## **Industrial Inline Control for Advanced Vacuum Roll to Roll Systems**

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### **ABSTRACT**

Production for the future FED (**F**lexible **E**lectronic **D**evices) and security markets must begin to take advantage of roll to roll coating. To guarantee high productivity and high quality, inline control of layer properties are required. This paper presents spectrometric and new ellipsometric inline measuring methods developed for a variety of different vacuum coating systems. Selected applications include the preparation of copy protect layers employing color shift and high quality diffusion barrier layers for FED in both Solar and OLED.

#### **INTRODUCTION**

Today's roll to roll systems are well known in the field of packaging and capacitor manufacture. They offer high productivity, reasonable coating cost and good reliability. New advanced Al coating systems for packaging reach coating widths of up to 4.5 m and web coating speeds of the order of 10 m/s. New markets for Solar, OLED (display and lighting) and security devices must begin to take advantage of new coating technologies to reduce the price per m² in order to be fully implemented in mass production. For roll to roll production with coating lengths of several 1000 m inline quality measurement and control is therefore essential. This is easy to understand for products which have to guarantee long lifetimes e.g. solar films. Examples including large area diffusion barrier layers for packaging, copy protect layers and ultra high diffusion barrier layers for FED employed in Solar and OLED will be therefore shown.

The transfer of the necessary measurement techniques from the laboratory scale up to industrial production scale under fast moving inline conditions represents a large challenge particularly in terms of determining the optical properties of a complex layer stack. This paper will therefore also present specific advances made in the development of an inline ellipsometric quality measurement and control system required for the deposition of high density, high quality diffusion barrier layers for FED employed in the Solar and OLED industries.

#### **HIGH PRODUCTIVE LAYERS FOR PACKAGING**

The three most commonly used roll to roll coating technologies are evaporation, sputtering and PECVD respectively. As far as the food packaging industry is concerned the reduction of the film coating cost per m² is of the upmost importance. The most commonly used coating technique within this industry is the thermal evaporation of Al using boat systems in order to deposit barrier films. Most production systems reach coating widths of up to 4.5 m at coating speeds of the order of 10 m/s. Last year Applied Materials introduced the world's largest Al boat evaporation system with 4.45 m coating width (Fig. 1). The annual output of such a system is approx. 11,000 t per year. These gigantic machines demonstrate the possibilities of roll to roll film production. Inline measurement and control of these production tools is typically achieved by monitoring the optical absorption characteristics of the deposited Al layers.

#### **COPY PROTECT LAYERS BY HIGH RATE ELECTRON BEAM EVAPORATION**

Annual losses through counterfeiting, product piracy and brand diversion are estimated to now account for as much as 7% of world trade, costing between \$240 and \$360 billion USD in lost revenue [1]. Several anticounterfeiting elements are used for brand labels and banknotes such as holograms, security inks and water marks etc. Up to now the production of security layers based on the optical Fabry Perot effect suffered from high production costs using sputter technology. The tolerance of the optical layers can easily be achieved by sputter technology but the deposition rates are too low for cost effective production. An alternative method for layer deposition is thermal evaporation with a high power Electron Beam gun. The main challenge for this technique is the required layer thickness accuracy.

#### **FABRY PEROT (FP) LAYER SYSTEM**

In optics a Fabry Perot interferometer is typically made of a transparent layer with two reflecting surfaces. Fig. 2 shows a thin film layer system with a fully reflecting base-layer (e.g. Al), a transparent middle layer (e.g. SiO2 or Al2O3) and a semi-transparent top layer (e.g. Al). This creates "color" as a consequence of optical interference in much the same way as color generated by an oil film on water. The base color is

dependent on the thickness of the middle layer [2]. The color is also dependent upon the viewing angle. For example the coated film sample shown in Figure 3, has for a perpendicular viewing angle  $(0^{\circ})$  a green color. By turning the film sample to 45° the **color shifts** to a blue color (Fig. 3). This type of layer system (color shift film) can therefore easily be integrated into a bank note or medical package (Fig. 4).

A rather simple structure for a Fapry Perot thin film system consists of three layers in the order Al /  $Al_2O_3$  / Al respectively deposited onto a plastic film. For each of the layers deposited, the starting material is pure Aluminum evaporated by a Electron Beam reacting only with oxygen when the  $A_1O_3$  layer is required (Fig.5). Typically a 20 µm thick PET film is used as the substrate material. The coating speeds are in the range of 10 m/s for the metallic layers and 0.5 m/s for the Al<sub>2</sub>O<sub>3</sub> layer. In the electron beam coater the spectral reflectance is measured with sensor heads equally spaced along the web width (Fig. 5 and 6). Figure 7 shows the measured optical reflectance curves for the most important  $A<sub>1</sub>O<sub>3</sub>$  layer during start up when the layer itself is not uniform. This results in the observation of coatings displaying a wide variety of different colors. This optical reflectance data is transferred to a highly advanced electron beam control system (ESCOSYS from Applied Materials) which varies the electron beam power delivered to the evaporation crucible in order to obtain the desired optical characteristics of the deposited thin film (Fig. 7). By employing this optical monitoring and control technique it was possible to obtain thickness uniformities within the range of +- 2 % thereby resulting in coatings of uniform color. Consequently typical coating costs can be reduced from 18 \$ /m² for the standard FP sputtering process to 0.8  $\frac{6}{m^2}$  using EB evaporation [2]. This therefore opens up a variety of new cost effective potential applications for optical security layers for high rate EB evaporation based processes.

#### **ULTRA HIGH DENSITY, HIGH QUALITY BARRIER LAYERS FOR SOLAR AND OLED FED'S**

Solar and OLED layer systems need ultra high quality diffusion barrier layers to protect against damage associated with water vapor and oxygen. The required barrier properties are several orders of magnitude more stringent than currently used in standard packaging films. One possibility is to produce a separate ultra high quality diffusion barrier film and laminate this film on to the flexible solar or OLED film. Typical diffusive flux values for water vapor diffusing through food packaging are of the order of 1 g/m²/day. This must be reduced to values of  $\sim 10^{-4}$  down to  $10^{-6}$  g/m²/day depending on the desired application and lifetime. Ultra high quality diffusion barrier films usually consist of a multilayer structure containing both inorganic and organic layers. The organic or "cross linked" layer, can be produced in vacuum by a PECVD process or alternatively at atmospheric pressure using special lacquers (e.g. ORMOCER – ISC/FhG). The inorganic layer (e.g. SiOx or  $A<sub>1</sub>O<sub>3</sub>$ ) is produced by a vacuum process. It is important to choose the right process in order to fulfill the necessary requirements (Fig. 8). High rate electron beam evaporation (e.g. for SiOx) offers a balance between reasonable coating cost and a moderate coating lifetime. Sputtering however provides a higher coating quality but with the disadvantage of a higher coating cost.

### **INLINE ELLIPSOMETRIC CONTROL FOR SOLAR ABD OLED FED BARRIER LAYER'S**

To reach the high barrier properties and lifetime guarantees required for both Solar and OLED applications an advanced inline control and quality management system is necessary. Ellipsometry is a well known lab measurement technology for determining layer properties of oxides such as SiOx orAl<sub>2</sub>O<sub>3</sub>. The challenge has been to develop an ellipsometric inline measurement system for vacuum high speed EB evaporation and sputter coating systems.

Fig. 9 shows the measurement principle of the ellipsometer in a SMART WEB sputter machine. Measurement with the ellipsometer requires highly precise alignment and stability of the measurement lightbeam (less than 1° on the moving film). The development of the UFMWE (ultra fast multi wavelength ellipsometer) was carried out by Jobin whereas the subsequent integration and testing in vacuum coating systems was performed by Applied Materials. This work is currently funded by the EU project FLEXONICS (Ultra high barrier for r2r encapsulation of flexible electronics - EU partners are: Alcan, Applied Materials, Jobin Horiba, IVV / ISC –FhG, Isovolta, Siemens, Konarca, Uni. Graz, University of Thessaloniki). Fig. 9 also shows the ellipsometer installed on the SMART WEB sputter machine. To be able to obtain the layer thickness and the refractive index from the ellipsometric measurement rough data ( Ψ / ∆ ) mathematical models of the deposited layers are required. These models must be specially designed for the system layer stack/film e.g. SiOx or Al<sub>2</sub>O<sub>3</sub> on a 50 µm thick PET substrate. This work was performed by the University of Thessaloniki. Professor Logothetidis will give a brief description of ellipsometric theory and the mathematical models used in the same TAPPI session.

Besides the integration of the ellipsometer in the sputter machine, the ellipsometer was also integrated into a high speed electron beam deposition system (Fig. 10). The UFMWE from Jobin was essential for single measuring times lasting only seconds. Fig. 11 shows the graph of a SiOx barrier coating on a PET film produced using an Electron Beam evaporation and inline control (ESCOSYS) using the thickness data obtainined by ellipsometric measurement and modeling. Offline check measurements of the coated SiOx film confirm the ellipsometric data.

## **CONCLUSION**

Advanced vacuum coating technology and the use of the latest developments in fast inline measurement and control opens new possibilities in the flexible Solar, OLED and copy protection markets. Indeed, Fabry Perot color shift layers deposited by high rate electron beam evaporation using an advanced inline control with spectroscopic reflection measurement have been demonstrated. It is possible to use ellipsometric inline control for vacuum coating of flexible webs in production machines. This will provide new possibilities for production and quality control in the preparation of ultra high quality diffusion barrier layers for Solar and OLED based applications.

#### **REFERENCES**

- 1. Security Labels.com
- 2. H.G. Lotz, R.Ludwig and G.Steiniger, "Production of Color Shift Layers with Electron Beam Technology for Identification and Decorative Applications",2007 Society of Vacuum Coaters, 50TH Annual Technical Conference Proceedings

#### **Figures:**





Fig. 2 Principle of the Fabry Perot layer system.



Fig. 3 Fabry Perot Layer with green color  $(0^{\circ})$ , and turned to  $45^{\circ}$  changes to blue color.





Copy protection mark or stripe in future possible with color shift layers.



Fig. 4 Various colors created using the Fabry Perot layer system on a PET film (e.g. Al- Al<sub>2</sub>O<sub>3</sub>-Al).



Fig. 5 Electron beam evaporation with reflection measurement and inline control . guns, 2,1 m coating width



 Fig. 6 Electron beam system with two (TOP BEAM-Applied Materials).

# Layer thickness control by the electron beam





# Functional Layers for FED\*:

# Multi-layer stag for a barrier film for display and solar applications

Inorganic layers : Al2O3, SiO2:



\*FED : Flexible Electronic Devices

Fig.8 High barrier multilayer stack. (web speed – approx. : evaporation  $m/s$  sputtering  $m/min$ )



Fig. 9 Ellipsometer integrated in the SMART WEB sputter system.

# Advanced inline measurement by ellipsometer at a high speed electron beam evaporation system

Installation of the ultra fast multi-wavelength ellipsometer at the TOP BEAM lab system of **Applied Materials** 

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Fig.10 Ellipsometer integrated in a high speed electron beam evaporation system



Fig. 11 Graph of SiOx electron beam coating with closed loop inline control using ellipsmeter measurement data for the layer thickness