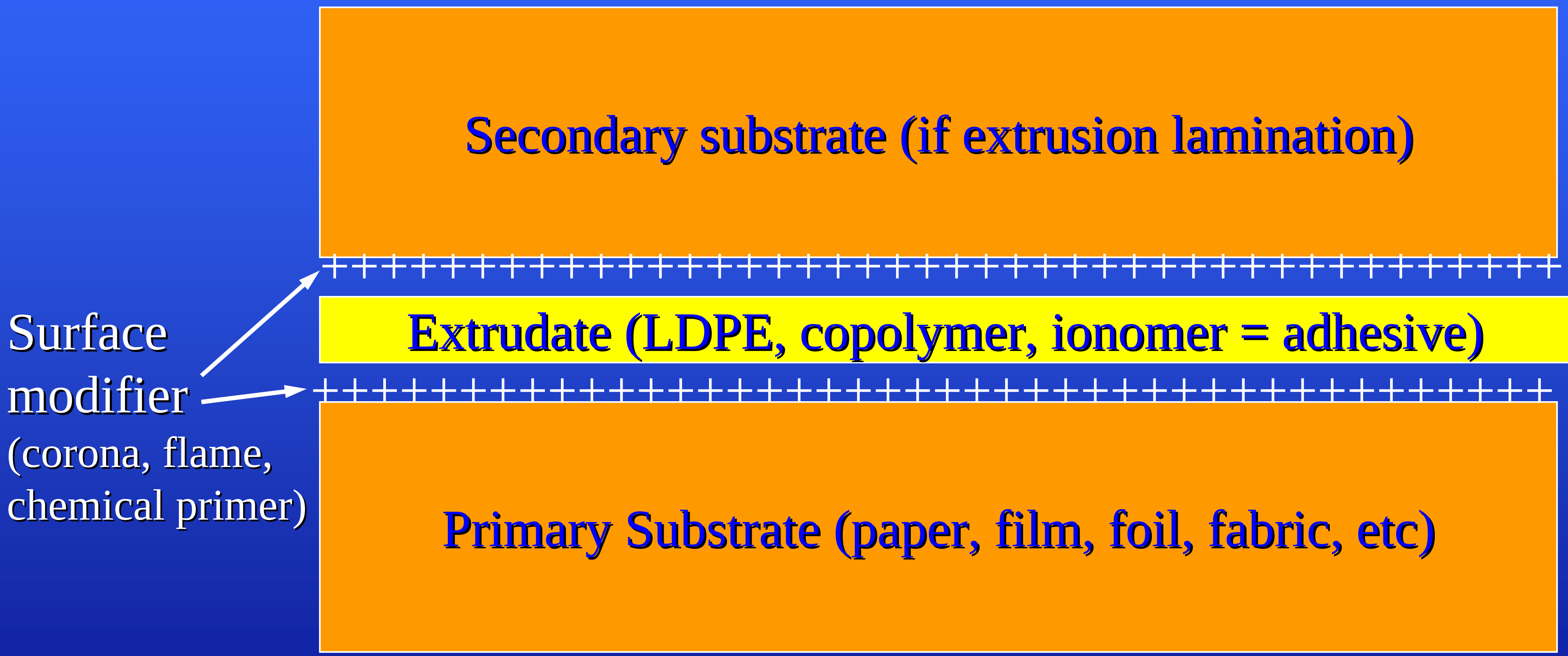
Adhesion in Extrusion Coating & Laminating - the Importance of Machine Variables

Bruce Foster
Mica Corporation

Outline

- Identify key factors that affect adhesion
- Examine Machine Variables that affect these factors

The Extrusion Coating System:



Two Key Components for Adhesion

1. ***Intimate contact*** between adhesive & substrate, which permits:

2. ***Chemical bonding*** between adhesive & substrate

Factors Influencing Intimate Contact

- Adhesive Thickness (thicker = more easily deformed, longer solidification time)
- Adhesive mobility/ deformability (viscosity, modulus, solidification temp., wet-out)
- Substrate morphology (rough surface = less intimate contact)
- Substrate wettability (poor wetting = less intimate contact)

Machine Factors That Can Improve Intimate Contact

- Extrudate thickness
- Nip conditions (pressure & length)
- Melt temperature

Factors Influencing Chemical Bonding

- Degree of intimate contact
- Substrate surface chemistry
- Adhesive surface chemistry
- Adhesive relaxation phenomena
- Migratory chemicals (in adhesive or substrate)
- Environmental stresses

Machine Factors That Can Improve Chemical Bonding

- Substrate treatment conditions (corona or flame power level, or primer application variables)
- Choice of extrudate type
- Extrudate thickness
- Air-gap distance
- Melt temperature of extrudate
- Extruder back-pressure
- Ozone assist

Intimate contact

+

Chemical bonding

=

Durable Adhesion

Now, Examine Each Machine Factor in More Detail…

Machine Factors That Can Improve Intimate Contact

1. Extrudate thickness
2. Nip conditions (pressure & length)
3. Melt temperature

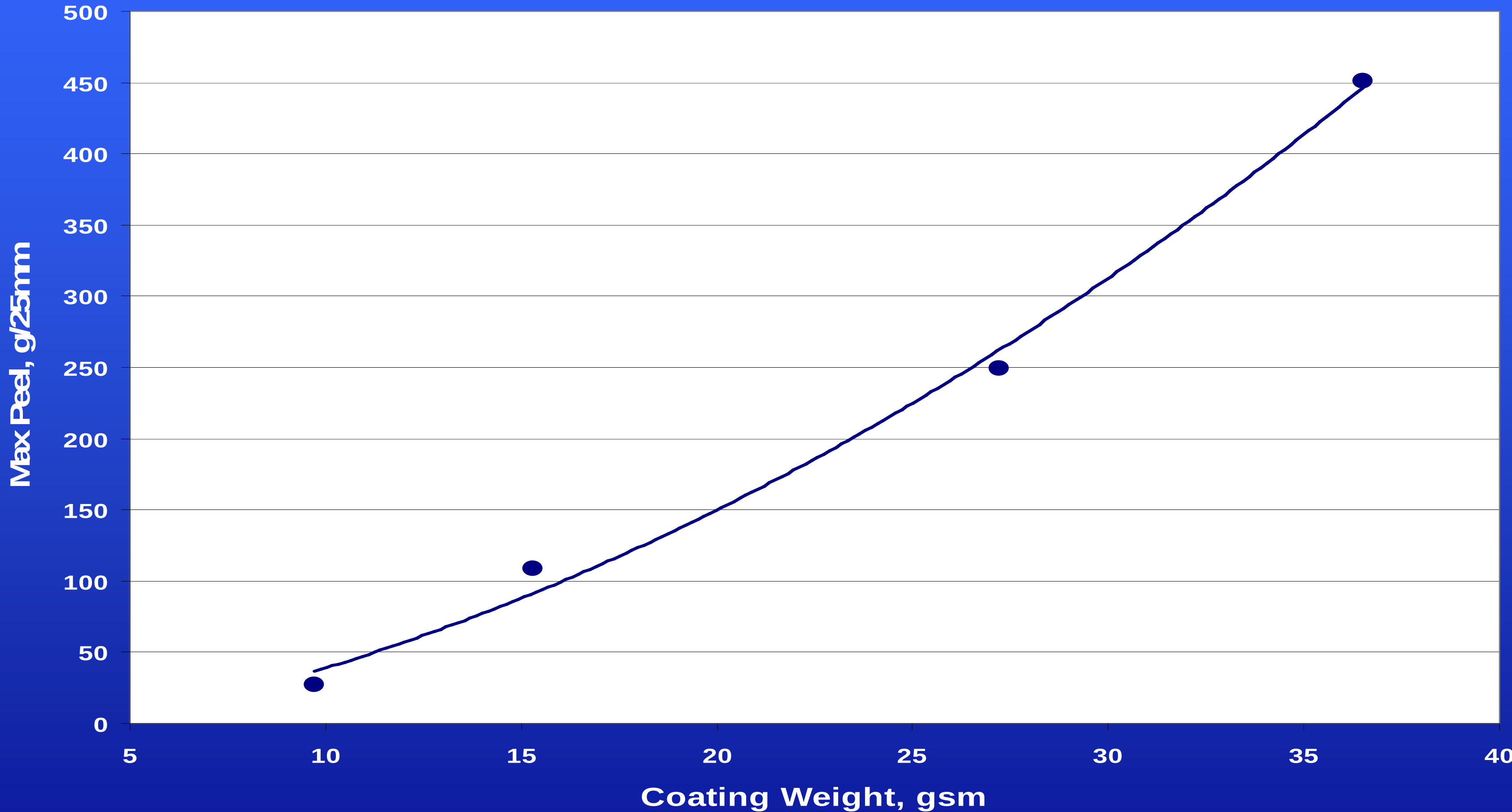
Extrudate Thickness

Single most important variable in achieving good adhesion*

Thicker is almost always better

*(Assumption: Other extrusion conditions are in "normal" ranges)

Fig. 1 - Typical Adhesion Response to Coating Thickness

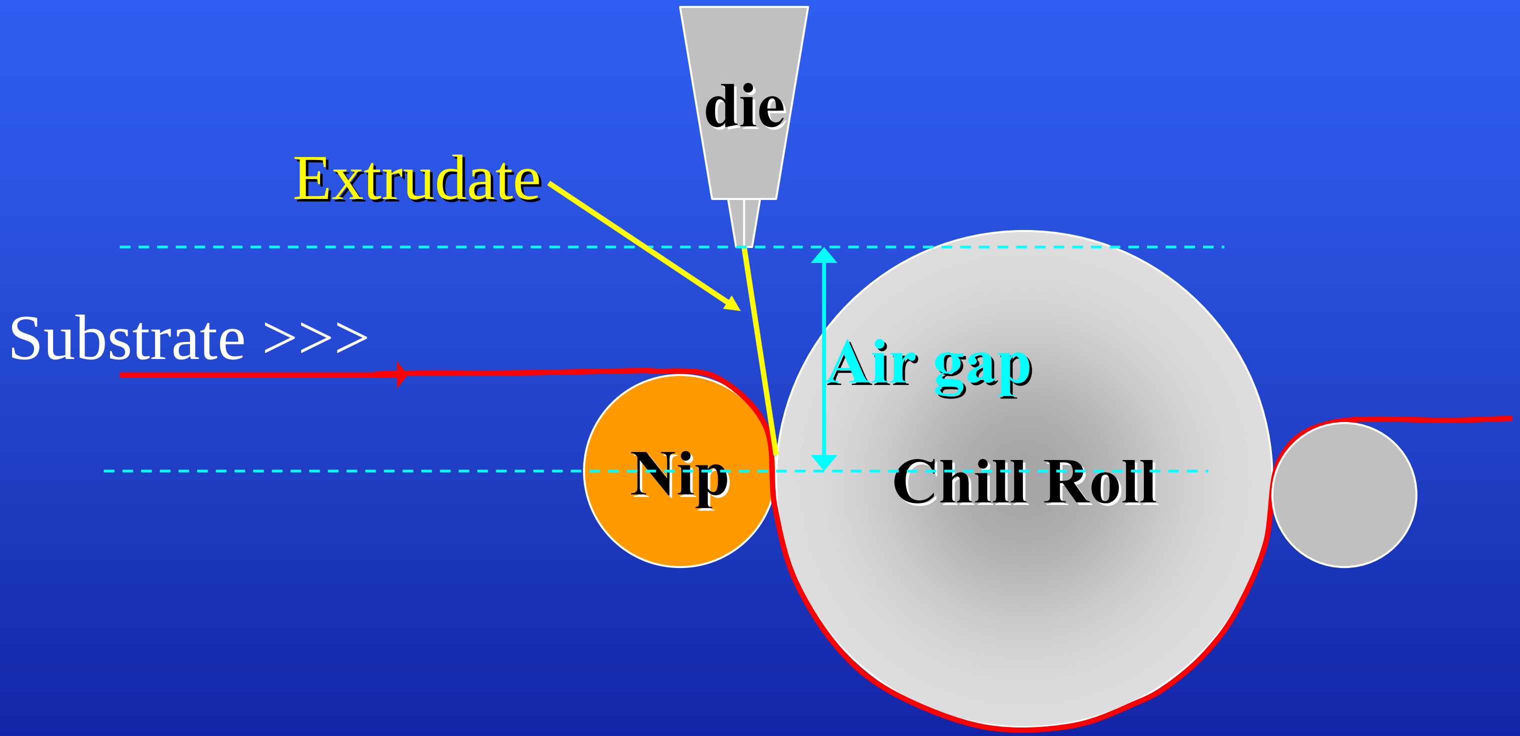


Ref. 1 - Foster, Bruce ; Baker, Mike, Effect of Extrusion Parameters on Adhesion of Polyester – A Line Study, 2001 TAPPI Polymers, Laminations, Coatings Conference Proceedings, TAPPI PRESS, Atlanta, paper 17-2

Why Thickness is Important

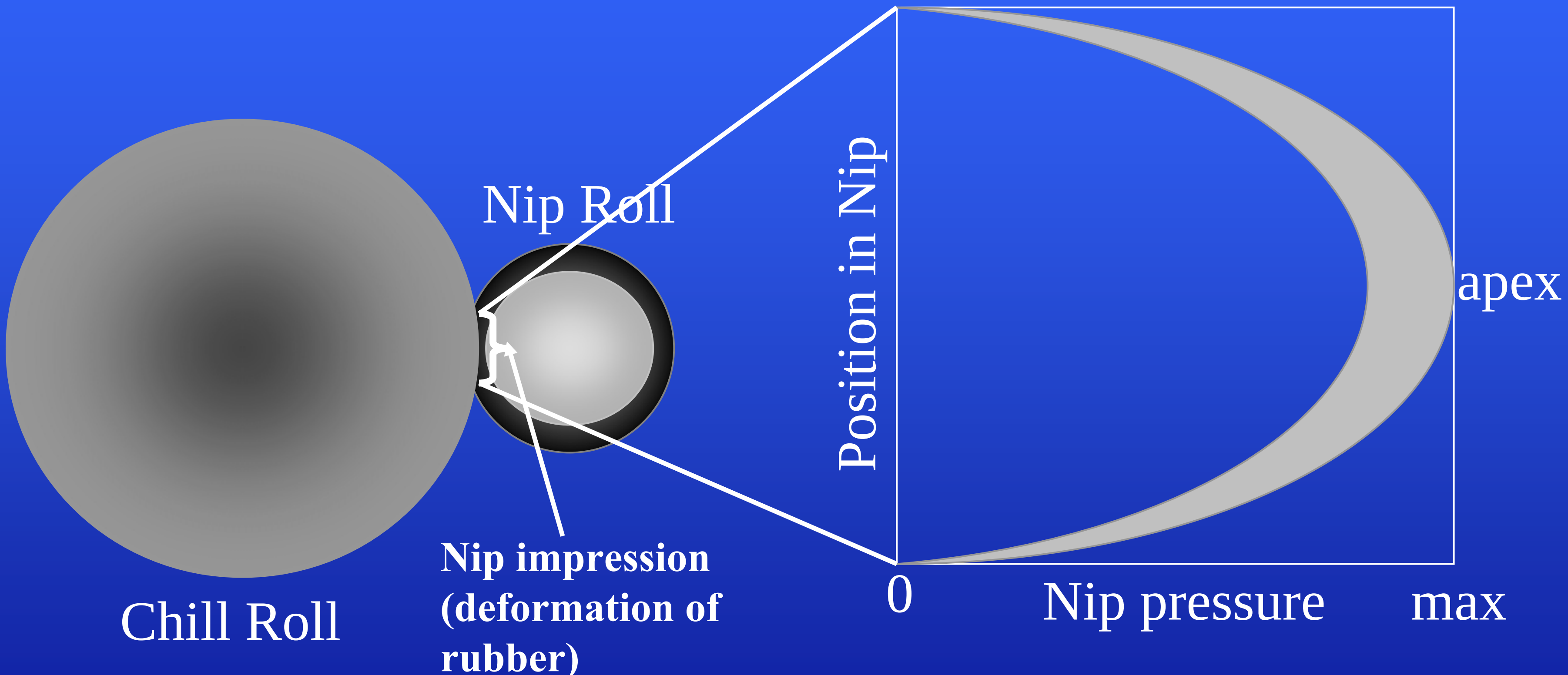
- Transfer of thermal mass to insure intimate contact
- Thermal mass aids in surface oxidation of the extrudate
- Thicker = more compressible, important for non-smooth substrates

Why Thickness is Important



Objective: Transfer thermal energy from die to nip

Fig. 2 - Transfer of Thermal Energy



$$\text{Time in Nip, msec} = \frac{\text{60 x Nip length, mm}}{\text{Line speed, mpm}}$$

Transfer of Thermal Energy

Solidification Model Gives Insight….

1-dimensional unsteady-state heat conduction equation:*

Model predicts solidification of polymer in the nip

*Ref. 2 - Trouilhet, Yves; Morris, Barry A, Prediction of Temperature Profiles Across Coating and Substrate in the Nip, 1999 TAPPI Polymers, Laminations, Coatings Conference Proceedings, TAPPI PRESS, Atlanta, p. 457.

Fig. 3 – Modeling Polymer Solidification Time vs. Thickness

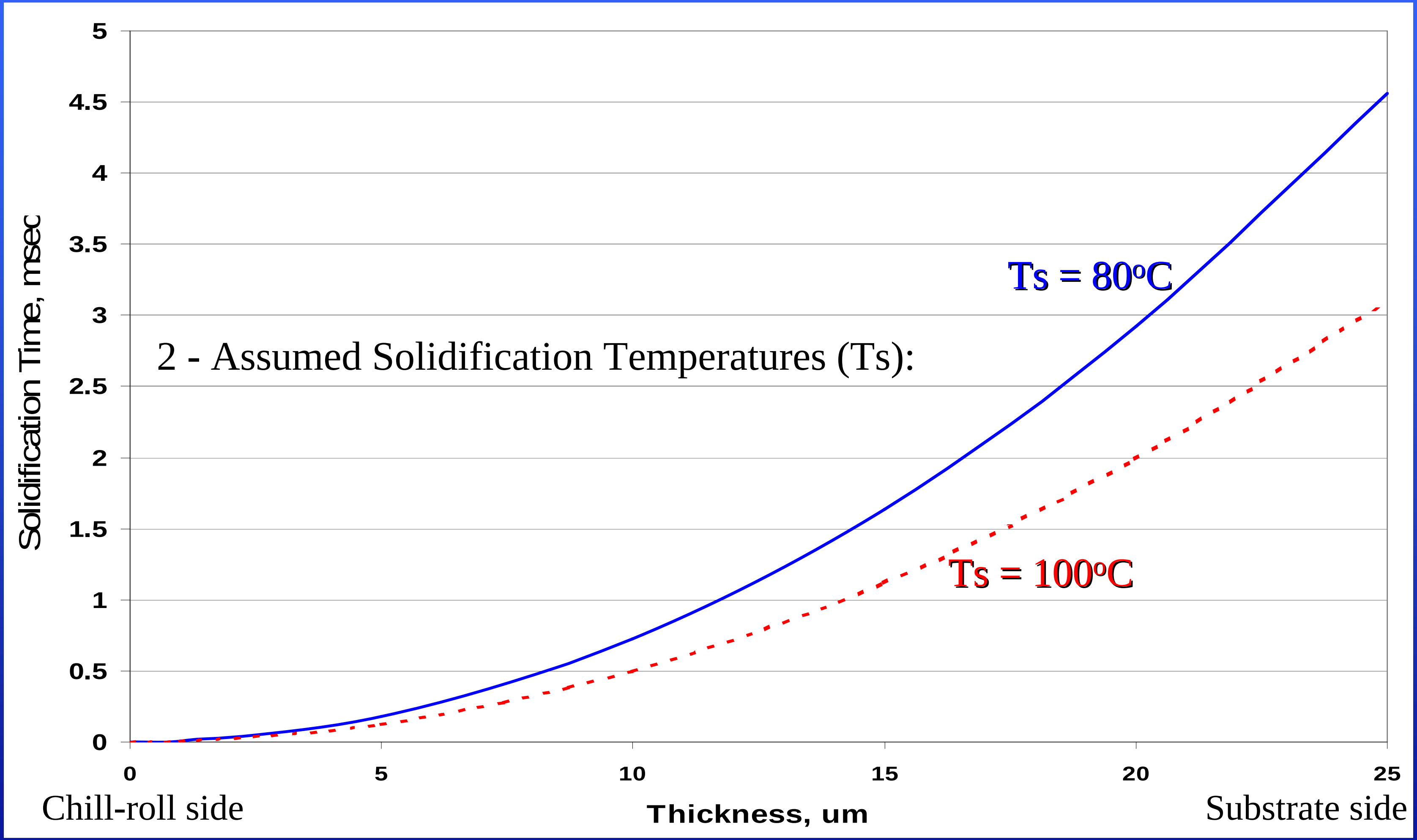


Fig. 4 – Solidification Time vs. Melt Temperature

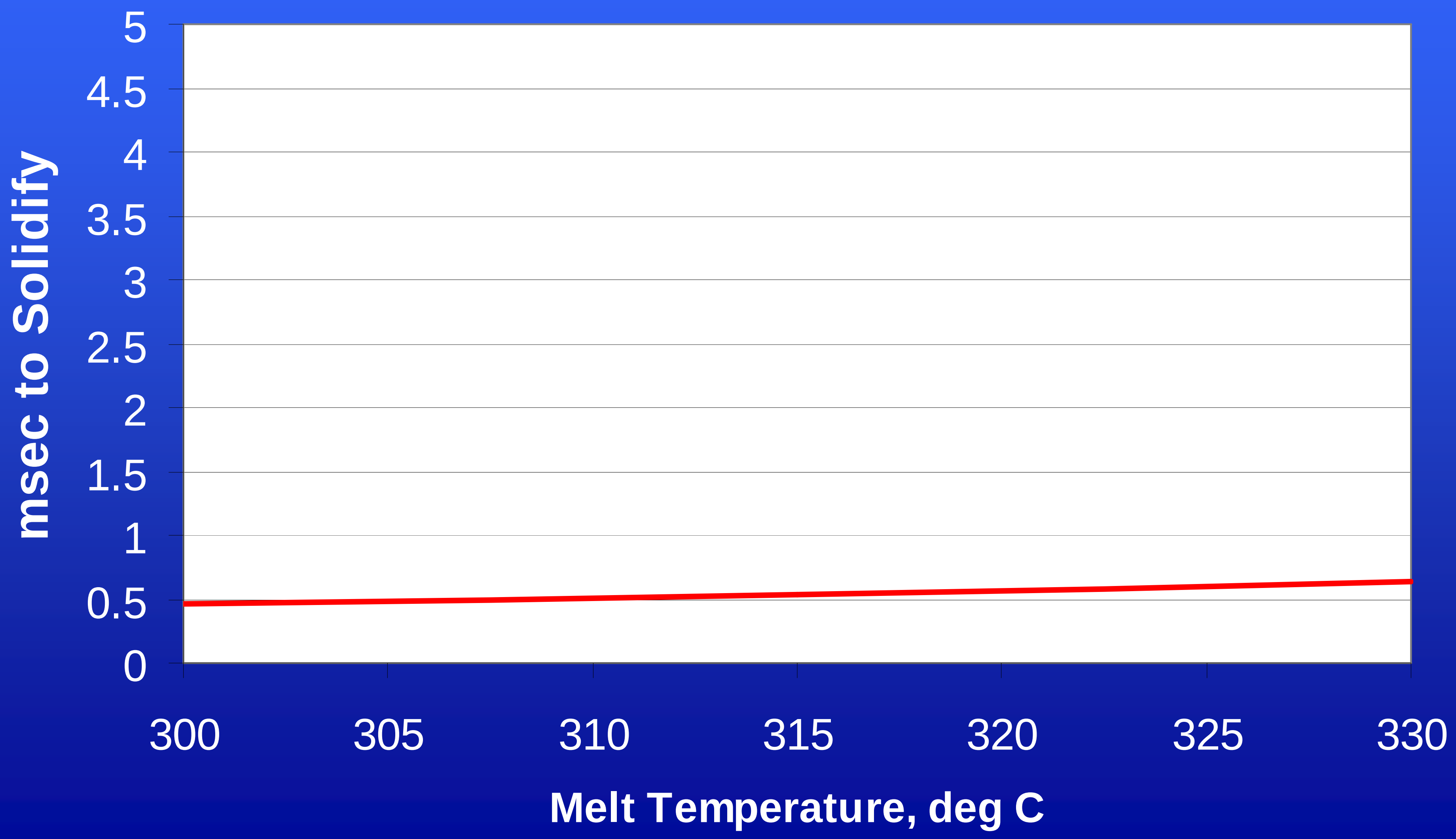
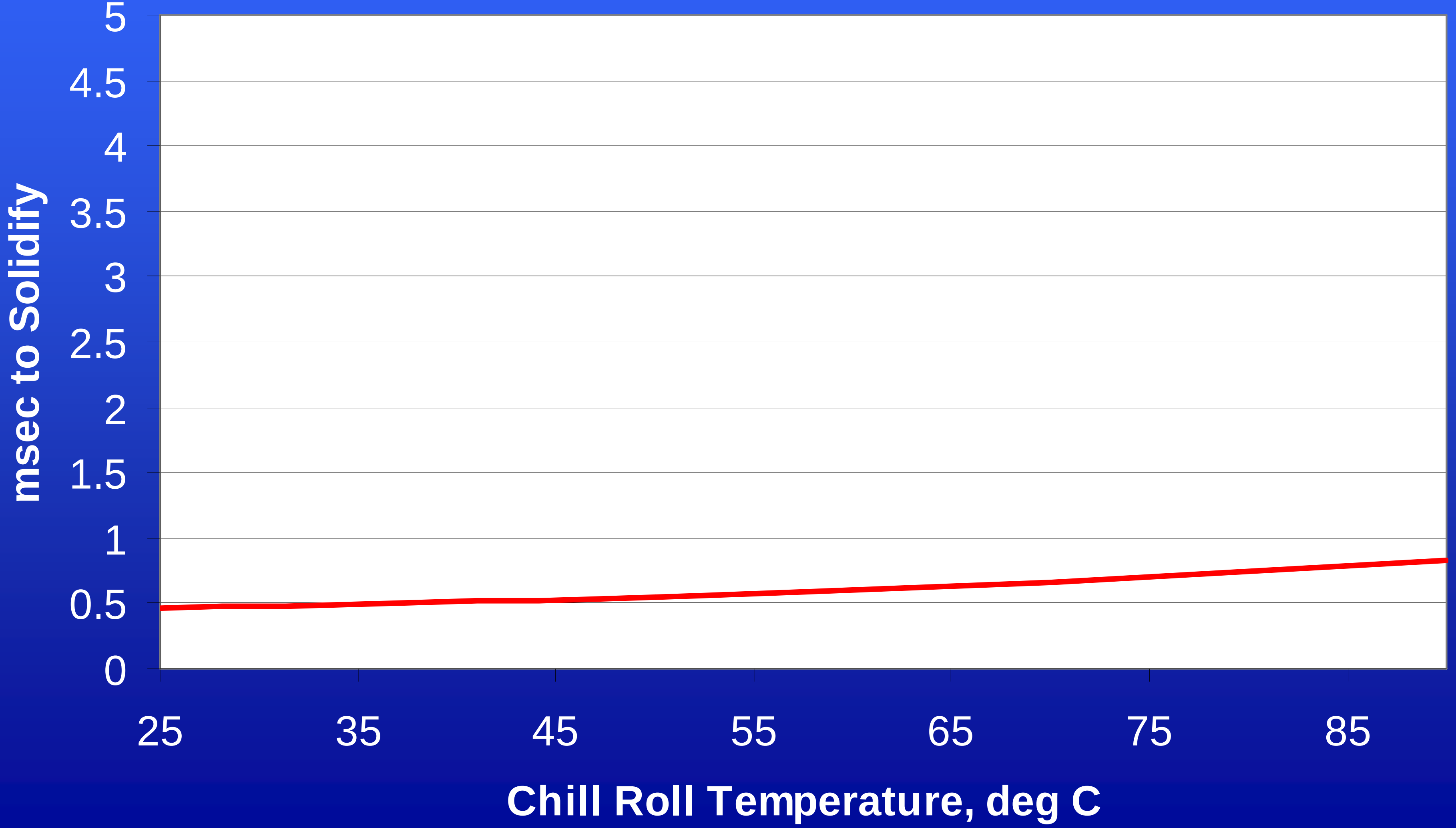


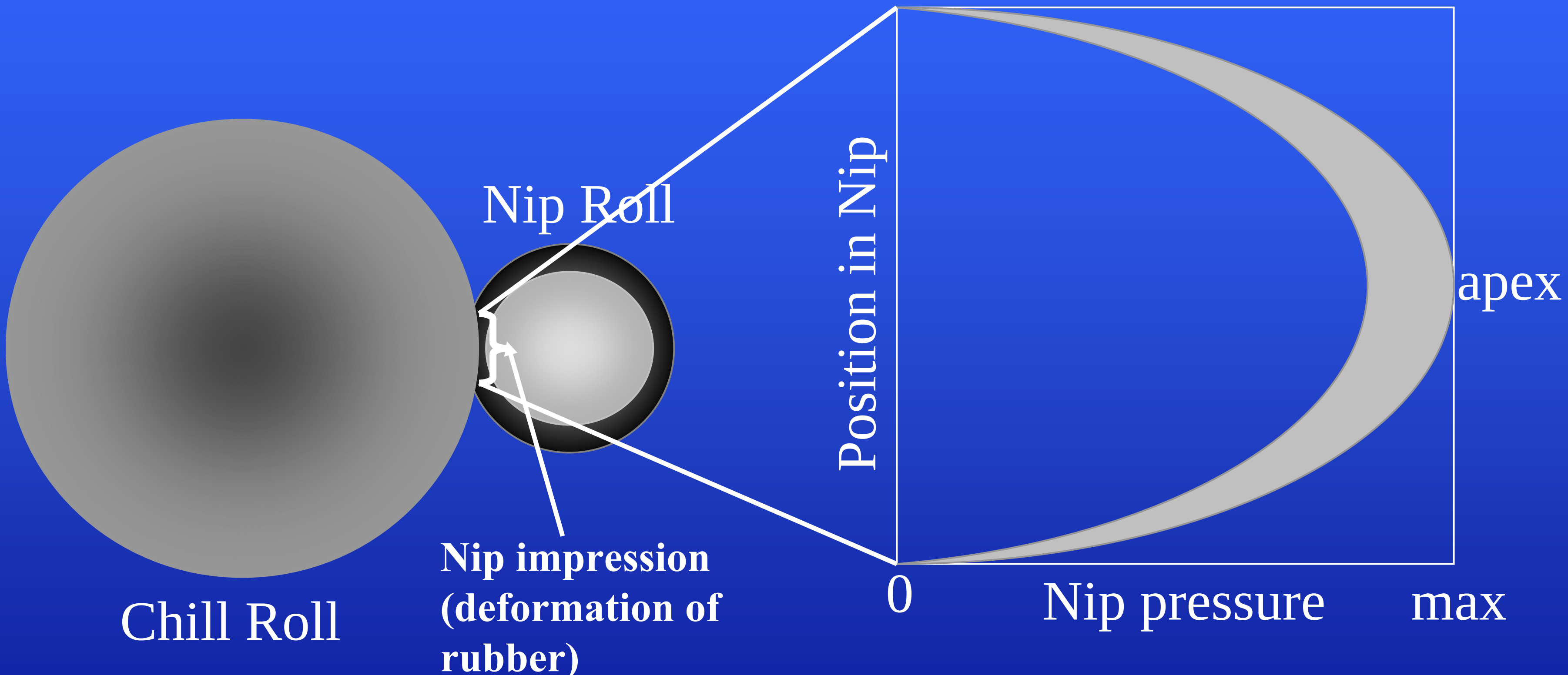
Fig. 5 – Solidification Time vs. Chill Roll Temperature



Machine Factors That Can Improve Intimate Contact

1. Extrudate thickness
2. **Nip conditions (pressure & length)**
3. Melt temperature

Fig. 6 – Nip Conditions



$$\text{Time in Nip, msec} = \frac{\text{60 x Nip length, mm}}{\text{Line speed, mpm}}$$

Fig. 7
Effect of Nip Length & Line Speed

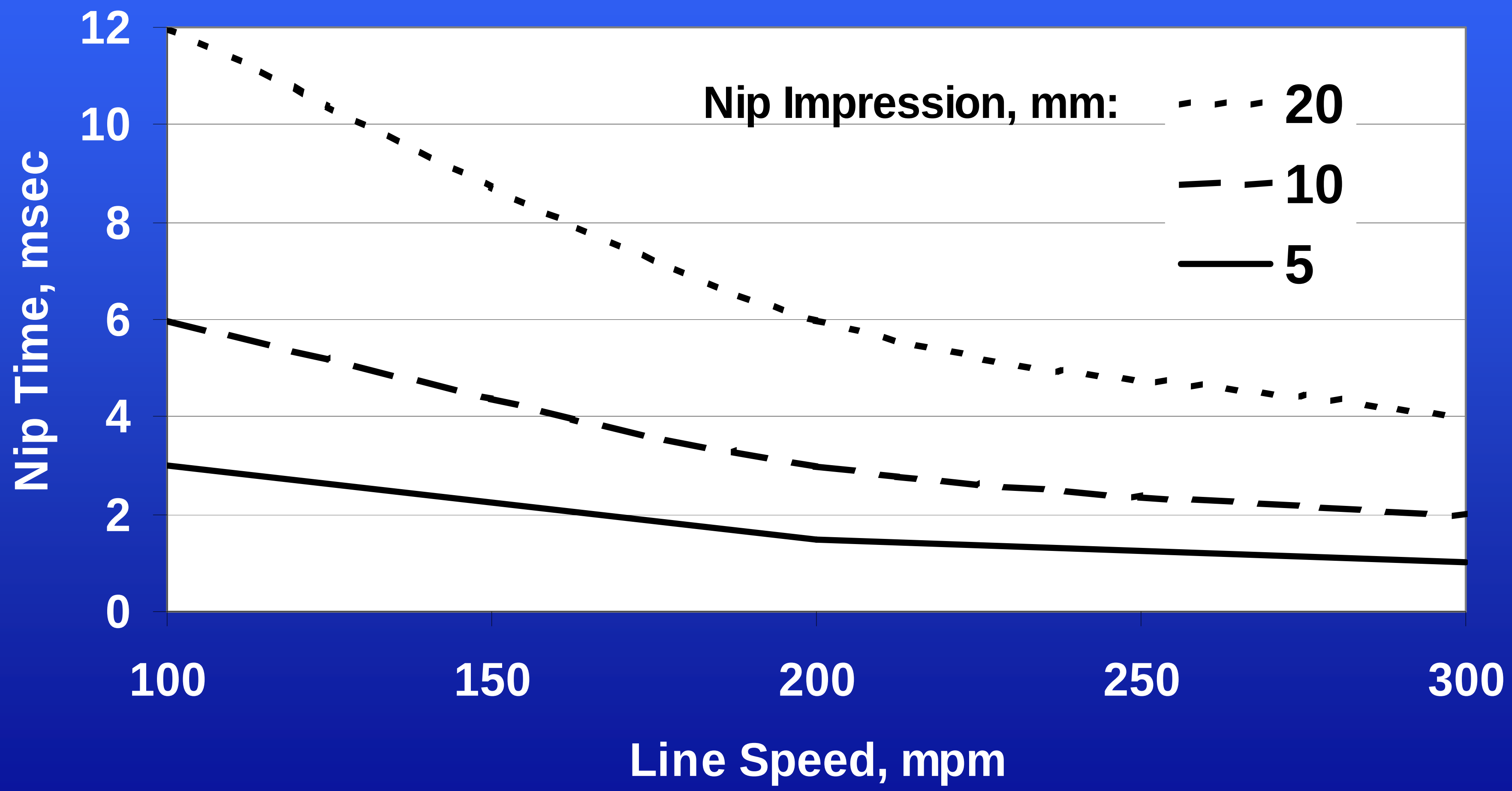
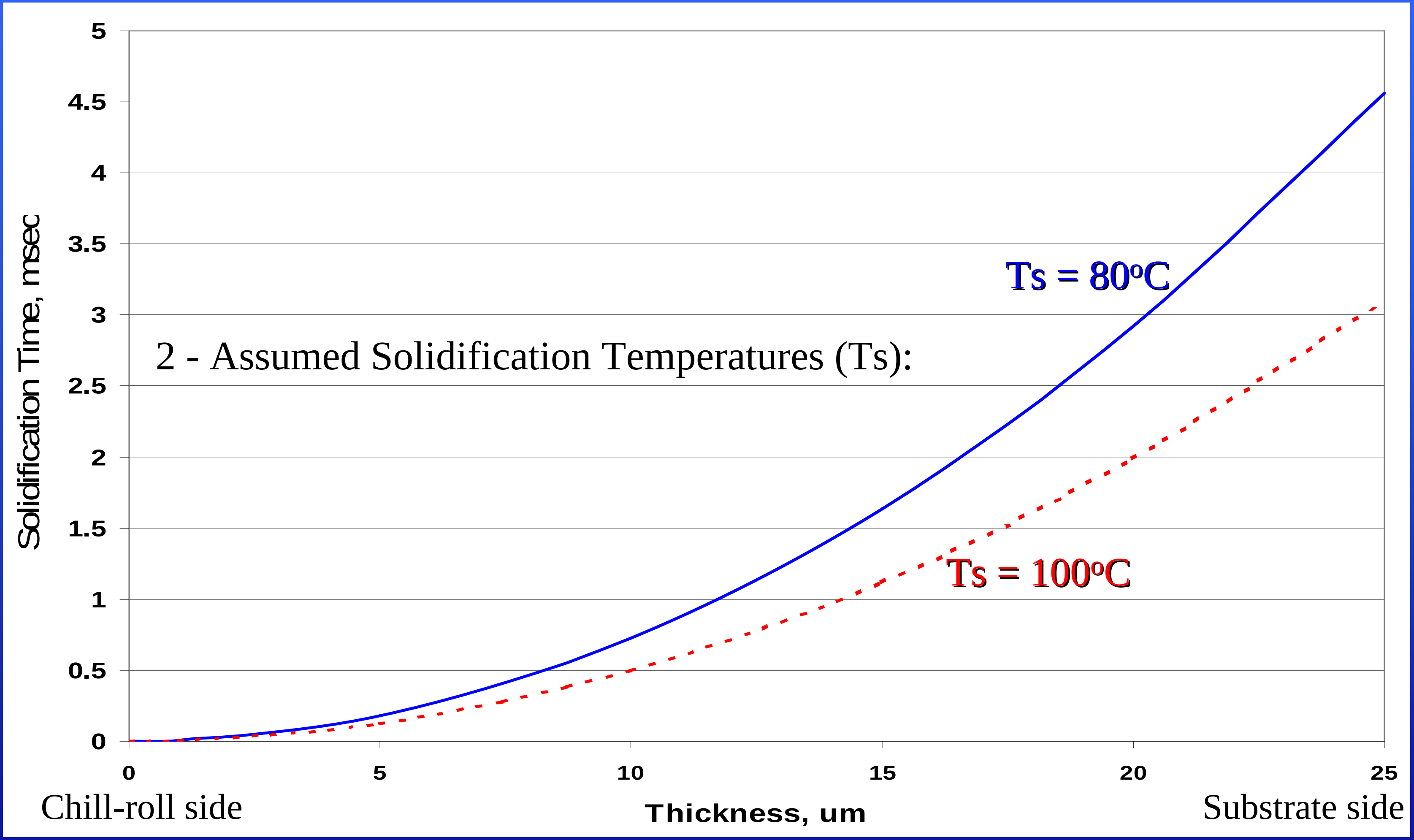


Fig. 3
Solidification Time vs. Thickness



Guidelines for Nip Length & Pressure

- Softer Polymers (e.g.: LDPE, EVA, EMA)
  - Longer impression, lower pressure (softer nip roll)
- Harder :Polymers (e.g.: HDPE, PP, PET)
  - Shorter impression, higher pressure (harder nip roll)

Machine Factors That Can Improve Intimate Contact

1. Extrudate thickness
2. Nip conditions (pressure & length)
3. **Melt temperature**

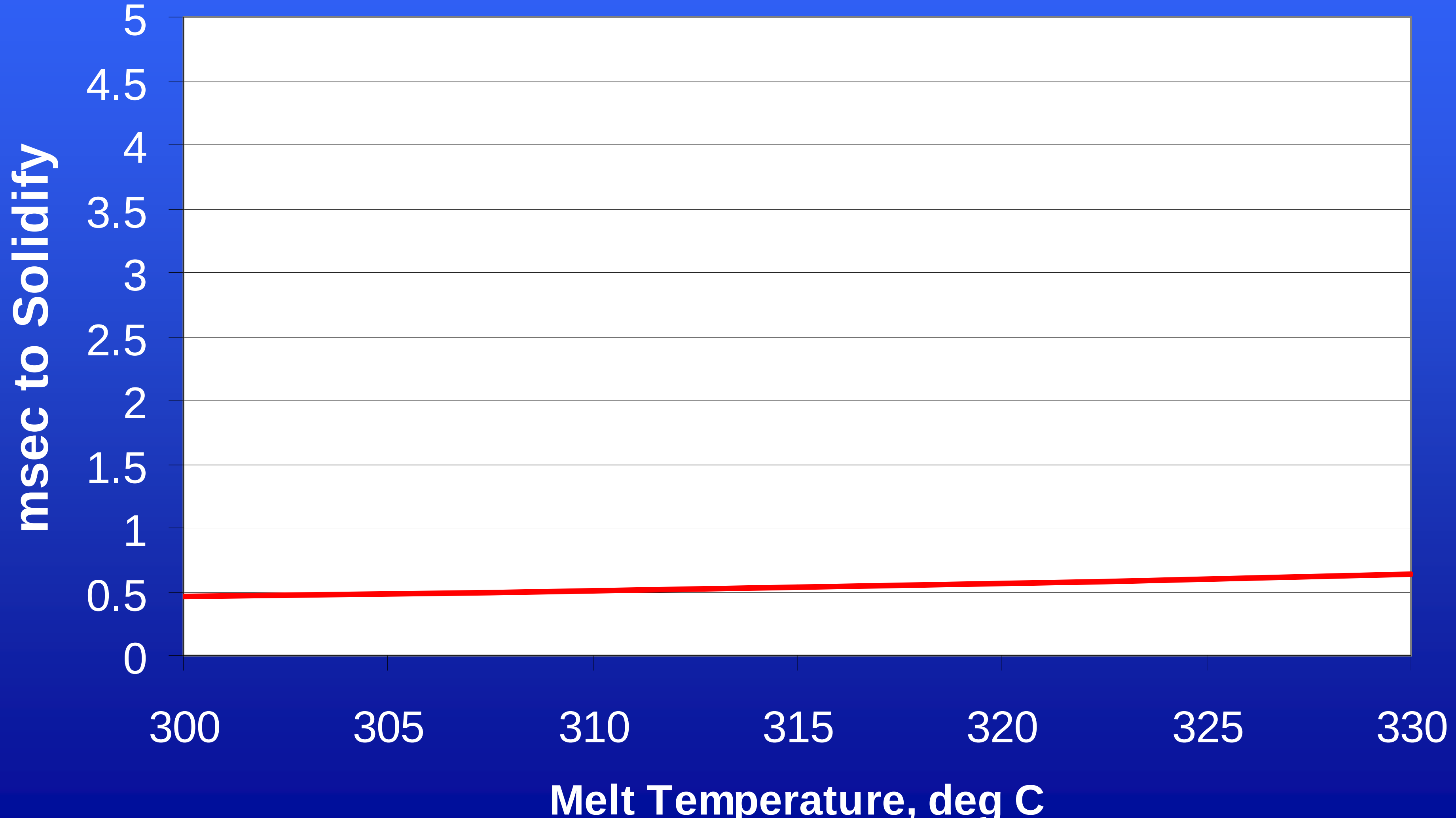
Melt Temperature (T)
& Intimate Contact

↑ **T** → Lower Viscosity **....Yes!**

↑ **T** → Improve intimate contact? **....No!**

Well…… maybe………..

Fig. 4 – Solidification Time vs. Melt Temperature



Melt Temperature (T) & Intimate Contact

↑ **T** → More surface oxidation

↑ Surface oxidation →

Better wetting of substrate

Machine Factors That Can Improve Intimate Contact

1. **Extrudate thickness**
2. **Nip conditions (pressure & length)**
3. **Melt temperature**

Intimate contact

\+

Chemical bonding

=

Durable Adhesion

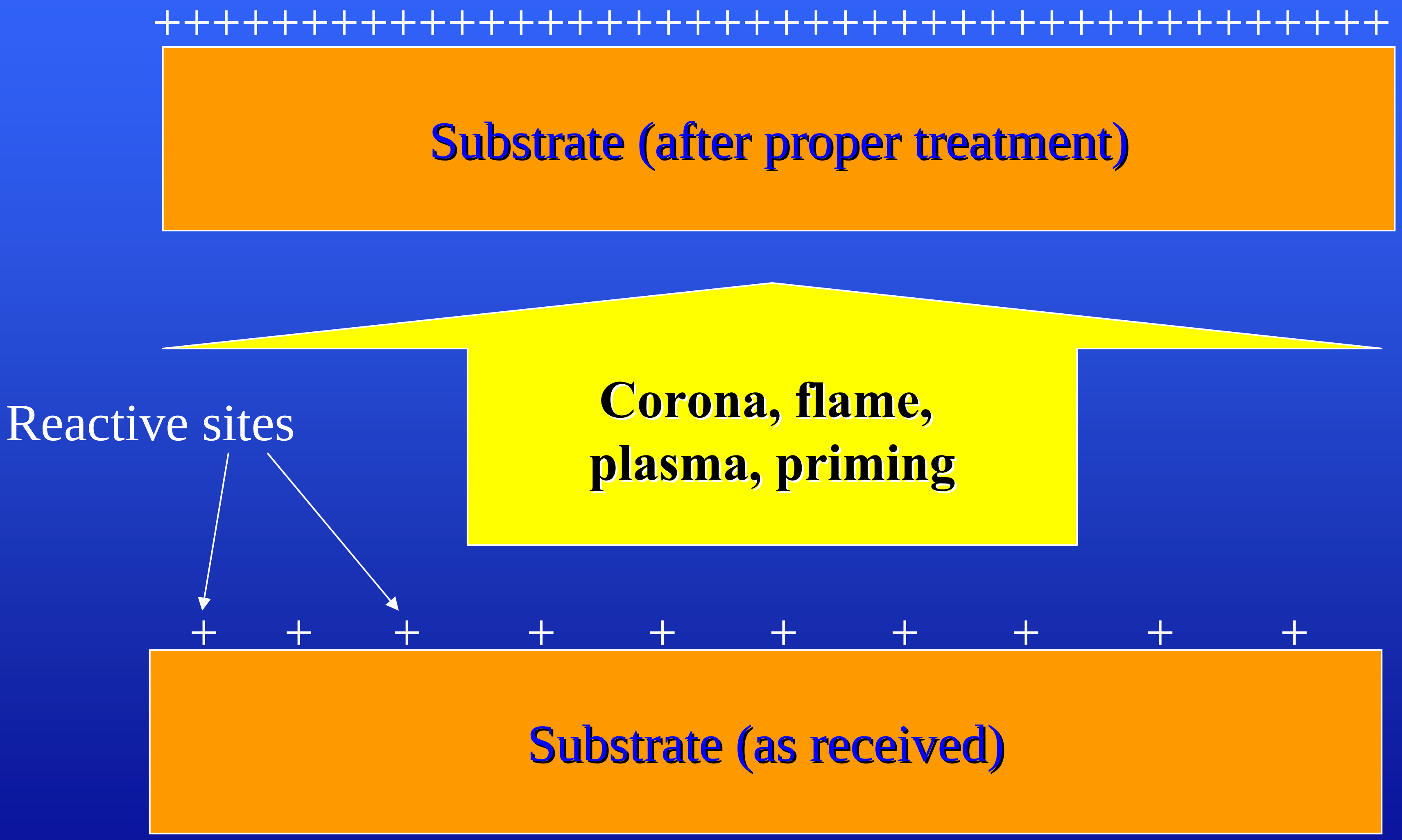
Machine Factors That Can Improve Chemical Bonding

- Substrate treatment conditions (corona or flame power level, or primer application variables)
- Choice of extrudate type
- Extrudate thickness
- Air-gap distance
- Melt temperature of extrudate
- Extruder back-pressure
- Ozone assist

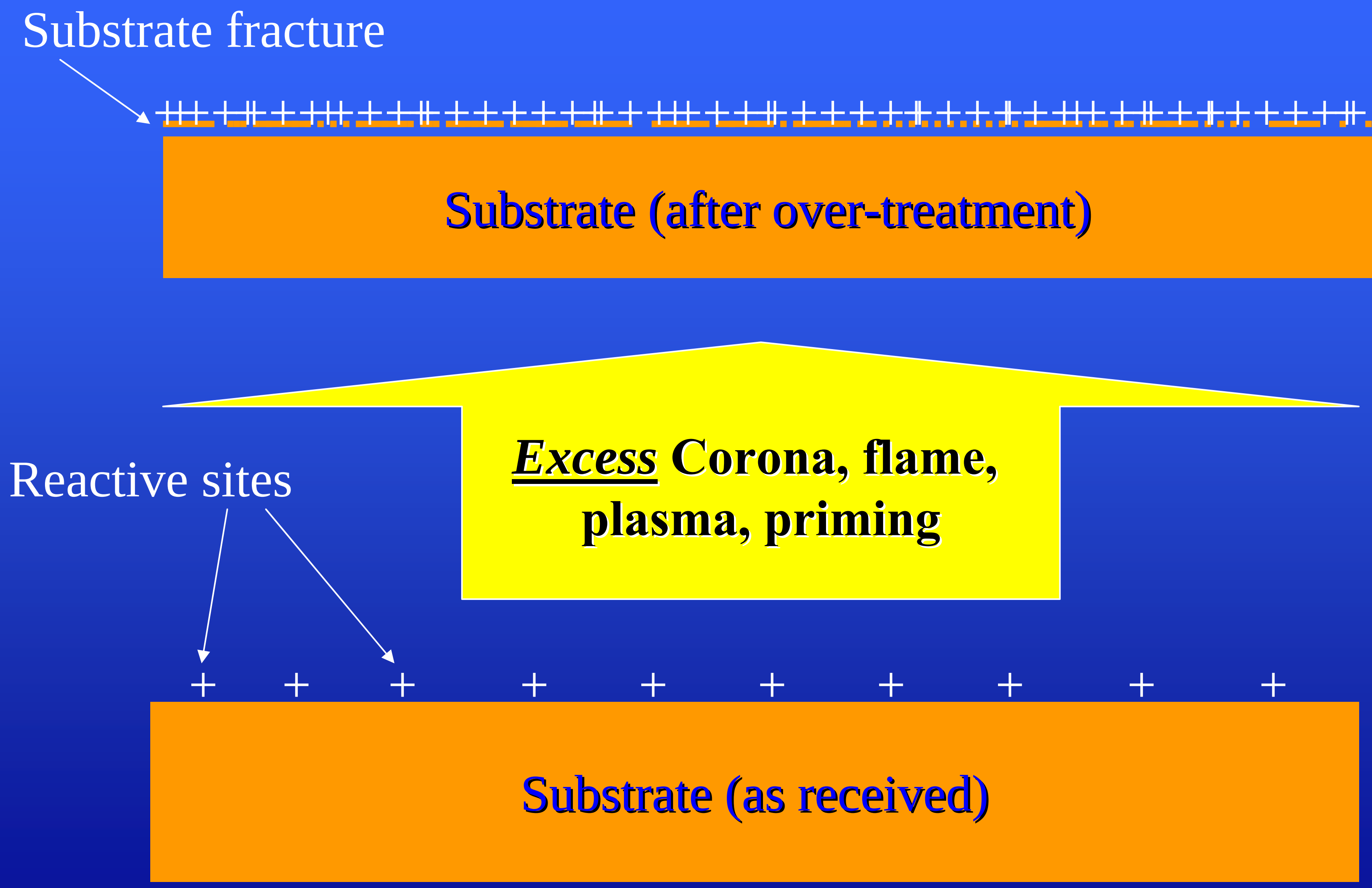
Substrate Treatment

- Treatment adds reactive sites to substrate
- Consult the supplier for PROPER treatment levels for your substrates
- Avoid over-treatment

Substrate Treatment



Avoid Over-Treatment!



Corona Treatment of Films
Use Proper Watt-Density

Watt-Density = Power / area / time

Metric Units: W/m^2/minute

[KW setting x1000] / [Line Speed (m/min) x width (m)]

Example: Treater is 3.0kW, line speed is 130mpm, and the treater width is 1400mm (1.4m), then the watt-density is:

(3.0 x 1000) / (130 x 1.4) = 16.5 W/m^2/min

<u>Important:</u> You must determine optimum watt-density for each film and each set of running conditions. Some starting suggestions:

BOPP films: 30 – 40 WD
OPET films: 15 – 20 WD
BON films: 5 – 15 WD

Other Treatment Methods

- Flame – proper air/gas ratio & manifold position
- Chemical priming – primer choice, proper amount & complete drying
- Plasma – ask the experts!

Machine Factors That Can Improve Chemical Bonding

- Substrate treatment conditions (corona or flame power level, or primer application variables)
- Choice of extrudate type
- Extrudate thickness
- Air-gap distance
- Melt temperature of extrudate
- Extruder back-pressure
- Ozone assist

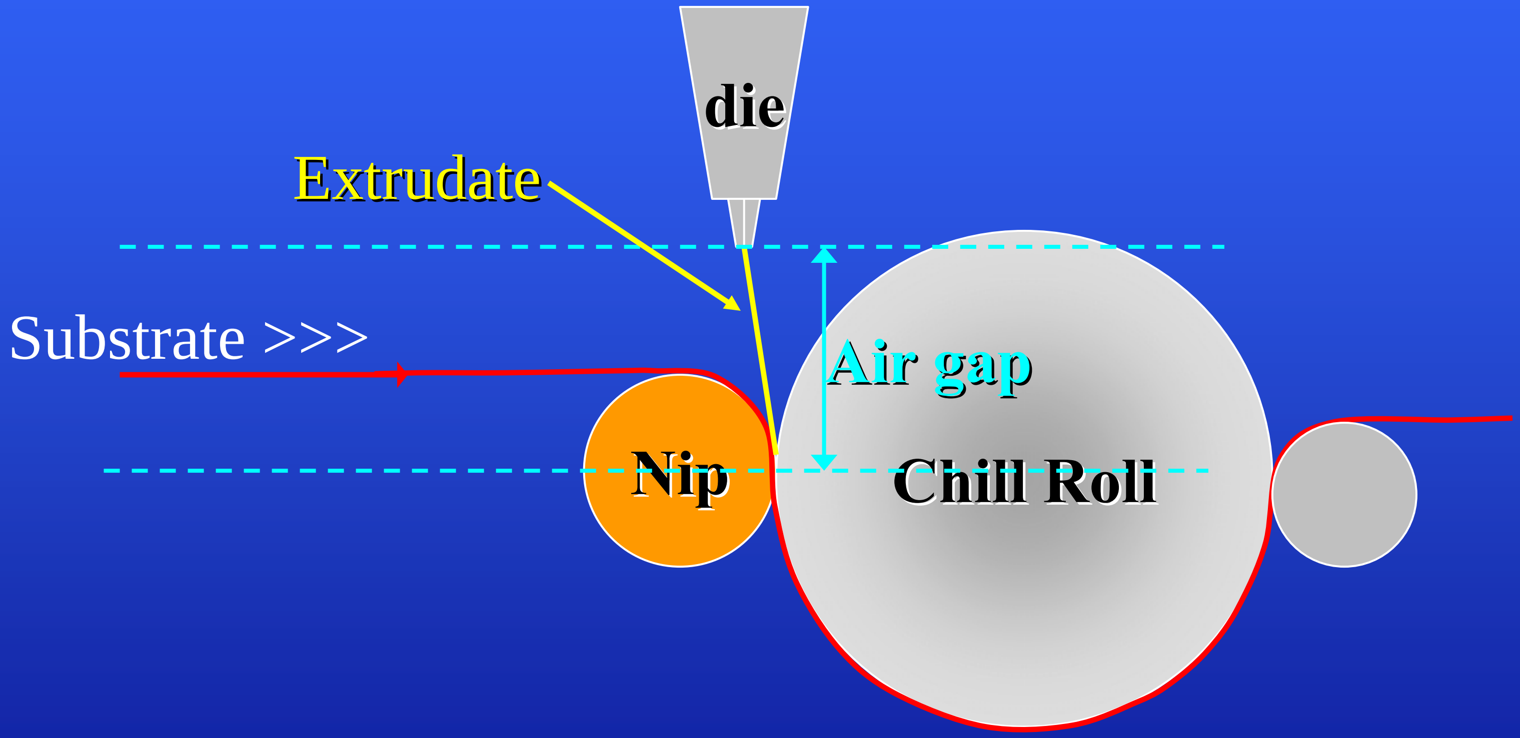
Choice of Extrudate
(General "Rules of Thumb")

- LDPE – for paper, primed plastics & primed foils
- Acid copolymers – for metal substrates & primed films
- Ionomers – for metal & primed substrates
- Acrylate copolymers – for PP & PET films, & primed surfaces

Machine Factors That Can Improve Chemical Bonding

- Substrate treatment conditions (corona or flame power level, or primer application variables)
- Choice of extrudate type
- Extrudate thickness
- Air-gap distance
- Melt temperature of extrudate
- Extruder back-pressure
- Ozone assist

Thickness, Air-Gap, Melt Temp



4 – References:

Ref. 3 - W.J. ***Ristey*** and R.N. ***Schroff***; The Degradation and Surface Oxidation of Polyethylene During the Extrusion Coating Process, 1978 TAPPI Paper Synthetics Proceedings, TAPPI PRESS, Atlanta, p.267

Ref. 4 - V. ***Antonov*** and A.M. ***Soutar***; Foil Adhesion With Copolymers: Time in the Air Gap, 1991 TAPPI Polymers, Laminations, and Coatings Conference Proceedings, TAPPI PRESS, Atlanta, p.553

Ref. 5 - B.A. ***Morris*** & N. ***Suzuki***; The Case Against Oxidation as a Primary Factor for Bonding Acid Copolymers to Aluminum Foil, 2000 TAPPI Polymers, Laminations, and Coatings Conference Proceedings, TAPPI PRESS, Atlanta

Ref. 6 – B. ***Foster***; Effect of Extrusion Coating Parameters on Activation of a Primed Film, 2002 TAPPI PLACE Proceedings, TAPPI PRESS, Atlanta Paper 14-2.

Ristey & Schroff - 1978

- Studied Effects of:
  - Air-Gap
  - Melt Temperature
  - Thickness
- On These Properties:
  - Molecular Weight Changes
  - Surface Oxidation

Ristey & Schroff - 1978

- Most Significant Conclusion:

25mm ↑air-gap = 5.5^{o}C ↑melt temp

For LDPE surface oxidation between 321 – 338^{o}C

4 – References:

Ref. 3 - W.J. ***Ristey*** and R.N. ***Schroff***; The Degradation and Surface Oxidation of Polyethylene During the Extrusion Coating Process, 1978 TAPPI Paper Synthetics Proceedings, TAPPI PRESS, Atlanta, p.267

Ref. 4 - V. ***Antonov*** and A.M. ***Soutar***; Foil Adhesion With Copolymers: Time in the Air Gap, 1991 TAPPI Polymers, Laminations, and Coatings Conference Proceedings, TAPPI PRESS, Atlanta, p.553

Ref. 5 - B.A. ***Morris*** & N. ***Suzuki***; The Case Against Oxidation as a Primary Factor for Bonding Acid Copolymers to Aluminum Foil, 2000 TAPPI Polymers, Laminations, and Coatings Conference Proceedings, TAPPI PRESS, Atlanta

Ref. 6 – B. ***Foster***; Effect of Extrusion Coating Parameters on Activation of a Primed Film, 2002 TAPPI PLACE Proceedings, TAPPI PRESS, Atlanta Paper 14-2.

Antonov & Soutar - 1991

Introduced the concept of:

Time In the Air-Gap (TIAG)

Simplifying Assumption: Extrudate Speed = Line Speed

$$\text{Then, TIAG (msec)} \cong \frac{\text{Air-Gap (mm)} \times 60}{\text{Line Speed (mpm)}}$$

Antonov & Soutar - 1991

- Most Significant Conclusion:

Air-gap time of ca. 80 – 120 msec

To allow oxidation of EAA & thereby get good adhesion to aluminium foil

Subsequently found to be a good "rule of thumb" for LDPE resins

Time In the Air-Gap

Has been & continues to be a useful tool for troubleshooting adhesion problems in extrusion coating & laminating processes

But....

4 – References:

Ref. 3 - W.J. ***Ristey*** and R.N. ***Schroff***; The Degradation and Surface Oxidation of Polyethylene During the Extrusion Coating Process, 1978 TAPPI Paper Synthetics Proceedings, TAPPI PRESS, Atlanta, p.267

Ref. 4 - V. ***Antonov*** and A.M. ***Soutar***; Foil Adhesion With Copolymers: Time in the Air Gap, 1991 TAPPI Polymers, Laminations, and Coatings Conference Proceedings, TAPPI PRESS, Atlanta, p.553

Ref. 5 - B.A. ***Morris*** & N. ***Suzuki***; The Case Against Oxidation as a Primary Factor for Bonding Acid Copolymers to Aluminum Foil, 2000 TAPPI Polymers, Laminations, and Coatings Conference Proceedings, TAPPI PRESS, Atlanta

Ref. 6 – B. ***Foster***; Effect of Extrusion Coating Parameters on Activation of a Primed Film, 2002 TAPPI PLACE Proceedings, TAPPI PRESS, Atlanta Paper 14-2.

Morris & Suzuki - 2000

Most significant conclusion:

Oxidation is *not* the mechanism for EAA* adhesion to aluminium foil, but

Time in the air-gap *does* influence the adhesion

* For Nucrel grades of 9% & 12% acid

Morris & Suzuki - 2000

Another conclusion:

For LDPE & low-acid EAA copolymers,

Oxidation *is* a key mechanism for adhesion

4 – References:

Ref. 3 - W.J. ***Ristey*** and R.N. ***Schroff***; The Degradation and Surface Oxidation of Polyethylene During the Extrusion Coating Process, 1978 TAPPI Paper Synthetics Proceedings, TAPPI PRESS, Atlanta, p.267

Ref. 4 - V. ***Antonov*** and A.M. ***Soutar***; Foil Adhesion With Copolymers: Time in the Air Gap, 1991 TAPPI Polymers, Laminations, and Coatings Conference Proceedings, TAPPI PRESS, Atlanta, p.553

Ref. 5 - B.A. ***Morris*** & N. ***Suzuki***; The Case Against Oxidation as a Primary Factor for Bonding Acid Copolymers to Aluminum Foil, 2000 TAPPI Polymers, Laminations, and Coatings Conference Proceedings, TAPPI PRESS, Atlanta

Ref. 6 – B. ***Foster***; Effect of Extrusion Coating Parameters on Activation of a Primed Film, 2002 TAPPI PLACE Proceedings, TAPPI PRESS, Atlanta Paper 14-2.

Experiment Set #1 – Feb '02

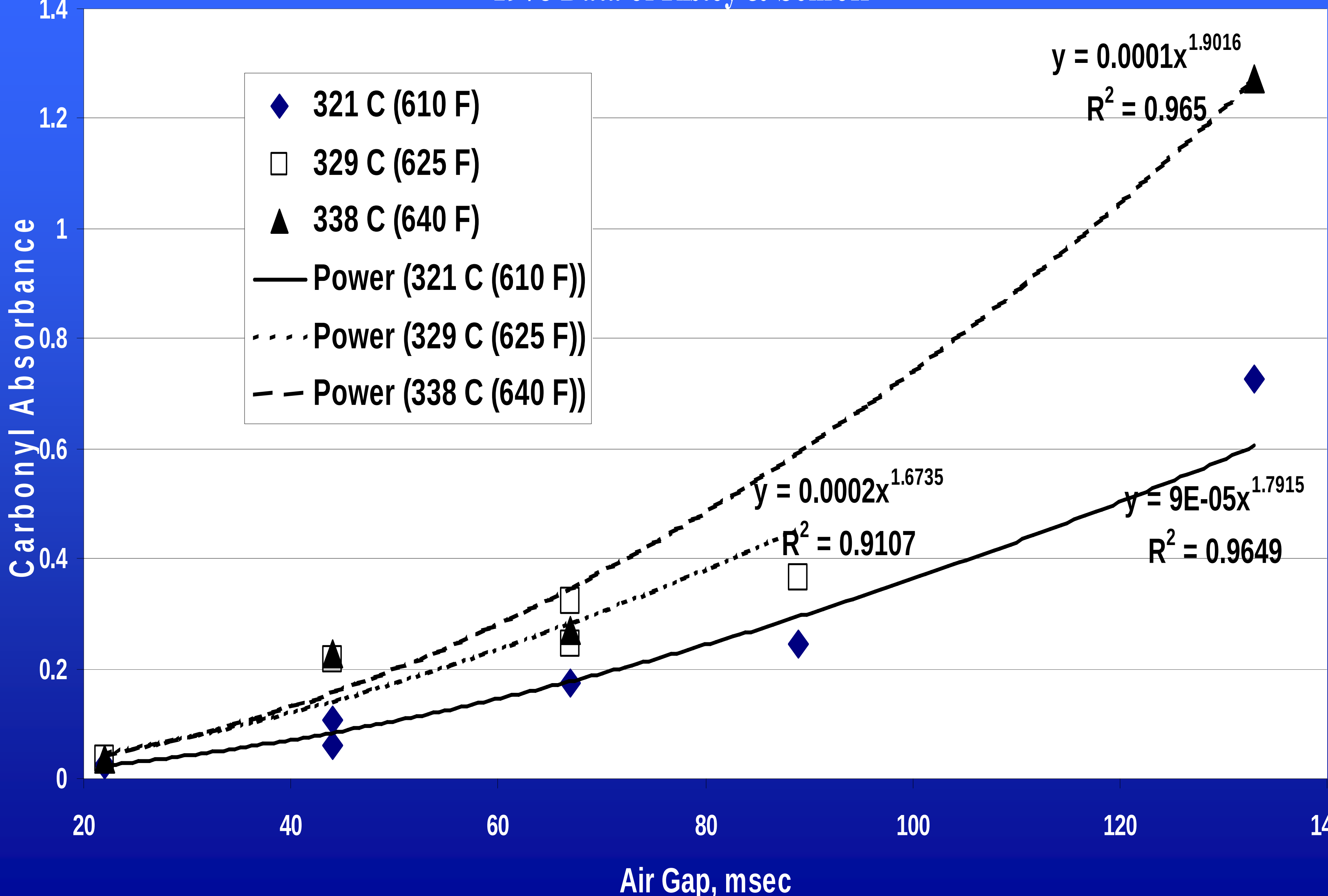
Experimental Design - Modified Box-Benkin (center and edge-centers)						
Variables: Melt temp, Acid#, Coat Wt, Offset						
Fixed Line-Speed @26mpm (85fpm) & Air-Gap at 44mm (100msec)						
Point #	**Melt Temp**	**Acid #**	**Coat Wt**	**Offset**	**Adhesion**	**Comments**
	deg C (deg F)		**gsm (#/rm)**	**(0, -0.5)**	**(g/25mm)**	
1	**271 (520)**	**0**	**20 (12)**	**0 & -0.5**	**0**	
2	**271 (520)**	**3**	**10 (6)**	**-0.5**	**FT @300**	
3	271 (520)	3	30 (18)		-	
4	**271 (520)**	**9**	**20 (12)**	**0**	**FT @2000**	
5	**293 (560)**	**0**	**10 (6)**	**0 & -0.5**	**FT @300**	
6	**293 (560)**	**0**	**30 (18)**	**0 & -0.5**	**FT @900**	
7	293 (560)	3	20 (12)		-	
8	293 (560)	9	10 (6)		-	Degraded melt
9	293 (560)	9	30 (18)		-	
10	315 (600)	0	20 (12)		-	
11	315 (600)	3	10 (6)		-	
12	315 (600)	3	30 (18)		-	
13	315 (600)	9	20 (12)		-	Max T= 302 (575)
					- not tested	

Experiment Set#2 – May '02

Following are designed to vary air-gap distance & TIAG, all with LDPE only: (and the primed stock is now 4 months old!)							
Pt. #	Melt Temp deg C (F)	Line Speed mpm(fpm)	Coat Wt gsm (#/rm)	Air-Gap mm (inch)	Air Gap, msec	Adhesion (g/25mm)	Failure Mode
1	293 (560)	15 (50)	10 (6)	25 (1.0)	100	40	
2	293 (560)	26 (85)	10 (6)	44 (1.7)	103	35	
3	293 (560)	15 (50)	10 (6)	89 (3.5)	350	300	FT
4	293 (560)	15 (50)	20 (12)	25 (1.0)	100	50	
5	293 (560)	26 (85)	20 (12)	44 (1.7)	103	500	FT
6	293 (560)	26 (85)	20 (12)	64 (2.5)	147	500	FT
7	293 (560)	26 (85)	20 (12)	89 (3.5)	206	500	FT
8	293 (560)	15 (50)	20 (12)	89 (3.5)	350	500	FT
9	282 (540)	15 (50)	10 (6)	25 (1.0)	100	6	
10	282 (540)	26 (85)	10 (6)	44 (1.7)	100	7	
11	282 (540)	26 (85)	10 (6)	89 (3.5)	206	70	
12	282 (540)	15 (50)	10 (6)	89 (3.5)	350	70	
13	282 (540)	15 (50)	20 (12)	25 (1.0)	100	6	
14	282 (540)	26 (85)	20 (12)	44 (1.7)	100	9	
15	282 (540)	26 (85)	20 (12)	89 (3.5)	206	500	FT
16	282 (540)	15 (50)	20 (12)	89 (3.5)	350	500	FT
17	282 (540)	26 (85)	30 (18)	44 (1.7)	100	12	

Oxidation -vs- Air Gap Time

1978 Data of Ristey & Schroff



Foster -2002

- Most Significant Conclusion:

  (For oxidation of LDPE in air gap):

  - **Air-gap time > thickness > melt temp**

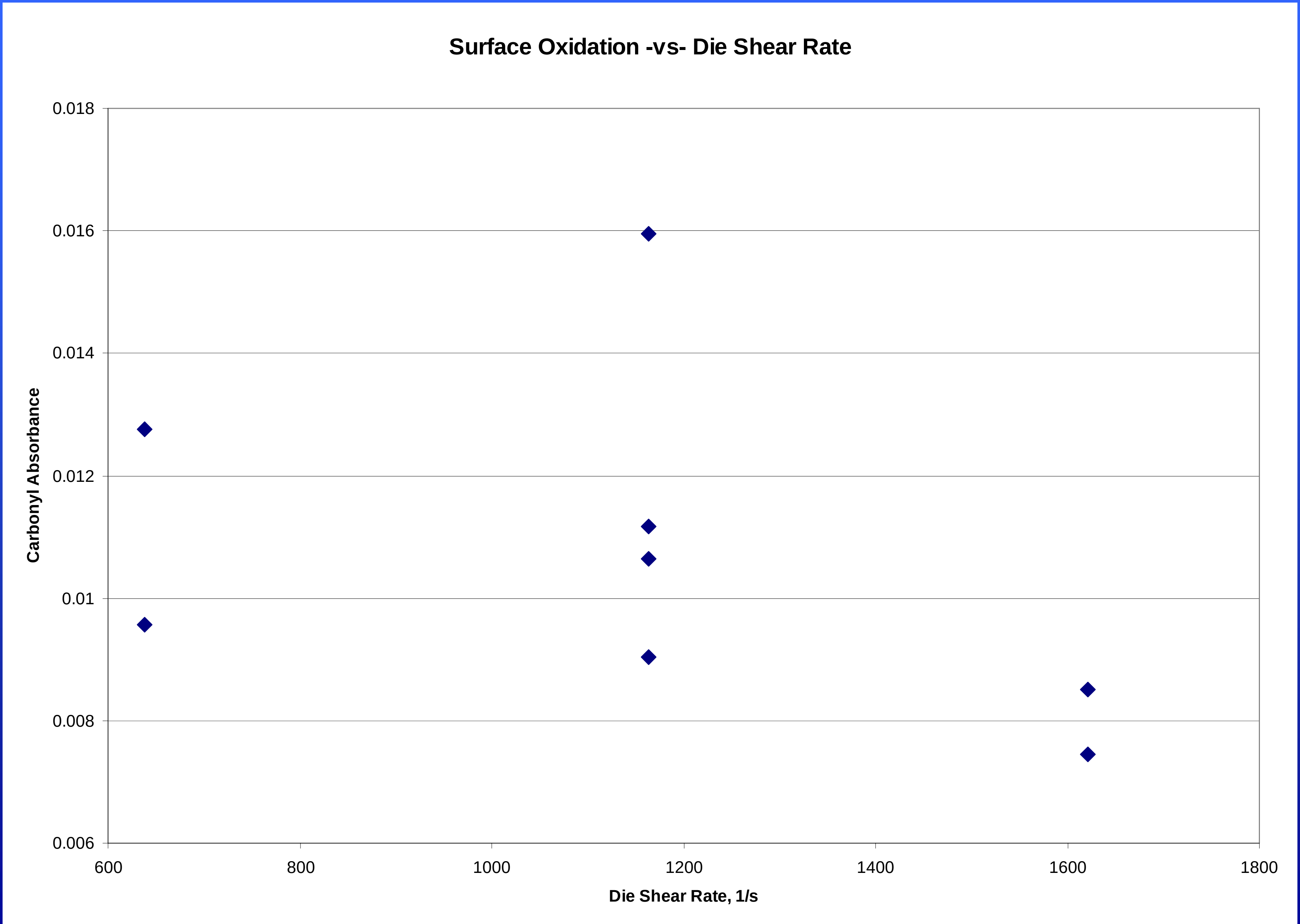
Scale-Up Differences..

Parameter	Pilot-Line Conditions	"Typical" Production Conditions
Line speed:	26m/min (85ft/min)	100-400m/min (~300-1300ft/min)
Air-gap:	44mm (1.75 inches)	150-300mm (6- 12 inches)
Die shear rate:	190sec^{-1}	500-2500sec^{-1}

Foster – 2002

Q – Is die shear rate an important factor?

Recent Experiment – Jan '05



Q – Is die shear rate an important factor?

A – Maybe! More work needed…

Machine Factors That Can Improve Chemical Bonding

- Substrate treatment conditions (corona or flame power level, or primer application variables)
- Choice of extrudate type
- Extrudate thickness
- Air-gap distance
- Melt temperature of extrudate
- Extruder back-pressure
- Ozone assist

Extruder Back Pressure

↑ Back Pressure:

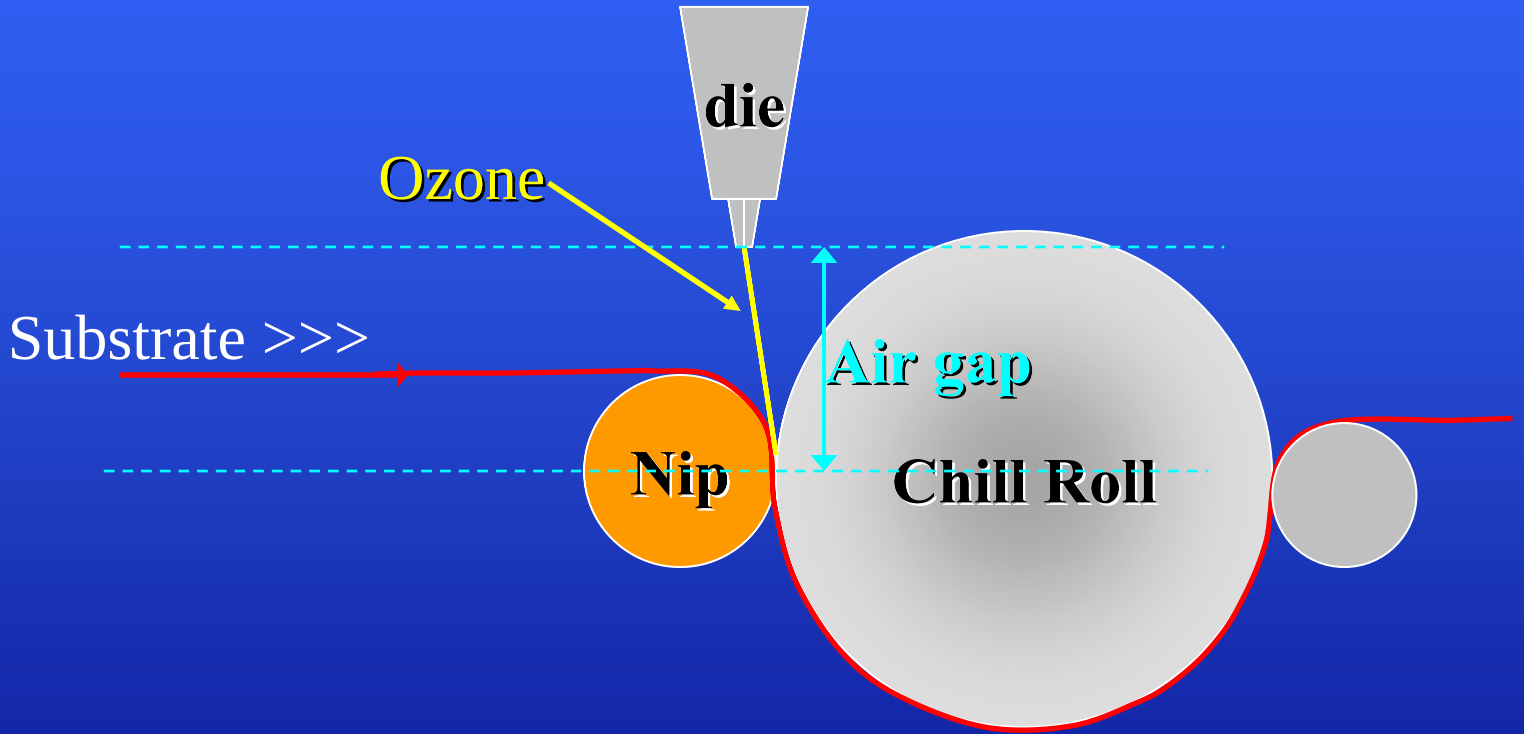
- **Creates more shear**
- **More polymer chain ends**
- **More oxidation**

- **More degradation! (possible heat-seal & bond aging problems)**

Machine Factors That Can Improve Chemical Bonding

- Substrate treatment conditions (corona or flame power level, or primer application variables)
- Choice of extrudate type
- Extrudate thickness
- Air-gap distance
- Melt temperature of extrudate
- Extruder back-pressure
- Ozone assist

Ozone Assist:



Ozone Assist

- Aids surface oxidation
- Lower melt temps and/or thinner coat weights may be possible
- Many References available

Conclusions

- Many factors affect adhesion
- More research needed to better understand the oxidation / mechanical process in the air-gap.