

Polypropylene Crystallization Effects on Film Forming

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ABSTRACT

Polypropylene (PP) is finding many new uses in film applications due to its finished product properties (e.g. clarity, stiffness, etc.). The crystallization characteristics during and after processing can cause problems in web handling and roll formation. This study investigates the effects of quenching temperatures, tensions, and air gap on finished product properties for a film grade PP homopolymer and random copolymer. Increases in melt temperature and processing tension are found to aggravate the web handling and roll formation difficulties.

KEYWORDS

Polypropylene, Cast Film, Crystallization, Quenching, Web Handling, Homopolymer, Random Copolymer

INTRODUCTION

Polypropylene (PP) offers the film converter many enhanced properties over low density polyethylene (LDPE) including high tensile strength and elongation at break, good clarity and gloss with low haze, as well as lower permeability and good chemical resistance. These properties coupled with low prices and ample capacity¹ have increased the use of PP film in many packaging and specialty applications. Although the PP films can be produced on conventional cast film equipment the processing characteristics challenge the converter.

The crystallization kinetics and morphology of PP have been widely studied and documented^{2, 3}. Due to the slower crystallization rate of PP compared to LDPE, processing

conditions will have a dramatic impact on final crystallinity and corresponding film properties. Increases in density during aging due to secondary crystallization also affect final film properties. Schaefer reported an increase in film modulus and a decrease in impact strength and COF with increased crystallinity⁴. Goins, Sun and Miller demonstrated that an increase in MFR yields a decrease in tensile strength and haze⁵. They also conclude melt temperature has an equivalent effect. Other factors including chill roll temperature, line speed, and film thickness had only a minor impact on final film properties. Steward and Bradley have shown how MFR increases due to processing conditions especially temperature⁶. These and other studies provide much guidance for tailoring the process to yield the desired film characteristics but little has been published to aid the processor in avoiding processing defects. Alsdorf identifies "knots" or mounds in the wound roll and camber, the tendency of the wound film to track to one side when unwound, as common undesirable features of cast PP film⁷. The mounds initiate around a melt defect or wound in contaminant and may grow due to processing conditions. Typically the processor ages the film to allow further crystallization and densification of the film before rewinding and slitting to the final roll configuration.

The object of this study is to evaluate the effects of quench conditions, web tension, and air gap (i.e. distance from die exit to chill roll contact) on the shrinkage or densification of the film. The conventional belief is that the long term increase in density increases the wound roll defects (i.e. "knots", mounds, etc.) hence the practice of aging before final winding. This density change, although typically less than 1% of the final density⁸, is inherent in PP and can not be stopped by the processor. This study will show that processing conditions do have an influence on overall densification as measured by shrinkage. The interactions with several other final film properties is reported, although heat seal and barrier properties were not investigated.

EXPERIMENTAL

Equipment

The equipment used for these experiments was an 88.9 mm (3.5") diameter, nominal 30:1 L/D extruder designed and manufactured by Black Clawson. This extruder was equipped with six sets of barrel heater/coolers configured as six control zones. These zones were controlled individually with their own thermocouple and proportional-integral-derivative (PID) loop controller through the supervisory control system. The first 20 mm (8 inch) of the extruder barrel were covered by a water-cooled

feed cylinder. The extruder was equipped with a 100 HP, 480 volt, 1750 RPM base speed motor rated at a maximum amperage of 170 amps. The 14:1 ratio gear box supplied a maximum screw speed of 125 RPM. The feed screw was a long melt barrier design with dispersive and distributive mixers. The extruder head was fitted with a flange mounted screen changer/restrictor valve. An offset transfer line conveyed the polymer from the valve to the combining adapter. The transfer line was fitted with a six element static mixer prior to the combining adapter. The five layer co-extrusion combining adapter was arranged for single supply single layer extrusion. The valve, transfer line, and combining adapter were arranged for nine (9) heat only temperature control zones. An internally deckled T-slot die was used for film formation. The 1200 mm (47.2 inch) slot with pre-set 0.75 mm (0.030 inch) gap was provided with seven (7) zones of heat only temperature control.

Two 69 Mpa (10,000 psi) pressure transducers were positioned on the extrusion system one at the head of the extruder prior to the screen changer/valve and one in the transfer line before the combining adapter. A variable depth thermocouple was located in the transfer line 216 mm (8.5 inch) from the exit of the screen changer/valve.

The haul-off equipment for these experiments consisted of a combination casting/coating station with pull rolls, a thickness gauging station, a corona treater/pull roll station, and a robotic winder/roll changer. The 457 mm (18 inch) diameter matte finished chill roll was of a double shell spiral fluted design supplied with 757 lpm (200 gpm) of cooling water. The water temperature was controlled by an individual PID loop controller. A vacuum box was fitted between the die and chill roll to provide a uniform laydown and pinning of the extrudate to the roll. Static pinners were applied to the web edges.

The chill roll, treater/pull roll, in-feed pull roll at the roll changer, and winder spindle were each independently driven and controlled by a DC drive. The casting unit pull roll was mechanically driven through a PIV from the chill roll. All process parameters were measured, monitored, controlled and recorded through the supervisory control system.

Procedure

Two polypropylene resins were used for this evaluation.

- 1) A nominal 8.0 MFR random copolymer (RCP), nominal 3.7% ethylene (melting point 142 °C (288 °F))
- 2) A nominal 8.8 MFR homopolymer (HPP)

Each polymer was provided with the same medium level of slip and antiblock additives. Slip additives (typically

erucamides) improve surface characteristics for subsequent web handling while antiblocks (typically silica) roughen the film surface preventing the intimate contact between wound layers that tend to bond with amorphous films⁹.

Quench conditions (melt temperature and chill roll temperature), web tension and air gap were studied. Table 1 lists the nominal high and low values used. (Note that when subsequent tables and discussions refer to low quench that is low melt temperature and high chill roll temperature, similarly high quench refers to high melt temperature and low chill roll temperature.) The extruder was operated at nominally 90 RPM making 2 mil film at 39.6 inpm (130 fpm). Extruder settings were adjusted for each resin to obtain a uniform high quality melt. Temperature variation with respect to position and time was less than 15 °F for all runs. Discharge pressure variations were less than 2% total pressure.

The properties evaluated were shrinkage, modulus, tensile strength, elongation, haze, gloss, clarity, and impact. Table 2 lists these properties and the test procedure used. The evaluation of shrinkage was accomplished by cutting random samples of film from the finished roll immediately after wind up. Samples were stored together and re measured after 72 hours. Average percent reduction in length, machine direction (MD) and trans machine direction (TD), was recorded.

TABLE 1

PARAMETER	LOW VALUE	HIGH VALUE
Melt Temp	272 °C (522 °F)	291 °C (556 °F)
Chill Roll Temp	32 °C (90 °F)	16 °C (60 °F)
Web Tension	35.6 N (8 lbf)	89.0 N (20 lbf)
Air Gap	2.5 cm (1 inch)	7.6 cm (3 inch)

TABLE 2

PROPERTY	ABBREVIATION	PROCEDURE
Shrinkage-MD	S-MD	Black Clawson
Shrinkage-TD	S-TD	Black Clawson
Modulus-MD	M-MD	ASTM-D882
Modulus-TD	M-TD	ASTM-D882
Tensile-MD	TB-MD	ASTM-D882
Tensile-TD	TB-TD	ASTM-D882
Elongation-MD	EB-MD	ASTM-D882
Elongation-TD	EB-TD	ASTM-D882
Haze	H	ASTM-D523
Gloss 45°	G-45	ASTM-D523
Gloss 60°	G-60	ASTM-D523
Clarity	C	ASTM-D523
Spencer Impact	SI	ASTM-D3420

TABLE 3
Random Copolymer Results

RUN	QUENCH	AIR GAP	TENSION	S-MD (%)	S-TD (%)	M-MD (psi)	M-TD (psi)	SI (in-lb/mil)
4	LO	HI	HI	-0.59	-0.25	98900	99600	9
5	HI	HI	HI	-0.96	-0.05	99300	97800	16*
6	HI	HI	LO	-0.64	-0.29	92600	101600	12.2
8	HI	LO	LO	-0.85	-0.2	95600	94600	16*
9	LO	LO	LO	-0.75	-0.29	98700	104300	10.3
10	LO	LO	HI	-0.48	-0.2	95600	97000	6.8

RUN	TB-MD (psi)	TB-TD (psi)	EB-MD (%)	EB-TD (%)	H (%)	G-45	G-60	C (%)
4	4850	4430	788	777	3.7	74	126	69
5	4600	4590	747	790	1.0	90	138	74
6	4630	4550	769	769	0.9	91	138	74
8	4660	4500	754	775	1.1	93	139	71
9	4770	4570	728	778	2.5	83	127	70
10	4930	4560	765	800	2.6	84	131	68

*Sample did not break at maximum load of 16 in-lb/mil.

Results and Discussion

During these experiments we did not observe any roll "knots" or mounds. Our experience has shown that the source of these problems are melt defects and contaminants. These can be minimized with a three step approach. First a high quality melt must be insured through a screw design that offers dispersive as well as distributive mixing. Extruder operating conditions must then be optimized for the application. Second the chill roll must be kept free of contaminants such as erucamides which can bloom to the film surface and plate out on the roll. A rubber covered herringbone grooved roll nipped against the chill roll prior to strip off is an effective means of insuring that the plate out is carried away with the film in a uniform manner. A wiper roll in the recovery zone of the chill roll is also effective as is frequent cleaning of the roll. Third a properly applied vacuum box in addition to providing a uniform quench reduces the entrainment of air and volatiles from the molten polymer between the film and the chill roll. Each of these three techniques were applied during the experiments.

Tables 3 & 4 summarize the results of the experiment for the RCP and HPP respectively. It can be seen that the effects of melt temperature as reported by Schael and Goins is confirmed here. With increasing melt temperature, tensile strength is reduced and haze values are lowered. Just as melt temperature appears to have the greatest effect on physical properties of the final film it also showed the most significant and consistent effect on shrinkage. Figure 1

shows the change in MD shrinkage for four pairs of runs as a result of quench condition changes only. Tension and air gap have a complex interaction on this shrinkage. Figure 2 compares runs showing the shrinkage in relation to tension change. Three of these indicate an increase of shrinkage due to an increase in tension. The RCP run at low quench and low air gap yielded a decrease in shrinkage. The varied results of this run may be due to increased orientation during solidification (low melt temperature, high rate of extension) and bears further investigation.

Figure 3 displays the correlation between air gap and optical properties in comparison to temperature effects averaged for both polymers. Temperature has the more significant effect however a similar effect is accomplished with an air gap change. This could allow the converter to adjust temperature for improved handling while adjusting air gap to compensate for optic requirements.

This study has attempted to show correlations between operating parameters and shrinkage as a measure of crystallinity in PP films. The level of crystallinity will impact the wound roll stability after forming and may necessitate aging and rewinding. It has been shown that low tension and low quench temperatures will reduce the subsequent shrinkage. Loss of optical properties due to low temperatures may be offset by reducing the air gap. The film forming process is a complex interaction of many variables which can be balanced to provide the desired mix of physical properties and processibility. The slow rate of

TABLE 4
Homopolymer Results

RUN	QUENCH	AIR-GAP	TENSION	S-MD (%)	S-TD (%)	M-MD (psi)	M-TD (psi)	SI (in-lb/mil)
18	LO	HI	HI	-0.32	-0.05	132700	137500	2.5
17	HI	HI	HI	-0.43	-0.2	119200	114800	2.4
16	HI	HI	LO	-0.27	-0.25	117500	119700	3.1
14	HI	LO	LO	-0.16	-0.15	113900	113100	3.2
12	LO	LO	LO	-0.16	-0.15	113700	107700	3.0
13	LO	LO	HI	-0.21	-0.0	115900	113200	3.3

RUN	TB-MD (psi)	TB-TD (psi)	EB-MD (%)	EB-TD (%)	H (%)	G-45	G-60	C (%)
18	5510	5630	754	816	18.5	50	76	38
17	5540	5330	774	786	3.6	79	123	72
16	5550	5800	779	818	3.6	76	113	71
14	6010	5570	767	794	2.3	82	129	73
12	5590	5360	754	844	7.6	67	108	62
13	6600	5500	787	813	7.6	67	105	63

crystallization of PP requires the converter to regulate the process for not only final properties but on machine handling as well.

Future studies to further analyze the interactions of air gap and tension as well as web pinning mechanisms and line speed are planned.

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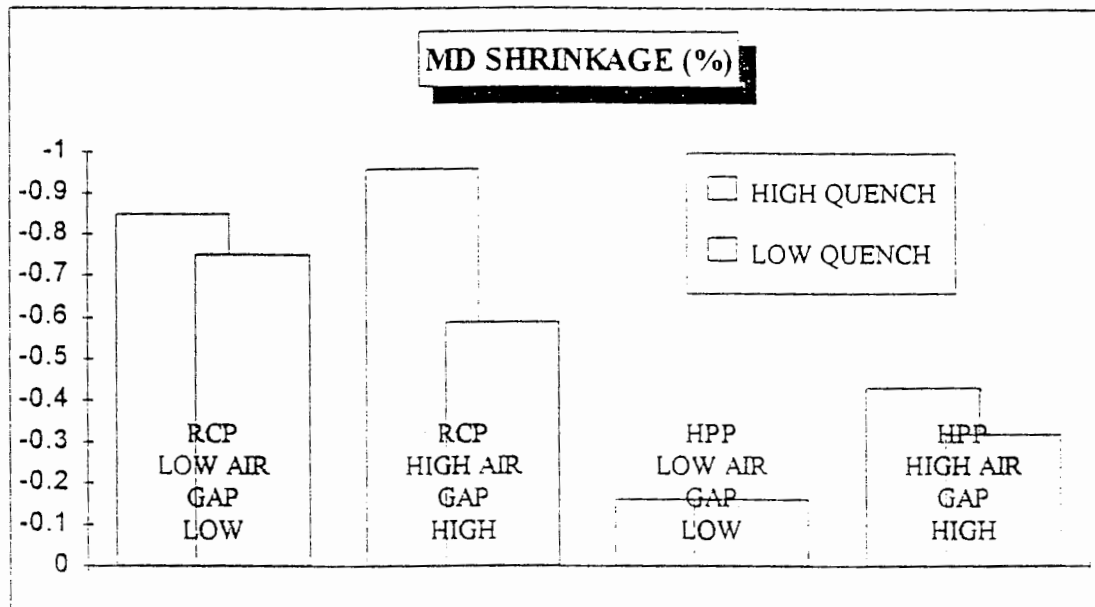


Figure 1

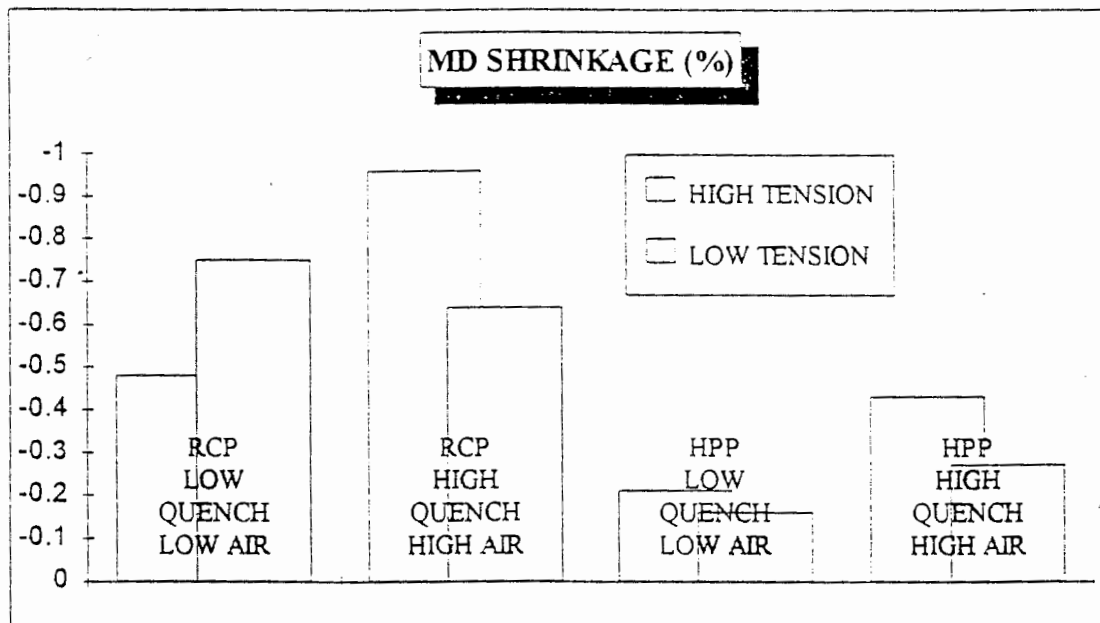


Figure 2

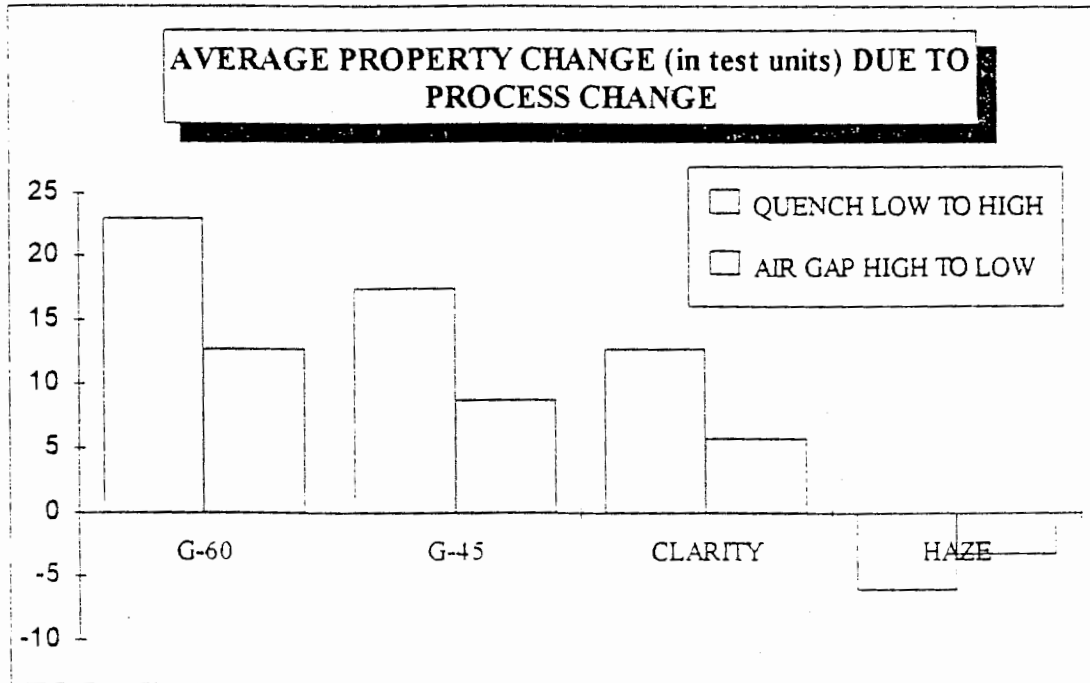


Figure 3