University of Huddersfield Repository

Blunt, Liam, Fleming, Leigh, Elrawemi, Mohamed, Robbins, David and Muhamedsalih, Hussam

In-line metrology of functional surfaces with a focus on defect assessment on large area Roll to Roll substrates

Original Citation


This version is available at http://eprints.hud.ac.uk/id/eprint/18216/

The University Repository is a digital collection of the research output of the University, available on Open Access. Copyright and Moral Rights for the items on this site are retained by the individual author and/or other copyright owners. Users may access full items free of charge; copies of full text items generally can be reproduced, displayed or performed and given to third parties in any format or medium for personal research or study, educational or not-for-profit purposes without prior permission or charge, provided:

* The authors, title and full bibliographic details is credited in any copy;
* A hyperlink and/or URL is included for the original metadata page; and
* The content is not changed in any way.

For more information, including our policy and submission procedure, please contact the Repository Team at: E.mailbox@hud.ac.uk.

http://eprints.hud.ac.uk/
In-line metrology of functional surfaces with a focus on defect assessment on large area Roll to Roll substrates

L. Blunt¹, L Fleming¹, M. Elrawemi¹, D. Robbins², H. Muhamedsalih¹
¹ University of Huddersfield, UK,
² Centre for Process Innovation, Sedgefield, UK
l.a.blunt@hud.ac.uk

Abstract
This paper reports on the recent work carried out as part of the initial stages of the EU funded NanoMend project. The project seeks to develop integrated process inspection, cleaning, repair for nano-scale thin films on large area substrates. Flexible photovoltaic (PV) films based on CIGS (Copper Indium Gallium Selenide CuInₓGa₁₋ₓSe₂) have been reported to have light energy conversion efficiencies as high as 19%. CIGS based multi-layer flexible devices are fabricated on polymer film by the repeated deposition, and patterning, of thin layer materials using roll-to-roll processes (R2R), where the whole film is approximately 3µm thick prior to final encapsulation. The resultant films are lightweight and easily adaptable to building integration. Current wide scale implementation however is hampered by long term degradation of efficiency due to water ingress to the CIGS modules causing electrical shorts and efficiency drops. The present work reports on the use of areal surface metrology to correlate defect morphology with water vapour transmission rate (WVTR) through the protective barrier coatings.

1.0 Introduction
To address the PV degradation problem a thin (~40nm) coating of Al₂O₃ has been implemented to provide the environmental protection (barrier) for the PV cells. The highly conformal aluminium oxide barrier layer is produced by atomic layer deposition (ALD) onto a Polyethylene naphthalate (PEN) substrate. The surface of the starting polymer is further planarised to give a high quality smooth surface prior to ALD. The presence of surface defects, pin holes and debris particles on the uncoated film can create significant defects within the, aluminium oxide, barrier layer. This paper reports the results of measurements conducted to characterise the uncoated and barrier coated polymer film surface topography using segmentation
feature parameter analysis. The presence of defects is then correlated with the water vapour transmission rate as measured on representative sets of films using a standard MOCON test. The paper also shows initial measurement taken on a prototype in process, high speed, environmentally robust optical interferometer instrument developed to detect defects on the polymer film during manufacture. These results provide the basis for the development of R2R in process metrology devices for defect detection.

2.0 Barrier Substrate

A series of 4 coated substrates were produced having a 40nm ALD Al₂O₃ barrier coating. An area of 80mm² was used for testing of the barrier properties using a standard MOCON test, fig 1. This test measures the steady state WVTR for a barrier coating under defined conditions. The system places the substrate in a sealed unit where one side of the substrate is subject to high humidity and the other side is defined as the dry side. The dry side is purged with a carrier gas which carries away any transmitted water vapour to an infrared sensor which records the transmission rate. The steady state rate was recorded along with the time to stable transmission.

3.0 Results

Table: 1 Water vapor transmission rate at specified conditions 38°C and 90% RH

<table>
<thead>
<tr>
<th>Sample No</th>
<th>WVTR (g/m²/24 hrs.)</th>
<th>Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>2701</td>
<td>1.1x 10⁻³</td>
<td>11 days</td>
</tr>
<tr>
<td>2702</td>
<td>1.3 x 10⁻³</td>
<td>11 days</td>
</tr>
<tr>
<td><strong>2705</strong></td>
<td><strong>4.1x 10⁻³</strong></td>
<td><strong>5 days</strong></td>
</tr>
<tr>
<td>2706</td>
<td>2.0x 10⁻⁴</td>
<td>5 days</td>
</tr>
</tbody>
</table>

The WVTR results show that sample 2705 had a significantly higher WVTR than the other specimens. Following WVTR testing the surface topography of all samples (including uncoated) was measured using laboratory based coherence correlation interferometry.
700 measurements, equating to 14% of the total surface area of all the specimens was measured. The results showed the presence of defects both particulate and pin hole type in all specimens. Typical examples are shown in fig 2. The surface roughness of defect free samples was ~0.6nm. Areal topography characterisation was carried out using the feature parameter set ISO 25178-particle. In particular the parameter Sfd was used (where Sfd = the number of significant hills + significant dales); significance was defined as any peak/pit greater than 20% of the total peak to valley roughness (Sz). Using this default significance value for the defects the results showed no clear correlation with the WVTR results. However when the significance criteria was increased for hills (Peaks) and dales(pits) (over +/- 3xRMS roughness and >15µm lateral dimension) the Sfd parameter could be used to count only the most severe defects over the total measured area. In this case the correlation was clear (figure 3b). The results indicate the presence of small numbers of large defects dominate the WVTR of the barrier layer. ALD coating is highly conformal and is likely to coat particulate debris and down deep pits. The mechanism for increased WVTR would be that debris on the surface or within pits become detached exposing uncoated PEN to water ingress.

Figure 2 Typical morphology of substrate defects a) large hole 60µm wide b) particulate debris 30nm high c) White light scanning interferometry (WLSI) measured defect
The aim of these results is to implement on line metrology for the roll to roll ALD process and using the knowledge gained from the present work implement areal feature analysis to carry in-line metrology and process control. The above analysis is now combined with a robust wavelength scanning interferometry instrument having internal environmental compensation to carry out the measurement work using parallel sensors to cover large areas of the substrate surface.

Conclusions
The present work has shown the potential of areal feature segmentation to detect functionally significant defects present in roll to roll produced ALD barrier coatings of $\text{Al}_2\text{O}_3$. The results show a good correlation between the presence of small numbers of large defects and WVTR. The analysis provides the basis for in process metrology for roll 2 roll production of barrier coatings for flexible PV arrays and is a first demonstration of in process use of feature parameters. Work is continuing to check repeatability of these tests and produce “cleaner” substrates.

Acknowledgement
The authors would like to thank the EC for providing funds to carry out this work via the NanoMend project NMP4 LA-2011-280581.

References: