

3M Optical Systems Division

# Barrier Films and Adhesives for Display Applications

CCR OLED Workshop, June 8, 2011, U of MN



# 3M Company Overview

- Sales: \$23.1 B
- Net income: \$3.2 B
- R&D investment \$1.3 B (5.6% to sales)
- International sales \$14.6 B (63% of company total)
- Companies in more than 60 countries
- Sales in nearly 200 countries
- ~ 75,000 employees
- ~ 10,000 technical employees
- 55,000+ products
- 45 established Technology Platforms
- > 2000 patents issued in 2009
- 40,000 issued and pending patents

## Six Market Leading Businesses



Consumer and Office

Display and Graphics

Electro and Communications

Safety, Security and Protection Services

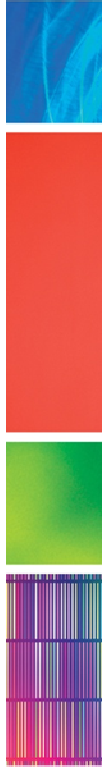
Health Care

Industrial and Transportation

# Display and Graphics Business

Innovative Solutions for light management and visual communications

■ Sales: \$3.1 billion



Commercial Graphics

Optical Systems



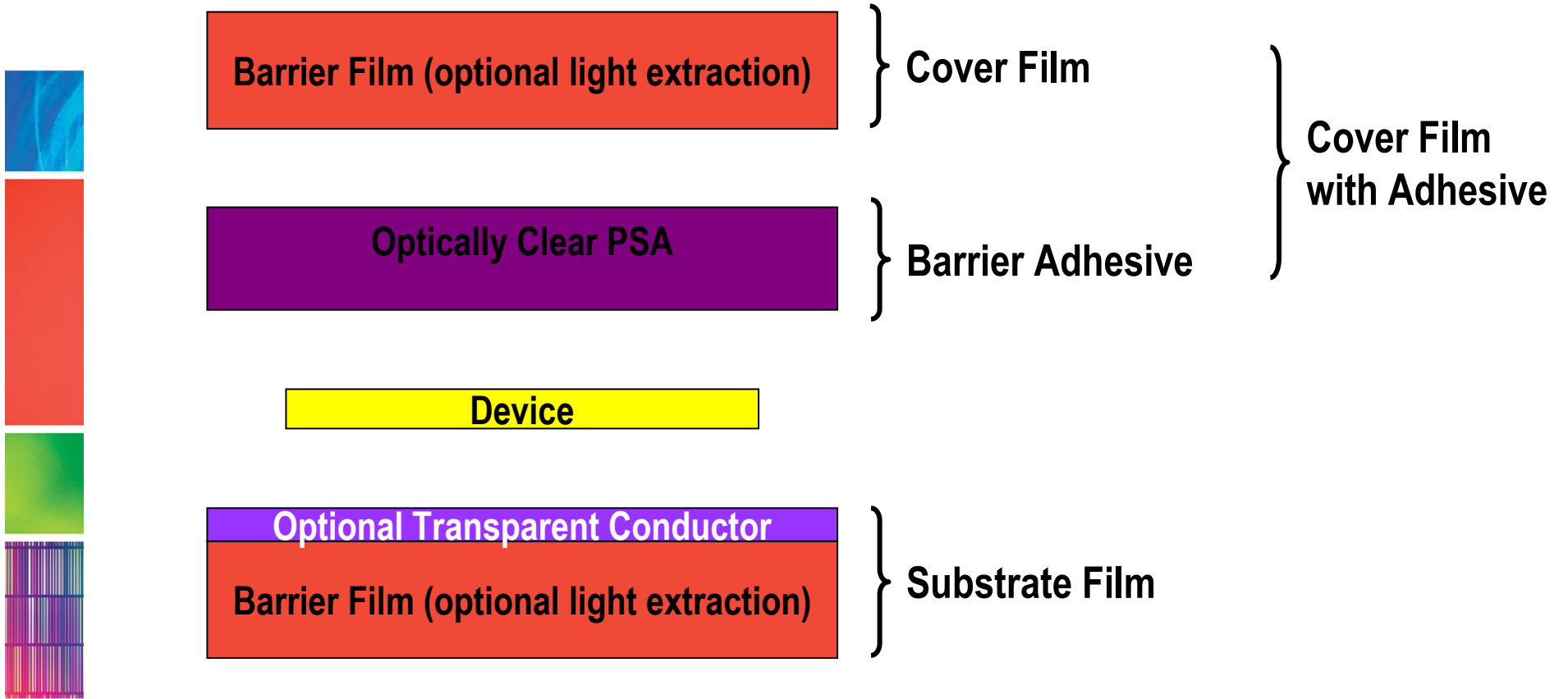
Traffic Safety



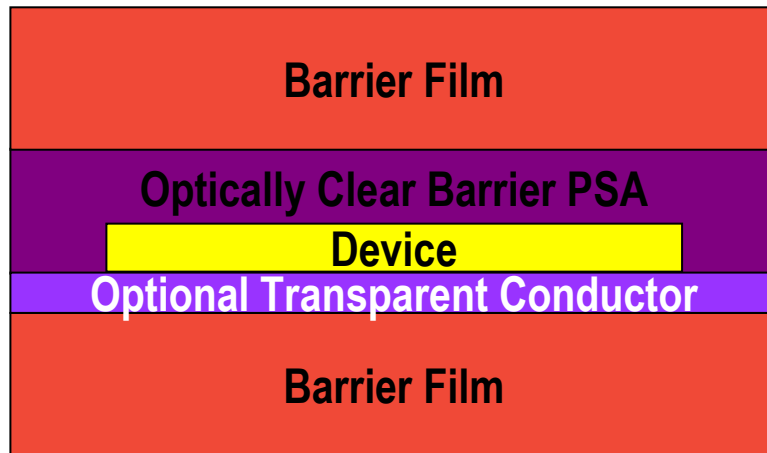
Touch Systems



# Barrier Products Under Development....

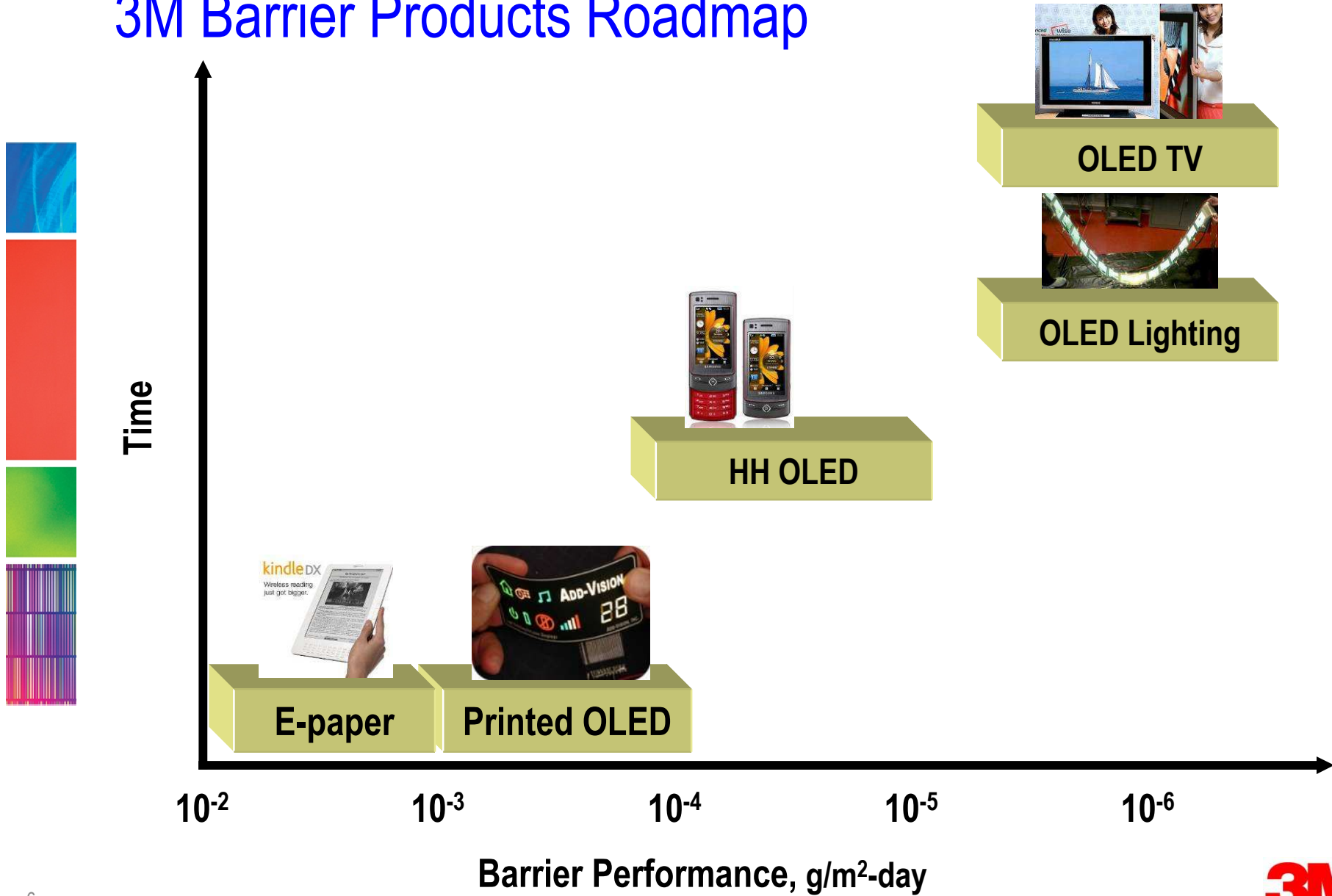


## .....Creating a Flexible Encapsulation System



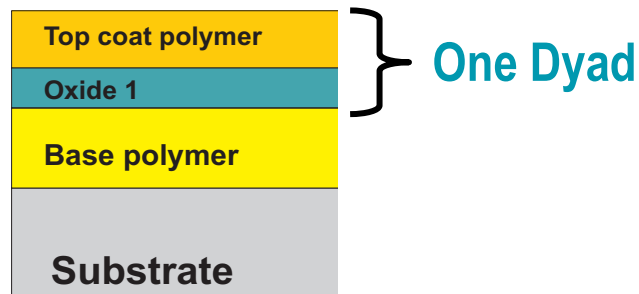
- Enabling Fully Flexible Displays and Solid State Lighting**
- Can also be used on rigid devices**

# 3M Barrier Products Roadmap

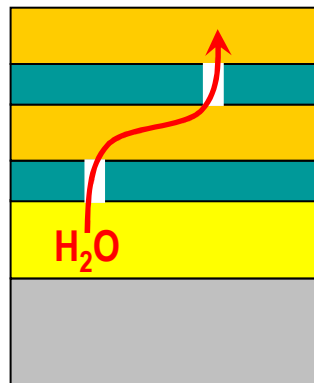


# 3M Barrier Film Technology

- All vacuum roll to roll process
- Barrier properties are provided by oxide/polymer pairs:

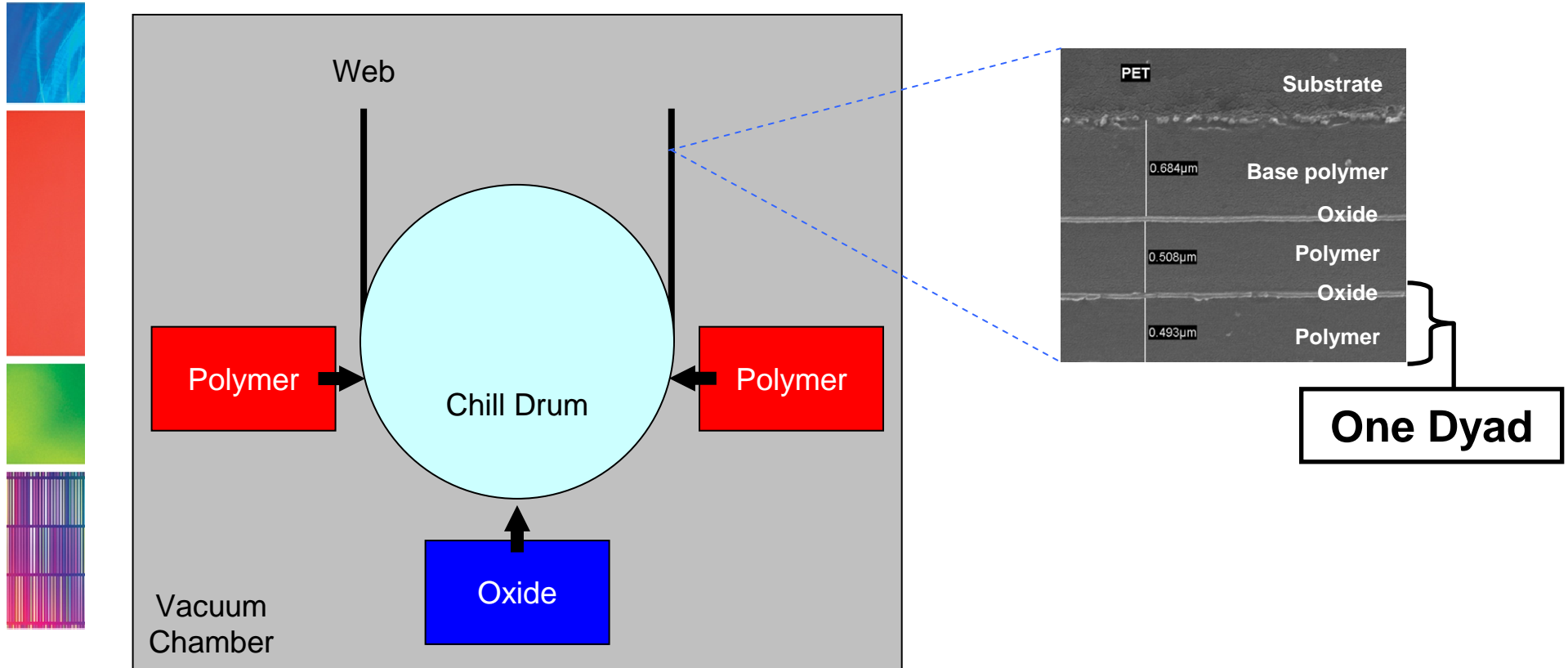


- High barrier properties are obtained by using multiple dyads:



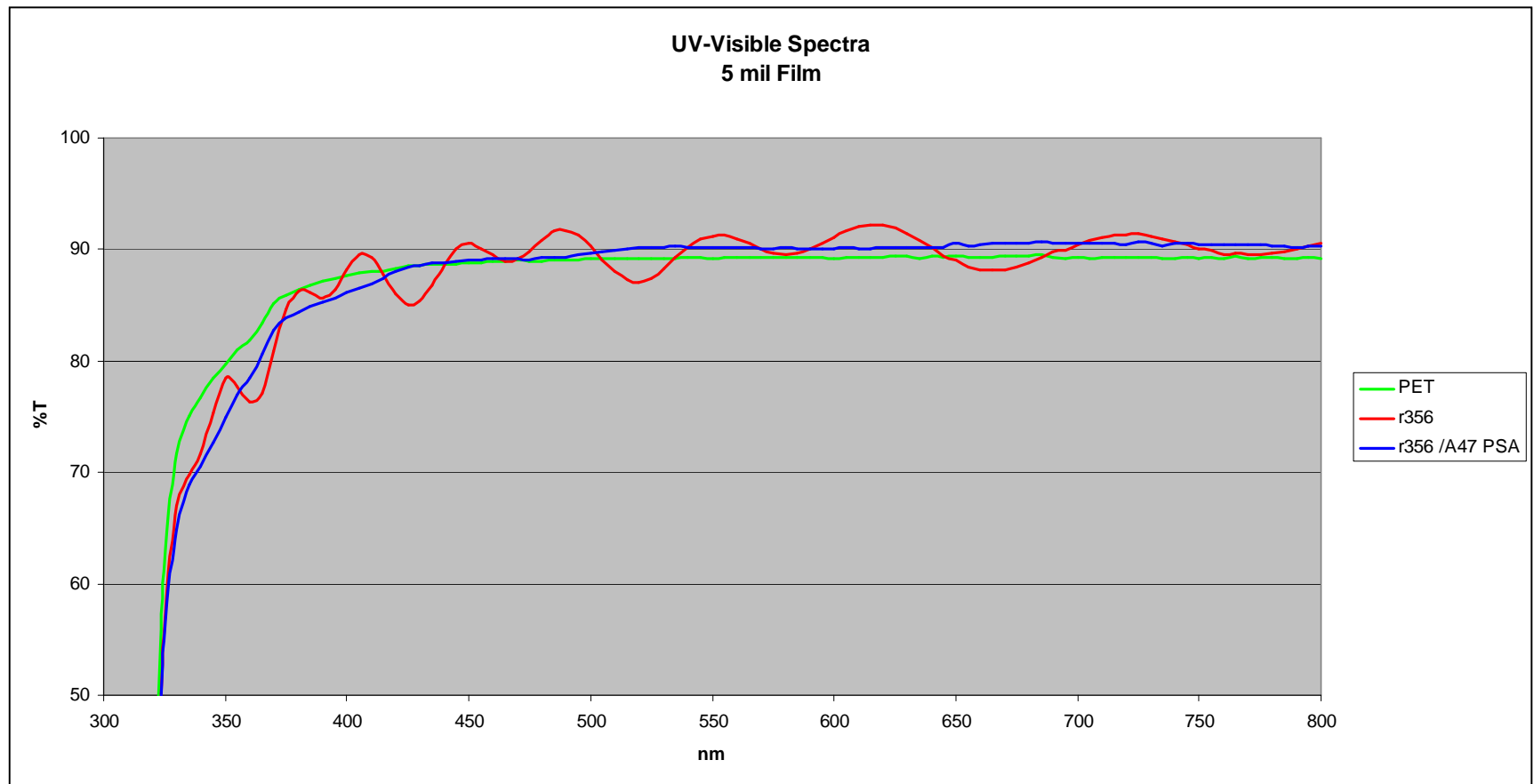
Multi-Layering creates a tortuous path through defects for water and oxygen

# Multilayer vacuum web process:





# Optical Absorption



# Preliminary Barrier Film Specifications



- **< 10<sup>-3</sup> g/m<sup>2</sup>-day WVTR Barrier (FTB3-125 and FTB3-50)**
  - *Suitable for E-Paper*
  - *>87%T in the visible range, <2.5% haze*
  - *PET substrate*
  - *Can incorporate AG/HC*
  - *A4 sheets or 300mm wide rolls*
  
- **< 10<sup>-5</sup> g/m<sup>2</sup>-day WVTR Barrier**
  - *Under development, suitable for OLEDs*
  - *PET substrate*
  - *Non-birefringent substrates under development*
  - *A4 sheets or 300mm wide rolls*

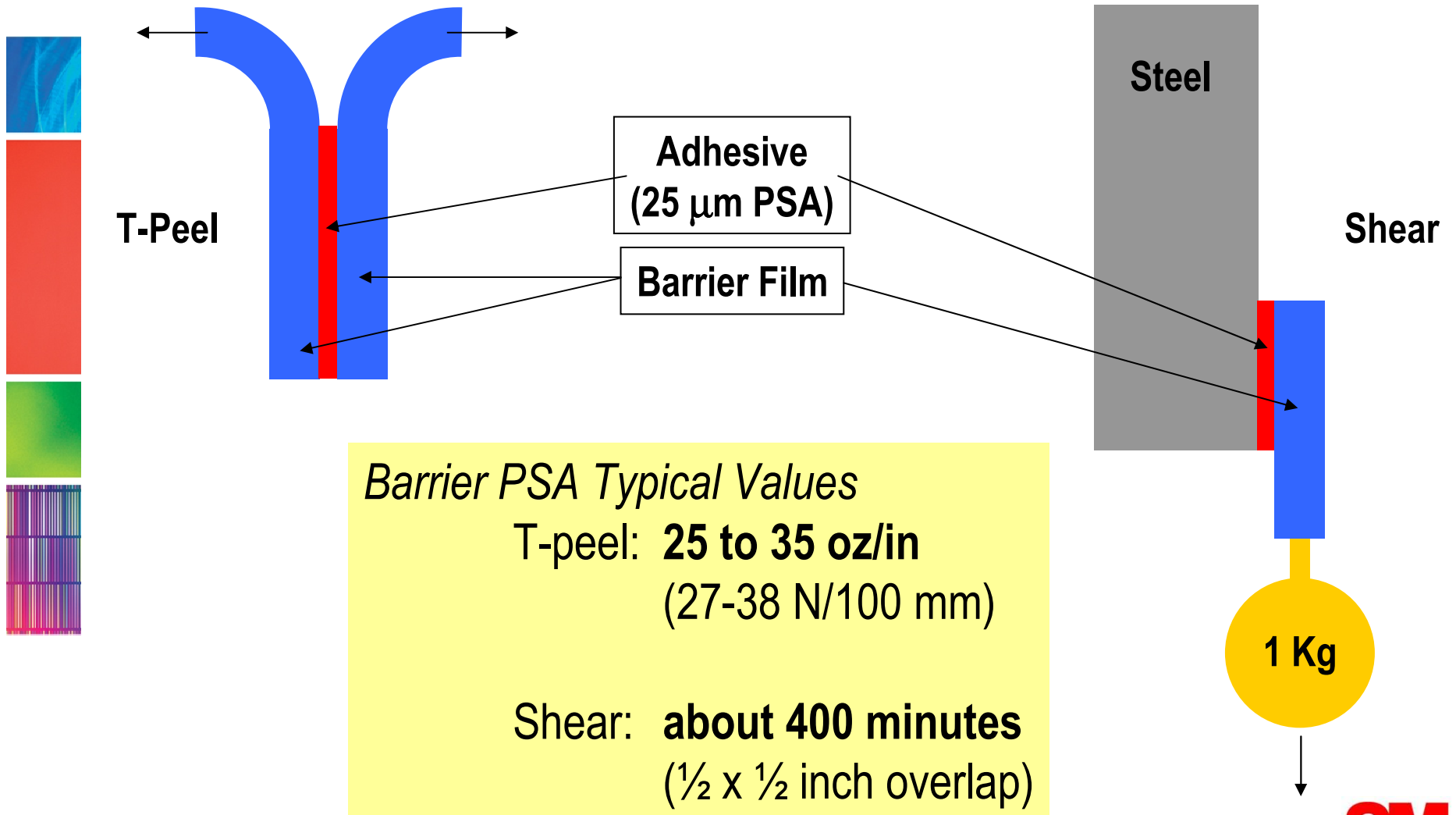
## Barrier Adhesives

- 3M has focused on transfer adhesive films:



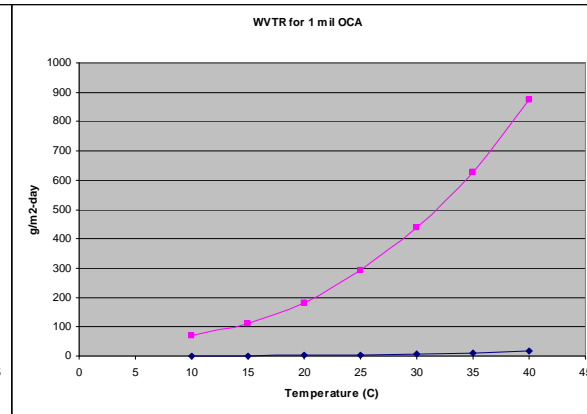
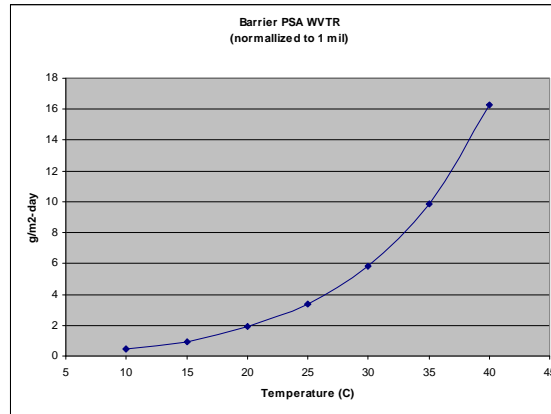
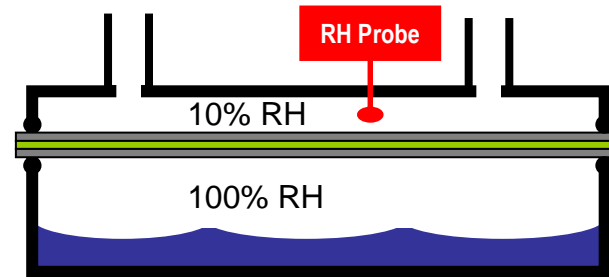
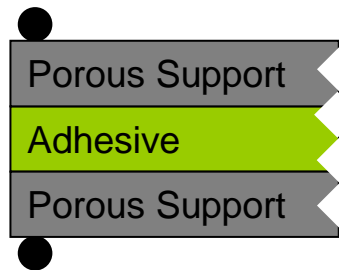
- Pressure Sensitive Adhesives (PSAs)
  - *Non-reactive towards common device materials*
  - *PSAs are used in aircraft, automobiles and buildings as structural adhesives*

# Adhesion Testing



# Adhesive Bulk WVTR

Using Mocon Permatran 150 (accumulation mode), 90% RH differential, ASTM E398. Adhesive laminated between water permeable film supports:



**For a 25 μm thick Barrier PSA:**

**For 25 μm thick typical OCA:  
(optically clear adhesive)**

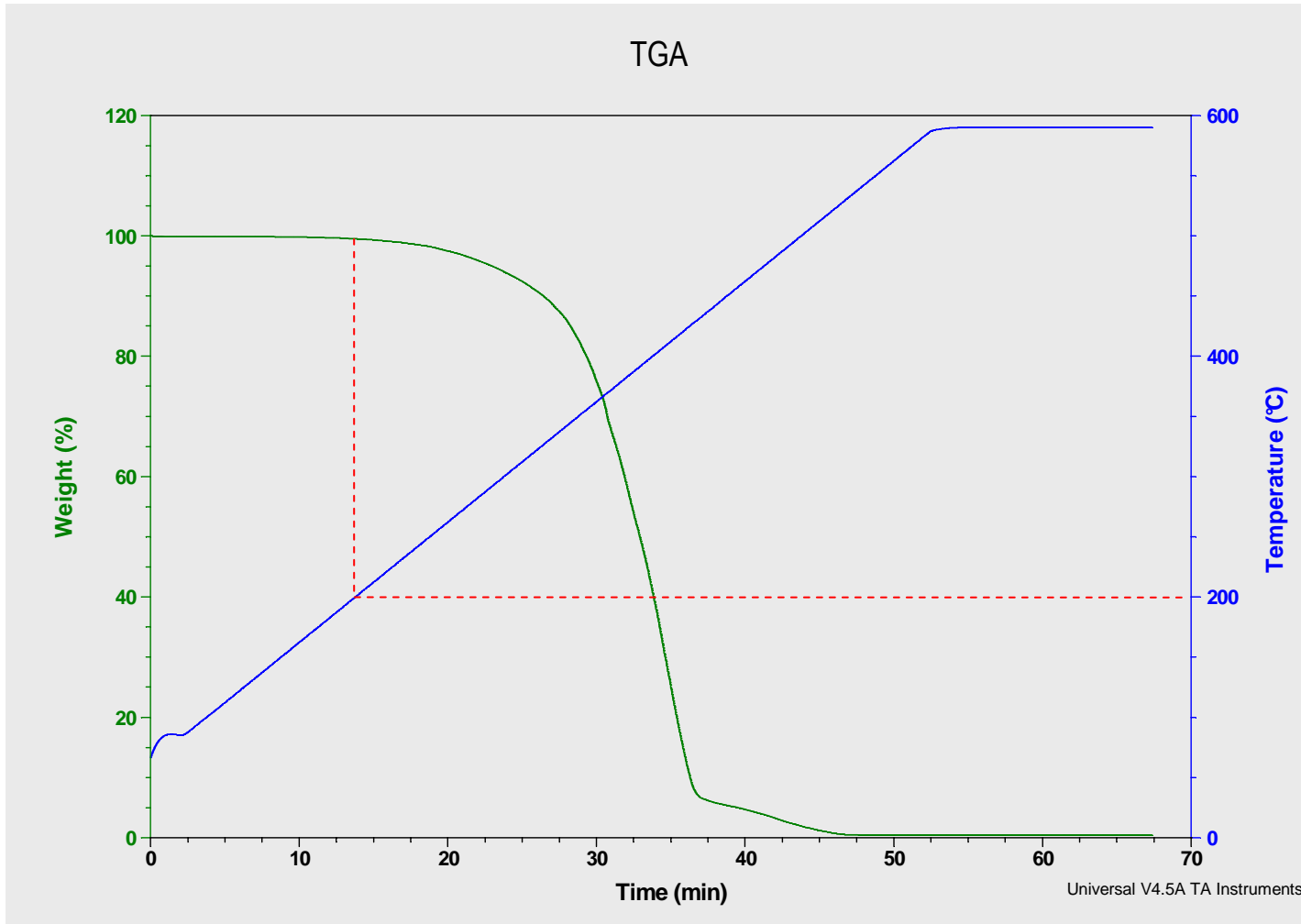
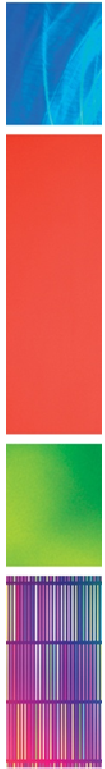
WVTR at 20 °C      1.9 g/m<sup>2</sup>-day  
 WVTR at 25 °C      3.4 g/m<sup>2</sup>-day

182 g/m<sup>2</sup>-day  
 292 g/m<sup>2</sup>-day



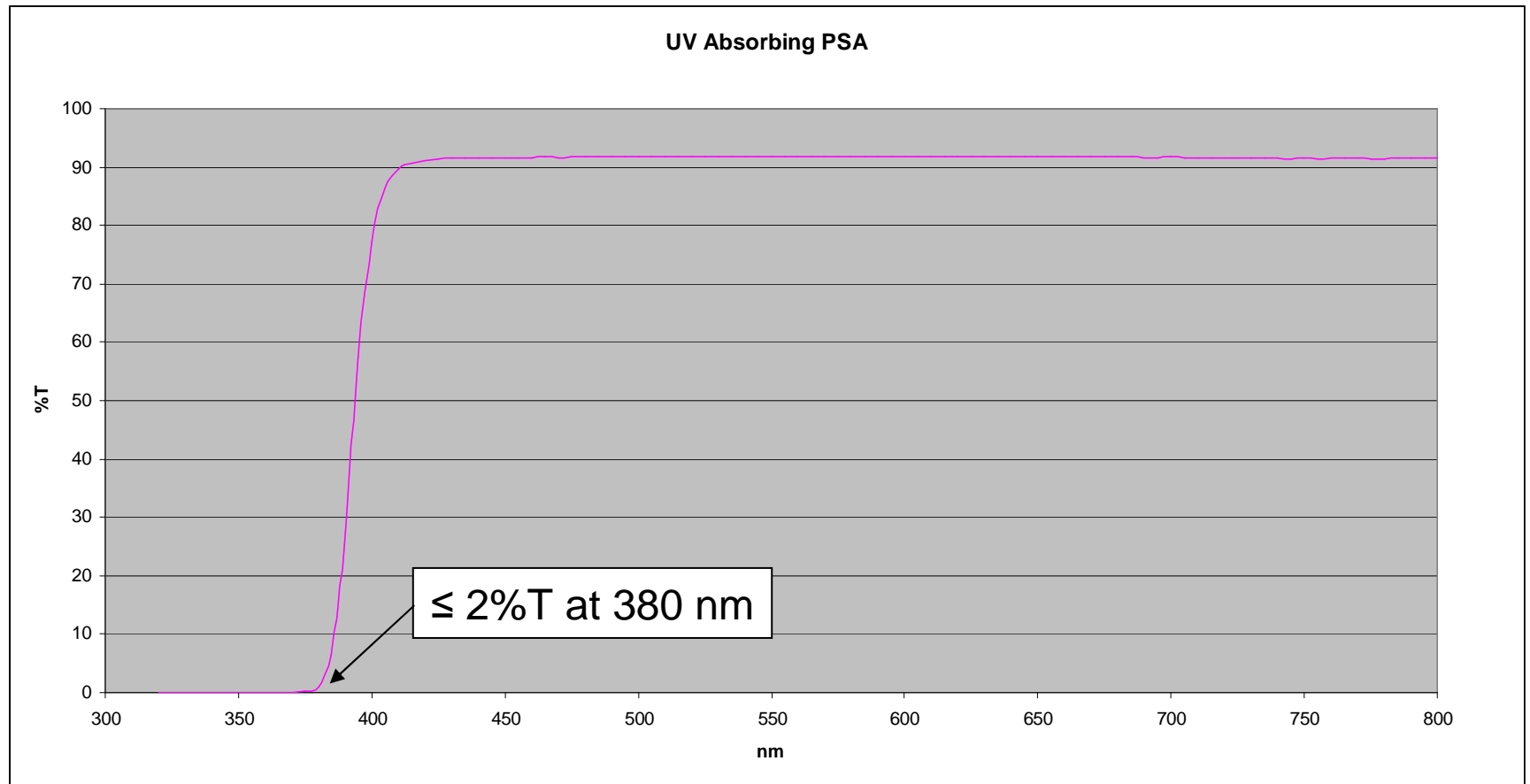


# Adhesive Thermal Stability



**10% weight loss at about 300 °C, little or no loss up to 200 °C**

# Adhesive Optical Properties (25 $\mu\text{m}$ Barrier PSA)

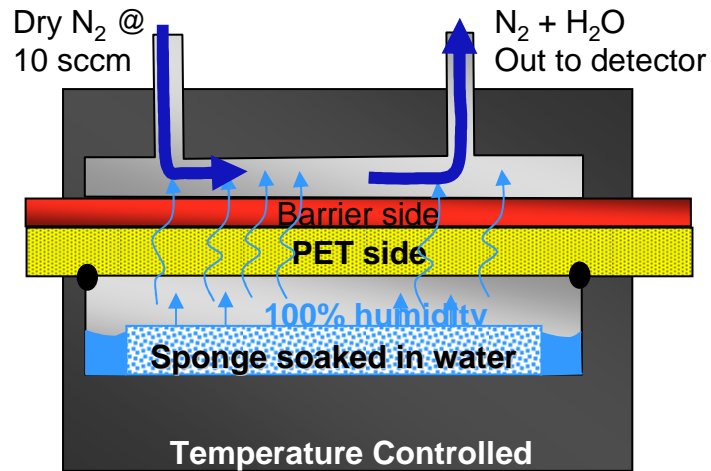


# Preliminary Barrier PSA Specifications



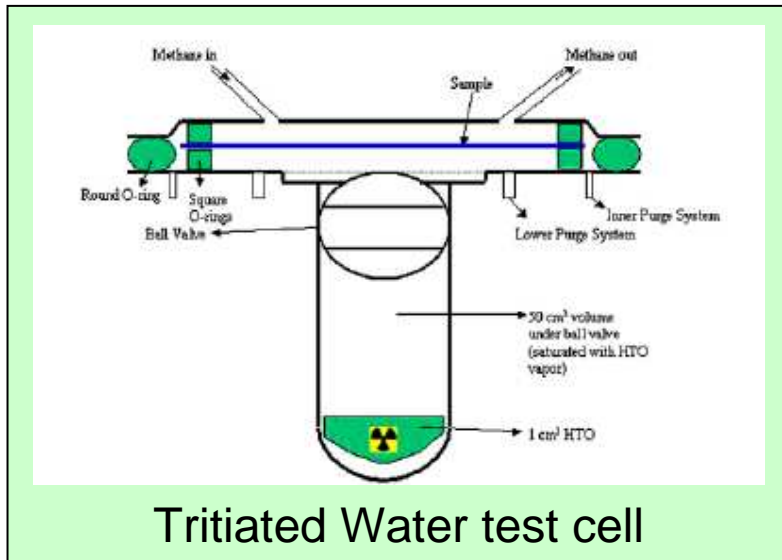
- FTBA-25
- 25  $\mu\text{m}$  standard thickness,  $\sim 10\text{-}50 \mu\text{m}$  also possible
- Between liners or pre-applied to barrier film
- A4 sheets or up to 650mm wide rolls
- 2-4  $\text{g}/\text{m}^2\text{-day}$  bulk adhesive WVTR (Mocon 150)
- $>87\%T$  in the visible range,  $<2.5\%$  haze
- UV cutoff feature;  $<2\%T @380\text{nm}$
- Stable to 200C
- Peel and shear properties typical of optical laminating adhesives

# WVTR - Mocon and HTO Methods

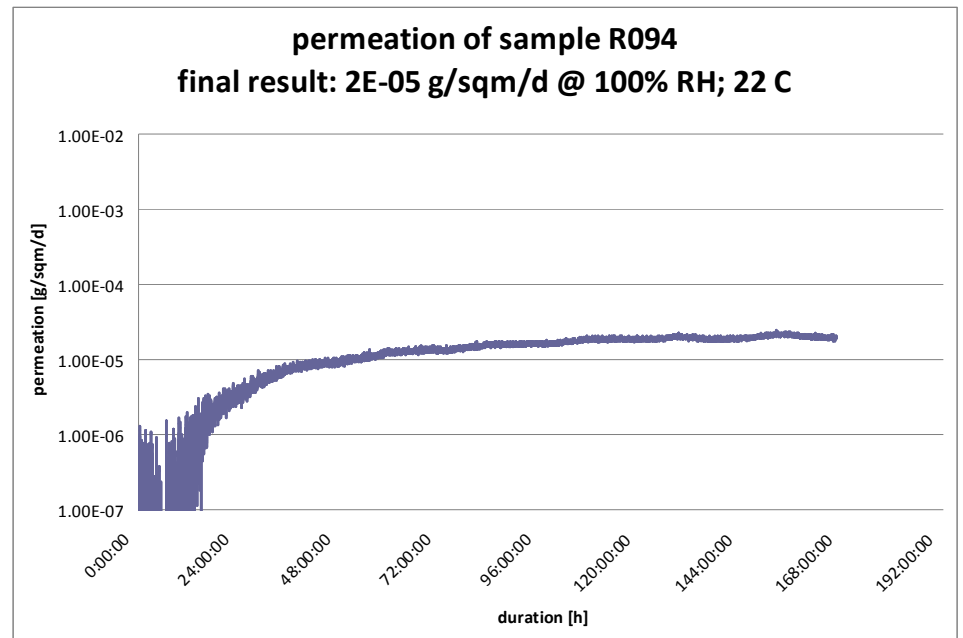


Mocon test cell

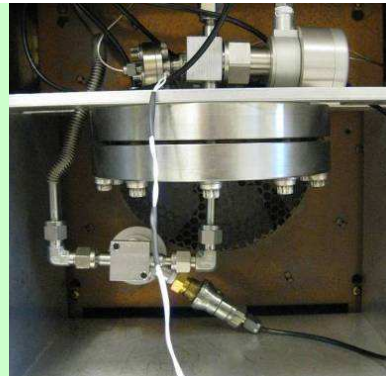
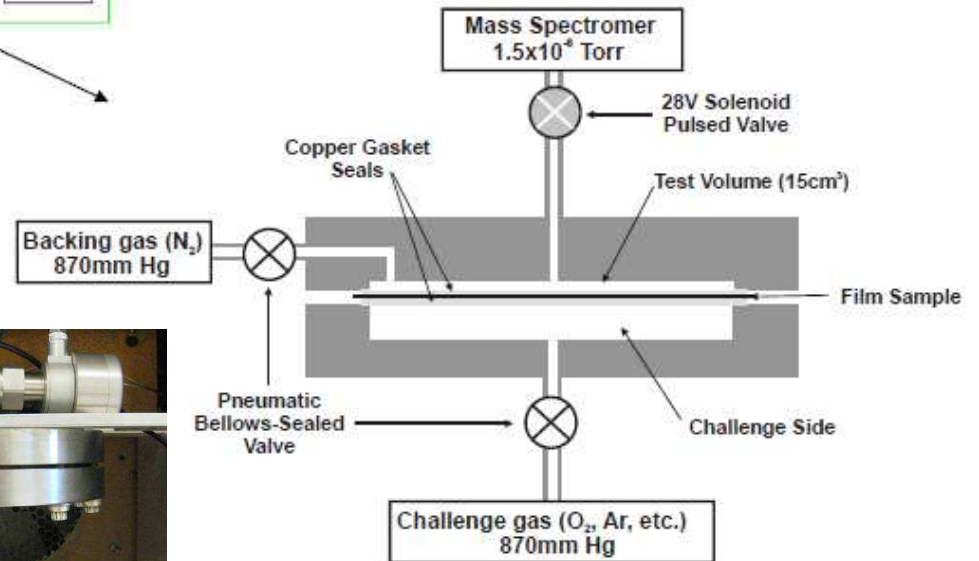
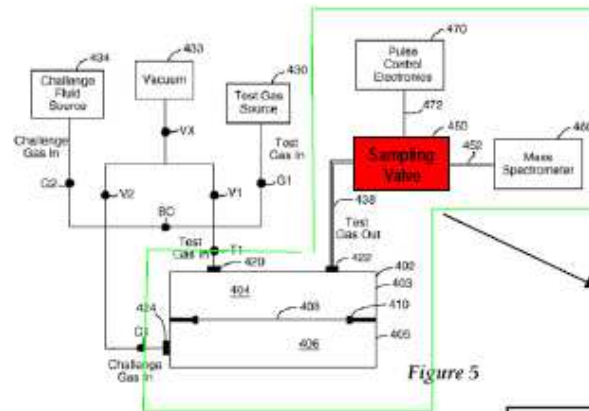
- Same basic technique
- Different detectors
- Different detection limits
- 3M 1-dyad films are-
  - Below Mocon detection
  - $10^{-5}$  by HTO ↘



Tritiated Water test cell



# WVTR – 3M Mass Spec Method



## Key features

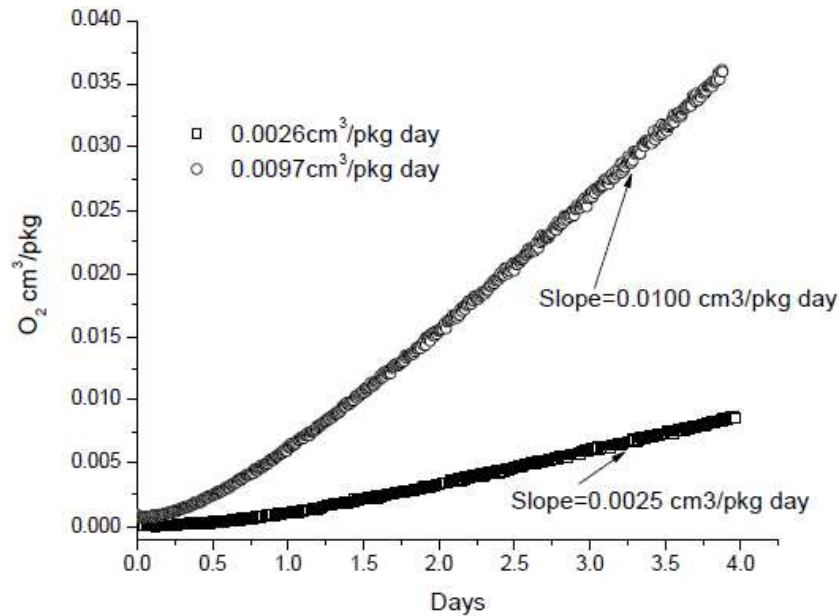
- Precise, small volume sampling valve
- Accumulation Mode
- No pressure gradient
- Calibrate with known gas standards
- Monitor several gases simultaneously
- US Patents 7,552,620 and 7,555,934



# WVTR – 3M Mass Spec Method



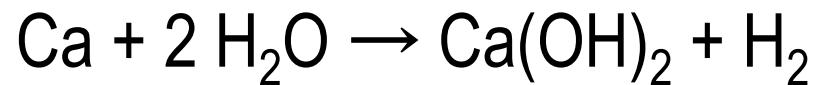
- Tested system versus Oxygen Certification Films-



- Now exploring Helium as the challenge gas
  - *Fast, < 4 hour test time*
  - *Measuring defect density?*
  - *Need to correlate He transmission rate with WVTR*

## WVTR – 3M Calcium Test Method

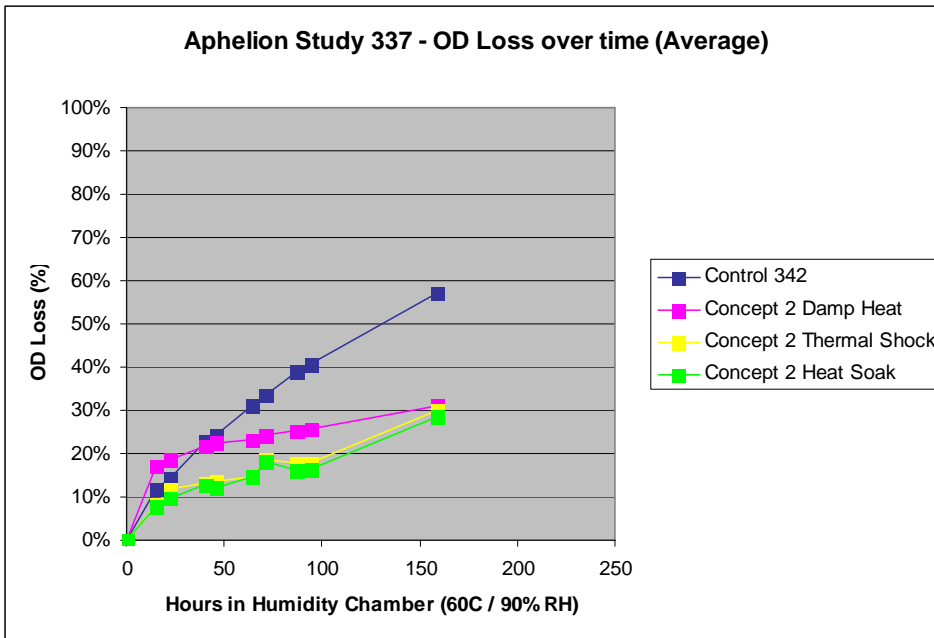
- Use thin film of Calcium as an indicator for moisture



- Encapsulate Calcium and monitor loss
  - *Optical Density or Transmission*
    - *Metal mirror → clear oxide*
  - *Resistivity*
    - *Conductive metal → insulating oxide*
- 3M using optical density measurements and image analysis

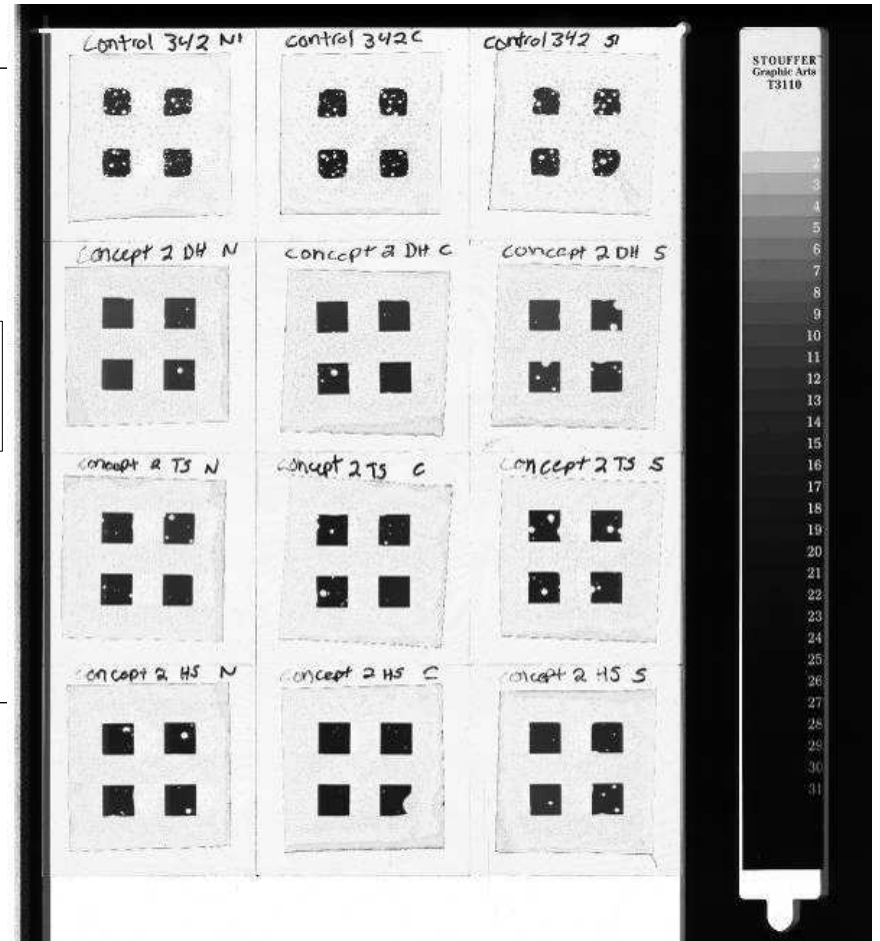
# Calcium Test Example

## EPD Laminated Barrier/HC Films

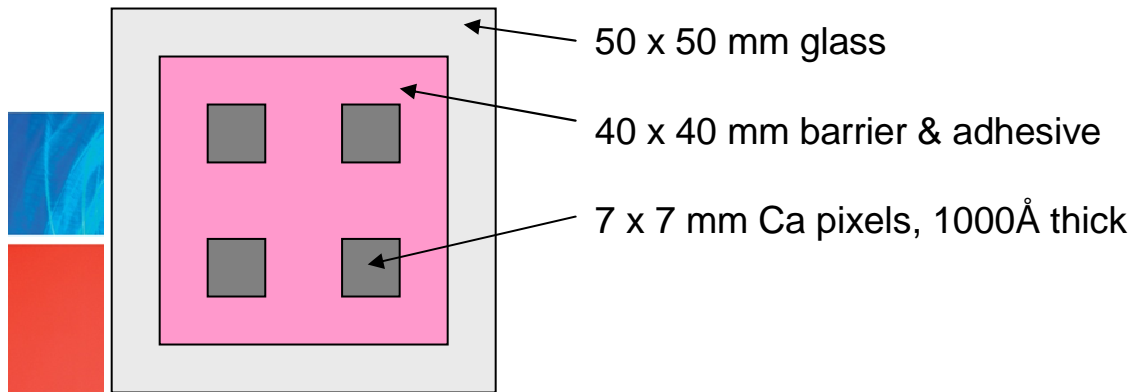


Can also track-

- Number of pinholes
- Pinhole area
- Ca pixel area



# Deriving WVTRs from Ca Data

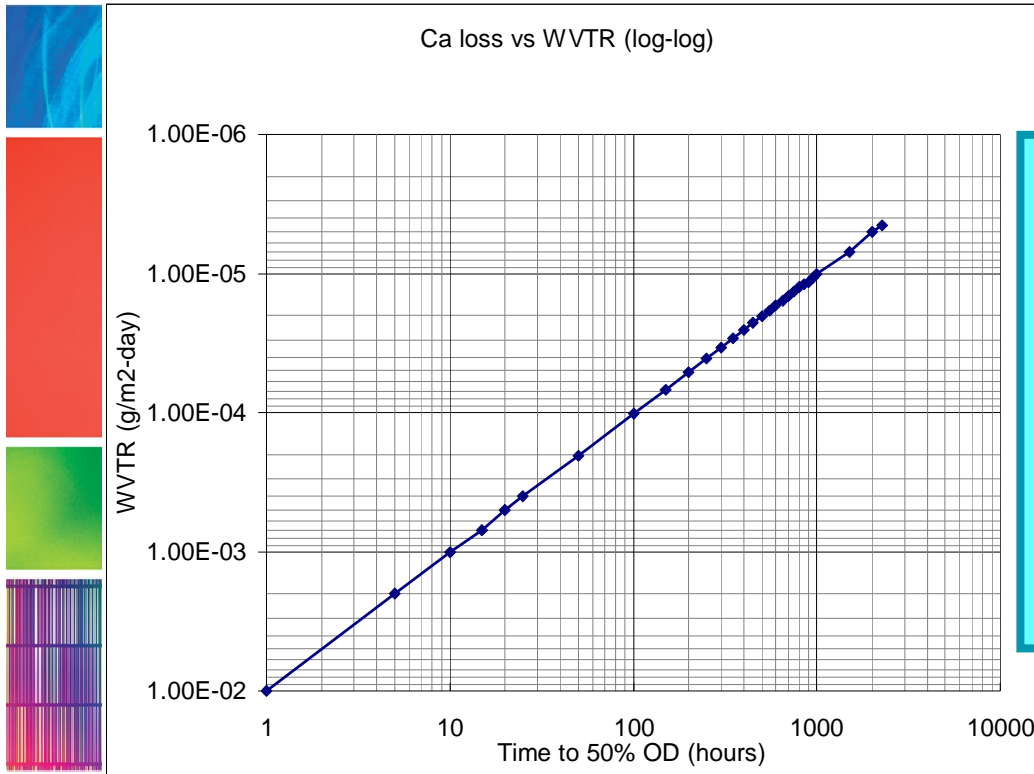


Place in 60/90 environmental chamber and monitor Ca optical density loss

- Amount of Calcium in sample is known
- Reaction stoichiometry is known:  $\text{Ca} + 2 \text{H}_2\text{O} \rightarrow \text{Ca(OH)}_2 + \text{H}_2$
- Area of barrier film/adhesive is known
- Assume Ca half optical density corresponds to consumption of half the Ca in sample
- Assume an acceleration factor of 20
  - generally accepted that 1 hour at 60/90 is equivalent to 24 hours at ambient

$$\frac{\text{Water required to consume one half of the Ca}}{(\text{Area of barrier \& adhesive}) (\text{Time to half optical density})} = \text{WVTR of the film plus adhesive g/m}^2\text{-day}$$

# Deriving WVTRs from Ca Data



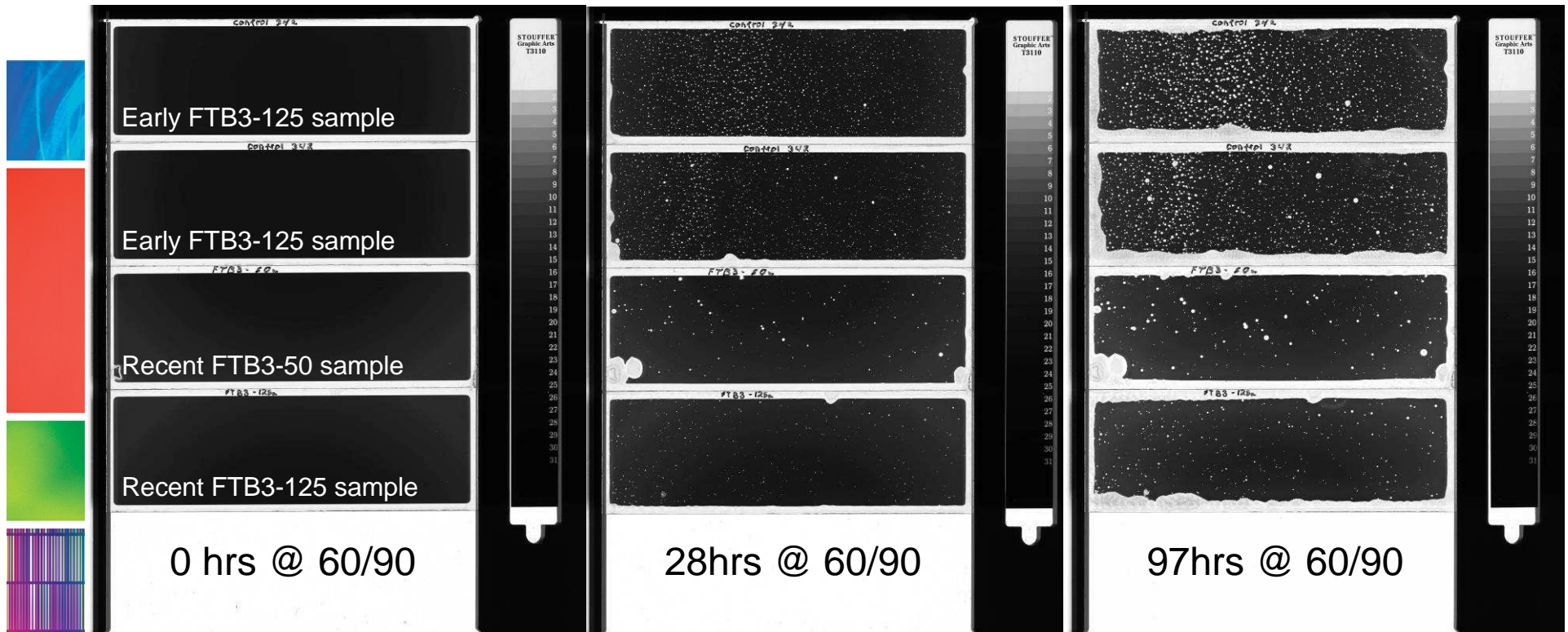
**Time to Ca half density at 60/90-**

- 10 hr =  $1 \times 10^{-3}$  g/m<sup>2</sup>-day WVTR
- 100 hr =  $1 \times 10^{-4}$  g/m<sup>2</sup>-day WVTR
- 1000 hr =  $1 \times 10^{-5}$  g/m<sup>2</sup>-day WVTR
- 10000 hr =  $1 \times 10^{-6}$  g/m<sup>2</sup>-day WVTR

- at ambient conditions

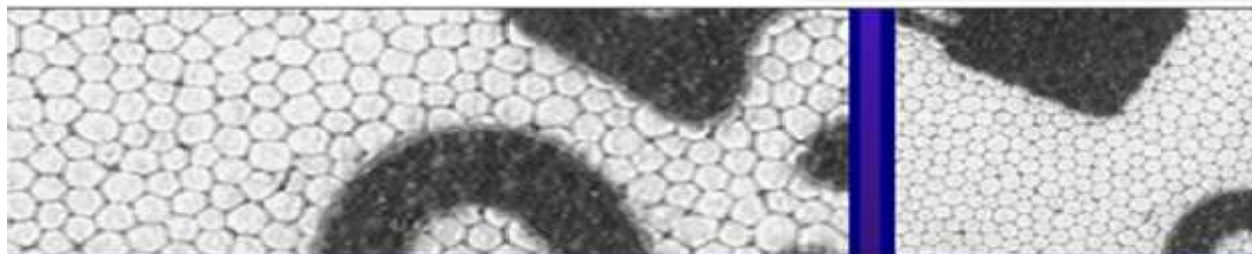
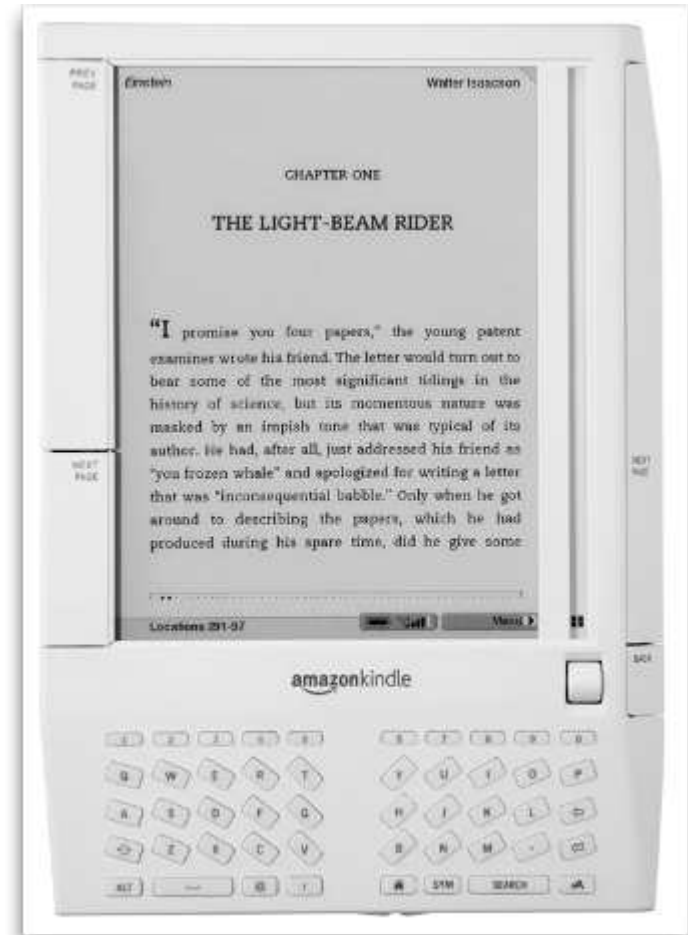
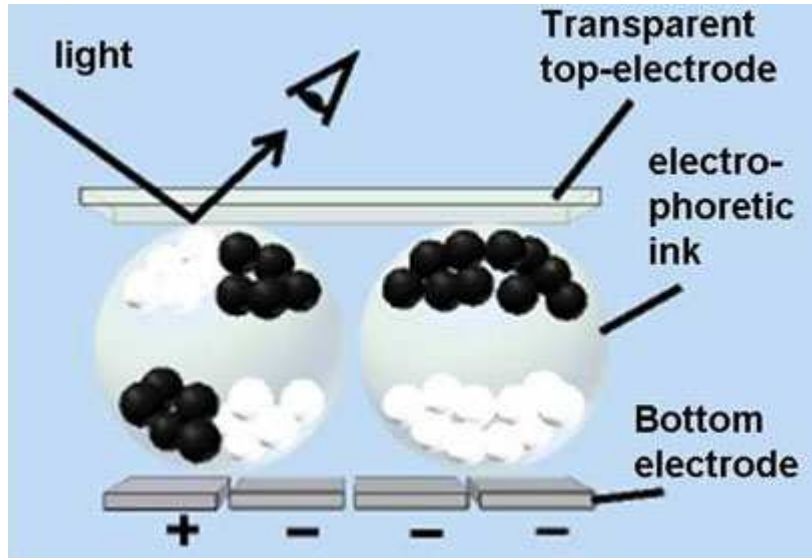


# Large Area Ca Test Under Development

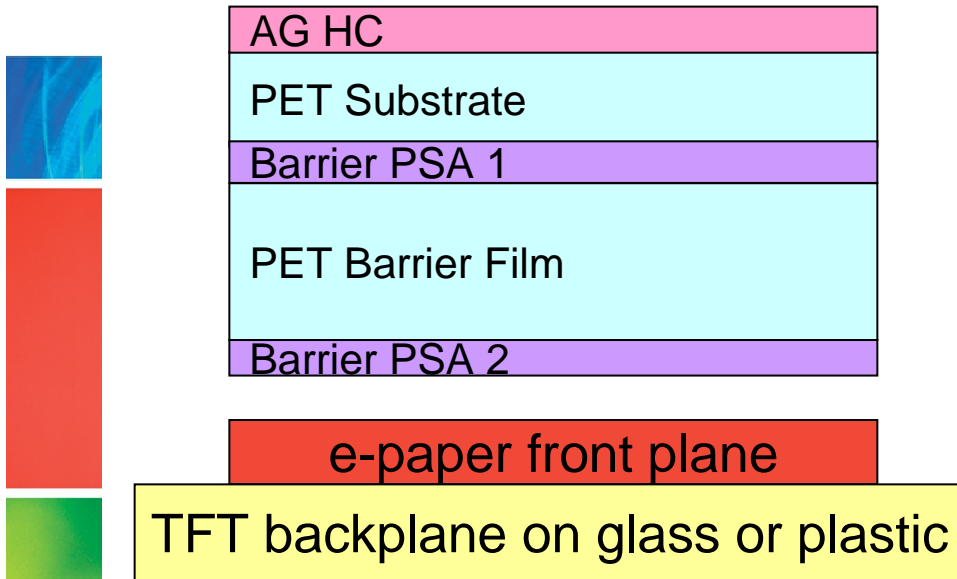


- 150 x 50 mm glass and barrier film & adhesive
- 140 x 40 mm calcium area
- Can reveal patterns of defects and scratches
- Apply image analysis techniques
- Shows steady improvement in barrier film performance

# Amazon Kindle E-Book



# Barrier Laminates for E-Paper Displays



## Film requirements

- $\leq 10^{-2}$  g/m<sup>2</sup>-day WVTR
- Low pinhole defect density
- 3H pencil hardness
- Antiglare (5-10% haze)
- 200-300  $\mu$ m thick
- Optional PSA 2
- UV cutoff at 380 nm

3M Film	WVTR g/m <sup>2</sup> -day at 50C	Avg %T 550 nm	Avg %T 450-650nm	Avg %T 380 nm	Avg %Haze	Clarity	Pencil Hardness
A	< 0.005	89.47	89.55	0.14	9.79	63.05	3H
B	< 0.005	90.86	89.45	1.86	8.66	64.80	3H
C	< 0.005	91.49	91.33	1.85	8.59	65.46	3H

# Shelf Tags with 3M Barrier Film



**Pervasive Display, Inc.**  
**ECO-Signs**  
([www.pervasivedisplays.com](http://www.pervasivedisplays.com))  
Deployed in Grocery stores in Taiwan



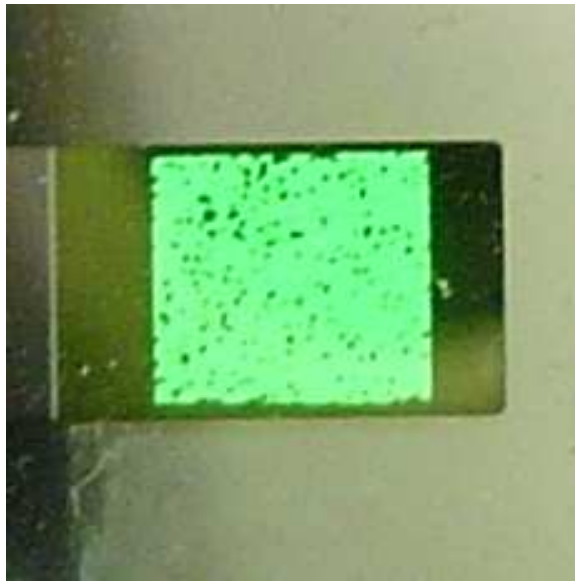
- 2" shelf label
- Wireless data refresh



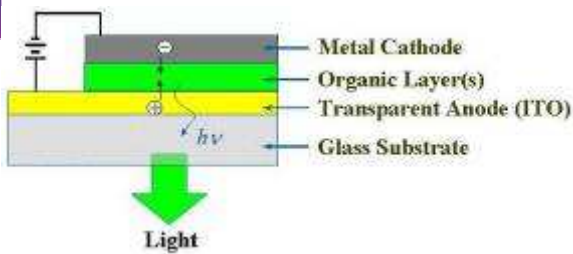
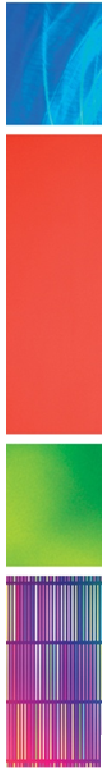
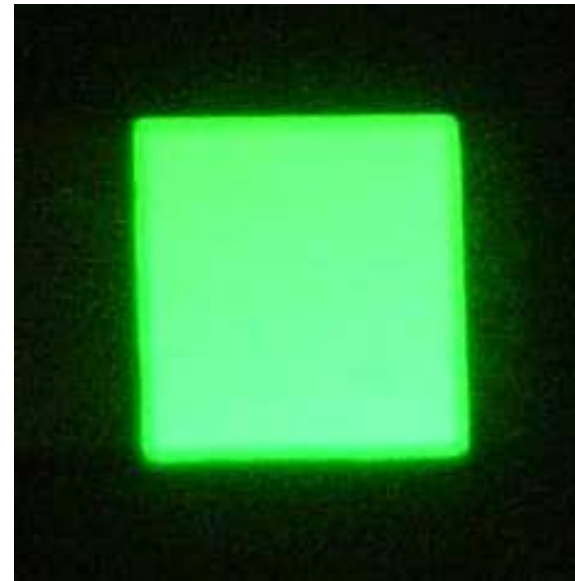


# Moisture & Oxygen Cause OLED Dark Spots:

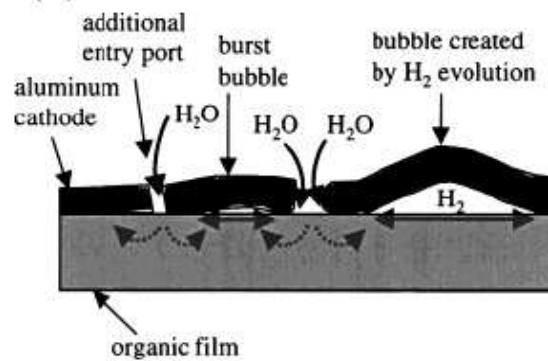
Barrier film with conventional PSA



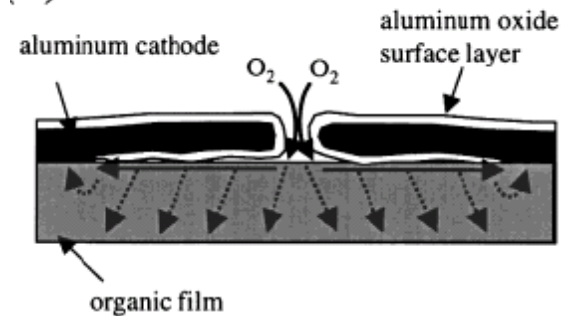
Barrier film with barrier PSA



(a)



(b)

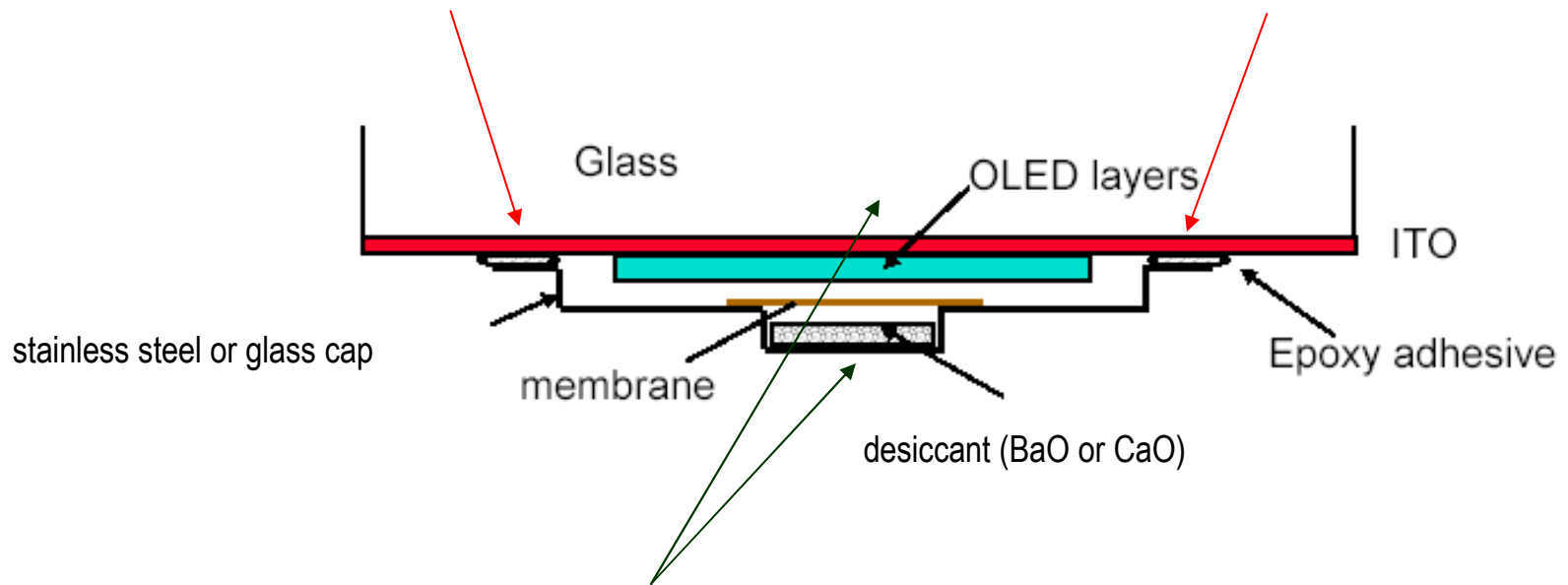


From Schaer et al., Adv. Funct. Mater. 2001, 11, 116-21

# Conventional OLED encapsulation

Rigid encapsulation: glass(metal) cap + adhesive + desiccant

**Replace epoxy with 3M barrier PSA**



**Replace rigid substrate and cap with 3M Barrier Film**



# OLED Flexible Barrier Requirements



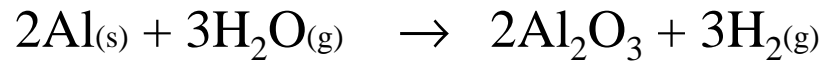
- Flexible barriers offer advantages
  - *Thin & rugged rigid devices*
  - *Fully flexible devices*
  - *Simple lamination encapsulation processing*
  - *Enables roll-to-roll processing*
  
- $10^{-6}$  g/m<sup>2</sup>-day WVTR
  - *Generally accepted as the OLED barrier target*
  - *Calculation based on aluminum cathode corrosion*
  - *May be dependent on device design and cathode choice*
  - *Thin film encapsulation and desiccants can compensate for higher WVTR barriers*
  
- **OLED displays using circular polarizers need non-birefringent barriers**
  - *3M is pursuing non-birefringent transparent barrier films*
    - *TAC, COP, PC.....*
  - *OLED lighting may be able to use PET*
  
- Adhesive needs
  - *What WVTR is required?*
  - *How to incorporate desiccant into flexible encapsulation schemes?*
  - *Epoxy edge seal plus desiccant seems to work for glass-glass OLED encapsulation*



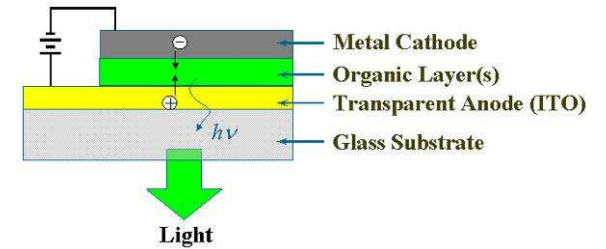
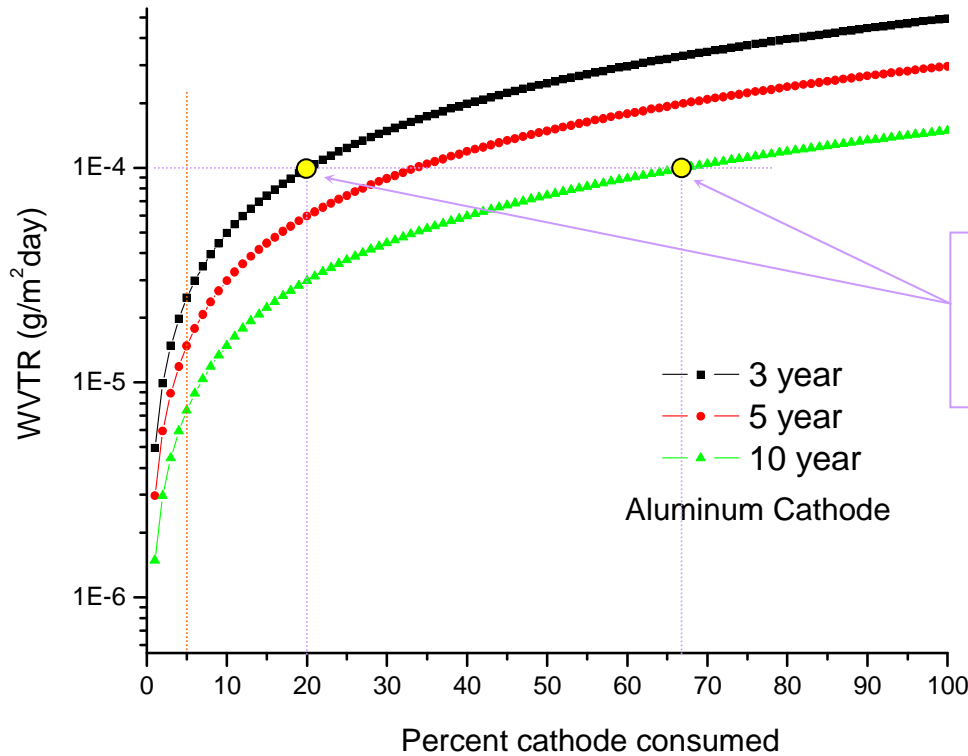
# Why $10^{-6}$ g/m<sup>2</sup>day?



Number based on consumption calculation for a 2000Å thick aluminum cathode

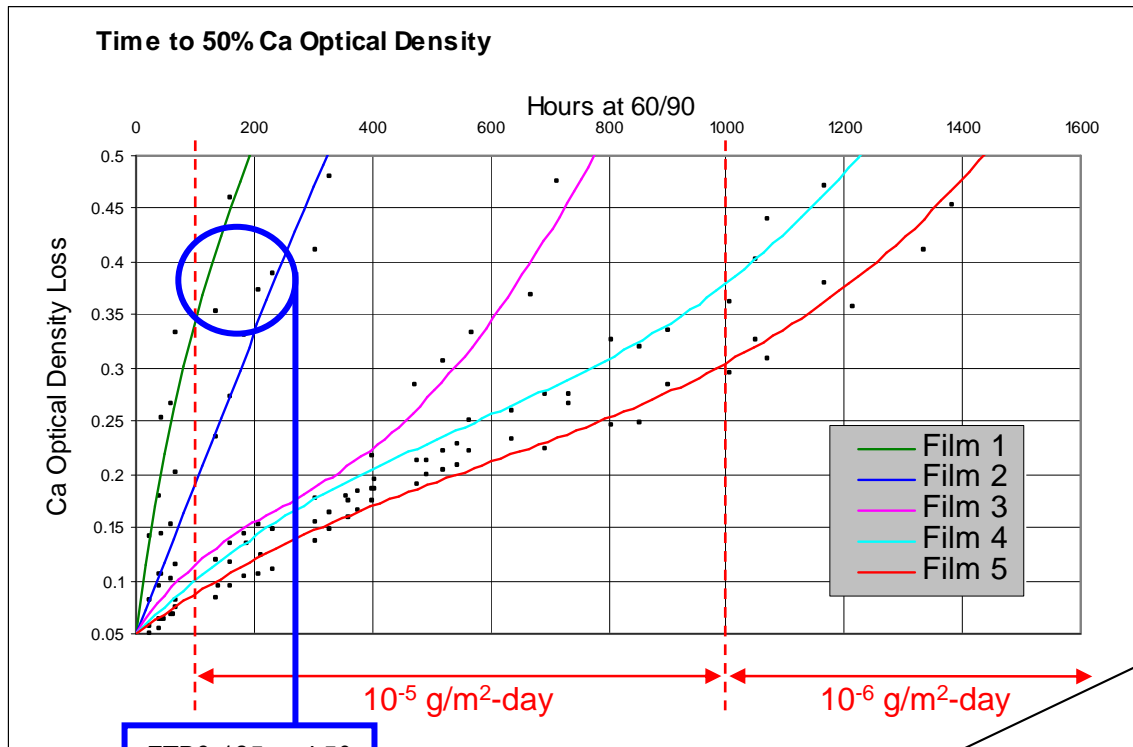


Calculated WVTR vs Percent Cathode Consumed

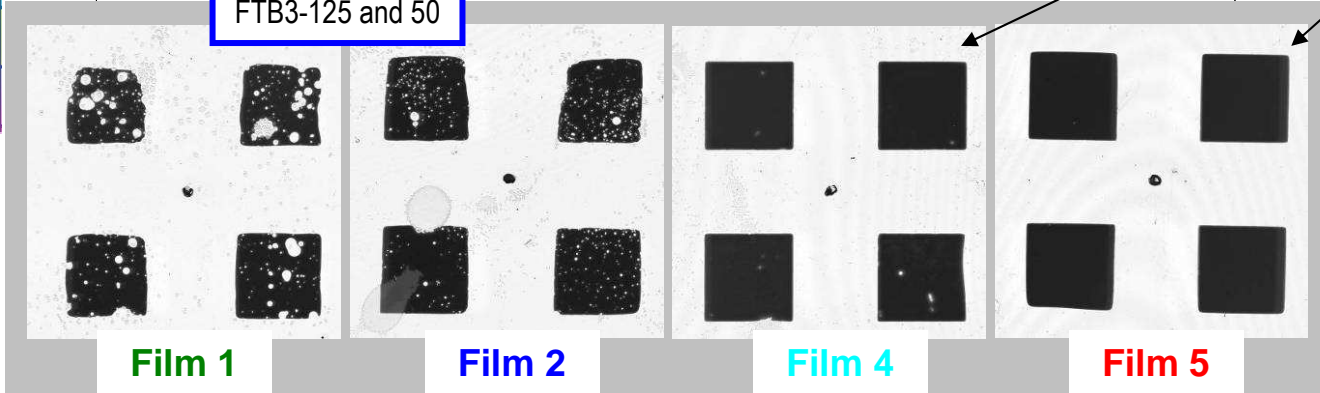
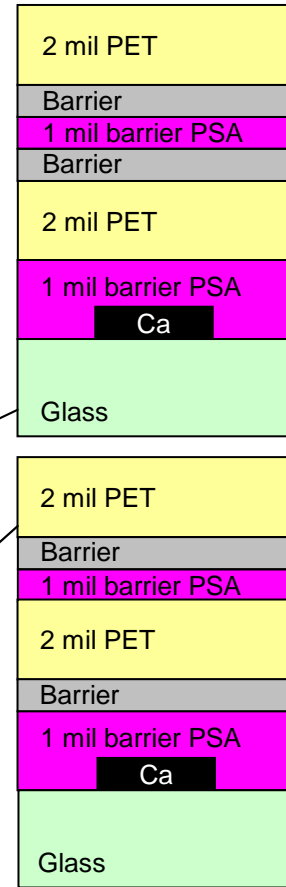


For a  $10^{-4}$  barrier, about 20% of the cathode would be consumed in 3 years and about 67% in 10 years

# Ca WVTRs Showing $10^{-6}$ Barrier Performance

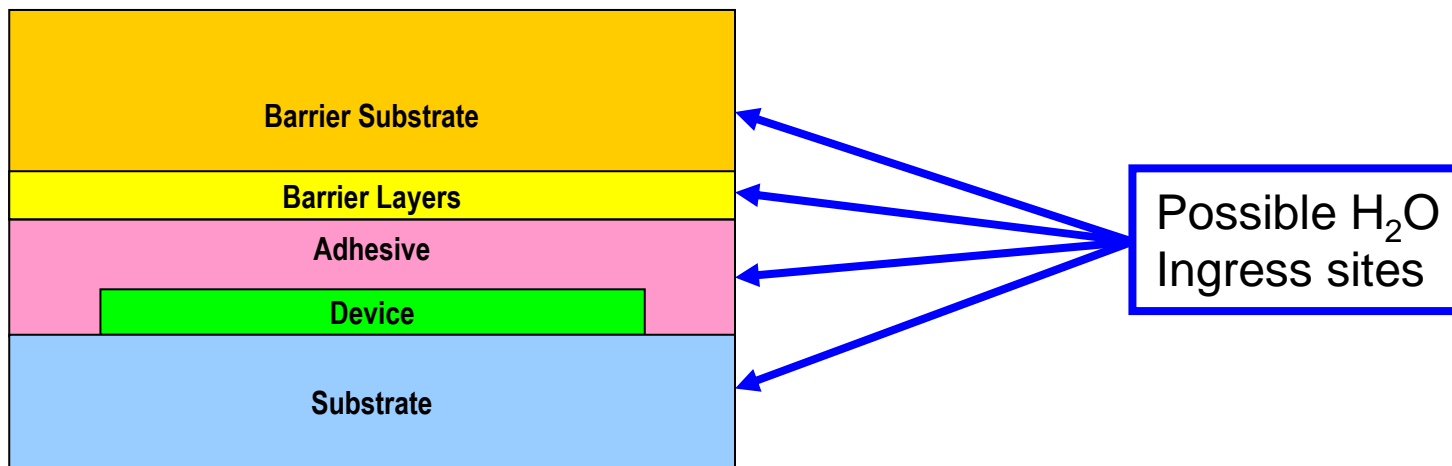


FTB3-125 and 50

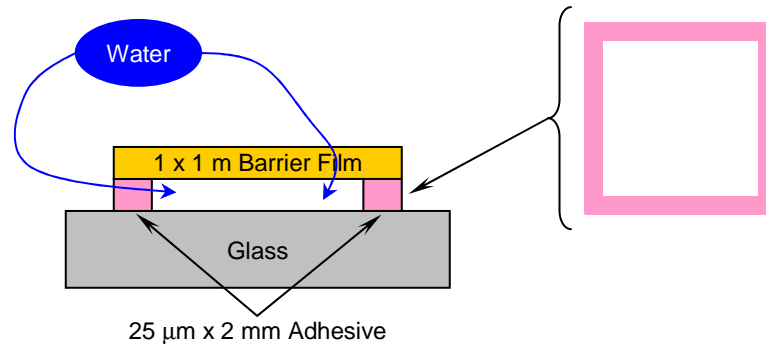


# Edge Effects

- How critical is moisture ingress from exposed film or adhesive edges?
- Knowing bulk WVTR properties calculate contribution from each component in device of interest:



# Adhesive Bondline WVTR Calculations



- Calculate the amount of water passing through a 2 mm wide bond line of a 1 by 1 meter device
- Compare result to the barrier film WVTR

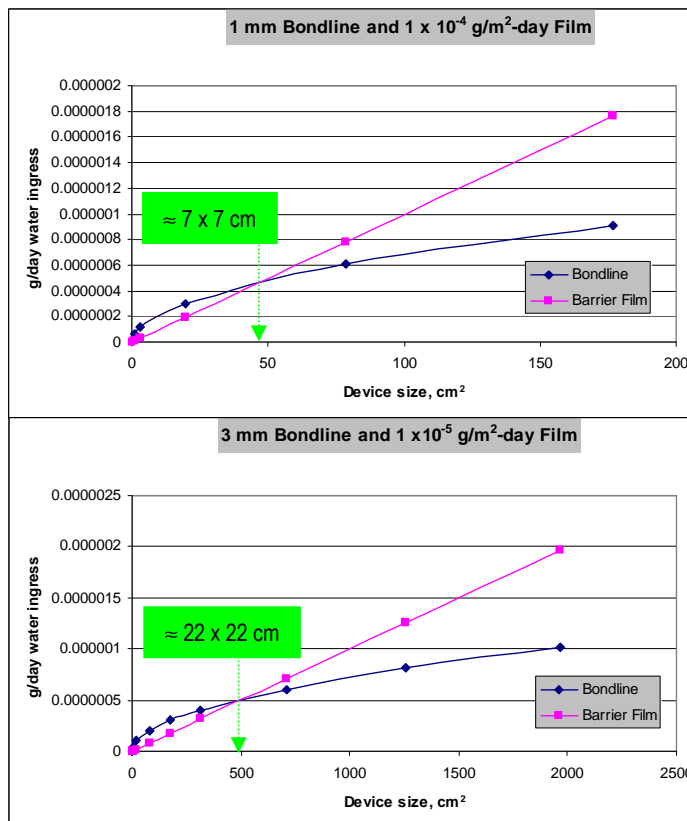
*In this example-*

The amount of water entering the through the bond line is  $5 \times 10^{-6}$  g/day

**3M's barrier PSA is well matched to "OLED barrier" film requirements in large device sizes.**

# Bondline WVTR Calculations – Smaller Devices

- Device perimeter and area do not scale linearly with device size
- Calculate the water leakage due to film and bondline
  - Different film WVTRs, bondline widths, and circular device sizes
- Determine where the water leakage rates for the film and bondline are the same:

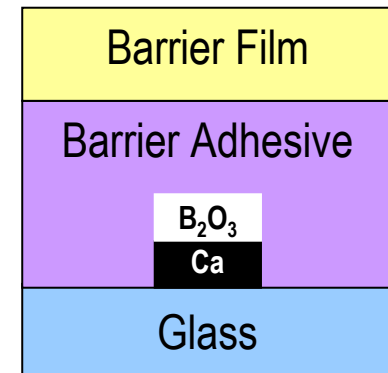
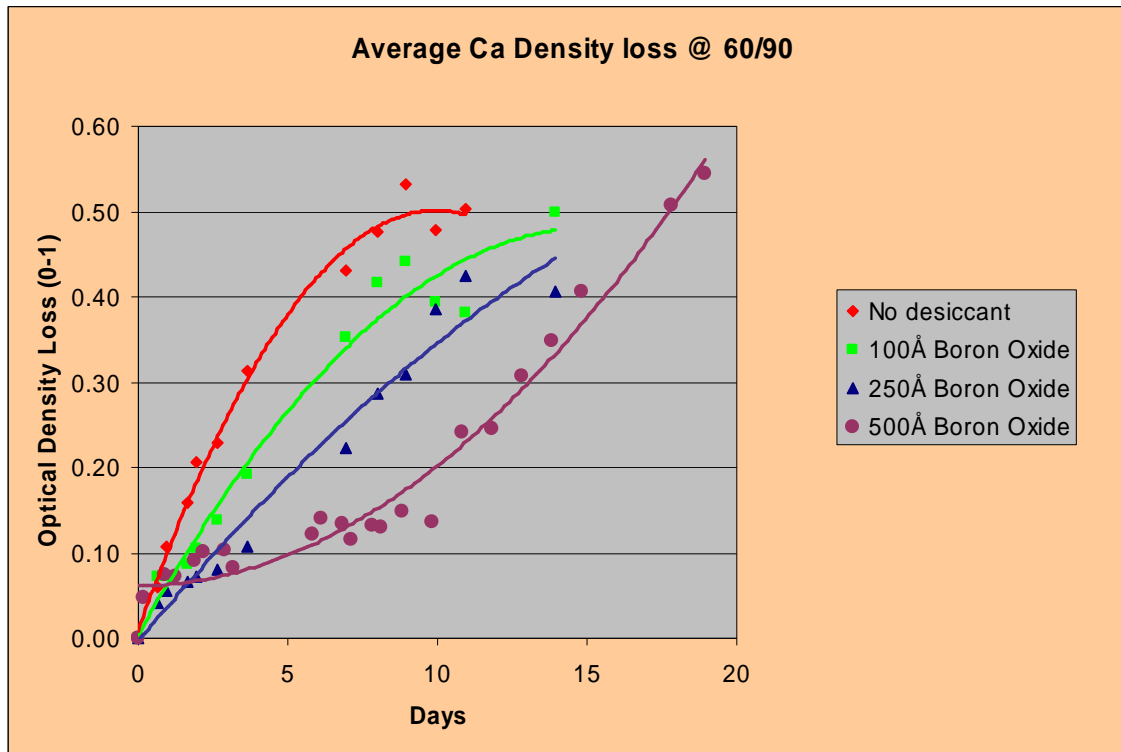


*Bond line requirements can be calculated for essentially any device size and shape*

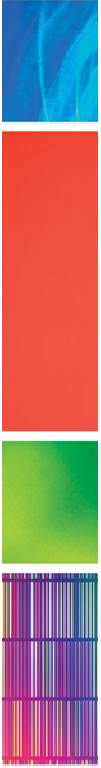
*Barrier property mismatch can be addressed with desiccants, thin film encapsulation, more stable display materials and electrodes*

# Desiccant Effect

- Boron oxide thermally evaporated over Ca coupons
  - Improves Ca durability at 60C/90%RH
  - Improves OLED device lifetimes
- $B_2O_3 + 3 H_2O \rightarrow 2 B(OH)_3$
- Boron Oxide forms clear glassy film; can be in the optical path



# Summary

- 
- 3M is developing a family of barrier products
    - *Wide range of applications*
      - *Targeting EPD and OLED markets*
  - Encapsulation system
    - *Barrier Film*
    - *Adhesive*
    - *Value add features; AG/HC, UV cutoff*
  - Sampling and R&D sales now, EPD barriers commercial in Q4 2011
    - *A4 sheets, 300mm wide rolls*
  - Wide width production coater in early 2012
    - *Up to 2 meter wide*
  - Unique WVTR test methods developed
    - *Calcium image analysis and Mass Spec*



**Important Notice**

3M does on occasion provide internal 3M test data as a service to our customers. 3M does not certify the accuracy or validity of this information and 3M is not responsible for Customer's interpretation of or use or misuse of the provided information. For example, Customers should not use this information for business purposes, including but not limited to setting test criteria, developing a specification or evidence of meeting a specification, or as a basis for determining if a product is fit for a particular application or will have particular attributes. Product attribute claims should not be based on this information.

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