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Barrier properties of plastic films coated with an Al_2O_3 layer by roll-to-toll atomic layer deposition

Terhi Hirvikorpi [a,*], Risto Laine [a], Mika Vähä-Nissi [b], Väinö Kilpi [a], Erkki Salo [b], Wei-Min Li [a], Sven Lindfors [a], Jari Vartiainen [b], Eija Kenttä [b], Juha Nikkola [c], Ali Harlin [b], Juhana Kostamo [a]

[a] Picosun Oy, Tietotie 3, FI-02150 Espoo, Finland
[b] VTT Technical Research Centre of Finland, Biologinkuja 7, Espoo, P.O. Box 1000, FI-02044 VTT, Finland
[c] VTT Technical Research Centre of Finland, P.O. Box 1300, FI-33101 Tampere, Finland

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ABSTRACT

Thin (30–40 nm) and highly uniform Al_2O_3 coatings have been deposited at relatively low temperature of 100 °C onto various polymeric materials employing the atomic layer deposition (ALD) technique, both batch and roll-to-roll (R2R) mode. The applications for ALD have long been limited those feasible for batch processing. The work demonstrates that R2R ALD can deposit thin films with properties that are comparable to the film properties fabricated by in batch. This accelerates considerably the commercialization of many products, such as flexible, printed electronics, organic light-emitting diode lighting, third generation thin film photovoltaic devices, high energy density thin film batteries, smart textiles, organic sensors, organic/recyclable packaging materials, and flexible displays, to name a few.

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1. Introduction

Atomic layer deposited (ALD) Al_2O_3 has proven to be effective in enhancing the moisture and gas barrier properties of various plastic films and coatings [1–7]. The key challenge in several applications is to find a flexible, reliable, and cost efficient material to protect sensitive goods from ambient atmosphere. The demand for continuous ALD processing stems from the fields of flexible, printed electronics, organic light-emitting diode lighting, third generation thin film photovoltaic devices, high energy density thin film batteries, smart textiles, organic sensors, organic/recyclable packaging materials, and flexible displays, to name a few.

“Barrier property” refers to a material's capability to resist the diffusion of a specific species (molecule, atom or ion) into and through the material. To be a good gas and vapor barrier, the material needs to be pore-free. When considering polymeric materials, the water vapor transmission rate (WVTR) is affected by e.g. the thickness of the polymer film as well as the temperature and humidity of the surroundings [8–10]. The common polymers used in flexible electronics include e.g. polyethylene terephthalate and polyethylene naphtalene. Hygroscopic materials, such as many biopolymers, typically lose their barrier properties at high relative humidity due to water absorption [11].

The ALD technique is a surface-controlled layer-by-layer deposition process based on self-limiting gas–solid reactions. It is well suited to produce inorganic gas barrier coatings on various materials [12]. The most common ALD-grown gas and water vapor barrier material has been Al_2O_3 [1–3,13–17]. In these studies the Al_2O_3coatings have mainly been fabricated using the TMA-H_2O process, but studies also show that O_3 can also be used as the oxygen source when depositing on polymers [7,18]. The advantages of ALD-grown Al_2O_3 coating are superior moisture protection and relatively low deposition temperature. For the purposes of protecting electronic parts, water vapor transmission rates of the order of $1*10^{-3}$ g/m^2/day and oxygen transmission rates below $5*10^{-3}$ cm^3/m^2/10^5 Pa/day have been reported for less than 25 nm thick Al_2O_3 coatings on plastic [2]. In addition, Park et al. [14] reported a water vapor transmission rate of 0.03 g/m^2/day at 38 °C and 100% relative humidity for an ALD-grown Al_2O_3 barrier that was 30 nm thick and deposited on both sides of a poly(ethersulfone) substrate, whereas Carcia et al. [17] showed that 25-nm thick Al_2O_3 barrier coatings on poly(ethylene naphthalene) substrates can have a water vapor transmission rate of less than $1*10^{-5}$ g/m^2/day.

We demonstrate in this paper the deposition trials on plastic films with a roll-to-roll (R2R) process. This study demonstrates that a thin

* Corresponding author. Tel.: +358 40 770 6648; fax: +358 20 722 7012.
E-mail addresses: Terhi.Hirvikorpi@picosun.com (T. Hirvikorpi), Risto.Laine@picosun.com (R. Laine), Mika.Vaha-Nissi@vtt.fi (M. Vähä-Nissi), Vaino.Kilpi@picosun.com (V. Kilpi), Erkki.Salo@vtt.fi (E. Salo), Wei-Min.Li@picosun.com (W.-M. Li), Sven.Lindfors@picosun.com (S. Lindfors), Jari.Vartiainen@vtt.fi (J. Vartiainen), Eija.Kentta@vtt.fi (E. Kenttä), Juha.Nikkola@vtt.fi (J. Nikkola), Ali.Harlin@vtt.fi (A. Harlin), Juhana.Kostamo@picosun.com (J. Kostamo).

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Al_2O_3 layer deposited with continuous ALD process enhances the barrier performance as much as does the Al_2O_3 layer fabricated with the batch ALD process.

2. Experimental details

The substrates used were commercial plastics films presented in Table 1. From our previous thermogravimetric study performed for most of the present substrate materials [5] we may conclude that the materials do not degrade thermally at temperatures employed in our low-temperature ALD-Al_2O_3 process.

Biaxially oriented polylactic acid (PLA) film had a three-layer structure: one thick core layer sandwiched between two thin skin layers. Polylactic acid was thermoplastic aliphatic polyester in this case supplied by NatureWorks™. One side of the film was corona treated, which typically increases the number of oxygen groups and surface energy. Cellophane film (CEL) was a non-commercial uncoated film. Polyimide film (PI) was an aromatic all-polyimide film with a wide processing temperature window. Cellophane is antistatic and dimensionally stable. PLA has glass transition temperature between 60 and 65 °C, while for cellophane this is typically higher and undefined. A second order transition occurs in PI between 360 and 410 °C assumed to be the glass transition temperature. PLA will likely have the highest strain at elevated temperatures.

Substrates were obtained as small reels. Top layers were simply disregarded before sampling the reels in order to avoid possible dirt particles, finger prints and other contaminants. No other cleaning procedure was carried out before deposition.

Silicon wafers, cellophane, polylactide, and polyimide film substrates were deposited onto with Al_2O_3 at 100 °C using both a batch PICOSUN™ reactor chamber and a PICOSUN™ R2R chamber. The chambers were used in the same PICOSUN™ R-200 ALD reactor. The precursors were trimethyl aluminum (TMA, electronic grade purity, SAFC Hitech) and H_2O. High purity nitrogen (99.9999% N_2) was used as a carrier and purge gas. The operating pressure was 5–10 hPa (mbar) in both reactor chambers. The precursor pulsing sequence for batch process was: 0.1 s TMA pulse, 6 s N_2 purge, 0.1 s H_2O pulse, and 6 s N_2 purge. For the R2R ALD process the precursor pulsing sequence was: 0.1 s TMA pulse, 4 s N_2 purge, 0.1 s H_2O pulse, and 4 s N_2 purge. The number of ALD cycles used for both batch and R2R process run was 500.

The R2R add-on was specially designed to fit to the basic ALD reactor for achieving fully comparable results than with the batch process (Fig. 1). Fig. 2 presents the configuration of the R2R mode. The substrate, unwinding and winding units and an R2R coating channel are inside a batch chamber. The precursors are pulsed into the coating channel similarly to a batch process, while the space with the winding units is purged with nitrogen. The substrate is moved in and out of the coating channel through narrow slits. The narrow slits keep the precursors in the coating channel, and purging gases are fed into a vacuum line. Only the reaction chamber, reaction chamber lid and vacuum chamber lid of the basic ALD reactor were changed. The substrates coated in batch chamber were ca. 100 × 100 mm^2 in size, while the substrates coated by R2R ALD were 70 mm wide and continuously moving at speed of 200 mm/h, from the unwinding roll to the winding roll. The servo motors which rotated the unwinding and winding rolls were located outside of the vacuum chamber. The sizes of the reaction chamber and vacuum chamber lids were the same in both batch and R2R processes; however, the R2R chamber lid was specially designed for the R2R mechanics. The basic controlling software was the same for both configurations, except the additional control panel for servo motors in the R2R software.

Table 1
The commercial plastic materials employed as substrates.

Code	Description
PLA	Biaxially oriented polylactide film (EVLON (R) EV) from BI-AX International Inc., Canada, one-side treated, thickness 40 μm
PI	Kapton® HN polyimide film from DuPont, thickness 127 μm
CEL	Cellophane film from Innovia Films, thickness 27 μm

Fig. 1. R2R setup assembled to a batch ALD reactor (A) and the R2R mechanism (B).

Coatings were grown on one side in batch processing and on both sides of the substrates in the R2R process. The Al_2O_3 deposited samples were characterized for their barrier and surface characteristics. The thickness of the fabricated Al_2O_3 coatings was estimated by growing the Al_2O_3 films on silicon wafer and measured by single wave-length ellipsometer. Because of the different surface chemistries of different polymers, the actual thickness may somewhat deviate from that

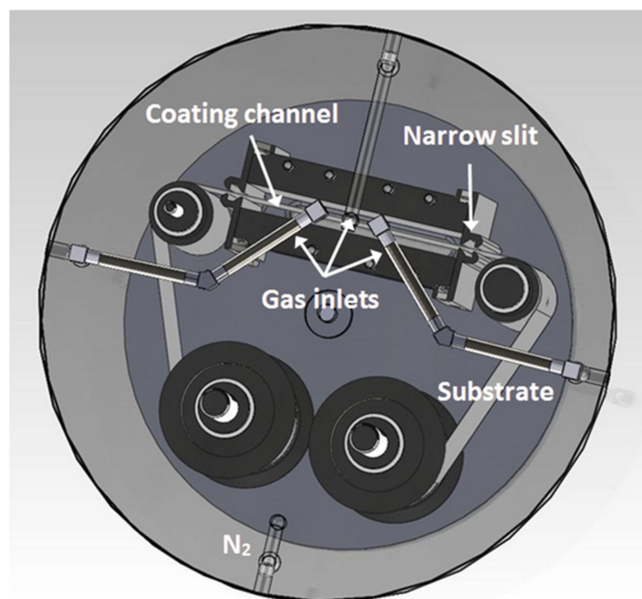

Fig. 2. The R2R mechanism in the batch ALD chamber.

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