Defect Detection in Thin-film Photovoltaics; Towards Improved Efficiency and Longevity

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***Abstract*— The Photovoltaic (PV) industry is seeking to increase efficiency and functional lifetime of PV modules manufactured on polymer substrates. High resolution and high speed surface inspection for the quality control of the manufacture of large area flexible PV modules are necessary to guarantee maximum quality, longer lifetime and enhanced product yield. Flexible PV films are the newest development in the renewable energy field and the latest films have efficiencies at or beyond the level of Si-based rigid PV modules. These modules are fabricated on polymer film by the repeated deposition, and patterning, of thin layer materials using roll-to-roll technology. However, they are at present highly susceptible to long term environmental degradation as a result of water vapor transmission through the protective encapsulation to the active layer. To reduce the WVTR the PV encapsulation includes a barrier layer of amorphous Al_2O_3 on a planarised polymer substrate. This highly conformal barrier layer is produced by atomic layer deposition (ALD). Nevertheless water vapour transmission is still facilitated by the presence of micro and nano-scale defects in these barriers which results in decreased cell efficiency and reduced longevity.**

Analytical techniques including: White Light Scanning Interferometry (WLSI), Atomic Force Microscopy (AFM) and Scanning Electron Microscopy (SEM) were used to characterise the water vapor barrier defects. Areal surface texture parameter analysis allows the efficient separation of small insignificant features from significant defects. This parametric analysis is then correlated with the water vapour transmission rate as measured on typical sets of films using standard MOCON test. The paper finishes by drawing conclusions based on analysis of WVTR and defect size, density and distribution, where it is postulated that small numbers of large features have more influence on the deterioration of water vapor transmission rates than large numbers of small features. This result provides the basis for developing roll-to-roll in process metrology devices for quality control.

Keywords—Photovoltaic; Aluminum Oxide; ALD; Defects; WVTR.

I. INTRODUCTION

The development of low cost, efficient and reliable photovoltaic devices is a major technological challenge which demands suitable materials and fabrication processes as well as quality control systems. In today's industry, the most common type of solar photovoltaic (PV) cell is fabricated from either crystalline silicon or thin-film materials [1]. 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 the present time [2].

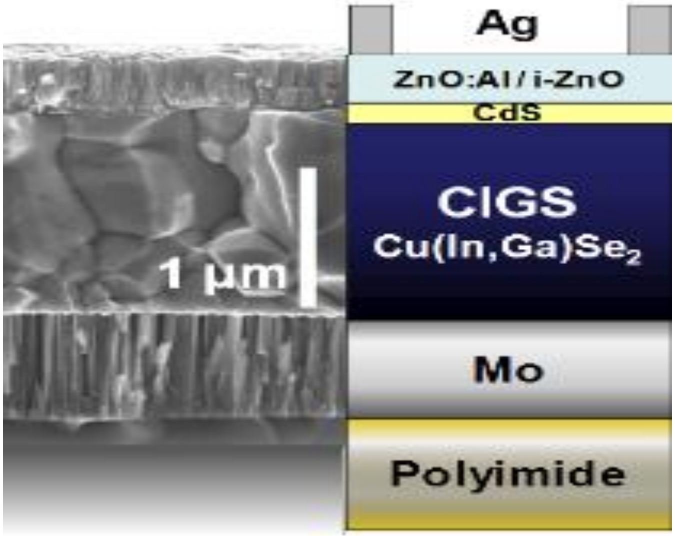

Fig. 1. Typical structure of flexible PV module (Courtesy of Flisom, Switzerland)

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 Cº and 85 % relative humidity. One solution to the moisture problem is to encapsulate CIGS cells with rigid glass in order to achieve a lifetime of at least 20 years, which is the standard for Si PV cells, but this increases weight and does not allow for flexibility to be maintained. 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. Studies regarding the encapsulation technologies have been carried out by Carcia *et al.* [3], 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 Cº and 85 % RH, with simulated solar illumination. 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 Cº and 85 % RH, whereas the CIGS cell protected with the 55 nm ALD Al_2O_3 barrier 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_2O_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.

A. Flexible PV module

Flexible solar modules comprise four functional layer groupings as shown in Fig 2. 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). The ALD is a controlled layer-by-layer deposition technique that enables the deposition of thin, smooth, and highly conformal films with atomic layer precision and typical thickness ranges between 1 and 100 nm [4].

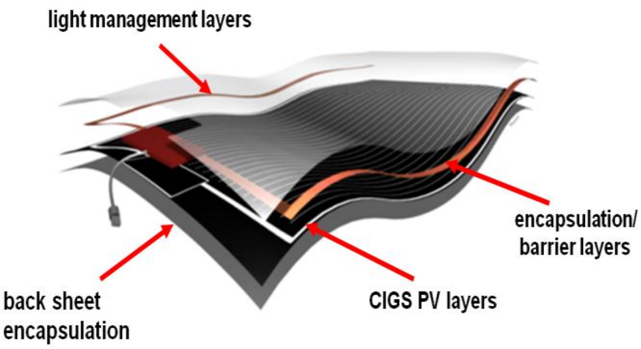

Fig. 2. Schematic of the flexible PV Module (Courtesy of Flisom, Switzerland)

II. 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 ALD Al_2O_3. The samples were prepared in a clean room under conditions shown in table I.

TABLE. I TEST SAMPLES AND PRE-COATING CONDITIONS

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

III. WATER VAPOUR TRANSMISSION RATE ASSESSMENTS

Prior to the surface measurements, the 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 Fig 3.

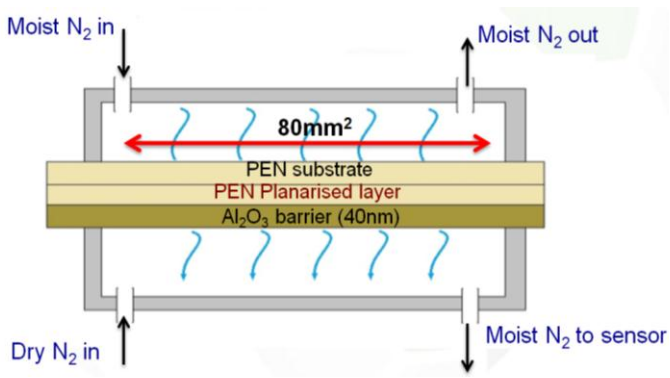

Fig. 3. Schematic representation of WVTR test

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 [5].

IV. DEFECT DETECTION METHODOLOGY

The samples in section II, which were at first examined for WVTR, the results 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 Scanning Electron Microscopy, Atomic Force Microscopy and White Light Scanning Interferometry.

A. Scanning electron microscopy analysis

A FEI Quanta 250 field emission gun environmental scanning electron microscope (FEG-ESEM) was used for imaging the Al_2O_3 barrier layers. When performing secondary electron imaging, the spatial resolution of the instrument in the environmental mode was approximately 1.4 nm at 30 keV. To investigate the Al_2O_3/polymer structure, 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. A cross-sectional electron micrograph was obtained near the edge of the Al_2O_3/polymer structure as shown in Fig (4a). The image shows the 40-nm Al_2O_3 barrier layer that exhibits pit-like defects between 100 nm and 500 nm in diameter on the surface. In Fig (4b) an image of a typical hole defect, approximately 4 μm in diameter, in the Al_2O_3 layer is shown. 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 penetration.

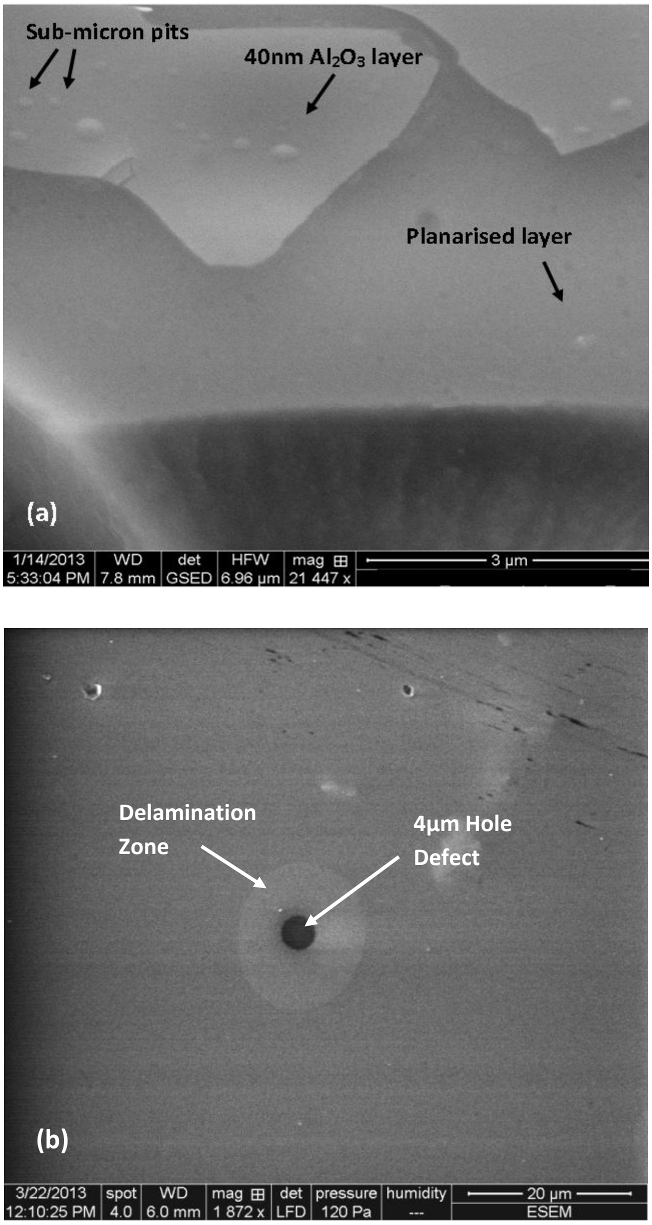

Fig. 4. ESEM of defects in Al_2O_3 barrier layer (a) Al_2O_3/polymer cross-section, (b) typical hole type defect

B. Atomic force microscopy analysis

Although the object targeting and the image acquisition can be more rapid using the SEM, polymers are difficult to examine as they have a tendency to rapidly charge, even with a conductive coating, 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. 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 Dimension Icon AFM was used as an independent measure of the Al_2O_3 ALD film. It allows much smaller defect sizes to be examined than would be possible by the SEM. It can also be used to determine the size of the cracks over the films which were not detectable by the CCI or the optical microscopy techniques. Fig 5 shows crack type defect captured by this technique.

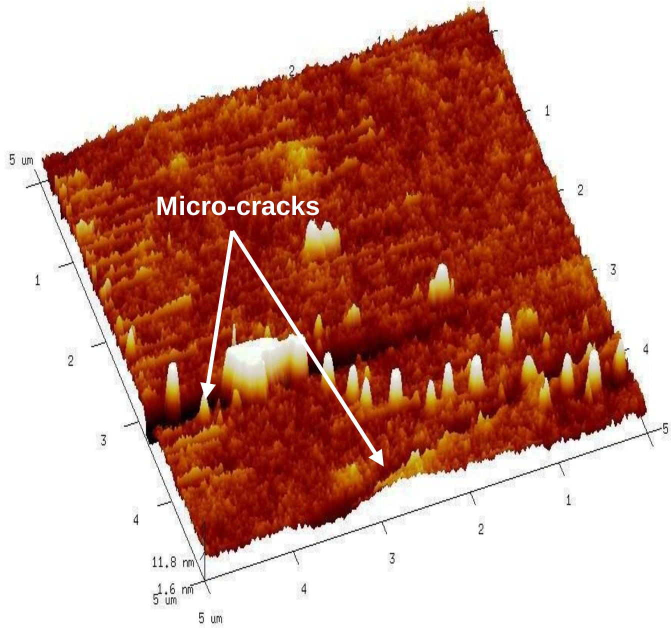

Fig. 5. AFM image-micro-crack type defect

C. White Light Scanning Interferometry analysis

In a previous published study, white light scanning interferometry (Ametek Taylor Hobson) Coherence Correlation Interferometer (CCI) 6000 [6] 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 [2], 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 CCI instrument.

The method of 'Wolf pruning' [7] is then utilised to perform topographic segmentation. This method provides a reliable approach for extracting the features of functional interest by accurately excluding insignificant geometrical features that are induced, such as measurement noise. 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.

V. WVTR RESULTS

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

TABLE II. WVTR RESULTS

Sample No	AlO_x thickness	WVTR ($g/m^2/24$ hrs.)
1	40nm	$5x10^{-4}$
2	40nm	Below detectable level
3	40nm	$1x10^{-3}$
4	40nm	Below detectable level
5	40nm	$6x10^{-4}$
6	40nm	Below detectable level

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

VI. SURFACE TOPOGRAPHY ANALYSIS

The surface roughness average of a defect free sample Fig. 6 was measured to be ~0.6nm. 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 Sq= 0.6nm height and 15µm width. Effective discrimination of significant and non-significant defects was recorded.

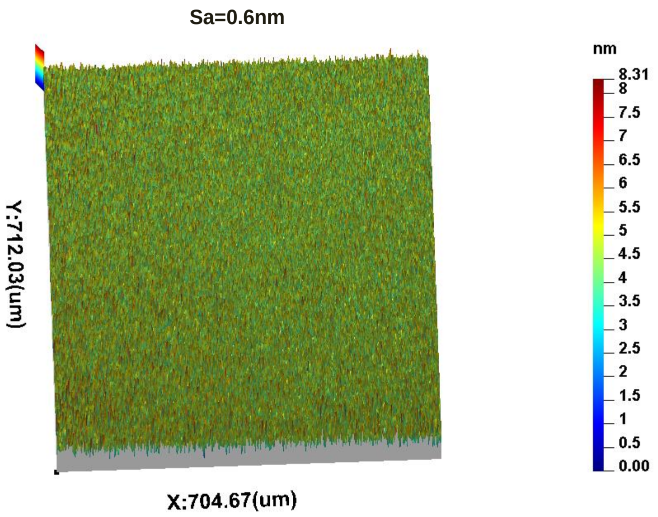

Fig. 6. Non-defective sample (Sa = 0.6nm)

VII. RESULTS DISCUSSION

The results in Fig 7. Showing 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 of (6 σ where Sq= 0.6 nm height and 15µm width) 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 concludes that large defects are the dominant factor for determining the WVTR.

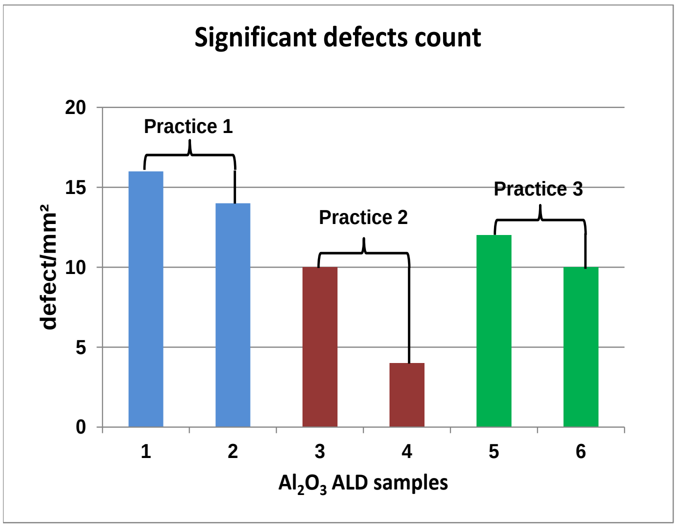

Fig. 7. Shows the significant defects count

VIII. PRE-COATING PROCESS EFFECT

The study 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 MOCON 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 II). 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 ≈ 1×10^{-3} $g/m^2/day$. This sample has larger defects than the other investigated samples (typical example is shown in Fig. 8, which may have had a negative effect on the barrier properties thus giving an increase in the WVTR.

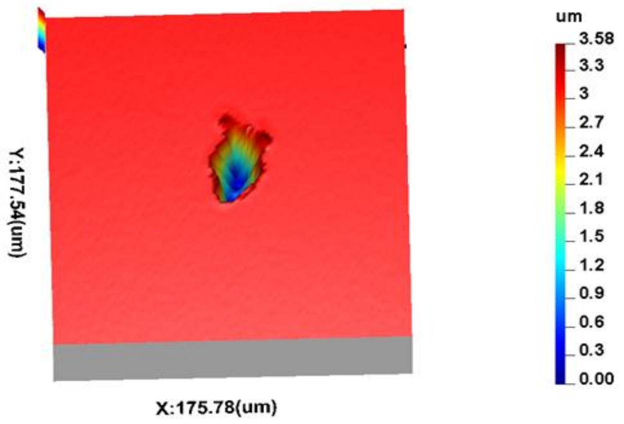

Fig. 8. shows a typical example of large defect

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.

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. 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 1 recommended).

IX. COCLUSION

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.

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REFERENCES

[1] P. F. Carcia, R. S. McLean, and S. Hegedus, "ALD Moisture Barrier for Cu (InGa) Se2 Solar Cells," in *Meeting Abstracts*, 2010, pp. 1435-1435.

[2] M. Igalson and A. Urbaniak, "Defect states in the CIGS solar cells by photocapacitance and deep level optical spectroscopy," *TECHNICAL SCIENCES*, vol. 53, 2005.

[3] P. F. Carcia, R. S. McLean, and S. Hegedus, "Encapsulation of Cu(InGa)Se2 solar cell with Al2O3 thin-film moisture barrier grown by atomic layer deposition," *Solar Energy Materials and Solar Cells*, vol. 94, pp. 2375-2378, 12// 2010.

[4] Thein, M. "Atomic layer deposition (ALD) ". A Tutorial by Cambridge NanoTech Inc. Cambridge, MA 02139 USA, 2006.

[5] J. Wennerberg, "Design and stability of Cu (In, Ga) Se_2-based solar cell modules," Uppsala University, 2002.

[6] W. K. a. C. Lukianowicz, "Coherence Correlation Interferometry in Surface Topography Measurements," in Recent Interferometry Applications in Topography and Astronomy, I. Padron, Ed., ed Poland Koszalin University of Technology, 2012.

[7] ISO25178-2, "Geometrical product specifications (GPS) -- Surface texture: Areal -- Part 2: Terms, definitions and surface texture parameters," 2 ed, 2012.