Applied Research for Vacuum Web Coating: What is Coming Next?

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presenter’s perspective
R&D services for the industry and flexible products
Service along the R&D chain for flexible products

- testing substrate materials
- coating technologies
- key components
- application
- technology transfer
- pilot production
Hot R&D topics

- ultra-thin flexible glass
- high-rate PECVD
- advanced packaging films
- encapsulation of flexible electronics
- flexible materials for batteries
- functional films for buildings and outdoor use
## Ultra-thin flexible glass

<table>
<thead>
<tr>
<th>product, application</th>
<th>ultra-thin flexible glass for displays, wearables, sensors, batteries</th>
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<tbody>
<tr>
<td>state of the art</td>
<td>increasing availability of flexible glass</td>
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<tr>
<td>R&amp;D need</td>
<td>surface functions</td>
</tr>
<tr>
<td>approach, solution</td>
<td>roll-to-roll and sheet-to-sheet vacuum coating technologies</td>
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**latest R&D results at FEP**

- installation of a vacuum pilot roll coater (ARDENNE GmbH)
- roll-to-roll coating technology development: magnetron sputtering (e.g. ITO)
- Flash lamp annealing (FLA)
Pilot roll-to-roll coater FOSA labX 330 glass

- Flexible glass, polymer film, metal foil
- up to 330 mm deposition width
- Substrate temperature up to 350 °C
- up to 4 coating zones
- Dual Anode Sputtering
- Front-side touchless

Interleaf winding system
Rewinder
Unwinder
ei./opt Inline-Monitoring
Heater
Rotatable Magnetron
Ultra-thin flexible glass
Project KONFEKT: R2R Coating on Flexible Glass

Objective
- Development of adapted coating equipment
- Application development
- Establishing cooperation with glass makers and lamination facilities

Technology
- Sputtering & heating
- Sputtering and lamination processes

project funded by BMBF, contract Nr. 13N13818
## High rate PECVD process

<table>
<thead>
<tr>
<th>product, application</th>
<th>protective or adhesion promoting layers for various applications</th>
</tr>
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</table>
| R&D need             | • increase of productivity  
|                      | • technology tailoring for specific applications               |
| approach, solution   | arcPECVD: high-rate PECVD process                             |
| latest R&D results at FEP | • protective layer on barrier film  
|                      | • adhesion promoting layer  
|                      | • anti-fingerprint  
|                      | • color coatings                                                 |
High rate PECVD: arcPECVD

- Low pressure PECVD (0.1 – 5 Pa)
- Very high coating rates (> 2000 nm m/min)
- Plasma sources for web widths > 2.85 m
Protective coating on a barrier layer by arcPECVD

- Roll-to-roll in-line deposition of both barrier layer and protective layer: in-line combination of sputtering and arcPECVD
- Protective layer provides significant protection of barrier layer
## Advanced packaging films

<table>
<thead>
<tr>
<th>product, application</th>
<th>Transparent barrier films for packaging</th>
</tr>
</thead>
</table>
| state of the art     | • increasing need for transparent barrier films  
|                      | • AlOx technology available             |
| R&D need             | • advanced product quality (barrier, convertability)  
|                      | • wide range of polymer films (including biopolymers)  
|                      | • retortable packaging                  |
| approach, solution   | HAD-AlOx technology: Plasma-supported reactive evaporation of Al from boats |
| latest results at FEP| • several industrial installations together with Applied Materials WEB Coating GmbH  
|                      | • advanced barrier performance on a wide range of polymer films |
**HAD-AlOx technology**
*(HAD: Hollow cathode Activated Deposition)*

Plasma assisted AlO\textsubscript{x} evaporation

- High density oxygen plasma expands into evaporated Al plume
- Molecular oxygen strongly dissociated & incorporated at growth surface
- High degree of control of *energetic* particle flux to growth surface significantly expanding process window
## Barrier performance: comparison with conventional AlOx technology

<table>
<thead>
<tr>
<th>Substrate</th>
<th>Uncoated WVTR</th>
<th>Standard AlO$_x$ WVTR</th>
<th>Plasma Assisted AlO$_x$ WVTR</th>
<th>Uncoated OTR</th>
<th>Standard AlO$_x$ OTR</th>
<th>Plasma Assisted AlO$_x$ OTR</th>
</tr>
</thead>
<tbody>
<tr>
<td>PET (12 µm)</td>
<td>40-50</td>
<td>≤ 0.7</td>
<td>≤ 0.35</td>
<td>100-140</td>
<td>≤ 1.6</td>
<td>≤ 0.8</td>
</tr>
<tr>
<td>BOPP (17 µm)</td>
<td>4-7</td>
<td>≤ 7</td>
<td>≤ 0.30</td>
<td>2000-2500</td>
<td>≤ 50</td>
<td>≤ 35</td>
</tr>
</tbody>
</table>

WVTR: Water Vapor transmission rate, measured in g(m$^2$ d) at 38°C, 90 % r. h.
OTR: Oxygen transmission rate, measured in cm$^3$/(m$^2$ bar day) at 23°C, 0 % r. h.

- Optical transmission ≥ 98%
  (measured inline during coating process)
- Web speed 8 m/s

Source: Neil Morrison, Applied Materials WEB Coating GmbH
Presentation at AMI Coral Springs, Florida, USA, 2017
Barrier performance for “non-conventional” substrates

<table>
<thead>
<tr>
<th>Polymer film type</th>
<th>OTR [cm³/m² × d × bar] (23°C, 0 % r. h.)</th>
<th>WVTR [g/m² × d] (38°C, 90 % r. h.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PLA 20 µm</td>
<td>25</td>
<td>25</td>
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<tr>
<td>CPP 20 µm</td>
<td>50</td>
<td>0.5</td>
</tr>
<tr>
<td>PE 20 µm</td>
<td>40</td>
<td>0.9</td>
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</tbody>
</table>

Optical transmission ≥ 98%

Web speed 8 m/s

Barrier values may vary depending on substrate and process conditions.
### High barrier films for encapsulation of flexible electronics

<table>
<thead>
<tr>
<th>product, application</th>
<th>transparent high barrier films for encapsulation of flexible electronics</th>
</tr>
</thead>
</table>
| state of the art                  | • increasing number of flexible electronic products (like flexible organic solar cells)  
                                       • increasing need for encapsulation |
| R&D need                          | • barrier, optical performance, low defects rate  
                                       • reduction of cost  
                                       • production equipment |
| approach, solution                | • systematic investigation of sputtering process  
                                       • development of substrate smoothing layer based on electron beam curable coatings |
| latest R&D results at FEP         | • reduction of defect rate in sputtering processes  
                                       • optimized winding procedure  
                                       • optimization of layer composition |
Project OPTIPERM: Encapsulation films for flexible electronics

<table>
<thead>
<tr>
<th>Objective</th>
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<tbody>
<tr>
<td></td>
<td>Simplified barrier layer</td>
<td></td>
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<tr>
<td></td>
<td>stack</td>
<td></td>
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<tr>
<td></td>
<td>Robust technology concept</td>
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<table>
<thead>
<tr>
<th>Technology</th>
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<tr>
<td></td>
<td>Wet coating and electron</td>
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<td></td>
<td>beam curing</td>
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<td></td>
<td>Sputtering</td>
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**Wet coating**  
**Sputtering**

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**Planarizer**  
**Barrier**  
**PET-Substrate**

**WVTR [g m\(^{-2}\) day\(^{-1}\)]**

- **SiO\(_2\)**
- **Si\(_3\)N\(_4\)**

Substrate: PC without planarizer

= measurement limit Brugger

**Project partners**

- VON ARDENNE
- 3D Micromac
- GfE Fremat
- IOT
- Vision optics

Funding contract Nr. 100236574/3160
**Flexible materials for batteries**

<table>
<thead>
<tr>
<th>Product, Application</th>
<th>Future batteries with improved energy density</th>
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<tbody>
<tr>
<td>R&amp;D Need</td>
<td>Cost efficient technologies for thin functional layers</td>
</tr>
<tr>
<td>Approach, Solution</td>
<td>Vacuum roll-to-roll technologies</td>
</tr>
</tbody>
</table>
| Latest R&D Results at FEP | • Si anode layers on copper foils for Lithium-Sulfur batteries  
|                      | • Protective layers on current collectors for Lithium Metal Polymer batteries  
|                      | • Plasma supported coating technologies for solid state electrolytes |
Vacuum deposited thin films: potentials and R&D needs

- Anode: Si, Li
- Cathode: e.g. LiCoO$_2$
- Interfacial layer on current collector
- Solid electrolyte
- Thin film coated separator
- Collector
- Graphite layer
- Metal oxide layer
- Charging and discharging
Silicon anodes for Li-S-Batteries

Copper foil with silicon anode layers – SEM picture of an ion beam prepared cross-section

Capacity vs. charge cycles after additional structuring of Si anode layers and testing in test cells
Roll-to-roll process for high-rate deposition of solid electrolyte layers on metal foils

Schematic layout of an arrangement for depositing a LiPON solid electrolyte layer

Coating drum

Coated metal foil

Plasma system

Flange unit including 4 inductive evaporators
4 reactive gas inlets
High-rate deposition of solid electrolyte: Influence of the plasma activation

**Without plasma activation**
No nitrogen integration

**With plasma activation**
high nitrogen integration
## Functional films for architecture and outdoor use

| product, application | functional films for outdoor use like  
|                      | • flexible solar cells  
|                      | • integration in membrane roofs and façades |
| R&D need             | vacuum PVD and PECVD on outdoor-stable substrates |
| approach, solution   | optimization of vacuum coating processes to special properties of fluoropolymer substrates |
| latest R&D results at FEP | • sputtered permeation barriers on ETFE with same performance as on PET  
|                       | • outdoor-stable anti-reflective surface treatment |
Permeation Barrier Performance on Fluoropolymers

reactive dual magnetron sputtering zinc-tin-oxide (ZTO)

WVTR at 38 °C / 90 % r.h. [g/(m²d)]

- ZTO on PVDF PVDF-1008, 50 µm
- ZTO on PET Melinex 400, 75 µm
- ZTO on ETFE 6235-Z, 100 µm

ZTO layer thickness [nm]
Reactive Plasma Etching of ETFE surfaces to promote adhesion thin layers

- top-coat deposition
- thin SiO₂ layer (< 10 nm)
- seed layer deposition
- thin SiO₂ (< 10 nm)
- oxygen plasma etching
- process drum
Summary

R&D for vacuum web coating

- ultra-thin flexible glass
- advanced packaging films
- encapsulation of flexible electronics
- flexible materials for batteries
- functional films for buildings and outdoor use
- high-rate PECVD

I thank you for your attention