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

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ABSTRACT

Transparent barrier films based on vacuum deposited aluminum oxide (AlO\textsubscript{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\textsubscript{x} producers and the wider converting industry. In this work, AlO\textsubscript{x} coated PET films have been converted in long duration industrial-scale trials via topcoating, printing, lamination and finally pouch making and filling. Throughout this process, each conversion step has been investigated for its effects on the barrier performance, visual appearance and adhesion of the AlO\textsubscript{x} coated film. Additionally, Gelbo-flex testing, barrier retention on elongation evaluation as well as SEM cross-sectioning were conducted.

1 INTRODUCTION

Inorganic transparent barrier layers such as AlO\textsubscript{x} or silicon oxide (SiO\textsubscript{x}) are still in demand for clear barrier packaging materials, with applications ranging from food stuffs, which have rather moderate barrier requirements, to electronic products such as displays, where ultra-high barrier levels are essential. A number of different techniques are available to deposit such inorganic layers including atomic layer deposition (ALD), plasma enhanced chemical vapor deposition (PECVD), sputtering and thermal evaporation processes (electron beam and boat evaporation) \[1-3\]. Whilst sputtering and ALD are mainly of importance for high-end applications, PECVD and thermal evaporation techniques are the processes of interest with regards to the cost-sensitive food packaging market. Reactive evaporation using resistively heated boats represents an especially promising candidate with great market potential due to the low associated capital investment, the use of inexpensive raw materials and the high process speeds that can be achieved, without disadvantageous effects to the barrier performance in comparison to the other deposition techniques.

The market for transparent oxide coated films has an estimated compound annual growth rate of 7.7 \% (year 2015), compared to only 0.8 \% for aluminum foil and even -0.3 \% for PVdC coated films \[4\], a polymer based transparent barrier film that can potentially be replaced by oxide coated film. Nevertheless, the market volume for these oxide based barrier films is currently rather small, around 5 \% the size of the aluminum foil market and approximately 10 \% the size of the market for PVdC coated films. To date, the clear barrier flexible packaging market for AlO\textsubscript{x} and SiO\textsubscript{x} transparent barrier films is still dominated by Japanese material producers, such as Toppan, Toray, DNP, Mitsubishi and others with well-known film grades such as GL Film, Barrialox, IB-Film or TECHBARRIER \[5-8\], although Camvac’s Camclear \[9\] and Amcor’s Ceramis \[10\] have also become

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well-established products. The Japanese producers predominantly apply electron beam evaporation or PECVD techniques for the deposition of the barrier layers [11], which entail a lot higher production and investment costs and more technical complexity including potential reliability issues compared to reactive boat evaporation. Furthermore, their products are generally topcoated with a material that not only protects the inorganic AlOₓ or SiOₓ barrier layer through handling and conversion, but also significantly enhances its barrier performance. This, in addition with the retortability of many of these materials, makes them very high performance products, which comes at a certain cost level and prevents real volume growth for cost-sensitive volume market applications. The reactive boat evaporation process has been developed over the last few decades and is now well-established [1, 2, 12-14]. Its vast economic potential of low cost AlOₓ production leads to new players continuously pushing to enter the attractive transparent barrier market and trying to obtain market share from the more advanced Japanese players. However, unlike the Japanese products, most of these ‘new’ products do not have a protective topcoat and their conversion is challenging with inevitable barrier deterioration.

In order to obtain a fully viable commercial AlOₓ product, the successful conversion of the coated film also has to be taken into account. Thereby, it is essential that barrier loss upon conversion (coating, slitting, printing & lamination) is avoided or at least minimized to achieve the barrier requirements of the target application. In our case, it has been found that the printing step, especially rotogravure printing, is the most damaging conversion process with regards to the AlOₓ barrier performance, which has also been identified by other researchers [15]. This paper therefore focusses on developing a suitable offline topcoat solution, which is not only capable of protecting the AlOₓ barrier layer through all conversion steps (including pouch making and filling), but also offers additional barrier functionality. Once this topcoat is successfully applied using an optimized/modified platform to prevent barrier loss upon web conveying and coating application, the topcoated AlOₓ PET films can be converted on standard industrial equipment, without the need for changes and without any subsequent barrier deterioration. Trials have been conducted using AlOₓ coated PET film and each process step was characterized with regards to its post-processing barrier performance. Additionally, flex-durability (Gelbo-flex) testing as well as barrier retention upon elongation investigations were carried out.

2 EXPERIMENTAL

2.1 COATING/CONVERSION PROCESSES AND PLATFORMS

A standard low-cost commodity grade PET film (12 μm film thickness; 1250 mm width; corona treated by the film manufacturer) was chosen for this investigation and AlOₓ coating was conducted via reactive thermal evaporation using a Bobst Manchester Ltd (Heywood, United Kingdom) K5000 vacuum metallizers with an optional AlOₓ coating system (boat-type). The AlOₓ deposition onto the 32 km reels was carried out at 600 m/min, whilst also applying in-line plasma pre-treatment using a medium frequency plasma source with magnetically enhanced water cooled electrodes. To achieve enhanced convertibility, the original AlOₓ process was further optimized in order to facilitate the handling of the AlOₓ coated film via increasing its flexibility. Thereby, the thickness of the coating layer was reduced from an average value of 10 nm [16, 17] to an estimated value of around 8 nm, at an optimized oxidation rate.
Topcoating of the produced AlOₓ PET reels was performed on an AlOₓ optimized Bobst CL 850D coater/laminator at Bobst Italia SpA (San Giorgio Monferrato, Italy) using a gravure coating system at speeds up to 250 m/min. The two coatings chosen are water-based and were supplied by the project partner Michelman SARL (Windhof, Luxembourg). These coatings are: a protective topcoat (offering protection through conversion only) and a barrier topcoat (offering protection through conversion as well as additional barrier improvement). Both coatings are compliant with relevant food contact legislations.

The rotogravure printing was also conducted at Bobst Italia using a standard Bobst RS 4003MP 8 color rotogravure printing press at a speed of 200 m/min. For this work, a high performance commercially available ink system was used, which was supplied by the project partner Flint Group Italia SpA (Cinisello Balsamo, Italy).

Flexo printing was carried out on a standard Bobst Bielefeld GmbH (Bielefeld, Germany). Printing was conducted at 200 m/min using the flexo version of the high performance ink system used in the gravure printing trials, supplied by Flint Group Germany GmbH (Willstätt, Germany).

Lamination of the topcoated and printed material was again carried out at Bobst Italia SpA using solvent-based adhesive lamination on a Bobst CL 850D coater/laminator. A commercially available high performance, two component polyurethane adhesive (supplied by the project partner DOW/Rohm and Haas Italia Srl (Mozzate, Italy)) was used and applied via a flexo trolley coating application system at a speed of 150 m/min. A 32 μm corona treated PE sealant film supplied by the project collaborator Printpack Inc. (Atlanta, US) was used as secondary material.

All further conversion steps (slitting, pouch making via HFFS (horizontal form fill & seal) and pouch filling) were arranged and managed by Printpack using their facilities, contacts and customer base.

### 2.2 Analytical Methods

Barrier properties in terms of oxygen and water vapor transmission rates (OTR/WVTR) were analyzed in accordance with ASTM F 1927 and ASTM F 1249/ISO 15106-3 using a Mocon Oxtran 2/20 and Systech Illinois 8001 for oxygen permeation and a Mocon Permatran-W 3/33 and Systech Illinois 7001 for water vapor permeation. Test conditions are 23 °C and 50 % relative humidity (RH) for OTR measurements and 37.8 °C and a gradient of 90 % RH for WVTR measurements.

A Zeiss Supra 40VP field emission gun scanning electron microscope (FEG SEM) was used to acquire cross-sectional images of the laminated film samples. Cross-sections were cut on a Leica RM2125 microtome and, subsequently, samples were examined on the SEM using a low acceleration voltage to avoid the need for coating with a conductive layer.

The flex durability or Gelbo-flex (ASTM F392 [18]) of the laminated AlOₓ coated films was investigated using a Gelbo-Flex tester model 5000 manufactured by United States Testing Co., Inc. For this investigation 1, 5 and 20 flexing cycles were performed and the barrier performance was determined successively.

For the evaluation of the barrier retention on elongation behavior, the films were stretched to a pre-defined strain (between 0.5 and 5 %, stretching in the machine direction) by a tensile testing
unit (Hounsfield H10KS with QMat 5.52 software) and subsequently the barrier properties were measured (test for stretch durability/resistance as suggested by Felts [19], however samples are analyzed in the relaxed state).

The laminate bond strength was investigated by Printpack. Two independent sample sets were tested for each of the topcoat laminates.

3 RESULTS AND DISCUSSION

3.1 TOPCOAT DEVELOPMENT

Before conducting industrial long-duration topcoating and conversion trials, material screening trials were performed in order to find a suitable coating material (polymer) and assess the material properties required. These investigations were conducted on a laboratory-scale as well as pilot-scale prior to moving onto industrial-scale. Thereby, the coating application technique, adhesion, coating laydown, coating optics and coating thickness had to be assessed and optimized. An effective laboratory-scale conversion simulation technique was developed in order to subject the topcoated AlOₓ PET samples (from laboratory, pilot and industrial coating work) to the levels of damage and stress that they would generally endure during conversion processes such as gravure printing. Once refined to match the effects of conversion, this method could then be used to examine and test topcoated samples for the protective properties of the applied topcoat without the need of having to conduct actual printing trials. Thereby, it was possible to determine the optimum/minimum dry coat weight required in order to give protection during conversion (i.e. maintain barrier properties) whilst still providing a cost efficient topcoat solution. When the step was taken to industrial-scale, the right coating application technique had to be selected and adjusted and the web handling system also had to be adapted in order to optimize the platform for the coating of AlOₓ PET material.

Results of some of the initial investigations conducted are summarized in Table 1. As can be seen, after the conversion simulation test on unprotected AlOₓ PET, OTR and WVTR are drastically increased with the OTR rising from a value of around 1 cm³/(m² d) to more than 10 cm³/(m² d) and WVTR from values of less than 1 g/(m² d) to an average value of 5.62 g/(m² d). In fact, when these values are compared with the results obtained during print tests on a standard non-modified Bobst RS 4003MP gravure press (non-topcoated AlOₓ PET), one can see that the simulation test is even more destructive. Nevertheless, this test is assumed to simulate the worst case scenario. Jahromi [20], for example, has reported comparable values for printed unprotected AlOₓ PET (OTR around 11 cm³/(m² d) and WVTR around 6 g/(m² d)), although these samples were flexo printed (which in our case has been found to be significantly less destructive than gravure printing).
Table 1 – Barrier performance – Effect of conversion simulation test

<table>
<thead>
<tr>
<th>Structure</th>
<th>Description</th>
<th>OTR  cm³/(m² d)</th>
<th>WVTR g/(m² d)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PET/AlOₓ</td>
<td></td>
<td>1.07 ± 0.10</td>
<td>0.66 ± 0.05</td>
</tr>
<tr>
<td>PET/AlOₓ After conversion test</td>
<td></td>
<td>13.47 ± 2.32</td>
<td>5.62 ± 2.12</td>
</tr>
<tr>
<td>PET/AlOₓ/ink</td>
<td>After rotogravure printing</td>
<td>3 – 6</td>
<td>1.5 – 3</td>
</tr>
<tr>
<td>PET/AlOₓ/protective topcoat</td>
<td></td>
<td>0.73 ± 0.12</td>
<td>0.58 ± 0.02</td>
</tr>
<tr>
<td>PET/AlOₓ/protective topcoat</td>
<td>After conversion simulation test</td>
<td>0.66 ± 0.11</td>
<td>0.74 ± 0.03</td>
</tr>
<tr>
<td>PET/AlOₓ/barrier topcoat</td>
<td></td>
<td>0.15 ± 0.06</td>
<td>0.36 ± 0.01</td>
</tr>
<tr>
<td>PET/AlOₓ/barrier topcoat</td>
<td>After conversion simulation test</td>
<td>0.27 ± 0.08</td>
<td>0.44 ± 0.04</td>
</tr>
</tbody>
</table>

Also displayed in Table 1 are the barrier performances obtained before and after conversion simulation tests for the two topcoats, which were selected for the conversion/pouch making trials (see next section). As can be seen, the protective topcoat only offers a small OTR improvement (from OTR values > 1 cm³/(m² d) to values consistently < 1 cm³/(m² d)), whilst the barrier topcoat increases the oxygen barrier properties remarkably. Furthermore, the barrier topcoat also offers some water barrier improvement, from a typical value of around 0.7 g/(m² d) to values around 0.4 g/(m² d). The barrier improvement obtained by applying the topcoat can be attributed to two aspects; a potential pore-filling effect as suggested by Affinito and Hilliard [21] (something we have previously reported for acrylate topcoats [22]) and the effect of the permeability/barrier properties of the topcoat material (in comparison to the PET substrate), which is now adjacent to the inorganic barrier layer [23]. With regards to the conversion simulation test, it can be seen that the barrier is unchanged or only marginally increased after conducting this test. The latter is the case for the barrier topcoat (refer to oxygen barrier) and indicates that the coat weight needs to be slightly increased. As will be shown later, the results obtained here are very reproducible and are consistently achieved in the subsequently conducted long duration industrial trials.

3.2 Conversion trials

After the initial coating material screening trials (laboratory and pilot) as well as successful short-run industrial topcoating trials, the next step was to conduct long-duration conversion trials, whereby the full downstream conversion chain was investigated, from topcoating, via printing, lamination and slitting to the final packaging structure (such as pouch/bag or lid) including the filling of this packaging material with actual food stuffs. Being able to directly print onto the topcoated AlOₓ PET material and hence achieve a two-ply laminate structure instead of a three-ply structure helps to fulfill the demand for cost reduction and down gauging. In order to promote the oxygen barrier performance of AlOₓ, nuts, which require a certain level of oxygen barrier properties for their packaging material, were found to be a suitable product. Appropriate inks were selected based on printability and ink adhesion studies carried out by the ink manufacturer. Furthermore, compatibility tests were conducted with the inks selected in order to avoid any negative effects on the barrier performance due to a potential incompatibility between topcoats and inks. A schematic of the final material structure produced during the conversion trials is shown in Figure 1, whilst an actual SEM image of a cross section is displayed Figure 2. The latter picture clearly shows the individual layers added in each conversion step. For the printing process, one can also distinguish between the
different colored inks, based on the larger pigments in the white ink. Finally, even the thin AlOₓ layer can be made out as lighter colored line. This is thought to be caused by electrons backscattered from the aluminum (higher atomic number of aluminum compared to atoms in the surrounding PET film and polymer topcoat) in the AlOₓ barrier layer.

![Image of laminate structure](image1.png)

*Figure 1 – Two-ply laminate structure of final pouch material*

![Image of SEM cross-sectional image](image2.png)

*Figure 2 – SEM cross-sectional image of final laminate structure*

As mentioned previously, the oxygen and water barrier was assessed after each individual conversion process step and results are summarized in *Table 2* for the protective topcoat and in *Table 3* for the barrier topcoat (note: the values stated are average values obtained from measurements conducted on samples taken across the film width). These results are very much in
agreement with the barrier performance obtained during the topcoat development stages (see Table 1).

**Table 2 – Barrier performance through conversion – Gravure printing (protective topcoat)**

<table>
<thead>
<tr>
<th>Conversion process</th>
<th>Structure</th>
<th>OTR cm³/(m² d)</th>
<th>WVTR g/(m² d)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AlOₓ coating</td>
<td>PET/AlOₓ</td>
<td>1.0 – 1.5</td>
<td>&lt; 1.0</td>
</tr>
<tr>
<td>Topcoating</td>
<td>PET/AlOₓ/topcoat</td>
<td>0.65 ± 0.09</td>
<td>0.56 ± 0.10</td>
</tr>
<tr>
<td>Printing</td>
<td>PET/AlOₓ/topcoat/ink</td>
<td>0.78 ± 0.10</td>
<td>0.70 ± 0.05</td>
</tr>
<tr>
<td>Lamination</td>
<td>PET/AlOₓ/topcoat/ink/ad/PE</td>
<td>0.77 ± 0.10</td>
<td>0.69 ± 0.06</td>
</tr>
<tr>
<td>Slitting</td>
<td>PET/AlOₓ/topcoat/ink/ad/PE</td>
<td>0.59 ± 0.02</td>
<td>0.69 ± 0.01</td>
</tr>
</tbody>
</table>

**Table 3 – Barrier performance through conversion – Gravure printing (barrier topcoat)**

<table>
<thead>
<tr>
<th>Conversion process</th>
<th>Structure</th>
<th>OTR cm³/(m² d)</th>
<th>WVTR g/(m² d)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AlOₓ coating</td>
<td>PET/AlOₓ</td>
<td>1.0 – 1.5</td>
<td>&lt; 1.0</td>
</tr>
<tr>
<td>Topcoating</td>
<td>PET/AlOₓ/topcoat</td>
<td>0.09 ± 0.04</td>
<td>0.42 ± 0.03</td>
</tr>
<tr>
<td>Printing</td>
<td>PET/AlOₓ/topcoat/ink</td>
<td>0.12 ± 0.08</td>
<td>0.45 ± 0.06</td>
</tr>
<tr>
<td>Lamination</td>
<td>PET/AlOₓ/topcoat/ink/ad/PE</td>
<td>0.11 ± 0.07</td>
<td>0.44 ± 0.13</td>
</tr>
<tr>
<td>Slitting</td>
<td>PET/AlOₓ/topcoat/ink/ad/PE</td>
<td>0.11 ± 0.09</td>
<td>0.41 ± 0.01</td>
</tr>
</tbody>
</table>

Furthermore, if one compares the barrier properties measured after each individual conversion step, it is clear that no oxygen or water vapor barrier deterioration took place through the conversion exercise. This is the case for both, the protective and the barrier topcoat. Some fluctuations in the barrier data are noticeable, which are, however, put down to typical fluctuations within the large film reels converted and the samples being taken at different positions (lengthwise). In the manner of a round robin test, barrier evaluations were also conducted by two other collaborators whose measurements were in-line with the data presented here and hence confirmed and validated the work conducted.

For the flexo printing, results are presented in Table 4 and Table 5. Also here, the topcoated barrier performance is of the same order as seen before and no barrier deterioration after printing is visible. This was to be expected, since the flexo printing is by far a less aggressive and damaging printing technique compared to gravure printing. Unfortunately, further conversion with the flexo printed material has not yet been conducted.

**Table 4 – Barrier performance through conversion – Flexo printing (protective topcoat)**

<table>
<thead>
<tr>
<th>Conversion process</th>
<th>Structure</th>
<th>OTR cm³/(m² d)</th>
<th>WVTR g/(m² d)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AlOₓ coating</td>
<td>PET/AlOₓ</td>
<td>1.0 – 1.5</td>
<td>&lt; 1.0</td>
</tr>
<tr>
<td>Topcoating</td>
<td>PET/AlOₓ/topcoat</td>
<td>0.67 ± 0.01</td>
<td>0.64 ± 0.01</td>
</tr>
<tr>
<td>Printing</td>
<td>PET/AlOₓ/topcoat/ink</td>
<td>0.72 ± 0.06</td>
<td>0.67 ± 0.17</td>
</tr>
</tbody>
</table>
3.3 **Gelbo-Flex Testing and Barrier Retention on Elongation**

Laminated samples of the conversion trials with gravure printing have been further investigated for their flex-durability (Gelbo-Flex). During this test, the laminate is repeatedly twisted and crushed, which serves the purpose of simulating the strain that the laminated material may be subjected during further conversion (i.e. folding and forming into packaging structures) and whilst handled in the typical transport, storage and retail environments as finished packages of food products. Results of this investigation are shown in Figure 3.

![Figure 3 – Barrier performance of printed and laminated AlOₓ PET material after Gelbo-Flex testing](image)

As can be seen from Figure 3, the laminate with the barrier topcoat reveals very good barrier performance. The OTR is unaffected, even after 20 Gelbo-Flex cycles, whilst WVTR is slightly increased after 20 cycles from values around 0.4 g/(m² d) for the unflexed laminate to values around 0.7 g/(m² d). This flex-durability performance is comparable to or even outperforms data published by some of the large and more established transparent barrier film producers [5, 7, 8]. In the case of the protective topcoat, the effect of the Gelbo-Flex test is more pronounced. OTR increases from 0.77 cm³/(m² d) for the unflexed sample to an average of 3.87 cm³/(m² d) after 20 cycles, whilst WVTR rises from 0.68 g/(m² d) to 1.44 g/(m² d). This performance is, however, still remarkable in regards to the destructiveness of this test. Finally, it should also be noted here that, as stated previously [24], the specific effects of the Gelbo-Flex test are influenced by many factors, such as the type/chemistry of the inorganic barrier layer [25] and its thickness, the deposition process [26], the substrate used and the characteristics of the lamination process [27] (type of adhesive, secondary material, duplex/triplex laminate etc.).
The laminates were additionally tested for their barrier retention on elongation behavior, whereby the samples are subjected to uniaxial deformation in the machine direction. This test has been previously used in our research in order to assess how the AlOₓ coated film can withstand downstream processing in terms of web tension [14, 24]. In the case of conducting this test with laminates, the objective is similar, as also the laminate is also further converted to a packaging structure (pouch) via HFFS. On these types of machinery, the laminate may be subjected to stretching due to high web tensions and it is important to exclude any barrier deterioration based on this process step.

![Figure 4 –Barrier retention on elongation behavior for printed and laminated AlOₓ PET material](image)

The barrier results from this test, presented in Figure 4, indicate that oxygen barrier remains unchanged for both topcoats up to 2 % elongation. After this, the OTR rises for the barrier topcoat to an average value of only 0.65 cm³/(m² d) at 5 % elongation. As observed for the flex-tests, this again shows the durability of the barrier topcoat laminate when subjected to different forms of stress and tensile strain. In the case of the protective topcoat, however, the increase in OTR for 3 % elongation onwards is drastic. When investigating the water barrier retention for both topcoats, it was found that water barrier properties are maintained up to 1.5 % elongation. For elongation values of 2 % and onwards, the WVTR increases; although, the barrier topcoat performs significantly better than the protective topcoat.

### 3.4 LAMINATE BOND STRENGTH

Results of the laminate bond strength analysis are summarized in Table 6. The bond strengths obtained for both topcoat laminates are good and fulfill requirements for most general food packaging applications. In the case of the protective topcoat, the bond strength measured even exceeds necessary levels by far.
Table 6 – Laminate bond strength (analysis conducted by Printpack)

<table>
<thead>
<tr>
<th>Sample description</th>
<th>Laminate bond strength</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>g/inch</td>
</tr>
<tr>
<td>Protective topcoat laminate</td>
<td>741 ± 84</td>
</tr>
<tr>
<td></td>
<td>589 ± 169</td>
</tr>
<tr>
<td>Barrier topcoat laminate</td>
<td>261 ± 13</td>
</tr>
<tr>
<td></td>
<td>245 ± 8</td>
</tr>
</tbody>
</table>

4 SUMMARY AND CONCLUSIONS

It has been shown with the work presented here that AlOₓ coated PET films can be converted without barrier deterioration using standard equipment once a protective offline topcoat has been applied. Extensive research has been conducted in developing and characterizing optimized topcoats as well as optimizing their properties and application method. Thereby a step-by-step approach was taken, going from laboratory and pilot coating to industrial-scale coating trials and lastly running longer campaigns. Two different topcoat variations have finally been established and investigated, with one topcoat giving protection through conversion only, and the other topcoat offering additional and significant barrier enhancement. Long duration conversion trials, including printing (flexo and gravure), lamination, slitting and pouch making/filling were successfully carried out with both topcoats. Furthermore, tests on flex-durability and barrier retention on elongation also showed that the barrier topcoat laminate has remarkable good properties when subjected to this kind of repetitive or uniaxial strain.

5 FUTURE WORK & DEVELOPMENTS

Using the Bobst integrated approach and synergies within the product lines vacuum, flexo, gravure and coating, further work is currently conducted in order to optimize the associated platforms to minimize conversion damage without the aid of an offline protective topcoat. This will reduce process steps and, hence, cost implications in order to target more cost sensitive volume applications, which previously were unable to be serviced with the existing products on the market.

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6 REFERENCES


