

# Oxygen Permeability Enhancements in Octene LLDPE Film Affected by LDPE Blending and Crystalline Orientation Changes

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**APPLICATION STATEMENT:** This study provides practical methods for increasing oxygen and moisture vapor transmission rates of thin gauge monolayer or coex films containing Octene LLDPE blended with LDPE. The blends discussed are particularly useful for flexible packaging films used to package respiring fresh-cut produce.

## ABSTRACT

The permeability of blown films made from Zeigler-Natta and single-site catalyzed octene linear low-density polyethylenes blended with branched low-density polyethylene was studied. At thinner film gauges, oxygen and moisture permeation rates of certain blends were significantly higher than the expected values. The films with the highest permeability had a corresponding higher degree of polymer crystal orientation in the film TD – ND plane. While PE film permeability rates typically decrease with increasing orientation, orientation in low crystallinity PE may align crystalline platelets parallel to the direction of permeant flow effectively increasing oxygen and moisture transmission rates. Certain blends of Octene LLDPE and LDPE showed higher levels of orientation and permeation in thin gauge films. Increased polymer relaxation times of these blends suggest that the LDPE helps induce and/or maintain orientation. Thus by increasing film draw-rate to maximize TD crystalline orientation and selectively blending LDPE to maintain orientation, the permeability of Octene LLDPE can be increased significantly.

## INTRODUCTION

Fresh-cut vegetables and other produce products continue to respire after harvest and will spoil rapidly if free oxygen in surrounding environment drops below critical concentrations. To maximize produce quality and shelf life, the produce must be refrigerated to slow respiration rates yet be packaged in an aerobic environment that allows respiration to continue at slow rates. Polyethylene films used to package fresh produce generally have relatively high oxygen and moisture permeation rates that match the respiration rates of the produce maintain an optimal environment within the package headspace.

The required permeability of the PE packaging film depends on the type of produce, package dimensions and several other factors. High permeability polymers such as elastomers and very low-density linear polyethylenes (densities < 0.910 g/cc) are frequently used to package high-respiring produce such as broccoli florets and certain leaf lettuces. While these materials provide high oxygen transmission rates (OTRs > 850 cc-mil/100in<sup>2</sup>-day-atm), they are relatively expensive and are often difficult to process and convert. Antiblock and other additives may alleviate certain processing, converting deficiencies but can negatively impact permeability, optics and sealability.

Blending high-pressure, low density polyethylene (LDPE) into Zeigler-Natta or Single Site catalyzed linear low-density polyethylene (LLDPE) is a common practice used in blown and cast film manufacturing to improve processability without sacrificing key film properties. LDPE blending can also reduce overall costs for elastomer and other expensive specialty resins. The presence of LDPE however can reduce the permeability of elastomer films. The Oxygen Transmission Rate (OTR) of PE polymer blends is usually predicted by weight averaging the Oxygen Permeability Values (OPV) of the individual components.

$$\text{Predicted Film OTR} = [(A)(X) + (B)(Y)]/t$$

Where:

A = Oxygen Permeability Value (OPV) of LLDPE base resin  
O<sub>2</sub>, or OPV units: cc-mil/100 in<sup>2</sup>-day-atm

B = OPV of LDPE blend resin

X = Weight percentage of LLDPE in the blend

Y = Weight percentage of LDPE in the blend

t = Final film thickness

Example: 2 mil film made from a blend of 80% LLDPE with 600 OPV and 20% LDPE with 500 OPV

Expected OTR = [(600)(.80) + (500)(.2)]/2 = 290 cc's O<sub>2</sub>/ 100 in<sup>2</sup>-day-atm

This equation provides relatively accurate OTR predictions for films with gauges of 1.5 mils or greater. The measured transmission rates of thinner gauge films however, have been shown to deviate from the predicted values. Thin gauge coextrusions of polypropylene and polyethylene frequently have lower than expected transmission rates presumably due to the higher PP chain orientation while thin gauge monolayer PE films have been shown to have higher OTR rates than predicted. These deviations suggest that the OPV (OTR normalized for film gauge) is not necessarily constant for certain polyolefins.

Recent studies have demonstrated that the OPV of monolayer blown films made from Octene LLDPE resins remained relatively constant with increasing film take up speed. (Increasing take up increases draw down ratio and decreases film gauge). When the same LLDPE resins were dry blended with 10 to 20 weight-percent high-pressure LDPE, the film OPV increased as take up speed increased/film gauge decreased. The OPV increases were more pronounced in films produced wider die-gap suggesting the increased permeability rates may be related to overall polymer draw ratios and thus polymer chain orientation.

This study examined the permeability rates of various Zeigler-Natta and single-site catalyzed Octene LLDPE monolayer blown films alone and in blends with two low-density polyethylenes. Specifically, the relationship between film morphology /orientation and permeation values were studied.

## THEORY

The Oxygen Permeability Value of a polymeric material is been shown to be the product of the diffusion coefficient and solubility of the permeant within the polymer matrix (1):

$$P = D \times S$$

This equation suggests that the movement of permeant molecules through a polymeric film has both chemical and physical aspects that must be considered. The diffusion coefficient corresponds to the velocity at which the permeant is able to travel through one side to the other side of the polymer film. A PE crystal is packed too densely for most permeant molecules to pass through. Thus the diffusion velocity in PE films is influenced by the percentage crystallinity and crystalline platelet orientation as the permeant moves along the path of least resistance. The solubility coefficient corresponds to the degree of electro-chemical compatibility between the polymer and permeant and is not significantly affected by the orientation of PE crystals.

The ability to change or control orientation of polymer crystals should thus enable one to control diffusion kinetics. While blending low levels of LDPE into a LLDPE rich blend is not expected to change overall levels of crystallinity substantially, a compatible and well-interspersed LDPE may affect the orientation of PE crystals (3). Several researchers have shown that polyethylene crystals form by regular polymer chain

folding into fairly uniform 10nm thick lamellae or platelets. These crystalline lamellae form orderly planes that radiate perpendicularly outward from the non-crystallized or amorphous polymer chains. When a PE melt is subject to the longitudinal stresses of film draw, the amorphous chains will align or orient along the machine direction of the film forcing the crystalline platelets to align parallel to the film TD (6). The degree of polymer orientation is proportional to the film draw rate introduced into the LLDPE melt. As the polymer melt relaxes, the polymer chains and crystalline platelets will revert to a random orientation.

Crystalline lamellae platelets aligned parallel to the direction of permeant flow: TD or normal direction (ND), create less barrier to diffusion compared with platelets aligned perpendicular to permeant flow direction. Therefore, the ability to maintain TD or ND orientation of crystalline platelets as the polymer melt relaxes should result in films with higher permeability. Low levels of LDPE blending resin in octene LLDPE increases polymer melt relaxation times which could subsequently maintain the TD or ND crystal platelet orientation preferred for breathable fresh produce packaging films.

The primary purpose of this study was to evaluate the effect of LDPE blending resins on the orientation of crystalline platelets in octene LLDPE and relate differences to film OPV. The secondary purpose was to identify film processing methods and LDPE blends that maximize permeability in films made octene LLDPE.

## MATERIALS AND METHODS

Three linear low-density polyethylene resins were used in this study. All three are nominal 1 dg/min melt index (MI), octene copolymers produced in a solution reactor. Two of these are advanced Zeigler-Natta catalyzed octene LLDPE produced in a single reactor and sold under the SCLAIR® brand name. The grade coded as zLL-912 has a nominal density of 0.912 g/cm<sup>3</sup> and the grade coded as zLL-920 has a nominal density of 0.920 g/cm<sup>3</sup>. The grade coded as sLL-917 is single-site catalyzed octene LLDPE produced in a parallel dual reactor with a nominal density of 0.917 and sold under the SURPASS® brand name. Properties for the LLDPE resins are summarized in slide 15.

Three tubular low-density polyethylenes were used as blending resins. The grade coded as LD-320 is a nominal 0.25 MI, 0.920 g/cm<sup>3</sup> density. The grade coded as LD-819 is a nominal 0.8 MI, 0.919 g/cm<sup>3</sup> density. The grade coded as LD-0222 is a nominal 2.0 MI, 0.922 g/cm<sup>3</sup> density LDPE. The LDPE resins are summarized in slide 16.

### Blown Film Production

Monolayer test films were produced on a Macro® blown film line with an 8-inch diameter spiral die and a 3.5 inch single screw, L/D=30. The line is equipped with a dual lip air ring and Internal Bubble Cooling (IBC). All films were run at a 2.5:1 blow-up ratio (BUR). LL and LD resins were dry-blended in a V-mixer and fed directly into the extruder hopper. Resin melt temperatures were held between 420 F (215 C) and 440 F (227 C). Most films were produced using a die gap of 100 mils and several films were produced using a die gap of 35 mils as a test variable. All other film processing conditions such as extruder output and temperature profiles were kept constant. Film gauges were changed by increasing or decreasing bubble takeup speeds at constant extruder output. Test films of similar blends were typically produced at the thickest gauge (2.0 mil) first and then progressively thinner gauges.

### Oxygen and Moisture Permeability Measurements

Test films were aged at 50% relative humidity and 23C for at least 1 week before testing. Oxygen permeation rates were tested under to ASTM D3985-05 conditions using a Mocon OX-TRAN®. Thickness for specific film test specimens was measured prior to testing. Individual test films were tested in triplicate and the average results were reported.

### Polymer Orientation by FTIR Dichroic Ratios

The degree of polymer chain orientation has been shown to correlate with dichroic ratios using various spectroscopic methods (2). The dichroic ratio is the ratio of absorbed energy for a polarized electromagnetic beam emitted parallel to the film MD and parallel to the film TD.

$$D = A_{//} / A_{\perp}$$

$A_{//}$  = Energy absorbance for beam transmitted parallel to the film machine direction

$A_{\perp}$  = energy absorbance for beam transmitted beam normal to the film surface

For this study, the dichroic ratios were calculated using transmission polarized FT-IR wavelengths of 720  $\text{cm}^{-1}$  and 730  $\text{cm}^{-1}$  corresponding to  $\text{CH}_2$  rocking frequencies. This method is similar to the method used by McRae and Maddams (2). The dichroic ratio D720 gives the relative PE unit cell b-axis orientation in the film MD and the amorphous chain orientation in the MD. As D720 decreases the b-axis (and amorphous chains) become less oriented with the MD and thus rotate toward the TD or ND. The Dichroic ratio D730 gives the relative orientation of the PE unit cell a-axis along the film MD. Since the D720 and D730 provide only relative directional changes for single axis within the PE unit cell and/or crystal, they cannot be used to provide a specific positioning of the PE crystal. If both D720 and D730 decrease however, the large crystal face is most likely aligning more along the film TD-ND plane.

### Polymer Orientation by X-ray diffraction

The three dimensional orientation of polymer crystals was characterized by wide-angle X-ray scattering using Relative Film Planar Orientation or RFPO.

X-ray reflection patterns from a PE powder show continuous concentric rings that represent random orientation of crystalline lamella. As crystals become oriented, the intensity of certain rings will change and degrade into segments or arcs indicating focused directional reflections from more orderly crystalline planes. In blown and cast films, the PE unit cell c-axis would align preferentially in the machine direction and certain reflection patterns would be more pronounced.

Relative Film Planar Orientation or RFPO is procedure for evaluating the relative alignment of (200) and (020) unit cell planes with the film MD. The degree of MD orientation is obtained from the degeneration of X-ray reflective rings into arcs. This degeneration is quantified by obtaining the intensity difference between the maximum and minimum for each reflection normalized to the minimum intensity. The extent of degeneration of the continuous concentric rings into arcs that become shorter as the orientation increases can be used to describe the degree of orientation. The intensity as a function of the azimuth angle will yield two maxima at the equatorial reflections, where the half peak width is used as a measure of the degree of orientation.

The RFPO (200) diffraction provides a relatively strong signal and may be the best diffraction to be used to monitor the degree of orientation. The value of RFPO for (200) is an indication of the degree of orientation of the a-axis in the MD direction. Considering the orthorhombic cell structure of PE crystals, lower RFPO (200) provides an indication crystal plane orientation in the film TD. As RFPO (200) values decrease, the crystal face will be less oriented in the film MD.

## RESULTS AND DISCUSSION

### Oxygen Permeation:

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Oxygen permeability results of the base components and blends are shown in Slides 22 and 23. The blends showed similar trends to those previously noted. OPV values for the non-blended samples remained fairly constant at different gauges while OPV values for the LDPE blended samples were significantly higher as drawdown increased/ film gauge decreased. One exception was the 100% zLL 0.912 density sample that, like the blended samples, had higher OPV at thinner gauges.

The type and amount of LDPE blending resin had some effect on OPV changes. In general, samples containing the maximum of 20% LD blend resin had more significant OPV increases at thinner gauges. Samples containing fractional melt-index LDPE had more significant OPV increases at thinner gauges than samples containing the 2.0 melt-index LDPE. Overall increases in OPV tended to be more significant for LL-LD blend combinations with similar viscosities at 230C and 10 sec-1 shear rate.

#### **Dichroic Ratios:**

The dichroic ratios showed two trends. The D720 ratios decreased with decreasing film thickness for both the 100% LL films and LL-LD blends. This indicates that the b-axis becomes less oriented in the MD and is rotating towards the TD or ND as drawdown is increased. The D730 ratios for the LL-LD blends typically decreased with decreasing film gauge indicating that the a-axis becomes less oriented in the film MD as drawdown. However, the 100%, non-blended LL films all showed increasing D730 ratios as film gauge decreased.

The D720 and D730 considered together suggest as the film gauge of the blended samples is reduced, the flat surfaces of the crystals are likely orienting parallel to the film TD-ND plane. This new orientation would lead to a reduction in path tortuosity that may account for the increase in film OPV for the thinner gauge blended films.

The decreasing D720 and increasing D730 ratios for the non-blended LL films suggest that the crystal orientation is changing with drawdown but not toward the TD-ND plane like the blended samples. The desired b-axis rotation away from the MD is likely offset the a-axis rotation toward the MD resulting in an orientation that leads to similar permeant path tortuosity for the sLL-917 and zLL-920 samples. This does reorientation not account for the unexpected increase in OPV for the zLL-912 samples. A possible explanation for this increase is that the b-axis becomes more oriented in the favorable ND direction although dichroic ratios does not account for ND orientation of the b-axis.

#### **X-Ray Diffraction: RFPO (200):**

Relative Film Planar Orientation results (200) are provided in chart 26 and graphs 27, 28 and 29. For all blended film sample sets, the OPV was inversely related to the RFPO (200) value. I.e. as the a-axis becomes more aligned with in the MD, permeability decreases. This trend was consistent with the D730 dichroic ratios. As the a-axis becomes oriented along film MD (large RFPO 200) the b-axis orientation would have more effect on path tortuosity and permeability. It is therefore not surprising that as the RFPO 200 increased, the data showed more scatter of data points from the trend line. It is likely that the samples with high RFPO 200 and higher OPV had crystal b-axis oriented more toward the ND.

## **CONCLUSIONS**

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Oxygen and moisture permeability rates of thin gauge blown films made from Ziegler Natta or single-site catalyzed octene LLDPE are significantly affected by the combined influence of polymer melt draw rates and the presence of certain LDPE blend resins. The increase in permeability is related to orientation of crystal lamellae within the polymer film. When the large crystal surface is aligned along the MD-TD plane, the permeant path is very tortuous and permeability rates are relatively low. Permeability rates increase as the crystal face aligns away from the MD-TD plane and toward either the MD-ND plane or MD-ND plane.

Film manufactures can generally increase the permeability of a thin gauge film made from Ziegler Natta or single-site catalyzed octene LLDPE by running thicker die gaps to increase polymer melt draw rates and blending in low levels of certain low-density PE to maintain the optimal orientation of crystal lamellae. A low-density blending resin with similar melt viscosity will generally provide the most significant improvement in film permeability.

#### **ACKNOWLEDGEMENTS**

The authors acknowledge the support from NOVA Chemicals Corporation and the assistance of several colleagues this study.

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# Oxygen Permeability Enhancements in Octene LLDPE Films Affected by LDPE Blending and Crystalline Orientation Changes

Presented by:

**Dan Ward – Technical Service Specialist**

 **NOVA** Chemicals®



# Permeability Requirements for Fresh Produce Flexible Packaging

Product	Respiration Rates* at 15°C (mg O <sub>2</sub> / kg product / hr)	Typical OTR minimum required for packaging film
Onion	< 10	100 – 200
Cabbage, Tomato	10 – 20	200 – 400
Lettuce, Radish	40 – 70	250 – 500
Spinach, bean	70 – 100	400 – 700
Broccoli Florets	> 100	> 600

\*From B.S. Luh, *COMMERCIAL VEGETABLE PROCESSING* 2nd edition: AVI/Van Nostrand Reinhold, New York, NY 1988

# Permeability Units and Conventions

- **WVTR**; Water Vapor Transmission Rate
  - **Units**; grams/100 in<sup>2</sup>-day
- **OTR**; Oxygen Transmission Rate
  - **Units**; cc's/100 in<sup>2</sup>-day-atm
- **OPV**; Oxygen Permeation Value  
(OTR normalized for a specific film gauge)
  - **Units**; cc-mil/100 in<sup>2</sup>-day-atm

# Permeability Estimates for Polymer Blends

## Polymer A:

LLDPE with Oxygen Permeability Value = 600 cc-mil/ 100 in<sup>2</sup>-day-atm

## Polymer B:

LDPE with Oxygen Permeability Value = 500 cc-mil/ 100 in<sup>2</sup>-day-atm

## Polymer Blend:

= 80% A LLDPE + 20% B LDPE

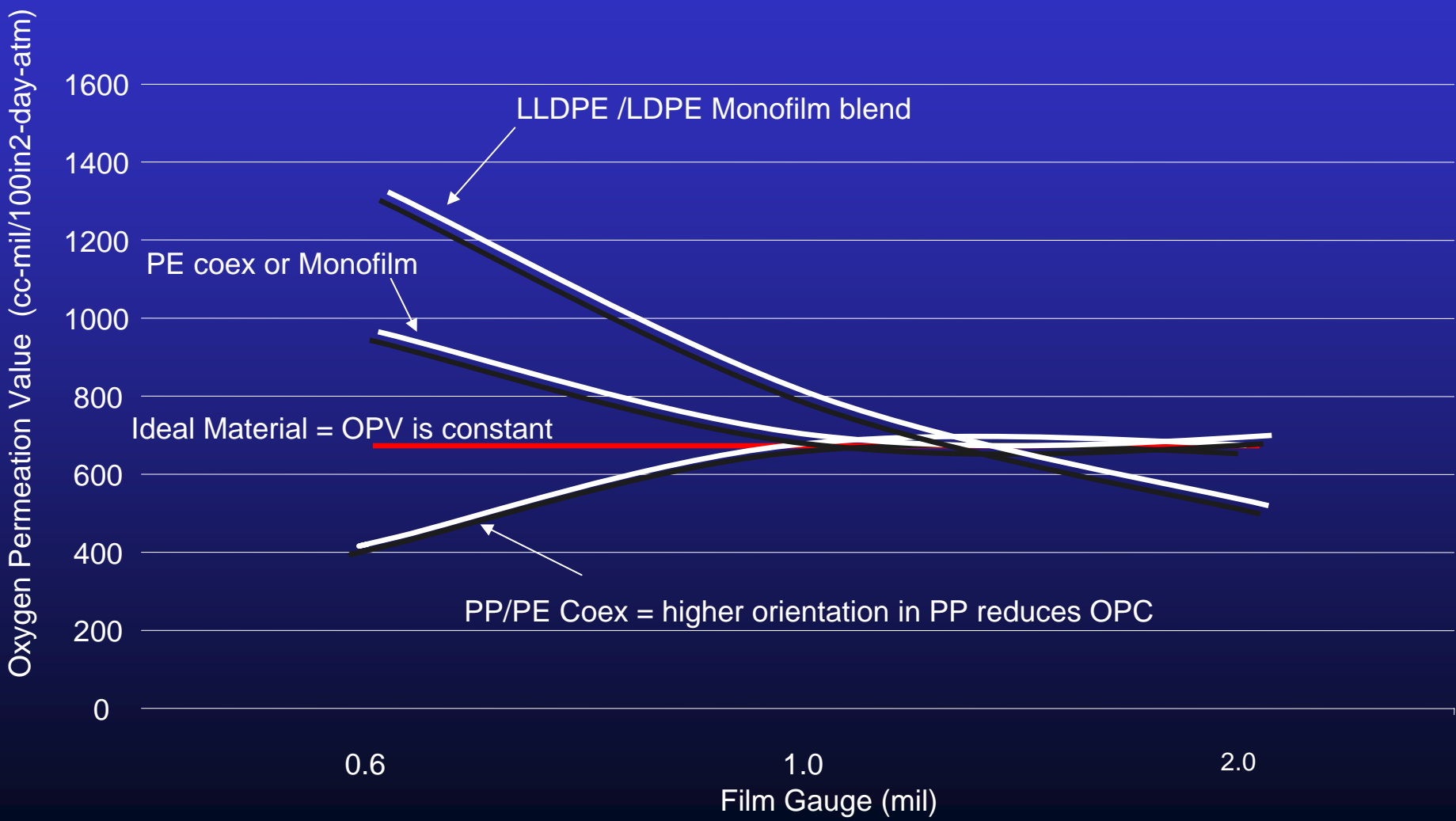
## ESTIMATED OTR FOR 2 MIL FILM:

= [(0.8)\*(600) + (0.2)\*(500)] / 2

= 290 cc's/100 in<sup>2</sup>-day-atm

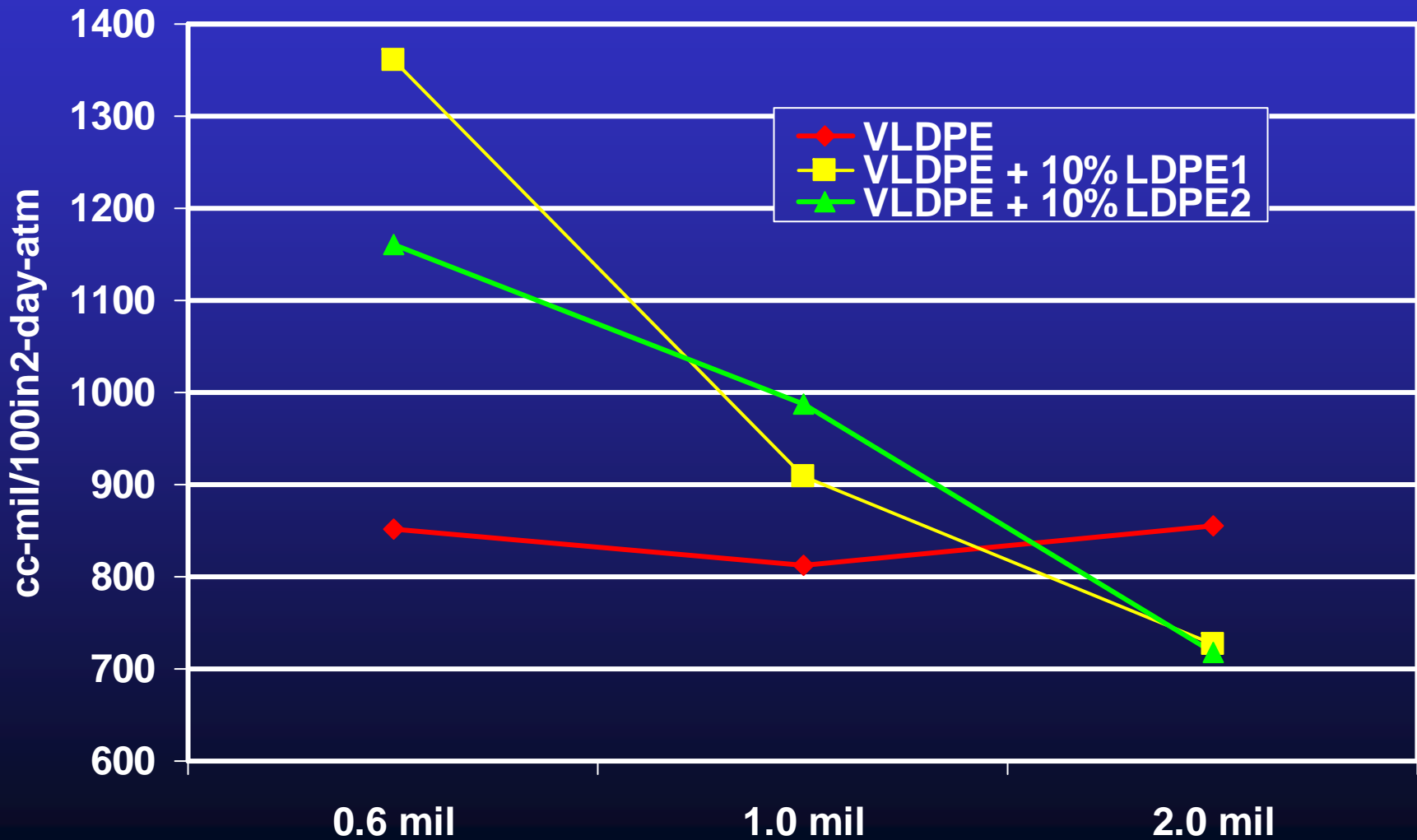
Blend Ratio	Gauge (mil)	OTR Est.	OPV Est.
80/20	2	290	580
80/20	1	580	580
80/20	0.5	1160	580

# OPV Deviations from Linearity in Thin-Gauge Films



# OPV for VLDPE Films and Blends

## Monolayer Blown Films - 100 mil die gap



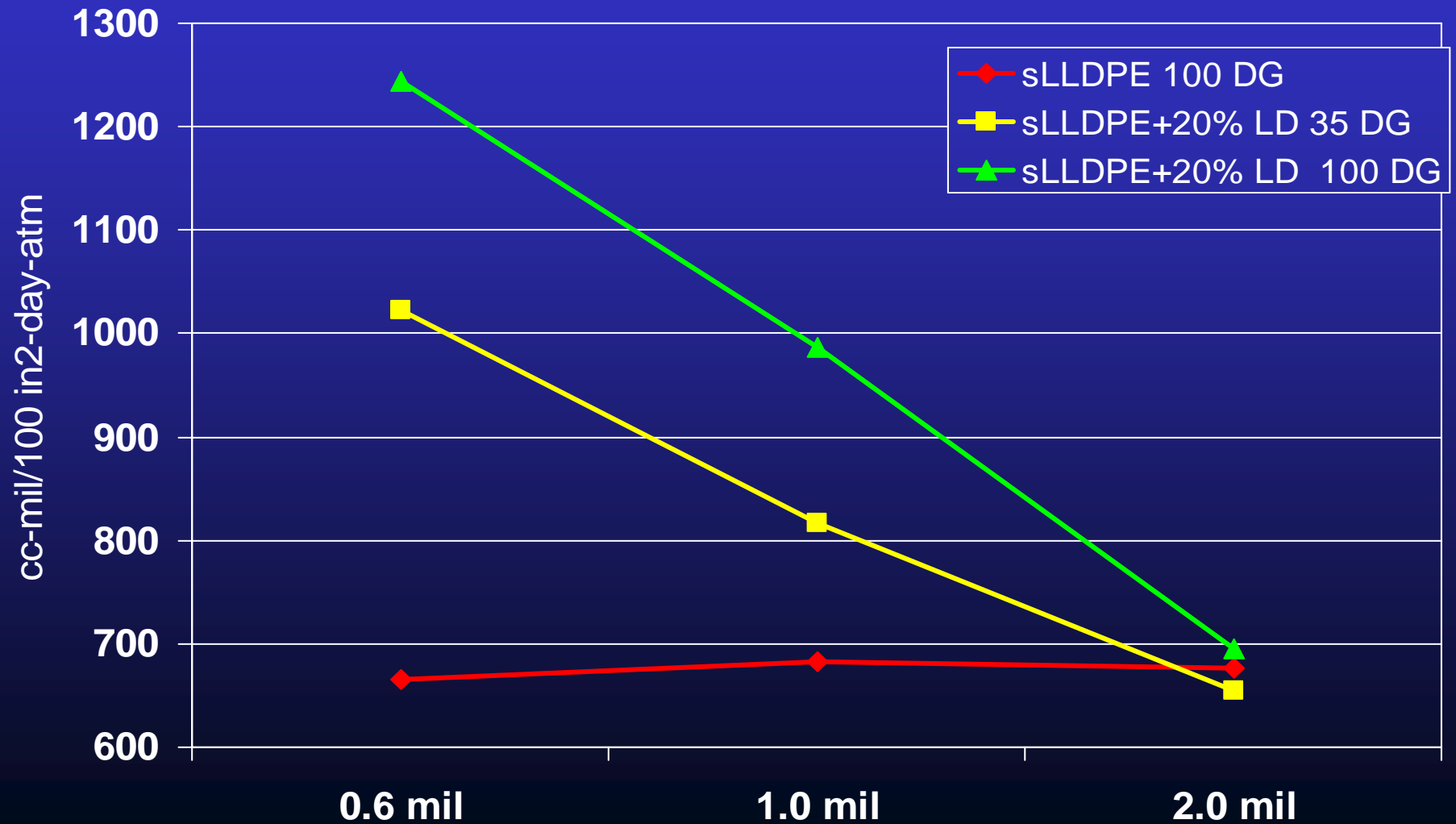
# **% Total Crystallinity vs. OPV for Ziegler-Natta LLDPE Test Films**

<b>LDPE blend Amount</b>	<b>% Crystallinity DSC Second Heat</b>	<b>OTR cc/100 in<sup>2</sup>-day-atm</b>
0	44.7	<b>703</b>
10% LD1	43.7	<b>670</b>
15% LD1	44.2	<b>730</b>
10% LD2	44.3	<b>593</b>
15% LD2	44.3	<b>730</b>

**Observation:** Overall crystallinity does not change significantly in these blends. Crystallinity variations do not account for OPV changes.

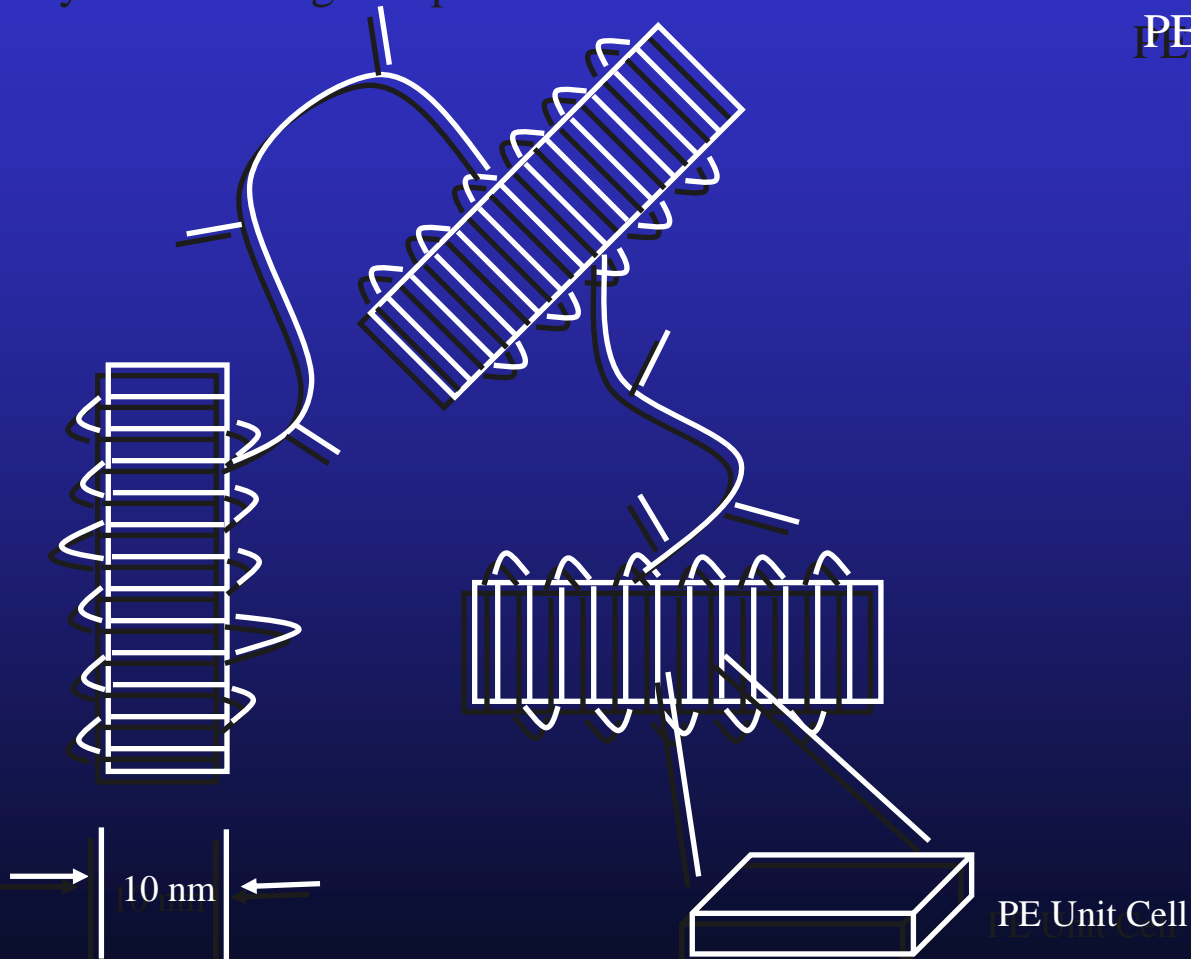
# OPV for sLLDPE Films and Blends

## 100 and 35 mil die gaps

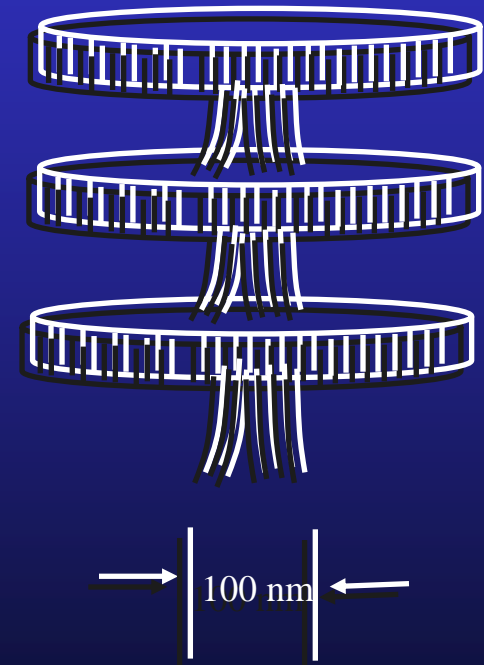


# PE Semi Crystalline Morphology

PE Crystal Weaving like pattern of folded chains



PE Macro "Shish-Kebab" Chains



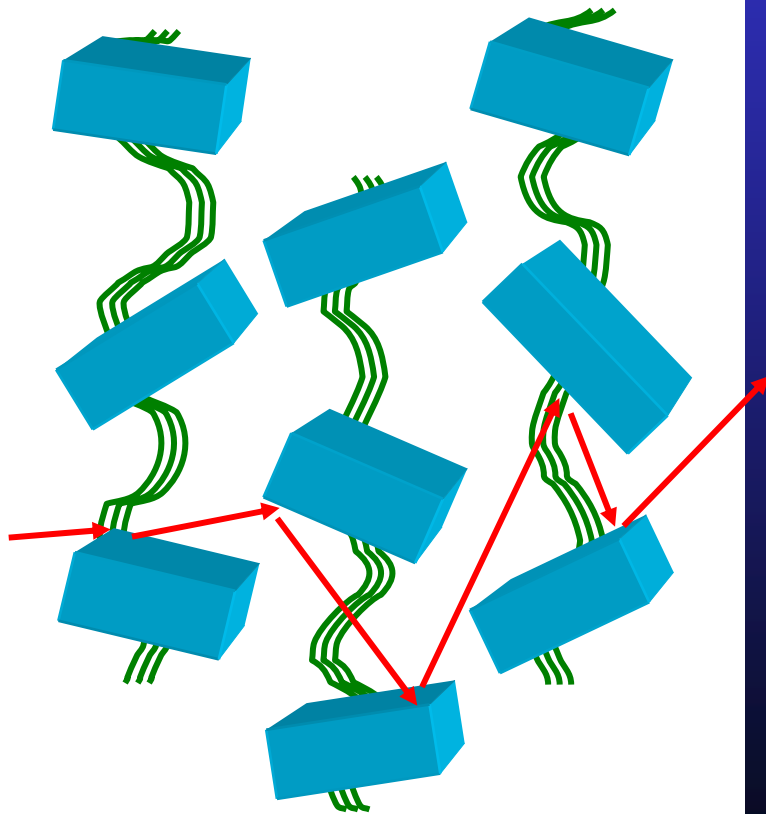
From: D.J. Williams, *Polymer Science and Engineering*, Prentice Hall Inc., Englewood Cliffs, NJ 1971

T. A. Kavassalis' and P. R. Sundararajan *Macromolecules* Vol. 26, No. 16 4144

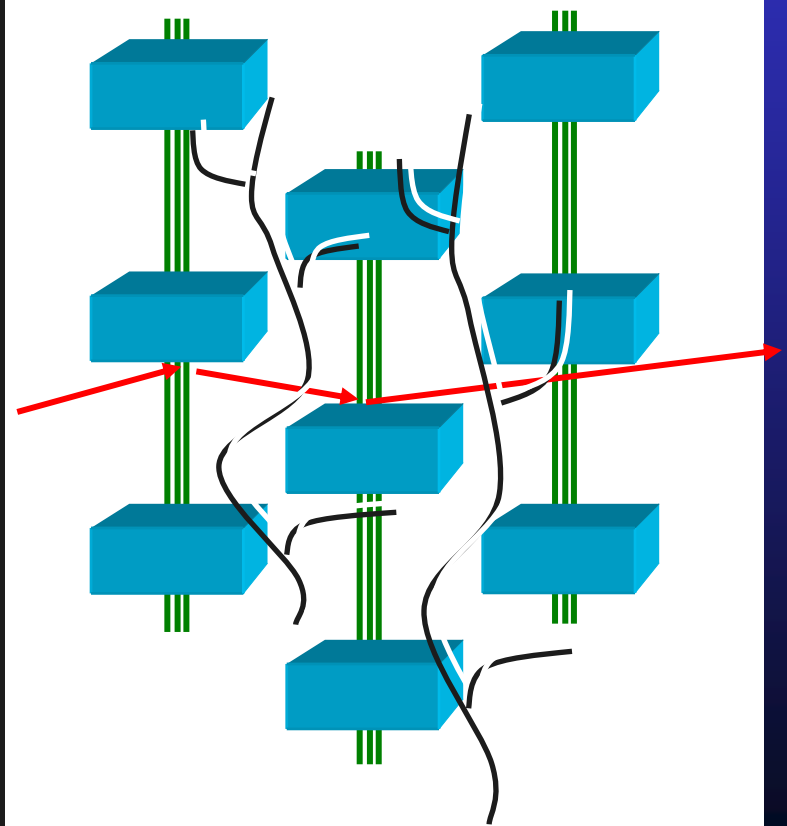


# Shish-Kabob Permeability Model for Low Crystallinity Blends

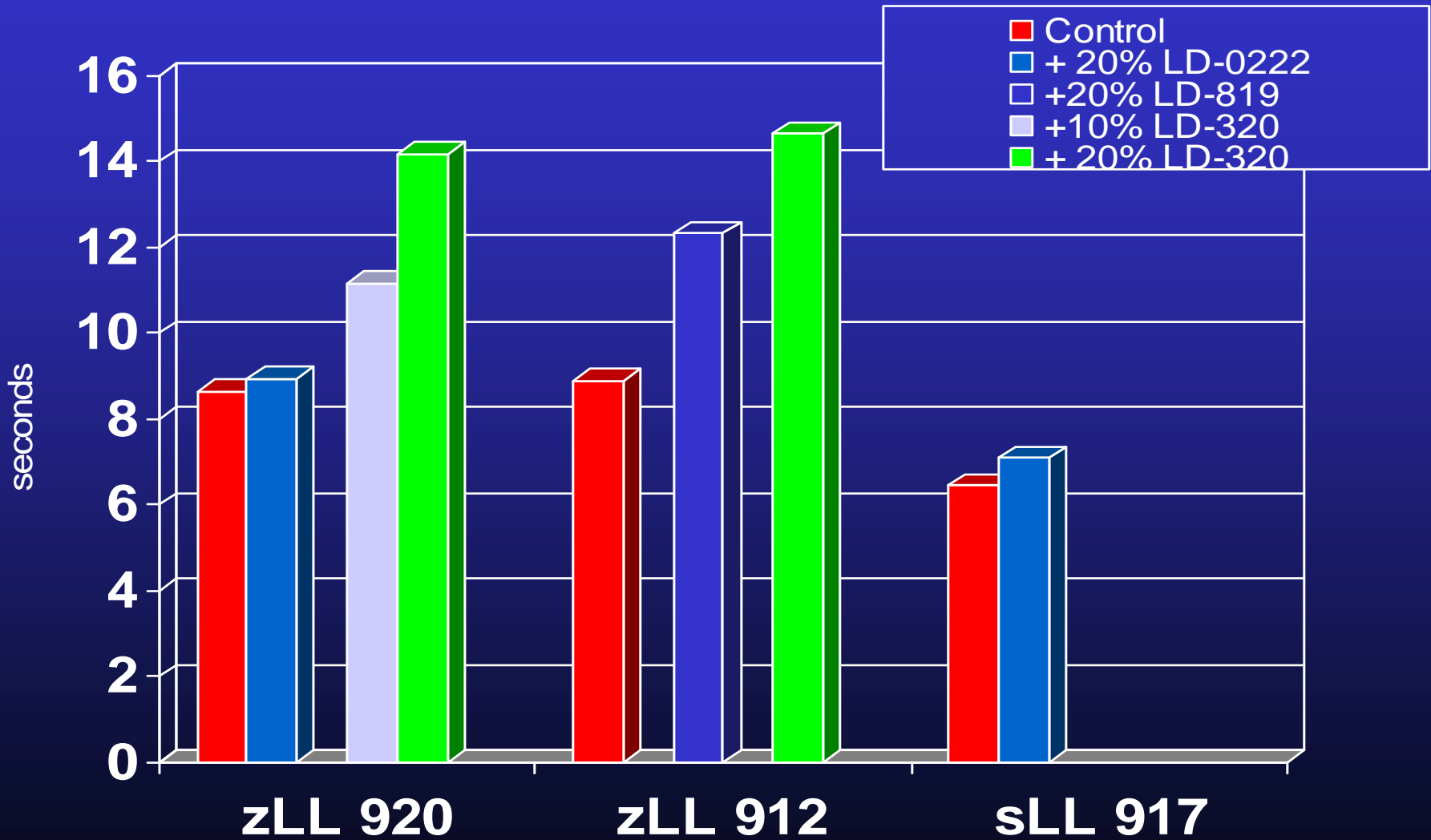
Low orientation



High orientation



# Melt Relaxation Times



# **Theory: Increased Orientation Leads to Increased Permeability of Blends**

- Draw induced orientation forces the crystalline platelets to align parallel to permeant flow.
- Low levels of certain LDPE blended into octene LLDPE facilitates and/or maintains parallel crystalline plate alignment.

# Objectives

- Evaluate differences between polymer morphology and crystal alignment in octene LLDPE / LDPE blends and relate differences to film OPV.
- Identify film processing methods and LDPE blends that maximize permeability in films made with octene LLDPE.

# Octene LLDPE Base Resins

Material	Catalyst	Melt Index (g/10 mins)	Density (g/cc)	Designation
SCLAIR FP120-A	Zeigler-Natta	1.0	0.920	zLL-920
SCLAIR FP112-A	Zeigler-Natta	1.0	0.912	zLL-912
SURPASS FPs117-A	Single Site	1.0	0.918	sLL-918

# Tubular LDPE Blend Resins

Material	Melt Index (g/10 mins)	Density (g/cc)	Designation
NOVAPOL LF-Y320-C	0.25	0.920	LD-320
NOVAPOL LF-Y819-A	0.8	0.919	LD-819
NOVAPOL LF-0222-A	2.0	0.922	LD-0222

# Film Production

- **Commercial-Scale Blown Film Line**

- 8 inch diameter die with internal bubble cooling
- 3.5 inch 30:1 L/D smooth bore extruder
- High Performance Dual Lip Air Ring

- **Film Production Variables**

- Die gaps: 100 and 50 mil
- Film gauges: 2.0, 1.0, 0.6 mil

- **Film Production Constants**

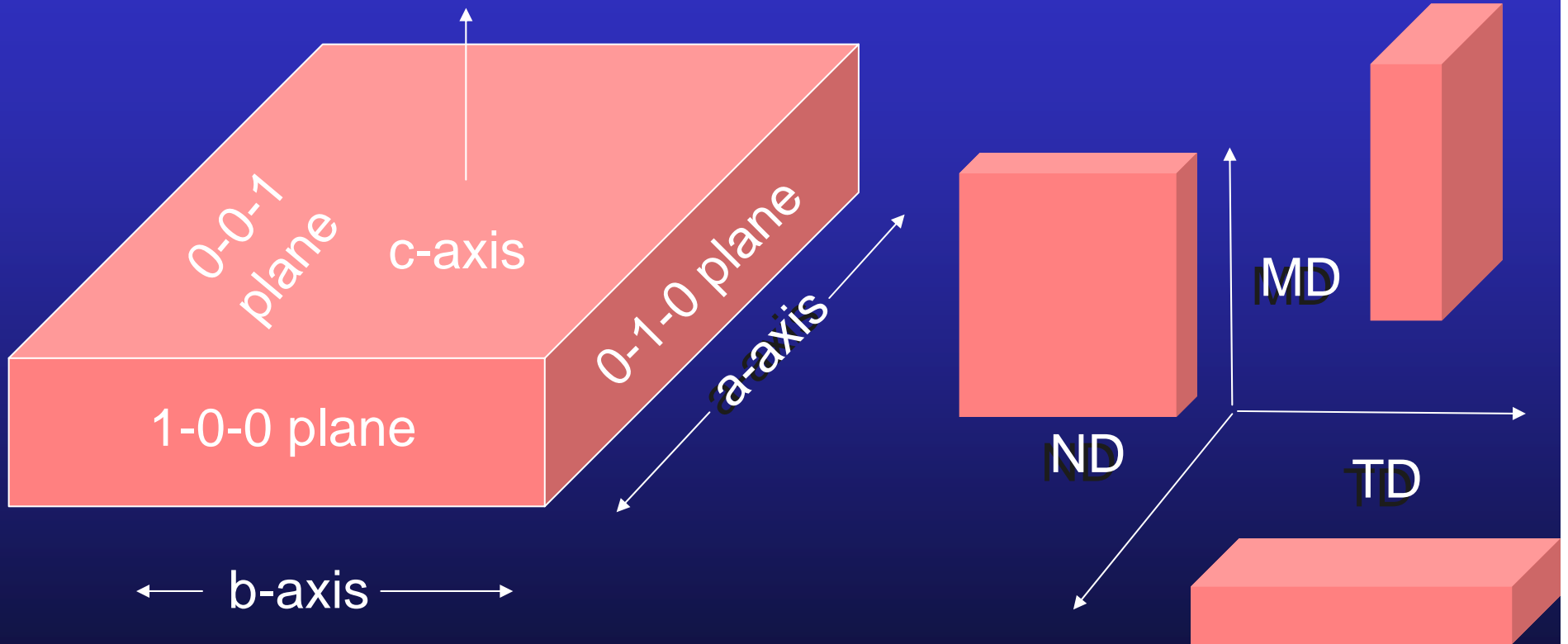
- **Blow Up Ratio 2.5 : 1**
- **Output rate: 250 pph.**
- **Target Melt Temps 215 – 227°C**
- **Frost Line Height = 10 – 14 inches**

# Analytical Tests

- **Oxygen Permeability**
  - Mocon OXTRAN® 23C, 50% RH, > 48 hr aging.
- **Orientation**
  - FTIR: Dichroic Ratio
  - X-Ray Diffraction
    - RFPO: *Relative Film Planar Orientation*
    - GADDS: **General Area Detector Diffraction System**



# PE Unit Cell Axes and Film Directions

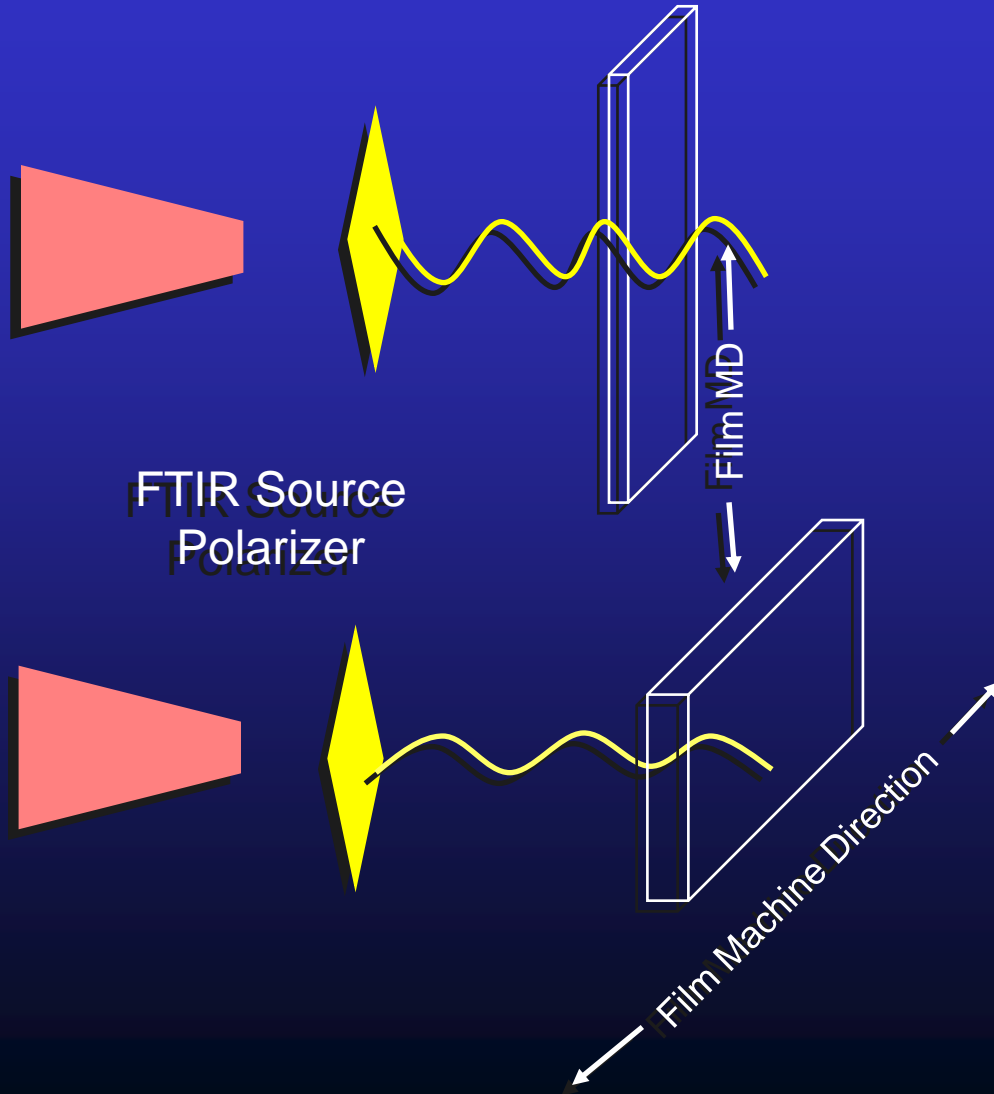


$a - 7.41 \text{ \AA}$     $b - 4.92 \text{ \AA}$     $c - 2.53 \text{ \AA}$

Orthorhombic unit cell  $a \neq b \neq c$

# Dichroic Ratio by FTIR

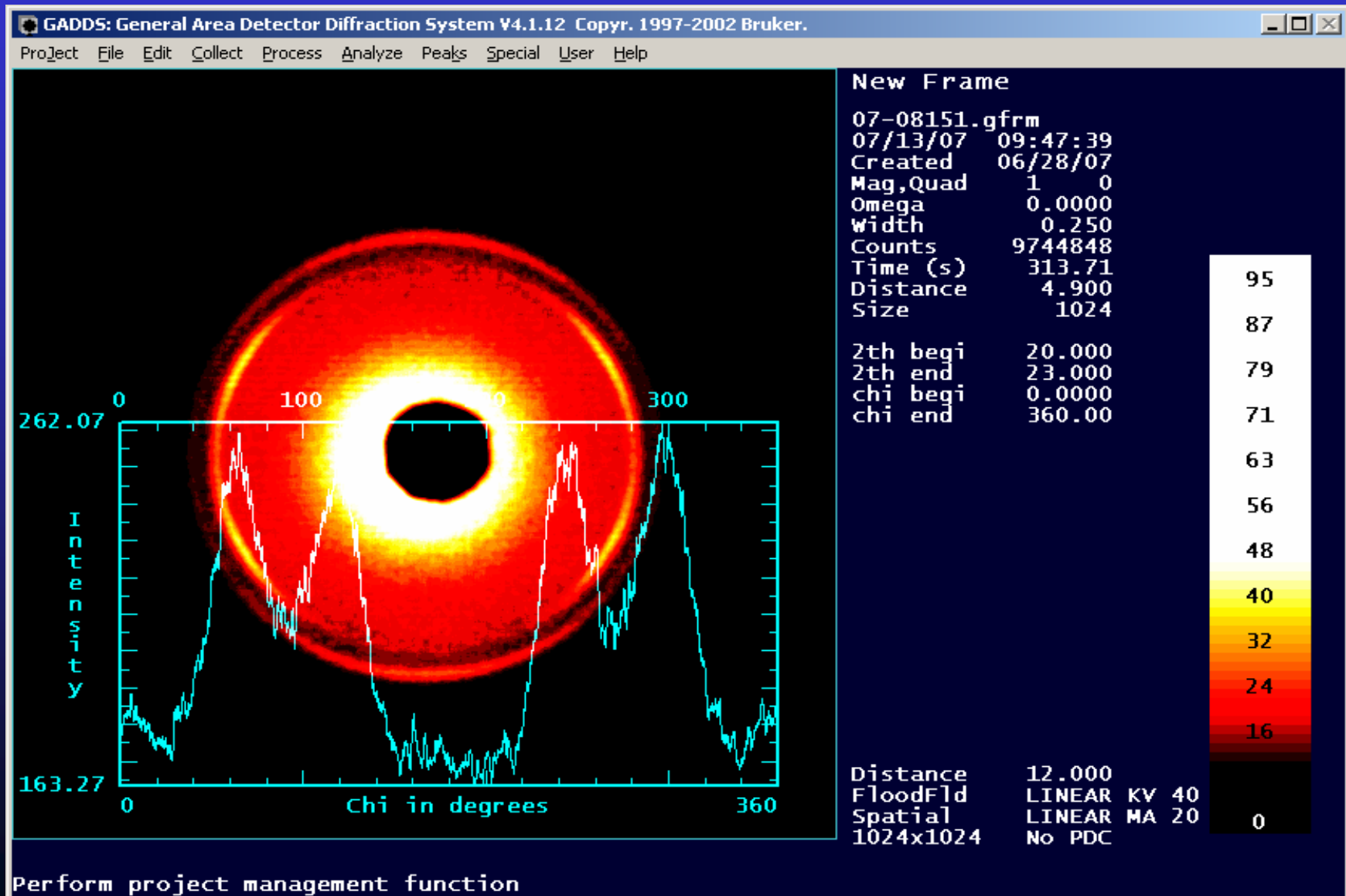
$$D = A_{\parallel} / A_{\perp}$$



730 cm<sup>-1</sup> Absorbance relates to crystalline and amorphous orientation along a-axis relative to MD

720 cm<sup>-1</sup> Absorbance relates to crystalline orientation of b-axis relative to TD

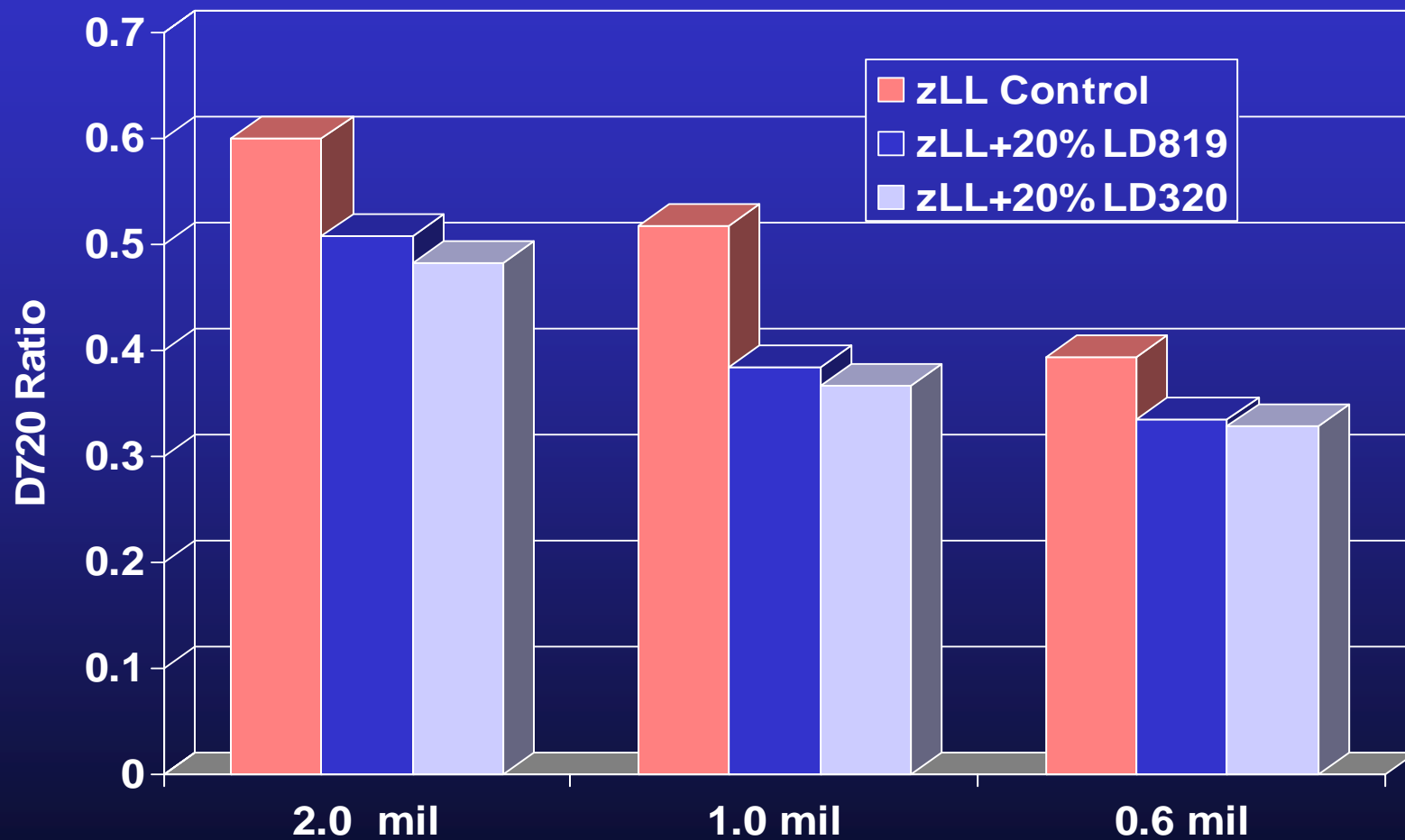
# X-Ray Diffraction



# OTR and OPV for Selected Films and Blends

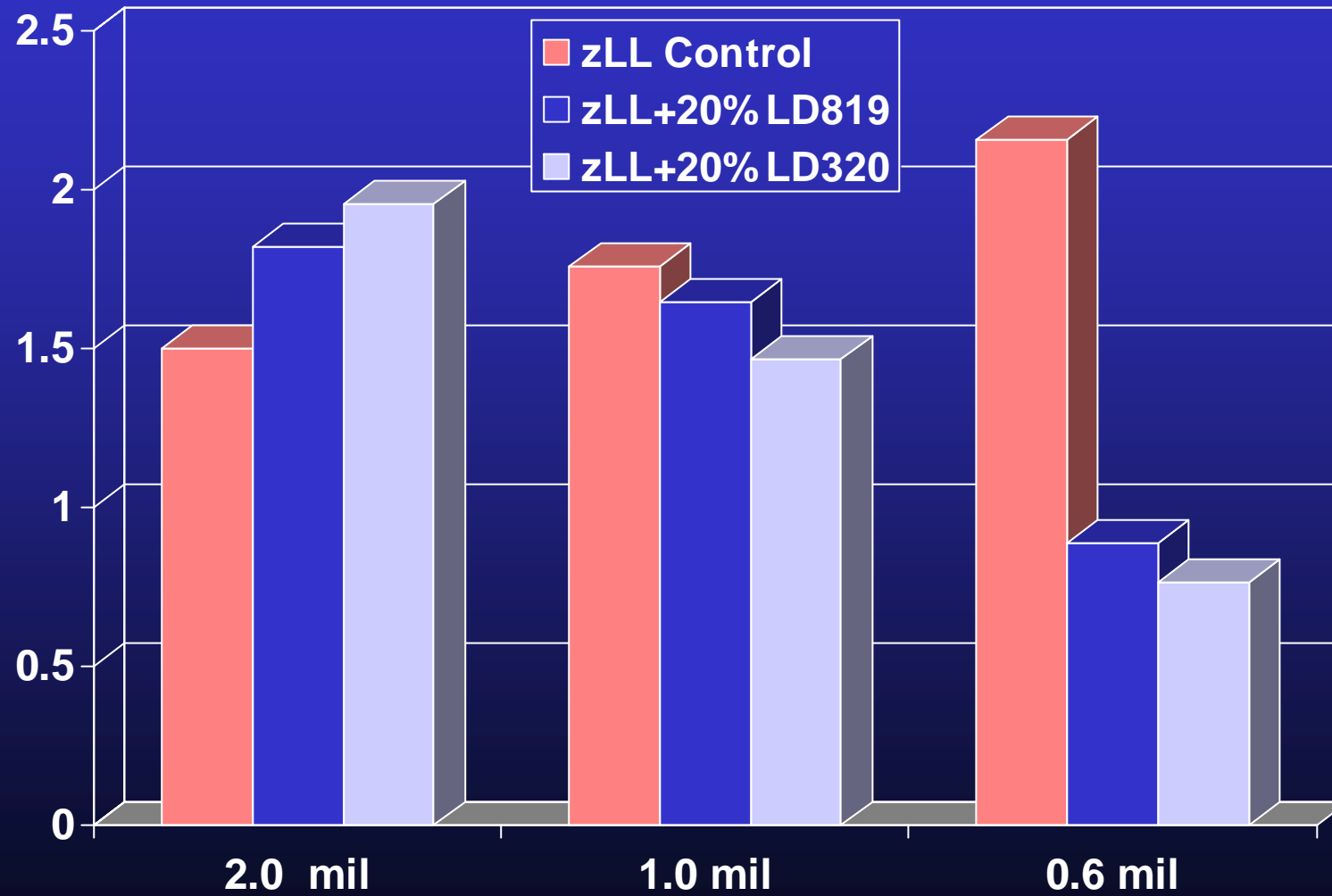
Film	Gauge (mil)	OTR (cc/100in-day-atm)	OPV (cc-mil/100in-day-atm)
sLL 917 Control	2.0	295	619
sLL 917 Control	1.0	626	626
sLL 917 Control	0.6	890	623
sLL 917+ 20% LD 0222	2.0	297	594
sLL 917+ 20% LD 0222	1	673	673
sLL 917+ 20% LD 0222	0.6	1468	881
zLL 912 Control	2.0	453	907
+ zLL 912 Control	1	799	799
zLL 912 Control	0.6	1918	1151

# zLL-912 Dichroic Ratios $720\text{ cm}^{-1}$



Decreasing D720: B-axis (crystal growth direction) is less oriented toward film MD

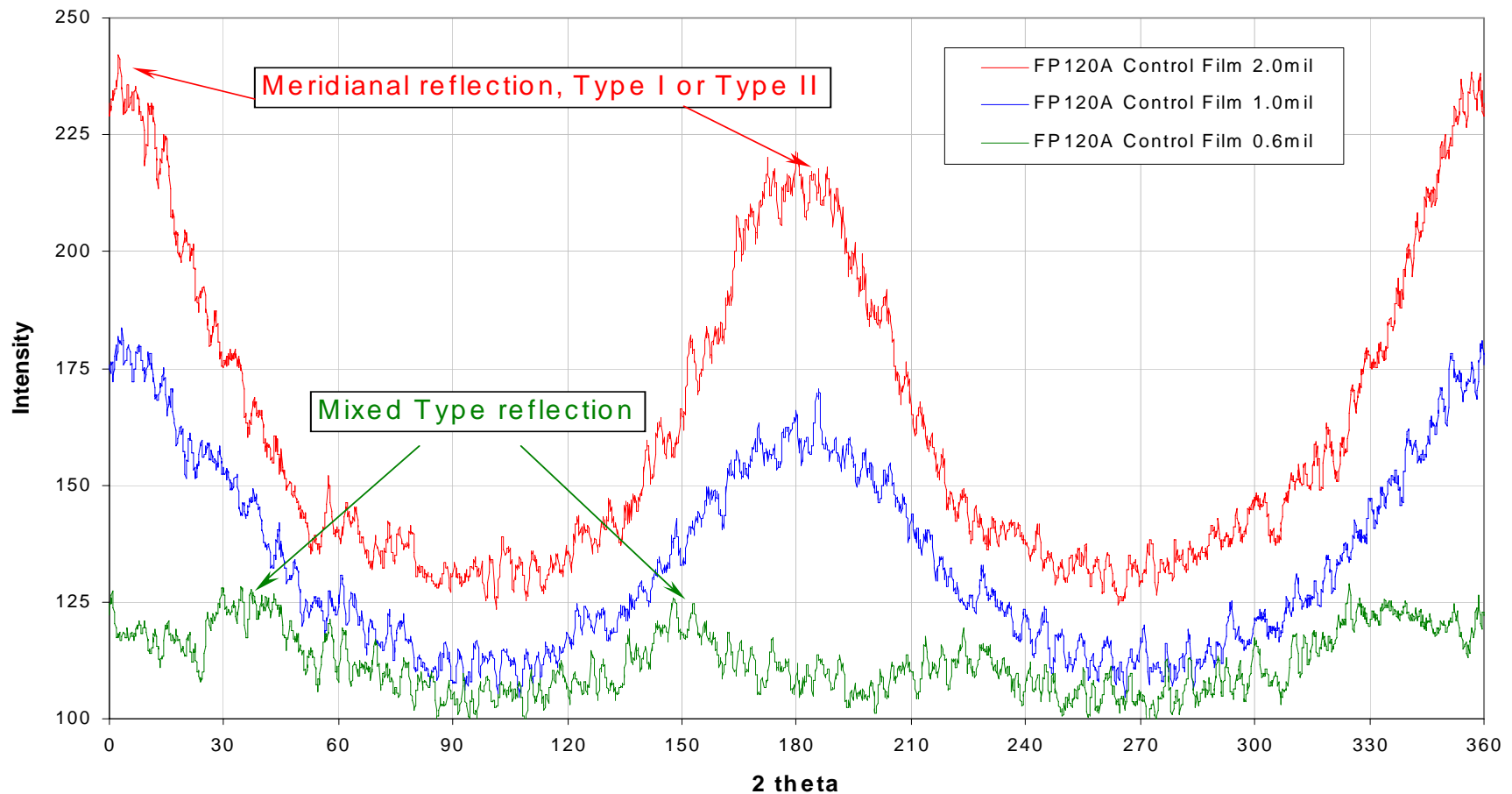
# zLL-912 Dichroic Ratios - 730



# X-Ray Diffraction Patterns

## PE Crystal Unit Cell: A-Axis

RFPO 200 (a-axis orientation)



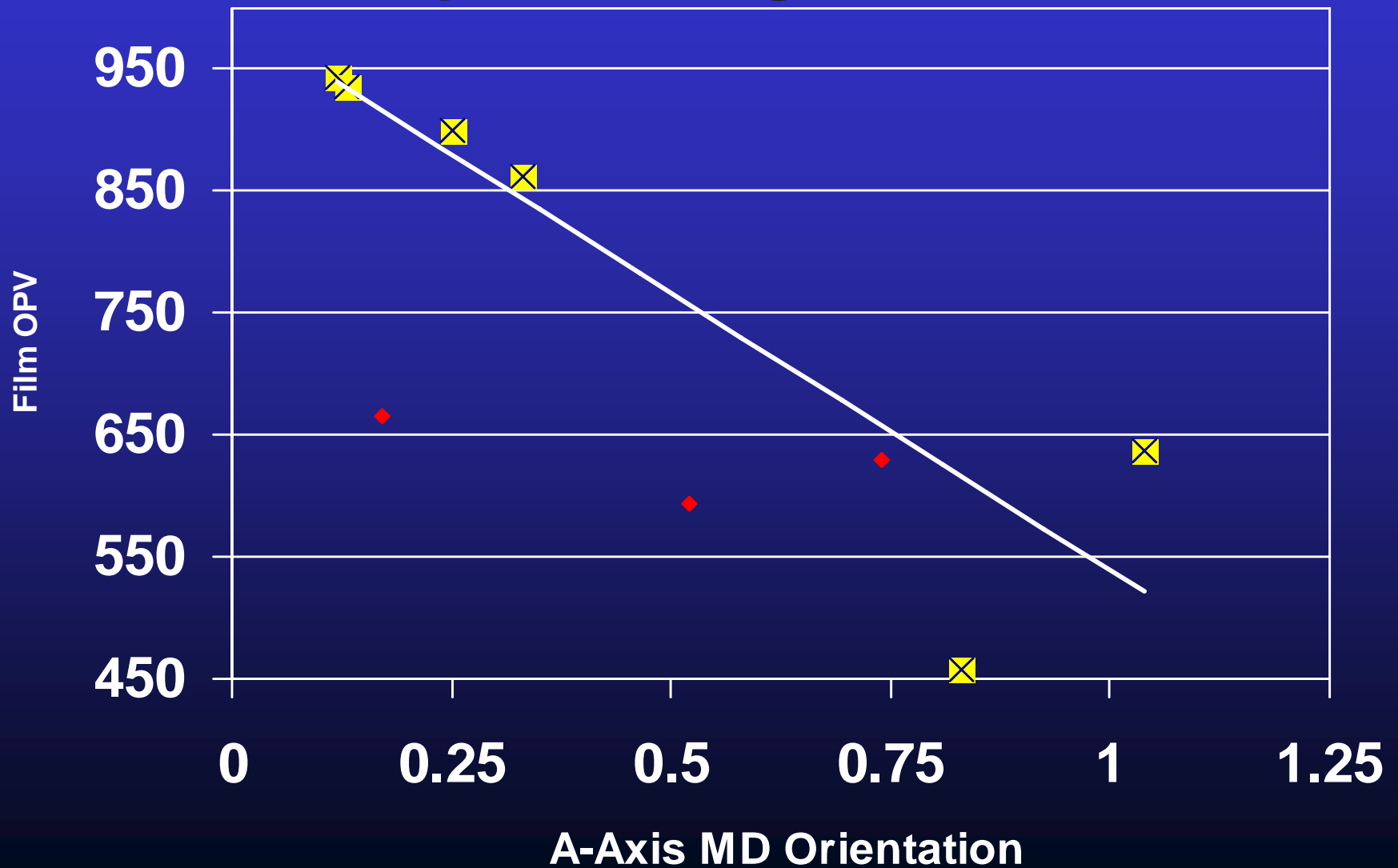
# RFPO 200 OPV of Film Blends

## RFPO 200: A-Axis Alignment with Film MD

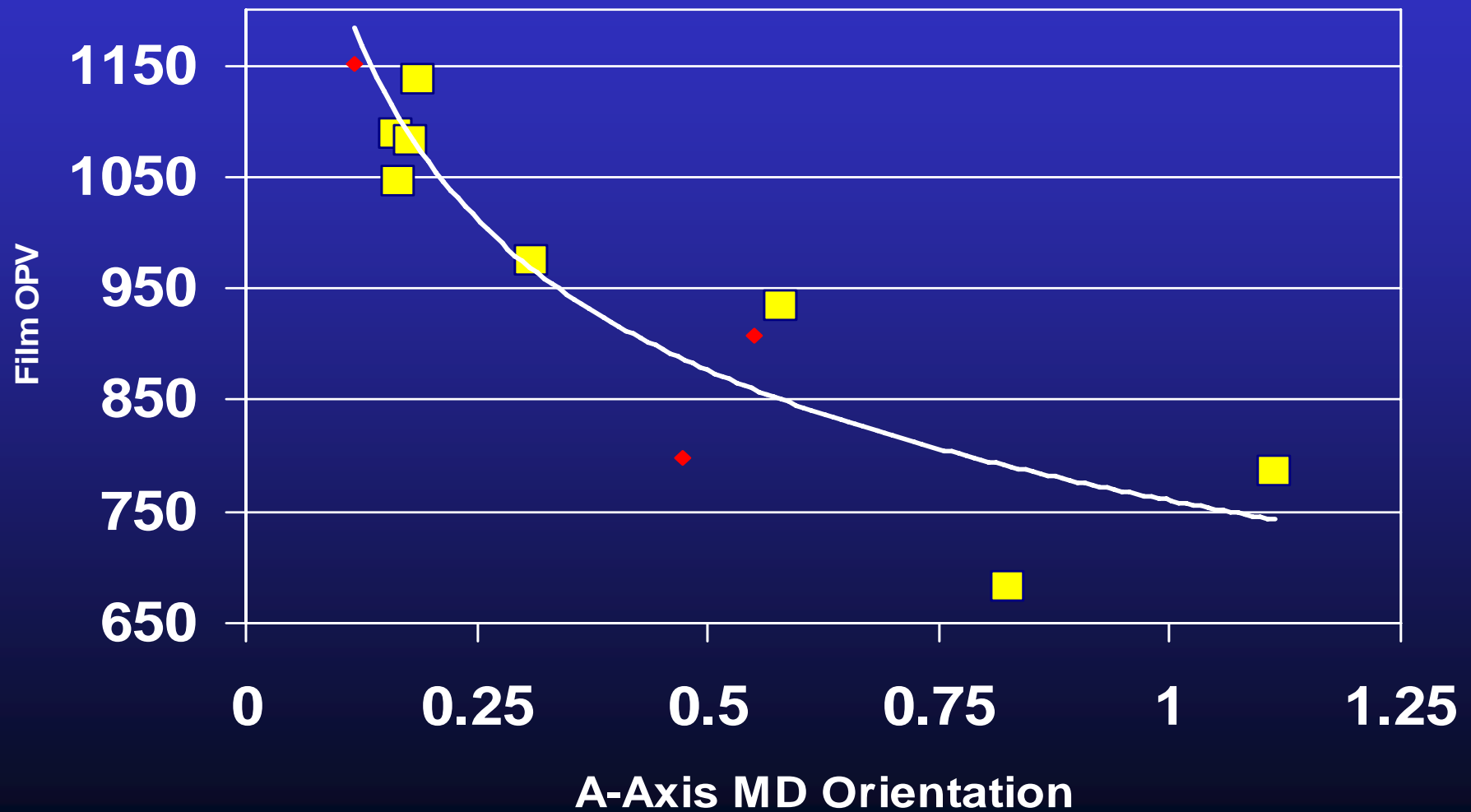
	Gauge	RFPO 200	OPV
zLL 920 control	2	0.74	630
	1	0.52	594
	0.6	0.17	666
zLL 920 +20% LD320	2	1.04	638
	1	0.25	900
	0.6	0.12	943
zLL 920 +10% LD320	2	0.83	457
	0.6	0.13	936



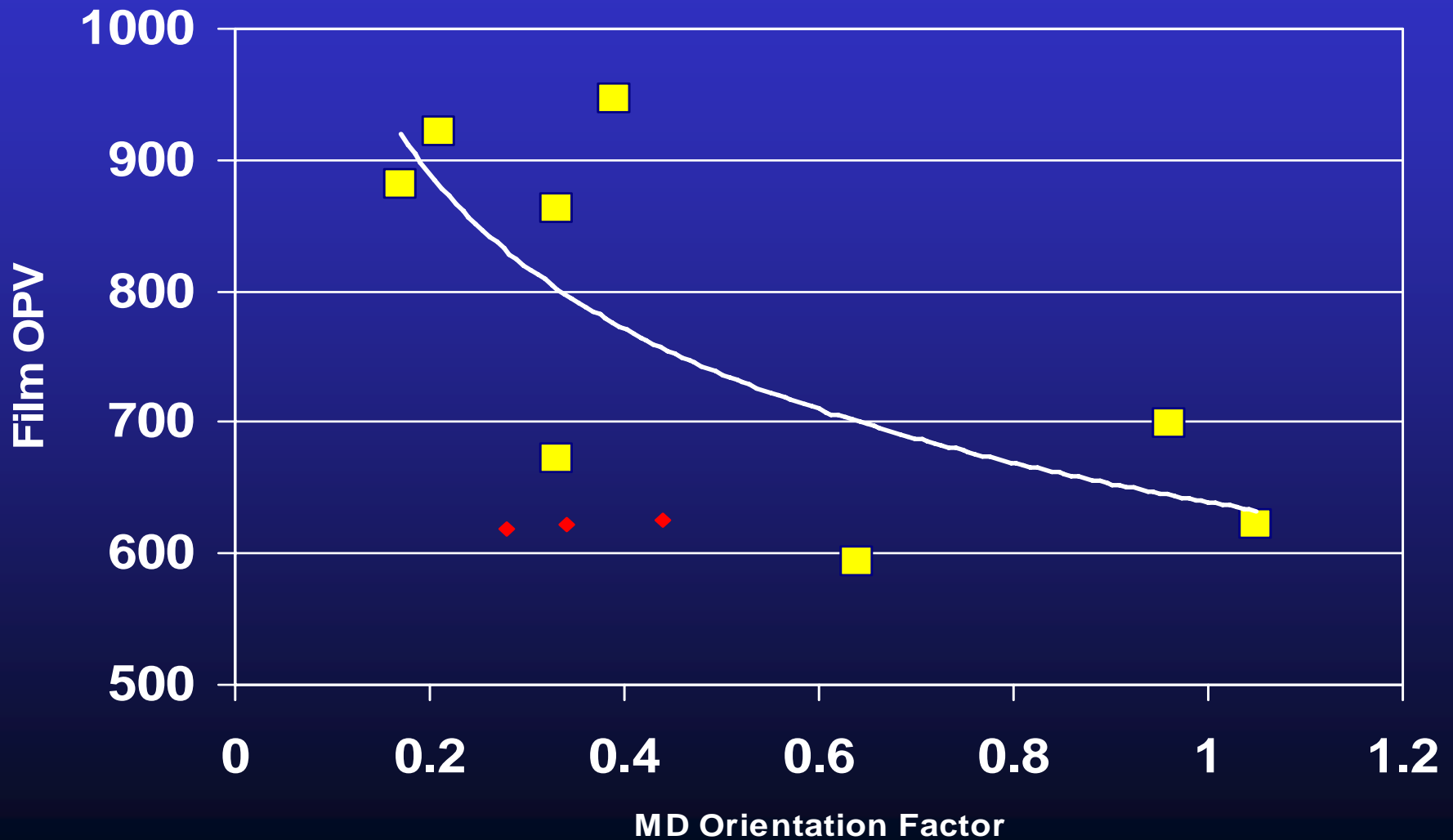
# zLL 920 A-Axis MD Alignment vs. Film OPV



# zLL 912 A-Axis MD Orientation vs. Film OPV



# sLL 917 A-Axis MD Alignment vs. Film OPV



# Conclusions

- The final orientation of crystalline platelets in octene LLDPE films is affected by draw rates and the presence of LDPE blend resins.
- The OPV of films is improved when crystalline platelets are aligned parallel to the film TD and ND.
- Film manufactures can increase OPV of octene LLDPE films by:
  - Maximizing MD draw ratios (larger die gaps)
  - Blending in low levels of LDPE with similar viscosities to LLDPE

# Acknowledgements

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- **Co Authors: Joo Teh, Kam Ho, Norm Aubee, Jamie Marler**

# Thank you

PRESENTED BY:

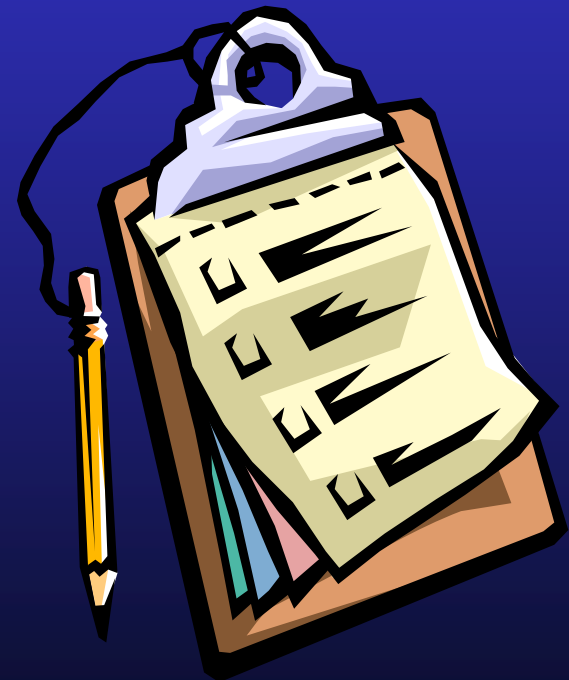
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