

Prof Liam Blunt

In process surface metrology for roll to roll manufacture of printed electronic devices

EPSRC Future Metrology Hub
University of Huddersfield, UK



Integrated Metrology for Precision Manufacturing Conference
22 - 23 January 2019, Knowledge Transfer Centre
Advanced Manufacturing Research Centre, Sheffield, UK

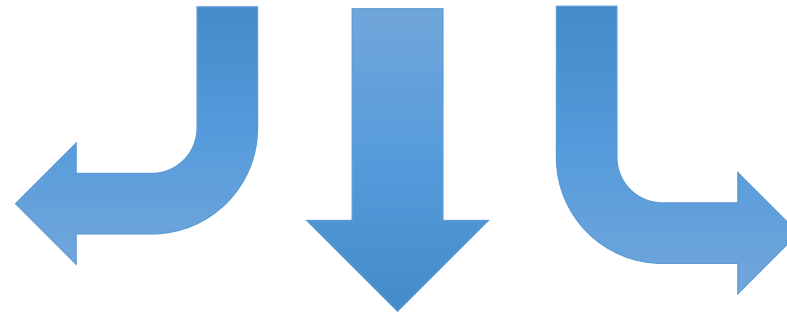
High Added Value Manufacture

Manufacturing contributes over \$11tr to the global economy¹

High value manufacturing is the application of leading-edge technical knowledge and expertise to the creation of products, production processes, and associated services which have strong potential to bring sustainable growth and high economic value to developed economies

Specific Technology Drivers

Products are usually high precision

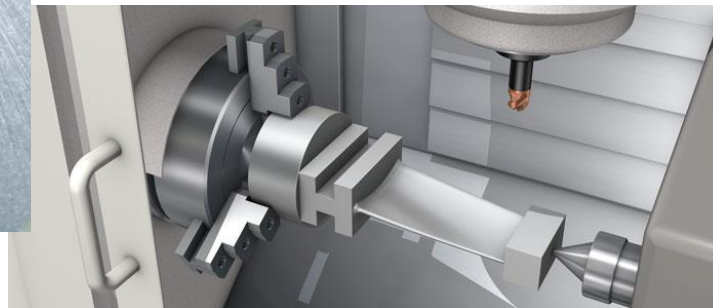


Expensive production facilities

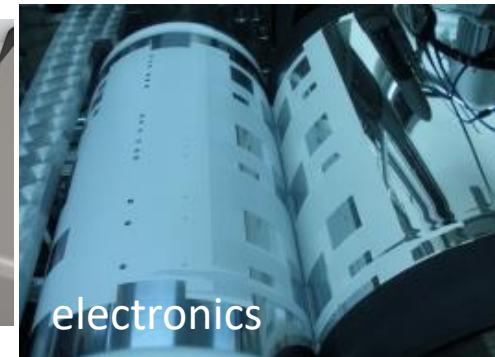
In process metrology desirable for maintaining quality with fast throughput



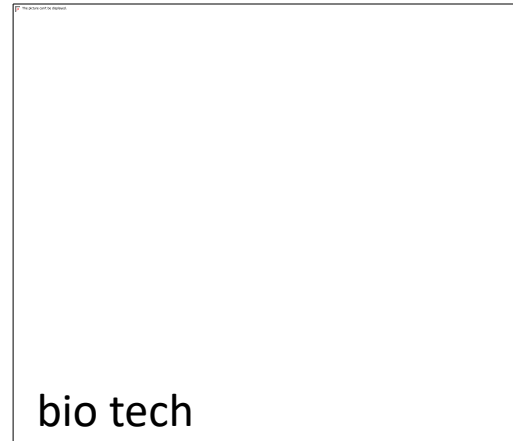
optics



aerospace



electronics



bio tech

¹ United Nations Statistics Division, United Nations National Accounts Main Aggregates Database, Value added by Country, 2010

Sensor/Instruments for future Manufacturing

- Contained within a machine tool/production line
- **Robust in the manufacturing environment**
- Achieve the same level of accuracy as lab-based measurement systems

Ideal Measurement systems

- ***Speed: More than KHz***
- ***Robustness: Operate in dynamic manufacturing systems***
- ***Sensor/instrument size: Miniaturisation***
- ***Measurement ratio: Coverage; high dynamic range***
- ***Accuracy & traceability: Same level as a standard lab instrument***
- ***Cost: Relatively inexpensive***

In Process Surface Metrology Challenges ||||| The Future

Measurement Speeds matched to process
< 500m/min or even kHz,

*Ultra fast camera technology and powerful
light source power (LUX)*

Calibration/verification
in situ

Challenging but feasible with clever design

Environmental effects

*Reduce effects, use enclosures, cleverer design,
fast acquisition, compensation*



Too much data to
handle!



Try to use only what is really important!

Exemplar case study;

Manufacture of printed electronic devices

- Printed Flexible photovoltaic cells
- In-process measurement of – protective *barrier coatings*
- Functional Specification for Barrier Properties
 - Water Vapour Transmission Rate WVTR
- Defect classification and correlation with function
 - Advanced Surface Metrology
- In-process measurement solution.....

Nanomend



||||| The Future



EU project: To develop technologies that are able to detect and correct micro and nano-scale defects in Roll to Roll produced Photo Voltaic films and polymer coated fibre products, without slowing production speed.

Roll to Roll Manufacture?



Roll-to-roll processing (R2R), is the process of creating electronic devices on a roll of flexible polymer sheet or metal foil. It refers to any process of applying coatings, printing and joining foils.

The process starts with a rolls of flexible material and involves re reeling after processing to create an output roll.

Metrology and Cleaning/Repair needs to integrate with film processing e.g. post ALD atomic layer deposition of Al_2O_3 (40nm)

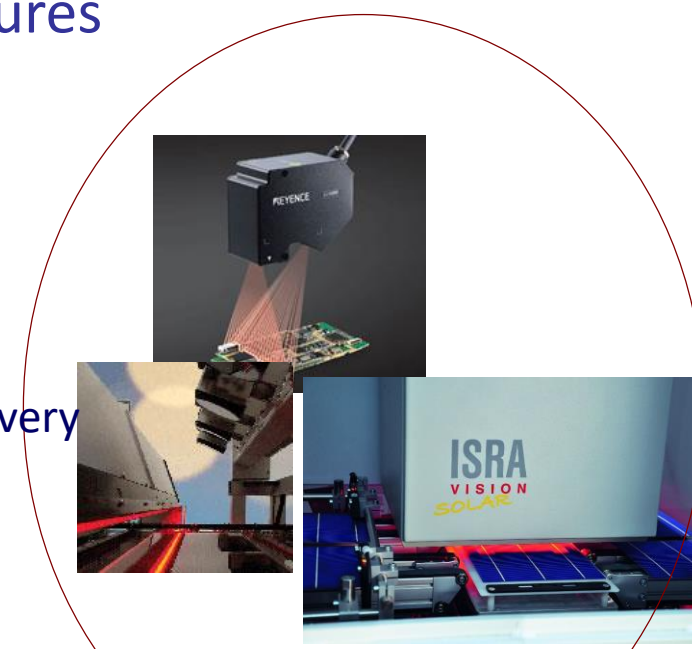
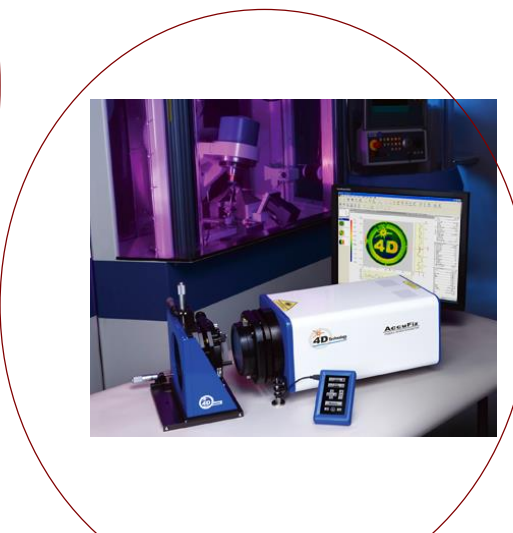
Conventional Measurement Solutions

A number solutions have derived from lab based instrument manufacturers or are camera based looking for specific targeted features

Robust conventional instrumentation
(expensive, slow!)



Fast acquisition! ~30μsec (very expensive)



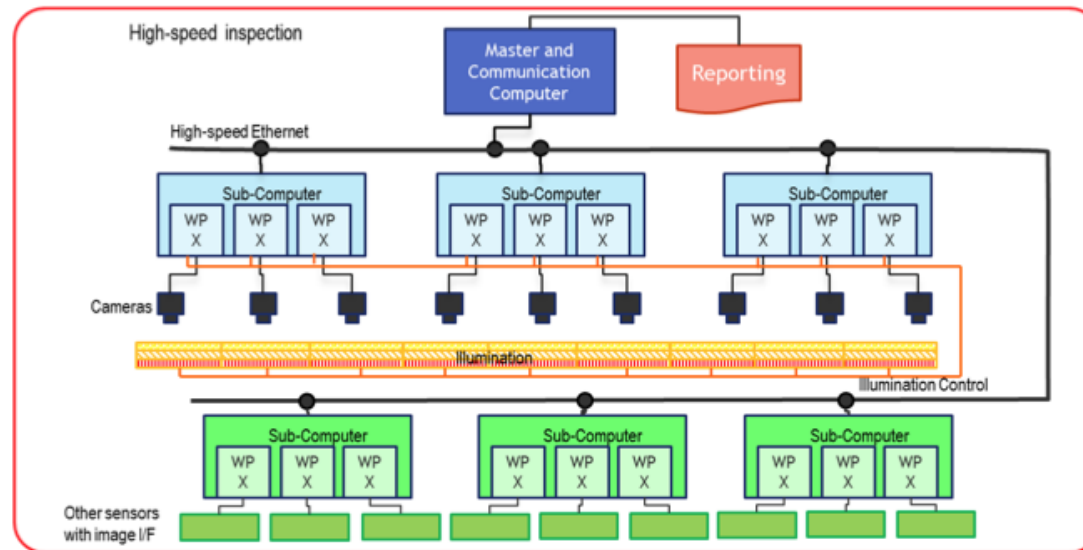
High res, high speed cameras
(less expensive low resolution)



Roll2 Roll Surface Measurement

Need multiple or moving sensors to give roll coverage BUT :-

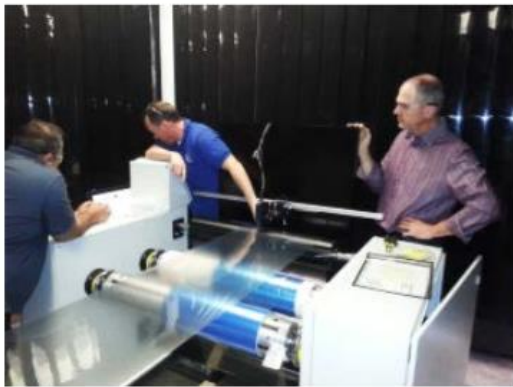
- Sensors need registering in a single coordinate system.
- Sensors need overall control
- Targeted on specific surface defects/characteristics
- Individual sensors relatively inexpensive
- Generate **masses** of redundant data multiple TB/min



Courtesy of ISRA Vision

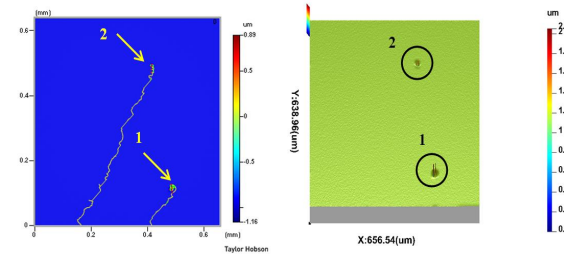
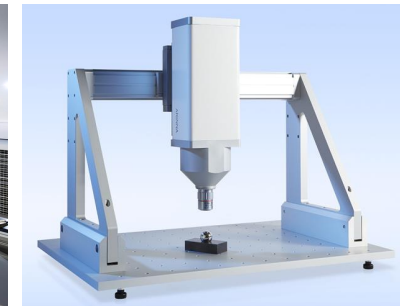
New Generation Solutions Arising

FlexCam 4D Technologies, USA



Vitriflex presentation from AIMCAL conference 2014.

WSI (IBSPE, Huddersfield NL/UK)



NanoMend

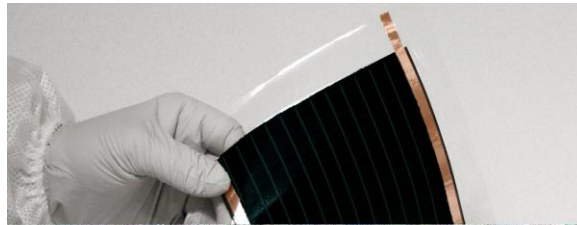
Photovoltaics, Rigid and Flexible

||||| The Future



Rigid Si based Photo
Voltaics (expensive/m²)

(crystalline or amorphous Si)

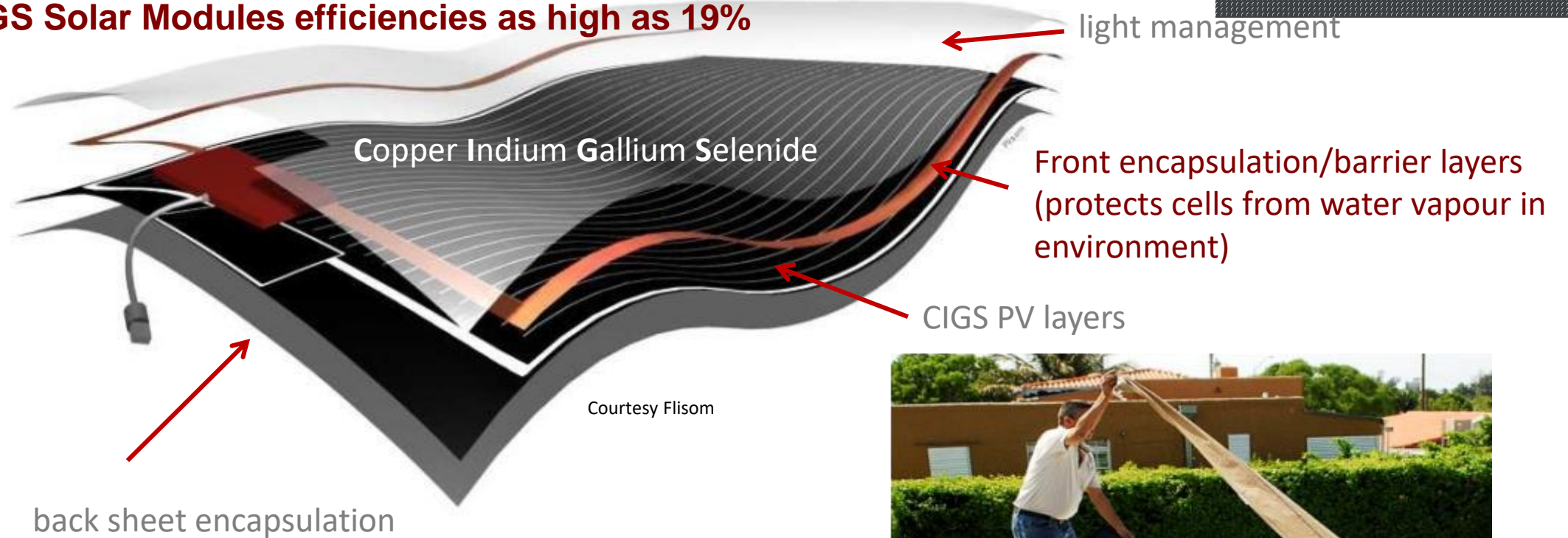


Flexible
Photovoltaics
(CIGS) inexpensive/m²



Flexible Solar Modules, CIGS; basic layer groupings

- **Highly suitable for Building Integration**
- **Latest CIGS Solar Modules efficiencies as high as 19%**

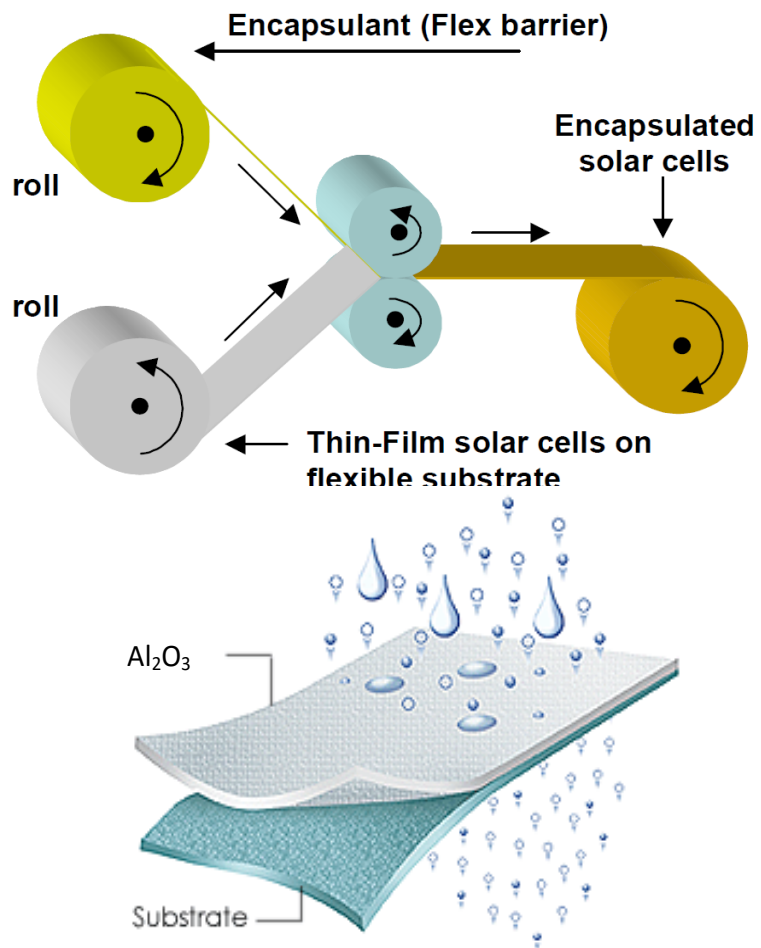


Front sheer barrier/encapsulation layer is the most expensive element per m².



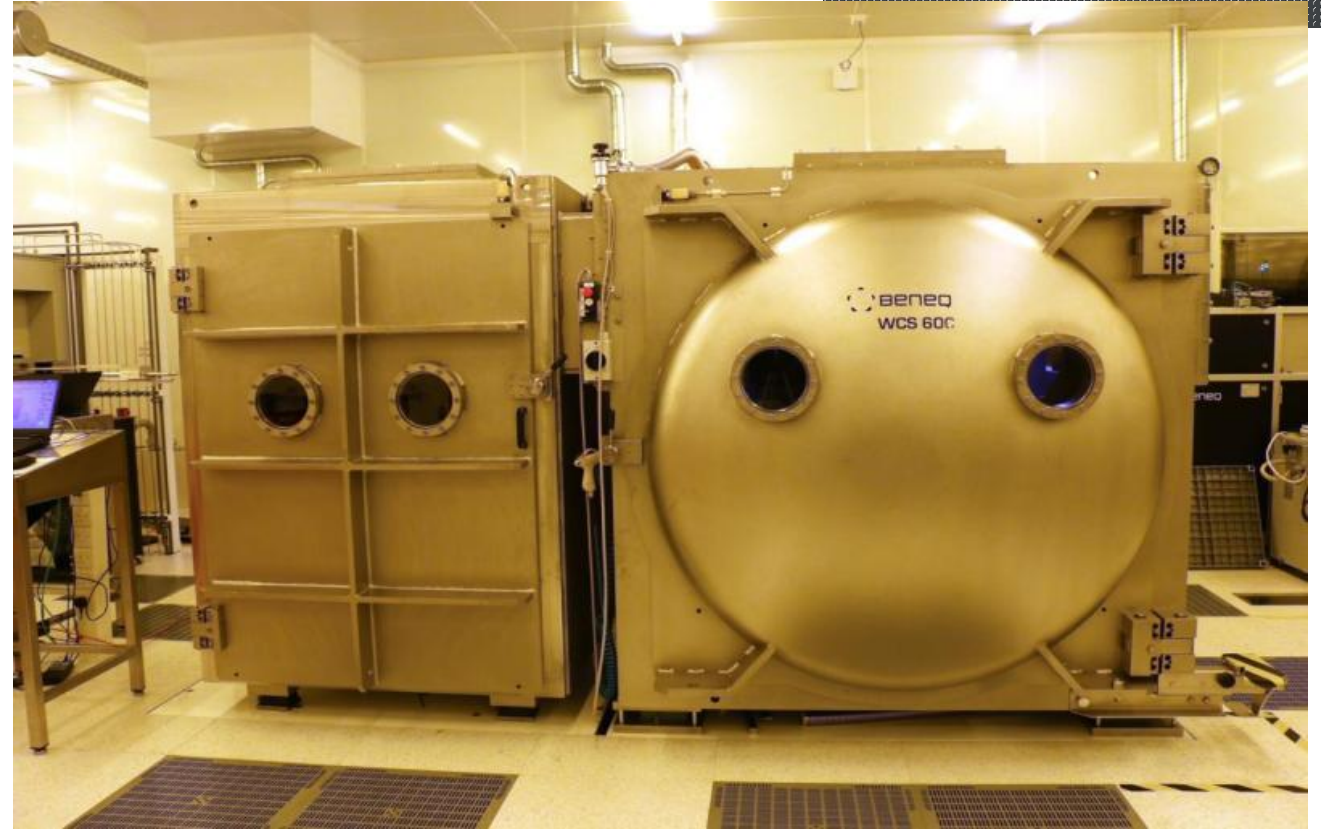
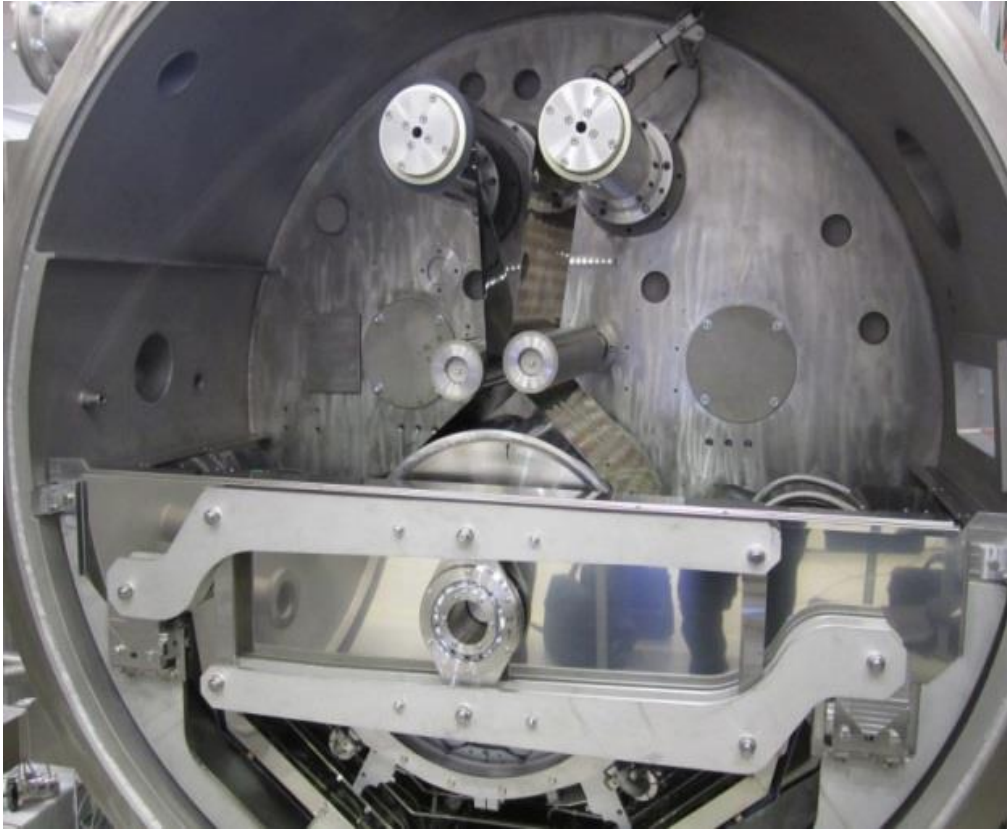
Flexible PV Modules

- Flexible PV modules are manufactured using roll to roll (R2R) technology.
- BUT these modules require a flexible barrier material to prevent water vapour ingress.



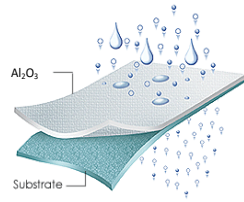
Roll2Roll Atomic Layer Deposition for Barrier Layers, Single Al_2O_3 layer 40nm thick

||||| The Future

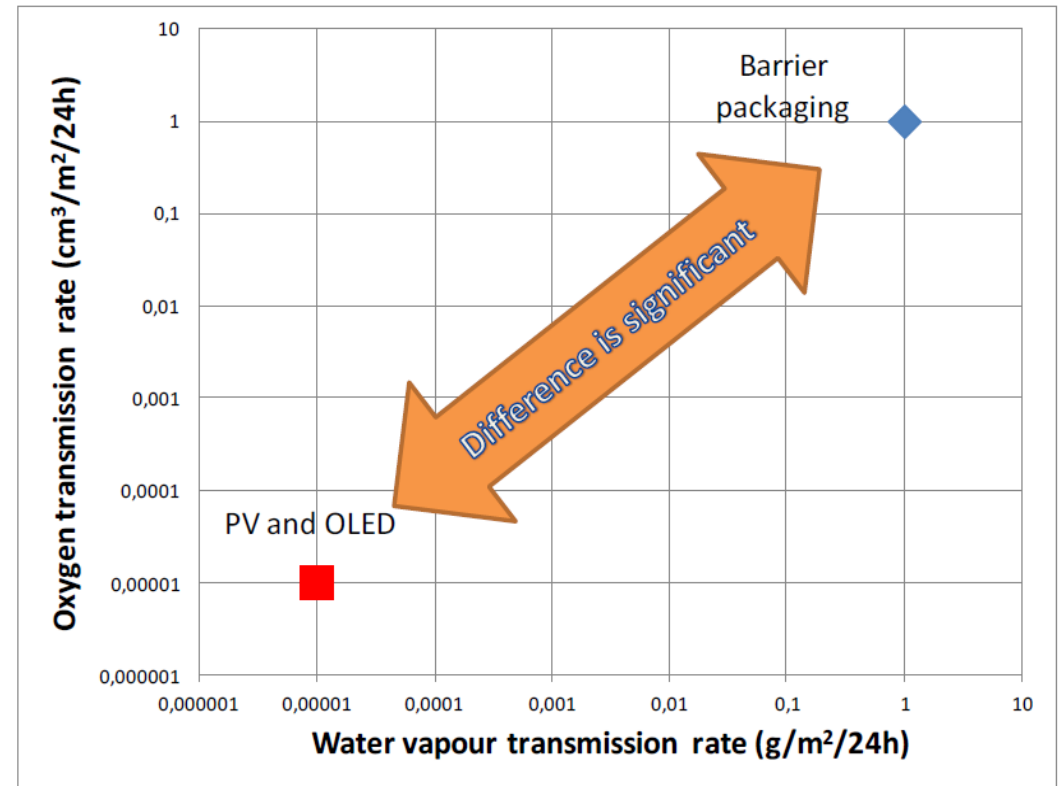


World first demonstration in NanoMend

PV Encapsulation Requirements



- 20 years lifetime needs WVTR @ $<10^{-4}$ g/m²/day.
- Current single barrier layer capable of $\sim 10^{-1}$ g/m²/day.
- A robust, improved and transparent flexible encapsulation method for flexible PV modules is needed.



IEC61646-2, "British Standards Institution," in *Thin-film terrestrial photovoltaic (PV) modules – Design qualification and type approval* ed: BSI, 2008, pp. 5-36.

WVTR= Water Vapour Transmission Rate

Manufacture of single flexible barrier layer for PV's

Requires; efficient in-process metrology based on understanding and modeling the function of the the surface to be measured;



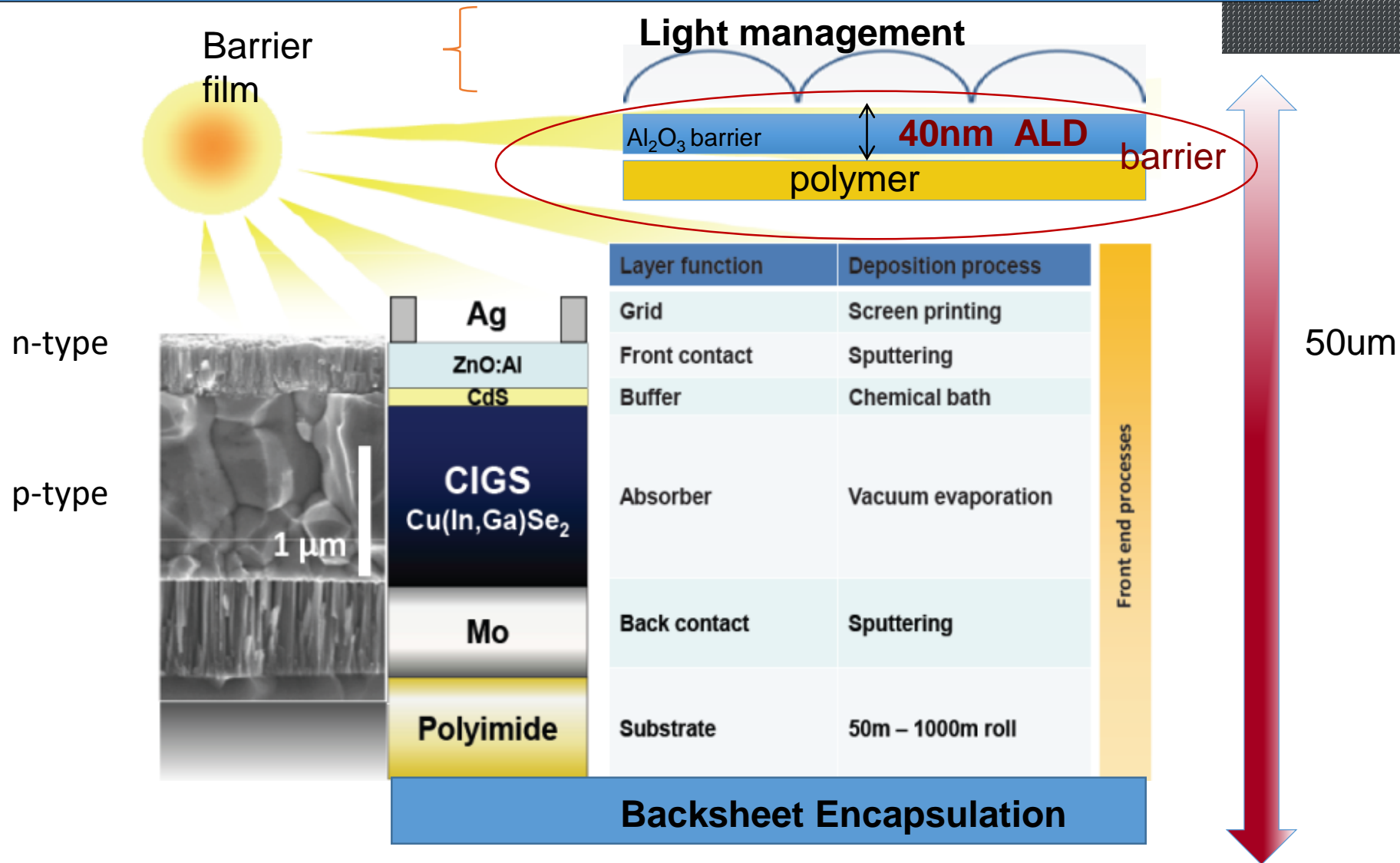
Investigation and correlation of **Water Vapour Transmission Rates (WVTR)** through defects in barrier substrate as applied to flexible PV's.

Facilitates in process metrology in R2R manufacture

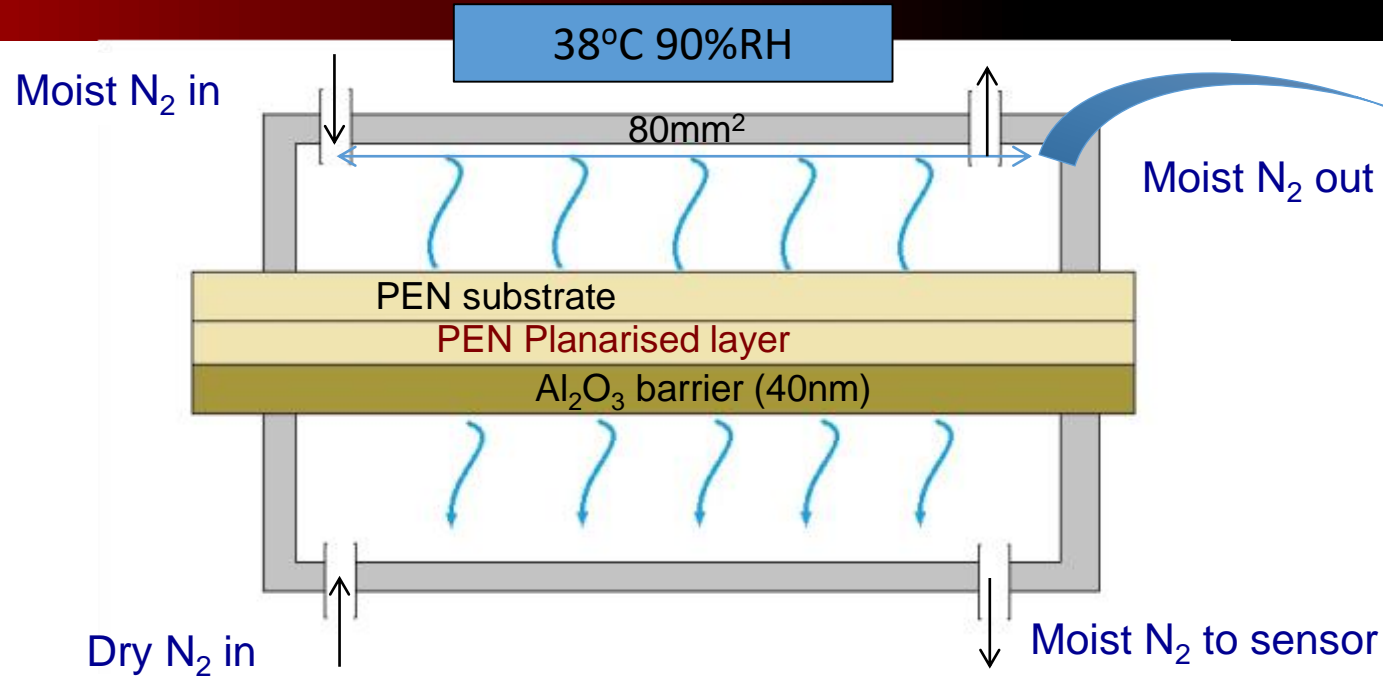
- Measured and catalogue defects
- Measuring and modeling which defects are most functionally significant
- Tailoring the process measurement

Functional elements of flexible photovoltaic cells

Al₂O₃ barrier layer produced by atomic layer deposition ALD (290 cycles)



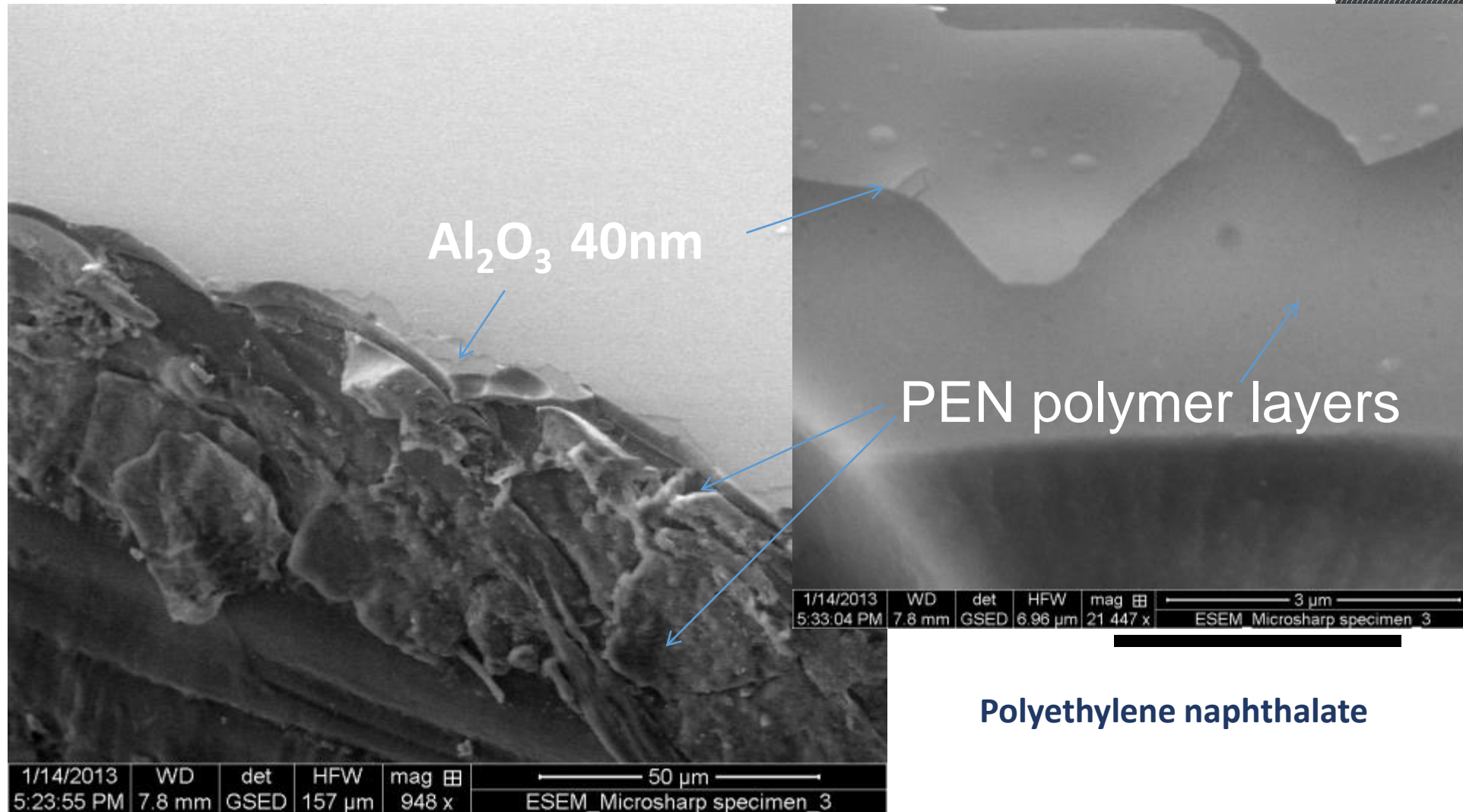
Water Vapour Transmission



Typical result set

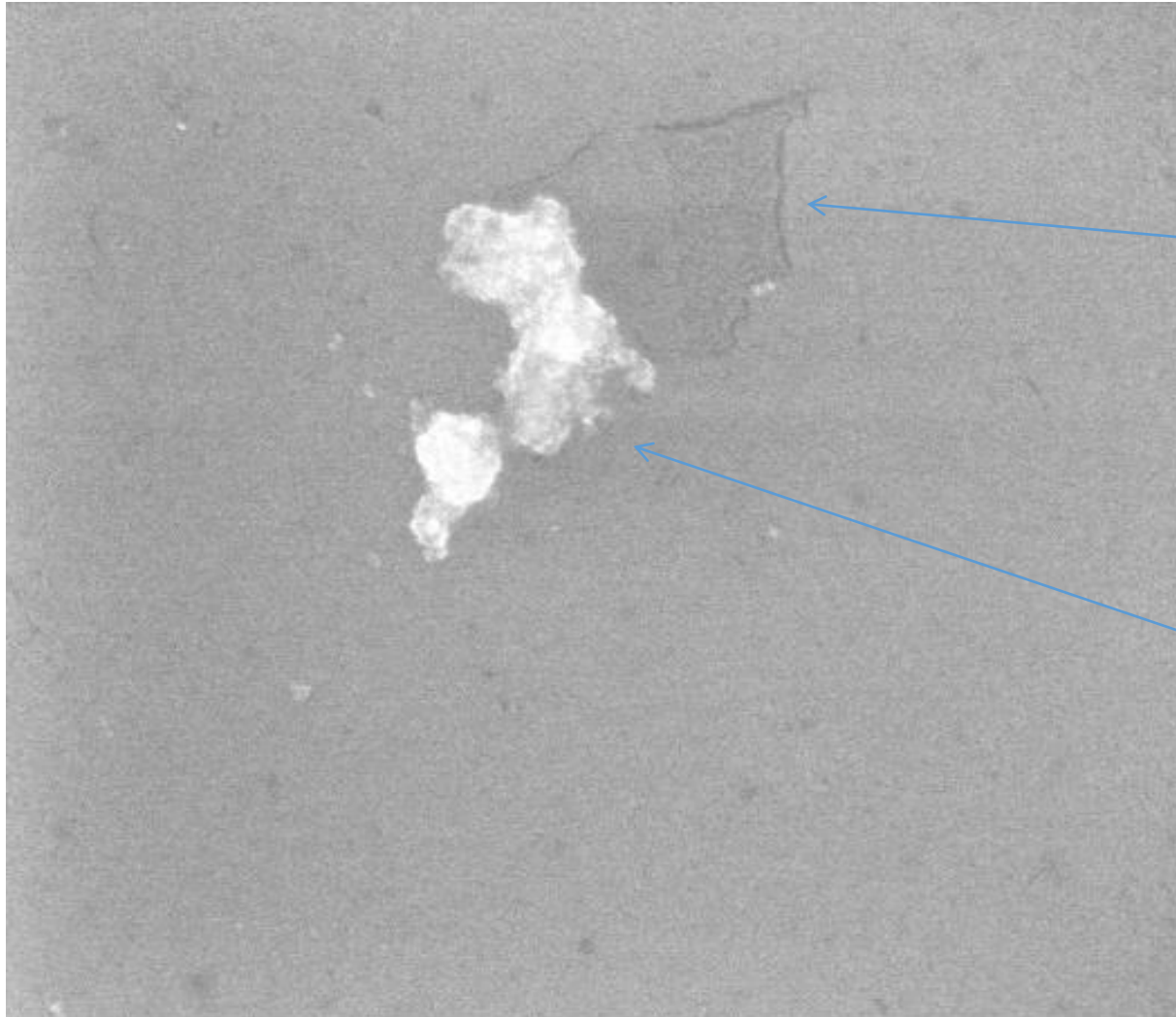
Sample	Water vapour transmission rate (g/m ² /day)	Stabalisation time (days)
2701	1.1x10 ⁻³	11
2702	1.3x10 ⁻³	11
2705	4.1x10⁻³	5
2706	2.0x10 ⁻³	5

Al₂O₃ by Atomic Layer Deposition (ALD) 40nm thickness (ESEM)



Polyethylene naphthalate

Defects: Particles and Cracks

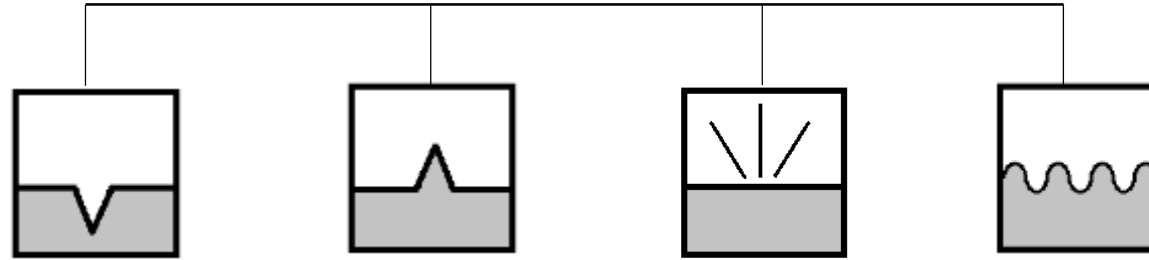


Cracking

Particulate debris

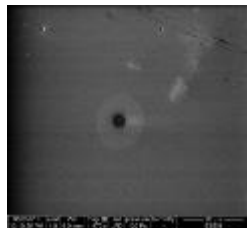
WD	mag	pressure	vac mode	HV	spot	2 μm
6.6 mm	27 917 x	200 Pa	ESEM	10.00 kV	3.0	ESEM Al2O3 2702

Defect Classification System



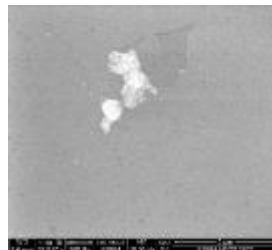
Inwardly directed defect

- Pin holes
- Holes
- Cracks/scratches



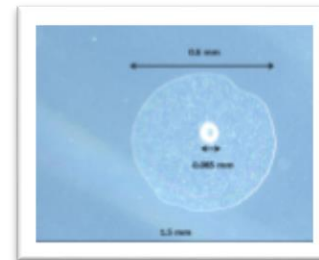
Outwardly directed defect

- Particulate debris



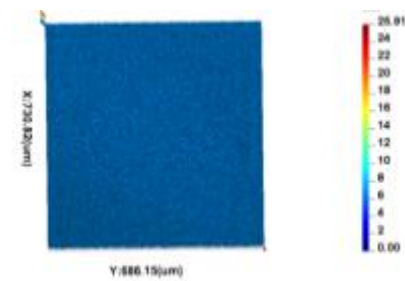
Differing appearance to surroundings

- Delamination



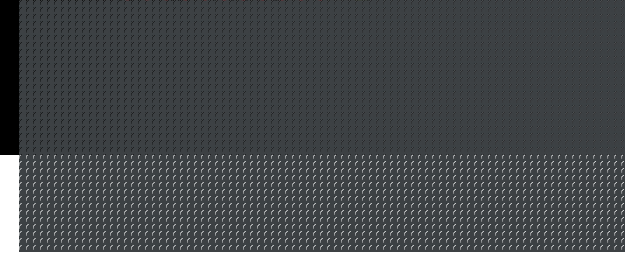
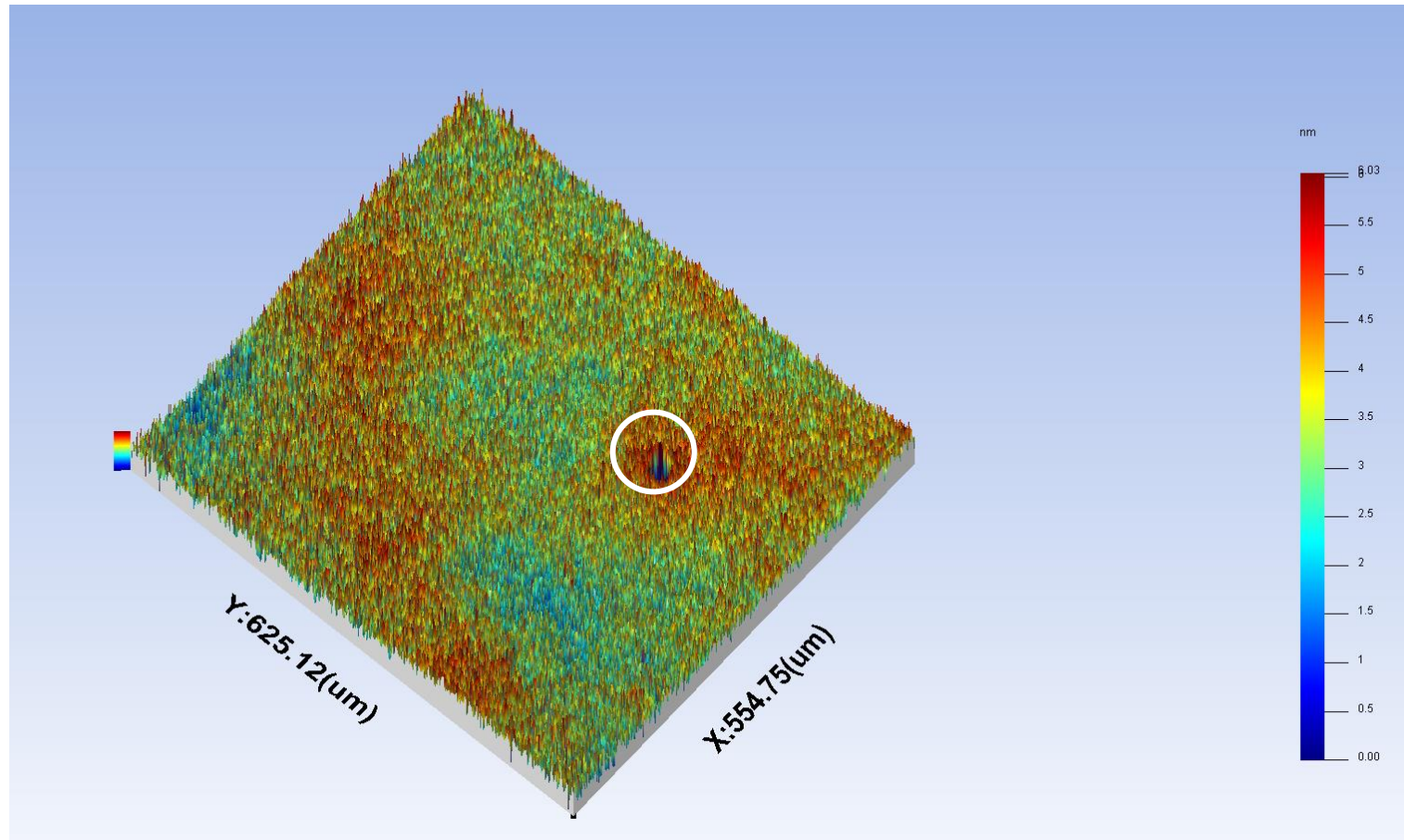
Surface relief

- High roughness



Surface measurement of barrier coating Optical Interferometer

The Future



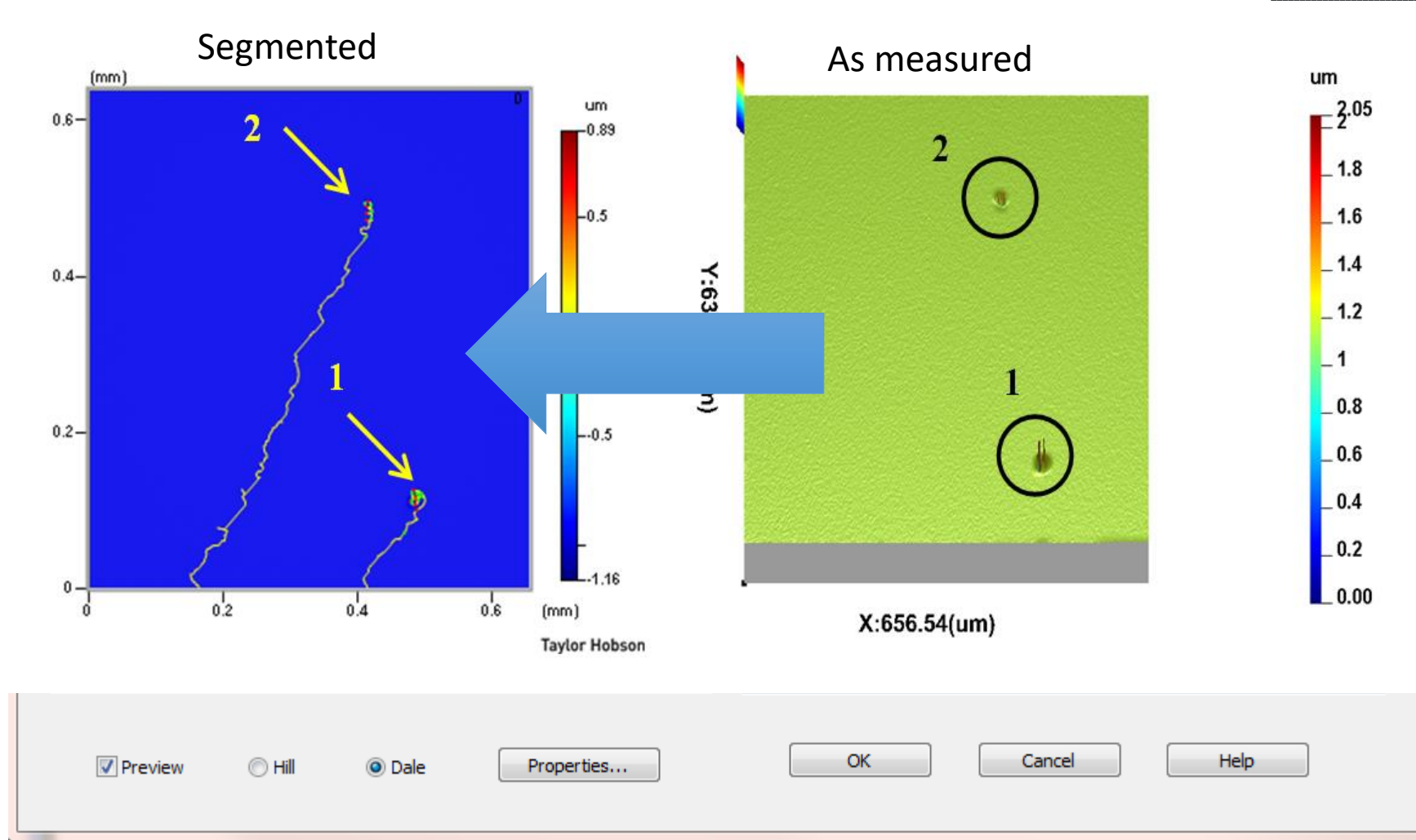
Surface Texture Standards

Roughness Parameters (ISO 25178)

Roughness Parameter	Physical meaning	
Sa/ μm	Average areal surface roughness height	
Sq/ μm	Areal root means square roughness height	
Ssk	Skewness of surface height distribution (-ve = valley dominated)	
Spd/ mm^2	Density of Peaks	Feature parameters
Sdd/ mm^2	Density of dales (pits)	
Ssd/ mm^2	Density of significant defects Ssd = Sdd + Spd	

- ◆ 15% Area coverage ~ on each substrate, 3500 measurements!
- ◆ coherence correlation interferometry

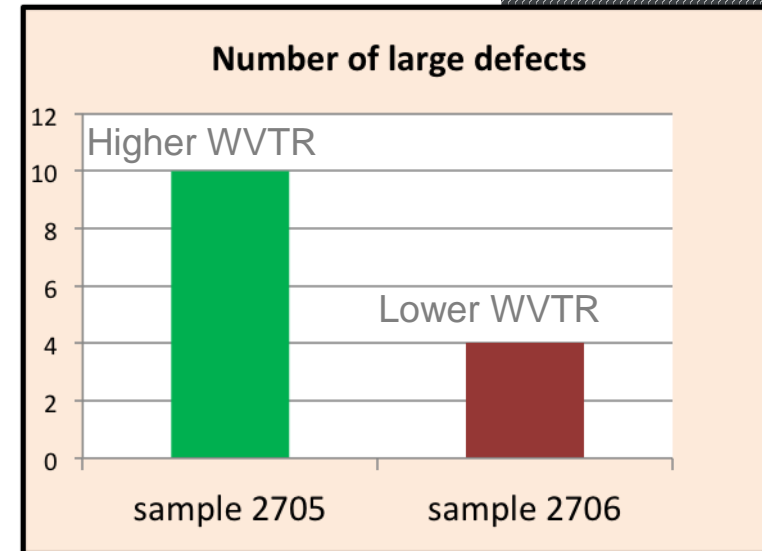
Feature Parameter Software ISO 28178



Defect Characterisation Trial :- Feature parameters

ISO 25178

Only significant defects (6σ ($Sq=\sigma=0.8nm$) height & width $> 5\mu m$) (i.e. area and height pruning/segmentation) considered significant.

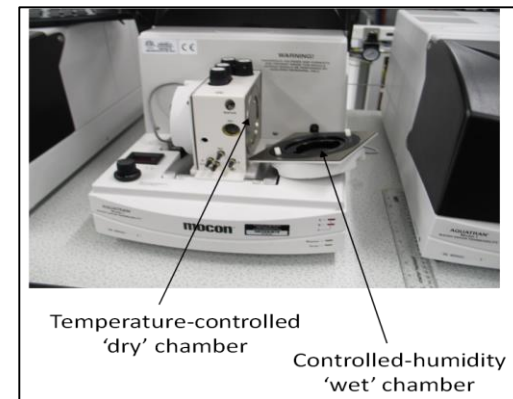
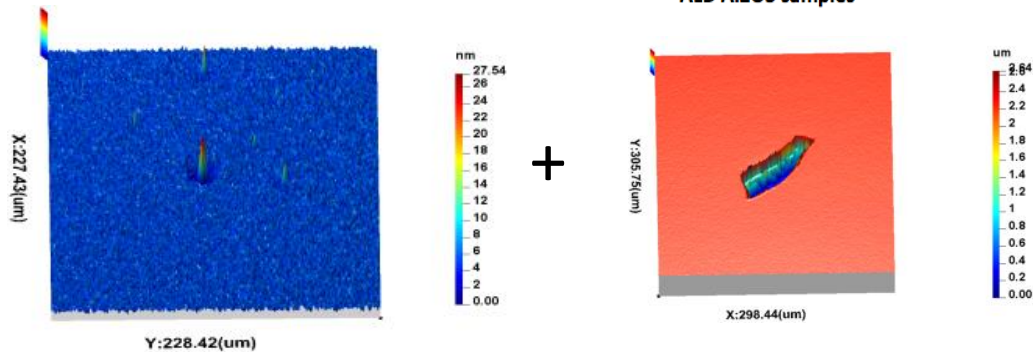
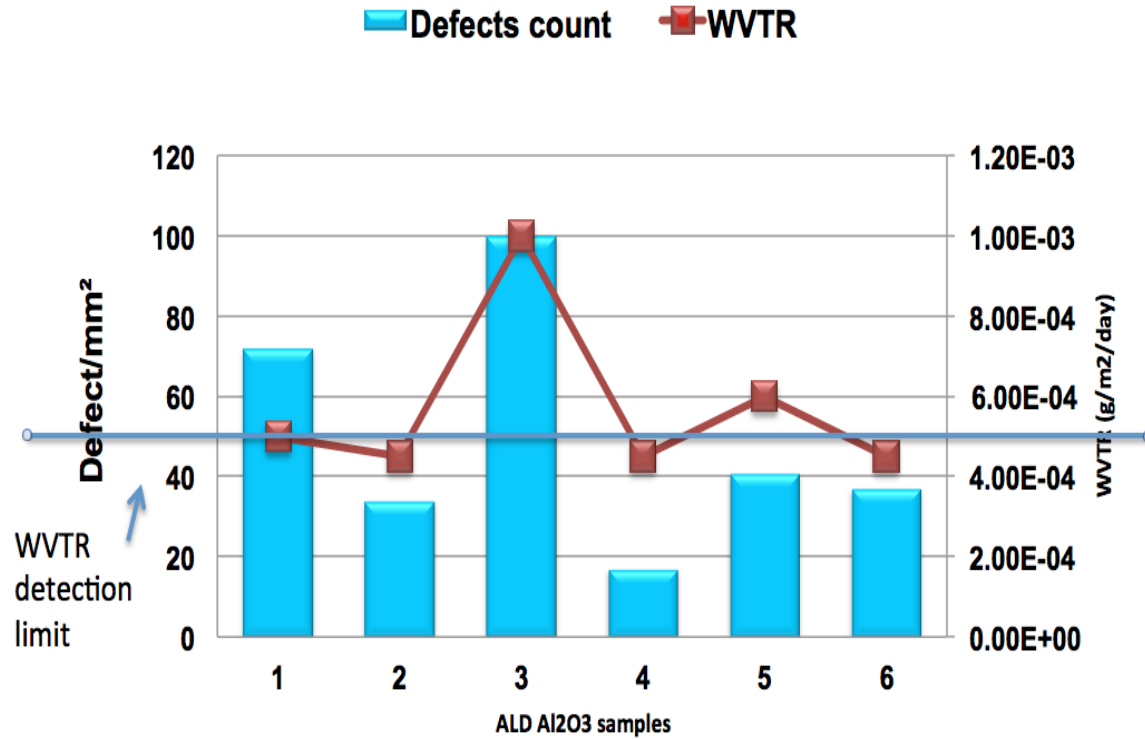


- Sample 2705 has 10 large defects; 5 dales + 5 peaks
- Sample 2706 has 4 large defects 2 dales + 2 peaks

- The presence of small numbers of larger defects seems to correlate with WVTR (no clear distinction between peaks and dales)
- Exercise repeated on a several sample sets with same results

Functional Correlation Defects of WVTR Trial

The Future



Modeling the WVTR

Can a model approach confirm the experimental WVTR results?

From Da Silva Sobrinho: amount of permeant traversing the polymer and through the hole per unit time, Q

$$Q = \frac{q_H}{t} = \frac{\rho R_0^2 D \Delta c}{L}$$

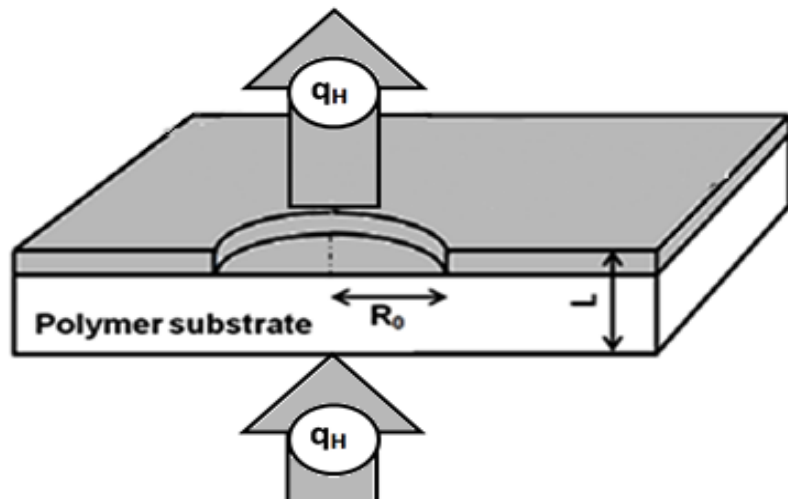
Where R_0 is the hole radius, D is the diffusion coefficient of the film (cm^2/s), Δc is the water vapour concentration (g/cm^3) and L is the combined film thickness, q_H amount of the water vapour, leaving the barrier film

$$WVTR = \frac{Q}{A} \quad (\text{g} / \text{m}^2 / \text{day})$$

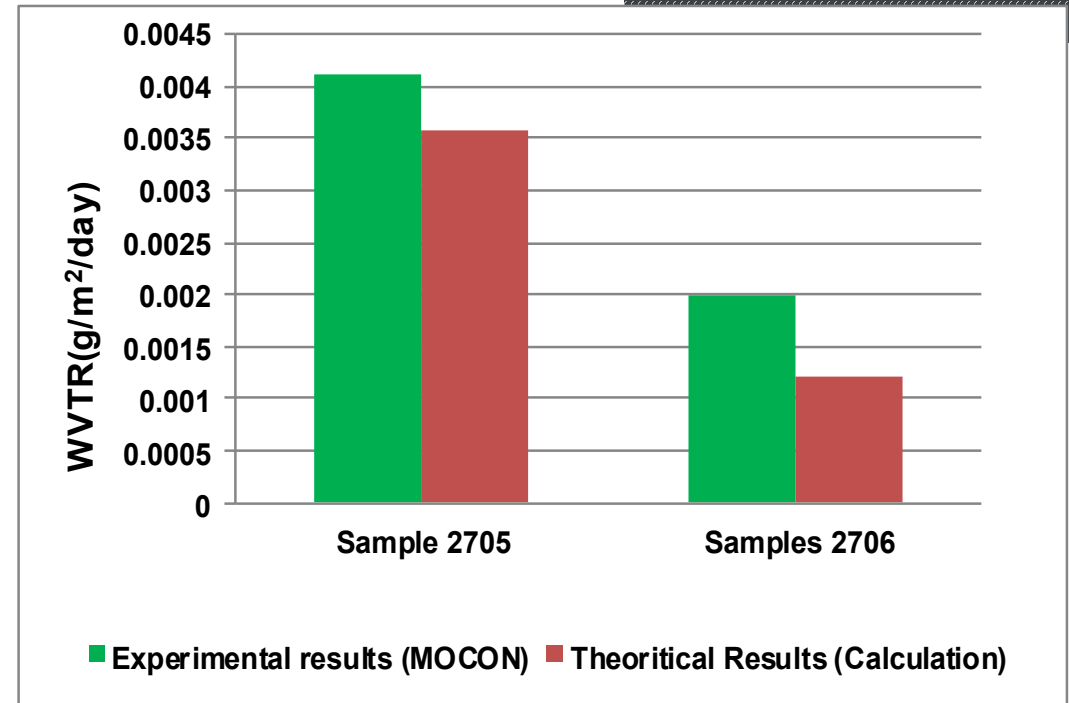
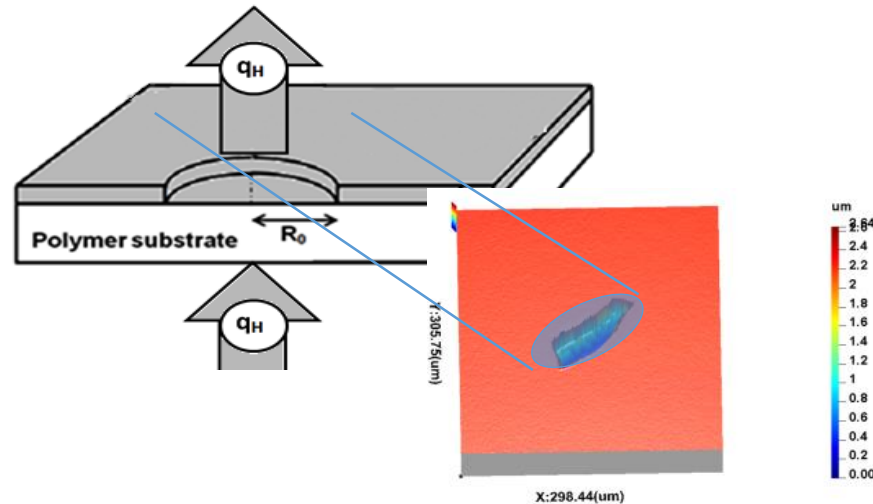
Where; Q is the amount of the water vapour passing through a film at the defect location where the thickness is L and area A during time t being driven by a partial pressure differential P across the film

For many defects (real case) then

$$WVTR = \int_0^N \left(\frac{Q}{A} \right) N$$



Modeling the WVTR



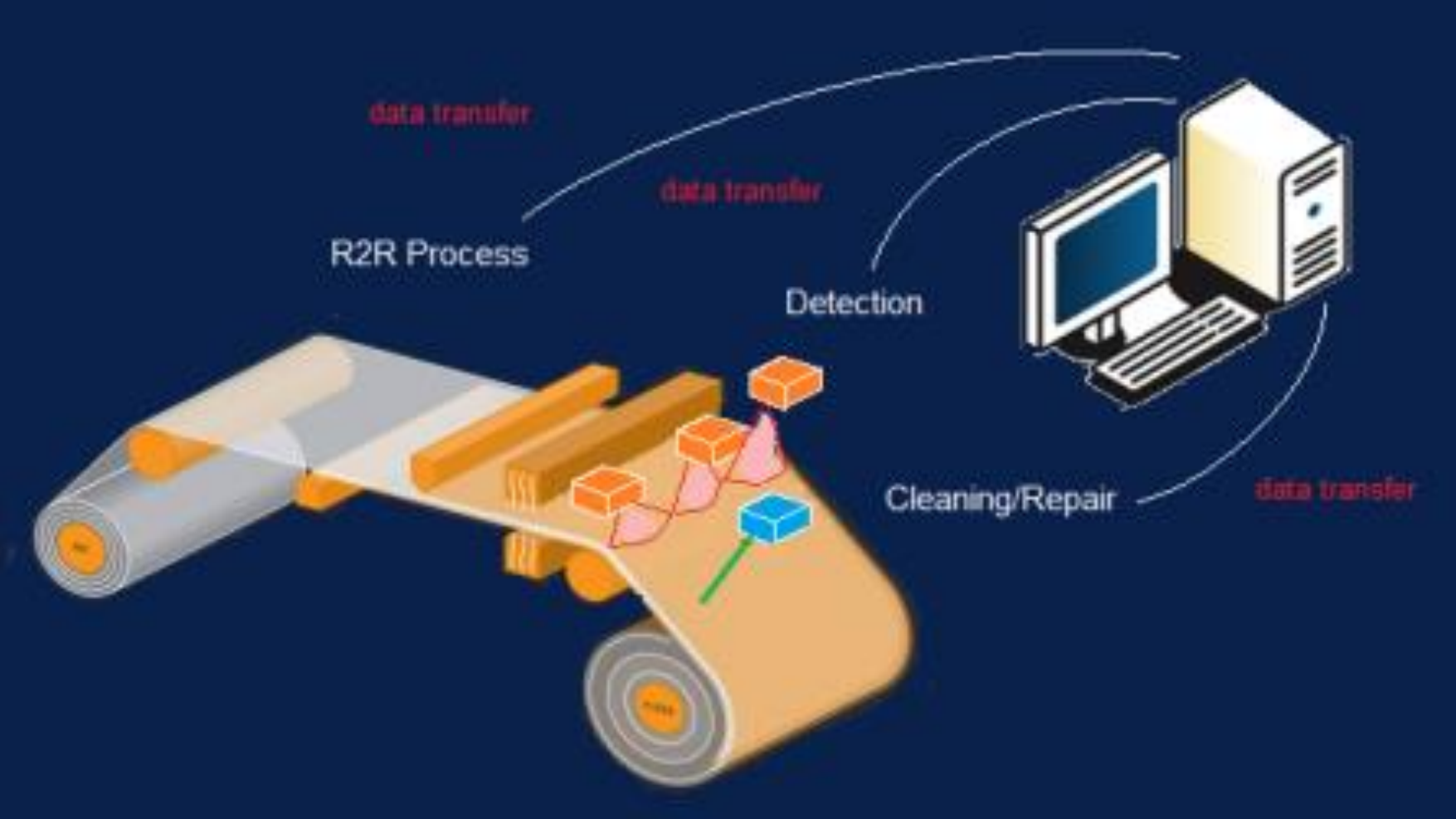
For many defects (real case) then

$$WVTR = \dot{a}_0^N \left(\frac{Q}{A} \right) N$$

Using surface area of defects calculated from the surface maps give a good correlation

Concentrate on measuring functionally significant defects! < 3um diameter

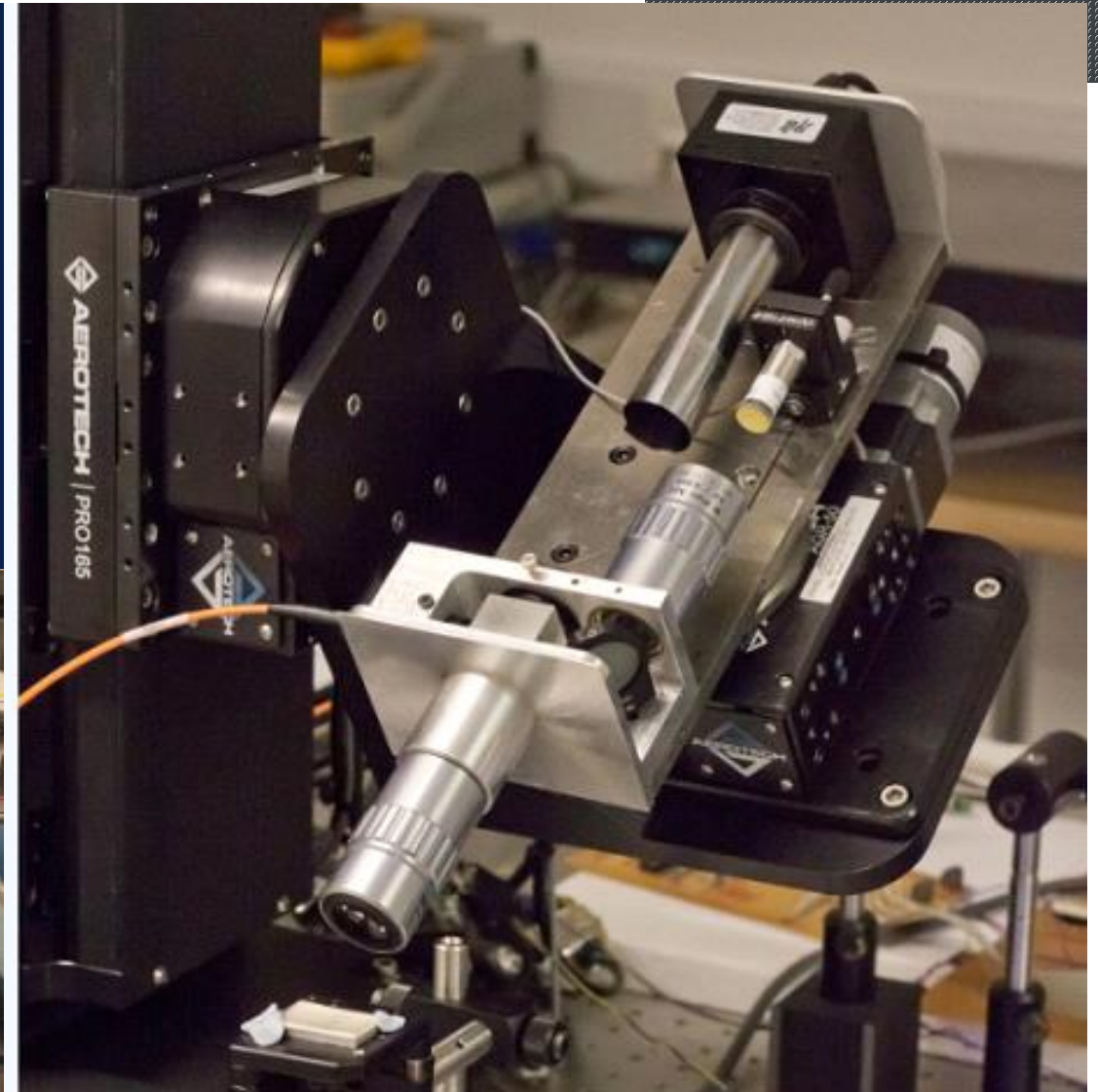
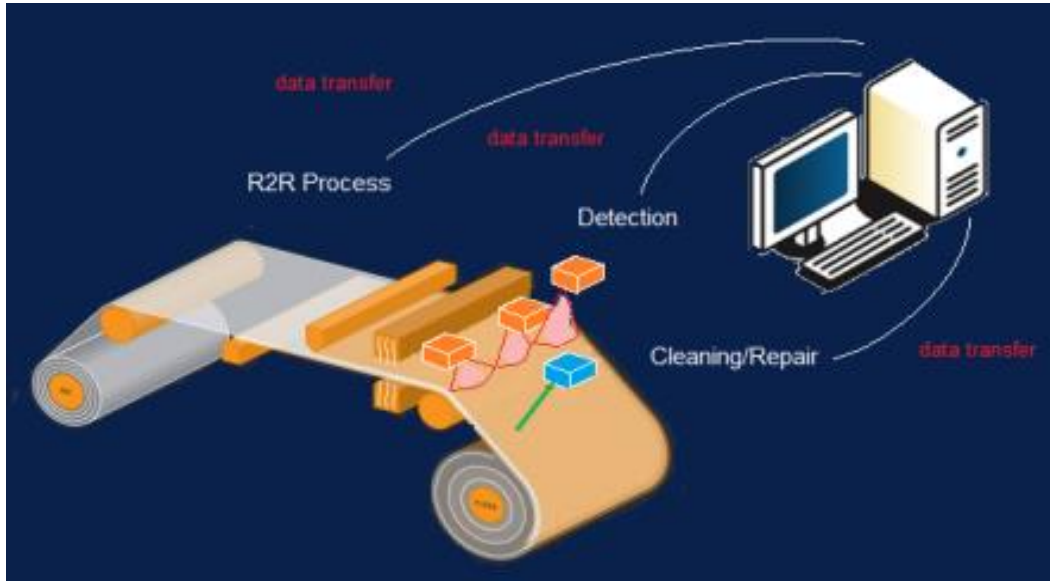
In-process Metrology :- Proof of Concept



In-process Metrology:- Requirements

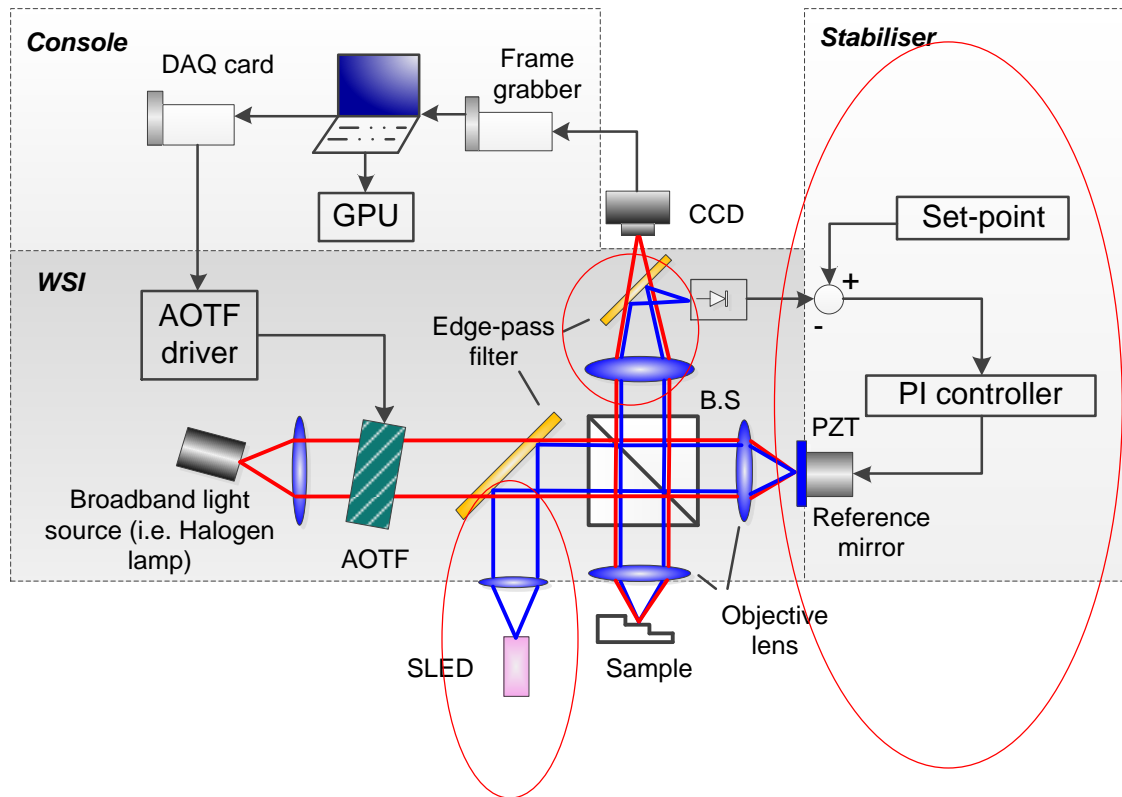
- Non Contact
- Fast acquisition ≤ 0.5 sec
- DYNAMIC BETTER
- Robust to environmental vibrations
- Substrate vertical position control
- Auto-focus of optics
- On line calibration
- Massive amounts of data!! (1 strip 900Mb)

In Process Systems; Wavelength Scanning Interferometry, WSI

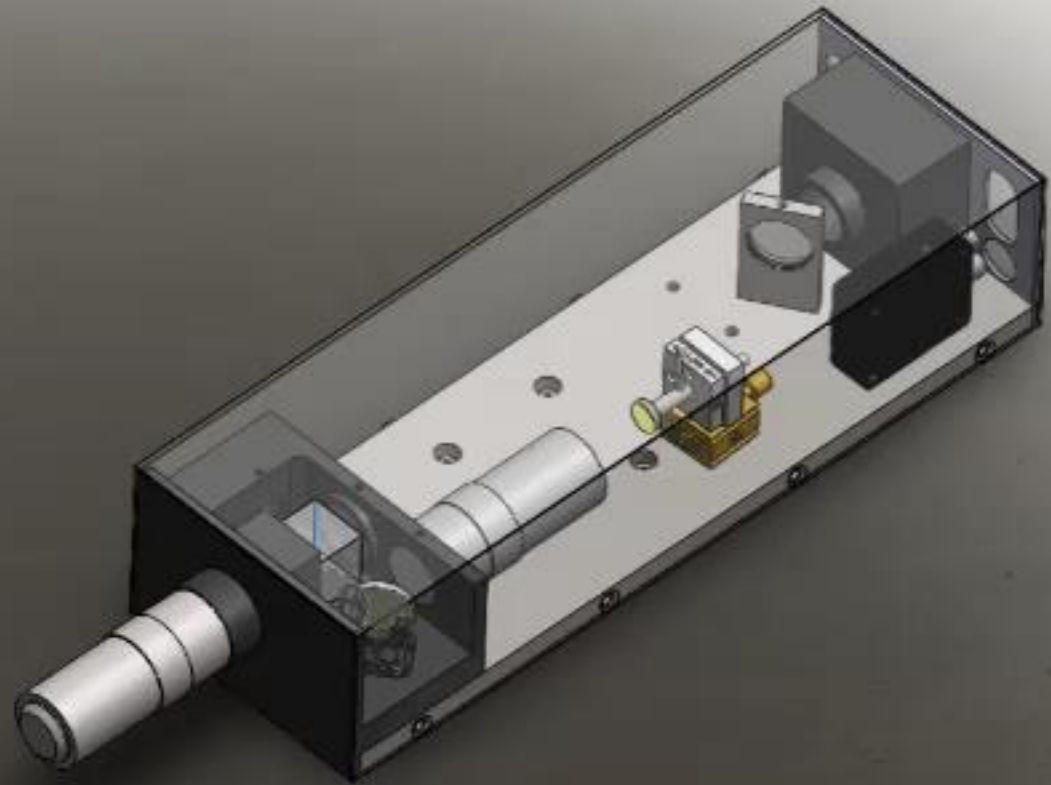


Wavelength Scanning Interferometer

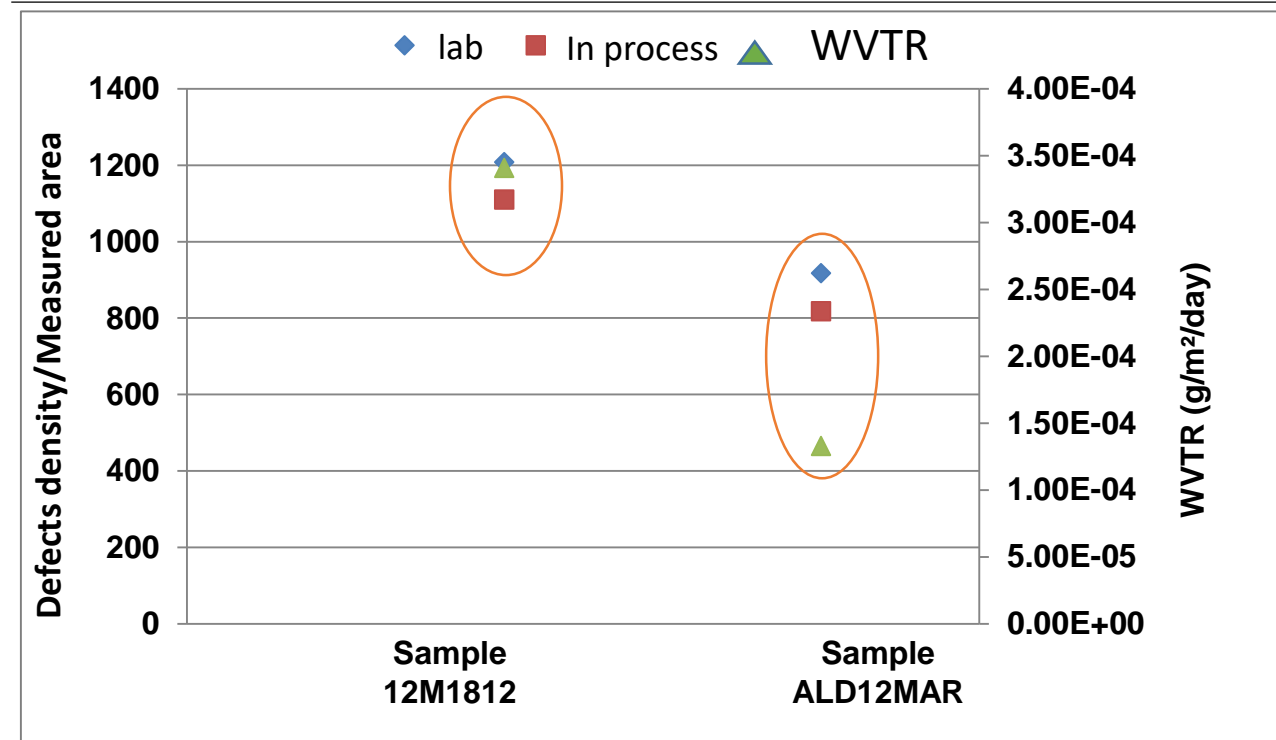
Static Measurement < 2secs



- Varying the wavelength of the incident light using AOTF (530-680nm) 1s acquisition (Acousto-Optic Tunable Filters)
- Dual path system (IR and visible) measurement allows for compensation of environmental vibrations
- GPU and CUDA processing of interferograms



Defects density (2 techniques) vs. WVTR (Trial 3)

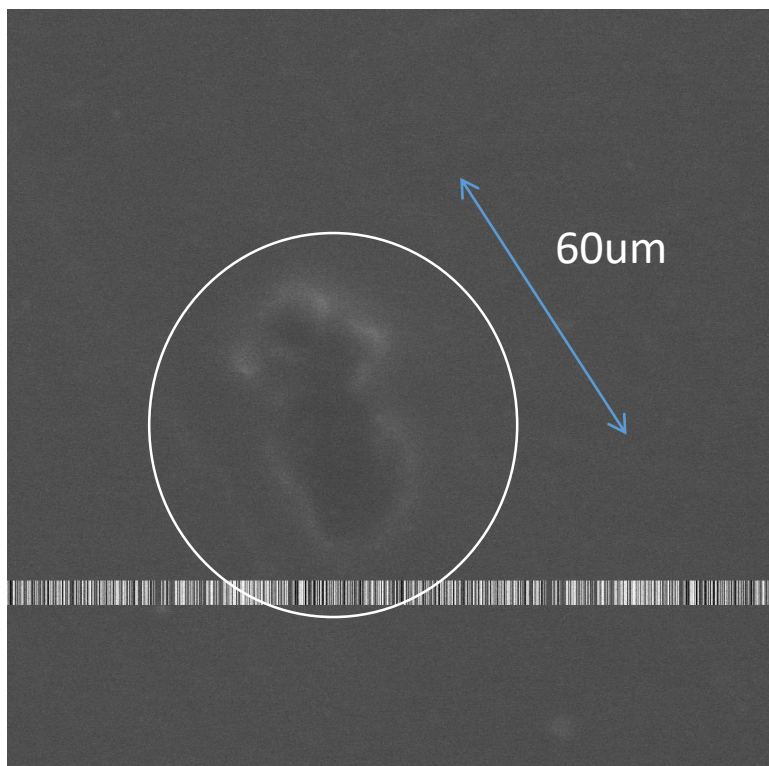


The analysis of the results appears to indicate that the sample with higher density of defects ($> 3 \mu\text{m}$ lateral spacing) exhibits inferior barrier properties. Clear difference in density values for two techniques

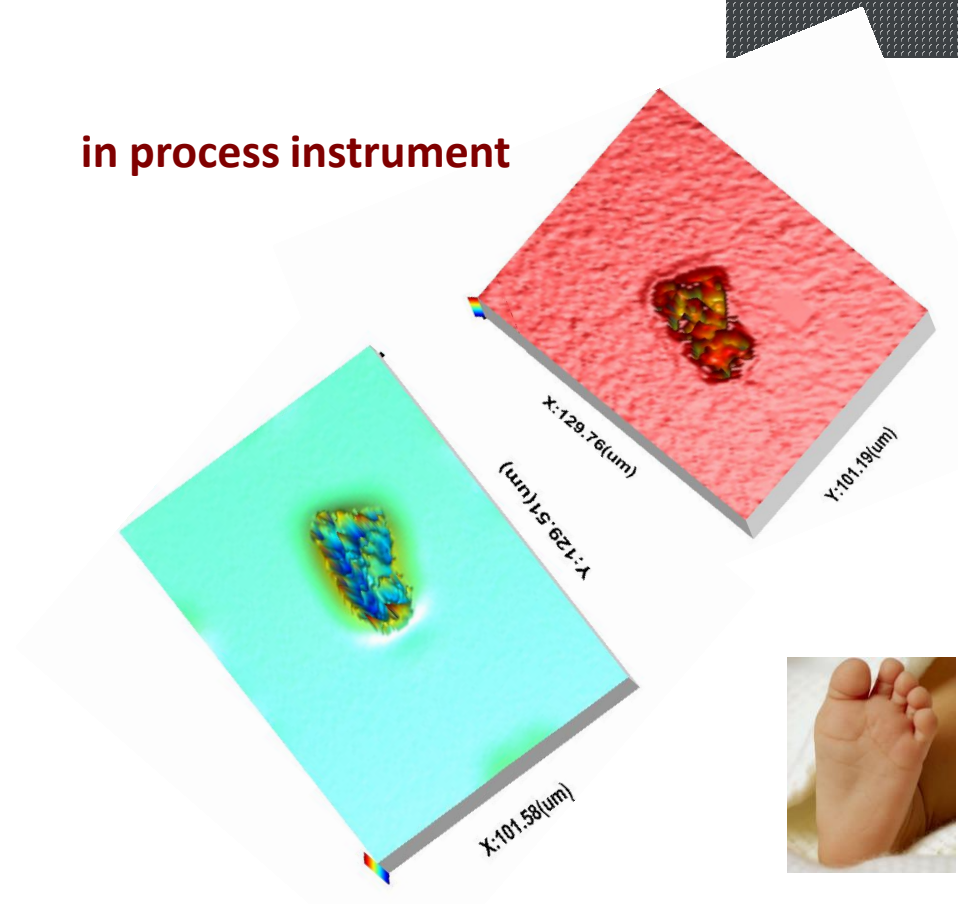
Comparative Measurements “baby foot”

The Future

ESEM



in process instrument



Lab based instrument

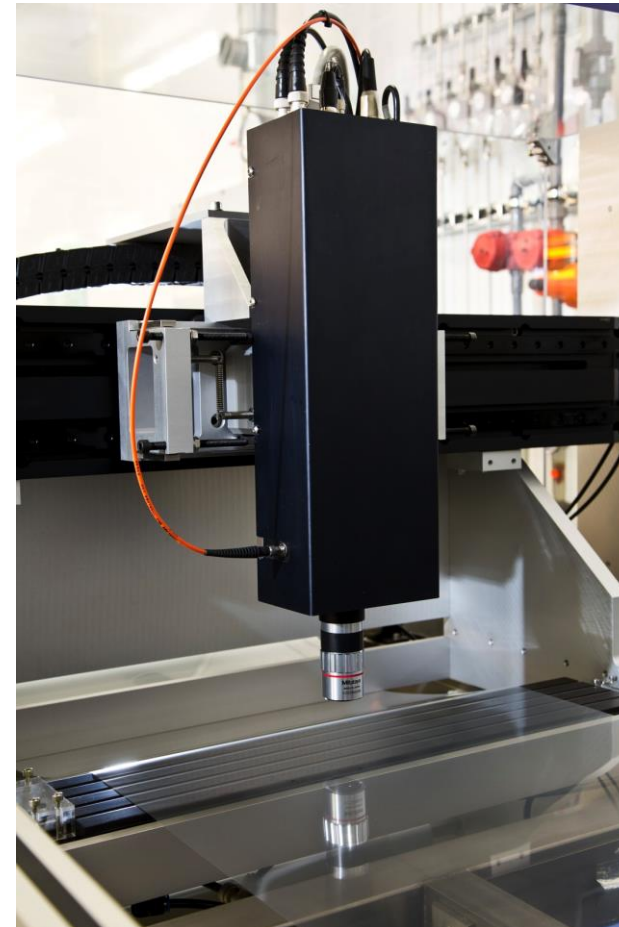


Test platform for web inspection of R2R ALD barrier film (stones)

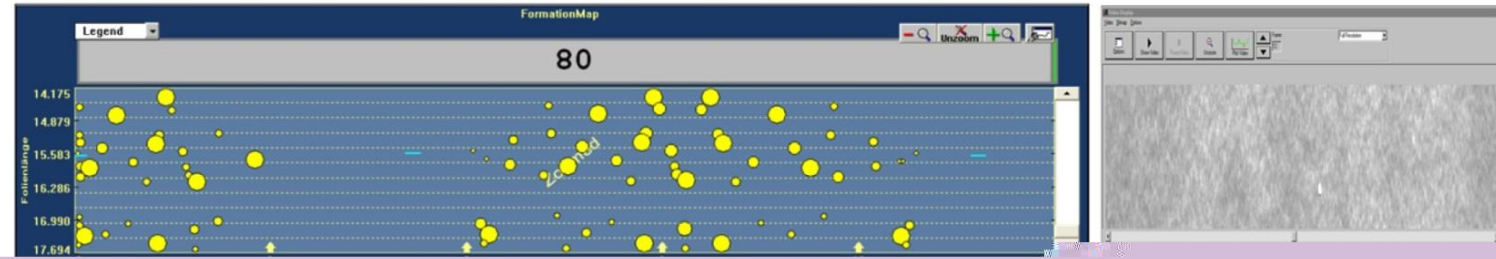
The Future

CPI- UK Catapult, Centre for Process Innovation

- Roll-to-roll web cleaning platform (CPI)
- Up to 500mm web-width spooling capability (CPI)
- “Substrate holding” system (IBSPE)
- In situ calibration artefact (NPL)

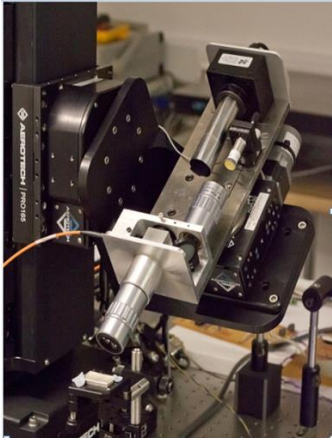


Required Analysis for implementation: Running defect map (mortar)

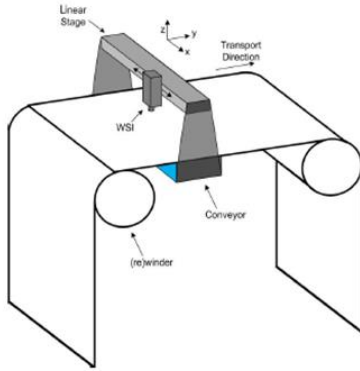
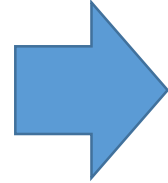


Map and collate defect data for
production control or defining areas
of substrate rejection

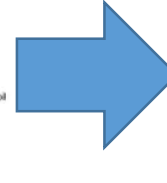
WSI development - evolution during the project



Lab-based system at Hud



IBSPE development for web hander

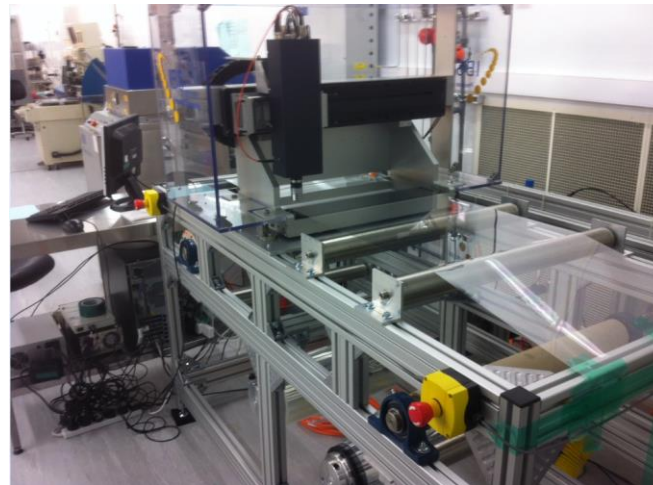
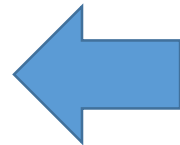
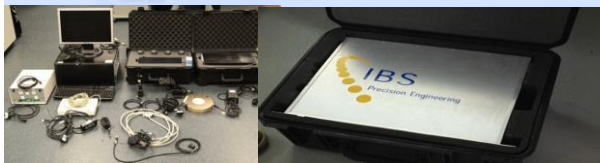
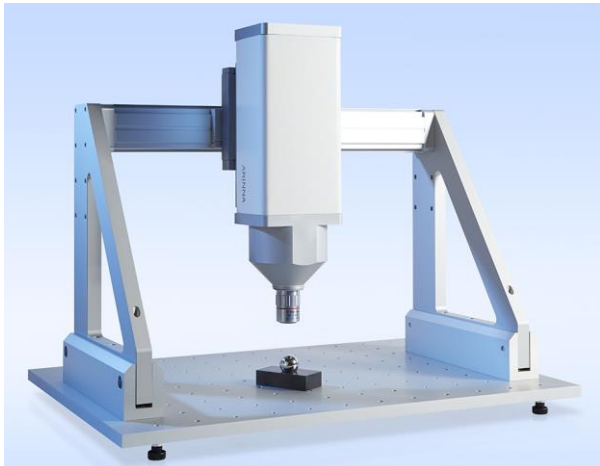


Proof of concept system at CPI



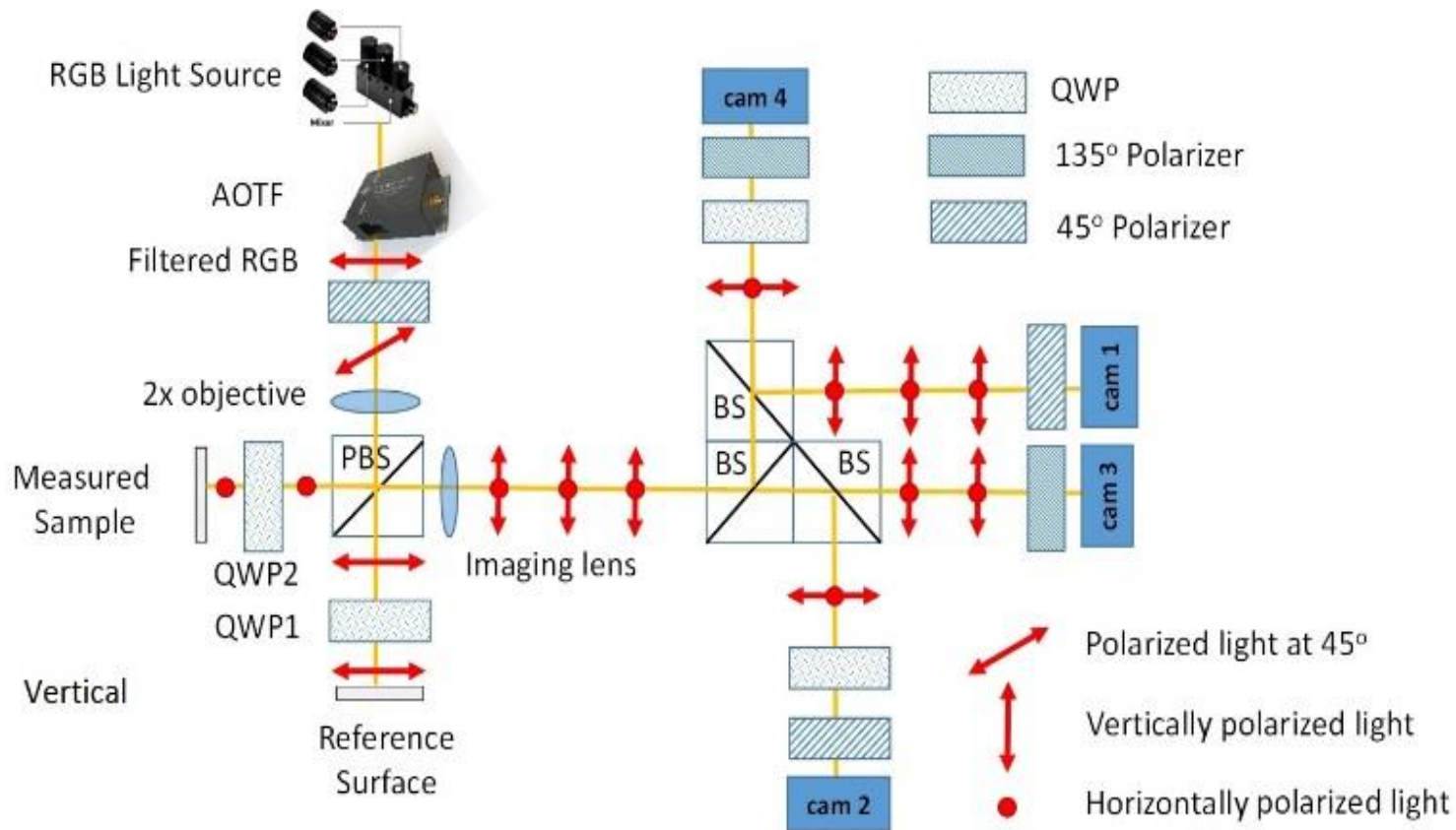
Development of improved web winder at CPI with calibration in line (NPL)

Arinna (IBSPE)



Multi-wavelength Polarising Interferometer

Single Shot Dynamic Measurement



- polarisation technique introduces the essential phase shift
- Each detection arm has a colour CMOS camera placed after a polarization arrangement to generate a single RGB interferogram shifted by $\pi/2$ sequentially.
- The polarization arrangement in each arm introduces a phase difference equal to $\pi/2$ between the beams and makes them coplanar in order to interfere

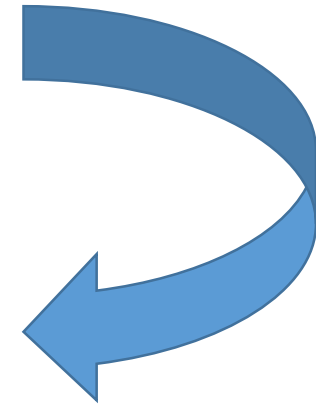
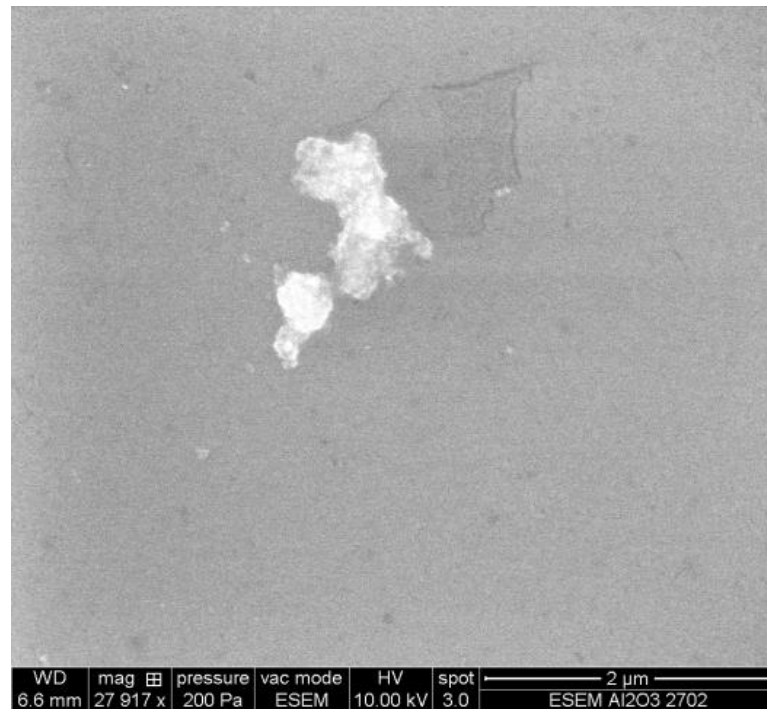
Patent pending

Conclusions

- In process metrology for large area substrates a strong technology driver
- Non contact robust measurement the obvious option
- For PV barrier coatings small numbers of large defects correlates with WVTR
- Modelling focuses metrology task
 - Simplifies the number of measurands
 - Reduces the amount of data needed
- Moving Substrates 1-10m/min
- More research needed on data handling

Case Study; Take Home

“Understand what needs to be measured and only measure that”



Acknowledgements

- EPSRC Sponsorship for Future Manufacturing Hub
- Colleagues
 - Prof Xiang Jiang, Prof Paul Scott, Dr Mohamed Elrawemi, Dr Hussam Muhamedsaleh, Dr X Lan

EU; Nanomend Grant Agreement No. *280581*

- Nanomend Partners