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Paper:

Implementation of in Process Surface Metrology for R2R Flexible PV Barrier Films

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Thin functional barrier layers of aluminum oxide (Al_2O_3) that are used particularly in photovoltaic (PV) modules to prevent the possibility of water vapor ingress should be applied over the entire PV surface without any defects. However, for barrier layer thicknesses within the sub-micrometer range (up to 50 nm) produced through the atomic layer deposition (ALD) method, it is common for defects to occur during the production process. To avoid defective barriers from being incorporated in the final PV unit, defects need to be detected during the barrier production process.
In this paper, the implementation of in process inspection system capable of detecting surface defects such as pinholes, scratches, or particles down to a lateral size of 3 μm and a vertical resolution of 10 nm over a 500 mm barrier width is presented. The system has a built-in environmental vibration compensation capability, and can monitor ALD-coated films manufactured using roll-to-roll (R2R) techniques. Ultimately, with the aid of this in process measurement system, it should be possible to monitor the coating surface process of large-area substrates, and if necessary, carry out remedial work on the process parameters.

Keywords: metrology, defects, photovoltaic, thin-film and aluminum oxide

1. Introduction

Transparent barrier films made from materials such as Al_2O_3 are being used to prevent water vapor permeation into flexible PV modules. However, the presence of small defects in the barrier surfaces has been shown to have the potential to facilitate water vapor ingress [1], thereby reducing the cell efficiency and durability [2]. Consequently, improvements to the quality of such barrier films are critical for reducing the susceptibility of PV systems to environmental degradation. This paper reports on the deployment of an innovative in-line optical metrology technology to aid in the identification of micro- and nanoscale defects without reducing the level of productivity.

The research conducted in this paper was based on an investigation into Al_2O_3 atomic layer deposition (ALD) sample substrates with defects present so as to create a comprehensive database that will aid in the development of the required defect sensors. Output from such an in-line system will provide a critical tool for process improvements.

2. Challenges in Defect Detection

The relationship between the surface morphology, defect density, and water vapor permeability through 40 nm Al_2O_3 barrier coatings produced using ALD [3] on 125-μm polyethylene naphthalate (PEN) substrates has to date only been studied in a laboratory [4, 5]. However, detecting defects off-line is difficult and time consuming. New methods for manufacturing barrier films using R2R methods are emerging, as shown in **Fig. 1**; however, such procedures can result in large quantities of barrier films being manufactured and integrated into a PV unit before any defects are detected and the process parameters optimized. Furthermore, the quality requirements and line speed in the PV manufacturing industry predicate the use of off-line defect detection methods. Therefore, it is desirable to make use of non-contact optical-based in-line inspection systems to detect defects during the Al_2O_3 ALD manufacturing processes. Nevertheless, implementing a high-resolution in-line (optical) measurement system in a R2R production environment is challenging because the positioning and stability requirements to be within the focal depth of the objective lens (e.g., less than 14 μm for a 5X objective lens) and less than the vertical resolution of the instrument (i.e., a few nanometers), respectively, are very demanding.

To facilitate in process measurement for R2R-produced substrates, two challenges need to be addressed: i) the measurement must be fast and non-contact, and ii) the measurement must be able to be carried out in a “noisy” working environment; one of the only feasible measurement solutions currently available is optical interferometry. However, interferometric measurement techniques

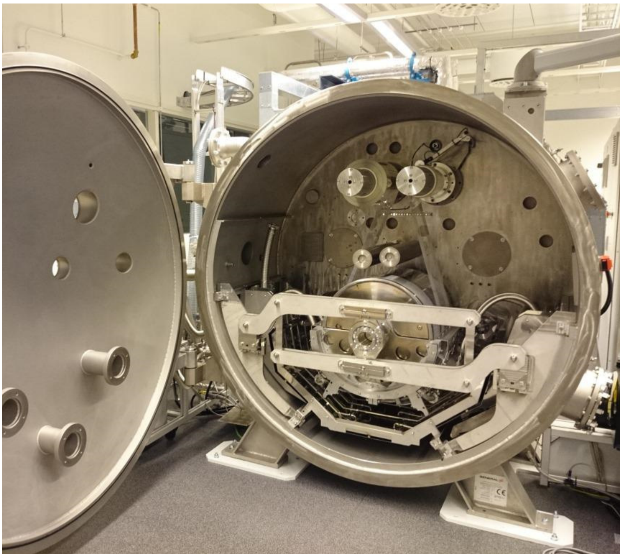


Fig. 1. Beneq WCS 500 R2R ALD reactor (Courtesy of Center for Process Innovation, UK).

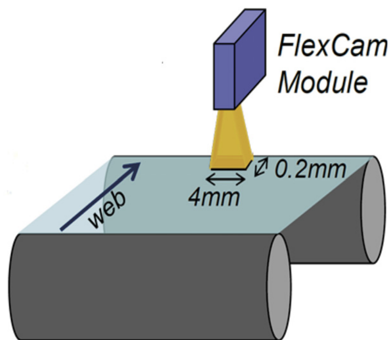


Fig. 2. In-line defect detection system for R2R barrier films of PV modules [7].

are extremely sensitive to environmental noises such as mechanical vibrations, air turbulence, and temperature drift. Thus, controlling the impact of noise on the measurement quality is essential if an interferometric approach is to be implemented for a process measurement.

A variety of groups and corporations are pursuing various in-line measurements for the flexible display industry, including bright-field inspection systems and scanning interferometers for 3D measurements [6, 7]. A single-shot interferometry system called "FlexCam" developed by 4D Technology is currently being used to detect defects for PV barrier films manufactured through R2R technology [7], as shown in **Fig. 2**.

This interferometric method relies on laser interferometry to acquire 3D surface data. It acquires all phase-shifting information quickly, the acquisition time depending solely on the camera exposure time (i.e., the frame rate). The goal of FlexCam is to provide about a 1.2-μm lateral resolution and a 2-nm vertical resolution over a field of view (FOV) of roughly 4 mm $\times$ 0.2 mm. This technology can provide areal measurements in milliseconds; however, it has a limited vertical range, which may pose a problem when identifying and classifying large

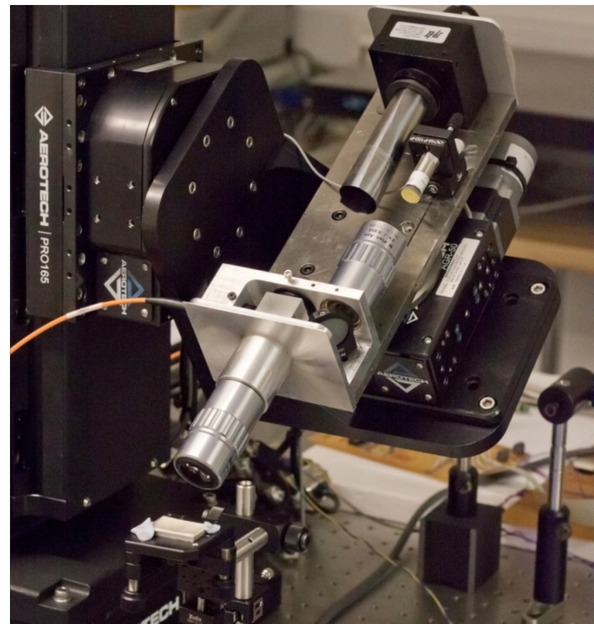


Fig. 3. WSI in the test setup.

vertical defects [7]. This paper reports on the deployment of a novel in-line interferometric optical technique based on wavelength scanning interferometry (WSI) for detecting PV barrier defects (up to 2.9 μm in lateral size) with a vertical range of 100 μm. The instrument has a built-in environmental vibration compensation capability, providing areal measurements at a high speed of less than 1 s per field of view (FOV). The technique is currently being deployed using a demonstration system at a R2R production facility. The results show the capability of the WSI to be used as a quality assurance tool in R2R production lines, where the results compare favorably with results obtained through off-line optical techniques (i.e., the Coherence Correlation Interferometer (CCI) 3000 of Taylor-Hobson, Ltd).

3. Wavelength Scanning Interferometry

The current industry standard interferometry systems generally work by mechanically shifting the phase of the fringes by scanning the position of an objective lens relative to the substrate/surface being measured. Such a technique is too slow to conduct large numbers of measurements required to enable a defect detection analysis for large-area thin-film barriers. The WSI shown in **Fig. 3** can take areal measurements without mechanical movement by simply changing the wavelength of a broadband light source in the interferometer setup [8]. This wavelength scanning process can ensure a phase shifting operation faster than conventional mechanical scanning methods. In addition, defects can be measured effectively in a factory environment because the WSI has an active closed-loop vibration compensation system [9].

The current time for data acquisition of the WSI is approximately 2 s, where a 3D measurement analysis is

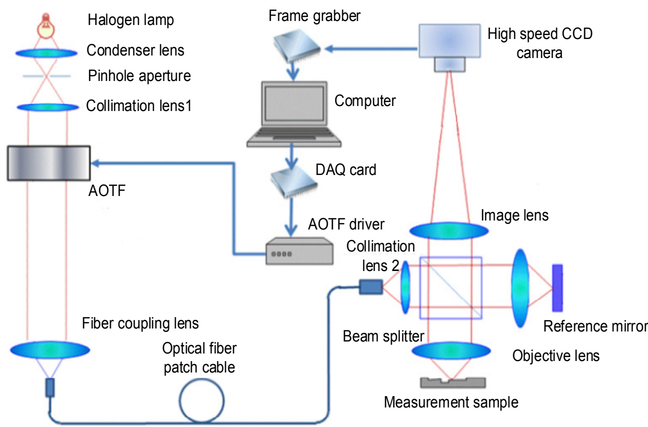


Fig. 4. Schematic representation of WSI system [9].

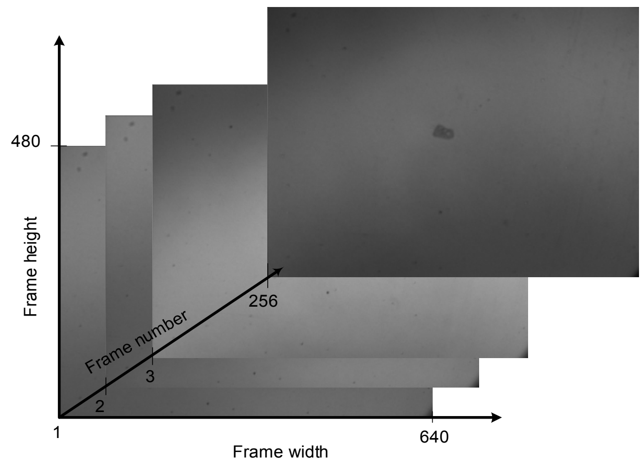


Fig. 5. Interferograms collected from a single pixel.

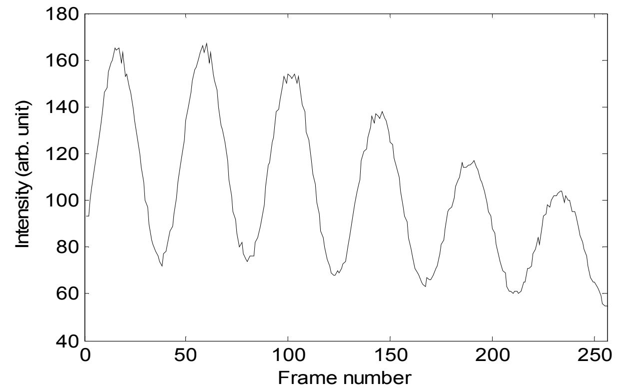


Fig. 6. Change of intensity across a single pixel.

achieved using a graphics processing unit (GPU) to accelerate the computing process to less than 1 s per FOV. However, this measurement time can be effectively reduced to less than 1 s if a faster CCD is employed and an up-to-date GPU is used.

4. WSI Measurement Principle

The measurement principle of the WSI is based on determining the phase shift of a constructed interference fringe while the wavelength of the illuminating light is scanned [9]. The wavelengths of white light, obtained from a halogen lamp, are scanned through a visible range of 683.42 to 590.98 nm by filtering a narrow spectral band using an acousto-optic tunable filter (AOTF). The system comprises two interferometers that share a common optical path, as shown in **Fig. 4**. The interferometer uses a filtered white-light source for measuring the surface topography. Another interferometer using a super-luminescent diode (SLED) is used to monitor changes in an unwanted optical path from barrier surface movement or temperature drift, and acts as a feedback sensor for an active servo control to cancel out such changes in the optical path [10].

The noise cancellation is achieved by moving the reference arm mirror using a piezo-electric translator (PZT), which acts according to the manipulated feedback signal. The noise cancellation can ensure the fringe stabilization before the wavelength scanning measurement process is carried out [8]. As such, any alteration to the optical path (vertical) will be considered as a noise source and can be compensated by the active control loop if the noise range is within the stabilization bandwidth. The stabilization bandwidth is typically dependent on the translation range of the PZT and frequency response of the system. In this application, a PZT type P-840 from PI Ceramic with a travel range of up to 15 μm and a control frequency response of up to 3 kHz is used.

Typically, 256 interferograms can be captured by current CCD cameras; each pixel in an obtained interferogram represents a specific point on the sample surface, as shown in **Fig. 5**. The number of interferograms taken depends on the specific precision and range requirements of the measurements.

By isolating a single pixel (which corresponds to a specific point on the sample) from a set of interferograms, a sinusoidal change of intensity with respect to the wave number (reciprocal of the wavelength) is apparent, as shown in **Fig. 6**. The overall phase shift across the wavelength scan range can be obtained from the intensity signal using Fourier transforms. The height of the point represented by a pixel can thus be calculated by

$$h(x,y) = \frac{\Delta\varphi(x,y)}{4\pi\left[\frac{1}{\lambda_{\max}} - \frac{1}{\lambda_{\min}}\right]}, \quad \text{(1)}$$

where $h(x,y)$ is the height of the specific pixel; $\lambda_{\max}$ and $\lambda_{\min}$ are the upper and lower filtered wavelengths of the scan range, respectively; and $\Delta\varphi(x,y)$ is the calculated phase shift over the scan range [9]. As a result, the height can be calculated after extracting the overall phase change produced by scanning the wavelength through a spectrum bandwidth of approximately 100 nm within the visible region. However, to guarantee a sufficient phase change and avoid a zero optical path difference, an offset distance with a magnitude of 5 μm has been introduced into the interferometer between the beam splitter and the reference objective lens (i.e., unbalanced arms produced for the interferometer) [10].

Table 1. Technical specifications [10, 11].

Specifications	Method	
	CCI 3000	WSI
Area (objective dependent)	0.3–7.2 mm^2	0.5–1.8 mm^2
Vertical resolution	0.01 nm	15 nm
Vertical range	100 μm	100 μm
Lateral resolution	0.36 μm	2.98 μm
Repeatability of surface (noise)	0.003 nm	7 nm
Typical measurement time	10–20 seconds	< 1 second

5. WSI System Verification

To verify the WSI system capability, samples of 40-nm Al_2O_3 coated onto a 125-μm PEN layer were first measured using an off-line commercial metrology technique (coherence correlation interferometry using a Taylor-Hobson, Ltd. CCI 3000) to detect and measure any defects and compare them later with the WSI measurement results. **Table 1** shows the technical specifications of each technique.

Typically, the lateral range and resolution are varied for different objective lenses and imaging sensor sizes. The WSI has the potential to increase the current lateral range and resolution by simply changing the CCD senor size and lens objectives. In this study, both systems (CCI and WSI) are calibrated and should yield closely comparable results. A 5X objective lens giving a sample spacing of 1.19 μm was used in the WSI, and for the CCI, a 20X objective lens providing a sample spacing of 0.9 μm was used. The initial measurement procedure was as follows: more than 100 typical defects were measured by both the CCI-3000 and WSI techniques. These defects were found to have lateral dimension ranges of approximately 20 to 60 μm, as shown in **Fig. 7**.

The measurement results also reveal that the surface roughness parameter value for a defect-free sample measured by the WSI is higher when compared to the results obtained using the CCI-3000 method, as shown in **Figs. 8** and **9**.

The high roughness value of the WSI data was due to the high noise floor level generated during the operation process of the WSI technique. This noise is most likely generated from accumulative effects of environmental noise, the WSI resolution, and measurement uncertainties. However, this tolerance in the magnitude of the surface roughness does not affect the defect detection capability or the characterization because the coating thickness of the Al_2O_3 layer is approximately 40 nm. Moreover, **Figs. 10** and **11** show the same defect measured by both the CCI-3000 and WSI techniques. Both instruments give similar values for the average vertical defect height, which is approximately 1 μm.

As an initial assessment for the WSI measurements, it was found that the results are very comparable to the results of the off-line CCI-3000 technique. For such thin barrier layers, the lateral dimensions are of critical functional importance. Examples of the defect size/scale cap-

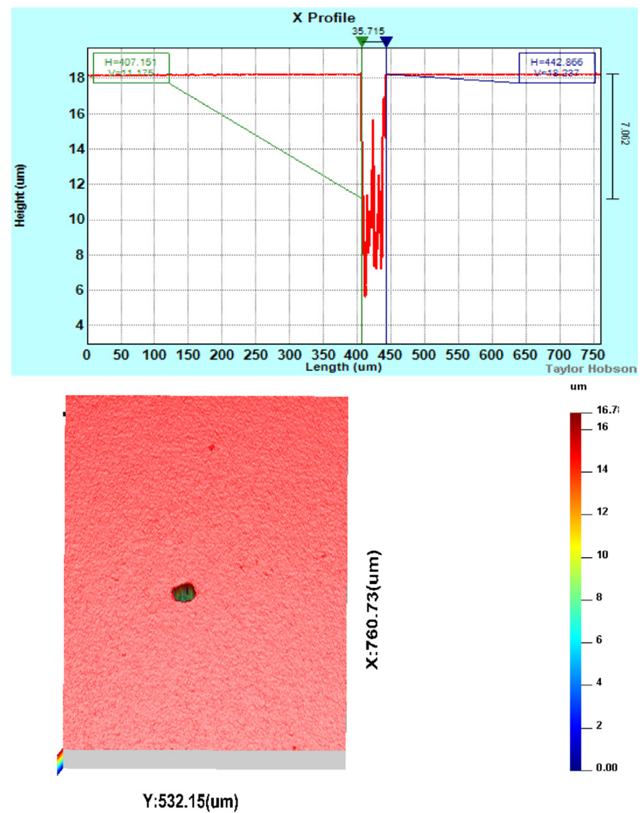


Fig. 7. Defects measured using the WSI system.

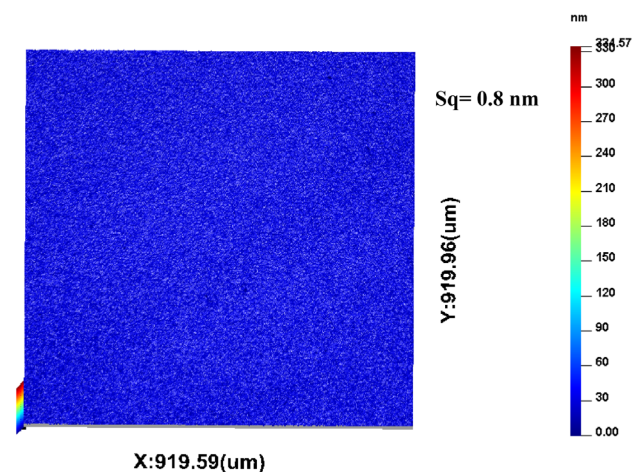


Fig. 8. Surface roughness of non-defective area measured using the CCI method.

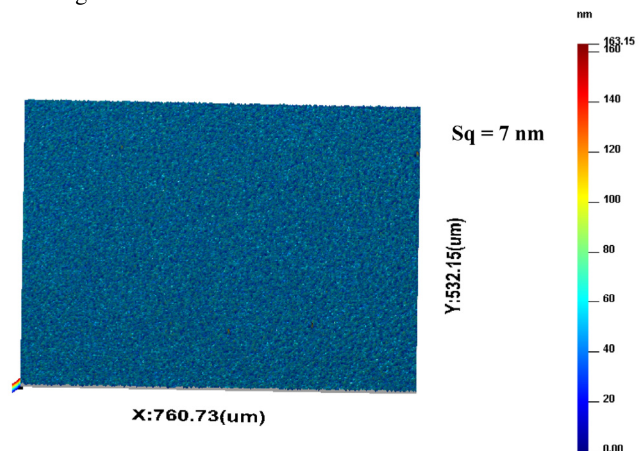
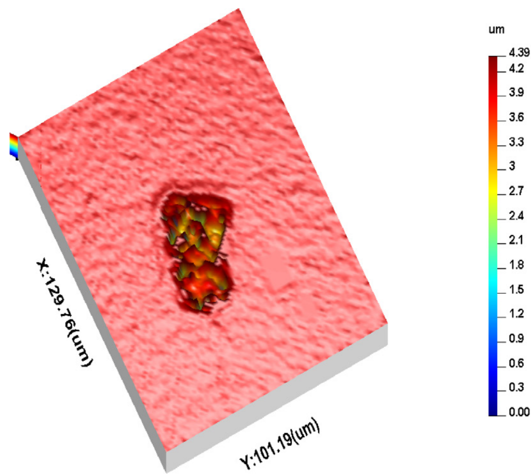


Fig. 9. Surface roughness of non-defective area measured by the WSI technique.



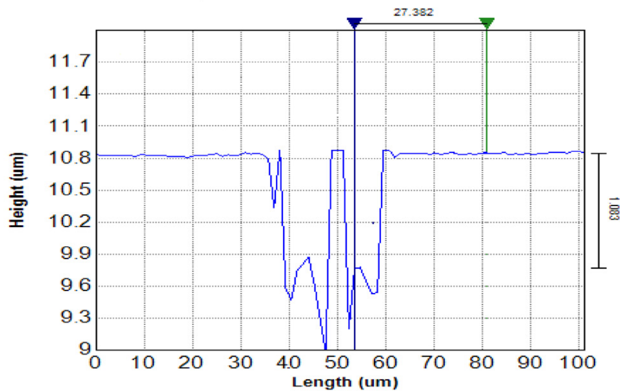
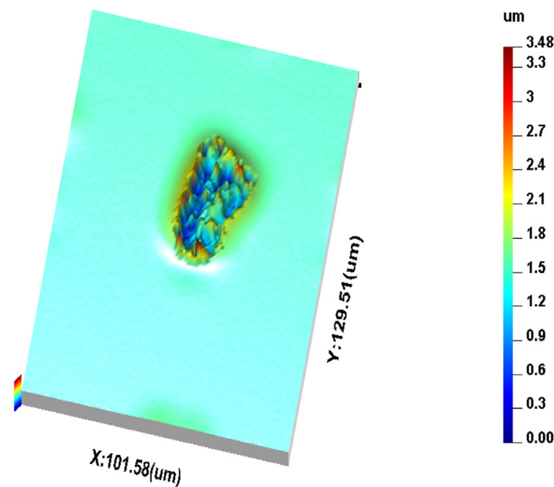


Fig. 10. Defect measurement using WSI.



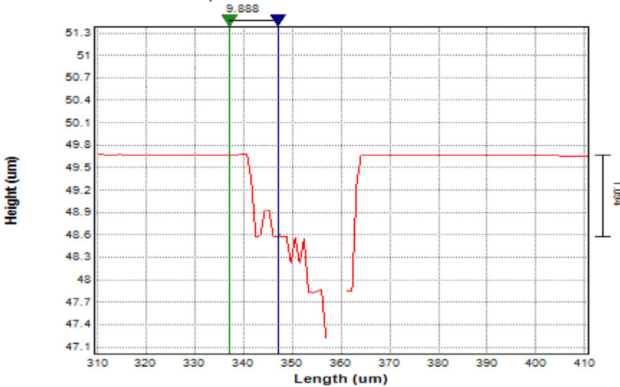


Fig. 11. Defect measurement using CCI.

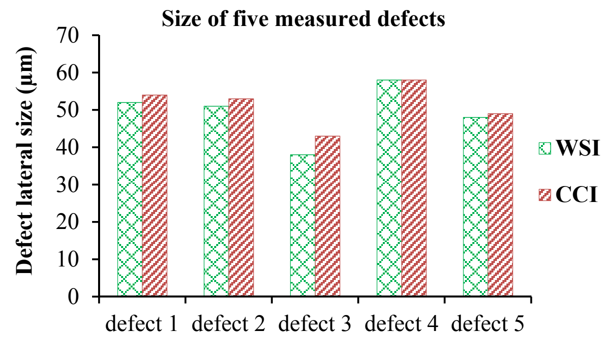


Fig. 12. Lateral size of defects measured in both off-line and in-line processes.

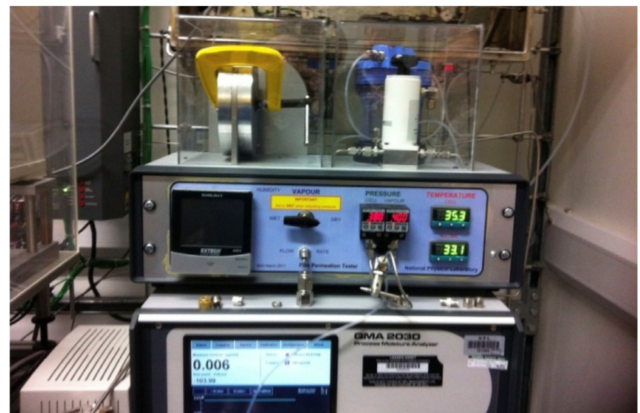


Fig. 13. Traceable in-house WVTR measurement instrument.

tured by the applied techniques are shown in **Fig. 12**. The results show that the WSI system accurately and reliably captures defects that were previously detected by the CCI instrument. Therefore, the WSI technique can be an efficient and optimal system for defect inspection in the processing of thin film barriers.

6. WSI Performance Study Results

A further analysis was conducted on two other Al_2O_3 ALD-coated samples to assess the capability of the WSI technique to distinguish between PV substrates with high and low water vapor rates. In this case, the correlation is based on the density of the detected defects. The samples have a 72.75 cm^2 area, and were initially measured for the water vapor transmission rate (WVTR) using a traceable in-house developed instrument at the National Physical Laboratory (NPL), as shown in **Fig. 13**. The instrument has a higher WVTR sensitivity limit of 3×10^{-5} g/m^2/day than the commercial MOCON® test. **Fig. 14** shows the WVTR test results for the two samples.

Following the WVTR test, the samples were first measured using a CCI-3000 instrument with a 20X objective lens, covering about 13% of the total sample area in a clean room (class 10000). The protocol used in this study

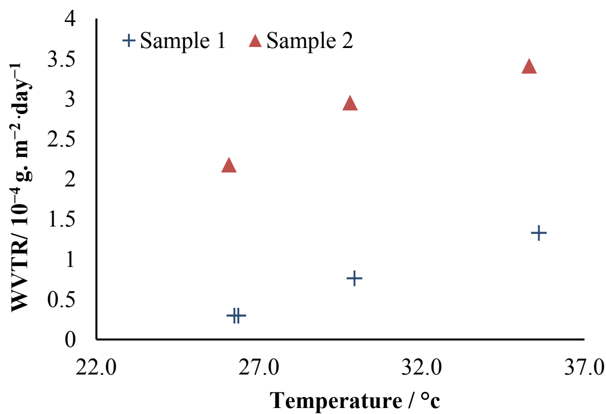


Fig. 14. WVTR test results.

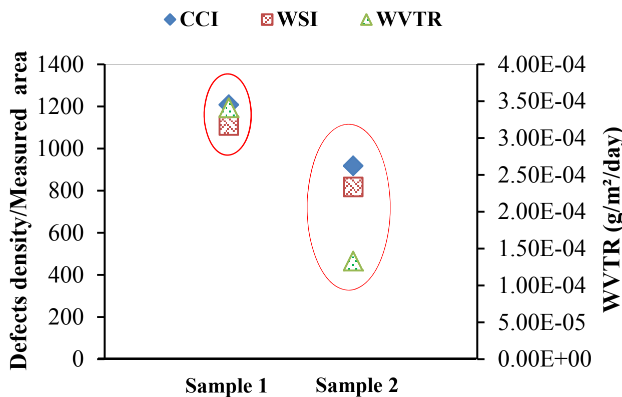


Fig. 15. Defects density versus WVTR for two samples.

measures all visible defects over the substrates. Following that, the WSI was used with 5X lens objective in a clean room (class 10000), to measure the same measured area that was previously measured by the CCI, and covering 13% of the total sample area. The collected data from both the CCI and WSI instruments were analyzed through a segmentation analysis [12] using a Surfstand software package, and the criteria used to segment and count only significant defects were (3 $\times$ Sq vertical $\approx$ 20% Sz) and 3 μm laterally, which is in accordance with previous studies conducted by the authors [13]. The results of the analysis, shown in **Fig. 15**, indicate a clear correlation between the defect size, density, and measured WVTR, as well as the capability of the WSI to capture such defects. The analysis also appears to indicate that a sample with a higher density of defects that satisfy the criteria of greater than 3-μm lateral spacing and 3 $\times$ Sq vertically exhibits inferior barrier properties, and a clear difference in the density values for two samples can be distinguished.

The results indicate that the WSI system accurately and reliably captures the morphology of defects that were detected by the offline CCI technique. The WSI technique is consequently considered to be an efficient and optimal system for defect inspection when processing thin film barriers.

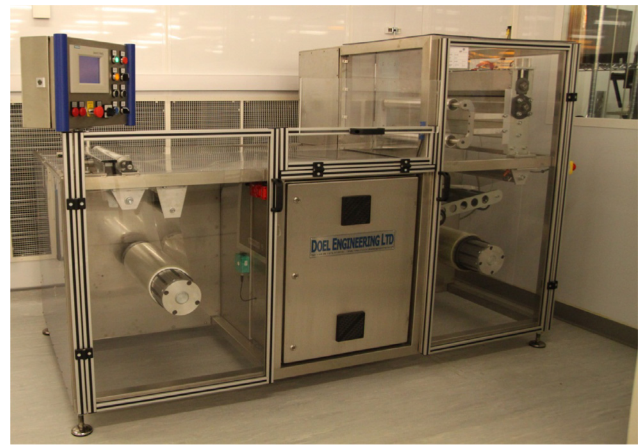


Fig. 16. Photograph of DOEL demonstrator/re-winder.

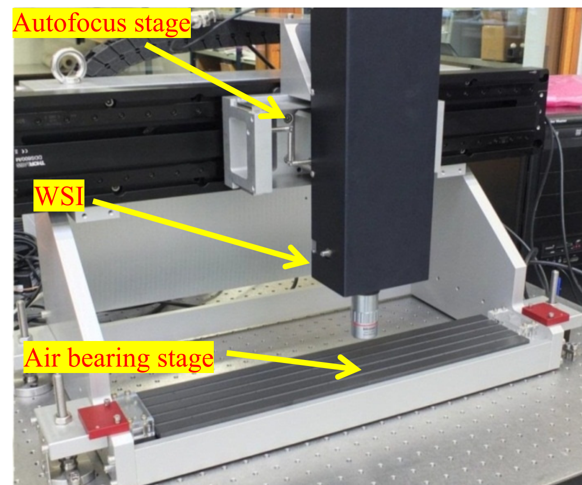


Fig. 17. *In situ* WSI integration.

7. WSI Integration in a R2R Demonstration System

To show the capability of the high-precision WSI system, a demonstration system was constructed *in situ* as a proof of concept at the Center for Process Innovation (CPI) in the UK. The entire demonstration system is contained in a class 1000 clean room. The Al_2O_3 ALD foil translation in the y-direction is facilitated by a foil re-winder, as shown in **Fig. 16**. The re-winder setup allows a large working area, where the high-precision WSI system can be implemented.

The instrument was integrated with an auto-focus linear stage mounted on a transverse stage, as shown in **Fig. 17**, so that the full width (500 mm) of the foil can be covered. In addition, deploying the WSI system when considering the movement of a substrate film has been a further challenge. In this case, an air-bearing guidance system based on a NEWWAY air bearing was employed, as shown in **Fig. 17**, in collaboration with IBS Precision Engineering (Netherlands). Under optimal conditions, the height deviation of the substrate can be kept within 5 μm. **Fig. 18** shows the changes in surface height across a 50.5-mm substrate when stabilized within the measurement zone.

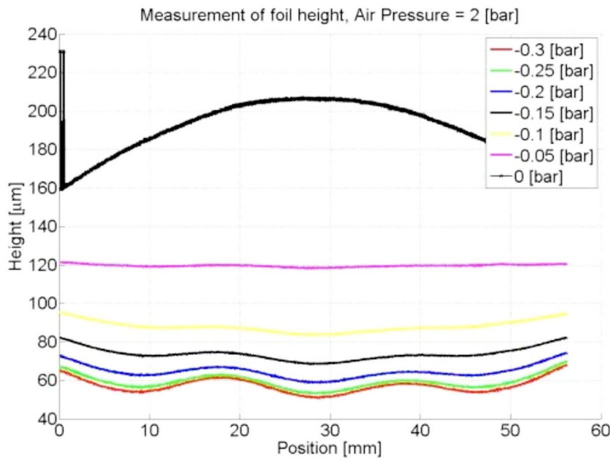


Fig. 18. Height variation across the substrate when applying an air bearing.

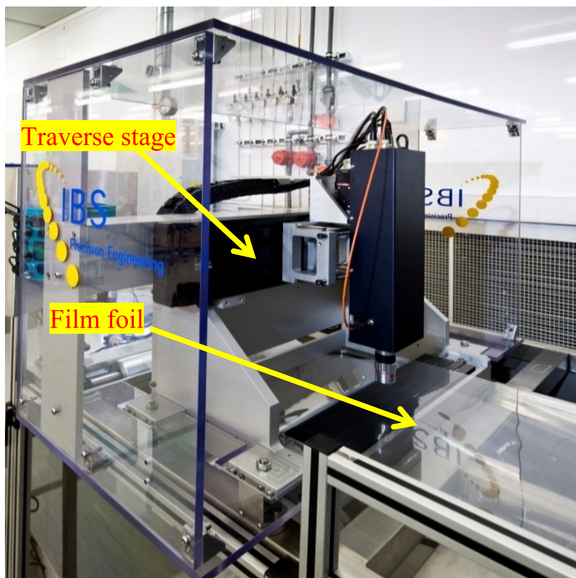


Fig. 19. A schematic view of the proof of concept system and WSI head.

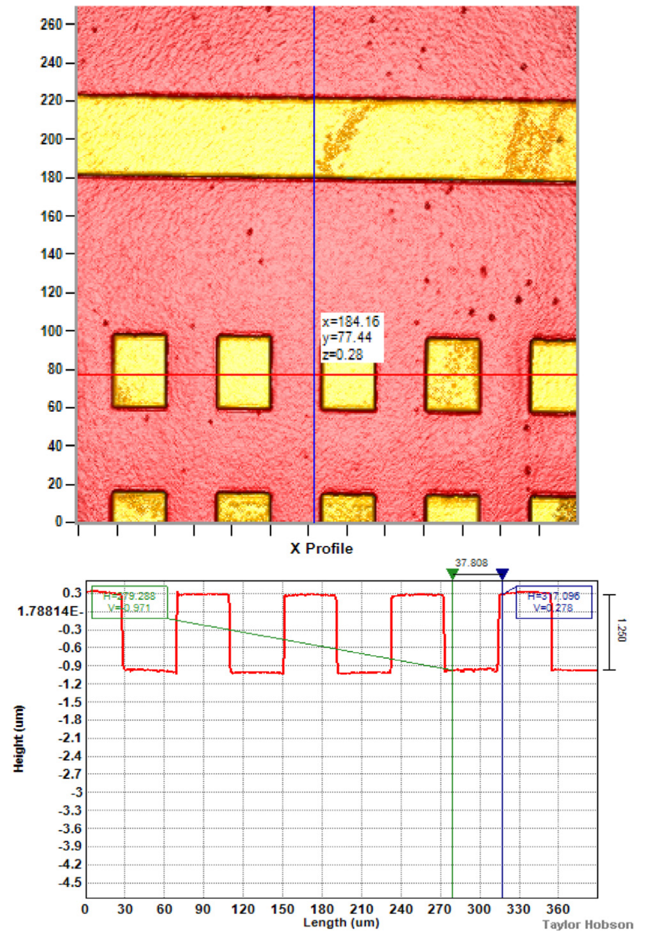


Fig. 20. Measured results for 2-μm standard step height sample using the WSI.

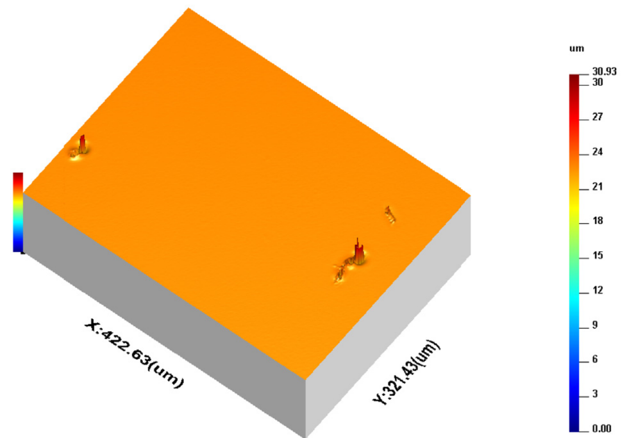


Fig. 21. *In situ* measurement of coated film.

One of the inspection challenges is conducting a high-fidelity surface measurement despite the possible vibrations introduced by the air-bearing stage. This challenge can be overcome by using the built-in active control loop in the WSI to compensate for any environmental disturbances.

The system setup of the proof of concept system and WSI head is shown in **Fig. 19**. The WSI is combined with a traverse stage to provide full coverage of the foil despite the limited FOV of the instrument. During the operation process, the sheet product will be scanned laterally by shifting the WSI by one FOV (including a 10% overlap) for each measurement step.

The initial implementation of the system will acquire a series of static images and thereby allow a significant area, but not the entire substrate surface, to be measured. The WSI system was implemented in a R2R demonstrator, as shown in **Fig. 19**, and calibrated by measuring a 1.2-μm standard step height sample (Bento Box) supplied by NPL. **Fig. 20** shows the results of the calibration artifact measurements.

A gold-coated PET film was measured to obtain the preliminary results *in situ*, and three defects were detected, as shown in **Fig. 21**. The system was then run to capture a series of static images (areal measurements) over the Al_2O_3 ALD foil, with 216 measurements taken in 1 h, which allowed a significant area, but not the entire substrate surface, to be measured.

8. WSI Data Handling Procedure

One of the main challenges for the application of such a metrology instrument is how to assess large and multiple measurement datasets. When measuring the surface topography over large-area substrates (i.e., approximately a 500-mm film foil width) with a limited FOV of the WSI, the procedure will produce hundreds or thousands of measurement files. Assessing each file individually to find and analyze defects manually is time consuming and impractical. Therefore, a computerized solution to assess these files by monitoring the surface topography parameters was developed, as shown in **Fig. 22**. Comparing the parameter values to an experimentally determined threshold value, obtained from extensive lab-based measurements for an ALD coating that were previously published in [4] and [13], the average roughness of a Al_2O_3 ALD coating without defects using the WSI was found to be Sq ≈ 7 nm, as shown in **Fig. 9**.

Previous studies [4, 13] have demonstrated that significant defects will increase the roughness Sq (with defect) larger than 3 $\times$ Sq (no defect) i.e., Sq (with defect > 21 nm). Moreover, segmentation analysis results [5, 13] appear to indicate that the major contributing factor for determining the WVTR is the total number of larger defects, where a sample with a higher density of defects of greater than 3 μm in lateral dimension and a 3 $\times$ Sq vertical dimension exhibits inferior barrier properties. These criteria are considered to be the basis for developing the segmentation "toolbox" shown in **Fig. 22**. Accordingly, if the Sq of the measured surface is greater than the threshold limit, the surface will be subject to a data segmentation procedure for extracting significant defects. Otherwise, the measurement will treat the surface as being defect free. For data segmentation, edge processing using Wolf and area-pruning methods is employed to identify and characterize the defects.

The Wolf pruning method, as defined in ISO 25178-2 [12], allows the detection of significant features on barrier surfaces and the determination of their morphology parameters (dimension, area, volume, etc.). The protocol used for characterizing the barrier films used in this study is described in a flow chart, where the surface was first filtered to eliminate data noise, which is achieved using box filtering (Gaussian filtering) with a cut-off of 2^n points, where n is the level of smoothness (from 1 to 5), and was specified as 5 herein. After the smoothing process, edge processing was applied on the data using a Sobel type operator [14]. The edge data were then "pruned" using Wolf pruning [12]. Following the Wolf pruning, an "area pruning" was applied, where the area was found to have a lateral diameter of up to 3 μm (which calculated based on a mathematical model published in [13] was deemed insignificant). **Fig. 23** shows the segmentation process and the power of the procedure for extracting defects from the surface data.

The procedure can be repeated to analyze all measured files to extract and count the defects and determine the chosen surface parameters (Sq, Sp, and Sv) as given in the output text file shown in **Fig. 24**.

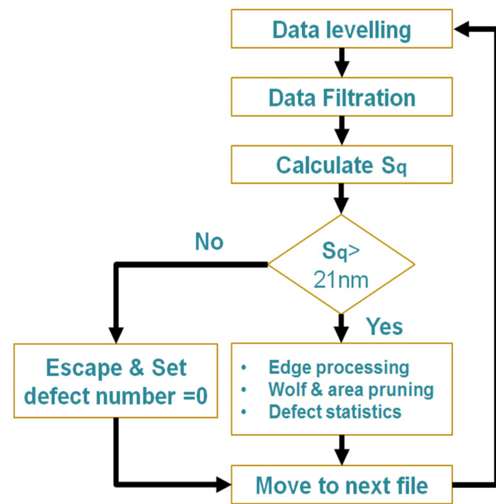


Fig. 22. Extraction of Al_2O_3 ALD defect statistical procedure using WSI.

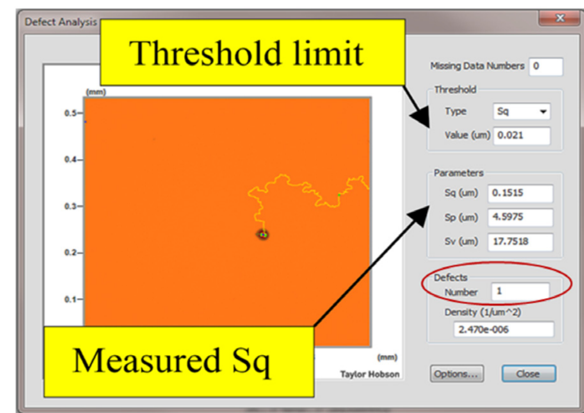


Fig. 23. Defect extraction using data segmentation (edge processing and Wolf pruning).

DefectsOutput - Notepad

File Name	Missing Data	Sq value(um)	Sp value(um)	Sv value(um)	Defects Number	Defects Density(1/um^2)
p1	0	0.013512	0.167268	1.211728	0	0.0000000
p10	0	0.008776	0.178558	0.376893	0	0.0000000
p100	0	0.054110	1.428052	8.203407	1	2.305e-006
p101	0	0.066044	8.092217	6.069404	9	2.075e-005
p102	0	0.036756	1.428892	4.900036	2	4.610e-006
p103	0	0.155251	4.074205	18.013100	2	4.610e-006
p104	0	0.092417	4.716363	9.277656	8	1.844e-005
p105	0	0.160350	3.587061	16.407406	4	9.221e-006
p106	0	0.103912	2.741945	13.174541	22	5.071e-005
p107	0	0.109479	5.027643	10.032352	5	1.153e-005
p108	0	0.122309	3.211112	15.662971	1	2.305e-006
p109	0	0.127985	3.537587	15.200192	2	4.610e-006
p11	0	0.048769	1.113212	10.223041	2	4.940e-006
p110	0	0.073023	1.900081	10.368506	1	2.305e-006
p111	0	0.112940	2.949974	12.328129	2	4.610e-006
p112	0	0.095846	3.194979	11.642429	1	2.305e-006
p113	0	0.048052	7.984729	6.021550	11	2.536e-005
p114	0	0.097775	2.405353	12.302823	1	2.305e-006
p115	0	0.062832	1.495073	8.417302	9	2.075e-005
p116	0	0.041706	0.858395	5.494088	3	6.916e-006

Fig. 24. Defect extraction output of the computerized method.

9. Conclusion

It has been well established that the performance of flexible PV modules is compromised by the presence of defects in the barrier layers. Lab-established measurements have indicated that defects with a lateral size of up to 3 μm have a very detrimental effect on the barrier film functionality. Metrology methodologies based on optical interferometery and a segmentation analysis method have proved to be powerful tools in an Al_2O_3 ALD barrier surface characterization. To implement in process metrology

during the production of a R2R barrier coating, optical interferometery must be applied. To overcome any environmental noise, a novel compensated wavelength scanning interferometry system has been developed, and this system has shown the ability to overcome environmental vibrations and additionally conduct high-speed areal measurements in situ in less than 1 s per FOV.

The results of this research show that a wavelength-scanning interferometer can be a viable solution to the challenges facing R2R barrier film defect measurements. The proposed system was demonstrated herein, and the output results were shown to compare favorably with results obtained with an optical-based commercial-lab instrumentation and functional assessment. Consequently, the WSI is considered to be a strong candidate for integration into quality-assurance systems for developing the field of R2R manufacturing.

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