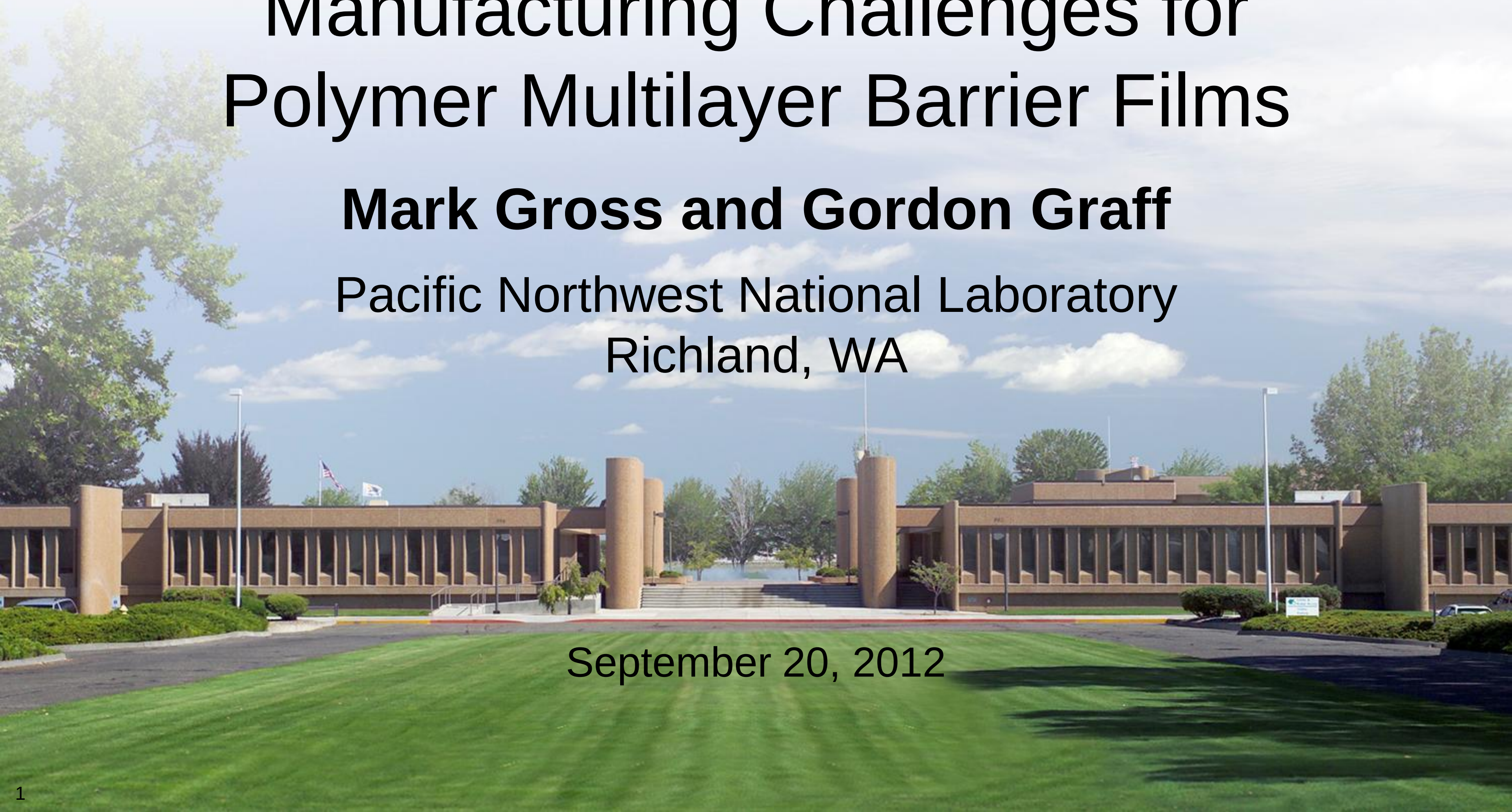
Barrier Technologies Workshop

Manufacturing Challenges for Polymer Multilayer Barrier Films

Mark Gross and Gordon Graff

Pacific Northwest National Laboratory
Richland, WA

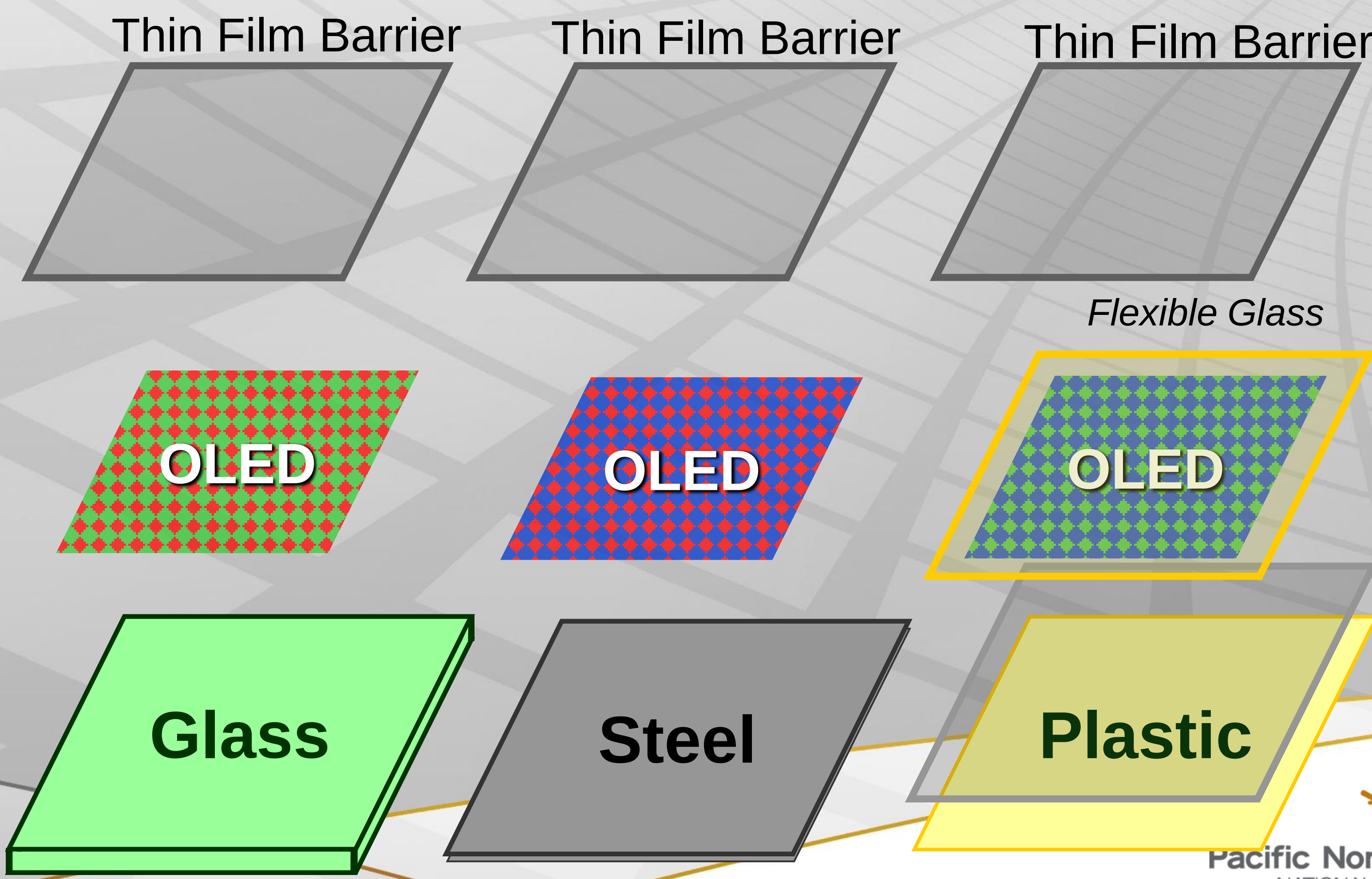
September 20, 2012



Overview

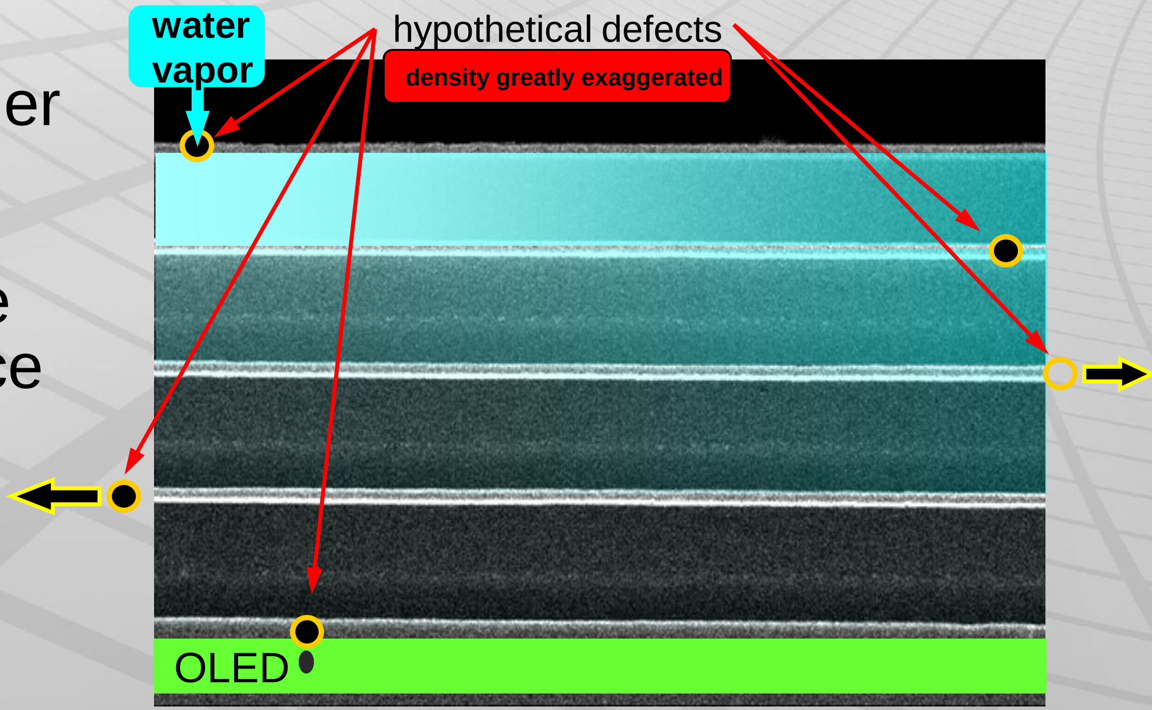
- Polymer Multilayer Introduction
- Two Polymer Multilayer Technologies
  - Thin Film Encapsulation (TFE) or Direct Encapsulation
  - Barrier on Film – flexible web
- Manufacturing Challenges of Polymer Multi-Layer Barrier Films
  - Defects
  - Machine Design
  - Process Scale up
  - Reduced Number of dyads
- Summary

TFE Configurations and Barrier Films



Barrier Function of Film Structure
Current Focus – Reduced Number of Dyads

- Water vapor migrates through defects in barrier layers
- Defect density and size determines performance
- Performance is determined by lag time (transient regime)



Model* prediction with 3 dyads

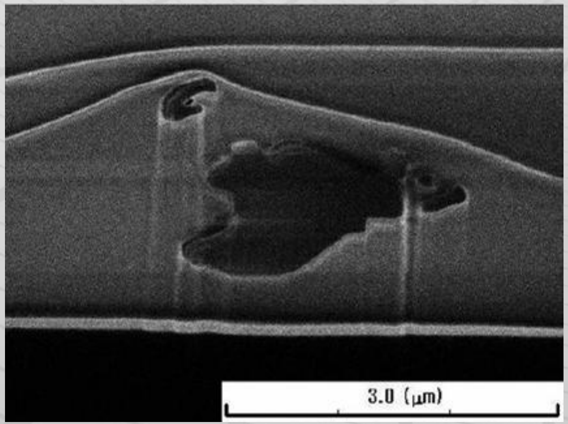
Lag time > 2 years for defect density of ~ 1 / mm^2

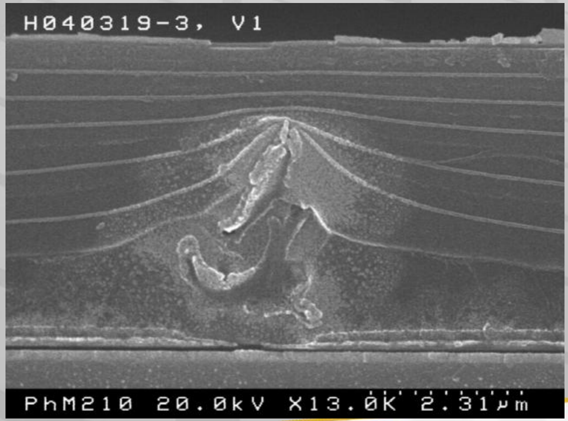
Pacific Northwest NATIONAL LABORATORY

Proudly Operated by Battelle *Since 1965*

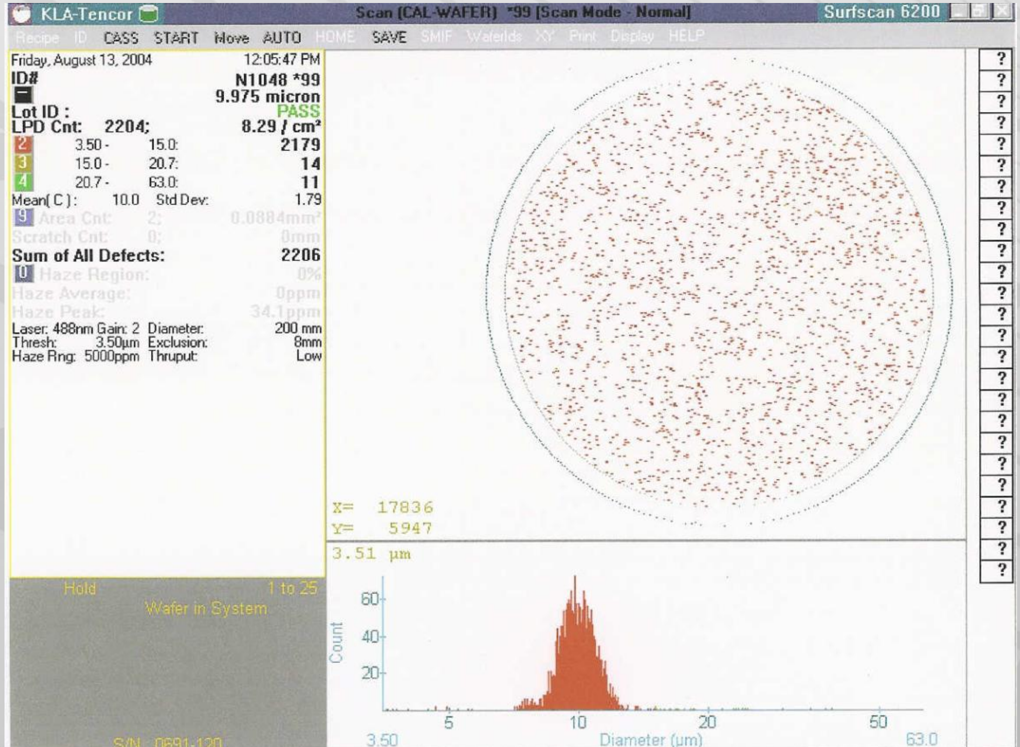
Defects - Particles

- Intrinsic defects
  - Quality of inorganic layer is a compromise with process compatibility for OLEDs devices
    - **Process conditions and hardware configuration**
    - **Maximized process control (high C_{pk} of oxide and polymer process)**
    - **Optimized materials**
- Extrinsic defects (= particles)
  - Present on samples surface due to fabrication and transport
    - Reduced with encapsulation tool in line with OLED deposition
  - **Related to hardware configuration of the deposition tool**





Barrier Performance: Particle Size and Density Experiments

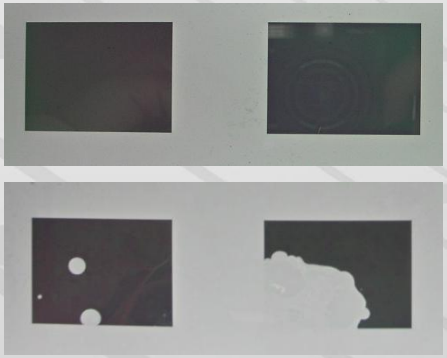


Designed an experiment to determine multilayer barrier performance with particle size and density

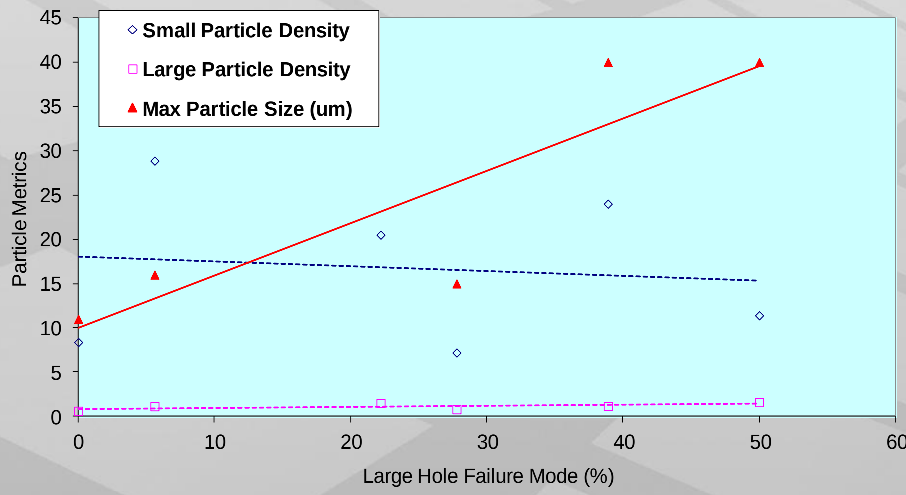
200 mm Si Wafers pre-coated with polystyrene latex (PSL) spheres in size of 1 to 10 um at a density of 16/cm^2

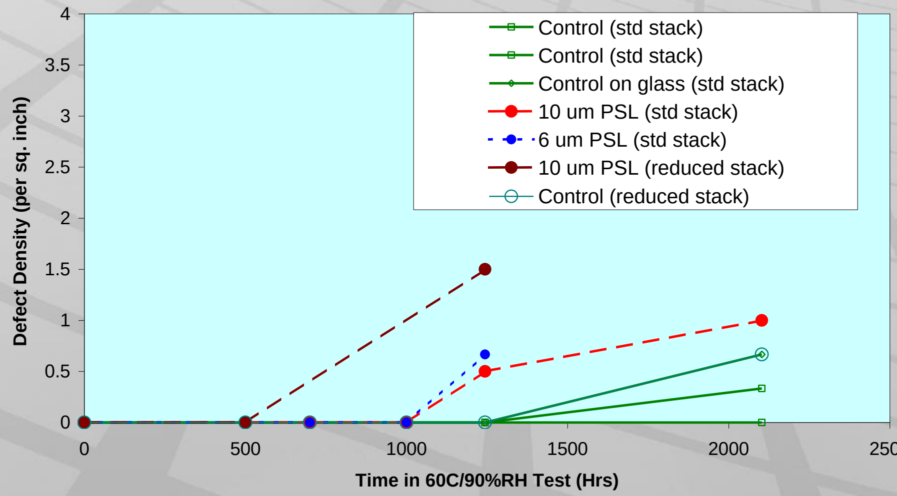
*Chu et al. IDW 2004

Calcium Test Results



Accelerated test result of Barix™ encapsulated Ca on glass substrate. Transmission change (@633nm) is <10% after 2000 hrs in 60°C/90RH conditions. The early failures shown in the pictures were correlated to particles visible on the substrates

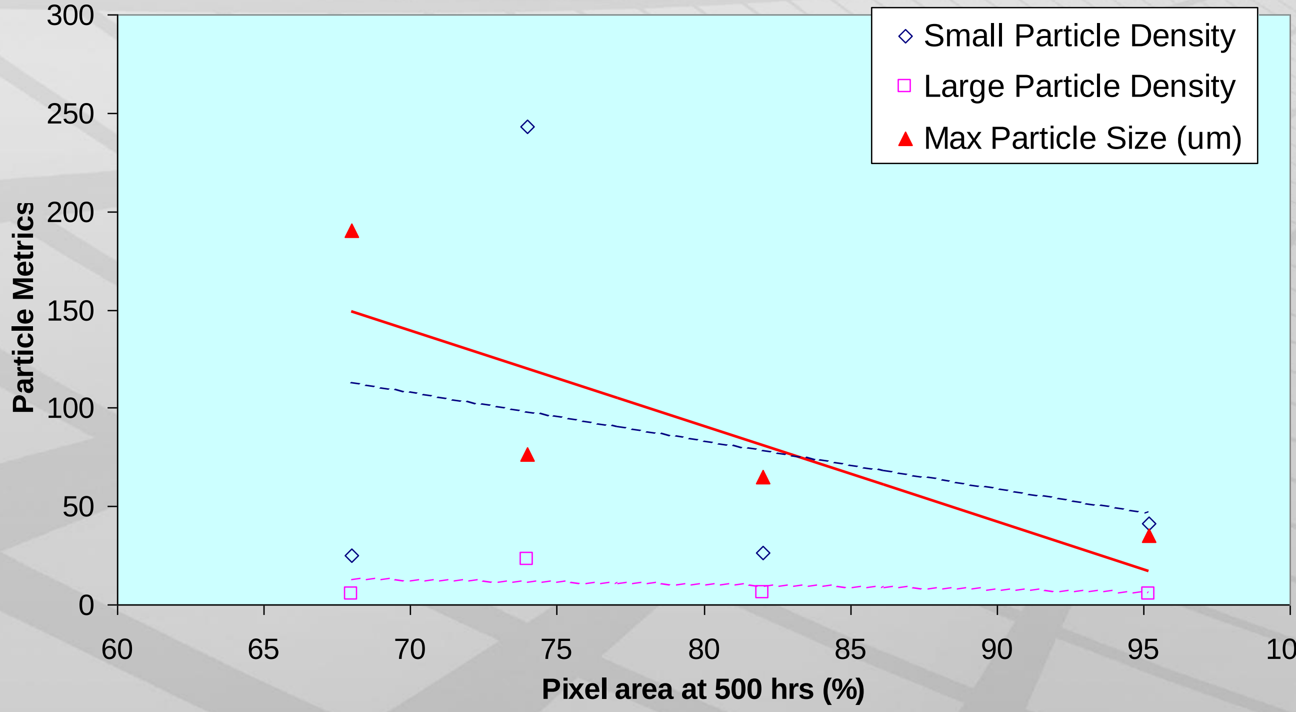




Correlation of Ca test failure due to large hole, particle density, and maximum particle size observed on the samples. Frequency of large holes on Ca samples correlates well with the largest size of particles (> 10 µm) observed on the samples.

*Chu et al. IDW 2004

OLED Barrier Performance: Particle Size

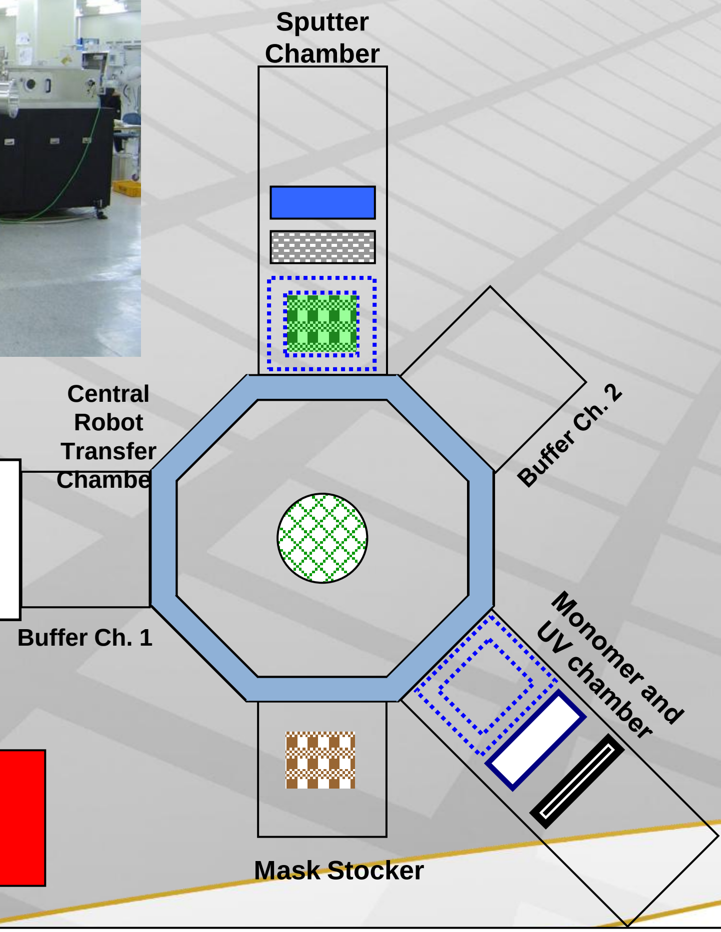


Correlation of TOLED pixel shrinkage, particle density, and maximum particle size observed on the samples. The pixel area remaining after 500 hrs testing at 60°C/90RH showed good fit to the largest particle size found in the film

*Chu et al. IDW 2004

Machine Design – Direct Encapsulation

Vitex cluster tool design



Polymer multilayer in production

- Process separation
- Device handling
- Particle control
- Monomer process on/off
- Monomer capture
- Edge seal
- Integration to OLED line
- Scale to G4 and beyond
- Through put / yield
- Maintenance

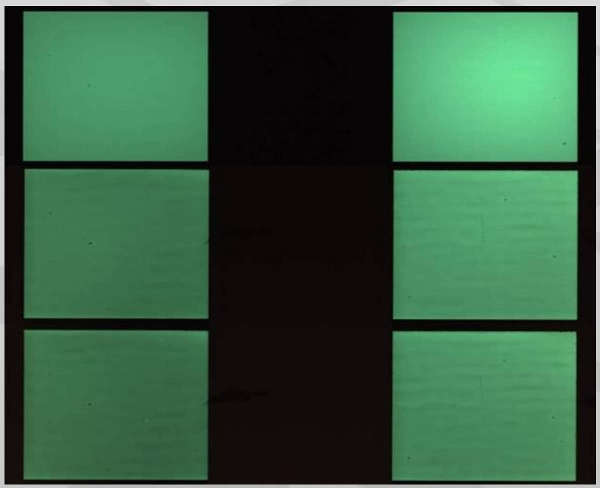
- **R&D and Pre-pilot**
  - 200mm x 200mm
  - 400mm x 400mm
  - Linear or cluster
- **Mass Production**
  - 15 substrates per hour
  - Up to 730mm x 920mm

Note: Cluster tool could also function to deposit barrier on film

Edge Seal

- An impermeable seal is necessary to prevent moisture from entering through the sides
- Protection of the sides of the display is obtained by making the oxide mask wider then the polymer mask

Edge Sealed Samples



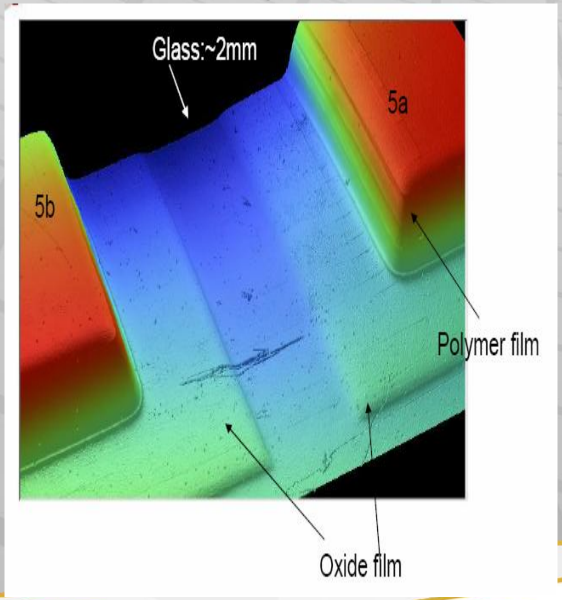
t0

300h
60ºC/90%RH

500h
60ºC/90%RH

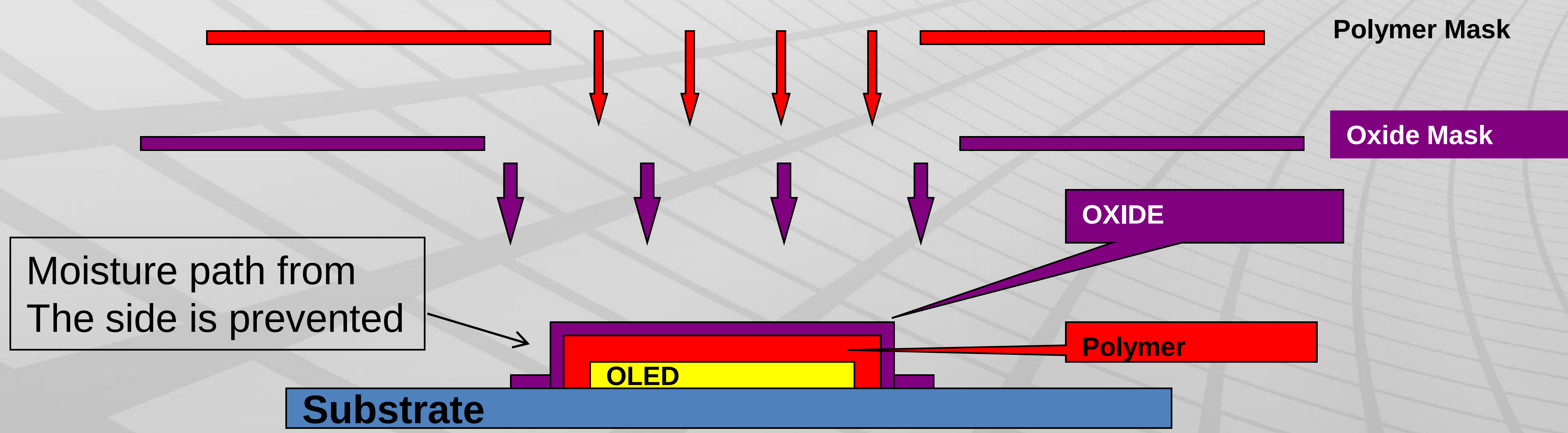
No Edge defects: 100% Yield 18 OLED samples

3D Optical Profile of the Edge Seal



- 1.5 mm edge seal
- CCD alignment will enable edge seal to go below 1mm

Edge Seal: Dual Mask Method



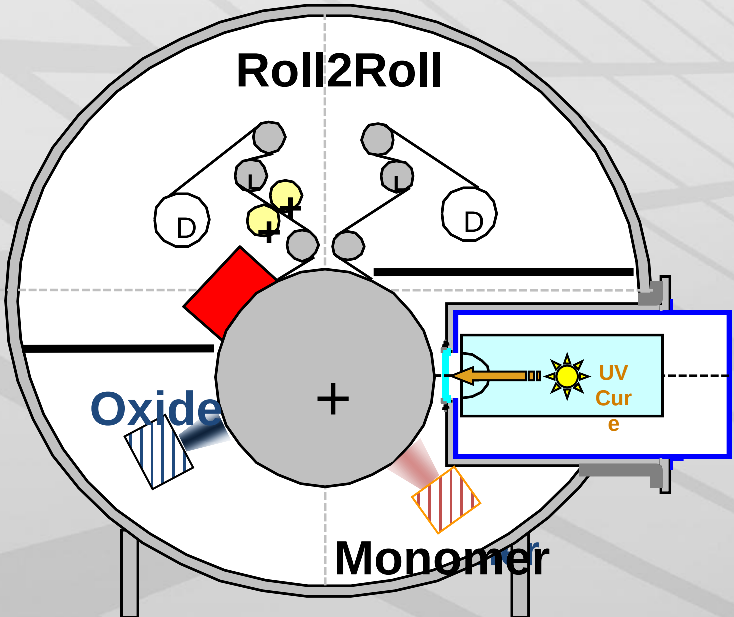
Typical dimensions:

•OLED Display:	2” diagonal
•Polymer thickness:	~1 micron
•Oxide thickness:	~30-50 nm
•Edge seal width: (CCD alignment) :	0.45 mm

Pacific Northwest NATIONAL LABORATORY

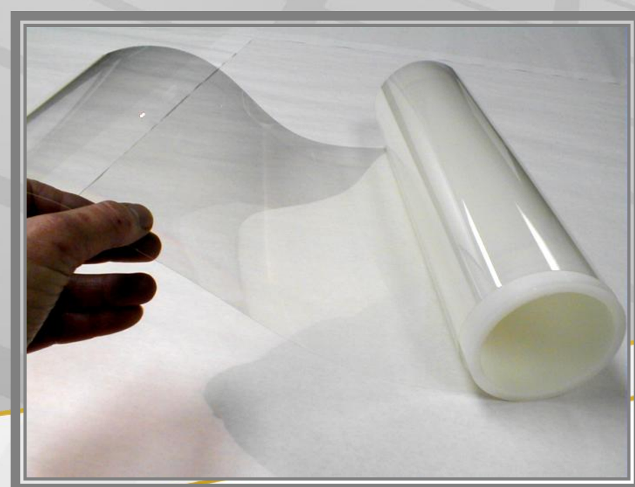
Proudly Operated by Battelle *Since 1965*

Machine Design – Barrier on Film



- Substrate Quality
- **Web Handling**
- **Particle control**
- Process Separation
- Temperature Control
- Monomer efficiency
- Edge seal
- Through put / yield
- Maintenance

Polymer multi-layer in production



Machine Design Barrier on Film: Effect of Roller Contact & Particles

- Objective of grooved roller experiment:
  - To evaluate the relative importance of web contact to rollers & effect of repetitive winding on barrier properties & defects

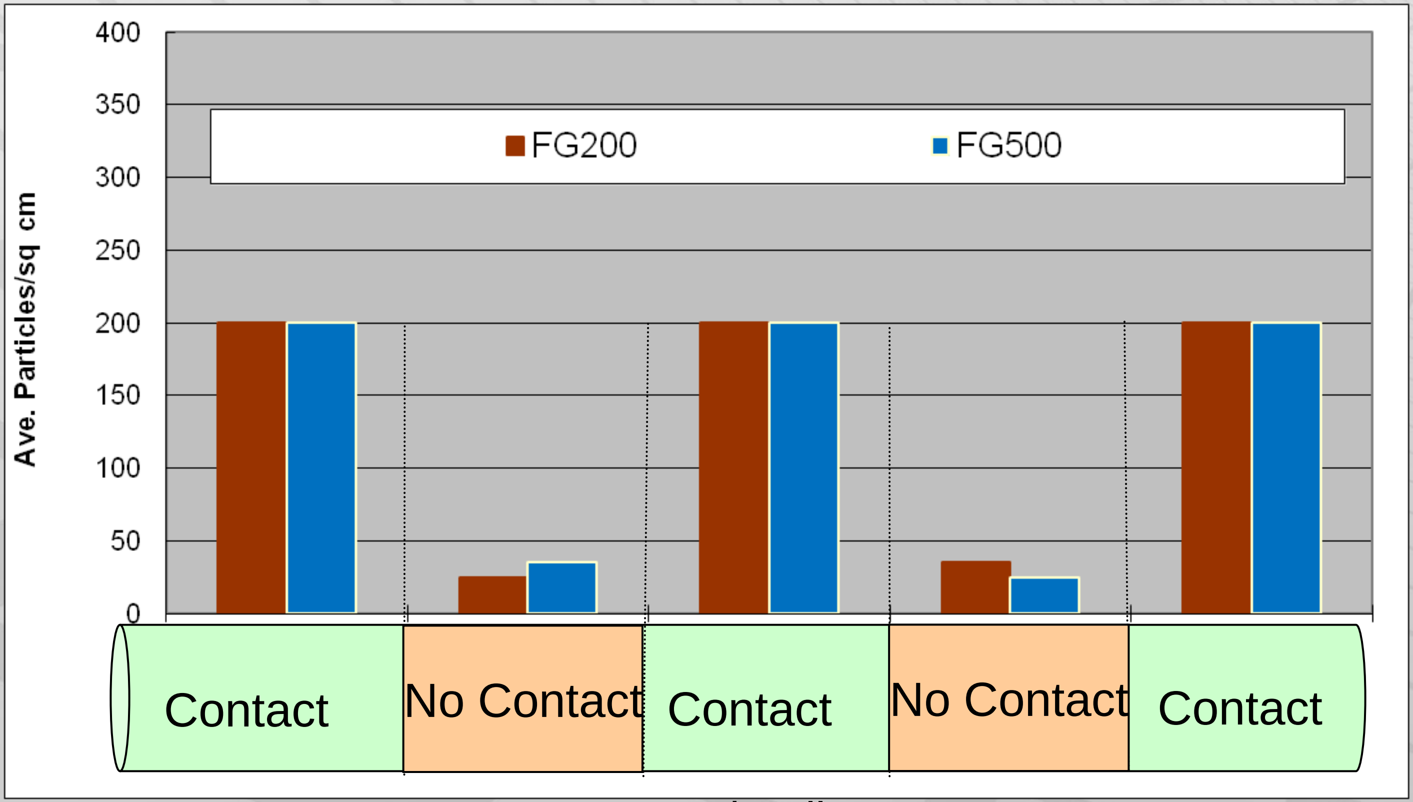
Grooved Roller

Metrics:

Barrier properties (calcium degradation) & number of particles (optical microscopy)

5 areas - 3 where film has contact, 2 with no contact

Grooved Roller Particle Results



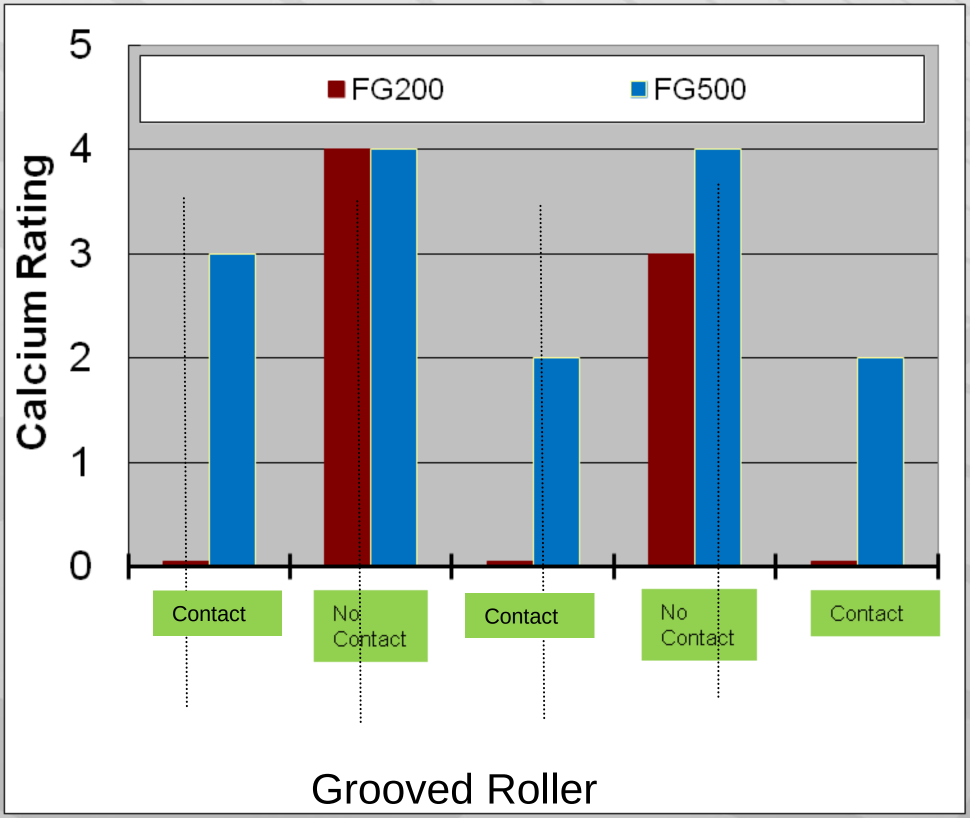
Grooved Roller

- Non contact region have significantly lower particles than contact regions
- Contact on the rolls is the dominant factor in the addition & transport of particles

*Kapoor et all SVC 2006 505/856

Grooved Roller Results: Calcium Test

200h Accelerated Aging Conditions 85ºC; 50% RH



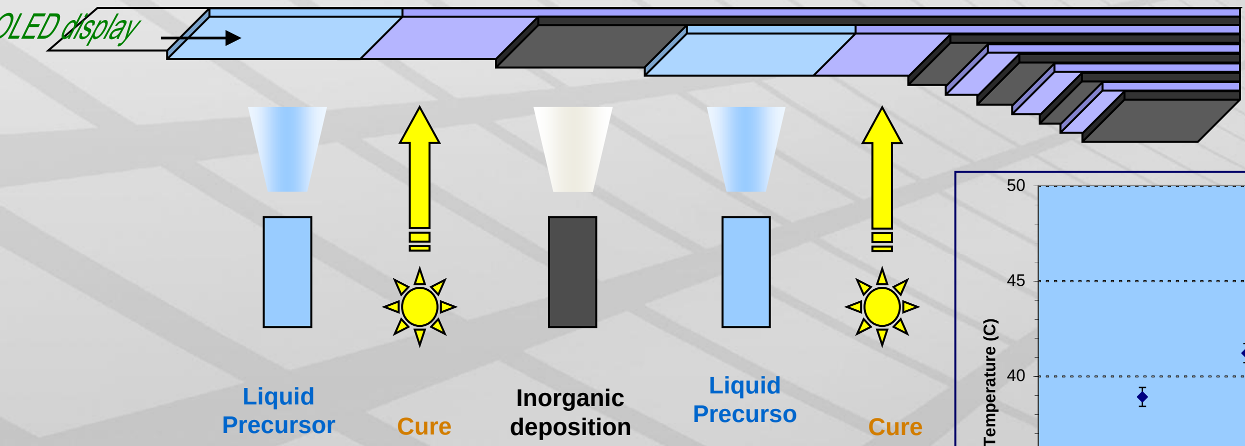
- **Non contact region has substantially better barrier properties than contact area**
- **Extra winding dominates failure of barrier properties – surface contact**
- **Extra layers (FG500) shows best barrier properties**
- **FG200 from non-contact section meet "pass" criteria for production FG500's**
- **FG500's from non-contact section had residual Ca @ 900+hr**

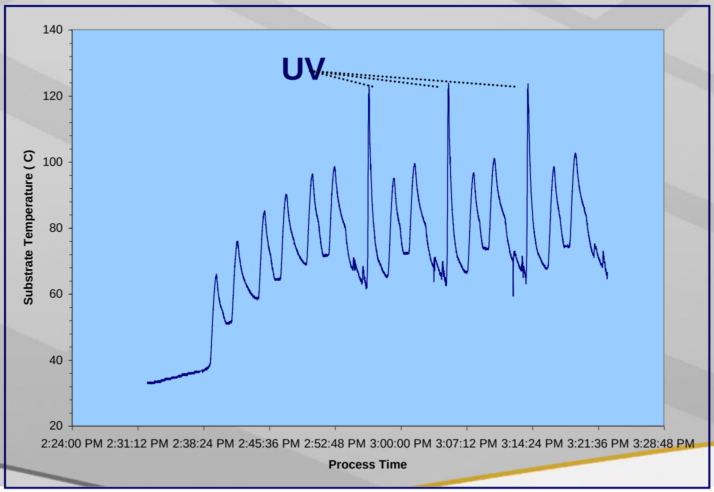
*Kapoor et all SVC 2006 505/856

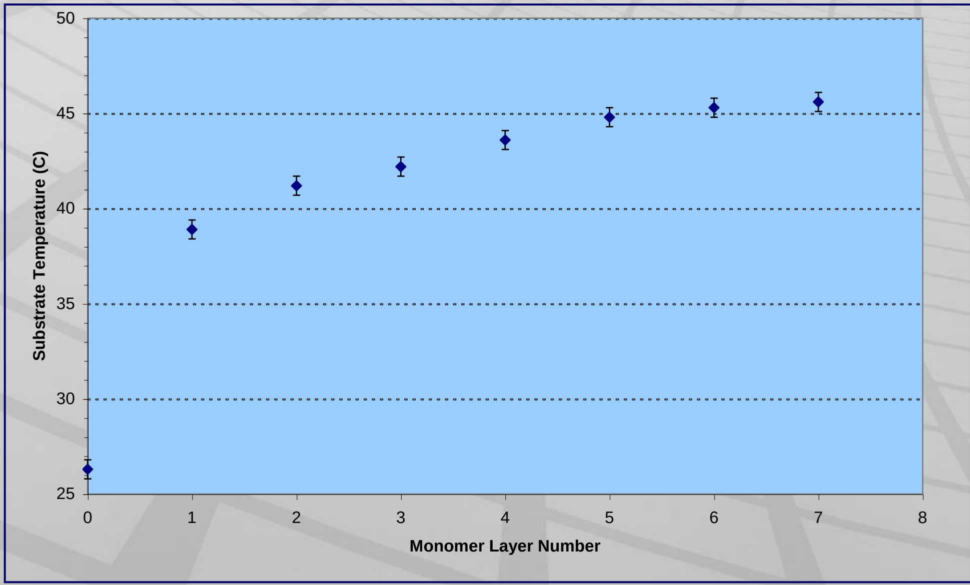
Process Scale Up

- Increased through put with high yield
  - More samples in less time – Lower TAC time
  - Balance faster process conditions with higher power and larger devices or web
    - Damage is application specific – OLED more sensitive than PV to plasma damage and heat load (direct encapsulation vs. lamination of barrier film)
  - Fewer number of dyads
- Which Layer to focus on
  - Organic
  - Inorganic

Barix™Technology: Low Temperature Process







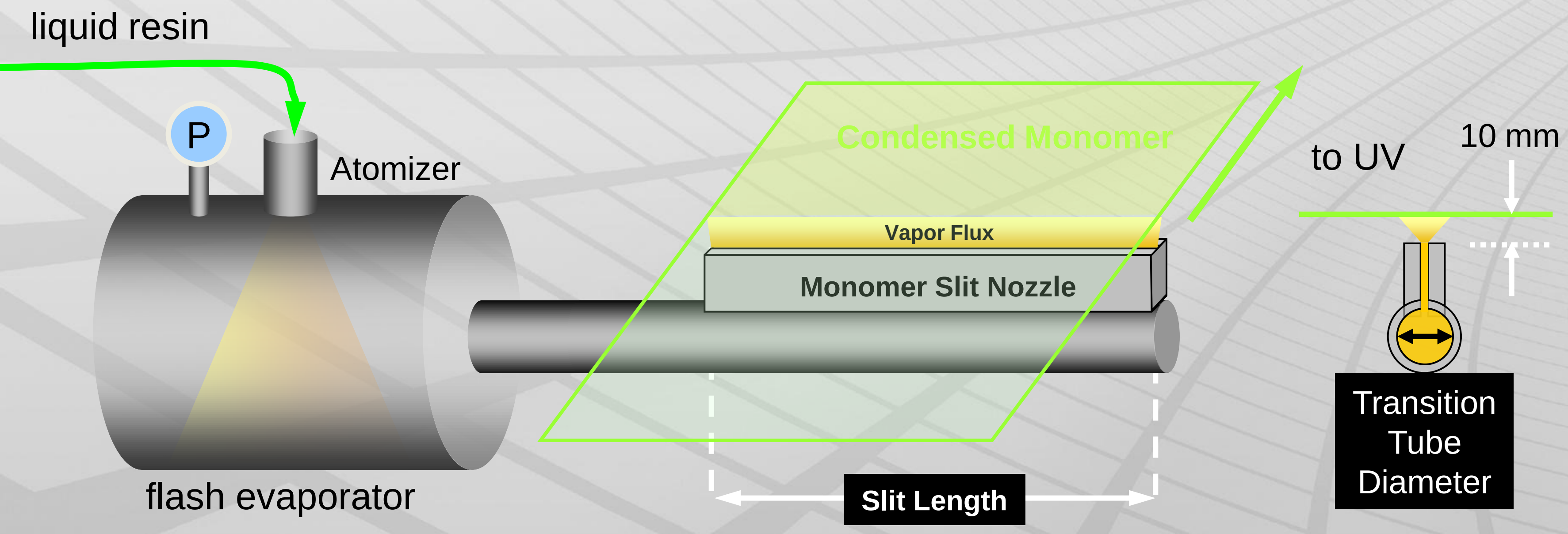
Average substrate temperature is far below 100ºC. LED cure source operates at lower temperature

Organic Deposition

- Purpose: Decoupling layer between inorganic layers
- Challenge: Deposition of a thin non-conformal flexible layer that closely matches the index of refraction of the inorganic layer
  - Areas of concern: Substrate non-uniformities, Particles, Device morphology, Ease of deposition, Edge seal, Low temperature process <100^{o}C, and adhesion

Process	Vacuum	Current Scale	Edge Seal	Manufacturing Issues
Acrylate evaporation	Yes	Production	Yes	Many issues solved, Chemistry limits
Spray / Ink Jet	No	Research	Not proven – could work	May work – broader chemistry,
CVD	Yes	Research	Difficult	Contamination-Up time
Parylene	Yes	Research	Not proven	Contamination
Gravure	No	Research	Not proven	Film thickness control for thin film

Polymer Source



- Flux distributed through long, narrow slit
  - Scalable to 2.0M+
- Experimental observations
  - Insensitive to nozzle-to-substrate distance variation
  - Small end effect (Slit length 210 mm for 200 mm substrate)

Inorganic Layer Deposition Technologies

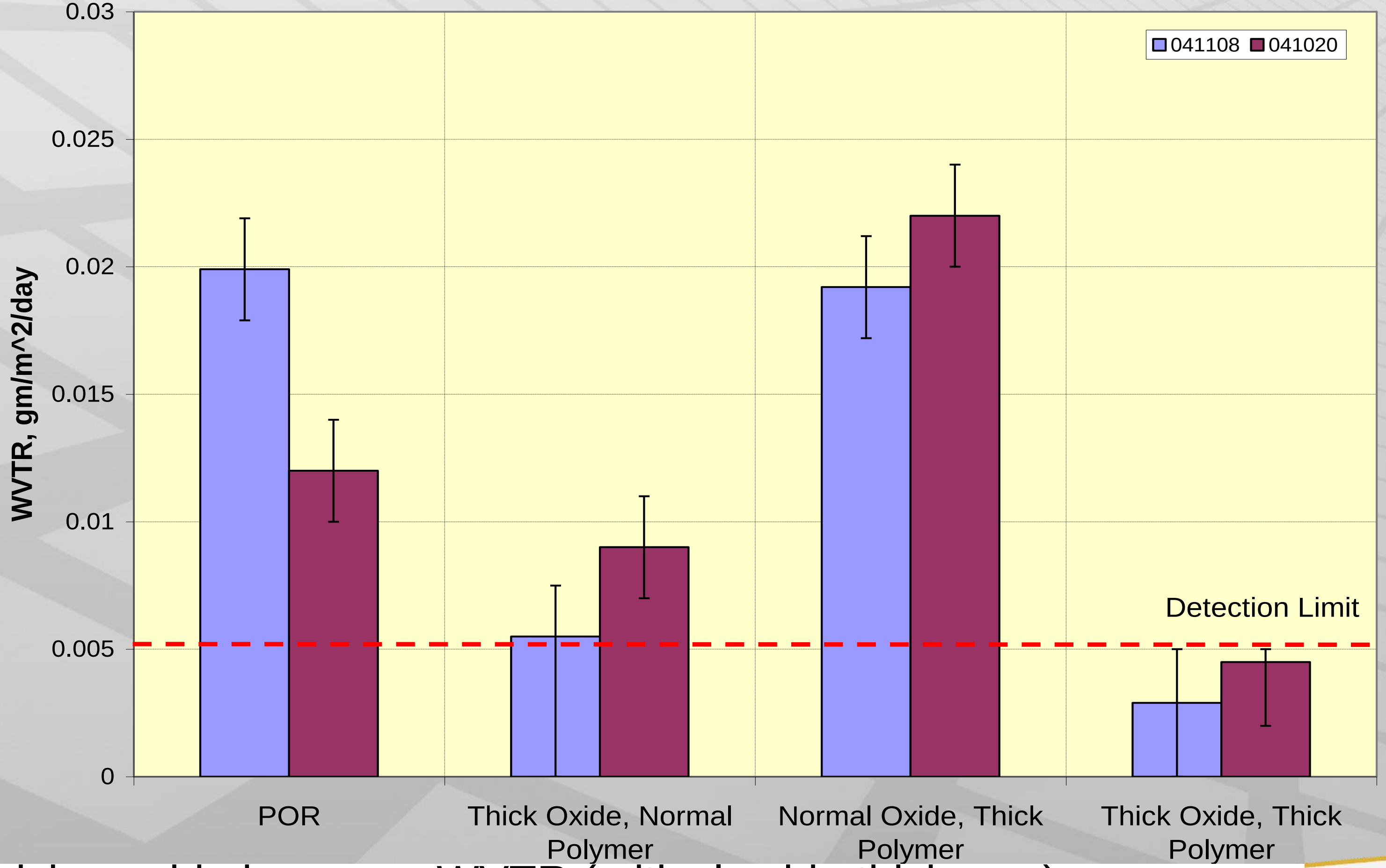
- Purpose: Determines barrier performance
- Challenge: Thin, dense, uniform, low particulate layer

  - deposited at low temperature with high rate

  - What is the critical inorganic layer thickness – machine and application specific

Technology	Structure	WVTR	Deposition rate	Production Scale
Reactive magnetron sputtering	Multilayer	$<10^{-6}$ g/m^2/day	High	Yes (off the shelf)
PECVD	Graded layer	5×10^{-6} g/m^2/day	High	Yes
ALD	Single layer	1.5×10^{-5} g/m^2/day	Low – but improving	Not Yet – promising

Barrier Effect of Critical Oxide Layer Thickness

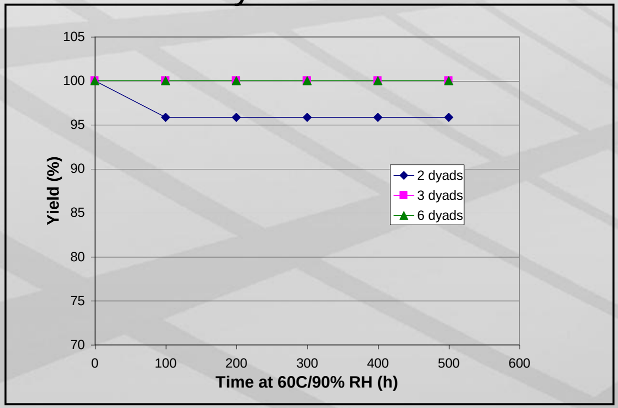


- Thicker oxide improves WVTR (critical oxide thickness)
  - Thickness is application specific: TFE 20-30nm, barrier on film 50nm
- Combination of best oxide and polymer thickness is best

*Kapoor et al. SVC 2006 505/856

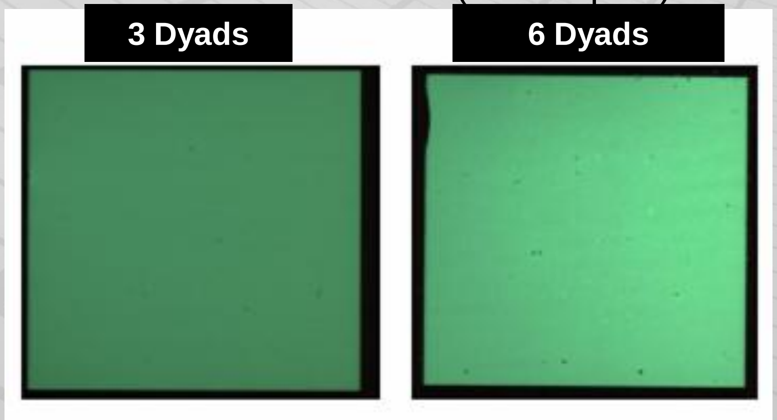
Barrier Performance on OLEDs With Reduced Number of Dyads - Cost

OLED Test: 3 Dyads Have >100% Yield!
2 Dyads have 95% Yield



3 Dyads structure tested on more than 150 test pixel devices

3 vs. 6 Dyads Yield after 1000 Hrs
60ºC/90 %RH: 95% (18 samples)



- Reduced number of layers from 5~6 to 2~3 dyads (polymer/oxide pair) in the typical Barix structure
  - Met telecom specs with 2~3 dyads
- Proven OLED results with 3 dyads

*Moro et al. Vitex Systems report

Why Isn't the Technology Widely Adopted

- Many issues have been resolved and the technology is proven on OLEDs, PV, Lithium thin film battery, etc.
- Several machines both pilot and production are in use
  - 16 Vitex System tool installed in Europe and Asia
  - 3M, Materion, and several other companies developing encapsulation technologies
- Two key points that have not changed in several years
  - Integration with production line for barrier on film
  - Commitment from industry – who will be the first

Summary

- Several companies and research institutes are working to develop an effective moisture barrier technology adaptable to large scale manufacturing
- Direct Encapsulation: In production for OLED displays
- Barrier on Film: Well suited for large scale manufacturing for OLED, PV and solid state lighting but struggling to penetrate the market
- Several technologies work at lab scale but can they be easily scaled to 24/7 manufacturing with an acceptable cost of ownership

Acknowledgements

- Marty Rosenblum – Vitex Systems
- Xi Ch - Vitex Systems
- Nicole Rutherford - Vitex Systems
- Robert Visser - Vitex Systems
- Sanjiv Kapoor – Vitex Systems

- Lorenza Moro – Samsung Cheil
- Damien Boesch – Samsung Cheil

- Robert Newton – Materion Advanced Materials

Pacific Northwest NATIONAL LABORATORY

Proudly Operated by Battelle Since 1965