SOLIDIFICATION PROCESS IN EXTRUSION COATING

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ABSTRACT: The work to design equipment and resins for the extrusion coater is enormous. The extrusion coater can negate all this work in a fraction of a second by not setting the parameters at the coating station properly. The region from the die, where the polymer is first extruded, to where the material leaves the chill roll is critical for good coatings. In this region the solidification process occurs and is discussed herein. Attention to details and an understanding of the interactions in this region will help design, setup and troubleshoot the coating process. A clearer understanding of this region will hopefully help the processor to produce a quality product consistently, as well as, enable the processor to more quickly troubleshoot his problems.

KEY WORDS: Extrusion Coating, Solidification, Chill Rolls, Nip Rolls, Process Variables

I. INTRODUCTION:

As noted in Figure #1, a great many variables interplay from the time the resin leaves the die to the time it is pulled off the chill roll. This is the solidification region. In this region, we can influence adhesion, barrier properties (pinholes, etc.), optical and mechanical strengths. Considering these phenomena occur in a matter of 5 to 10 feet of travel, at speeds upwards of several hundred feet per minute, one realizes the set up and understanding of this region is imperative to high speed quality production. We will attempt to view the critical elements from the design of the chill roll to process troubleshooting guidelines. Literature review, personal experiences and results of designed experiments are the background for the discussion. The work of R.T. Van Ness (6-8) and D. Djordjevic (4) is used throughout this paper. Their work is useful and highly recommended for the novice and the experienced practitioner. The variables we will review are:

- Die Parameters
  - Die Height
  - Die Lead
- Nip Roll
- Gap Settings/Draw Ratio
- Web Pretreatment
- Chill Roll Design
- Chill Roll Temperature
- Stripper Roll
- Temperature Differences
- Line Speed
- Substrate Characteristics

These parameters and their adjustments are necessary to run an extrusion coater for optimum results.

The heart of solidification in extrusion coating occurs on the chill roll. Understanding the transport phenomenum occurring in this region is important. As shown in Figure # 2, the heat transfer occurs across multiple boundaries. In its simplest form, heat put into the system by molten polymer needs to be pulled out by the cooling water. Two interfaces need to be crossed and a rate of heat transfer established across the mass (chill roll) when the system is at steady state.

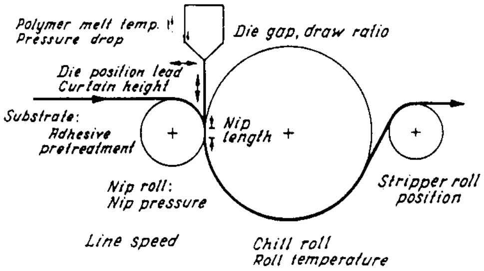

Figure #1
Process Variables

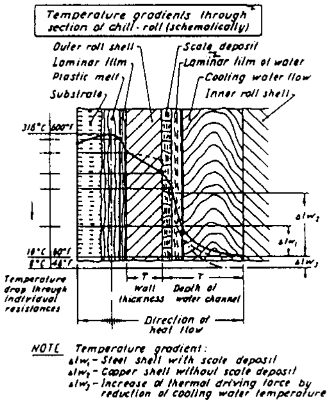

Figure #2
Chill Roll Gradiants Through Shell

In reality, this region is very complex. Scale or rust built up on the inside of the roll will create interfacial transfer problems. Air or water vapor entrained on the surface of the chill roll will change the heat transfer. Thermophysics of metals, thermodynamics and transport phenomena all present a very complex situation.

In its simplest form, heat in will equal heat out. The quantity of heat to be extracted is given by:

$$Q_{in} = m_k \cdot \Delta h \qquad (1)$$

where;

Q_{in} = heat quantity $_{in}$

M_k = mass throughput

Δh = enthalpy

Figure #3 shows the specific enthalpy of various polymers. Q_{out} needs to be extracted by the fluid media which is governed by:

$$Q_{out} = M_{Tm} \cdot C_{Tm} \cdot \Delta T_{TM} \qquad (2)$$

where;

Q_{out} = heat quanity $_{out}$

M_{Tm} = mass thruput of transfer medium

C_{Tm} = heat capacity of transfer medium

ΔT_{Tm} = change in temperature

Thus, at steady state:

Heat in = Heat out

or,

$$M_K \cdot \Delta h = M_{Tm} \cdot C_{Tm} \cdot \Delta T_{Tm} \qquad (3)$$

As an approximation, we can use h = heat capacity of polymer times T, the temperature difference, thus:

$$M_K \cdot C_p \cdot \Delta T_p = M_{Tm} \cdot C_{Tm} \cdot \Delta T_{Tm} \qquad (4)$$

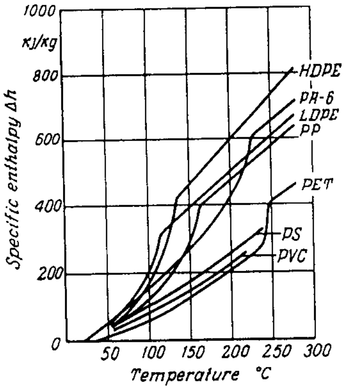

Figure #3
Special Enthalpy for Various Polymers

The standard ΔT_m used as a guide is 3°F. Thus, we can calculate the appropriate cooling system requirements (M_{Tm}) from this equation by knowing the material constants and rate of extrusion.

However, the real art of design is encountered when one becomes concerned with the interfaces. The governing equation is:

$$Q = A (T_F - T_W)/s \qquad (5)$$

where,

Q = heat quantity through interface

λ = thermal conductivity of surface

A = contact area

T_F = film temperature

T_W = roll temperature
S = film thickness

The amount of heat at steady state will be governed by equation #1. The area will be governed by the design geometry of the roll and the process. Temperature of the film is relative to strippability. T_W is governed by line speed and requires recovery time from film strip point to nip or curtain contact. However, T/S is very dependent on roll condition and requires experience in using the appropriate design factors. Equipment manufacturers can provide the appropriate constants. Through experience, one must realize that if the rolls are not maintained properly, these constants will change and the design efficiency of the roll will deteriorate. Figure #4 A, B, and C depict common overall heat transfer coefficients.

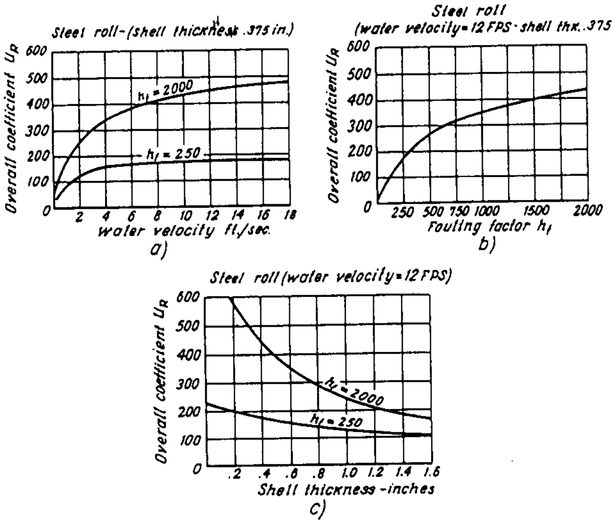

Figure #4
Overall Heat Transfer Coefficients

In many respects, solidification is the inverse of melting. As such, this step is a complex relationship of heat transfer and flow governed by geometric conditions and boundary conditions. (See Figure #5) In the simplest form, we are trying to balance a solidification front by a flow front, i.e. as the material freezes and gains in density, by decreasing volume, we need to be filling in the internal volume by the melt flow. The outside solidifies first and may be constrained by adhesion to the substrate and chill roll. If the center volume decreases and is not filled by the melt flow, a void will be created. This freezing or solidification creates problems such as curl, optical clarity and mechanical strengths. Figure #6 shows a theoretical change in temperature for a crystalline polymer under isothermal conditions. As can be seen, we will obtain a 12.5% change in density in less than a second. (Figure #7) These figures and theories are presented to show the variables influencing solidification and their interrelationship to actual processing. The equations and boundary equations to investigate the theory are available (1-3) and are more rigorous then space allows in this discussion.

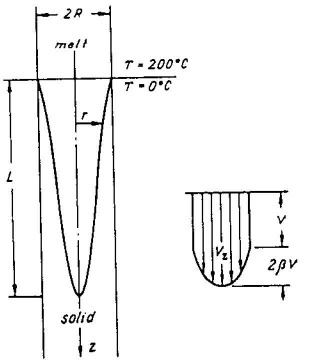

Figure # 5
Solidification Profile during Isothermal Solidification

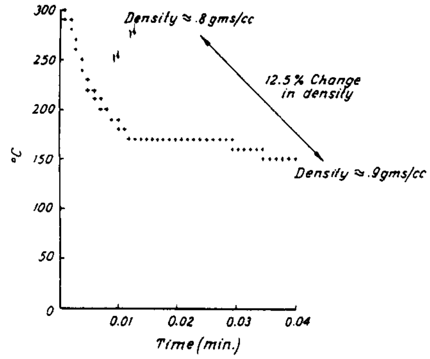

Figure #6
Isothermal Cooling Time

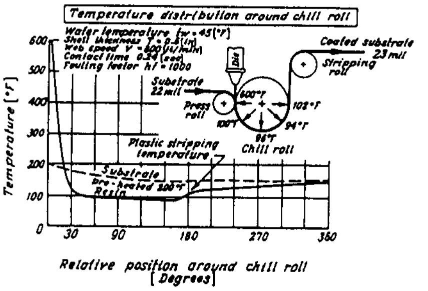

Figure 7
Chill Roll Temperature

EQUIPMENT: **Chill Roll**

Typical chill rolls have a double walled system. The inner wall is 3/4" to 1 1/4", depending on length and stiffness desired. The outer wall is 3/8" to 3/4" thick, with varying surface finishes. Rotary unions are connected to the two ends to allow passage of water. The design types can be varied from elementary chill cans to double walls with spirals of decreasing pitch. Usually the inner workings are designed to keep the temperature drop across the face to 3°F and to keep the flow rates at 12 to 15 ft./second.

Typically, a double walled spiraled chill roll is used. The widths of the spirals would be given by:

$$\text{Width of Passage} = \frac{\text{GPM X in}^3\text{/gal}}{\frac{\text{sec}}{\text{min}} \text{ X (velocity of water) X } {}^{\text{in}}\text{/ft. X depth between shells ( ) X no of passages}} \qquad (6)$$

The pitch of the spiral is then given by:

$$\text{Pitch} = (\text{width of passage} + \text{wall thickness}) \text{ X \# of passages} \qquad (7)$$

As mentioned previously, running clean fluid or soft water through the system is needed to keep the inner interface clean. In addition, periodic flushing and cleaning should be preformed to reduce rust and scale.

To aid in this cleaning, certain manufacturers offer removable shells. The outer surface of the chill roll can be removed and cleaned thoroughly. The advantage of such a system is that one base and associated hardware can be used for many surface shells. The disadvantage being that time, care and equipment is required in replacing shells. If change over time is essential, having individual rolls with matching hardware is more convenient.

Different roll surfaces are used for three primary reasons:

(1.) Release of extruded film
(2.) Optical properties of the film
(3.) Coefficient of friction of the film

The four basic patterns used are:

(1.) Gloss or Polished
(2.) Mirror Pocket
(3.) Satin (semi-gloss)
(4.) Matte

The polished roll has a low peak to valley surface finish (3-5 microinch finish). These rolls produce the highest opticals (high gloss, transparency and low haze), have higher coefficients of friction and have poor strippability. Matte rolls have a high peak to valley surface finish (40 to 180 or higher microinch finishes). These rolls produce the poorest opticals (low gloss, transparency and high haze), however, they produce films with low coefficients of friction and have excellent strippability. Adhesive layers, such as EMA, NBA and iononers, need to be extruded onto these rolls for best results. Satin (semi-gloss) rolls are intermediate to gloss and matte rolls, with associated intermediate properties. Mirror pocket rolls are similar to matte rolls except there is a plain surface having a majority of surface gloss interdispersed with small depressions. (Figure #8)

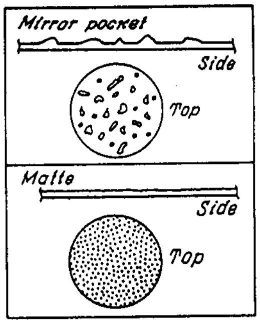

Figure #8
Mirror Pocket and Matte Finishes

Mirror pocket rolls can be produced with various surface finishes. Thus, we can find a compromise to C.O.F., optics and release using the mirror pocket rolls. Relative gloss and C.O.F. values are listed in Tables I and II. Release is usually proportional to the C.O.F. values with higher C.O.F. representing harder release.

Table I. Gloss Reading for Various Chill Rolls

	Kraft	*Semibleached board*	*Bleached board*	*Clay coated board*
Commercial matte	...	1	...	...
Matte	1	1	1	1
Satin	2	...	5	...
Mirror Pocket No. 1	3	6	8	8
Mirror Pocket No. 2	4	10	...	...
Mirror Pocket No. 3	14	22	...	30
Commercial gloss	...	12–21	...	...
TSL gloss	14	28	35	50

Table II. Coefficient of Friction for Various Roll Surfaces

	Kraft paper PE/PE	Kraft paper PE/Steel	Semibleached board PE/PE	Semibleached board PE/Steel
Commercial matte	...	...	0.45	0.22
Matte	0.55	0.25	0.48	0.28
Satin	0.46	0.32	...	...
Mirror Pocket No. 1	0.45	0.25	0.46	0.23
Mirror Pocket No. 2	0.47	0.33	0.40	0.23
Mirror Pocket No. 3	0.48	0.14	0.37	0.17
Commercial gloss	...	...	0.65	0.26–0.37
TSL gloss	0.6–0.10	0.42	0.86	0.30

Many materials are used to manufacture nip rolls. Table III shows the relative merit of these systems.

Table III Evaluation of Roll Materials

MATERIAL	DUROMETER A	FINISH	APPROX. TEMP. RESIST., F	DURABILITY	RELEASE	REL. COST	TAPE RETENTION	CONFORM-ABILITY
Neoprene	60-90	G	200-225	F	P	1	G	G
Viton	75	G	400-500	F	P	9	G	G
Teflon in Viton	87	G	400-500	F	VG	15	G	G
SS Teflon in Neoprene	80	F-G	200-225	F	F	3	G	G
Teflon in Hypalon	80	G-	250	F	P	2	G	G
Filled Nordel EPDM	77	G-	300	F	F	2	G	G
Hycar Nitrile	60-90	G	225	F	P	1.2		G
Silicone	55-85	F	450-500	P	VG	3	P	G
Polyurethane	82	G	180	F	F	2	G	G
Teflon Covered	90+	F	?	P	Ex	1.5-4		F-G
Steel	>100	Ex	High	Ex	Ex	1		P
Hypalon	80	G	250	F	P	1.2	G	G
Natural Rubber	50		180	P	P	1		G

P = Poor, F = Fair, G = Good, VG = Very Good, Ex = Excellent

EQUIPMENT: NIP ROLL

Nip rolls play an important part in adhesion, coating integrity, film appearance and caliper. Standard nip rolls are water-cooled and rubber coated. Surface temperatures usually run from 100 to 190°c with typical nip pressures from 9 to 12.5 kgcmn (50 to 70pli). Thus, materials need to withstand these rigorous conditions and environs (ozone from treaters and fumes from additives). Usual hardness ranges from 60 to 90A (Shore), with most rolls in the 90A (Shore) range.

Figure #9 shows the relationship of size to pressure distribution across the web as a function of roll size. Also shown is the relationship of rubber thickness at a given size and pressure load. In general, the larger the roll, the greater the adhesion, propensity for pin holes and longer wrap around by the substrate.

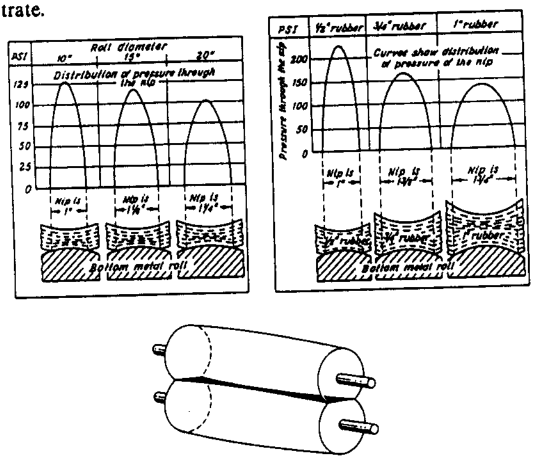

Figure #9
Effects of Nip Roll Size on Nip Pressure Distribution

Figure #10 shows the influence of roll hardness on the nip width. As expected the harder the roll, the narrower the nip gap and the sharper the distribution on the chill roll.

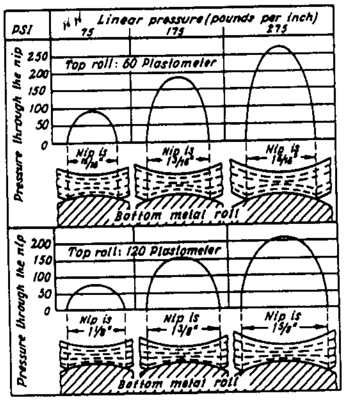

Figure #10
Effects of Nip Roll Hardness on Nip Pressure Distribution

As seen in Figure #11, the nip width will influence adhesion and all of its associated changes in appearance and barrier properties.

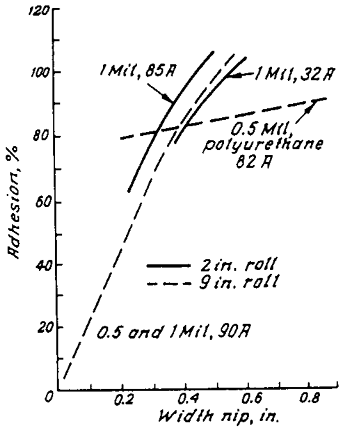

Figure #11
Nip Width vs. Adhesion

EQUIPMENT: ADJUSTMENTS

Five adjustments in the solidification region are important:

1.) Nip Pressure
2.) Nip Gap
3.) Stripper Roll Position
4.) Die Position
5.) Changeable Substrate Approach

Figure #12 depicts the number of pin holes in a coated paper as a function of nip pressure. As shown, nip pressure lead (die position), pretreatment and surface hardness will influence pinholes. Our experience shows these variables will also influence bubbles and voids when extruding on to a plastic substrate for barrier purposes.

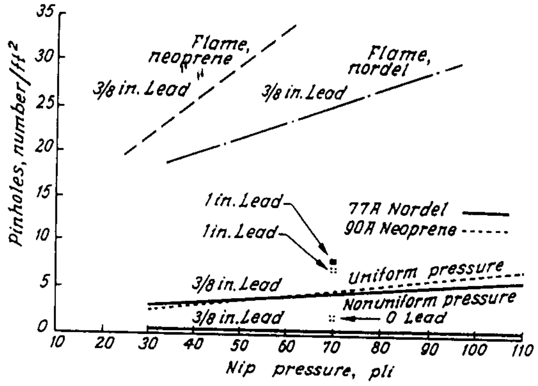

Figure #12
Effect of Nip Pressure

Figure #13 shows the relationship of nip pressure to adhesion with corresponding effects of melt temperature, roll size and hardness. Adhesion will increase with increased pressure. Note these figures are based on pli and not guage pressure. One must calculate pli from guage pressure through the mechanics of their own system.

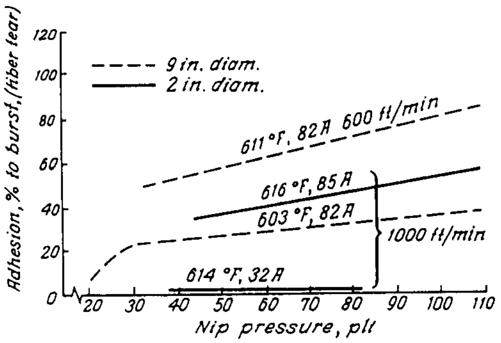

Figure #13
Nip Pressure vs. Adhesion

The nip gap or width of impression of the nip roll onto the chill roll will also influence adhesion and pinholes. As seen in Figures #10 and #11, higher contact will provide better adhesion. Another process variable related to nip width is coating weight vs substrate thickness. This factor is especially important when coating on plastic where shrinkage can cause wrinkles and curl. The general rule is the thinner the substrate, the narrower the width of the nip desired. If adhesion can be obtained, then one would want to extend this even to zero or a negative gap.

Stripper roll position and adjustments are also very important. Figure #14 shows the relationship of film thickness to cooling time and travel around the chill roll. As an example, a chill roll of 60°F, melt of 600° and stripping temperature of 105°F will give a value of .083 for Y. If we are using a constant diameter roll, one sees the time on the roll will have to vary if we are to keep strip temperature the same. This variation can be compensated for by speed or stripper roll position. Two other factors will influence stripper roll position; (1.) release of film and (2.) film stiffness. The angle of release needs to be adjusted to provide a uniform, straight release from the roll. For thin, flexible substrates, the roll may even have to be brought into contact with the chill roll or be driven to aid release.

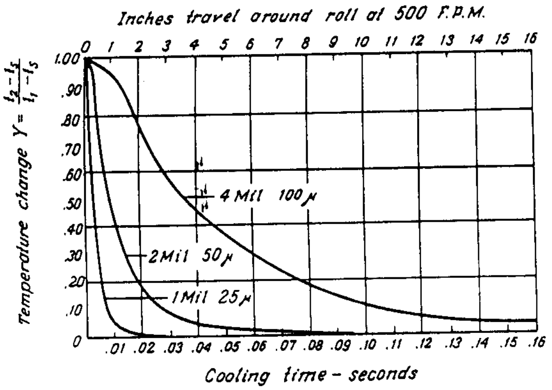

Figure #14
Effect of Thickness on Chilling

Die lead and die height need to be adjusted to aide processing. Figure #15 shows the interrelation of lead to adhesion. As shown, this is a bell curve which will vary based on resin, equipment and resin thickness. Die height increase will also act as a melt temperature increase. A one inch height adjustment will act as if the melt were raised 2 to 5°F (depending on resin). Thus die height and die lead can be adjusted to help increase adhesion without changing any other variable. One caution for heavier coatings, one may want to move the die towards the nip to avoid voids and bubbles.

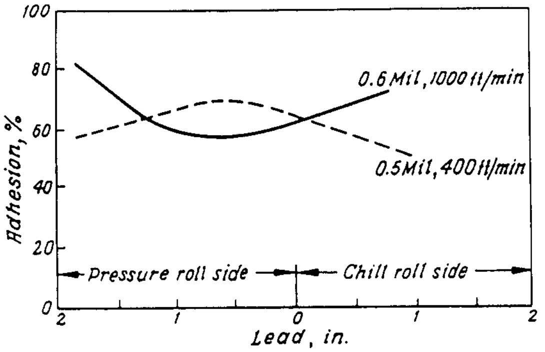

Figure 15
Lead vs. Adhesion

A valuable tool when running different substrates on the same line is variable substrate angle into the nip roll. For more flexible substrates and films that contract when coated, our experience shows the film should be raised, giving a high angle of approach and low wrap angle on the nip. For stiffer materials, the opposite holds true. Varying roll diameter and nip width can also help this situation. By varying these angles and wrap, we have found we can minimize wrinkles and aid the tracking of the films thru the line.

PROCESS VARIABLES

Based on investigated literature, discussion with equipment manufacturers and other converters and our own experience, Table #IV was compiled. This chart represents a sequencing guide for general problems on our line. Each area of concern is prioritized to the appropriate process variable. Some priorities are listed as to the speed of adjustment, as well as the importance of the variable. Different resin systems and equipment may change the priorities for a given system, but we believe this guide will work and aide converters.

MATERIAL PARAMETERS

Obviously, if all the solidification zone variables are set properly, the highest quality product at maximum speed is expected. However, resin choice (density, amorphous, crystaline, coating weight) and substrate type (stiffness, substrate contraction) will be significant factors in final product performance, especially curl. Changes in density from the melt to the solid builds up stresses because the substrate usually has less tendency to shrink. Thus, the coating is like a stretched rubber band attached to the substrate and released upon exiting the roll. Competing factors, such as higher melt temperature for adhesion, may increase curl, while we may negate this by faster quench or expending the substrate before coating. Understanding and knowledge of the resin and the mechanisms occurring in these few short seconds (solidification) can eliminate or create problems.

Knowledge of the resin's crystalline or amorphorous behavior will help determine quenching rates. Crystallization is not only a function of temperature and time but also a function of orientation and stress, which can be introduced during solidification. In essence, not all problems can be solved by adjusting process parameters, but one knows with good knowledge, we can help build a poorly designed product, but more importantly, without proper knowledge, we can ruin a well conceived product being processed on good equipment.

CONCLUSION

The solidification region is a complex area of extrusion coating. Due to the speeds and distance traveled, a great many phenomena can occur in a short time. This paper has highlighted the main points and presented a troubleshooting guide that is being used on our system and for our products. Experimentation and arrangement of this guide to any other situation is believed to act as a good guide to reduce scrap while increasing quality and productivity.

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TABLE IV PROBLEM VARIABLES: Solidification Variables

PROCESS VARIANCE	PRIMARY INFLUENCE	ADHESION	APPEARANCE	PINHOLES/ BARRIER	WRINKLES	CURTAIN STABILITY	GUAGE CONTROL	MECHANICAL PROPERTIES
I. CURTAIN POSITION (LEAD)	• Lead towards nip roll will help in adhesion, but care and experimentation needed. • Lead can help in avoiding voids and surface bubbles	3. Lead in of curtain can maximize adhesion (Unprodicable and needs to be explored on each system)			7. • Thick coating on thin substrate may cause contraction, if lead is to far towards nip roll			
II. CURTAIN HEIGHT	• Curtain height adds oxidation time to curtain. Each 1" of height acts as if melt were raise 2 to 5° F • Low curtain will aid curtain problems	2. Height relatable to oxidation - acts as increased melt temperature				3. • Curtain breaks, edge tears, draw resonance lower die • High neck-in lower die		7 • High curtain can aid oxidation of the coating • Orientation due to layer stretch distance
III. NIP PRESSURE	• Nip pressure increase aids adhesion • High nip pressure can create pinholes in paper coatings • Low nip pressure can aid bubbles and voids in plastic substrate coatings	4. Higher pressure produces higher adhesion	4. • More pressure will reflect surface of substrate (Fiber or rough surface)	1. • Coating on paper, fibers show through • Plastic film, bubbles and voids	4. • Uneven pressure on nip • Uneven pressure distribution		2. • Uneven pressure (clam distribution) creates distribution of melt • Side to side pressure variation will show gauge control	
IV. NIP WIDTH	• Wider width leads to better adhesion • Appearance and pinholes/barrier properties may be influenced • Shrinkage of base film needs to be compensated for by nip width • Flexible films need smaller widths	7. Larger gaps produce higher adhesion		4. • Longer nip width changes pressure distribution • Flexible films that contract need to have minimum nip width	3. • Thinner the gauge, narrower the nip width • Substrate contracting, allow for nip gap			
V. CHILL ROLL TEMPERATURE	• Quick solidification vs slower solidification will influence crystallinity, density • Curl, optical properties, mechanical properties • Strippability, release of coating • Sweating, moisture leads to defects	9. • Too fast a quench will hinder adhesion • Too slow could cause poor strippability	5. • Quenching will determine crystal structure	9. • Inadequate cooling will disturb coating integrity • Crystalline vs amorphous regions is a function of quench rate	5. • Uneven temperature can create uneven pull away from chill roll			2. • Premature cooling effects adhesion • Rate of cooling can influence crystallinity • Inadequate cooling causes stripping problems
VI. LINE SPEED	• High speeds require more cooling capacity • Higher flow rate polymer can influence curtain • Recovery time of chill roll must be considered	10. Higher speed at constant temperature and pretreat will drop adhesion	3. • Improper cooling • Tension too high to control web • Draw elongation is extended			4. • Slow line and extruder in ratio to help curtain stability • Reduce rate of stretch elongation		
VII. STRIPPER ROLL POSITION	• Position determines wrap angle on chill roll • Stripper roll aides even pull off • Flexible films may need aide by stripper roll for tension control	11. Poor angle or too hot a strip off temperature will disturb coating	7. Poor angle or too hot strip temperature will disturb coating	8. • Poor angle or too hot strip temperature will disturb coating	2. • Thin gauges need closer stripper roll • Hard pull - move roll closer			6 • Hot pull away will hurt coating integrity • Flexible films may need assistance from stripper roll • Poor pullaway angle will pull on coating (gauging roll)
VIII. PRETREAT FOR ADHESION	• Corona, ozone, flame greatly aid adhesion • Flame helps reduce pinholes or paper • High pretreat can change adhesion characteristic during solidification	1. Surface treatment needs to overcome wetability especially a plastic		3. • Flame treating on fibrous paper • Corona treat on plastic. Adhesion vs chilling cycle	6. Higher adhesion in nip - more contraction and curl, warpage			4. • Low treatment leads to poor adhesion, heat sealability • Over treatment changes adhesion characteristics during cooling
IX. DIE GAP/ DRAW RATIO	• Too small a gap can lead to melt fracture • High draw ratio can lead to curtain instability • High draw ratio leads to high polymer orientation		2. • Too small a gap can create melt fracture • Higher draw ratios will give higher orientation and corresponding optics	6. • Increase polymer thickness to cover problem • Melt fracture causing uneven coating		1. • Edge bead Size • Neck down due to too high a ratio • Draw resonance • Curtain breaks	4. • Too small a gap can cause melt fracture and uneven coating • Draw resonance creates wavy coating	3. • Draw ratio corresponds to orientation and corresponding mechanical changes • Die gap too small causes melt fracture
X. POLYMER TEMPERATURE	• Proper melt temperature and uniform flow is imperative to good curtain condition • Adhesion • Improper flow leads to poor guage • Degradation of polymer	5. Higher temperature provides more adhesion	1. • High temperature causes melt flow problems - Melt fractures - Curtain resonance • Crystalline rate	5. • Polymer degradation and over oxidation cause polymer problems • Low temperature leads to adhesion problems		2. • Lowering polymer melt increases viscosity and curtain stability (edge tear, resonance, curtain breaks)	1. • Temperature variations can lead to guage bands • Temperature too high can create melt and flow instability	1. • Polymer degradation leads to poor physicals • Oxidation due to high temperature • Poor heat sealability
XI. SUBSTRATE, CHARACTERISTIC	• Rough and uneven substrates create caliper and pinhole problems • Stiff substrates vs thin flexible substrates require different lead in angles to web	8. Surface characteristics determine amount of treatment or type of resin required	6. Rough surfaces will telescope to coating surface	2. • Rough fiberous surface • Surface characteristics relative to adhesion	1. Thickness vs. attack angle to nip - need to be adjusted to change wrap angle on nip roll		3. • Uneven substrate	3. • Rough surfaces create surface problems • Substrate characteristics need to be considered so inline tensions do not over stress • Substrate surface will influence adhesion
XII. SUBSTRATE PREHEAT	• Aids in Adhesion • Stripping temperature will be changed • Different cooling characteristics will influence curl with some substrates	6. Substrate preheat usually promotes better adhesion		7. • Adhesion vs pinhole barrier properties change with preheat • Preheat helps contract of plastic films				8. • Helps adhesion • Eases curl problems in some substrates