

## **Production Proven Vacuum Web Coating System with High Process Flexibility for Robust and Environmentally-Friendly Transparent Barriers**

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### **EXTENDED ABSTRACT**

The end market for transparent flexible barrier films is larger than for metallized films and currently is dominated by polymeric barrier layers. The thickness of such layers is in the range of several micrometers to several 10's of micrometers. Some of these coatings are under scrutiny and discussion since they contain chemicals like chlorine, which are not considered to be environmentally friendly.

An alternative for thick polymeric coatings can be a vacuum coating process with a coating thickness several orders of magnitude thinner and in the range of only ~ 10 nanometers achieving the same or better barrier properties compared with polymeric coatings. Another advantage of vacuum based processes, the source materials for the barrier layers are usually natural occurring oxides like SiO<sub>x</sub> or AlO<sub>x</sub>. By coating on substrates like PLA, fully biodegradable barrier films can be produced.

Environmental aspects, barrier and optical properties are all important, but equally important are cost aspects, process flexibility regarding different base films and coating robustness to survive the down stream converting processes.

The most universally used vacuum web coating processes are sputtering and evaporation.

- Based on high energy input from energetic particles during growing of the coating, sputtering provides the most dense layers and is used for many technical applications with very critical quality requirements. However compared with evaporation, sputtering technologies run at relatively low coating speeds resulting in higher coating costs.
- Evaporation provides coating speeds ranging from one to two orders of magnitude faster compared to sputtering while producing less dense layers.
- A combination of both high coating speed and production of dense layers can be achieved with plasma assisted evaporation.

Boat type evaporation is well known for metallization. Boat type evaporation can be modified to use reactive evaporation of Al to also produce transparent AlO<sub>x</sub> barrier layers, useable for different applications.

For other requirements such as base film flexibility and down stream robustness, boat type evaporation can be further modified to use plasma assisted reactive evaporation to produce denser, more sputter-like coatings. Such a process, based on HAD-plasma assisted evaporation was developed by FEP Fraunhofer in Dresden, Germany and scaled up jointly with Applied Materials to production scale.

Furthermore, for different applications an additional organic topcoat on the inorganic Al or AlO<sub>x</sub>-layer can be of advantage. For such applications DSM has developed the Freshure® process which, as well as the HAD plasma process, can be implemented in the TOPMET CLEAR system described below.

## TOPMET-CLEAR

Based on the market demand, a new type of vacuum web coater for production of barrier coatings, the TOPMET CLEAR with the following key features was developed:

- Basic system for metallizing with all advantages of state of the art metallizers like staggered boat arrangement, high speed winding, closed loop quality control, state of the art winding technology
- Optional process kit for reactive AlOx coating without plasma
- Optional Process kit for HAD-plasma assisted AlOx process for highest barrier and optical requirements. Layers with an optical transparency of up to 100 % can be achieved.
- Optional process kit for the Freshure® coating process which is patented by DSM and licensed by Knowfort Technologies BV

In TOPMET CLEAR, all this mentioned process kits are also available for future retrofit at customer's site.

It's possible to start with the AlOx process without HAD-plasma assistance and to retrofit the HAD process later if it is required to fulfil increasing quality, barrier or optical requirements or if other substrates should be used.

### Optical Inline Coating Control

To ensure the product quality, an inline control of the transparent coating is mandatory. This is especially the case for reactive processes, both with- or without plasma assistance. For metallized layers either the measurement of optical- or of electrical properties is possible. For non metallic oxide coatings, the only practical method for inline measurement and control is optically. Even with this method an inline coating control for the transparent AlOx coatings is not simple. For such thin coatings as used for barriers interference effects cannot be used. The only way of measurement is to use the absorption of the layers. So it is needed to select the right wavelengths of the light and also to run the process somewhat sub-stoichiometric. In case the resulting optical performance is then not sufficient it can be helpful to provide an additional plasma post treatment.

In principle also other methods like inline Ellipsometry or inline x-Ray Fluorescence XRF are possible. However, this is for boat evaporation processes not a real option, as here a separate measuring sensor for each boat is required and it would be space and cost wise not practical to install up to 20 or 30 Ellipsometer- or XRF-sensors into one machine.

### Barrier Properties:

Below is a summary of barrier data, achieved with different coating processes on different types of substrates. ( Conditions for the measurements: WVTR 38°C, 90%r.h., OTR 23°C, 0%r.h.)

PET substrate 12 µm	OTR [cm <sup>3</sup> /m <sup>2</sup> *day]	WVTR [g/m <sup>2</sup> *day]
AlOx using HAD-plasma assisted process	< 2	< 1
AlOx using reactive, non-plasma assisted process	< 3	< 3

Table 1: barrier data on PET for AlOx processes

OPP substrate 17 $\mu\text{m}$	OTR [ $\text{cm}^3/\text{m}^2\cdot\text{day}$ ]	WVTR [ $\text{g}/\text{m}^2\cdot\text{day}$ ]
AIOx using HAD-plasma assisted process	< 100	< 1
AIOx using reactive, non-plasma assisted process	300 - 1500	< 9

Table 2: barrier data on OPP for AIOx processes

With the HAD-plasma assisted process, barrier of the following additional substrates were tested in cooperation with Fraunhofer FEP:

Substrate type	OTR [ $\text{cm}^3/\text{m}^2\cdot\text{day}$ ]	WVTR [ $\text{g}/\text{m}^2\cdot\text{day}$ ]
CPP 20 $\mu\text{m}$	< 50	< 0.5
PLA 20 $\mu\text{m}$	< 25	< 25

Table 3: barrier data for HAD-plasma assisted AIOx process on different substrates

The Freshure® layer provides oxygen barrier as a single coating. Below is a summary of OTR values on different substrates with Freshure® coating, provided by DSM.

Substrate	OTR [ $\text{cm}^3/\text{m}^2/\text{day}$ , 23°C/0% RH]
20 $\mu\text{m}$ BOPP	< 20
12 $\mu\text{m}$ PET	< 2
15 $\mu\text{m}$ OPA	< 1
20 $\mu\text{m}$ PLA	< 40

Table 5: OTR of Freshure® single layer on different substrates

According to DSM, an additional Freshure® top coating on AIOx-coated PET or OPP leads to the improvement of oxygen barrier up to a factor of 3 and also to a mechanical protection of the AIOx layer, resulting in a better water barrier. Laminates out of AIOx + Freshure® coated PET and OPA/CPP are retortable.

#### First Products based on the New Processes

After the installation of the first vacuum web coater with the newly developed transparent barrier processes at Biofilm, as first product a transparent packaging for potato-chips was introduced by a major chip manufacturer in Mexico in March 2009. In the meantime different regional and national manufacturers in USA, as well as national manufacturers in Mexico and Columbia are using the new processes in laminated structures for applications like breakfast cereal, fried snacks, baked snakes, red pork rinds, popcorn and tortilla snacks. In addition coated monolayer film is used by a regional manufacturer in USA for packaging of soft cookies and by a national Columbian manufacturer for packaging of hard cookie.

## SUMMARY

These newly in a production web coater introduced vacuum based coating processes for transparent barrier layers provide interesting alternatives to the existing polymer solutions. The ecological properties and environmental impact are greatly improved. The amount of overall material needed for the barrier layer itself is only a small fraction compared to the amount needed for polymer barriers. The material itself is a natural oxide and not toxic or dangerous to the environment or individuals. Recycling and waste management is relatively cheap and simple. In combination with a compostable substrate, it can be even fully biodegradable.

The production process is based on a well accepted and reliable system. The machine layout provides optimum flexibility, depending on the actual needs of the customer and metallizing different kind of transparent barrier processes can be accomplished.

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