, 50% RH
- WVTR @ 38°C, 90% RH
Barrier Requirements for Electronic Applications

<table>
<thead>
<tr>
<th>Barrier Performance (g/m²/day)</th>
<th>Application</th>
</tr>
</thead>
<tbody>
<tr>
<td>10⁻⁶</td>
<td>OLEDs</td>
</tr>
<tr>
<td>10⁻⁵</td>
<td>Solar Cells</td>
</tr>
<tr>
<td>10⁻⁴</td>
<td>Thin-Film Batteries</td>
</tr>
<tr>
<td>10⁻³</td>
<td>Sensors, Electrophoretic Displays</td>
</tr>
<tr>
<td>10⁻²</td>
<td>RFID</td>
</tr>
<tr>
<td>10⁻¹</td>
<td>Electrochromic Displays</td>
</tr>
<tr>
<td>10⁻⁰</td>
<td>Medical Packaging</td>
</tr>
<tr>
<td>10⁰</td>
<td>Food Packaging</td>
</tr>
</tbody>
</table>

Barrier required to protect active layers from oxygen & water vapour
PECVD SiNₓ Barrier Coating on Polyimide @ 200°C

With a Film Thickness for Approximately 50nm WVTR decreases to 2.0E-2 g/m²/day
Summary

- High rate deposition process with excellent uniformity over complete coating width and length

- Different processes for AlOx based on thermal boat evaporation
  - Without plasma: For transparent barrier on PET
  - With plasma: For robust transparent barrier on PET, OPP, CPP, PLA

- Stable processes based on proven technology

- Environmental-friendly coatings

- High process flexibility for metallizing, different transparent AlOx-processes (depending on required coating properties) and for Freshure® top- or single coating

- Development of new R2R processes, e.g. PECVD, for future high-barrier requirements
Turning innovations into industries.™