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IN-LINE METROLOGY FOR DEFECT ASSESSMENT ON LARGE AREA ROLL TO ROLL SUBSTRATES

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Abstract:
This paper reports on work carried out as part of the EU funded research project "Nanomend". The project seeks to develop integrated process inspection, cleaning, repair for nano-scale thin films on large area substrates. In order to prevent water ingress into flexible PV modules they are coated with a protective barrier layer of Al_2O_3 using atomic layer deposition (ALD) technique. Unfortunately defects in this layer have been shown to reduce module efficiency over a period of time due to water vapour ingress. The present work concentrates on defect detection and reports on the use of areal surface metrology parameters to correlate defect morphology with water vapour transmission rate (WVTR) through the protective barrier coatings. The use of advanced segmentation techniques is demonstrated where topographic information on functionally significant defects can be extracted and quantified. The work also reports on the deployment of new in line interferometric optical sensors designed to measure and catalogue the defect distribution and size where they are present in the barrier film. The sensors have built-in environmental vibration compensation and are being deployed on a demonstrator system at a Roll2Roll production facility in the UK.

Keywords: Roll2Roll, surface metrology, defects, flexible photovoltaics

1. INTRODUCTION

Flexible photovoltaic (PV) films based on CIGS (Copper Indium Gallium Selenide $CuIn_xGa_{(1-x)}Se_2$) have been reported to have light energy conversion efficiencies as high as 19% [1]. Their adoption has many advantages in terms of applications and in particular building integration. These CIGS based multi-layer flexible devices are fabricated on polymer substrates by the repeated deposition, and patterning, of thin layer materials using roll-to-roll processes (R2R). The whole film is approximately 3µm thick prior to final encapsulation. The resultant films are lightweight and flexible, however wide scale implementation is hampered by long term environmental degradation of efficiency due to water vapor ingress to the CIGS modules through the polymer layers causing electrical shorts and corresponding efficiency drops and, ultimately failure.

One of the most effective methods to protect the CIGS cells is to apply a barrier coating of Al_2O_3 to the encapsulation material. The highly conformal Al_2O_3 barrier layer is produced by the atomic layer deposition (ALD) technique [2]. The surface of the encapsulation substrate polymer (PEN) film must be smooth; in order to achieve a high quality deposition, hence the substrate film is further planarised. Nevertheless this ALD barrier is not at present fully effective; water vapour can still permeate through the barrier due to the presence of micro and nano-scale size defects in the barrier films. This paper reports the results of measurements conducted to characterise 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. A robust interferometric technology is introduced which allows in-process measurement during R2R manufacture. The results presented within this paper provide the basis for the development of R2R in process metrology devices for defect detection. The specimens used represent substrates prepared under dissimilar levels of cleanliness and hence represent cases where differing levels of defects should be present. Table 1.

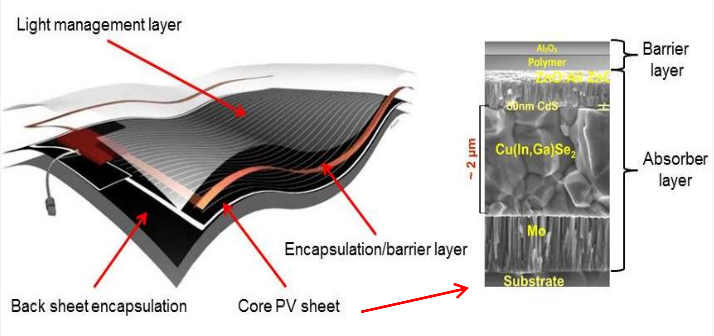


Fig 1: Functional layers of Flexible PV

2. WATER VAPOUR TRANSMISSION (WVTR)

2.1 WVTR Measurements

All barrier coated samples were measured for water vapor transmission rate (WVTR) using standard MOCON test instrumentation prior to the surface measurement. With this method, the test specimen is held within the instrument such that it separates into two sides of a test chamber. One side, the "wet side", is exposed to the gas or vapour to be measured. On the detector or the "dry side", the sample is subjected to zero relative humidity. 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 [3]. The steady state rate was recorded along with the time to stable transmission. The area exposed to the water vapour was approximately 80mm. The WVTR measurement for the tested substrates are shown in Table 2.

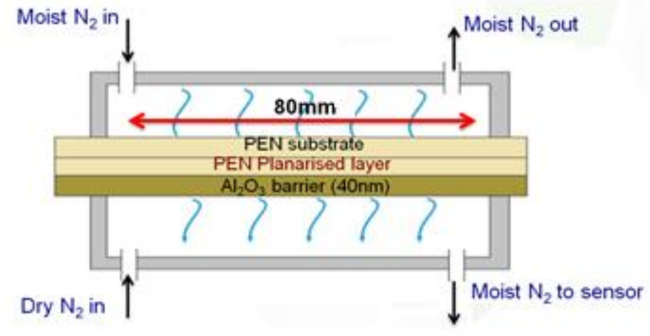


Fig 2: WVTR Measurement using MOCON method, exposed area 80mm.

Table 1 shows the WVTR for substrates prepared with differing levels of cleanliness.

Sample	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. Some visible scratches were reported on S3 (Practice 2).
5 6	Contact cleaning of the polymer before ALD (Practice 3).

Table. 2 WVTR test results

Sample No	AlO$_x$ thickness	WVTR (g/m²/24 hrs.)
1	40 nm	5×10^{-4}
2	40 nm	$< 5\times10^{-4}$
3	40 nm	1×10^{-3}
4	40 nm	$< 5\times10^{-4}$ level
5	40 nm	6×10^{-4}
6	40 nm	$<5\times10^{-4}$

3. SURFACE TOPOGRAPHY ANALYSIS

All surfaces were measured after WVTR testing using a combination of laboratory based coherence correlation interferometry (CCI) , SEM and AFM. A classification system previously reported [4] was used to classify the types of measured defect, Fig 3.

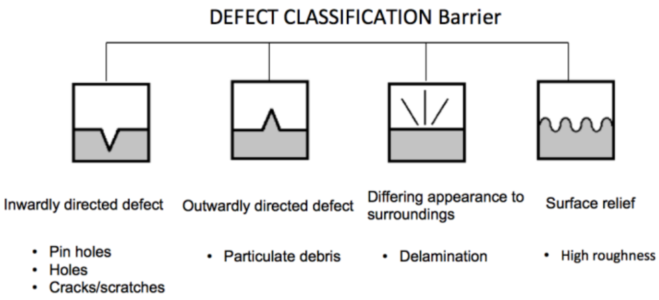


Fig: 3 defect Classification System

Figure 4a shows and examples of a relatively large pit like feature in the Al_2O_3 barrier. A typical peak type feature is shown in Figure 4b.

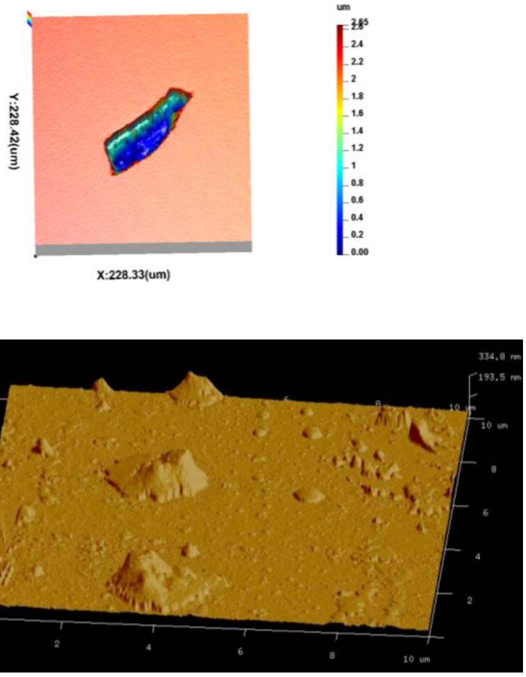


Figure 4 a) pit like feature in barrier largest later dimension 100um (interferometry) b) Peak feature approximately 3um width (AFM)

Despite being prepared under differing condition the average surface roughness values Sa, showed little difference between the substrates, mean 1.24. and standard deviation of 0.17nm As no clear difference between the samples was evident the substrates were subjected to segmentation analysis differences were noted between the samples.

Automated segmentation has been utilised in the present case, to distinguish between those features that are functionality significant from those which are non-functionally significant. Segmentation analysis allied to analysing large amounts of function related experimental data [5] provides a powerful tool for functional discrimination of surfaces.

3.1 Feature segmentation analysis

Wolf pruning, as defined in ISO 25178-2 [6], allows the detection of significant features on the barrier surfaces and their characterization in terms of dimension, area, volume, shape or morphology. Wolf pruning at different thresholds, produces different counts of the number of significant features, in this case pits + peaks. The present study is based upon the supposition that defects above a certain scale determine the water vapour transmission through the barrier coatings. To this end feature segmentation analysis [6]was implemented in order to separate the significant from non-significant surface topography features. Feature parameters are not specifically defined by an equation, but rather use a toolbox of pattern recognition techniques. The characterisation consisted of five steps; (1) selection of type of texture feature, (2) segmentation, (3) determine the significant features, (4) selection of feature attributes, and (5) quantification of feature attributes statistics. The segmentation was applied by means of an “iterative” process. The protocol used for characterising the barrier films was as follows. Firstly, 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 the smoothing process, edge processing was performed on the data using a Sobel type operator[5]. The edge data is then “pruned” by means of Wolf pruning [6]where all data elements below 1% of the Sz (of the edge filtered surface) value are deemed insignificant, and those elements higher than 1% Sz (of the edge filtered surface) were retained as significant. Following Wolf pruning an area prune was applied where if an area was found to be $\leq$ 5um lateral diameter (this area being based on optical and SEM analysis) it was deemed insignificant. Figure 5 shows an example “captured” defects following the segmentation process. The figure shows the power of the segmentation procedure for extracting defects from the surface data.

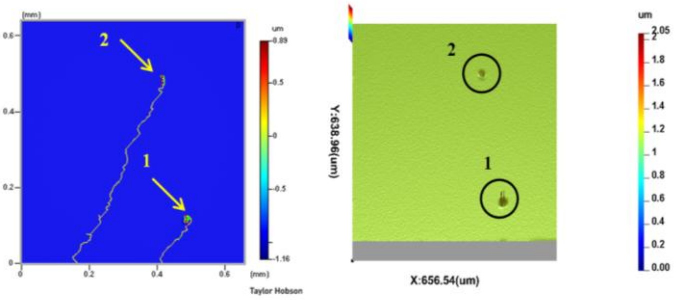


Fig 5: Segmentation analysis results for 2 significant defects based on measured data (right).

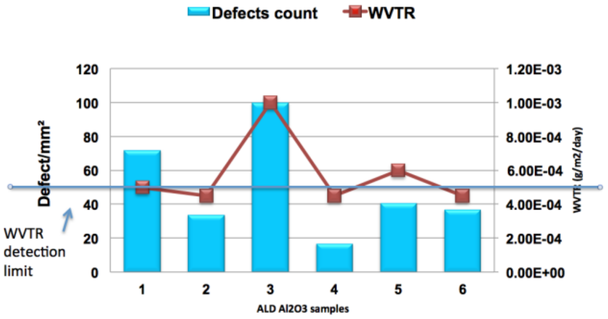


Fig 6: Defects density versus WVTR values

Following the implementation of the segmentation process the defect density for all the substrates under test was calculated. The results were then compared to the measured WVTR values, Fig 6.

The results show that for each pair of surfaces corresponding to a differing pre processing of the polymer prior to coating that, the sample with the highest WVTR values (sample 3) corresponds to the sample with the highest defect density. The samples with the lowest WVTR value (2,4,6) shows the lowest defects density. Sample 4 had the least opportunity for contamination and this is reflected in the defect density and the WVTR results. Finally, it is interesting to note that where visible large scratches were reported (sample 3) the highest defect density and WVTR occurred.

The results concur with previously reported work of the present authors that seems to show that for the Al_2O_3 ALD barrier coating a small number of $\geq$ 5um lateral diameter large defects dominates the WVTR and thus these defects should be the focus of any detection system[[7]

4. IN PROCESS SURFACE METROLOGY

To facilitate in process measurement of R2R substrates two challenges need to be addressed; i) the measurement must be fast and non-contact ii) the measurement must be carried out in a “noisy” working environment. With the general surface topography, Sa, of the substrates being of the order of $\leq$ 2nm the only feasible measurement solution is optical interferometry.

Unfortunately interferometric measurement techniques are extremely sensitive to environmental noise such as mechanical vibration, air turbulence and temperature drift. Thus, controlling the impact of noise is essential if the interferometric approach is to be adopted for in process measurement.

Consequently the authors have introduced an environmentally compensated interferometric technique for R2R barrier coating inspection based on the principle of wavelength scanning interferometry (WSI) as developed by Jiang et al [8].The working principle of this technique is shown in figure 7. WSI is employed to measure the surface topography of the barrier coating and is capable of generating surface maps with unambiguous height, without the well-known 2π phase ambiguity limitation. The interferograms are produced with no mechanical movement and are generated by means of scanning the wavelength of a halogen light in the visible region (683.4 nm-590.9 nm) using an acousto-optic tuneable filter (AOTF). Such a measurement methodology can provide significant enhancements in speed compared to comparable methods such as white light interferometry. In addition, WSI can be stabilised against environmental disturbances by using an active control of the reference arm, thus enabling nanometre scale measurements with large amounts of environmental isolation. This active control consists of a reference interferometer which provides positional feedback and a piezo-electric transducer (PZT) which moves the reference mirror. The PZT is driven by a PI controller to track the change in the optical path due to environmental disturbance such as mechanical vibration and refractive index drift.

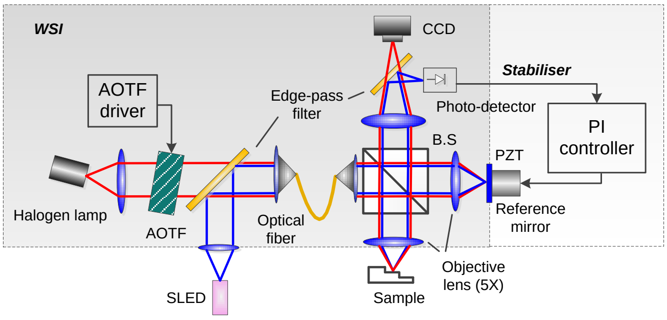


Figure 7: Configuration of the WSI

The system can compensate for disturbances in the optical path length up to several microns at 10^2 Hz. The reference interferometer is sourced by a super luminescent diode (SLED) light source having a central wavelength of 820 nm and sharing the same optical path as the rest of the WSI. The intensity of the generated fringes is detected by a photo-detector which monitors the SLED light only, via a dichroic filter that allows the scanned visible light to pass through to the CCD. A narrow optical band filter, with 3nm bandwidth and central wavelength similar to the SLED, is placed in front of the photo-detector to increase the coherence length of the reference interferometer, hence increasing the stabilisation range. During the wavelength scanning process, 256 interferograms are captured over a field of view of 640x480 pixels using 5X magnifications objective lenses. A periodic spectral interference pattern produced for each captured pixel is analysed individually using a Fourier transform algorithm. The full analysis of all the pixels is accelerated by parallelising the computation with a multi-core graphic processing unit (GPU). The WSI can capture and generate a full areal topography map in less than 3.7 seconds [9].

The vailidty of the WSI approach for measuring defects in barrier coating was tested for a range of Al_2O_3 ALD coated PEN substrates Exemplar samples were first measured using off-line metrology techniques (CCI) in order to detect and measure the defects and compare them later with the WSI measurement results. The coated samples used has comprised 80 mm diameter Polyethylene Naphthalate coated area with 40 nm thick Al_2O_3 film. The measurement protocol was as the following, 100 measurements were carried out by each of the techniques over the same sample area. Five different defects were collected from the sample. These defects have a lateral dimension ranges approximately (35-60) um. Table (1) shows the technical specifications of each technique. The specifications are given for the off-line CCI and in-line WSI.

Typically, the lateral range and resolution are varied for different objective lenses and imaging sensor sizes. The current WSI setup has CCD sensor size of 640x480 and objective lenses set of 2X and 5X magnifications. Whereas, the considered CCI sensor size is 1024x1024 pixels with objective lenses set of 5X, 20X and 50X magnifications and working distances shorter than the WSI equivalent objectives. Therefore, the WSI has the potential to increase the lateral range and resolution by simply changing the CCD senor size and objectives.

The table also shows that the vertical range for both instruments are equal, but this value mainly depends on the focus depth of the objective for the WSI. However, in this application, the defects vertical depth does not exceed several micrometres and are therefore within the limit of focus depth of high magnification objectives.

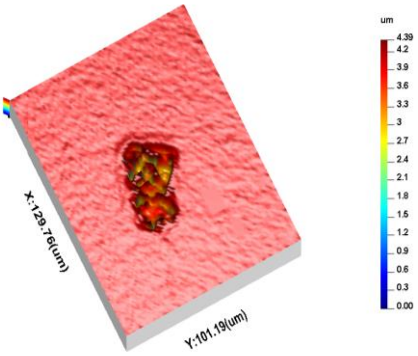
Table 3. Technical specification

Specifications	Method	
	CCI	**WSI**
Area (objective dependent)	0.3-7.2mm^2	0.5-1.8mm^2
Vertical resolution	0.001 nm	15 nm
Vertical range	100 um	100um
Lateral resolution	0.36 um	2.98 um
Repeatability of surface (noise)	0.003 nm	7 nm
Typical measurement time	10-20 seconds	<1 second

3.1 Comparison Results

Both systems (CCI and WSI) are calibrated and should yield closely comparable results. The work carried out for this research paper shows that, the surface roughness Sa, value for defect free sample measured by the WSI is higher when compared to the CCI method, see figures (4) and figure (5). Sa= 0.7nm (CCI) and 6.7nm (WSI). This discrepancy is due to the high noise floor level generated during the operation process of the WSI technique. This noise is considered to be generated from accumulative effects of environmental noise and WSI resolution and measurement uncertainty. However, this tolerance in the magnitude of surface roughness does not effect on defect detection ability nor characterisation since the coating thickness is approximately 40 nm.

Figure (6) and figure (7) show the same defect which has been measured by both CCI and WSI. Both instruments give similar values for the average vertical defect height which is approximately 1 um.



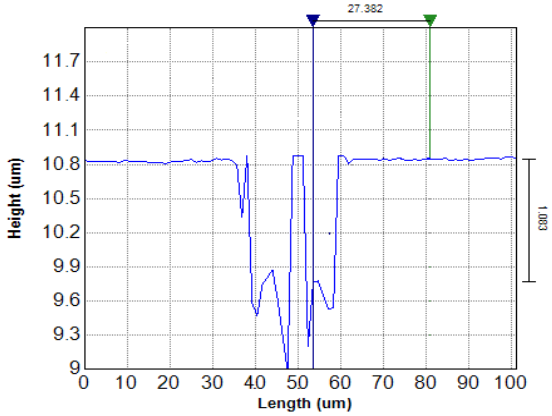


Fig. 8: Defect measurement using WSI

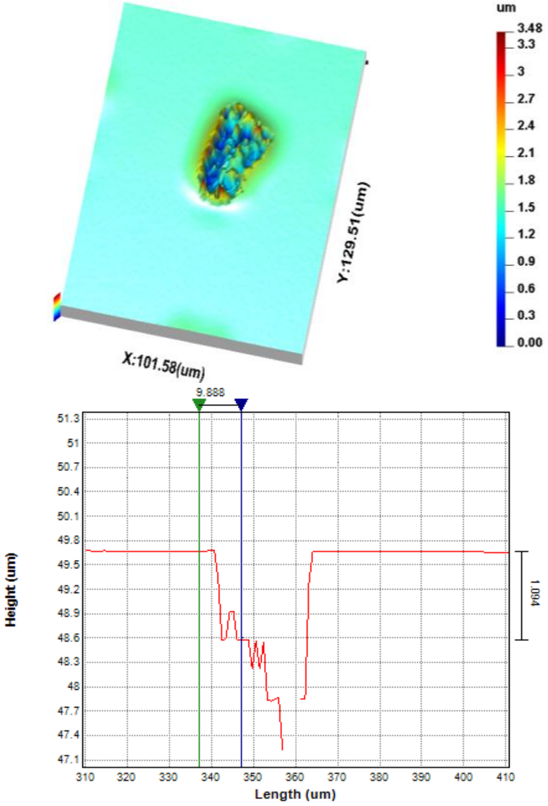


Fig. 9: Defect measurement using CCI

A 5X objective lens giving sample spacing of 1.19 um was used in the WSI, and for the CCI 20X objective lens giving sample spacing of 0.9 um was used. As an initial assessment for WSI measurement, it is considered that the results are comparable to the off-line technique (CCI). Example of the defects size/scale captured by the techniques is shown in figure (8).

Fig. 10: Defects lateral size measured offline and online process

The result in the above figure indicates that, the WSI system has accurately and reliably captured the morphology of defects that have been detected previously by the CCI. The WSI technique is consequently considered to be can be an efficient and optimal system to be used for in process thin film barrier defects inspection.

A proof of concept system is now under construction and is set to be deployed as a demonstration of the potential of the WSI technique. In order to deploy the WSI system consideration of the movement of the substrate film has been a further challenge. In this case a air bearing guidance system based on a New Way Air bearing PI ……… has been employed, in collaboration with IBS Precision Engineering, under optimal condition the substrate height deviation can be kept within < 5um. Figure 11 shows the surface height changes across a 50.5mm substrate when it is stabilised in the measurement zone.

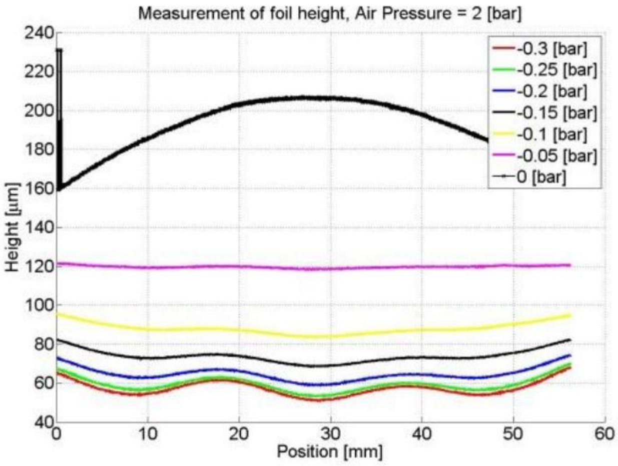


Fig: 11 Height variation across substrate when by air bearing

A schematic view of the proof of concept system and WSI head is shown in figure 12.Figure 12b shows the air bearing stage below the measurement head. In operation the sheet product will be scanned laterally at defined positions along the sheet figure 13. For the proof of concept system the sheet will remain static during data acquisition before being moved to the next measurement site

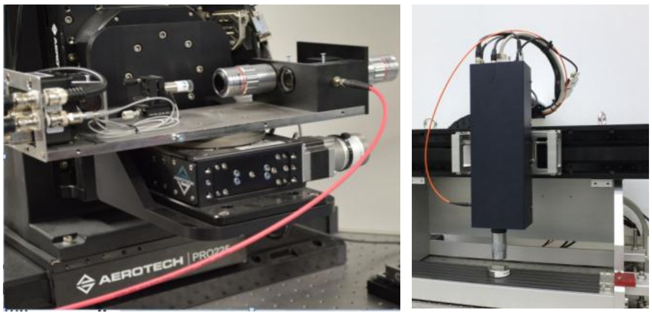


Fig 12 a) Realisation of WSI concept showing a) optical configuration b) WSI head above air bearing.

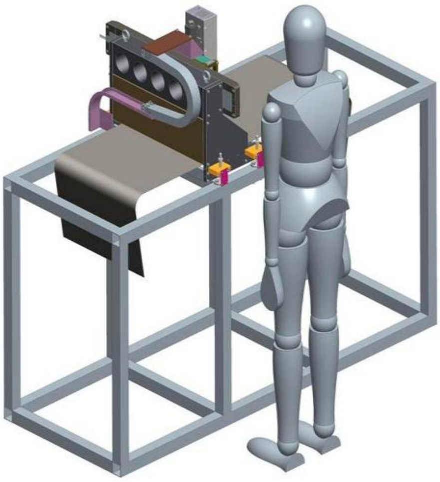


Figure 13 Schematic representation of WSI in line measurement system

5.0 Conclusions

It is well established that the performance of flexible PV is compromised by the presence of defects in the barrier layers. The present and previous work by the authors indicates that relatively large defects dominate the WVTR through the barrier layer. Metrology methodologies based on optical inferferometery and segmentation analysis has proved to be a powerful tool in surface characterisation.

Implementation of in-line defect detection systems requires fast and environmentally robust instrumentation. The white light Scanning inferferometer has been introduced as a solution to the measurement challenges the system has been demonstrated and the output results compare favourably with lab based instrumentation. The authors consider that WSI is a strong candidate for integration into quality assurance systems for developing the field of R2R manufacture. This is because WSI is a fast in-line defect measurement compared to commercial interferometric techniques such as CCI and robust against environmental disturbances.

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