Conversion of aluminium oxide coated films –
From a single layer material to the finished & filled pouch

Carolin Struller, Manchester Metropolitan University & Bobst Manchester
Peter Kelly, Manchester Metropolitan University
Nick Copeland, Bobst Manchester

2016 AIMCAL Web Coating & Handling Conference USA
The Peabody, Memphis, Tennessee, 9th – 12th October 2016
Outline

- Introduction
  - Motivation and market potential
  - Previous work & findings
  - AlOₓ conversion project
- Topcoat development
- Conversion trials
  - Machine platforms
  - Pouch design & structure
  - Barrier through conversion
  - Flex-durability & barrier retention on elongation
  - Laminate bond strength
- Summary and conclusions
Introduction
Motivation and market potential

**AlOₓ/SlOₓ packaging applications**

- Lidding material (meat, pasta, cheese …)
- Pouches (liquid & dry foods, sauce, meat …)
- Sachets (sauce)
- Pillow/flow packs (pasta, snack foods, bars, chocolate …)
- Retort applications
- Medical, pharmaceutical & personal care packaging

(Camvac and Amcor products)
Motivation and market potential

$\text{AlO}_x/\text{SiO}_x$ existing products and current producers

<table>
<thead>
<tr>
<th>Producer</th>
<th>Barrier layer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Amcor Flexibles</td>
<td>SiO$_x$</td>
</tr>
<tr>
<td>Biofilm</td>
<td>AlO$_x$</td>
</tr>
<tr>
<td>Camvac</td>
<td>AlO$_x$</td>
</tr>
<tr>
<td>DNP</td>
<td>SiO$_x$, AlO$_x$</td>
</tr>
<tr>
<td>JBF RAK</td>
<td>AlO$_x$</td>
</tr>
<tr>
<td>Mitsubishi Plastics</td>
<td>SiO$_x$</td>
</tr>
<tr>
<td>Mitsui Chemicals Tohcello</td>
<td>AlO$_x$</td>
</tr>
<tr>
<td>Oike Industrial</td>
<td>SiO$_x$</td>
</tr>
<tr>
<td>Reiko</td>
<td>SiO$_x$, AlO$_x$</td>
</tr>
<tr>
<td>Toppan Printing</td>
<td>SiO$_x$, AlO$_x$</td>
</tr>
<tr>
<td>Toray Advanced Film</td>
<td>AlO$_x$</td>
</tr>
<tr>
<td>Toyobo</td>
<td>AlO$_x$/SiO$_x$</td>
</tr>
<tr>
<td>Uflex</td>
<td>AlO$_x$</td>
</tr>
<tr>
<td>Ultimet Films</td>
<td>AlO$_x$</td>
</tr>
</tbody>
</table>

Dole Fruit pots

Dole Fruit pots

$\text{AlO}_x$

LifePAK Dietary Supplement

LifePAK Dietary Supplement

$\text{AlO}_x$

Uncle Ben's microwavable rice/
Fall River Wild Rice microwave pouch

SiO$_x$

Fairytale Brownies Individual Wrapper

Fairytale Brownies Individual Wrapper

SiO$_x$

https://www.flexpack.org/

http://www2.dupont.com/Packaging/en_US/news_events/16th_dupont_packaging_award_winners.html
Motivation and market potential
Market size and growth

Global market volume
(chart based on metric tons)

Annual growth rate

<table>
<thead>
<tr>
<th>Material Type</th>
<th>CAGR</th>
</tr>
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<tbody>
<tr>
<td>Aluminium foil</td>
<td>&lt; 1 %</td>
</tr>
<tr>
<td>PVdC coated films</td>
<td>Negative growth</td>
</tr>
<tr>
<td>AlOₓ &amp; SiOₓ coated films</td>
<td>7 – 8 %</td>
</tr>
<tr>
<td>Metallised films</td>
<td>4 – 5 %</td>
</tr>
</tbody>
</table>

UV barrier properties: metallised PET vs. AlOₓ coated PET

- Aluminium foil
- EVOH (coextruded & coated)
- PVdC coated films
- High barrier metallised films
- AlOₓ & SiOₓ coated films

Alloy Development Corp.
Applied Market information Ltd.
C. Reardon (AWA) AIMCAL Europe 2014
Summary: Previous work/results
Single layer AlOₓ & initial conversion work

Characterization of:
- Base film *surface defects* and *surface chemistry* and impact on barrier performance
- AlOₓ and base film *surface topography*
- AlOₓ *thickness, adhesion & surface energy*
- *Stretch durability* of AlOₓ coated films
- AlOₓ coating *composition/stoichiometry*
- *Lamination* of non-printed/non-topcoated material
- *Slitting* of AlOₓ coated material
Summary: Previous work/results
Single layer AlOₓ & initial conversion work

RESULTS Lamination

**AlOₓ coated BOPP**

**AlOₓ coated PET**

Shaded area: AlOₓ PET (before lamination)

OTR 23 °C, 50 % RH
WVTR 37.8 °C, 90 % RH
AIOₓ conversion project
Project aims/deliverables

Overall Project Aim: Offer the film producer and/or the convertor part or full turn-key conversion solutions utilising AIOₓ transparent barrier. This involves both the machine platforms and the associated raw materials (films, ink, adhesives, coatings etc.).

Drivers for project: Environmentally friendly packaging ‘chlorine free’/ sustainable packaging, cost reduction (triplex to duplex solutions), transparency/ product visibility, microwaveable, improved shelf life.

Project Funding: KTP – Innovate UK (formerly TSB) & Bobst Manchester

Duration: 3 years

Start Date: Oct 2013
**AIoₓ conversion project**

**Project team**

### Industrial Project Partners

**Equipment Providers:**
- **Bobst Manchester Ltd** – Metalliser platform provider
- **Bobst Bielefeld GmbH** – Flexo press platform provider
- **Bobst Italia SpA** – Rotogravure press/ coater/ laminator platform provider
- **Atlas Converting Equipment Ltd** – Slitting platform provider

### Film Producers:
- BOPP film producers
- PET film producers
- AIoₓ film producers

### Raw Material/ Consumable Suppliers:
- Coatings
- Inks
- Adhesives

### Academic Partners/ Support:
- Manchester Metropolitan University (MMU)
- Oxford University

### Convertors:
- **Printpack** – Large convertor supplying to BOs
AlO$_x$ benefits for conversion

- Optimized AlO$_x$ thickness & oxidation rate → Flexibility
- High AlO$_x$ adhesion to the substrate film → > 5 N/(15 mm)
- High AlO$_x$ surface energy → 60 – 65 mN/m (maintained over 2 years)
- AlO$_x$ PET stretch durability OTR → 3 %
- Ceramic nature → chemically inert & temperature resistant

AlO$_x$ drawbacks for conversion

- Ceramic nature → brittle (aluminium = ductile)
- AlO$_x$ transparency → Conversion damage not visible
- AlO$_x$ PET stretch durability WVTR → 1.5 %
Topcoat development
Topcoat development

Methodology

- Laboratory coating
  - Coating technique
  - Material screening
  - Test of protective properties
  - Optimum coating thickness

- Pilot coating
  - Material screening
  - Coating technique

- Industrial coating
  - Machine optimisation
  - Coating parameter optimisation
  - Coating technique optimisation

- Campaign running
  - At selected customer sites

- Marketing/Promotion
Topcoat development
Industrial trials

EXPERIMENTAL
- CL 850D coater/laminator (Bobst Italia)
- Topcoating of 12 µm AlOₓ coated PET
- Rotogravure coating application
- Coating speed 250 m/min

- Water based topcoats
- Different topcoat formulations (chemistries) tested
RESULTS

- Topcoats successfully applied without damaging AlOₓ
- Barrier performance post-topcoating depends on topcoat chemistry
- Barrier topcoat C improves OTR and WVTR significantly

**Barrier properties** of topcoat material and possible *pore filling effects* are important for barrier enhancement.
AlO_x conversion trials
AlO$_x$ conversion trials

Conversion steps

1. K5000
2. Topcoating
3. Printing
4. Slitting, pouch making & filling
5. Lamination

12 μm PET

AlO$_x$ Coating
**AIOₓ conversion trials**

**AIOₓ coating**

**EXPERIMENTAL**

- Bobst K5000 vacuum metalliser
- AIOₓ coating speed: 600 m/min
- In-line plasma pre-treatment

- Optimised AIOₓ process for increased convertibility of coated film
- Starting material: low-cost commodity grade 12 μm corona treated PET; 32 km roll length
**AIO$_x$ conversion trials**

**Topcoating**

**EXPERIMENTAL**
- Trials performed at Bobst Italia
- AIO$_x$ optimised Bobst CL 850D coater/laminator
- Topcoating of 12 µm AIO$_x$ coated PET
- Rotogravure coating application
- Coating speed 250 m/min

- Water based topcoats: ① protective topcoat and ② barrier & protective topcoat
- Coatings compliant with relevant food contact legislation
AlOₓ conversion trials
Rotogravure printing

EXPERIMENTAL
- Printing conducted at Bobst Italia
- Bobst RS 4003MP 8 colour gravure printing press (standard)
- Printing speed: 200 m/min
- High performance commercially available ink system (solvent based)
AIO$_x$ conversion trials

Flexographic printing

**EXPERIMENTAL**

- Printing conducted at Bobst Bielefeld
- Bobst 20SIX CI 8 colour flexo printing press (standard)
- Printing speed: 200 m/min

- High performance commercially available ink system (solvent based)
EXPERIMENTAL

- Bobst CL 850D coater/laminator (Bobst Italia)
- Solvent based adhesive lamination
- High performance 2-comp PU adhesive

- Flexo trolley adhesive coating system
- Lamination at 150 m/min
- Secondary material: 32 μm PE sealant web

Light microscope image
(after Microtome sectioning of laminated film)
**AI0x conversion trials**

**Analytical tests & equipment**

### Barrier performance
- Mocon Oxtran 2/20 and Permatran-W 3/33
- Systech Illinois 8001 and 7001
- OTR: 23 °C, 50 % RH
- WVTR: 37.8 °C, 90 % RH

### Flex-durability (Gelbo-Flex)
- Gelbo-Flex tester model 5000 (United States Testing Co., Inc.)
- 1, 5 & 20 cycles and subsequent barrier determination

### Stretch-durability
- Hounsfield H10KS tensile tester
- QMat 5.52 software
- Stretching in machine direction & subsequent barrier determination

### Scanning electron microscopy (SEM)
- Zeiss Supra 40VP field emission gun SEM
- Analysis of film surface and laminate cross sections
POUCH DESIGN

FRONT

LEADING INNOVATION IN AIOx CONVERSION

CLEAR WINDOW

BACK

INNOVATIVE CLEAR BARRIER FILM
AIOx deposition: K4000 & K5000
Top coating: CL 850D
Gravure printing: RS 4000 MP
Lamination: CL 850D

For more information, please contact sales.manchester@bobst.com
www.bobst.com

HONEY ROASTED CASHEWS

INGREDIENTS Cashews, Sugar, Honey
NUTRITIONAL INFORMATION Amount per 100 grams:
Carbohydrates 11g, Total Fat 4.5g, Total Carbohydrate 18g, Dietary Fiber 1g, Saturated Fat 0.5g, Protein 12.5g, Sodium 1mg
Allergy Advice: Contains cashew nuts. May contain traces of peanuts. May not be suitable for those allergic to tree nuts.

40g

POUCH DESIGN

4 side sealed pouch (not stand-up)
POUCH DESIGN

4 side sealed pouch (not stand-up)
AIOₓ conversion trials

Pouch structure

Inside pouch (food contact side)

- Outside pouch
  - PET (12 μm)
  - AIOₓ
  - Topcoat
  - Ink (reverse printing)
  - Adhesive
  - PE sealant (32 μm)

Inside pouch

- CL 850D
- 4003MP (20SIX)
- CL 850D
AlOₓ conversion trials

Pouch structure – SEM cross-section

- Topcoat
- PET film
- Pigments coloured ink (red)
- Pigments white ink (TiO₂)
- Adhesive
- Ink
- AIOₓ
### RESULTS

- **Barrier performance**

<table>
<thead>
<tr>
<th></th>
<th>OTR</th>
<th>WVTR</th>
</tr>
</thead>
<tbody>
<tr>
<td>OTR</td>
<td>1.0 – 1.5</td>
<td>&lt; 1.0</td>
</tr>
<tr>
<td>WVTR</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**PET/AlOₓ (pre-conversion)**

<table>
<thead>
<tr>
<th></th>
<th>OTR</th>
<th>WVTR</th>
</tr>
</thead>
<tbody>
<tr>
<td>OTR</td>
<td>0.65 ± 0.09</td>
<td>0.09 ± 0.04</td>
</tr>
<tr>
<td>WVTR</td>
<td>0.56 ± 0.10</td>
<td>0.42 ± 0.03</td>
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</tbody>
</table>

**Topcoating**

<table>
<thead>
<tr>
<th></th>
<th>OTR</th>
<th>WVTR</th>
</tr>
</thead>
<tbody>
<tr>
<td>OTR</td>
<td>0.78 ± 0.10</td>
<td>0.12 ± 0.08</td>
</tr>
<tr>
<td>WVTR</td>
<td>0.70 ± 0.05</td>
<td>0.45 ± 0.06</td>
</tr>
</tbody>
</table>

**Printing**

<table>
<thead>
<tr>
<th></th>
<th>OTR</th>
<th>WVTR</th>
</tr>
</thead>
<tbody>
<tr>
<td>OTR</td>
<td>0.77 ± 0.10</td>
<td>0.11 ± 0.07</td>
</tr>
<tr>
<td>WVTR</td>
<td>0.69 ± 0.06</td>
<td>0.44 ± 0.13</td>
</tr>
</tbody>
</table>

**Lamination**

<table>
<thead>
<tr>
<th></th>
<th>OTR</th>
<th>WVTR</th>
</tr>
</thead>
<tbody>
<tr>
<td>OTR</td>
<td>0.59 ± 0.02</td>
<td>0.11 ± 0.09</td>
</tr>
<tr>
<td>WVTR</td>
<td>0.69 ± 0.01</td>
<td>0.41 ± 0.01</td>
</tr>
</tbody>
</table>

**Slitting**

*PET/AlOₓ/topcoat*  
*PET/AlOₓ/topcoat/ink*  
*PET/AlOₓ/topcoat/ink/ad/PE*  

---

OTR in cm³/(m² d) @ 23 °C, 50 % RH  
WVTR in g/(m² d) @ 37.8 °C, 90 % RH
AIOₓ conversion trials
Barrier through conversion – Gravure printing

RESULTS

- Barrier uniformity across web width & length
- Tested for all conversion steps → reliability

<table>
<thead>
<tr>
<th>65 mm</th>
<th>313 mm</th>
<th>625 mm</th>
<th>938 mm</th>
<th>1185 mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>OTR</td>
<td>1.17 ± 0.08</td>
<td>1.40 ± 0.05</td>
<td>1.26 ± 0.08</td>
<td>1.21 ± 0.06</td>
</tr>
<tr>
<td>WVTR</td>
<td>0.75 ± 0.11</td>
<td>0.82 ± 0.01</td>
<td>0.83 ± 0.05</td>
<td>0.73 ± 0.01</td>
</tr>
</tbody>
</table>

PET/AIOₓ (pre-conversion)

<table>
<thead>
<tr>
<th>65 mm</th>
<th>313 mm</th>
<th>625 mm</th>
<th>938 mm</th>
<th>1185 mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>OTR</td>
<td>0.79 ± 0.22</td>
<td>0.71 ± 0.05</td>
<td>0.85 ± 0.01</td>
<td>0.75 ± 0.03</td>
</tr>
<tr>
<td>WVTR</td>
<td>0.69 ± 0.11</td>
<td>0.63 ± 0.08</td>
<td>0.71 ± 0.04</td>
<td>0.75 ± 0.01</td>
</tr>
</tbody>
</table>

PET/AIOₓ/topcoat/ink/ad/PE (protective topcoat)

OTR in cm³/(m² d) @ 23 °C, 50 % RH
WVTR in g/(m² d) @ 37.8 °C, 90 % RH
AIOₓ conversion trials
Barrier through conversion – Gravure printing

RESULTS

- Barrier topcoat

OTR in cm³/(m² d) @ 23 °C, 50 % RH
WVTR in g/(m² d) @ 37.8 °C, 90 % RH

- OTR improvement by factor > 10
- WVTR improvement by factor ≈ 2
AlO$_x$ conversion trials
Barrier through conversion – Gravure printing

RESULTS

- Protective topcoat

OTR improvement to < 1 cm$^3$/m$^2$ d)

OTR in cm$^3$/m$^2$ d) @ 23 °C, 50 % RH
WVTR in g/m$^2$ d) @ 37.8 °C, 90 % RH
RESULTS

- Barrier performance after Gelbo-Flex (laminate)

OTR in cm³/(m² d) @ 23 °C, 50 % RH
AlO\textsubscript{x} conversion trials
Flex-durability – Gravure printing

**RESULTS**

- Barrier performance after Gelbo-Flex (laminate)

![Graph showing WVTR in g/(m² d) @ 37.8 °C, 90 % RH vs. Number of Gelbo-Flex cycles with lines for Barrier topcoat and Protective topcoat.](image)
**AIO\text{x} conversion trials**
Barrier retention on elongation – Gravure printing

**EXPERIMENTAL & RESULTS**

- Characterization of stretch durability
- Stretching of laminate to a predefined elongation using tensile tester
- Assess capability of laminated film to withstand downstream processing

![Graph 1](#)

![Graph 2](#)
RESULTS

- Laminate bond strength investigations (conducted by project partner Printpack)

<table>
<thead>
<tr>
<th></th>
<th>Bond strength</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>g/inch</td>
<td>N/(15 mm)</td>
</tr>
<tr>
<td><strong>Barrier topcoat</strong></td>
<td>261 ± 13</td>
<td>1.5 ± 0.1</td>
</tr>
<tr>
<td></td>
<td>245 ± 8</td>
<td>1.4 ± 0.1</td>
</tr>
<tr>
<td><strong>Protective topcoat</strong></td>
<td>741 ± 84</td>
<td>4.3 ± 0.5</td>
</tr>
<tr>
<td></td>
<td>589 ± 169</td>
<td>3.4 ± 1.0</td>
</tr>
</tbody>
</table>

Picture source: ISO 11339
AlOₓ conversion trials
Barrier through conversion – Flexographic printing

RESULTS

- Barrier topcoat

OTR in cm³/(m² d) @ 23 °C, 50 % RH
WVTR in g/(m² d) @ 37.8 °C, 90 % RH

- OTR improvement by factor > 10
- WVTR improvement by factor ≈ 2
AlOₓ conversion trials
Barrier through conversion – Flexographic printing

RESULTS

- Protective topcoat

OTR improvement to < 1 cm³/(m² d)

<table>
<thead>
<tr>
<th>PET/AlOₓ</th>
<th>TOPCOATING</th>
<th>PRINTING</th>
</tr>
</thead>
<tbody>
<tr>
<td>OTR cm³/(m² d)</td>
<td>1.2</td>
<td>1.0</td>
</tr>
<tr>
<td>WVTR g/(m² d)</td>
<td>0.9</td>
<td>0.8</td>
</tr>
</tbody>
</table>

OTR in cm³/(m² d) @ 23 °C, 50 % RH
WVTR in g/(m² d) @ 37.8 °C, 90 % RH
Summary & conclusions

- Development of protective off-line topcoats for AlO$_x$ coated PET film (protective topcoat and barrier topcoat)
- Successful application of off-line topcoat on AlO$_x$-optimised industrial coater
- Full conversion of AlO$_x$ coated PET film to the final package, using standard industrial printing, laminating and pouch making equipment
- Barrier maintained successfully throughout conversion chain (across web width)
- Laminate shows very good flex & stretch durability (barrier topcoat)
- Final laminate fulfils market requirements in terms of bond strength
Acknowledgements

- BOBST Italia SpA and BOBST Bielefeld GmbH
- Manchester Metropolitan University
- Printpack Inc.
- Michelman SARL
  Collaborative work also presented in: ‘Unique High Oxygen Barrier Coatings for Food Packaging’, Vince Zabrocki, Michelman, SPE/FlexPackCon, Monday 9/10/2016, 2:30 pm – 3:00 pm
- Flint Group Italia SpA
  & Flint Group Germany GmbH
- Dow/Rohm and Haas Italia Srl
- Innovia Films Ltd
- RDM Test Equipment Ltd
- Innovate UK
Thank You!

Come and visit us on the BOBST TableTop for a nut filled AlOₓ pouch!!