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BLOWN FILM EXTRUSION OF CARILON EP POLYMER

by

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J.D. van Zomeren



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BLOWN FILM EXTRUSION OF CARILON EP POLYMER

(October 1994 - December 1995)

by

J.D. van Zomeren

Approved by: J.A. Verhave

SUMMARY

This report summarizes the results of film blowing trials carried out with CARILON EP polymer. After defining the operating window, CARILON polymer was successfully processed into a homogeneous film. Smooth, flat and wrinkle free films were produced having promising properties.

Based on the work done it is strongly believed that the combination - improved melt stability/properly designed equipment - will lead to the production of high quality film. In-house basic expertise for future/potential customer trials is now available.

December, 1995.

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BLOWN FILM EXTRUSION OF CARILON EP POLYMER

1 INTRODUCTION

CARILON EP polymer has promising market potential as an engineering thermoplastic (ETP). A variety of high performing components (e.g. gears, clips, snap fits,..) have already been produced successfully by injection moulding ¹.

Although the development of extrusion activities has a lower priority over the injection moulding one, previous experimental work has shown that, despite some problems, it would be worthwhile to explore that field in order to establish the necessary basic expertise for (future) customer trials. The limiting factors appear, to some extend, to be the melt stability of the polymer but more important an evident shortage of expertise in extruding Carilon polymers and other ETP's.

Anticipating on both, improved polymer properties and extrusion expertise a screening study was initiated to determined the possibilities to produce by extrusion pipes, films, profiles, rods,... from CARILON polymers.

A rather severe route of producing thin wall CARILON items, blown film extrusion has been selected for this screening work. The programme focused on the following:

- Determine "friendly" extrusion conditions suitable for melting, homogenising and transporting the polymer from the hopper to the die head.
- Investigate the extrudability of CARILON polymer by measuring the effects of variable extrusion parameters e.g. screw speed, throughput, melt temperature, melt pressure, etc.
- Investigate the possibility to convert a CARILON polymer into a thin wall film of various thicknesses, using two different die gap widths.
- Examine the effect of die gap width and film thickness on the mechanical and optical film properties.

2 EXPERIMENTAL

2.1 Materials

The CARILON polymer selected was the medium viscosity CARILON EP type (Lot 93.002) with an LVN of 1.95 and a melting point of 220°C.

Prior to extrusion melt strength measurements were carried out on Rheotens equipment at CRCSL². This melt strength evaluation indicate that CARILON polymers have a melt strength similar to a 1 MFR LLDPE. This suggested that the behaviour of CARILON polymer, in the film blowing technic, should be comparable.

2.2 Extrusion Procedure

The CRCSL 30 mm screw diameter, Plastic Machinebau (PM) laboratory film blowing line has been used for this work. The machine characteristics are given in Table 1. The equipment comprises an extruder fitted with: a breaker plate, an adapter, a central feed film die (two spiders), a film cooling unit, a take off and a wind-up unit. Pressure gauges are installed before and after the breaker plate, the first one indicating the extruder pressure the latter the die pressure.

In the absence of any other information, the extruder settings were first set on the basis of expertise with other polymers. In a stepwise approach, several corrective actions were taken to achieve stable extrusion conditions, in combination with a visual estimation of the film quality. After each individual adjustment, the bubble was allowed to stabilise completely.

The main actions taken were:

- adjustment of the temperature profile
- manual adjustment of the guiding rods and boards.
- setting the nip roll at a desired pressure.
- decreasing the blow up ratio, stepwise from 2.0 to 1.9 and then 1.8. Improvement of the bubble stability was clearly noticeable on decreasing blow up ratio.
- adjusting the air cooling volume at a desired level. Noticeable was the fast set up (crystallisation speed) of the CARILON polymer. Cooling air volume was reduced by half of what is normally used for polyolefins.

Based on the above mentioned experiments the extrusion parameters were installed and used as standard settings for all extrusion trials. A description of these settings are given in Table 2.

2.3 Film Testing

Film properties were determined as recommended by the following ASTM methods:

•	Haze	ASTM D1003-61 Procedure A
•	Gloss 45° Measured, in the machine direction of the film.	ASTM D2457-70
•	Falling weight impact strength(F50) Method A stair case	ASTM D1709-75
•	Elmendorf tear strength	ASTM D1922-67
■ .	Tensile properties Method A, initial grip separation 50 mm, rate of grip separation 500 mm/min.	ASTM D882-79
•	Young modules Initial grip separation 100 mm, rate of grip separation 10 mm/min.	ASTM D882-79
•	Puncture resistance Cross head speed 250 mm/min.	ASTM D4649

Where appropriate, these properties have been measured both in machine (MD) and transverse direction (TD).

3 RESULTS

3.1 Extrusion Behaviour

The extrusion behaviour of CARILON polymer over the whole screw speed range was investigated. The following extrusion parameters were determined: throughput (Figure 1), melt temperature (Figure 2), extruder and die pressure (Figures 3 and 4), axial pressure (Figure 5) and motor current development (Figure 6). All experiments were carried out with two different die gaps; 0.75 and 1 mm. The results of these measurements are summarized in Tables 3 and 4.

3.2 Film Blowing

The production of 25, 30 and 45 micron films was carried out with both die gap widths. The optimum extrusion conditions were determined by visually assessment, bubble stability, surface appearance and homogeneity of the film. This appeared to occur at a screw speed of 100 rpm. Subsequently at this condition films of different thicknesses were produced by varying the take-off speed. The extrusion conditions during the film manufacturing are given in Tables 5 and 6.

3.3 Film Properties

Despite the screening character of the trial, excellent films were produced having nice and smooth surface. This also implies that their is still room for further optimisation of the properties measured. The preliminary results are summarized in Tables 7 and 8 and Figures 7-18. A detailed discussion is given below.

4 DISCUSSION

4.1 **Processability**

4.1.1 Extrusion

Figure 1 shows a throughput curve versus screw speed using a die gap of 0.75 mm and 1.00 mm, respectively. Within the experimental error the throughput increases linearly with the screw speed range of 40 to 140 rpm. A maximum throughput of about 14 kg/h was obtained. The die gap width has no significant effect on the throughput.

The influence of the melt temperature against the screw speed is plotted in Figure 2. A significant melt temperature increase is noticed while raising the screw speed. The recorded melt temperature values ranged from 234°C to 254°C. The temperature increase is most likely due to the higher frictional heat generated at the higher screw speeds. However, the temperature increase did not appear to be detrimental for the process.

Extruder and die pressure development as a function of die gap width measured over a screw speed range from 40 to 140 rpm show a significant variation. The die and extruder pressure increase with the screw speed and appear to be die gap width dependent. The smaller the gap width the higher the pressure. This is expected on the basis of the higher resistance of the die with the smallest gap (see Figures 3 and 4).

The development of the axial load versus screw speed as a function of die gap width is plotted in Figure 5. The die gap width has a significant influence on the axial load. As expected with higher loads for the smaller gap.

The electrical power consumption versus screw speed and die gap are plotted in Figure 6. Although, some differences in motor current are noticed when using a die gap of 0.75 mm or 1.00 mm, it does not seem critical. The motor current increases with the screw speed. The somewhat higher values obtained for the 0.75 mm gap at 60 rpm and 80 rpm are thought to be due to experimental fluctuations.

4.1.2 Film blowing

For both die gaps film quality became acceptable at some 100 rpm. This processing condition was selected to manufacture film samples. Film blowing of CARILON polymer went smoothly and a good balloon stability was obtained. The balloon shape can be compared to that of LDPE (short neck). A blow-up ratio of 1.8 appeared to be optimal in terms of balloon stability. During the trials very few gel particles were observed. These particles had no effect on the bubble formation. Please note that film blowing with CARILON R1000 was totally impossible because of the extremely low melt strength.

The production of wrinkle free, flat films was possible. However, compared to e.g. LDPE, CARILON polymer appeared susceptible to draughty environment, main cause low melt strength of CARILON. Hence, special measures had to be taken to avoid any disturbance of the bubble. The film thickness variation of all produced films, measured at the circumference of the film can be judged as excellent. A standard deviation of around 2.5 micron for all tested film samples was achieved. Hence, the processability of CARILON film is good and to some extend, comparable with these of polyolefins on the same PM equipment.

Extrusion/film blowing of CARILON polymer was possible throughout a period of more than <u>10 hours without any complication</u>. Moreover, after purging with a high viscosity HDPE and equipment shut down for 24 hours an easy machine start-up was made.

After dismantling and manual cleaning of the die and extruder parts, the following observations were made: all <u>hard chromium</u> parts of the equipment including extruder barrel as well as the die head were easy to clean and no deposit of degraded polymer was detected on these parts. An exception has to be made for the connection between extruder and die where the breaker plate is situated. This part of the equipment is made of a <u>non</u> <u>chromium</u> plated steel quality and CARILON polymer was found to be accumulated and degraded on it. Cleaning of this specific machine part was necessary.

4.2 Film Properties

4.2.1 Optical film properties Haze

Figure 7 shows the effect of the die gap width and film thickness on the haziness. A first impression is that CARILON polymer does not score very well, rather hazy films are obtained. The film thickness appears to have a significant influence on the haze properties, the haze increases by almost 85% when the film thickness increases from 25 micron to 45 micron. The die gap width has virtually no affect on the haze properties.

Surface gloss

Figure 8 displays the effect of surface gloss as function of film thickness for a die gap width of 0.75 mm. Nice and glossy films were produced. However, a significant contrast between the surface gloss of the outer and inner side of the film is observed. On average, 30% higher gloss values are achieved for the inside surface.

This is probably due to the difference in cooling efficiency between the inside and outside face of the bubble. A similar picture is obtained when using a die gap width of 1.00 mm. The film thickness has only a marginal effect on the gloss.

The rather poor optical properties of CARILON films appear to be mainly due to its fast crystallization (fast set-up) but also to the presence of melt surface irregularities which freeze upon cooling. A way of achieving a smoother surface will be to operate at higher melt temperature, currently limited due to the low melt stability.

4.2.2 Mechanical properties

Dart impact

The dart impact strength as a function of film thickness for both die gap widths are graphically displayed in Figure 10. As expected the dart impact strength is high and shows a strong dependence on the film thickness. The dart impact strength increases by about 60% when film thickness increases from 25 micron to 45 micron. The die gap width has a small effect on the dart strength, leading to some preference for the larger gap.

Elmendorf tear strength

The Elmendorf tear strength properties as a function of film thickness for both die gap widths are shown in Figures 11 and 12. The values recorded are lower than expected with regard to the dart impact results. A priori, it indicates that CARILON films present a certain tear/crack sensitivity. The disagreement between the MD and TD values is also noticed and suggests that an unbalanced molecular orientation has taken place upon blowing.

Both MD and TD Elmendorf tear strength values increase with increasing film thickness. No real difference is noted when working with the 0.75 mm or 1.00 mm die gap width.

Tensile properties

Results of the tensile tests are graphically presented in Figures 13-16. The yield strength, the ultimate strength and the ultimate elongation as a function of film thickness for both die gap widths are displayed.

As expected the yield strength for both MD and TD are not affected by the die gap width and the film thickness. Values as high as 60 MPa were determined. The ultimate strength seems to be unaffected by the die gap width, however, the results in MD show a slight film thickness dependence.

Tables 7 and 8 summarise the ultimate stress values. As indicated, only a limited amount of samples could be tested for their ultimate stress values in TD. This was especially the case when the wider die gap was used (Figures 13 and 14). The disagreement between MD and TD values has already been noticed for the Elmendorf tear strength. This discrepancy has been attributed to an unbalanced molecular orientation upon film blowing. Notwithstanding this, Figure 13 indicates that the ultimate stress in TD seems to increase with increasing film thicknesses.

The results indicate that the die gap width has only a marginal effect on the ultimate elongation when measured in MD. Values in MD increase by 50% when the film thickness increases from 25 micron to 45 micron. Measurements in TD were not detectable in three cases, a brittle like failure was recorded in these cases.

Young modulus

Figures 17 and 18 display the young modulus values as a function of the die gap width and the film thickness. Very good results were obtained. On average both MD and TD show values approaching 1600 MPa. The 45 micron film presents a slightly lower modulus. The 1 mm die gap appears to have a beneficial effect, a 10% increase is recorded. The somewhat higher value achieved in TD may again be explained by a certain anisotropy in molecular orientation.

Puncture resistance of thin film

High puncture resistance properties were obtained. The results are displayed in Figures 19 and 20. The die gap width has only a marginal affect on the puncture resistance. On the contrary, as expected the film thickness has a strong influence on the puncture resistance. Both load at maximum and energy at break point increase significantly with the 45 micron film.

5 COMPARISON WITH OTHER POLYMERS

In order to position the Carilon film properties, a fast screening with LDPE and PP blown films has been made. The films selected have a thickness of 45 micron and a blow-up ratio of 1.8 (Table 9 and Figures 21-30).

CARILON films have a poor haze in comparison with LDPE and PP (Figure 21). This is probably due to the fast set-up (crystallisation speed) of CARILON polymers.

Although it is less pronounced, the surface gloss properties of CARILON film are lower than with LDPE. Noticeable is the significant difference between the outside and inside surface gloss of the CARILON film (Figure 22).

The dart impact strength of CARILON film is comparable with LDPE and scores three folded better than PP (Figure 23). The best tear strength properties were achieved for the LDPE film in both MD and TD. However, as indicated in Figure 24, CARILON polymer is characterised by higher tear strength values than PP (MD and TD). A higher LVN will improve this value.

CARILON appears to be superior to LDPE and PP for all tensile properties. CARILON films demonstrate a higher yield stress in both MD and TD (Figure 25), a higher ultimate stress at break (Figure 26) and on average a higher elongation at break (Figure 27). CARILON shows also higher young modulus than the other two materials. A 15% higher modulus than PP is recorded (Figure 28). As expected the puncture resistance results are superior for CARILON films (Figures 29 and 30).

Taking into account that the films are not manufactured in optimized conditions, it can be concluded that CARILON films potentially offer an attractive balance of properties. The apparent tear sensibility will be investigated in the future. A comparison with other engineering thermoplastics would give a clearer picture for a good positioning of the CARILON films.

6 CONCLUSIONS

This screening exercise on blown film extrusion of CARILON EP polymer shows that:

- After setting the extrusion temperature profile CARILON polymer could successfully be extruded on the PM laboratory film blowing equipment at CRCSL. The production of flat and wrinkle free film was possible without any major problems. Moreover, after two days of extrusion acceptable films were still produced.
- A processing window has been identified which allows production of CARILON film having a smooth surface.
- The two selected die gap widths have only a marginal effect on the extrusion behaviour as well as on the film properties, with on average a preference for the larger die gap.
- Mechanical properties increase with the film thicknesses, this is reversed for the optical properties.
- CARILON films yield interesting film properties, especially mechanical properties.
- Due to its crystallisation kinetics CARILON polymer has poor optical performances. However, surface gloss properties were acceptable.
- Comparison of CARILON, LDPE and PP blown film revealed superior mechanical properties for CARILON film.
- These results indicate that thin wall CARILON film can be successfully made and might be used in the packaging industry, especially where barrier and mechanical properties are requested.

7 FURTHER WORK

- Determine the orientation behaviour of CARILON film
- Modifications of the film blowing line:
 - replacing all non chromium plated machine parts by hard chromium.
 - modifying the connection between extruder and die, avoiding dead spots.
 - studying the effect of die land length on film properties as well on the extrusion behaviour.
 - studying the effects of different screw geometry on the extrusion performance, as well as on the film properties.
 - Evaluate higher LVN CARILON polymer which is expected to exhibit superior melt strength allowing to obtain films with still superior properties viz tear strength and puncture resistance.

Louvain-la-Neuve, November 1995.

References

- 1. A. Wakker, H.G. Kormelink, P. Verbeke and J.C.M. Jordaan, Kunststoffe 85 (1995) p.1056
- 2. J. De Clippeleir and P. Verbeke, "Rheological Characterisation of CARILON Polymers Rheotens and Capillary Rheology", LVGR.93.029.

Extruder size	:	30 mm
Screw L/D ratio	:	22
Compression ratio	:	3.5:1 (short compression)
		 11 D Feed section 2 D Compression 9 D Metering
Breaker plate	:	(standard)
Die diameter	:	50 mm
Die gap	:	0.75 and 1.00 mm
Land length	:	6.5 mm

Table 1: Characteristics of the PM film blowing line at CRCSL

Extruder	Breaker-plate	Neck	Blow-head	Die
[°C]	[°C]	[°C]	[°C]	[°C]
195 - 215 - 225	225	225	225	225

 Table 2: Standard extrusion temperature settings.

.

Screw speed	Throughput	Melt temperature	Extruder pressure	Die pressure	Axial load	Motor current
[rpm]	[kg/h]	[°C]	[bar]	[bar]	[kn]	[A]
40	3.72	235	100	69	65	6.7
60	5.52	239	113	74	72	8.6
80	7.20	243	130	85	82	9.8
100	9.54	248	145	95	94	10.1
120	11.70	251	167	111	109	10.7
140	13.92	254	188	125	120	11.2

Table 3: Extrusion parameters obtained for a 50 X 0.75 mm die

Screw speed	Throughput	Melt temperature	Extruder pressure	Die load [bar]	Axial load	Motor current
[rpm]	[kg/h]	[°C]	[bar]		[kn]	[A]
40	3.72	234	74	46	43	6.6
60	5.34	238	91	58	53	8.0
80	7.50	243	112	75	67	8.8
100	9.72	246	129	86	78	9.8
120	11.70	249	145	99	90	10.7
140	14.16	252	161	112	100	11.2

Table 4: Extrusion parameters obtained for a 50 X 1.00 mm die

Parameters	Units	Run 1	Run 2	Run 3
Melt temperature	°C	247	247	247
Screw speed	rpm	100	100	100
Output	kg/h	9.54	9.54	9.54
Motor current	А	9.7	9.7	9.7
Extruder pressure	bar	133	134	135
Die pressure	bar	98	101	100
Axial load	kn	82	77	87
Take-off speed	m/min	20	15	10
Film thickness	micron	25	30	45
Blow-up Ratio	1	1.8	1.8	1.8
Frost line height	mm	250	250	250

Table 5: Processing conditions film blowing using a die gap width of 0.75 mm

Parameters	Units	Run 4	Run 5	Run 6
Melt temperature	°C	246	246	246
Screw speed	rpm	100	100	100
Output	kg/h	9.72	9.72	9.72
Motor current	А	10.4	9.9	8.5
Extruder pressure	bar	126	118	136
Die pressure	bar	85	86	87
Axial load	kn	76	73	83
Take-off speed	m/min	20	15	10
Film thickness	micron	25	30	45
Blow-up Ratio	1	1.8	1.8	1.8
Frost line height	mm	250	250	250

Table 6: Processing conditions film blowing using a die gap width of 1.00 mm

Properties	Units	Run 1	Run 2	Run 3
Film thickness	micron	23.9	31.8	46.8
Coefficient of variation	%	9.6	9.4	6.8
OPTICAL PROPERTIES				
Haze	%	29	38	53
Gloss outside surface	units	33	32	27
Inside surface	units	45	45	43
MECHANICAL PROPERTIES				
Dart impact	g	39	82	136
	g/micron	1.63	2.57	2.90
Elmendorf tear strength M	D g	8	11	21
T	D g	43	40	48
Tensile strength				
Yield stress M	D MPa	55	54	55
T	D MPa	52	55	60
Ultimate stress M	D MPa	94	101	108
T	D MPa	ND	86	103
Ultimate elongation M	D %	294	361	445
T	D %	ND	488	528
Young Modulus M	D MPa	1675	1584	1464
	D MPa	1549	1525	1527
Puncture resistance				
Load at maximum	N	92	138	184
Energy to break point	J	2.50	4.00	6.80

Table 7: Film properties CARILON blown film extrusion(die gap 0.75 mm)

Properties		Units	Run 4	Run 5	Run 6
Film thickness	micron	25.2	31.5	46.5	
Coefficient of variation	%	9.9	11.4	9.2	
OPTICAL PROPERTIES					
Haze		%	29	37	54
Gloss outside surface		units	35	33	26
Inside surface		units	46	45	45
MECHANICAL PROPERT	IES				
Dart impact		g g/micron	64 2.53	96 3.00	138 2.96
Elmendorf tear strength	MD	g	7	12	22
	TD	g	36	43	53
Tensile strength					
Yield stress	MD	MPa	55	57	60
	TD	MPa	55	57	58
Ultimate stress	MD	MPa	93	111	111
	TD	MPa	ND	ND	85
Ultimate elongation	MD	%	284	373	425
	TD	%	ND	ND	470
Young Modulus	MD	MPa	1567	1586	1441
	TD	MPa	1647	1726	1355
Puncture resistance					
Load at maximum		N	112	156	199
Energy to break point		J	3.17	5.30	7.82

ND = Not detectable

Table 8: Film properties CARILON blown film extrusion
(die gap 1.00 mm)

Properties		Units	CARILON	LDPE	PP
Optical properties					
Haze		%	55	10	30
Gloss	outside inside	units units	30 45	55 60	25 30
Mechanical properties					
Dart impact		g	135	125	25
Elmendorf tear	MD TD	g	20 50	95 130	10 15
Yield stress	MD TD	MPa MPa	55 60	10 10	ND ND
Ultimate stress	MD TD	MPa MPa	110 105	25 20	40 40
Ultimate elongation	MD TD	% %	445 530	300 700	20 20
Young modulus	MD TD	MPa MPa	1465 1530	200 200	1300 1300
Puncture resistance					
Load at maximum Energy to break point		N J	185 6.80	68 3.30	89 1.58

	MFR g/10 min	Density kg/m²
LDPE	1.0	925
PP	3.0	930

 Table 9: Comparison of film properties between CARILON, Low density polyethylene and Polypropylene



Figure 1: Throughput versus screw speed (two die gap widths)



Figure 2: Effect screw speed on melt temperature (two die gap widths)



Figure 3: Effect of screw speed on extruder pressure (two die gaps)



Figure 4: Effect of screw speed on die gap pressure (two die gap widths)



Figure 5: Effect of screw speed on axial extruder load (two die gap widths



Figure 6: Effect of screw speed on motor current (two die gap widths)



Figure 7: Effect of film thickness on haze properties (two die gap widths)



Figure 8: Effect of film thickness on surface gloss (die gap of 0.75 mm)



Figure 9: Effect of film thickness on surface gloss (die gap width of 1.00 mm)



Figure 10: Effect of film thickness on dart impact (two die gap widths)



Figure 11: Effect of film thickness on tear resistance (die gap width of 0.75 mm)



Figure 12: Effect of film thickness on tear resistance (die gap width of 1.00 mm)



Figure 13: Effect of film thickness on tensile stress (die gap width 0.75 mm)



Figure 14: Effect of film thickness on tensile stress (die gap width 1.00 mm)



Figure 15: Effect of film thickness on ultimate elongation (die gap width 0.75 mm)







Figure 17: Effect of film thickness on modules (die gap width of 0.75 mm)



Figure 18: Effect film thickness on modules (die gap width of 1.00 mm)



Figure 19: Effect of film thickness on puncture resistance load at maximum (two die gap widths)







* 45 micron blown film Blow-up Ratio 1.8





Figure 22: Comparison of surface gloss properties



* 45 micron blown film Blow-up Ratio 1.8





Figure 24: Comparison of Elmendorf tear properties







Figure 26: Comparison of ultimate stress properties



Figure 27: Comparison of ultimate elongation properties



Figure 28: Comparison of young modulus.



* 45 micron blown film Blow-up Ratio 1.8





* 45 micron blown film Blow-up Ratio 1.8



APPENDIX 1

List of Abbreviations

ASTM:	American Society For Testing And Materials
CARILON EP:	Ethylene Polypropylene
HDPE:	High Density Polyethylene
LLDPE:	Linear Low Density Polyethylene
LDPE:	Low Density Polyethylene
PP:	Polypropylene
LVN:	Linear Viscosity Number
MFR:	Melt Flow Rate
MD:	Machine Direction
TD:	Transverse Direction
PM:	Plastic Machinebau
cN:	centiNewton
MPa:	Megapascal
G:	Gramme
mm	millimetre
J:	Joule
N:	Newton

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