

Correlations Between Processing Parameters, Morphology, and Properties of Blown Films of Linear Low-Density Polyethylene/Low-Density Polyethylene Blends. I. Crystalline Biaxial Orientation by IR and Mechanical Properties

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ABSTRACT: The change of the processing parameters of a blown film operation alters the mechanical and optical properties of the films. This work studied the influence of some of these parameters on the properties of blown films made of blends of linear low-density polyethylene (LLDPE) and LDPE. Correlations between the crystalline biaxial orientations of these films and the mechanical properties were found. The crystalline biaxial orientation was measured by IR following the Krishnaswamy approach. The *a* axis of the unit cell was oriented along the machine direction (MD) at all LDPE concentrations, and it was not affected by the blow-up ratio (BUR). In contrast, the *b* axis changed its

orientation from orthogonal to MD to along the transverse direction (TD), and it was affected by the BUR. Finally, the *c* axis changed its orientation from equiplanar between the MD and TD to along the thickness of the film, and it was influenced by the BUR. The decrease of the tensile mechanical properties along the MD with the increase in the amount of LDPE in the blends was attributed to the tilting of the *c* axis toward the film thickness. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 101: 3161–3167, 2006

Key words: films; blends; polyethylene; orientation

INTRODUCTION

Linear low-density polyethylene (LLDPE) as a blown film has superior toughness (tear strength, impact resistance, and puncture resistance) but higher haze than LDPE. Therefore, it is expected that the mixture of both polymers will produce blends with synergistic properties (i.e., good mechanical and optical properties).

Both properties are affected by the macromolecular orientation achieved during the blowing process. During this process, a macroscopic biaxial orientation is given to the film by the upward and circumferential drawings that are developed by the nip rolls and the air blow, respectively. At the same time, the “balloon” is cooled by the surrounding air from the air-cooling ring, and the blown film crystallizes under biaxial elongational tensions. Thus, both the amorphous and crystalline parts of the film will have a biaxial orien-

tation along the machine direction (MD) and along the transverse direction (TD).

Peterlin¹ pointed out that the biaxial orientation of the amorphous part is a good indicator of the amount of taut tie molecules present and hence is an excellent parameter for the description of the mechanical properties, being proportional to the elastic modulus and strength. Patel et al.² confirmed these observations in the study of blown films of LLDPE. Peterlin¹ also recalled that the crystallization in a blown film produces “row nucleated” structures and that the “row nuclei” with partially extended chains is the strongest element of this structure.

Conversely, the biaxial orientation of the crystalline part, mainly of the chain axes or *c* axis of the unit cell, can also be correlated with the mechanical properties like the tension at break. If the *c* axis is aligned parallel to the MD or TD, it is expected that the tensile strength at that particular direction will be high. Recent studies³ have shown that the TD tear resistance of LDPE blown films, for example, is proportional to both the MD *a*-axis and TD *b*-axis orientation. Other work⁴ on blown films of LLDPE/LDPE blends has found that the tensile properties of these films has a positive synergism; that is, the values of those properties are

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higher than the ones expected from the additive rule, especially the LLDPE-rich blends.

The measurement of the crystalline biaxial orientation can be done by X-ray diffraction (WAXD) using pole figures. However, the procedure as a quality control tool is complex and expensive. Therefore, other less expensive techniques, like IR spectroscopy, IR, and birefringence measurements, can be used.

The use of IR dichroism to measure the crystalline uniaxial orientation function is a common practice. However, the calculation of the crystalline biaxial orientation function by IR is less common, because it requires extra measurements and knowledge of the films' morphology and crystallinity. Thus, the objective of the first part of this series is to correlate the influence of the processing parameters with the crystalline biaxial orientation of blown films of LLDPE/LDPE blends as measured by IR and to correlate this orientation with the mechanical properties of the films. The second part of this series⁵ will report the measurement of the crystalline and amorphous biaxial orientation of these films by X-ray pole figures. The correlation between the rheological properties, morphology, and optical properties of these films was published in a recent article.⁶

Stein and Wilchinsky⁷ developed methods to measure the crystalline biaxial orientation from pole figures generated from WAXD measurements. White et al.^{8,9} modified the Stein and Wilchinsky equations, which resulted in the well-known White and Spruiell biaxial orientation factors (f^B) in the MD ($f^B_{MD,j}$) and TD ($f^B_{TD,j}$) of the crystallographic axis j (a , b , or c), given by

$$f^B_{MD,j} = 2\cos^2\phi_{MD,j} + \cos^2\phi_{TD,j} - 1 \quad (1)$$

and

$$f^B_{TD,j} = 2\cos^2\phi_{TD,j} + \cos^2\phi_{MD,j} - 1 \quad (2)$$

where j is the crystallographic axis (a , b , or c), $\cos^2\phi_{i,j}$ is the mean square cosine of the ϕ angle that the j crystallographic axis has with respect to the i processing direction, and B is the biaxial orientation.

In the case of PE, it is known that the unit cell is orthorhombic and has the following parameters: $a = 0.736$ nm, $b = 0.495$ nm, $c = 0.254$ nm, and $\alpha = \beta = \epsilon = 90^\circ$. However, studies^{10,11} have shown that branch type and distribution and drawing can affect these parameters. For LDPE, drawing has been shown to reduce the value of a , whereas for LLDPE, drawing has the opposite effect. For both polymers the drawing decreases the b and c parameters.

Recently, Krishnaswamy¹² developed an IR method to characterize these biaxial orientation factors in PE blown films. The method considers that the morphol-

ogy of blown films of PE made at low elongational rates is a shish-kebab type or row nucleated, as described by Keller and Machin in Peterlin.¹ The shish or row nuclei are formed by macromolecules aligned parallel to the main flow direction, whereas the kebabs or lateral lamellae are formed by twisted lamellae. The b axis corresponds to the crystal growth direction, whereas the c and a axes are randomly oriented. Therefore, the b axis can be assumed to be orthogonal to the MD.¹ The resultant equations of the method are¹² the following:

$$f^B_{MD,a} = 2(X_3 + X_5) + (X_2 + X_6) - 1 \quad (3)$$

$$f^B_{TD,a} = 2(X_2 + X_6) + (X_3 + X_5) - 1 \quad (4)$$

$$f^B_{MD,b} = 2(X_4 + X_6) + (X_1 + X_5) - 1 \quad (5)$$

$$f^B_{TD,b} = 2(X_1 + X_5) + (X_4 + X_6) - 1 \quad (6)$$

$$f^B_{MD,c} = 2(X_1 + X_2) + (X_3 + X_4) - 1 \quad (7)$$

$$f^B_{TD,c} = 2(X_3 + X_4) + (X_1 + X_2) - 1 \quad (8)$$

where the X_k ($k = 1, 2, \dots, 6$) is the fraction of each of the six orthogonal configurations with respect to the axis of a blown film in which the a , b , and c axes of PE can be aligned.

In order to apply the resultant equations from the Krishnaswamy method to blown films of LLDPE/LDPE blends, it will be assumed that the crystals of both polymers are immiscible, because their morphology is row nucleated¹³; the IR band at 730 cm^{-1} will be polarized along the a axis of both unit cells; and the crystalline component of the 719 cm^{-1} band will be polarized along the b axis of both unit cells.

EXPERIMENTAL

Materials

The LLDPE (comonomer 1-butene) and the LDPE were from Braskem S. A. (Triunfo, Brazil). Their molecular weight distributions were obtained by gel permeation chromatography using a Waters model 150-CV apparatus with Ultrastaygel columns. Trichlorobenzene was used as a solvent at 150°C , and the

TABLE I
Molecular Weight Distributions

Polymer	M_n (g/mol)	M_w (g/mol)	M_z (g/mol)	M_w/M_n	M_z/M_w
LLDPE	8522	90328	264929	10.59	2.93
LDPE	7066	88357	295662	12.50	3.34

TABLE II
Extrusion and Blowing Parameters

Mass rate	90 kg/h
Freeze line height	80 cm
Cooling air temperature	28°C
Die diameter	200 mm
Die gap	1.8 mm
Screw diameter	75 mm
Screw length/diameter ratio	24
Screw speed	55 rpm
Mass pressure	226 kgf/cm ²
Take-up roll pressure	6 bar
Average final film thickness	30 μm
Blow up ratio final film radius/die radius	2/1 and 3/1

machine was calibrated with monodisperse polystyrene standards. Table I provides these results.

Blown films

The blown films of the blends were produced in a Carnevali (model CLD-75) tubular extrusion line at LLDPE/LDPE compositions of 100/0, 90/10, and 80/20 wt %. The extrusion and blowing parameters were kept constant regardless of the blend composition and are shown in Table II.

Depending on the blend composition, the extruder temperatures and take-up velocities were set as provided in Table III.

Amount of crystallinity

The weight fraction crystallinity (W_c) was measured by differential scanning calorimetry (DSC) using a PerkinElmer DSC 7 after heating at 10°C/min from 20 to 140°C. The calibration was made with indium.

The amount of crystallinity of the blends was calculated from the following equations:

$$W_{c,LLDPE} = (\Delta H_{LLDPE} / \Delta H_{100\%}) \quad (9)$$

$$W_{c,LDPE} = (\Delta H_{LDPE} / \Delta H_{100\%}) \quad (10)$$

where ΔH_{LLDPE} and ΔH_{LDPE} are the heats of fusion of the LLDPE and LDPE in the blends, respectively;

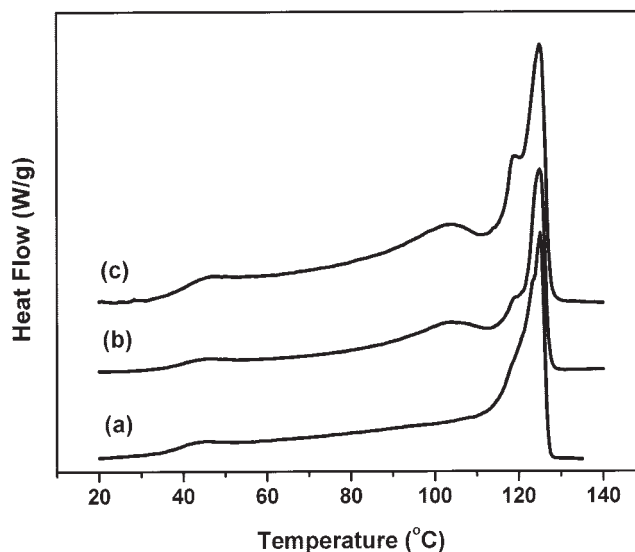


Figure 1 DSC curves of the (a) 100/0, (b) 90/10, and (c) 80/20 LLDPE/LDPE films at BUR = 3.

$\Delta H_{100\%}$ is the heat of fusion of a 100% crystalline sample (68.5 cal/g)¹⁴; and $W_{c,LLDPE}$ and $W_{c,LDPE}$ are the weight fraction crystallinities of LLDPE and LDPE in the blends, respectively.

Biaxial orientation factors by IR spectroscopy

To measure the biaxial orientation factors of the blown films by IR spectroscopy, a Fourier transform IR instrument (PerkinElmer Spectrum 1000) with a gold mesh polarizer was used. Samples for the IR measurements were cut along the height of the blown films. The measurements were made from 600 to 4000 cm⁻¹ with a resolution of 4 cm⁻¹. The peak absorbancies were measured from the absorption bands at 730 and 719 cm⁻¹ with the films' MD aligned parallel and perpendicular to the polarization of the incident IR radiation.

The absorption coefficients were measured from isotropic samples that were prepared by compression molding in a Schwing Siwa press after melting at 210°C for 5 min. The 2019 cm⁻¹ band was chosen for the thickness correction.¹⁵ The film thickness was cal-

TABLE III
Extruder Temperatures and Take-Up Velocities

LLDPE/LDPE blend	BUR	Temperature (°C)						$\dot{\epsilon}$ (s ⁻¹)
		Extrudate	Die	Zone 4	Zone 3	Zone 2	Zone 1	
100/0	2	190	209	167	164	163	156	0.84
	3	190	205	170	160	162	153	0.57
90/10	2	180	210	169	163	161	150	0.83
	3	180	205	169	160	160	150	0.52
80/20	2	180	209	166	163	162	150	0.82
	3	180	210	165	160	162	151	0.51

$\dot{\epsilon}$, elongation rate = $(V - V_o)/Z$, where V is the take-up velocity and V_o is the extrudate velocity.

TABLE IV
Thermal Transition Calculations

LLDPE/LDPE blend	BUR	$W_{c,LLDPE}$	$W_{c,LDPE}$	$T_{m1,LLDPE}$ $T_{m2,LLDPE}$ (°C)	$T_{m3,LLDPE}$ ^a (°C)	$T_{m1,LDPE}$ (°C)	$T_{m2,LDPE}$ (°C)
100/0	Isotropic	0.33	—	125.1 117.9	45.9	—	—
90/10	Isotropic	0.35	0.08	125.67 119.5	46.1	103.0	ND
80/20	Isotropic	0.32	0.11	125.5 118.8	46.3	103.1	ND
0/100	Isotropic	—	0.48	—	—	103.2	40.0
100/0	2	0.46	—	125.2 118.9	46.0	—	—
100/0	3	0.46	—	125.8 119.1	46.4	—	—
90/10	2	0.41	0.09	125.6 119.8	46.2	104.4	ND
90/10	3	0.41	0.10	125.0 119.3	46.0	104.0	ND
80/20	2	0.40	0.13	124.8 118.6	46.4	103.5	ND
80/20	3	0.39	0.14	124.3 118.3	47.0	103.3	ND

ND, not detected.

^a Attributed to the LLDPE.

culated from the fringe interference using the following equation:¹⁶

$$t = 1/2n(v_2 - v_1) \quad (11)$$

where $(v_2 - v_1)$ is the difference between the maximum and minimum of two adjacent waves and n is the refractive index.

Mechanical properties

The tensile properties of the blown films were measured along both directions (MD and TD) in a universal testing machine (model 5500) following the ASTM D 882-90 standard procedure. The tension samples were cut from the blown films using a rotating cylinder. The tension at break along the MD (σ_{MD}) and TD (σ_{TD}), the elastic modulus along the MD (E_{MD}) and TD (E_{TD}), and the elongation at break along the MD (ϵ_{MD}) and TD (ϵ_{TD}) were calculated from the tensile curves.

RESULTS AND DISCUSSION

Figure 1 shows standard DSC curves of the films at a blow-up ratio (BUR) of 3, and Table IV provides the results of the calculation of the thermal transitions. Note that both polymers showed multiple melting peaks, which are probably due to the effect of the type and distribution of branching on the thickness and perfection of the lamellas.

In the isotropic state the pure LDPE samples had two melting peaks, indicating the formation of two

different populations of crystals. The more perfect crystals of the isotropic LDPE melted at approximately 103°C, whereas the less perfect crystals melted at approximately 40°C. In contrast, the pure LLDPE samples showed two main melting peaks at approximately 46 and 125°C and a small shoulder at approximately 118°C.

The blowing of the pure LLDPE sample again produced three populations of crystals, which melted at the same temperatures as in the isotropic state. The blends also displayed multiple melting peaks. These were attributed to the LLDPE or the LDPE based on the peaks of the pure component; thus, the melting peak around 103–104°C was attributed to LDPE melting, whereas the peak at 118–125°C was attributed to LLDPE melting. From these main peaks, the $W_{c,LLDPE}$ and $W_{c,LDPE}$ were calculated from eqs. (9) and (10). Thus, it was concluded from these measurements that the crystalline part of these blends was immiscible.

It can be observed that the increase of BUR at each composition did not affect the amount of crystallinity and the main melting temperatures of the blend components.

Figure 2 shows standard IR spectra of the films at a BUR of 3. From these spectra each X_k was calculated for each composition and BUR; then, the biaxial factors were determined from eqs. (3)–(8). Table V provides these results.

The orientation factors were very small, indicating that the average biaxial crystalline orientation of the films was also very small. In addition, we observed that the standard deviations of the measurements

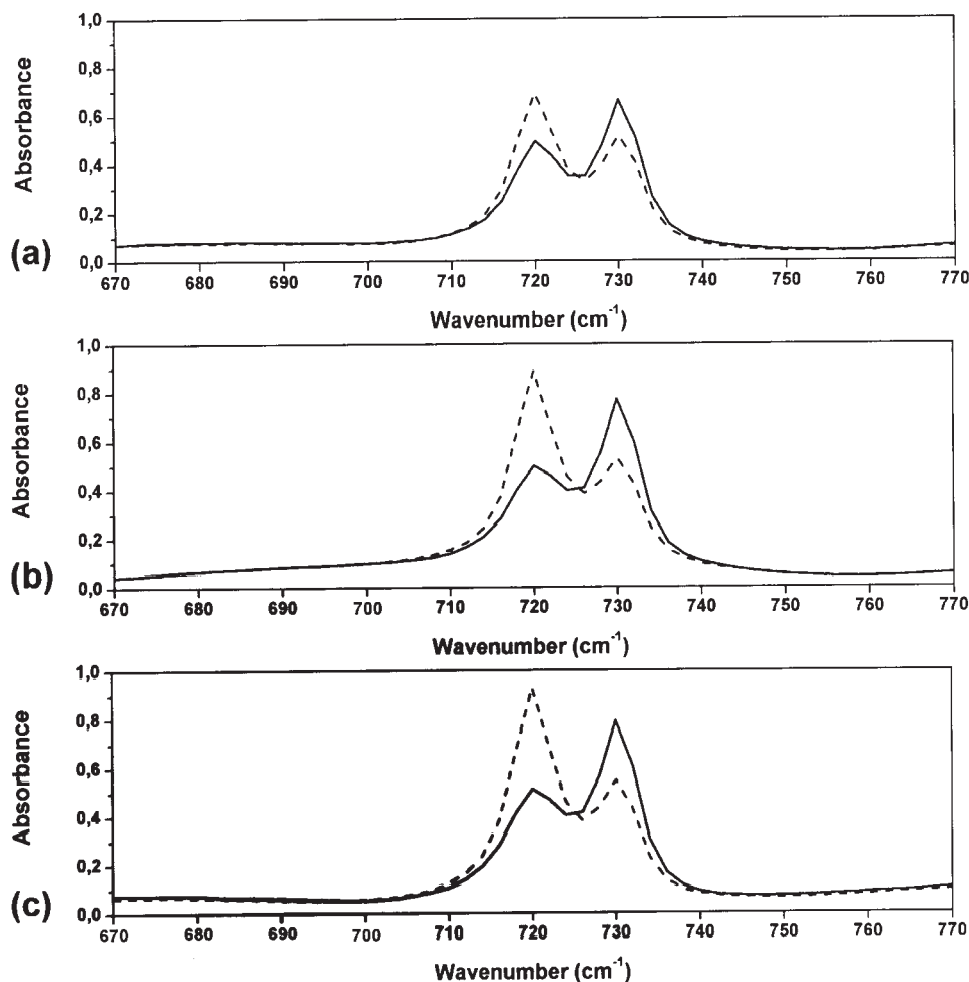


Figure 2 IR spectra of (a) 100/0, (b) 90/10, and (c) 80/20 LLDPE/LDPE films at BUR = 3 using polarized light (—) parallel and (- -) perpendicular to the MD.

were high. These errors can be credited to the high variation of the film thickness along the height.

Standard graphical representations, which are the White/Spruiell orientation triangle diagram,⁸ of these orientation factors are shown in Figures 3, 4, and 5 as a function of the composition and BUR. The dashed lines in these figures represent the equibiaxial orientation along the plane of the film. Figure 6 also shows the orientation factors at a BUR of 3. Observe that the *c* axis tilts toward the film thickness as the amount of

LDPE in the blend increases. From these figures and Table V the following conclusions were made.

Blends of 100/0 LLDPE/LDPE

The orientation of the *a* axis was not influenced by the degree of BUR; this axis was oriented between the MD and along the thickness of the film (ND), but closer to the MD, confirming other results.¹⁷⁻¹⁹ The orientation of the *b* axis was also not influenced by the degree of

TABLE V
Biaxial Factors

LLDPE/LDPE blend	BUR	$f_{MD,a}^B$	$f_{TD,a}^B$	$f_{MD,b}^B$	$f_{TD,b}^B$	$f_{MD,c}^B$	$f_{TD,c}^B$
100/0	2	0.054 ± 0.043	-0.110 ± 0.042	-0.212 ± 0.044	0.044 ± 0.047	0.158 ± 0.085	0.066 ± 0.088
	3	0.048 ± 0.043	-0.075 ± 0.064	-0.205 ± 0.077	-0.018 ± 0.089	0.157 ± 0.146	0.093 ± 0.153
90/10	2	0.191 ± 0.064	-0.009 ± 0.048	-0.112 ± 0.060	0.334 ± 0.071	-0.079 ± 0.120	-0.325 ± 0.119
	3	0.214 ± 0.044	0.008 ± 0.038	-0.119 ± 0.047	0.242 ± 0.054	-0.095 ± 0.091	-0.250 ± 0.091
80/20	2	0.234 ± 0.031	0.042 ± 0.025	-0.101 ± 0.030	0.356 ± 0.040	-0.133 ± 0.060	-0.398 ± 0.065
	3	0.300 ± 0.039	0.093 ± 0.038	-0.030 ± 0.039	0.359 ± 0.049	-0.270 ± 0.078	-0.452 ± 0.085

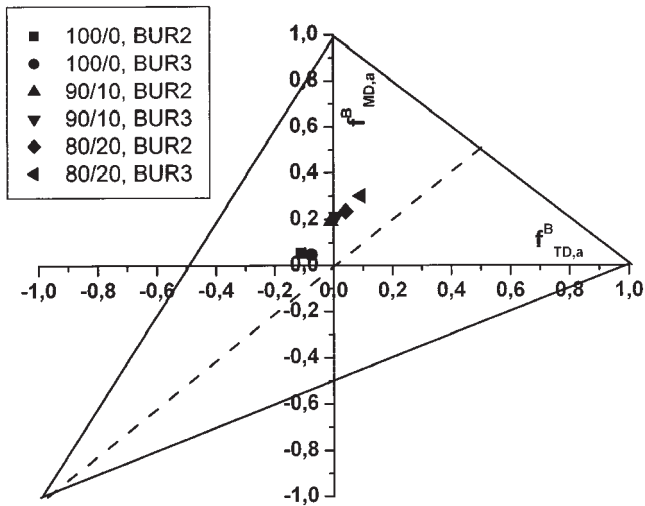


Figure 3 Biaxial orientation factors of the *a* axis of LLDPE/LDPE films at BUR = 2 and 3. (---) The equibiaxial orientation along the plane of the film.

BUR; this axis was oriented orthogonal to the MD (almost isotropic however). The orientation of the *c* axis was slightly influenced by the degree of BUR, decreasing with the increase in BUR. This axis had equal planar orientation between the MD and TD (equibiaxial orientation) at all BURs.

Blends of 90/10 LLDPE/LDPE

The orientation of the *a* axis was not affected by the degree of BUR; this orientation was along the MD. The orientation of the *b* axis was affected by the degree of BUR; the orientation was between the ND and TD, but closer to the TD. The *c*-axis orientation was again

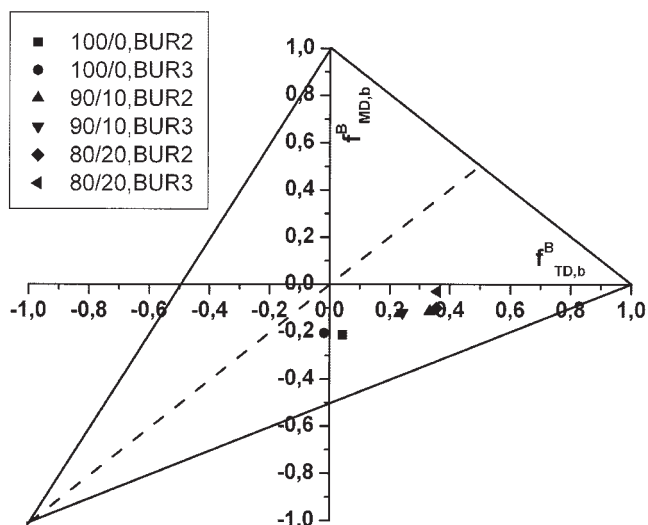


Figure 4 Biaxial orientation factors of the *b* axis of LLDPE/LDPE films at BUR = 2 and 3. (---) The equibiaxial orientation along the plane of the film.

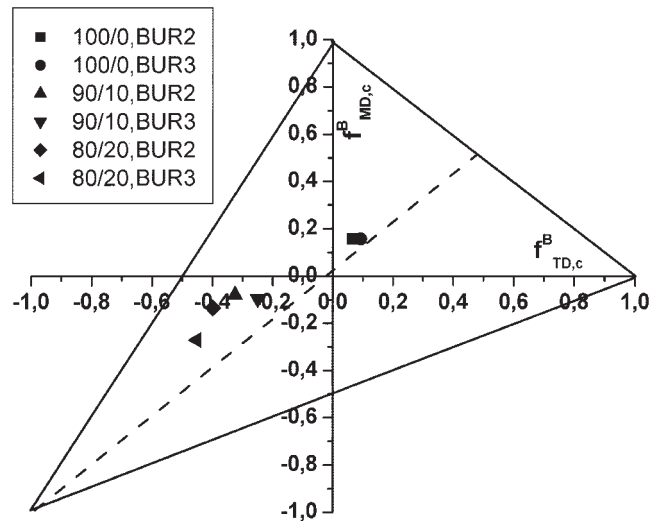


Figure 5 Biaxial orientation factors of the *c* axis of LLDPE/LDPE films at BUR = 2 and 3. (---) The equibiaxial orientation along the plane of the film.

slightly influenced by the BUR; its orientation was preferentially along the ND.

Blends of 80/20 LLDPE/LDPE

The *a*-axis orientation was affected by the BUR, increasing with increases in the BUR. This orientation was again preferentially along the MD. The *b*-axis orientation was affected by the BUR, increasing with increased BUR. Its orientation was along the TD. The *c*-axis orientation was also affected by the BUR. The orientation was along the ND.

Therefore, the *a* axis was preferentially oriented along the MD for all compositions. By contrast, the *b*

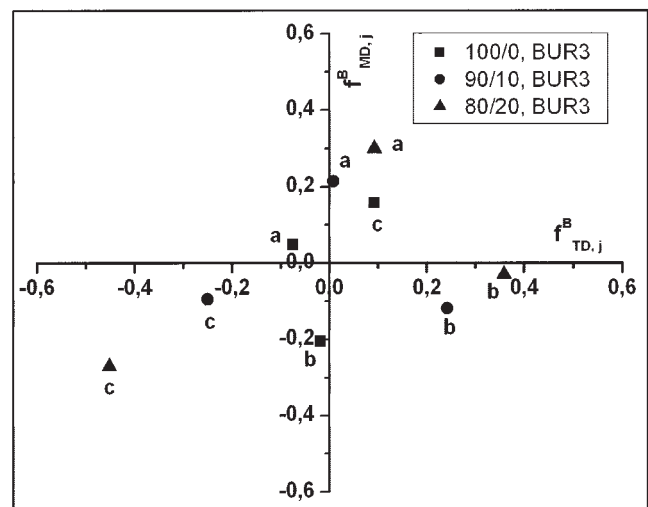


Figure 6 Biaxial orientation factors of the crystal unit cell axes of LLDPE/LDPE films at BUR = 3.

TABLE VI
Tensile Properties of Films Along MD and TD

LLDPE/LDPE blend	BUR	E_{MD} (MPa)	E_{TD} (MPa)	σ_{MD} (MPa)	σ_{TD} (MPa)	ε_{MD} (%)	ε_{TD} (%)
100/0	2	193.38 ± 14.40	232.12 ± 23.48	33.58 ± 1.81	26.49 ± 2.07	783.59 ± 25.38	1055.45 ± 61.99
	3	206.99 ± 7.50	228.03 ± 54.32	34.55 ± 3.33	29.18 ± 2.35	803.92 ± 30.47	916.41 ± 36.85
90/10	2	182.13 ± 13.05	224.44 ± 26.02	33.72 ± 2.09	22.59 ± 2.75	714.88 ± 21.95	947.84 ± 78.79
	3	121.09 ± 14.05	134.27 ± 16.83	26.89 ± 3.41	21.94 ± 2.44	810.33 ± 49.10	880.96 ± 49.82
80/20	2	174.30 ± 9.94	229.24 ± 13.49	32.45 ± 1.82	25.01 ± 2.85	745.86 ± 29.13	1012.15 ± 73.79
	3	187.45 ± 57.31	225.34 ± 58.64	31.38 ± 1.95	27.63 ± 3.03	760.31 ± 41.18	931.80 ± 59.80

axis changed its orientation gradually from orthogonal to the MD in the pure LLDPE film to along the TD in the blends. The chain axis also changed from an equibiaxial orientation in the pure LLDPE films to orientation along the film thickness in the blends; that is, the chain axes of the lamellae tilted.

Thibault et al.²⁰ studied blown films of blends of a metallocene LLDPE (mLLDPE) with a radicalar LDPE. They measured the crystalline orientation from pole figures and found the following behavior: LDPE > 70 mLLDPE/30 LDPE > 95 mLLDPE/5 LDPE > mLLDPE. The orientation factors showed also that the *c* axis was preferentially oriented along the film thickness.

Table VI lists the results of the tensile properties of the films along the MD and TD. The E_{MD} decreased with the increase in the amount of LDPE and with the increase in BUR; in contrast, the E_{TD} did not change with either composition or BUR.

Regarding σ_{MD} , we observed that it decreased with the increase in the amount of LDPE. The increase in BUR also decreased the σ_{MD} in the blends but increased the σ_{MD} in the pure LLDPE. By contrast, σ_{TD} decreased with both the increase in the amount of LDPE and the increase in BUR.

Finally, the ε_{MD} decreased with the increase in the amount of LDPE and increased with the increase in BUR. The behavior of ε_{TD} was erratic, and at the present no possible explanations can be found.

The decrease of E_{MD} , σ_{MD} , and ε_{MD} with the increase of the amount of LDPE in the blends can be credited to the change in orientation of the *c* axis that went from equibiaxial in the pure LLDPE films to oriented along the film thickness in the blends.

CONCLUSIONS

The calculation of the biaxial crystalline orientation factors of the blown films of LLDPE/LDPE blends using the Krishnaswamy approach was shown to be a valid one. The high standard deviations of the results were credited to the high variation of the film thickness along the height. We found that, at all concentrations, the *a* axis was oriented along the MD. Conversely, with the increase in the amount of LDPE in the blends, the *b* axis changed its orientation from orthogonal to the MD to

along the TD, whereas the *c* axis changed from equibiaxial between the MD and TD to along the ND.

The decrease of the tensile mechanical properties along the MD with the increase in the amount of LDPE in the blends was attributed to the tilting of the *c* axis toward the film thickness.

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