Characterisation techniques to assess functional properties of barrier coatings for flexible PV substrates

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Abstract

This paper details findings from recent work undertaken as part of the EU funded NanoMend project. The aim of the project is to develop integrated process inspection, cleaning, repair and control systems for nano-scale thin films on large area substrates.

The presence of surface irregularities on the uncoated film can induce “defects” within the, aluminium oxide, barrier layer, these features are in the order of magnitude of nanometres to a few microns. These “defects” are examined and quantitatively characterised. The measurements are carried out using a Taylor Hobson Coherence Correlation Interferometer an optical microscope and SEM. The efficient separation of small features from large features is achieved using data segmentation and feature parameter techniques. The presence of both large and small features is then correlated with the water vapour transmission rate as measured on representative sets of films using at standard MOCON (quantitative gas permeation) test. The paper concludes that, based on analysis of water vapour transmission rates (WVTR) and defect size that small numbers of large features have a more significant effect on the deterioration of water vapour transmission rates. It is the WVTR which indicates the potential longevity of the device as it is the effectiveness of this ALD barrier layer to water vapour which ensures the effective function of the core CIGS layers encapsulated in the device.

The ability to measure and effectively characterise the features which are significant defects in the ALD barrier layers provides novel information to enable automatic detection and correction of potential restrictors to device performance in the processes.

1 Background

The latest flexible PV (photo-voltaic) film technologies have efficiencies at or beyond the level of Si based rigid PV modules; this is thanks to significant investment in research activities in order to further develop these technologies. These flexible devices offer significant advantages over rigid Si based technologies as they offer reduced mass, and increased opportunity for building integration (BIPV). Devices currently available are susceptible to environmental degradation which limits their long term use. The devices are fabricated on polymer film by the repeated deposition, and patterning, of thin layer materials incorporating roll-to-roll processes. The thickness of the film is approximately 3um prior to encapsulation. The environmental degradation occurs when water vapor transmission occurs through the barrier layers to the CIGS (Copper Indium Gallium Selenide $CuIn_xGa_{(1-x)}Se_2$) PV cells thus causing electrical shorts, efficiency drops and ultimately failure.

One method of environmental protection for the GIGS cell is to apply a barrier coating of Al_2O_3 to the encapsulation material. The highly conformal Al_2O_3barrier layer is produced by atomic layer deposition (ALD). The surface of the encapsulation substrate polymer film must be of very high quality in order to ensure a robust effective barrier layer can be deposited, in order to achieve this high quality surface finish of the substrate prior to ALD coating, the substrate film is planerized

1.1 Flexible PV Modules

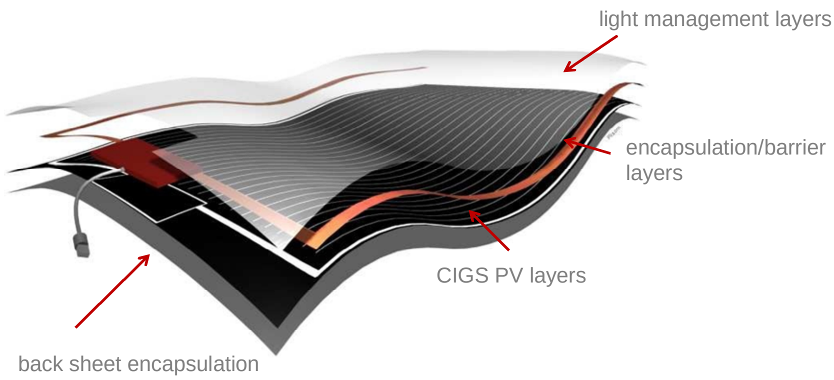


Figure 1: Schematic of the flexible PV Module

Figure 1 shows a schematic of the structure of the flexible PV modules, it is the encapsulation/barrier layer that is the focus of the investigaction reported in this paper. The layer is typically formed from a planerised PET sheet with an Al_2O_3 coating which is applied by atomic layer deposition which technique which provides a highly conformal precision layer that provides environmental protection in the form of a barrier to water vapour transimision to the critical internal layers of the modules. The efficiency and conformity of these barrier layers gives great potential to increase the longevity of these devices makeing them a viable and affordable technology.

1.2 Water Vapour Transmission Rate Assesment

Water vapour transmission is assesed by either quantitative calcium resistivity or through use of a MOCON® (figure 2) which is an instrument used to asses gas permeation rates of ultra high barrier films, this is an accurate method of obtaining quanititative assesment of WVTR (water vapour transmission rates) of the ALD coated barrier layers under assesment.

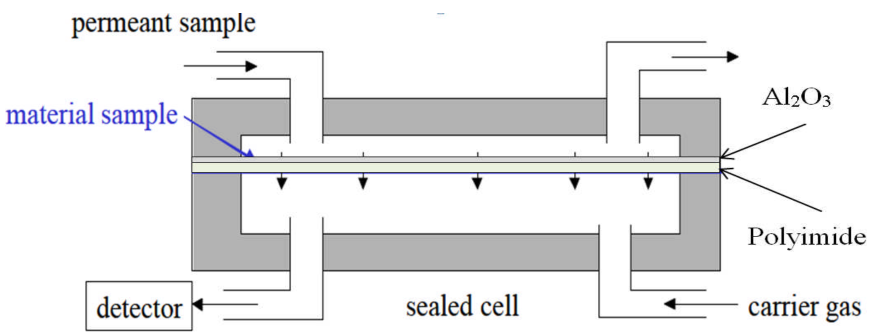


Figure 2: WVTR Measurement using MOCON method

The area of measurement over which the WVTR is assessed is limited to 80mm diameter. The main challenge that this presents for the current study is this large area in which defect detection must be completed. In order to investigate the surface at the required resolution to observe and quantify potential defects the area of measurement is restricted to a few mm at most.

2 Measurement Methodology

In total four samples all ALD coated with Al_2O_3 were assessed for WVTR, these along with a non coated substrate sample were then assessed to determine the presence and magnitude of features which had the potential to influence the WVTR showing the areas to be defective. The samples were initially examined blind with no knowledge of the WVTR for any of the samples.
Three key measurement techniques were employed to survey the surface at different scales of measurement in order to capture all potential features which

may be classed as “defects”. These techniques were Optical microscopy, White Light Scanning Interferometry and Scanning Electron Microscopy (SEM). The focus of this study will report the findings from the optical Interferometry.
An initial survey was completed using the optical microscope to determine the concentration of features which may be classified as defects and to asses the appropriate scale of measurement.

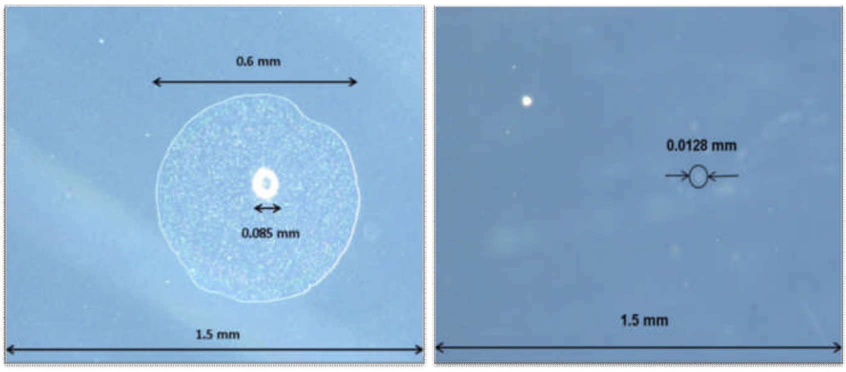


Figure 3 Optical image showing the scale of larger and smaller features observed

2.2 White Light Scanning Optical Interferometry

The scale of defects observed from the broad survey using the optical microscope determined the appropriate measurement protocol for measurements taken completed using white light scanning Interferometry.
The instrument used was the Ametek Taylor Hobson CCI 3000 (Coherence correlation Interferometer). A 20x magnification lens was used which gave a measurement area of apx 1mm x 1mm. this gave adequate spatial resolution to detect features at both the larger and smaller observed features.
At this scale of measurement it is not feasible to survey the entire surface so a structured approach to measurement was adopted in order to provide a statistically significant representation of the surface of each sample.

2.3 Measurement Protocol

A total of 3 studies were performed on each sample.

- 10 x 10 matrix of measurements to give an overall 2% of the tested area
- 100 measurements spaced evenly over the tested area
- A Repeat of the 10 x 10 measurement matrix

The measurements were completed on both the ALD coated samples and also the substrate polymer sheet.

A number of different types of feature were observed and catalogued, parametric analysis has been completed comparing areal surface texture parameters for each sample. Feature parameters were also used to determine the number and type of features for each sample. These parameters were subsequently compared with the WVTR for each sample.

3 Results

The WVTR (water vapour transmission rates) for each sample can be seen in table 1. There were two distinct groups of rates, where one sample showed a significantly higher WVTR, the other samples showed no statistical variation.

Table 1. Water vapour transmission rates

Sample	WVTR (High or Low)
2701	Low
2702	Low
2705	High
2706	Low

Although there was some variation with the standard areal surface texture parameters (table 3) the general texture of topography was of less interest than presence of isolated features which may result in a defective or compromised ADL coating. A range of typical features were noted and presumed to be potential defects in the substrate, the coating or both (table 2).

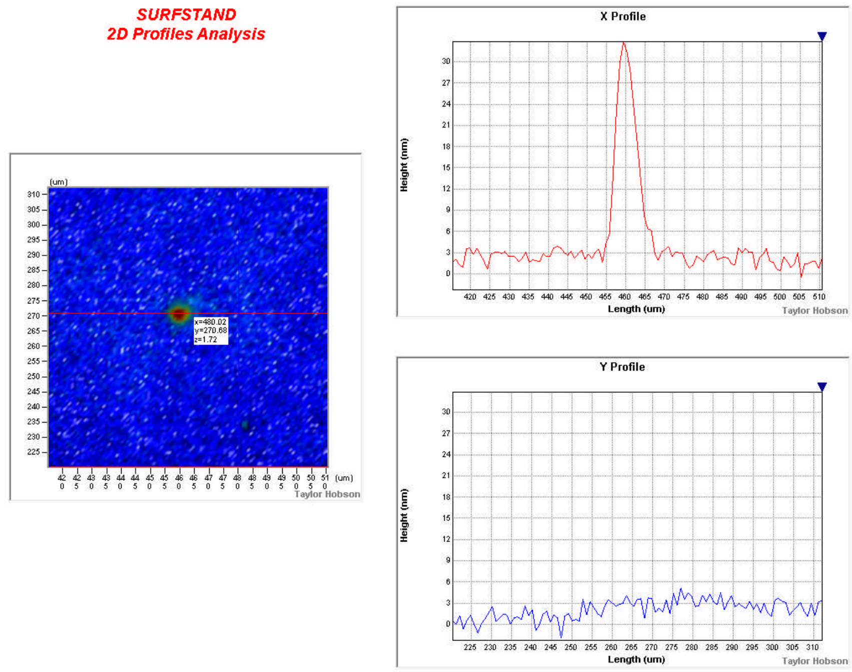
3.1 Catalogue of Features

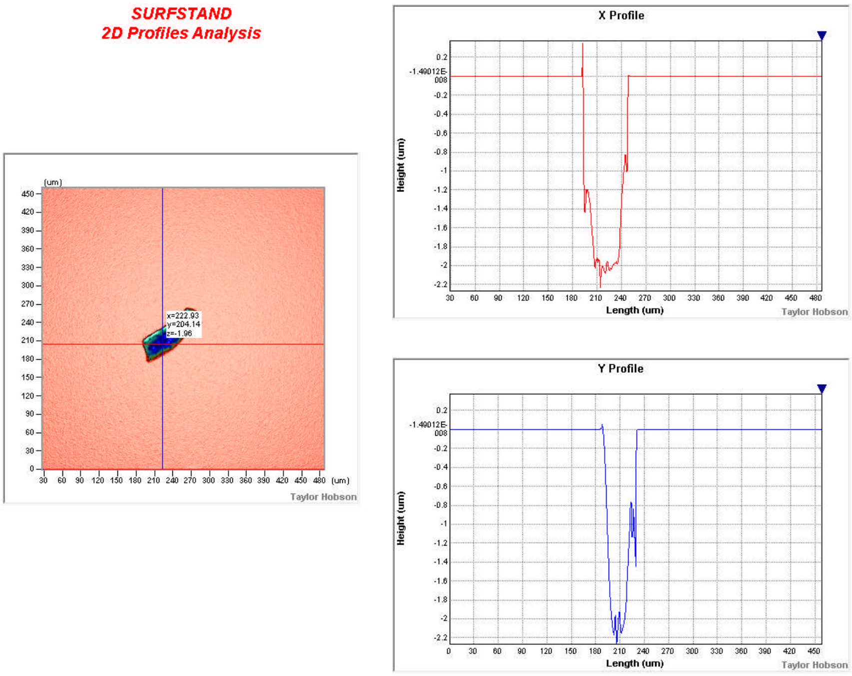
A number of different features were observed in the topography of the samples, these could be classified into the following types of feature dependant upon size and prominence. Figures 4 and 5 show two of these typical features peaks (figure 4) and holes (figure 5).

Table 2 Catalogue of features observed

Type of defect	Size
Spikes	> 700 nm in height
Cracks	-
Scratches	-
Ghost defects	30-50 µm lateral dimension
Pinholes	1 to 3 µm lateral dimension
Peaks	< 33 nm
Holes	60 µm lateral dimension

Ghost defects refer to features where it is not clear if they are raised "peaks" or depressed "pits".





3.2 Areal Surface topography assessment

Table 3 shows the variation in Sa – average roughness for the 3 studies completed. There is little variation in these figures and no correlation with the level of WVTR, what did correlate with WVTR was that the largest range in Sa was found on the sample with the highest WVTR. When interrogating the data from the first study, this large range was found to be from isolated features skewing the results. A method of setting appropriate boundaries in order to employ a wolf prune method to automatically detect hill and dale features was adopted. From this the density of peaks (figure 6), dales (figure 7) and significant features (figure 8) was determined.

Table 3 Sa – Average roughness of the samples from each of the 3 studies

Sa parameters mean value (nm)			
Sample No	Exercise 1	Exercise 2	Exercise 3
Sample 2701	0.909	0.841	0.875
Sample 2702	0.853	0.732	0.792
Sample 2705	0.789	0.776	0.783
Sample 2706	0.865	0.874	0.865
Uncoated /S	1.06	0.965	1.01

A peak is defined as a point on the surface which is higher than all other points within a neighborhood of that point (ISO 25178-2:2012 (E)).

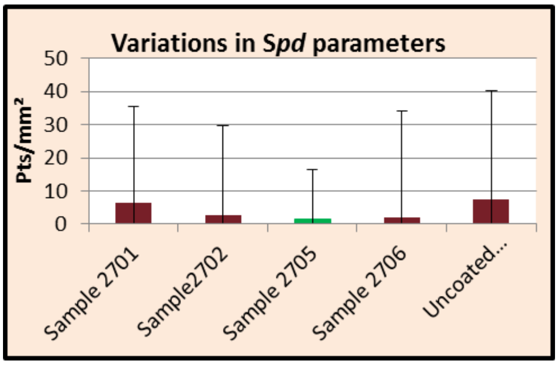


Figure 6 Variation in density of peaks

A dale is defined as a region around a pit such that all maximal downward paths end at the pit (ISO 25178-2:2012 (E)).

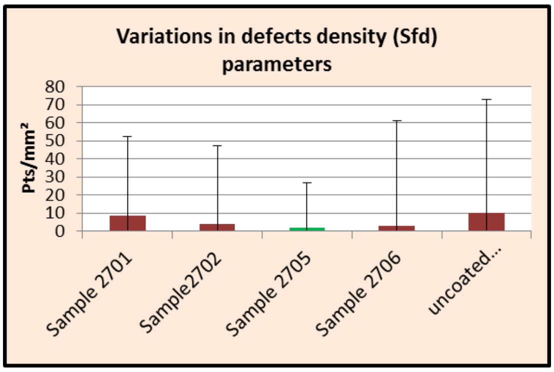


Figure 7 Variation in density of dales

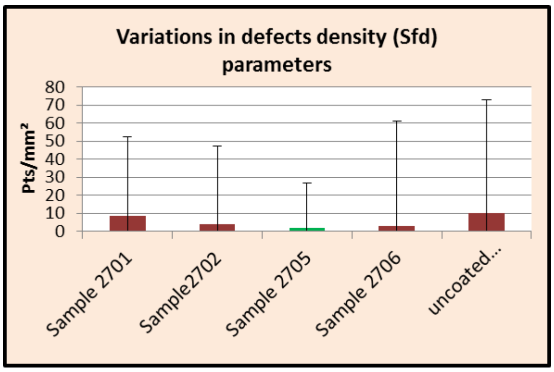


Figure 8 Variation in significant feature density (peaks + dales)

Although sample 2705 showed the highest WVTR it showed in all 3 measurement studies the lowest density of peaks, pits and significant features when taking all visible features into account.

Following these findings, a visual survey of the samples with the highest and lowest WVTR was completed. The surface was fully visually inspected and only those defects which satisfied the criteria of only large defects (6σ (Sq=0.8nm) height & width > 15um were measured and recorded.

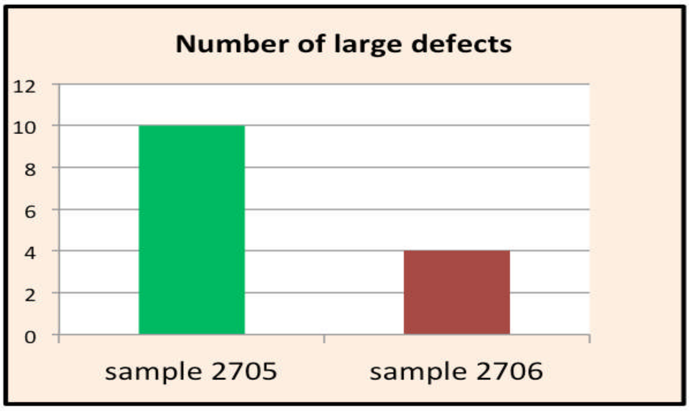


Figure 9 Comparison in large defect concentration

Sample 7205 (highest WVTR) showed a much higher number of these large, significant features than that of sample 7207 which had the lowest WVTR. (figure 9), this exercise will be repeated to ensure statistical significance in the results with a further batch of samples.

4 Conclusion

Optical Interferometry is an effective method for measurement of barrier layers in the development and assessment of flexible PV modules. Changes and irregularities in surface topography can be a useful predictor of performance of the barrier layer to gas permeation and water vapour transmission. The initial work has shown that there are methods for effective discrimination of significant and none significant features.

The findings show that small numbers of large defects may have more of an influence on function that large numbers of smaller defects. Further work is required to establish this hypothesis as statistically significant.

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References

[1] Jiang, X., Scott, P. J., Whitehouse, D. J. and Blunt, L. (2007). 'Paradigm shifts in surface metrology.' Part II. Proceedings of the Royal Society A. 463 (2), pp. 2071-2099.

[2] International Standard (2012). ISO 25178-2:2012(E), Terms, definitions and surface texture parameters. Switzerland. 1-47.

[3] Duncan, B. Urquhart, J and Roberts, S. (2005). Review of Measurement and Modelling of Permeation and Diffusion in Polymers. NPL Report DEPCR 012