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# Metrology and Characterisation of Defects on Barrier Layers for Thin Film Flexible Photovoltaics

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## Abstract

This paper reports on the recent work carried out as part of the EU funded NanoMend project. Part of the project seeks to develop integrated process inspection, cleaning, repair and associated control systems for the manufacture of large area, nano-scale thin films of CIGS (Copper Indium Gallium Selenide  $\text{CuIn}_x\text{Ga}_{(1-x)}\text{Se}_2$ ) flexible PVs.

Transparent barrier films such as aluminium-oxide ( $\text{Al}_2\text{O}_3$ ) can be used as a barrier to oxygen and/or water vapor permeation and such layers are of increasing interest for the encapsulation of flexible PV modules. However, the existence of micro and nano-scale defects in the barrier surface topography has been shown to have the potential to facilitate water vapor ingress, thereby reducing cell efficiency and causing internal electrical shorts in the CIGS cells. Therefore, improvement of the quality of these barrier films is critical to reduce the susceptibility of these photovoltaic systems to environmental degradation. This paper reports on the development of a characterisation method for defect detection and then correlates this with measured water vapor transmission rates (WVTRs). The results show small numbers of large defects play the dominant role in determining the WVTR. A unified classification system for defects is proposed where the classification breaks down the defects types into four main functional groupings.

## 1 Introduction

Thin layers of aluminium-oxide ( $\text{Al}_2\text{O}_3$ ), of the order of a few tens of nanometres, thermally evaporated onto polymeric web in roll-to-roll vacuum metallisers and deposited via the atomic layer deposition technique (ALD), have been introduced to allow PV modules transparency, flexibility and to provide protection from the ambient environment. These barrier films have an effective water vapor transmission rates (WVTR) of less than  $10^{-4}$  g/m<sup>2</sup>/day. However, the barrier properties are often influenced by a wide range of variables, making conclusive WVTR capabilities sometimes difficult to guarantee. Hence, in order to optimize the photovoltaic (PV)

module performance and lifespan, a study to understand the nature of surface defects (shape, size-scale, density, and morphology) in the barrier layer which allow water vapour and oxygen ingress to the active (absorber) layer has been carried out.

### 1.1 Barrier Preparation

A set of six coated substrates were produced having a 40nm ALD  $\text{Al}_2\text{O}_3$  and coded as shown in table (1). Each sample has an 80mm diameter, and was produced in accordance with previous studies [1]. However the pre coating procedures for the present samples were varied in order to investigate the effect of cleanliness on WVTR. The samples were measured for water vapor transmission rate (WVTR) at  $38\text{C}^\circ$  and 90% RH using Isostatic standard test (MOCON®) instrumentation prior to the surface measurements, Fig(1) [1]. Table (1) shows the WVTR for each sample after stabilisation time.

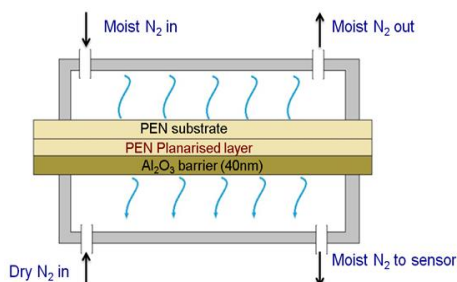


Figure 1: MOCON test set up [1]

## 2 Surface Characterisations

White Light Scanning Interferometry (WLSI) technique and Scanning Electron Microscopy (SEM) were employed for collecting large amounts of surface data from the PV barrier layers defects, where  $\geq 2000$  measurements were conducted on each sample. Wolf pruning as defined in ISO 25178-2 [2], was performed on the set of data by trying out various segmentation criteria. The segmentation was applied by means of an “iterative” process. The surface was filtered to eliminate data noise, where the box filtering (Gaussian filtering) uses a cut-off of  $2^n$  points; where n is the smooth level (from 1 to 5), and n was specified to be 5. After smoothing, edge processing was performed on the data using a Sobel type operator [2]. The edge data is then “pruned” by means of Wolf pruning [2], where all data elements below 1% of the  $S_z$  value are combined, and those elements higher than 1%  $S_z$  were retained as

Table 1: WVTR at  $38\text{C}^\circ$  and 90%RH

Sample No	$\text{AlO}_x$ thickness	WVTR ( $\text{g}/\text{m}^2/24$ hrs.)
*12k1001	40nm	$5 \times 10^{-4}$
*12k1002	40nm	$< 5 \times 10^{-5}$
**12k0902	40nm	$1 \times 10^{-3}$
**12k0901	40nm	$< 5 \times 10^{-4}$
***12k0803	40nm	$6 \times 10^{-4}$
***12k0804	40nm	$< 5 \times 10^{-4}$

\*Polymer surface unprotected before loading for ALD.

\*\*Polymer surface protected to the last moment before loading into ALD. However, some visible scratches were reported on sample 12k0902.

\*\*\*Contact cleaning of the polymer before ALD.

significant. Following Wolf pruning an area prune was applied such that if an area was found to be less than 2.5% of the total area (this area being defined by optical and SEM analysis) it was deemed insignificant and combined with its neighbouring region. Figure 2 (a-b) show defects before and following the

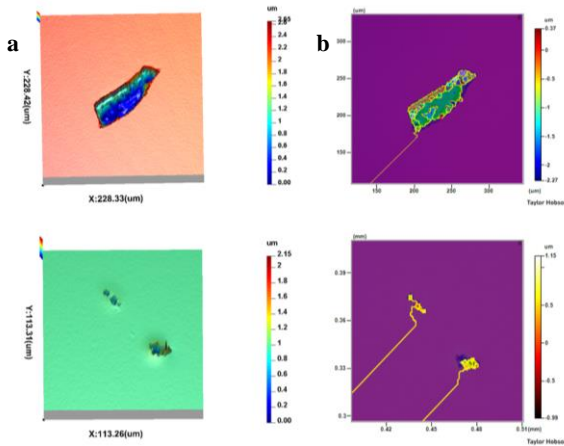


Figure 2: (a). Defects before after segmentation, (b). Defects after segmentation

## 2.1 Results and Discussion

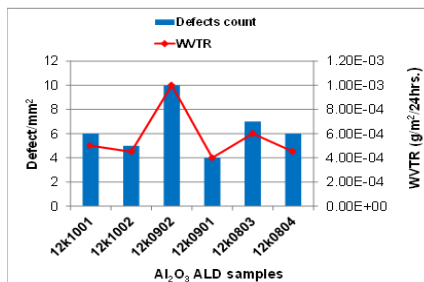


Figure 3: WVTR versus defect density

segmentation process for two different samples. The figure shows the power of the procedure for automatically extracting defects from the surface data. Following the extraction the defect density for significant defects can be simply calculated.

The results in figure (3) indicates that, sample with the lowest WVTR value (12k0901) shows the lowest defects density. This sample had the least contaminating processing prior to coating, it is also interesting to note that where visible large scratches were reported (sample 12k0902) the highest defect density and WVTR occurred.

Previous work [1] and data presented in this study seem to show that for the barrier coatings a small number of large defects dominate the WVTR shown in table (1), and thus these defects should be the focus of any detection system

## 1.2 Defects Classification System

A defect classification system was developed in order to enhance the interpretation of defect data within the NANOMenD project and across the large area substrate sector

in general. At present no such system exists however very recently a system for defect classification in the die polishing industry has been proposed [3]. This system has now been adopted for the NANOMenD project and allows a unified classification of defect to be implemented. This classification system lists the most detrimental defects correlating with high WVTR. The proposed system is based on breaking the defect types down into four main groupings and symbols; (i). Inwardly directed

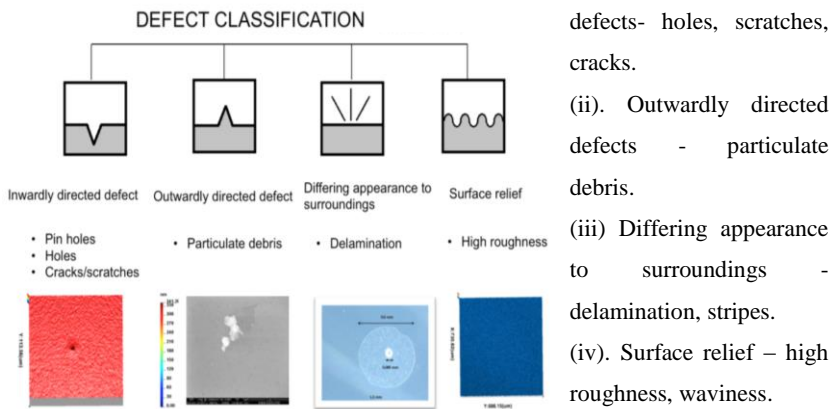


Figure 4: Classification of defects on barrier substrate

### 3. Conclusion

The study emphasises the importance of reducing contamination during ALD process (protection till just before ALD), and shows the potential of segmentation and edge process methods to detect the most significant defects present in the barrier coatings of the ALD Al<sub>2</sub>O<sub>3</sub>. The results have confirmed that small number of large defects dominates the WVTR and thus these defects should be the focus of any detection system. In addition, The classification system of the defects presented in this paper can be applied to all large area substrates to facilitate improved process control.

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