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Manuscript Details

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Title	Conversion of aluminium oxide coated films for food packaging applications – From a single layer material to the finished pouch
Short title	AlO _x conversion
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Abstract

Transparent barrier films based on vacuum deposited aluminium oxide (AlO_x) layers are continuing to create large interest in the market with regards to their use as food and healthcare packaging materials. Nevertheless, their post-metalliser conversion to the final packaging material still presents challenges to current AlO_x producers and the wider converting industry. In this work, AlO_x coated PET films have been converted in long duration industrial-scale trials via topcoating, printing, lamination and finally pouch making. Throughout this process, each conversion step has been investigated for its effects on the barrier performance. It was found that the printing processes, especially, induce significant damage to the ceramic barrier layer. However, by the use of a protective topcoat prior to any conversion step, the barrier properties of the AlO_x coated film were preserved, or could even be significantly enhanced, depending on the topcoat material. Furthermore, for a barrier topcoat, remarkable stretch- and flex-durability properties were achieved in the final laminate.

Keywords	Aluminum oxide; transparent inorganic barrier layers; conversion; printing; lamination
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Suggested reviewers	Charles Bishop, Hazel Assender

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Dear Editors,

We would like to submit our paper entitled “Conversion of aluminium oxide coated films for food packaging applications – From a single layer material to the finished pouch”, which describes the impact of standard converting processes on the barrier properties of AlOx-based transparent packaging material. We can confirm that this paper isn't under consideration with any other journal.

'Declarations of interest: none'

Dear Authors,

The manuscript is well written and has useful information. It can be accepted for publication in the journal after revisions are made.

Please see below:

1) line 47/48/147: PVdC (polyvinylidene chloride), EVOH, HFPS etc. should be written as polyvinylidene chloride (PVdC), please be consistent in formatting with the rest of the manuscript.

Response - corrected

2) Please define the acronym PET before first use.

Response - corrected

3) line 142: Rohm and Hass Italia Sri (Italy)... no need to be bold.

Response - corrected

Thanks

-Reviewer 2

- A well-presented summary of the effects on barrier performance of AlO_x/PET films of the various film conversion properties, and then an investigation of the benefits induced by two 'protection layers'. The absence of any information on the nature of these protective layer coatings and the paucity of information on their deposition process means that it is difficult to put any 'why' on the performance improvements observed, but this report does serve to illustrate what can be achieved.

I would recommend a rewording in lines 265-270 which seems unclear in its logic - the manuscript makes reference to a critical thickness of the adhesive that depends on the thickness of barrier layer and therefore concludes that the barrier performance of the adhesive dominates that of the laminated polymer. It is not clear why a critical thickness that depends on the barrier material should be critical in dictating the relative importance of the adhesive and laminated polymer.

Response – text amended:

The thickness of the adhesive layer was between 2.5 and 3 μm (determined via light microscopy of microtome cross sections of the laminate). This means that the adhesive thickness is above the so-called critical thickness, which depends on the defect size distribution of the inorganic barrier layer and typically is less than 2.5 μm for the type of industrially produced barrier layers discussed here (Langowski, 2002). According to Langowski (Langowski, 2008), if the thickness of the polymer layer directly adjacent to the inorganic barrier layer is thicker than the critical thickness, then this layer along with the inorganic barrier layer is dictating the barrier performance of the system. For the work

discussed here this means that in the final laminate, the barrier properties of the polyurethane adhesive (significantly better oxygen barrier properties compared to polypropylene) dominate over those of the material laminated on top (i.e. the CPP film), which is of importance for the above discussion on barrier enhancement due to adhesive lamination.

-Reviewer 3

- Well done investigation work. Very interesting from a practical point of view.

Conversion of aluminium oxide coated films for food packaging applications –

From a single layer material to the finished pouch

Carolin Struller ^{a, b}, Peter Kelly ^b, Nicholas Copeland ^a

Highlights

- Full industrial conversion including topcoating, printing, lamination and pouching
- Most conversion damage induced to ceramic AlO_x layer during printing process
- Successful industrial conversion of AlO_x PET via use of protective offline topcoat
- Outstanding flex and stretch durability when protective barrier topcoat is used

'Declarations of interest: none'



1 Conversion of aluminium oxide coated films for food packaging applications – From a single
2 layer material to a complete pouch

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10 **Abstract**

11 Transparent barrier films based on vacuum deposited aluminium oxide (AlO_x) layers are
12 continuing to create large interest in the market with regards to their use as food and
13 healthcare packaging materials. Nevertheless, their post-metalliser conversion to the final
14 packaging material still presents challenges to current AlO_x producers and the wider
15 converting industry. In this work, AlO_x coated **polyethylene terephthalate (PET)** films have
16 been converted in long duration industrial-scale trials via topcoating, printing, lamination and
17 finally pouch making. Throughout this process, each conversion step has been investigated
18 for its effects on the barrier performance. It was found that the printing processes, especially,
19 induce significant damage to the ceramic barrier layer. However, by the use of a protective
20 topcoat prior to any conversion step, the barrier properties of the AlO_x coated film were
21 preserved, or could even be significantly enhanced, depending on the topcoat material.
22 Furthermore, for a barrier topcoat, remarkable stretch- and flex-durability properties were
23 achieved in the final laminate.

24 **Keywords:** aluminium oxide; transparent inorganic barrier layers; conversion; printing;
25 lamination

26

27

28

29

30 1 Introduction

31 Inorganic transparent barrier layers such as aluminium oxide (AlO_x) or silicon oxide (SiO_x)
32 are still in demand for clear barrier packaging materials, with applications ranging from food
33 stuffs, which have rather moderate barrier requirements, to electronic products such as
34 displays, where ultra-high barrier levels are essential. A number of different techniques are
35 available to deposit such inorganic layers including atomic layer deposition (ALD), plasma
36 enhanced chemical vapour deposition (PECVD), sputtering and thermal evaporation
37 processes (electron beam and boat evaporation) (Groner, George, McLean, & Carcia, 2006;
38 Kelly, 1994; Schiller, Neumann, Morgner, & Schiller, 1994). Whilst sputtering and ALD are
39 mainly of importance for high-end applications, PECVD and thermal evaporation techniques
40 are the processes of interest with regards to the cost-sensitive food packaging market.
41 Reactive evaporation using resistively heated boats represents an especially promising
42 candidate with great market potential due to the relatively low associated capital investment,
43 the use of inexpensive raw materials and the high process speeds that can be achieved,
44 without disadvantageous effects to the barrier performance, in comparison to other deposition
45 techniques.

46 The market for transparent oxide coated films has an estimated compound annual growth rate
47 of around 7 to 8 %, compared to only 2 to 3 % for polyvinylidene chloride (PVdC) coated
48 films and 4 to 6 % for ethylene vinyl alcohol (EVOH) copolymer co-extruded films (Platt,
49 2016); both of which are polymer based transparent barrier films that can potentially be
50 replaced by oxide coated films. Nevertheless, the market volume for these oxide based barrier
51 films is currently a lot smaller, compared to the polymer based options of PVdC and EVOH.
52 To date, the clear barrier flexible packaging market for AlO_x and SiO_x transparent barrier
53 films is still dominated by Japanese material producers, such as Toppan, Toray, DNP,
54 Mitsubishi and others with well-known film grades such as GL Film, Barrialex, IB-Film or
55 TECHBARRIER (DNP America, 2017; Mitsubishi Chemical, 2018; Toppan USA, 2016;
56 Toray Advanced Film Co., 2018), although Camvac's Camclear (Camvac Limited, 2018) and
57 Amcor's AmLite (formerly Ceramis) (Amcor Limited, 2018) have also become well-
58 established products. The Japanese producers predominantly apply electron beam evaporation
59 or PECVD techniques for the deposition of the barrier layers (Naegli & Lohwasser, 2001),
60 which entail substantially higher production and investment costs and more technical
61 complexity, including potential reliability issues, compared to reactive boat evaporation.
62 Furthermore, their products are generally topcoated with a material that not only protects the

63 inorganic AlO_x or SiO_x barrier layer through handling and conversion, but also significantly
64 enhances its barrier performance. This, in addition to the retortability of many of these
65 materials, makes them very high performance products, which comes at a certain cost level
66 and prevents real volume growth for cost-sensitive volume market applications. The reactive
67 boat evaporation process has been developed over the last few decades and is now well-
68 established (Günther et al., 2009; Kelly, 1993, 1994; Schiller et al., 1994; Struller, Kelly,
69 Copeland, & Liauw, 2012). Its vast economic potential for low cost AlO_x production leads to
70 new players continuously pushing to enter the attractive transparent barrier market and trying
71 to obtain market share from the more advanced Japanese players. However, unlike the
72 Japanese products, most of these ‘new’ products do not have a protective topcoat and their
73 conversion is challenging with inevitable barrier deterioration.

74 In order to obtain a fully viable commercial AlO_x product, the successful conversion of the
75 coated film also has to be taken into account. AlO_x coated polymer films will always need to
76 undergo further conversion steps in order to obtain the final packaging structure. These
77 conversion steps will include all or most of the following downstream processes; slitting,
78 coating, printing and lamination. Since each of these steps can potentially have a detrimental
79 impact on barrier performance, it is essential that barrier loss upon conversion is avoided or at
80 least minimised to achieve the barrier requirements of the target application. In this study, it
81 has been found that the printing step, especially rotogravure printing, is the most damaging
82 conversion process with regards to the AlO_x barrier performance, which has also been
83 identified by other researchers (Jahromi, 2016). This paper is a unique study on optimising
84 and facilitating the conversion process of AlO_x coated films in order to meet market
85 requirements for food packaging applications (with the aim of dry food packaging
86 applications such as snack foods, confectionary, biscuits or cereal bars). The focus, therefore,
87 is on developing a suitable offline topcoat solution, which is not only capable of protecting
88 the AlO_x barrier layer through all conversion steps (including pouch making), but also offers
89 additional barrier functionality. Once this topcoat is successfully applied using an
90 optimised/modified platform to prevent barrier loss upon web conveying and coating
91 application, the topcoated AlO_x polyethylene terephthalate (PET) films can be converted on
92 standard industrial equipment, without the need for changes and without any subsequent
93 barrier deterioration. Trials have been conducted using AlO_x coated PET film and each
94 process step was characterised with regards to its post-processing barrier performance.

95 Additionally, flex-durability (Gelbo-Flex) testing as well as barrier retention upon elongation
96 investigations were carried out.

97 **2 Material and methods**

98 **2.1 Coating/Conversion processes and platforms**

99 Standard low-cost commodity grade PET films (12 μm film thickness; 1250 mm width;
100 corona treated by the film manufacturer) were chosen for this investigation (note: especially
101 in the pre-assessment phase, PET films from a variety of film supplies were used). AlO_x
102 coating was conducted via reactive thermal evaporation using a Bobst Manchester Ltd
103 (Heywood, United Kingdom) K5000 vacuum metalliser with an optional AlO_x coating
104 system. The vacuum coater has a deposition source consisting of resistively heated
105 evaporation boats (standard intermetallic composite) onto which aluminium is continuously
106 fed in the form of a wire. Oxygen is introduced into the aluminium vapour stream in order to
107 produce a transparent aluminium oxide coating and an optical monitor beam and closed loop
108 control system is used to achieve consistent optical properties of the coated film across the
109 web width and length. The pressure during aluminium oxide deposition is of the order of 0.05
110 Pa. AlO_x deposition was carried out at a web speed of 600 m/min, whilst also applying
111 optimised in-line plasma pre-treatment using a medium frequency plasma source with
112 magnetically enhanced water cooled electrodes. To achieve enhanced convertibility of the
113 AlO_x coated starting material, the original AlO_x process (Struller, Kelly, & Copeland, 2014)
114 was further optimised in order to facilitate the handling of the AlO_x coated film via increasing
115 its flexibility. Thereby, the thickness of the coating layer was reduced from an average value
116 of 10 nm to a thickness in the range of 7 to 8 nm. For the final conversion work (including
117 topcoating, printing and lamination), 32 km reels were AlO_x coated.

118 Topcoating of the produced AlO_x PET reels was performed on an AlO_x optimised Bobst
119 CL 850D coater/laminator at Bobst Italia SpA (San Giorgio Monferrato, Italy) using a
120 forward gravure coating system at web speeds up to 250 m/min. The two coatings chosen are
121 water-based and were supplied by the project partner Michelman SARL (Windhof,
122 Luxembourg). These coatings are: a protective topcoat (offering protection through
123 conversion only) and a barrier topcoat (offering protection through conversion and additional
124 barrier functionality). Both coatings are compliant with relevant food contact legislations.

125 The rotogravure printing was also conducted at Bobst Italia using a standard Bobst
126 RS 4003MP 8 colour rotogravure printing press at web speeds of up to 400 m/min. For the
127 final conversion work (using a topcoat prior to printing), a solvent-based high performance
128 commercially available ink system was used, which was supplied by the project partner Flint
129 Group Italia SpA (Cinisello Balsamo, Italy).

130 Flexo printing was carried out on a standard Bobst 20SIX 8 colour CI (central impression)
131 flexo printing press at Bobst Bielefeld GmbH (Bielefeld, Germany). For the final conversion
132 work, printing was conducted at web speeds of 200 m/min using the flexo version of the
133 solvent-based high performance ink system used in the gravure printing trials, supplied by
134 Flint Group Germany GmbH (Willstätt, Germany). Printing (flexo and gravure) was
135 conducted as 'reverse' printing with the white colour printed last, since the printed material
136 itself will be on the outside of the packaging structure with the ink embedded within the
137 laminate (i.e. one looks through the PET film at the print or, in areas of no print, at the
138 packaged food stuffs).

139 Lamination was again carried out at Bobst Italia SpA using a solvent-based adhesive on a
140 Bobst CL 850D coater/laminator. A commercially available high performance, two
141 component polyurethane adhesive (supplied by the project partner Dow/**Rohm and Haas**
142 **Italia Srl (Mozzate, Italy)**) was used and applied via a flexo trolley coating application
143 system at a speed of 150 m/min. For the lamination in the pre-assessment stage, a 40 µm
144 thick corona treated cast polypropylene (CPP) sealant film was used; whilst for the full
145 conversion work a 32 µm corona treated linear low-density polyethylene (LLDPE) sealant
146 film was applied as secondary material.

147 All further conversion steps (slitting, pouch making via HFFS (horizontal form fill & seal)
148 and pouch filling) were arranged and managed by collaborator Printpack Inc. (Atlanta, GA,
149 United States) using their facilities, contacts and customer base.

150 **2.2 Analytical methods**

151 Barrier properties in terms of oxygen and water vapour transmission rates (OTR/WVTR)
152 were analysed in accordance with ASTM F 1927 and ASTM F 1249/ISO 15106-3 using a
153 Mocon Oxtran 2/20 and Systech Illinois 8001 for oxygen permeation and a Mocon
154 Permatran-W 3/33 and Systech Illinois 7001 for water vapour permeation. Test conditions
155 were 23 °C and 50 % relative humidity (RH) for OTR measurements and 37.8 °C and a

156 gradient of 90 % RH for WVTR measurements. Barrier properties were typically measured at
157 several points across the 1250 mm web width and average values are given.

158 A Zeiss Supra 40VP field emission gun scanning electron microscope (FEG SEM) was used
159 to acquire SEM images. All samples were examined using a low acceleration voltage to avoid
160 the need for coating with a conductive layer (which could potentially mask important surface
161 detail). Cross-sections were cut on a Leica RM2125 microtome prior to SEM analysis.

162 The flex durability or Gelbo-Flex (ASTM F392) of the laminated AlO_x coated films was
163 investigated using a Gelbo-Flex tester model 5000 manufactured by United States Testing
164 Co., Inc. For this investigation 1, 5 and 20 flexing cycles were performed and the barrier
165 performance was determined successively.

166 For the evaluation of the barrier retention on elongation behaviour, the films were stretched
167 to a pre-defined strain (between 0.5 and 5 %, stretching in the machine direction) by a tensile
168 testing unit (Hounsfield H10KS with QMat 5.52 software) and subsequently the barrier
169 properties were measured (test for stretch durability/resistance (Felts, 1993), however
170 samples are analysed in the relaxed state).

171

172 **3 Results and discussion**

173 **3.1 Printing and lamination pre-assessment**

174 **3.1.1 Flexographic and gravure printing**

175 During the initial assessment, printing conversion processes were analysed for their impact
176 on the barrier performance of AlO_x coated PET film (i.e. when the non-protective topcoat is
177 present). Therefore, trials were conducted using standard ‘non-optimised’ conversion
178 equipment at Bobst Italia and Bobst Bielefeld. During each interaction, several trials were
179 conducted on the printing presses, in order to investigate the variability that can be obtained
180 during direct printing onto the sensitive AlO_x PET material. Additionally, trials were
181 conducted on different machines of the same model, i.e. the RS 4003MP as well as the 20SIX
182 presses were investigated on two different occasions, whereby the machines were of the same
183 model, but not ‘physically’ the same machines. This was also part of the pre-assessment to
184 show the range of barrier deterioration that can occur on the same machine type. No special
185 care was taken in regards to the sensitiveness of the AlO_x PET film, but the web was printed

186 using parameters and run conditions typical for 12 μm uncoated PET film. Additionally, trials
187 were also carried out at a customer site in China. The latter was conducted in order to acquire
188 true field data of a trial that has not been executed under the supervision and control of the
189 authors/team at Bobst as well as using non-Bobst and older equipment (Chinese supplied 7
190 colour gravure printing press; make, model and age of machinery unknown to the authors;
191 solvent based inks used from Chinese supplier).

192 For each of the assessments, 4 km long rolls of AlO_x coated PET film were produced and
193 used to carry out the conversion trials and the results are summarised in Table 1. Barrier
194 numbers before conversion are given as a range which is typical for the PET films coated in
195 this work. For the post printing barrier performance, a large spread of data was obtained in
196 the different assessments of the same machine types as well as the various trials run on the
197 same machine. Due to this variability of transmission rates, the post printing ranges obtained
198 are given for OTR and WVTR, rather than an average value. As can be seen, after printing
199 (gravure and flexo) the barrier is deteriorated in all cases, although to a larger degree for the
200 gravure printing. This is due to the longer web path and larger amount of web handling
201 involved in the gravure process (printing units inline), versus CI flexo printing (printing units
202 arranged around central impression drum). The fact that web handling can have a detrimental
203 impact on the barrier performance of ceramic inorganic barrier layers, such as AlO_x coatings
204 deposited onto a polymer web, has also been shown by a group of researchers (Lahtinen,
205 Lahti, Johansson, Seppänen, & Cameron, 2014) who investigated the effect of post deposition
206 film handling on atomic layer deposited aluminium oxide coatings on biaxially oriented
207 polypropylene film. Also, the barrier data obtained from a gravure printing field trial at a
208 customer site falls within the spread of post-printing data obtained on the Bobst gravure
209 platform. Furthermore, Jahromi (Jahromi, 2016), has reported comparable barrier values for
210 printed unprotected AlO_x PET (OTR 6 to 12 $\text{cm}^3/(\text{m}^2 \text{ d})$ and WVTR 2 to 7 $\text{g}/(\text{m}^2 \text{ d})$), although
211 these samples were flexo printed, which in our case has been found to be significantly less
212 destructive than gravure printing.

213 In order to investigate the nature and origin of the damage to the AlO_x layer, further analysis
214 was conducted. In principle, the damage to the AlO_x layer (and hence barrier deterioration)
215 could be caused by either chemical, thermal or mechanical stress to the film. However, AlO_x
216 as a ceramic material should be chemically inert and also temperature resistant, which was
217 investigated and proven via laboratory assessment trials. Therefore, mechanical damage to

218 the AlO_x layer appeared to be the most likely cause of the barrier deterioration. In this case,
219 damage to the AlO_x coating could be due to stretching the film and hence cracking of the
220 AlO_x layer or mechanically scratching/scuffing the brittle AlO_x due to contact with rollers
221 (driven or idler) or the gravure printing head in the web handling mechanism. With regards to
222 the stretch durability of AlO_x coated PET film, the authors have previously shown (Struller et
223 al., 2012) that OTR can withstand up to 3 % elongation, whilst water vapour barrier is
224 retained up to 1 % elongation before deterioration. Based on the web tensions used during the
225 printing process, this is very unlikely to be the cause of the barrier decline seen after printing
226 and, hence, samples of the printing trials were analysed via scanning electron microscopy in
227 order to discover any visible kind of mechanical damage in the AlO_x layer. An example SEM
228 micrograph revealing the damaged AlO_x layer is show in Figure 1. The image on the left side
229 is at lower magnification in order to show the location of the damage on a larger scale.
230 Obviously, damage to the AlO_x coating can only be detected in the unprinted area, since it
231 will be covered up by the ink in the printed areas. On the right hand side, a higher
232 magnification image of the scratch marked in the low magnification image can be seen. It is
233 clearly noticeable that the AlO_x layer is damaged. Scratches like this were found to cover all
234 post-printing samples (exhibiting barrier deterioration) that were investigated and their
235 typical orientation was the machine direction (i.e. movement direction of the film during
236 processing). Consequently, it can be concluded that the barrier loss induced during printing is
237 caused by mechanical damage of the AlO_x coating during web handling on the printing press.

238 **3.1.2 Lamination**

239 In addition to assessment of the two printing processes, the lamination process was also
240 investigated for non-protected AlO_x coated PET film using a standard ‘non-optimised’
241 laminator. A range of lamination trials was conducted, whereby the adhesive was applied
242 directly onto the AlO_x layer and a CPP film was used as the secondary web. Unlike the
243 printing assessment, the post-lamination barrier performance was comparable to pre-
244 lamination properties and, hence, average values and associated standard deviations are given
245 in Table 1 for the AlO_x PET as well as the laminated structure (note, the average given has
246 been obtained from several lamination trials). Overall, no significant barrier deterioration is
247 seen when non-protected AlO_x PET film is laminated via direct application of the adhesive
248 onto the ceramic barrier layer using standard industrial equipment. Nevertheless, an oxygen
249 barrier improvement effect is frequently observed by lamination of vacuum coated (i.e.

250 metallised) films when using polyurethane based adhesives, based on the good oxygen barrier
251 properties of the adhesive itself (Mueller, Schoenweitz, & Langowski, 2012). It should be
252 noted here that the aluminium layer of a metallised film is less prone to damage during
253 conversion due to its ductility, compared to an AlO_x layer which is ceramic and hence brittle.
254 Langowski (Langowski, 2008) states that for a multilayer system with an inorganic barrier
255 layer embedded between two polymer layers ‘when the two polymer layers have the same or
256 very similar permeation coefficients, the permeability (i.e. transmission rate) is halved
257 compared to a system comprising a single polymer layer and the inorganic layer’. As no
258 degree of barrier improvement was seen when laminating the AlO_x coated PET film, it may
259 well be that damage of the AlO_x layer took place during the conversion process of
260 lamination. However, as we obtain a post-lamination barrier level comparable to the pre-
261 lamination level, it is assumed that the barrier performance loss is recovered due to the barrier
262 properties of the adhesive, as well as by a possible infiltration of the adhesive into the
263 defects/damage in the AlO_x layer (Miesbauer, Schmidt, & Langowski, 2008). The thickness
264 of the adhesive layer was between 2.5 and 3 μm (determined via light microscopy of
265 microtome cross sections of the laminate). This means that the adhesive **thickness** is above
266 the so-called critical thickness, **which depends on the defect size distribution of the inorganic**
267 **barrier layer** and typically is less than 2.5 μm for the type of **industrially produced** barrier
268 layers discussed here (Langowski, 2002). **According to Langowski (Langowski, 2008), if the**
269 **thickness of the polymer layer directly adjacent to the inorganic barrier layer is thicker than**
270 **the critical thickness, then this layer along with the inorganic barrier layer dictates the barrier**
271 **performance of the system. For the work discussed here this means that** in the final laminate,
272 the barrier properties of the **polyurethane** adhesive (**significantly better oxygen barrier**
273 **properties compared to polypropylene**) dominate over those of the material laminated on top
274 (i.e. the CPP film), which is of importance for the above discussion on barrier enhancement
275 due to adhesive lamination.

276

277 Another important conversion step with the potential to damage is the slitting process. This
278 has been found not to impair AlO_x barrier performance when conducted in a controlled
279 manner on standard slitting equipment and has been discussed by the authors in more detail
280 elsewhere (Struller et al., 2015).

281 **3.2 Topcoat development**

282 After the initial assessment of conversion processes and their impact on the barrier
283 performance of AlO_x coated films, the approach taken in this research work was to apply a
284 topcoat prior to any damaging conversion (such as printing). This topcoat serves the purpose
285 of protecting the AlO_x layer during conversion, thus avoiding damage/barrier deterioration
286 and enabling the use of standard industrial equipment for downstream processing of AlO_x
287 coated films. In this manner, any conversion after topcoating can be decoupled. A topcoat can
288 be applied in two ways, either inline in the vacuum chamber or offline via an atmospheric
289 pressure ‘wet’ coating process. The former can, for example, be achieved by the use of an
290 acrylate-based topcoat, flash evaporated (Affinito, Eufinger, Gross, Graff, & Martin, 1997;
291 Affinito et al., 1996; Shipman, 2016) or flexo printed in vacuum (Ferrari, 2016), or also via a
292 melamine based topcoat (Jahromi, 2011). Acrylate coatings have been widely investigated,
293 including by the authors of this paper (Struller, Kelly, Copeland, et al., 2014). Nevertheless,
294 the guarantee of fully cured acrylate layers without any residual monomer (as would be
295 required for food safety reasons) has been the main hurdle and, despite recent advances,
296 acrylate topcoated films have not yet found a place in the market for food packaging
297 materials, but are predominantly used for industrial applications. Melamine based topcoats on
298 the other hand, also suffer from low market acceptance; here, though, mainly based on the
299 negative connotation associated with melamine. Hence, in this work, the focus lies on
300 investigating offline applied wet topcoats, which do not exhibit any of the above drawbacks.

301 The initial work started here with material screening trials, in order to find a suitable coating
302 material (polymer) and assess the material properties required. These investigations were
303 conducted on a laboratory-scale as well as pilot-scale prior to moving onto industrial-scale,
304 and won’t be discussed in further detail here. An effective laboratory-scale conversion
305 simulation technique was developed in order to subject the topcoated AlO_x PET samples
306 (from laboratory, pilot and industrial coating work) to the levels of damage and stress that
307 they would generally endure during conversion processes such as gravure printing. Once this
308 proprietary technique was refined to match the effects of conversion, this method could then
309 be used to examine and test topcoated samples for the protective properties of the applied
310 topcoat without the need of having to conduct actual printing trials. When the step was taken
311 to move onto industrial-scale, the right coating application technique had to be selected (a
312 forward gravure coating system) and adjusted and the web handling system also had to be
313 adapted in order to optimise the platform for the coating of the AlO_x PET material. This is in
314 order to avoid any damage of the sensitive AlO_x layer on the offline coater due to the web

315 handling before the coating head as well as during the coating application process.
316 Nevertheless, these platform and coating technique optimisations are of confidential nature to
317 Bobst and can't be discussed in further detail.

318 Results of some of the initial investigations conducted are summarised in Table 2. As can be
319 seen, after the conversion simulation test on unprotected AlO_x PET, OTR and WVTR are
320 drastically increased with the OTR rising from a value of around 1 cm³/(m² d) to more than
321 10 cm³/(m² d) and WVTR from values of less than 1 g/(m² d) to an average value of 5.62
322 g/(m² d). These values fall within the range of results obtained during print tests (refer to
323 Table 1) of non-topcoated AlO_x PET, which confirms the suitability of the conversion
324 simulation test developed.

325 Also displayed in Table 2 are the barrier performances obtained before and after conversion
326 simulation tests for the two topcoats, which were selected for the conversion/pouch making
327 trials (see next section). As can be seen, the protective topcoat only offers a small OTR
328 improvement (from OTR values > 1 cm³/(m² d) to values consistently < 1 cm³/(m² d)), whilst
329 the barrier topcoat increases the oxygen barrier properties remarkably. Furthermore, the
330 barrier topcoat also offers some water barrier improvement, from a typical value of around
331 0.7 g/(m² d) to values around 0.4 g/(m² d). The barrier improvement obtained by applying the
332 topcoat can be attributed to two aspects; a potential pore-filling effect (Affinito & Hilliard,
333 2004) (something the authors have previously reported for acrylate topcoats (Struller, Kelly,
334 Copeland, et al., 2014)) and the effect of the permeability/barrier properties of the topcoat
335 material itself (in comparison to the PET substrate), which is now adjacent to the inorganic
336 barrier layer (Langowski, 2008). With regards to the conversion simulation test, it can be
337 seen that the barrier is unchanged or only marginally increased after conducting this test. The
338 latter is the case for the barrier topcoat (refer to oxygen barrier) and indicates that the coat
339 weight needs to be slightly increased. As will be shown later, the results obtained here are
340 very reproducible and are consistently achieved in the subsequent long duration industrial
341 trials.

342 **3.3 Conversion trials**

343 After the initial investigations presented in the previous section, as well as successful short-
344 run industrial topcoating trials, the next step was to conduct long-duration conversion trials,
345 whereby the full downstream conversion chain was investigated, from topcoating, via
346 printing, lamination and slitting to the final packaging structure (such as pouch/bag or lid)

347 including the filling of this packaging material with actual food stuffs. Being able to directly
348 print onto the topcoated AlO_x PET material and hence achieve a two-ply laminate structure
349 instead of a three-ply structure helps to fulfil the demand for cost reduction and down
350 gauging. Appropriate inks were selected based on printability and ink adhesion studies
351 carried out by the ink manufacturer. Furthermore, compatibility tests were conducted with the
352 inks selected in order to avoid any negative effects on the barrier performance due to a
353 potential incompatibility between topcoats and inks. Two topcoats were used in the
354 conversion work:

- 355 - Protective topcoat: offering protection through conversion only
- 356 - Barrier topcoat: offering protection and additional barrier functionality

357 The final laminate structure produced during the conversion trials is shown in an SEM image
358 (microtome cross section) in Figure 2. The picture clearly shows the individual layers added
359 in each conversion step: AlO_x, topcoat, inks & adhesive, apart from the LLDPE sealant web
360 which would be on top of the adhesive. For the printing process, one can also distinguish
361 between the different coloured inks, based on the larger pigments (titanium dioxide) in the
362 white ink. Finally, even the thin AlO_x layer can be made out as a lighter coloured line. This is
363 thought to be caused by electrons backscattered from the aluminium (higher atomic number
364 of aluminium compared to atoms in the surrounding PET film and polymer topcoat) in the
365 AlO_x barrier layer.

366 The oxygen and water barrier was assessed after each individual conversion process step and
367 results are summarised in Table 3 for the gravure printing and in Table 4 for flexo printing
368 (note: not in all cases, the full conversion of the produced material was assessed). These
369 results are very much in agreement with the barrier performance obtained during the topcoat
370 development stages (see Table 2). If one compares the barrier properties measured after each
371 individual conversion step, it is clear that no oxygen or water vapour barrier deterioration
372 took place through the conversion exercise. This is the case for both the protective and the
373 barrier topcoat. Some fluctuations in the barrier data are noticeable, which are, however, put
374 down to typical fluctuations within the large film reels converted and the samples being taken
375 at different positions (lengthwise). Although not all conversion steps have been conducted
376 for the flexo printing, it is clear that also in this case, no further barrier deterioration can be
377 expected, especially since flexo printing has been found to be less aggressive and damaging
378 compared to gravure printing.

379 3.4 Gelbo-Flex testing and barrier retention on elongation

380 Laminated samples of the conversion trials with gravure printing have been further
381 investigated for their flex-durability (Gelbo-Flex). During this test, the laminate is repeatedly
382 twisted and crushed, which serves the purpose of simulating the strain that the laminated
383 material may be subjected during further conversion (i.e. folding and forming into packaging
384 structures) and whilst handled in typical transport, storage and retail environments as finished
385 packages of food products. Results of this investigation are shown in Figure 3. As can be
386 seen, the laminate with the barrier topcoat reveals very good barrier performance. The OTR is
387 predominantly unaffected, even after 20 Gelbo-Flex cycles, whilst WVTR is slightly
388 increased after 20 cycles from values around 0.4 g/(m² d) for the unflexed laminate to values
389 around 0.7 g/(m² d). This flex-durability performance is comparable to or even outperforms
390 data published by some of the large and more established transparent barrier film producers
391 (DNP America, 2017; Toppan USA, 2016). In the case of the protective topcoat, the effect of
392 the Gelbo-Flex test is more pronounced. OTR increases from 0.77 cm³/(m² d) for the
393 unflexed sample to an average of 3.87 cm³/(m² d) after 20 cycles, whilst WVTR rises from
394 0.68 g/(m² d) to 1.44 g/(m² d). This performance is, however, still remarkable in regards to
395 the destructiveness of this test. Finally, it should also be noted here that, as stated previously
396 (Struller, Kelly, Copeland, & Read, 2013), the specific effects of the Gelbo-Flex test are
397 influenced by many factors, such as the type/chemistry of the inorganic barrier layer (Chiba,
398 Mikami, Sakamoto, & Tsuchiya, 2006) and its thickness, the deposition process (Komada,
399 Oboshi, & Ichimura, 2000), the substrate used and the characteristics of the lamination
400 process (Abedin & Jopko, 2013) (type of adhesive, secondary material, duplex/triplex
401 laminate etc.).

402 The laminates were additionally tested for their barrier retention on elongation behaviour,
403 whereby the samples were subjected to uniaxial deformation in the machine direction. This
404 test has been previously used in our research in order to assess how the AlO_x coated film can
405 withstand downstream processing in terms of web tension (Struller et al., 2012). In the case
406 of conducting this test with laminates, the objective is similar, as also the laminate is further
407 converted to a packaging structure (pouch) via HFFS. On these types of machinery, the
408 laminate may be subjected to stretching due to high web tensions and it is important to
409 exclude any barrier deterioration based on this process step.

410 The barrier results from this test, presented in Figure 4, indicate that oxygen barrier remains
411 unchanged for both topcoats up to 2 % elongation. After this, the OTR rises for the barrier
412 topcoat to an average value of only 0.65 cm³/(m² d) at 5 % elongation. As observed for the
413 flex-tests, this again shows the durability of the barrier topcoat laminate when subjected to
414 different forms of tensile stress and strain. In the case of the protective topcoat. However, the
415 increase in OTR for 3 % elongation onwards is drastic. When investigating the water barrier
416 retention for both topcoats, it was found that water barrier properties are maintained up to
417 1.5 % elongation. For elongation values of 2 % and onwards, the WVTR increases; although,
418 the barrier topcoat performs significantly better than the protective topcoat. Looking back at
419 the barrier performance obtained for the structure with the barrier topcoat post pouch-making
420 (see Table 3), one can see that neither OTR nor WVTR is deteriorated after this conversion
421 step, hence indicating that no stretching beyond 1.5 % elongation occurs due to the web
422 tensions on the pouch making equipment.

423 **4 Summary and Conclusions**

424 It has been shown with the work presented here that AlO_x coated PET films can be converted
425 without barrier deterioration using standard equipment once a protective offline topcoat has
426 been applied. Extensive research has been conducted in assessing the various conversion
427 processes for their impact on the conversion of AlO_x coated PET film as well as developing
428 and characterising optimised topcoats. Two different topcoat variations have been established
429 and investigated, with one topcoat giving protection through conversion only, and the other
430 topcoat offering additional and significant barrier enhancement. Long duration conversion
431 trials, including printing (flexo and gravure), lamination, slitting and pouch making/filling
432 were successfully carried out with both topcoats. Furthermore, tests on flex-durability and
433 barrier retention on elongation also showed that the barrier topcoat laminate especially has
434 remarkably good properties when subjected to this kind of repetitive or uniaxial strain.

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444

445

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541 **Table and figure headings**

542 Table 1 – Barrier performance – Assessment of printing and lamination processes for
543 ‘unprotected’ AlO_x PET

544 Table 2 – Barrier performance – Effect of conversion simulation test

545 Table 3 – Barrier performance through conversion – Gravure printing

546 Table 4 – Barrier performance through conversion – Flexo printing

547

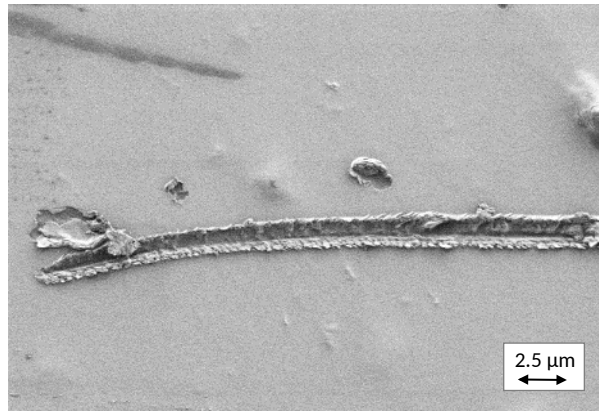
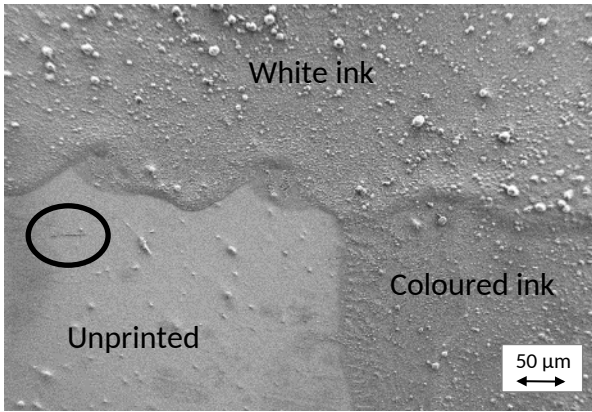
548 Figure 1: SEM images showing damage to AlO_x layer after gravure printing (note: machine
549 direction is horizontal)

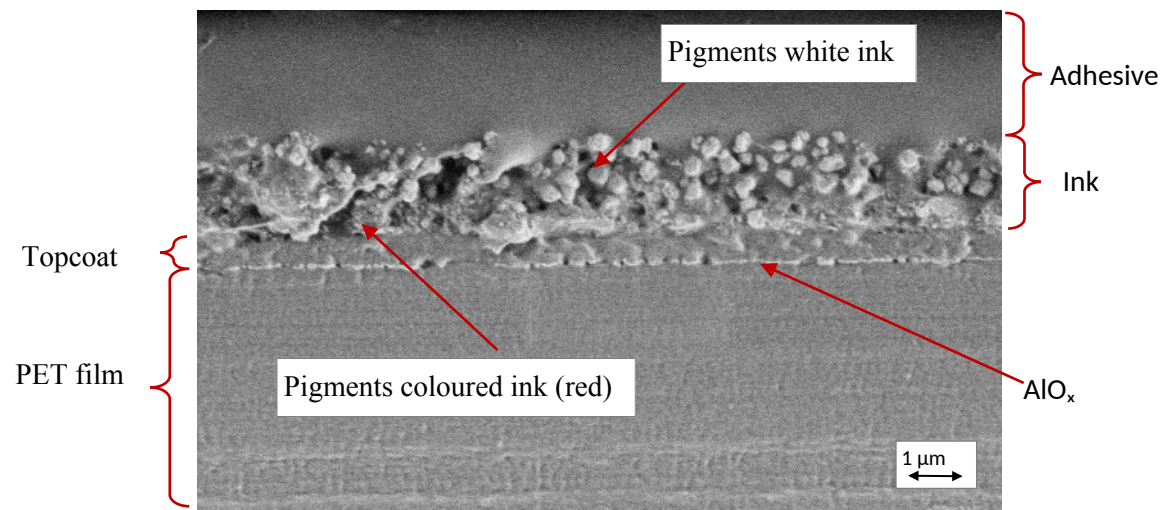
550 Figure 2: SEM cross-sectional image of final laminate structure

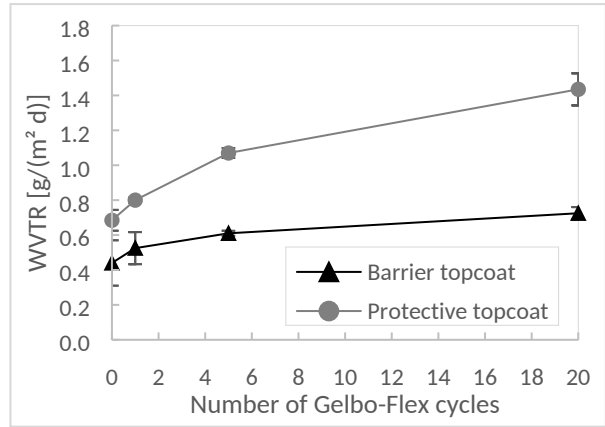
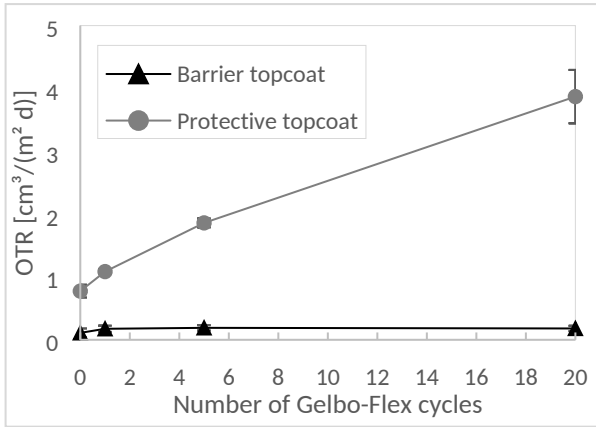
551 Figure 3: Barrier performance of printed and laminated AlO_x PET material after Gelbo-Flex
552 testing

553 Figure 4: Barrier retention on elongation behaviour for printed and laminated AlO_x PET
554 material

555







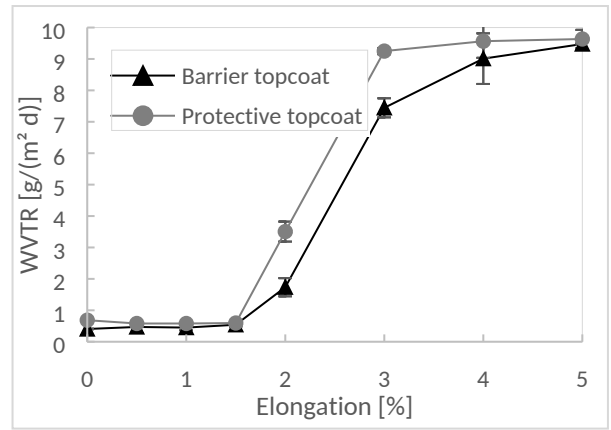
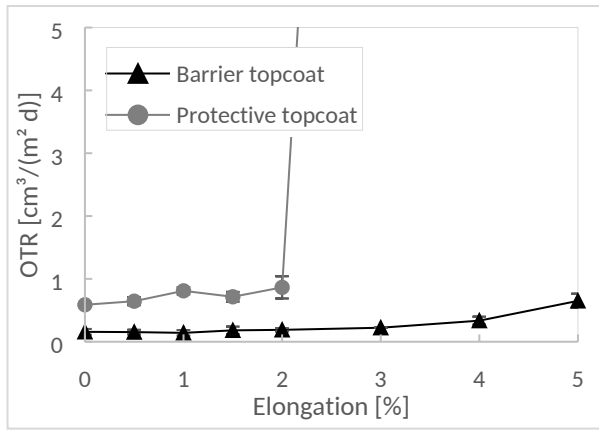


Table 1 – Barrier performance – Assessment of printing and lamination processes for
'unprotected' AlO_x PET

Structure	Description	OTR	WVTR
		cm ³ /(m ² d)	g/(m ² d)
PET 12 μm		≈ 120	40 – 50
PET/AlO_x	Before printing	1.0 – 1.5	0.5 – 1.0
PET/AlO_x/ink	Bobst Italia, gravure press RS 4003MP 250 m/min	2.5 – 18	1 – 12
PET/AlO_x	Before printing	1.0 – 1.5	0.5 – 1.0
PET/AlO_x/ink	Chinese customer, gravure press 200 m/min	15 – 20	8 – 10
PET/AlO_x	Before printing	1.0 – 1.5	0.5 – 1.0
PET/AlO_x/ink	Bobst Bielefeld, CI flexo press 20SIX 250 and 400 m/min	1.5 – 8	1 – 3.5
PET/AlO_x	Before lamination	1.29 ± 0.21	0.82 ± 0.11
PET/AlO_x/ad*/CPP	Bobst Italia, laminator CL 850D 150 m/min	1.34 ± 0.23	0.85 ± 0.10

*ad = adhesive layer

Table 2 – Barrier performance – Effect of conversion simulation test

Structure	Description	OTR	WVTR
		cm³/(m² d)	g/(m² d)
PET/AlO_x	-	1.07 ± 0.10	0.66 ± 0.05
PET/AlO_x	After conversion simulation test	13.47 ± 2.32	5.62 ± 2.12
PET/AlO_x/protective topcoat	-	0.73 ± 0.12	0.58 ± 0.02
PET/AlO_x/protective topcoat	After conversion simulation test	0.66 ± 0.11	0.74 ± 0.03
PET/AlO_x/barrier topcoat	-	0.15 ± 0.06	0.36 ± 0.01
PET/AlO_x/barrier topcoat	After conversion simulation test	0.27 ± 0.08	0.44 ± 0.04

Table 3 – Barrier performance through conversion – Gravure printing

Conversion process	Structure	OTR	WVTR
		cm ³ /(m ² d)	g/(m ² d)
-	PET 12 μm	≈ 120	40 – 50
AlO_x coating	PET/AlO _x	1.0 – 1.5	< 1.0
Protective topcoat			
Topcoating	PET/AlO _x /topcoat	0.65 ± 0.09	0.56 ± 0.10
Printing	PET/AlO _x /topcoat/ink	0.78 ± 0.10	0.70 ± 0.05
Lamination	PET/AlO _x /topcoat/ink/ad/P	0.77 ± 0.10	0.69 ± 0.06
Slitting	PET/AlO _x /topcoat/ink/ad/P	0.59 ± 0.02	0.69 ± 0.01
Barrier topcoat			
Topcoating	PET/AlO _x /topcoat	0.09 ± 0.04	0.42 ± 0.03
Printing	PET/AlO _x /topcoat/ink	0.12 ± 0.08	0.45 ± 0.06
Lamination	PET/AlO _x /topcoat/ink/ad/P	0.11 ± 0.07	0.44 ± 0.13
Slitting	PET/AlO _x /topcoat/ink/ad/P	0.11 ± 0.09	0.41 ± 0.01
Pouch making	PET/AlO _x /topcoat/ink/ad/P	0.11 ± 0.09	0.44 ± 0.01

Table 4 – Barrier performance through conversion – Flexo printing

Conversion process	Structure	OTR	WVTR
		cm ³ /(m ² d)	g/(m ² d)
-	PET 12 μm	≈ 120	40 – 50
AlO_x coating	PET/AlO _x	1.0 – 1.5	< 1.0
Protective topcoat			
Topcoating	PET/AlO _x /topcoat	0.67 ± 0.01	0.64 ± 0.01
Printing	PET/AlO _x /topcoat/ink	0.72 ± 0.06	0.67 ± 0.17
Barrier topcoat			
AlO_x coating	PET/AlO _x	1.0 – 1.5	< 1.0
Topcoating	PET/AlO _x /topcoat	0.12 ± 0.03	0.39 ± 0.05
Printing	PET/AlO _x /topcoat/ink	0.12 ± 0.03	0.39 ± 0.03