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Metrology of Al$_2$O$_3$ Barrier Film for Flexible CIGS Solar Cells

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ABSTRACT

Flexible Cu (In, Ga) Se$_2$ (CIGS) solar cells are very attractive renewable energy sources because of their high conversion efficiencies, their low cost potential and their many application possibilities. However, they are at present highly susceptible to long term environmental degradation as a result of water vapor ingress through the protective encapsulation layer to the absorber (CIGS) layer. The basic methodology to prevent the water vapor permeation is to combine an oxide layer (e.g. AlO$_x$) coating with suitable polymer substrates. Nevertheless, micro and nano-scale defects can appear at any stage of the coating process thus affecting the module efficiency and lifespan. The main aim of this research paper is to use surface metrology techniques including: White Light Scanning Interferometry (WLSI), Atomic Force Microscopy (AFM) and Environmental Scanning Electron Microscopy (ESEM) to characterise the aluminum oxide (Al$_2$O$_3$) barrier film defects, which appear to be directly responsible for the water vapor permeability. This paper reports on the development of a characterisation method for defect detection based on "Wolf Pruning" method and then correlates this with measured water vapor transmission rates (WVTRs) using standard MOCON® test. The results presented in this paper provided a detailed knowledge of the nature of micro and nano-scale defects on the Al$_2$O$_3$ barrier films which are responsible for water vapor and oxygen ingress. This result can then be used to provide the basis for developing roll-to-roll in process metrology devices for quality control of flexible PV module manufacture.
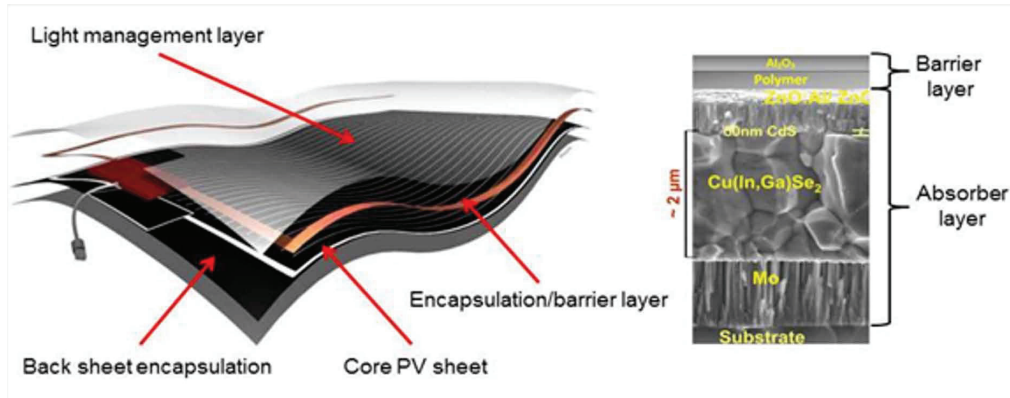
Keywords: ALD, Aluminum Oxide, Photovoltaic, Surface Defects, WVTR

1. INTRODUCTION

In today's industry, the most common type of solar photovoltaic (PV) cell is fabricated from either crystalline silicon or thin-film materials (P. F. Carcia, R. S. McLean, & S. Hegedus, 2010).

Figure 1. Schematic of the flexible PV module (Courtesy of Flisom, Switzerland)

The rigid construction of Si solar cells hampers their economic integration into residential and commercial buildings; however, thin film solar cell technologies may prove to be most appropriate with respect to cost and ease of manufacture, and it is anticipated that the next generation of photovoltaic devices will be based entirely on thin film technologies. These cells are based on the material $CuIn_{1-x}Ga_xSe_2$ (CIGS) as the absorber layer (p-type) and they are the most efficient cells at present (Igalson & Urbaniak, 2005). The key weakness of these cells is their moisture sensitivity. This is critical problem if this technology is to meet the requirements of international standard IEC61646, which requires that all PV modules survive 1000 hours in an environment of 85 C° and 85% relative humidity(IEC61646-2, 2008). Cost effective encapsulation facilitating stable outdoor performance for more than 20 years is still a challenge. The only cost-effective encapsulation possibility for long term stability available at the moment is to use rigid glass, where all benefits of flexibility and lightweight disappear (BrÃ, 2009). Therefore, a robust, transparent flexible encapsulation method for CIGS PV cells is needed. Meeting these requirements is a major concern for the manufacture of thin film CIGS cells.

1.1. Flexible PV Modules

Flexible solar modules comprise four functional layer groupings as shown in Figure 1. The main focus of the investigation in this paper is the barrier layer which is incorporated in the encapsulation layers. This layer is typically formed from a planarised Polyethylene Naphthalate (PEN) sheet with an amorphous Al_2O_3 barrier coating < 50 nm thick. The Al_2O_3 barrier coating is produced by Atomic Layer Deposition (ALD). This technique is a controlled layer-by-layer deposition that enables the deposition of thin, smooth, and highly conformal films with atomic layer precision and typical thickness ranges between 1 and 100 nm (S. M. George, 2009).

2. POLYETHYLENE-NAPHTHALATE AND ALUMINUM-OXIDE PROPERTIES

Polymer materials such as polyethylene naphthalate (PEN) consist of long straight-chain polymer molecules with weak chemical interaction between the chains. The molecular building block for PEN chains is shown in Figure 2. Therefore, small molecules such as water vapor and oxygen can diffuse around the material chains. Although polymers have strong covalent bonds (short range)

Figure 2. Structure of Polyethylene Naphthalate -PEN (Hansen, Myers, & Osakada, 1998)
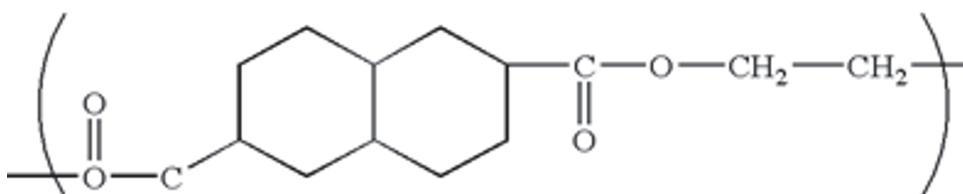
along the chains, they have weaker (long range) bonding between chains (Ayache, Beaunier, Boumendil, Ehret, & Laub, 2010).

The spaces between chains where there are no atoms make it easy for gas molecules to dissolve and diffuse in the PEN layer. In addition, these spaces make the density of the polymer relatively low. In contrast 'ceramic' materials such as AlO_x and SiO_x are crystalline lattices with strong inter-atomic and inter-molecular bonds (Kailas, 2006). The bonding between atoms in ceramics is a combination of short range ionic and covalent bonding. Even for amorphous ceramics there is a high instance of short range ordering in three dimensions, so there are few spaces between atoms for gas molecules to dissolve and diffuse, see Figure 3. The tight packing makes for high density, depending of course on the atomic weights of the constituent atoms. Although in an amorphous ceramic material, such as Al_2O_3, the ordering of atoms over a long range is lower than for the crystalline material (a less regular lattice structure) (Carter & Norton, 2007), the ALD AlOx still to be an effective barrier material to protect CIGS PV modules from water vapor and oxygen ingress.

2.1. Al_2O_3 ALD Barrier Film

Atomic layer deposition relies on sequential, self-limiting surface reactions and allows atomic level thickness control of deposition (S. George, Ott, & Klaus, 1996). Due to its unique characteristics, ALD has been used in this work as a diffusion barrier film. Al_2O_3 ALD thin films with only 25 nm thickness are able to reduce the water vapor transmission rate to 10^{-3} g/m²/day (Groner, George, McLean, & Carcia, 2006; Langereis, Creatore, Heil, Van de Sanden, & Kessels, 2006) or even lower (Carcia, McLean, Reilly, Groner, & George, 2006). However, the characterization and spatial density of defects that may occur during the deposition process have never been reported for Al_2O_3 ALD films.

Park et al. (2005) reported a water vapour transmission rate of 0.03 g/m²/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 Polyethersulfone (PES) substrate, whereas Carcia, et al. (2006) showed that 25 nm thick Al_2O_3 barrier films on poly (ethylene naphthalene) substrates can have a water vapour transmission rate of less than $1*10^{-5}$ g/m²/day.

Moreover, studies regarding the encapsulation technologies using Al_2O_3 ALD for CIGS PV modules have been carried out by (P. Carcia, R. McLean, & S. Hegedus, 2010), the authors compared the moisture sensitivity of CIGS cells protected by a 55 nm thick Al_2O_3 film, deposited by an ALD technique, with equivalent CIGS cells protected with a glass layer, and with an uncoated Polyethylene Terephthalate (PET) film. The study lasted for more than 1000 hours at 85 C° and 85% RH, with simulated solar illumination. The result indicated that, the CIGS cell protected with the PET layer lost about half its efficiency (12.5% → 6.6%) after ageing for 1020 h (42.5 days) at 85 C° and 85% RH, whereas the CIGS cell protected with the 55 nm ALD Al_2O_3 bar-

Figure 3. Structure of Al$_2$O$_3$ material
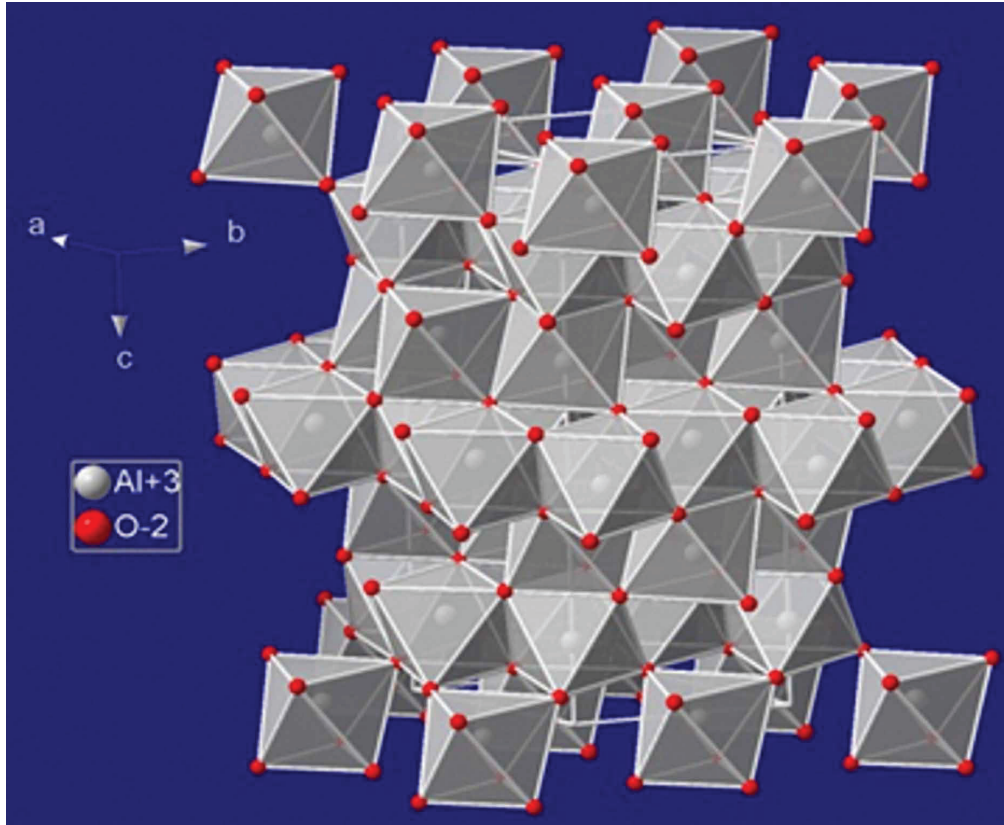

rier film and the cell with a glass layer showed only small net change (< 3%) in efficiency. This remaining degradation in efficiency is considered to be due to the presence of small defects on the barrier film. Therefore, quantitative information about key defects is required to fully specify the new inspection systems and identify cases where very high spatial resolution is required. This paper seeks to provide detailed knowledge of the nature of micro and nano-scale defects on the Al$_2$O$_3$ barrier film with respect to the water vapour transmission. The defect nature is analysed largely from lab-based high resolution measurements on small samples taken from the barrier.

2.2. Flexible PV Encapsulation Technology

Flexible encapsulants consist of multi-layer combinations of polymer and inorganic dielectric coating layers such as Al$_2$O$_3$. However, effective coating process is essential to reduce defects during roll-to-roll fabrication of flexible PV barrier films (see Figure 4).

At the present time, no cost effective, flexible transparent encapsulation products fulfill the PV modules requirements. The water-vapor transmission rate (WVTR) of present PV modules is in the range of 10^{-1} g/m^2/day, while it should not be higher than 10^{-4} g/m^2/day to assure life-times of 20 years and more (Fraunhofer ISC, 2004; Kempe, 2005). Several companies are currently working on this problem, but they still have difficulties to optimise roll-to-roll barrier layers manufacture.

Figure 4. Roll to Roll technology (Galagan & Andriessen, 2012)
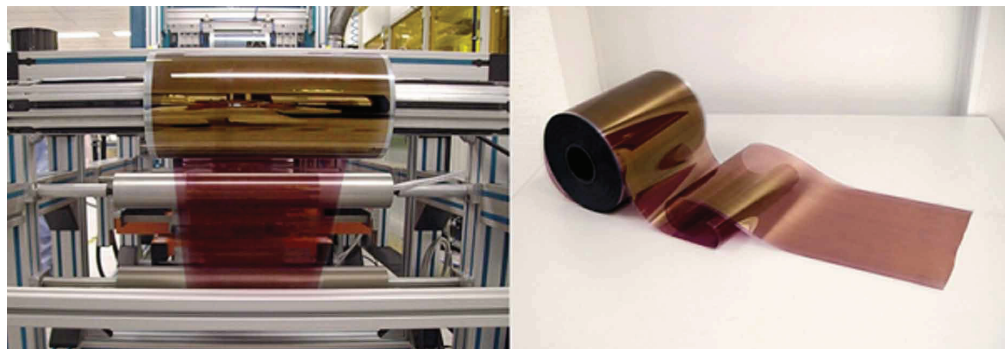
3. EXPERIMENTAL WORK (AL$_2$O$_3$ ALD SAMPLES PREPARATION)

In this study six barrier substrate samples were assessed after they were pre prepared under different conditions before ALD coating. The samples had an 80 mm diameter, and were coated with 40 nm amorphous ALD Al$_2$O$_3$. The samples were prepared in a clean room under conditions shown in Table 1. The pre-coating procedures for the present samples were varied in order to investigate the effect of the substrates cleanliness on WVTR.

3.1. ALD Deposition

The ALD depositions of this study were made using Oxford Instruments FlexAL tool. Trimethyl Aluminum (TMA) was used as the metal precursor. The reactor temperature used was 120 C° and the pressure was very low (<0.1mBar). 312 reaction cycles were made to produce 40nm Al$_2$O$_3$ layers on the PEN substrate. Figure 5 shows a typical Al$_2$O$_3$ ALD process.
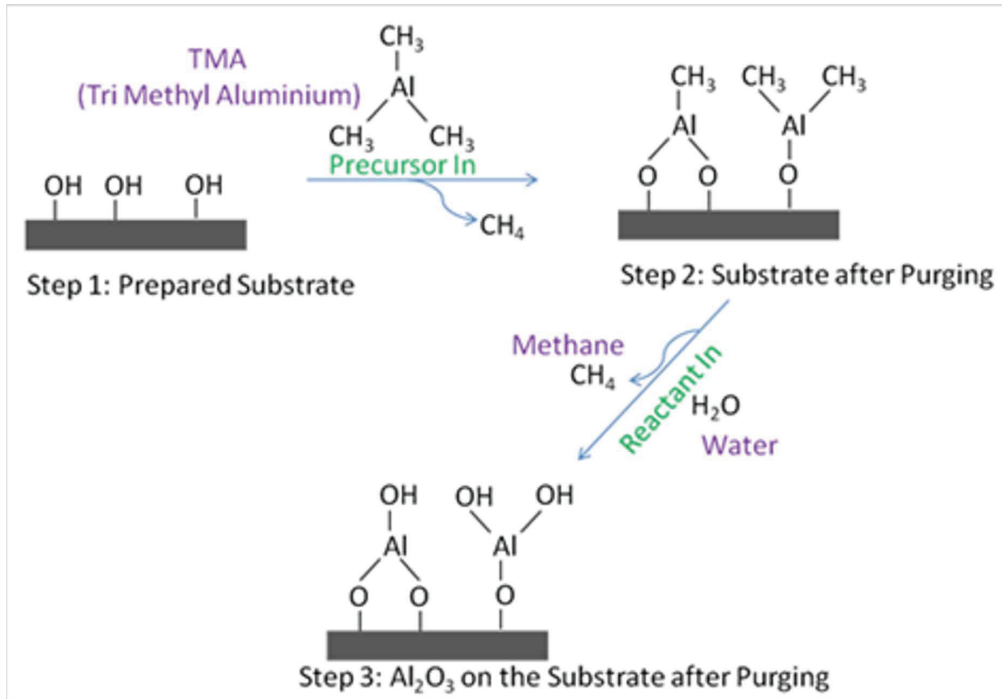
3.2. Water Vapour Transmission Rate Assessments

Since no thin film deposition technique and process are perfect, defect information such as their densities, sizes and locations in the Al$_2$O$_3$ ALD thin films are in high demand to evaluate the quality of the films and optimise the deposition process and PV system functional lifespan. The defect information is especially important for diffusion barrier applications. For example, in the gas diffusion barrier application, the measurable permeability is attributed to the defects in the barrier films that allow the leakage of reactive species (Graff, Williford, & Burrows, 2004;

Table 1. Test samples and pre-coating conditions

Sample No	Conditions
1 2	Polymer surface unprotected before loading for ALD (practice 1).
3 4	Polymer surface protected to the last moment before loading into ALD coater. However, some visible scratches were reported on sample 3 (Practice 2).
5 6	Contact cleaning of the polymer before ALD (Practice 3).

Figure 5. Shows a typical Al_2O_3 ALD process (S. M. George, Yoon, & Dameron, 2009)

Maiola, 2000). In this study, prior to the surface measurements, the Al_2O_3 ALD samples were measured for water vapour transmission rate (WVTR) using Isostatic standard test instrumentation (MOCON®) at specified conditions (38C° and 90% RH respectively). This method involves the test specimen being held in a sealed chamber such that it separates two sides of the chamber as shown in the Figure 6.

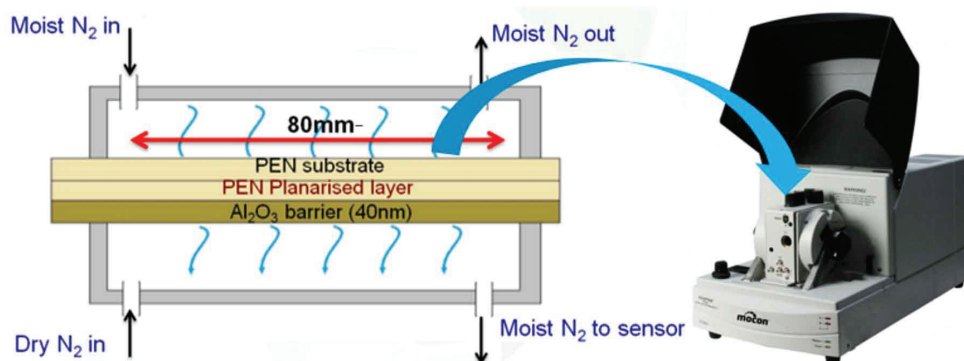*Figure 6. Schematic representation of WVTR test (Maiola, 2000)*

Table 2. WVTR test results

Sample No.	AlO$_x$ Thickness	WVTR (g/m^2/24 hrs.)
1	40 nm	5×10^{-4}
2	40 nm	Below detectable level
3	40 nm	1×10^{-3}
4	40 nm	Below detectable level
5	40 nm	6×10^{-4}
6	40 nm	Below detectable level

On one side of the sample a highly saturated wet environment is maintained at a constant temperature, whilst on the other, a dry nitrogen carrier gas is passed to a coulometric phosphorous pentoxide sensor where water vapour which enters is converted to a measurable charge (Wennerberg, 2002).

3.2.1. WVTR Results

The WVTR for each sample can be seen in Table 2. There were two distinct levels of WVTR. These were classified as over and below the detectable level of the MOCON instrument.

The WVTR results indicated that sample (3) has a higher WVTR value than the other samples. The study's hypothesis is that the presence of significant micro/Nano- scale defects might play a critical role in determining WVTR.

4. DEFECT DETECTION METHODOLOGY

The results of the WVTR test (MOCON®) for each sample were initially concealed so all metrology was conducted blind with no knowledge of the WVTR values. Following to that, three key measurement techniques were employed to characterise the samples' surfaces at different scales of measurements, in order to capture all the potential features which might be classified as "significant defects". These techniques were Environmental Scanning Electron Microscopy, Atomic Force Microscopy and White Light Scanning Interferometry.

4.1. Scanning Electron Microscopy Analysis

A field emission gun environmental scanning electron microscope (FEG-ESEM) Quanta 250 was used for imaging the Al_2O_3 barrier layers. When performing secondary electron imaging, the maximum spatial resolution of the instrument in the environmental mode was approximately 1.4 nm at 30 keV. To investigate the Al_2O_3/polymer defects, the low vacuum mode was employed using water vapour at pressures between 120 and 400 Pa. An off-axis large field detector was used to collect the amplified secondary electron signal emitted from the specimen; this detector has the advantage of providing images with a relatively large field-of-view. An image of a typical 'hole' defect, approximately 4 μm in diameter, in the Al_2O_3 layer is shown in Figure 7a. A region of differing contrast is observed in the area surrounding this hole; this is attributed to the delamination of the Al_2O_3 layer from the underlying polymer structure due to water vapor ingress. Figure 7b shows a particle type defect was captured in the Al_2O_3/polymer structure using

Figure 7. ESEM of defects in Al_2O_3 barrier layer (a) typical hole type defect (b) particulate type defect
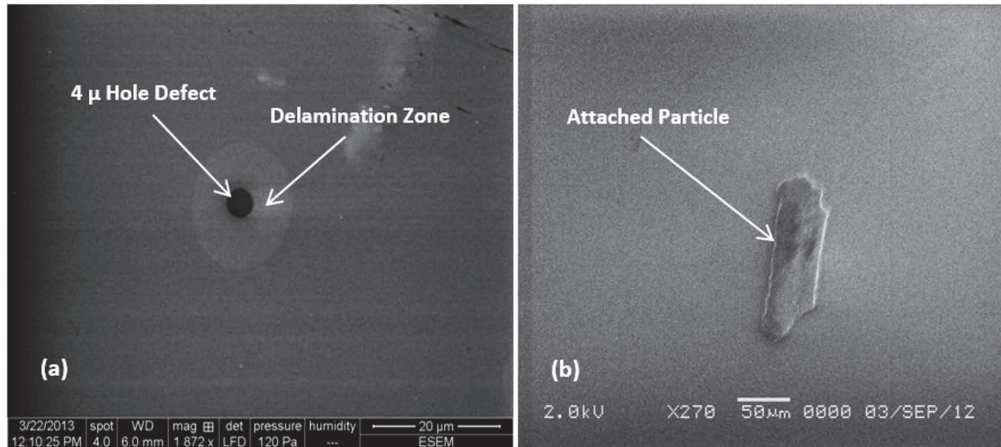
a JEOL 6060 LV scanning electron microscope operated in high vacuum mode. The image shows a particulate type defect of approximately 50 μm lateral dimension on the surface.

## 4.2. Atomic Force Microscopy Analysis

Although the object targeting and the image acquisition can be more rapid using the ESEM, aluminum oxide is difficult to examine as it has a tendency to rapidly charge, resulting in beam divergence and image degradation. In contrast, AFM can analyse the samples with minimal sample preparation, thus preserving surface textures and allowing repeat analyses without the risk of charging (Bottomley, 2012). The AFM can produce an equal or higher data resolution image of an equivalent area, but with the addition of absolute X, Y and Z for any features. In this study, a Bruker's Dimension icon® Atomic Force Microscope (AFM) was used as an independent measure of the Al_2O_3 ALD film. The technique allowed much smaller defect sizes to be examined than would be possible by the ESEM. It has been also used to determine the size of the peaks (particles) over the films which were not detectable by the ESEM or the conventional optical microscopy technique. Figure 8 shows peaks type defect captured by this technique.

4.3. White Light Scanning Interferometry Analysis

In a previous published study (Blunt, Elrawemi, Fleming, & Sweeney, 2013), white light scanning interferometry (Ametek Taylor Hobson) Coherence Correlation Interferometer (CCI) 6000 was used to conduct 3D surface analysis providing a number of important areal parameters. Based on the results of the study, it was concluded that small numbers of large defects that satisfy the criteria of measuring 6 σ (Sq = 0.6 nm) in height and 180 μm^2 in area, where Sq is the standard deviation of the sample (ISO25178-2, 2012), were the dominant factors in predicting WVTR. However, this technique showed its limitation of the measured area when using an X20 objective lens. This lens can measure an area of 1 mm^2. 100% of the overall area was measured on each sample using the CCI instrument with an X20 magnification lens.

The method of 'Wolf pruning'(ISO25178-2, 2012) is then utilised to perform topographic segmentation analysis. This method provides a reliable approach for extracting the features of

Figure 8. AFM image-peaks type defect
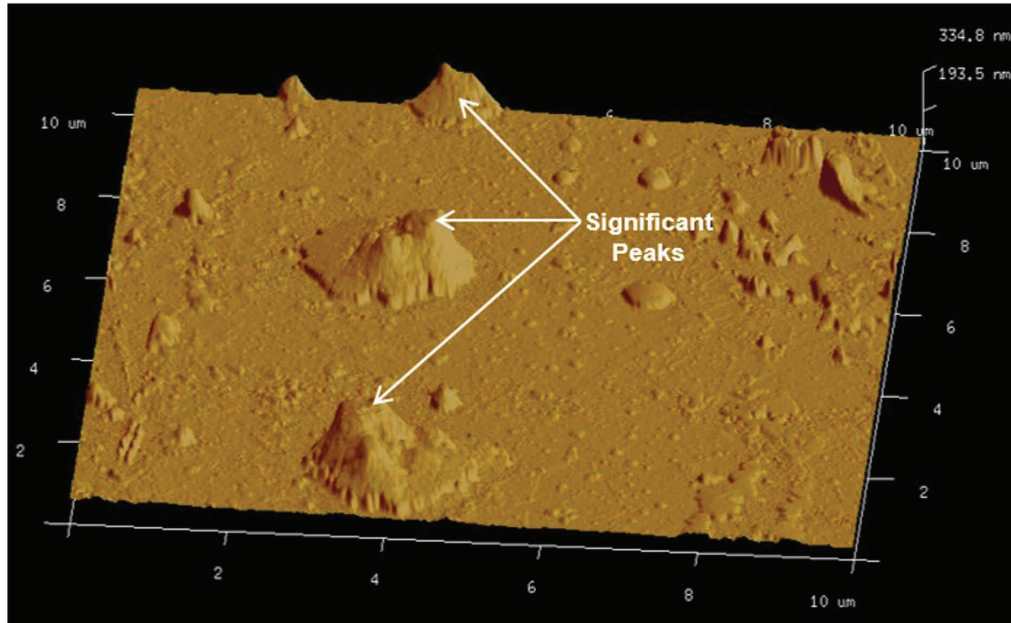
functional interest by accurately excluding insignificant geometrical features that are induced, such as measurement noise and errors. The procedure consists of finding the surface peaks and dales with the smallest height difference and combining them with the adjacent saddle point, where the cut-off can be selected to prune only insignificant peaks and dales. The technique is then used to delineate the large defects and record their presence. The most observed 'significant' defects which are postulated to be responsible for the high WVTR values are classified and published in previous studies (Elrawemi, Blunt, & Fleming, 2013).

4.4. Surface Topography Analysis

The surface roughness average of a defect free sample was measured to be (Sa ~ 0.6 nm), see Figure 9. Based on that, the Wolf Pruning method was conducted with prune conditions set for each sample in order to isolate only the largest defects. The criteria of being defined as a large defect was 6σ where ($6\sigma = \pm 3Sq = \pm 3 \times 0.8$nm height, and 15 µm width). Effective discrimination of significant and non-significant defects was recorded.

5. RESULTS DISCUSSION

The results in Figure 10 presenting significant defects count as analysed by the 'wolf pruning' method. Comparing each two sets of the samples results, feature segmentation analysis and the pruning condition applied to the data have provided a clear evidence for the correlation of surface defects size, defect density, and the transmission of water vapour through the barrier coating layers. The investigation seems to indicate that large defects of more than ($\pm 3Sq$ height, and 15µm width) are the dominant factor for determining the WVTR.

Figure 9. Non-defective sample (Sa = 0.6nm)
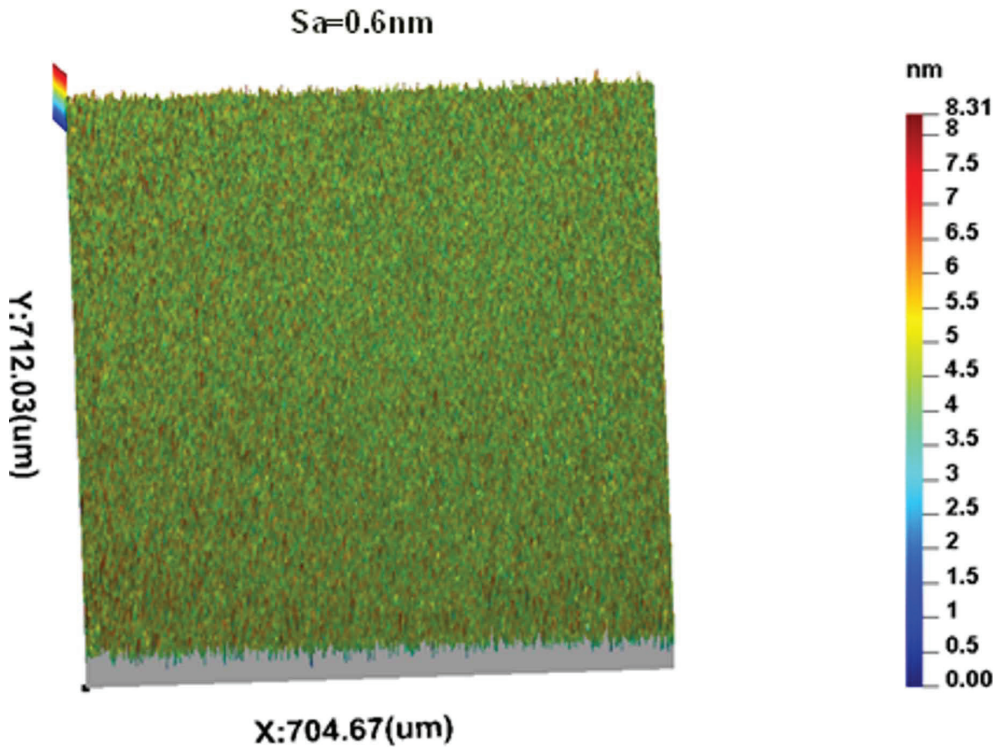
## 6. PRE-COATING PROCESS EFFECT

The results in Figure 10 has demonstrated those samples (1 and 2) which were deliberately left exposed in a clean room environment over night show higher numbers of defects, but the MO-CON tests show a low WVTR indicating that the films have collected particles, but the particles are not a significant impact on the WVTR.

Sample 4 has less large defects recorded than the other samples. This is believed to be attributed to the nature of sample handling conditions. The procedure for sample handling and purging/cleaning of the ALD coating equipment was optimised for this sample. This ensured few or even no particles were present on the surface prior to the ALD process. Therefore, the WVTR value was very low (see section 3.2.1). In contrast to this, sample 3 prepared with the same conditions as sample 4 demonstrated a lower large defect count than the other samples but still had the highest WVTR value $\approx 1 \times 10^{-3}$ g/m^2/day. This sample has larger defects than the other investigated samples (typical example is shown in Figure 11), which may have had a negative effect on the barrier properties thus giving an increase in the WVTR.

Lastly, sample (5 and 6) show evidence of more particles and scratches, the MOCON tests show a low WVTR, indicating that the web-roller used to clean the substrates before the ALD process, may increase the WVTR by causing scratches.

The investigation in this research paper seems to concur with previous published literature (Zhang et al., 2009), individual defects or pinholes are generally caused by particulate contami-

Figure 10. The significant defects count
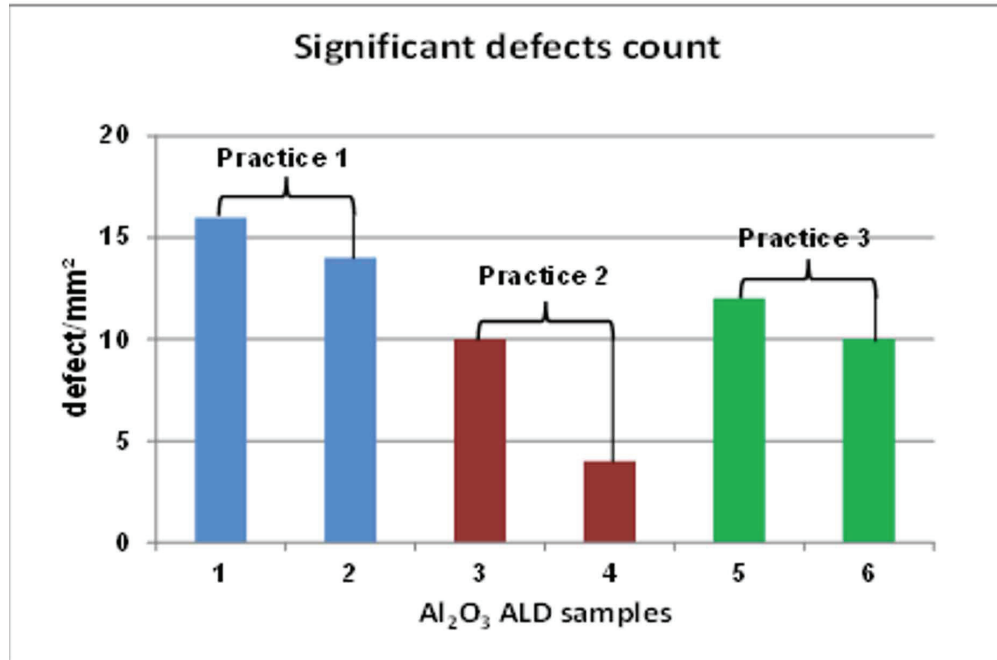
nation or the surface roughness of the substrate. Individual film defects are believed to be the critical features limiting the performance of barriers. These defects must be controlled to assure high barrier quality and efficient barrier manufacturing. The authors also note that these defects could be observed by (FE-SEM). However, the location and density likely result from particle contamination on the PEN substrate. The particles are believed to mask the polymer substrate and prevent effective Al_2O_3 ALD coating. The particles then move or are dislodged after the Al_2O_3 ALD coating process and leave an uncoated region of the polymer substrate resulting in high

Figure 11. A typical example of large defects
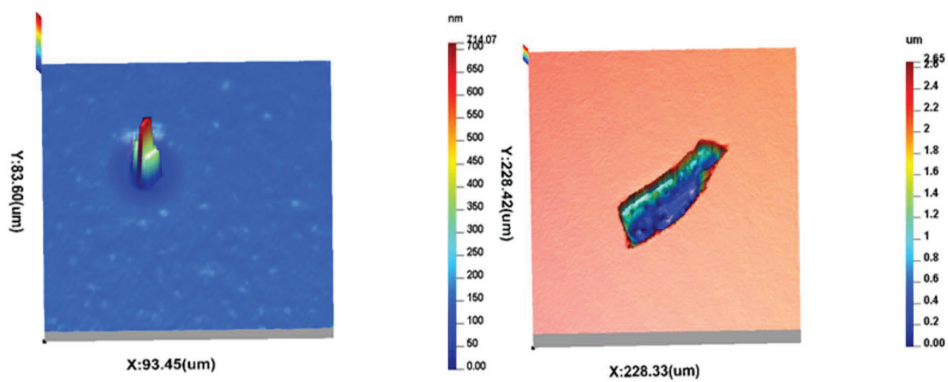
water vapor ingress. Hence, to achieve low WVTR value, longer lifespan and best efficiency the following criteria are recommended to be followed when preparing the polymer layer for ALD coating.

- Contamination must be avoided as much as possible (practice 1 not recommended).
- Cleaning has limited effect. Damage is very detrimental to WVTR so it must be avoided.
- Limiting atmospheric exposure ensures best WVTR results (Practice 2 recommended).

To summarise, this study gave a good insight into the best practice to be used when preparing the samples for Al_2O_3 ALD coating process, and the type of defects which has an effect on the WVTR value.

7. CONCLUSION

The Al_2O_3 barrier film is known to improve PV lifespan due to reduction in WVTR. This improvement can be seriously affected and potentially reduced when defects in this barrier film are present. In this study, surface metrology techniques have provided the ability to measure and effectively characterise these defects. The obtained results provide new information that enables automatic defect detection methods to be developed. Information has also been provided on what type of defects will impede PV performance and lifespan. This result provides important information which will be in valuable in the future development of an automatic in-line defect detection system. Work still continues to develop the optimal prune conditions which will help to determine the defects which have a significant effect on the WVTR.

ACKNOWLEDGMENT

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Liam Blunt has an honours degree in Materials Technology and a PhD in "The Metallurgy of Centreless Ground Surfaces" at Coventry University. He has formed extensive industrial collaborations in particular with Taylor Hobson Ltd a world leading metrology company. He is now the Director of the Centre for Precision Technologies. His research specialises in the applications of surface metrology to new production processes.

Leigh Fleming is a Senior Lecturer and researcher in The School of Computing and Engineering at The University of Huddersfield, her research is mainly focussed on the application of surface metrology to novel situations with an emphasis on improvements in engineering function and performance.

Francis Sweeney is a Post-doctoral researcher in the school of computing and engineering at the University of Huddersfield. He gained his PhD from the University of Strathclyde in 2005. In 2007 he moved to the University of Sheffield where he was involved in the development of electron ptychography in the Transmission Electron Microscopy. Currently, the main focus of his research at Huddersfield is the characterisation of the structure and morphology of materials by electron microscopy and associated techniques.

David Robbins from 1973 to 2004 was leader of research in thin film materials and devices for electronics at the UK Ministry of Defence laboratories and at QinetiQ in Malvern. During 1981-1982 he was seconded as Visiting Scientist at IBM, Yorktown Heights. In 2004 Dr Robbins was appointed Chief Technology Officer for Cenamps in Newcastle-upon-Tyne, and from 2008 he led research in Atomic Layer Deposition (ALD) at Centre for Process Industries (CPI). He is currently a consultant to CPI on ALD barrier layer technology, a Fellow of the Institute of Physics and has published over 150 papers and patents.

David Bird has 11 year experience working with film substrates and barrier materials. David completed an Engineering Doctorate in 2008 sponsored by DuPont Teijin Films on nano-scale coatings on films for flexible electronics. He is now a Senior Scientist at the UK's National Printable Electronics Centre, focussing on vacuum-deposition of barrier materials. David has 21 IP submissions (for Patent, Publication or internal Know-how), 15 of which relate to Barrier Materials and their integration to functional devices.