Advances in protection of Aluminum Oxide using inline vacuum deposited organic top coats

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Reactive evaporation of aluminum using an industrial ‘boat-type’ roll-to-roll metallizer offer a cost effective way to producing transparent barrier layers. Aluminum oxide (AlOx) layers produced in this way are brittle resulting in barrier deterioration during downstream processing. Figure 1 shows barrier values of a standard PET-AlOx measured after direct printing on AlOx layer using flexography followed by lamination.

Figure 1. Effect of direct printing on PET-AlOx followed by lamination with PE. The designation are as follows:

Input Reel: PET-AlOx single web before entering the printing machine

Clear Printed: Unprinted PET-AlOx single web after existing the printing machine
As can be seen in Figure 1, both oxygen and water barrier are deteriorated after direct printing on AlOx layer. The deterioration on the printed single web can be seen in printed areas, and also in clear sections that have not been in contact with inks. After lamination with PE water barrier somewhat recovers but oxygen transmission rate (OTR) remains high. The recovery of water vapor transmission rate (WVTR) can be attributed to water barrier properties of PE. This sealant film however, does not provide any oxygen barrier and therefore OTR values remain high after lamination.

Deterioration of AlOx barrier can be due to both mechanical (stretching and scratching) and chemical effects, i.e. interaction with inks components. Figure 1 shows damage to OTR and WVTR in clear area that have not been in contact with any inks. Deterioration in these areas can be solely attributed to mechanical effects. It should be noted that AlOx layer deposited on PET is only 10 nm. In addition to printing and lamination, our results show that this thin AlOx layer can be damaged by other downstream processing steps such as slitting and retort.

In order to maintain barrier values of AlOx during downstream processing, it is essential that AlOx is protected by a top coat layer. In principal there are two different ways of applying the top coat, i.e. offline or inline. The offline coating is carried out outside the vacuum chamber in a separate step. Inline top coating is conducted in 1x pumping, i.e. directly after AlOx coating in the same vacuum chamber. Needless to say that inline top coating is preferred option from operational and thus cost point of view.

Ideally the top coat should comply following stringent conditions:

1. Boost the initial OTR
2. Protect barrier during downstream processing steps.
3. Compatible with commercially available inks & adhesives
4. Cost effective, i.e. Inline process using inexpensive material
5. Compliant with FDA and EU regulations

DSM has developed an inline coating process, branded as Freshure®, which complies with all above requirements. Using this process organic compounds are vacuum deposited in 1x step, i.e. in 1x pumping, on top of AlOx layer. Without additional steps, such a UV-curing, these compounds form crystalline layers with intrinsic oxygen barrier. Inline application of such coatings boost the initial oxygen barrier of AlOx and more importantly protects the barrier during downstream processing steps. Furthermore the use of such vacuum deposited organic layers is compliant with both EU and FDA regulations for food packaging applications.

Figure 2 shows barrier values of PET-AlOx top coated with inline organic top coat after different stages of downstream processing.
Figure 2. Effect of direct printing on WVTR (a) and OTR (b) of PET-AlOx coated with inline organic top coat followed by lamination with PE. For reference barrier values of unprotected AlOx (without top coat) are also shown. Designations are the same in Figure 1.
Figure 2 shows clearly that with inline organic top coat both OTR and WVTR are protected in clear and printed sections with finished laminate retaining barrier values below 2.

The results in Figure 2 are obtained using standard corona treated PET. Barrier values can be further improved by using PET film with smoother surface topology. OTR results for special grade PET coated with AlOx+inline organic top coat are shown in Figure 3.

**Figure 3.** Effect of direct printing on PET-AlOx coated with inline organic top coat followed by lamination with PE. For these trials higher quality PET films are used with smoother surface topology as compared with standard corona treated PET films the result of which are shown in Figure 1 and 2. Designations are the same in Figure 1.

Results in Figure 3 clearly demonstrate that by optimizing surface topology of PET film barrier values as low as 0.5 can be achieved. Further improvement of barrier can be envisaged but additional substrate and process optimization.

Next to good barrier after printing and lamination, it is important that the laminates have sufficiently high bond strengths. By using the right selections of inks and adhesives we have been able to achieve bond strengths values above 3N/25mm. It should be added that all inks and adhesives used in our trials are of commercially available grades.

In summary results shown here demonstrate that without protection barrier values of AlOx deteriorate dramatically specially after direct printing. Inline top coat process developed by DSM boost the initial barrier of AlOx and protects it during various downstream processing steps. By optimizing PET film quality barrier values as low as 0.5 can be achieved for finished printed laminates demonstrating bond strength values above 3N/25mm. Such performance opens various applications such as replacement of Aluminum foil in flexible packaging.