Injection-Moulded Blends of a Thermotropic Liquid Crystalline Polymer with Polyethylene Terephthalate, Polypropylene, and Polyphenylene Sulfide

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SYNOPSIS

The properties of various blends of a polyester-type thermotropic liquid crystalline polymer (LCP) with polyethylene terephtalate, polypropylene, and polyphenylene sulfide were investigated. The polymers were blended in a twin screw extruder after which samples were injection moulded. The mechanical properties, morphology, and thermal properties of the blends are discussed.

INTRODUCTION

Thermotropic main-chain liquid crystalline polymers (LCPs) consist of linear semirigid rodlike molecules that are capable of aligning to a very high degree during melt flow, and thus form a highly ordered melt phase. When the polymer melt is cooled, the orientation is retained, which leads to a highly ordered fibrous structure and anisotropic properties. The properties of thermotropic LCPs, like high modulus, are based on this oriented structure.^{1,2} The degree of orientation can be influenced by the processing conditions, especially by mould design, or by applying elongational forces to the molten polymer. The processing of LCPs, therefore, plays an important part in their final characteristics.^{3,4}

LCP mouldings can exhibit a layer structure and a skin core morphology depending on the processing conditions. Near the surface there is a skin layer with extensive highly ordered fibers, while in the core there is less orientation and fiber formation.³⁻⁷

By blending thermotropic main-chain LCPs with engineering thermoplastics, the highly ordered fibrous structure and good properties of LCPs could be transferred into the more flexible matrix polymer. LCPs are blended with thermoplastics mainly to reinforce the matrix polymer or to improve its dimensional stability. A small amount of a thermotropic LCP can also make some thermoplastics easier to process due to its relatively low melt viscosity.

Thermotropic LCP and thermoplastic blends consist of two separate phases and are mainly immiscible. If the polymers are quite similar, however, e.g., a polyester-type LCP and a polyester, they can be partly miscible.^{8,9}

Similar skin-core structures can be found in the blends as in pure LCPs. In the skin layer there are often more oriented fibrous LCP phases, while in the core the LCP phases are less oriented or in spherical form.¹⁰⁻¹² LCP content and processing orientation determine whether fibers are formed or not. The morphology and the resultant properties of the blends are also highly dependent on the composition of the two polymers. The mutual compatibility of the polymers and the interfacial adhesion between them play important roles here. The ratio of the melt viscosities of the polymers affects their miscibility and fiber formation.^{13,14}

In the solid state blends of LCPs and thermoplastics can exhibit a compositelike fiber structure if sufficient orientation of the molten LCP phases during processing is achieved.^{15,16}

Several researchers have studied blends of thermotropic LCPs and thermoplastics during recent years. Commercial or experimental LCPs [mainly aromatic polyesters, polyamides (PA), or their copolymers] have been blended with many thermo-

^{*} To whom correspondence should be addressed. Journal of Applied Polymer Science, Vol. 44, 1051–1060 (1992) © 1992 John Wiley & Sons, Inc. CCC 0021-8995/92/061051-10\$04.00

plastics, e.g., PC,^{12,13,15-19} PET,^{8,10,19-22} PBT,^{9,15} PA6,²⁵ PA12,^{26,27} PA66,^{15,28,29} amorphous PA,¹¹ PES,^{15,30} PPS,³¹ PSU,³² PEI,^{15,18,33} PEEK,^{15,30} PVC,³⁴ HDPE,²⁶ and aromatic polyesters.¹⁵

The blends have been processed by extrusion, melt spinning, injection, and compression moulding. Due to the melt behavior of thermotropic LCPs, extrusion and melt spinning are the best methods to achieve uniaxial orientation and good LCP fiber formation in the matrix polymer. However, in situ composites of LCP and thermoplastics can also be formed by injection moulding, although controlling LCP orientation and achieving good fiber formation is more complicated.

In this work we focus on twin screw extruder blended and injection-moulded blends of a polyestertype thermotropic LCP with polyethylene terephtalate, polypropylene, and polyphenylene sulfide. Polypropylene and polyphenylene sulfide are matrix polymers that have not been widely studied before. The mechanical and thermal properties of the blends are discussed here in relation to their morphology and rheology.

EXPERIMENTAL

Materials

The liquid crystalline polymer used in this work was Vectra A 950 produced by Hoechst Celanese. It is a totally aromatic polyester-type thermotropic mainchain LCP. The following properties of the LCP are given by the manufacturer: density 1.40 g/cm³, melting point 280°C, tensile strength 165 MPa, elastic modulus 9700 MPa, and elongation at break 3.0%.³⁵

The matrix polymers used in these experiments were polyethylene terephtalate (PET Melinar P100: ICI), polypropylene (PP VB 65-11B: Neste), and polyphenylene sulfide (PPS Fortron 0214P1: Hoechst Celanese). All these polymers were supplied as extruded pellets.

Blending

The LCP was first manually mixed with the matrix thermoplastics as dry pellets in a barrel. The amounts of LCP used in the blends were 5, 10, 20, 30, and 50% by weight. The mixed polymer pellets were then carefully dried before the melt blending stage in a Conair Micro-D dehumidifying dryer. The drying conditions for the blends shown in Table I were chosen according to the matrix polymers.

Table I I	Drying	Conditions	for	the	Blends
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	T	t	
Blend	(°C)	(h)	
LCP	155	5	
LCP-PET	160	5	
LCP-PP	50	2	
LCP-PPS	140	3	

The melt blending of the materials was performed by a Berstorff ZE $25 \times 33D$ corotating twin screw extruder at temperatures ranging from 285 to 290°C. The screw speed used was 150 rpm. The extrudate was immediately quenched in a water bath and repelletized and redried before processing.

Processing

Both the blends and the pure polymers were injection moulded into the form of test specimens after proper drying in a Conair Micro-D dehumidifying dryer. The drying conditions were the same as shown in Table I.

Injection moulding was carried out in an Arburg Allrounder 20-75A 220 D injection moulding machine. The pure polymers were processed at conditions recommended by the manufacturers. As similar conditions as possible were chosen for the blends as for the matrices. The processing conditions were not optimized for every individual blend composition, but similar conditions were used for each blend type. The most important processing parameters are shown in Table II.

Testing

The moulded specimens consisted of standard test bars for tensile, bending, and impact tests. Before testing all samples were conditioned according to ISO 291 at 23°C and 50% relative humidity for at least 88 h.

Tensile properties were measured according to ISO 527 using an Alwetron F 5000 testing machine. The strain rate was 5 mm/min for tensile strength and elongation measurements and 1 mm/min for determining elastic modulus. The dimensions of the test bars were length 154 mm, width of narrow part 10 mm, and thickness 4 mm.

The Charpy impact strength of unnotched test specimens was determined according to ISO 179 using a Zwick 5102 pendulum-type testing machine. The dimensions of the specimens were $4 \times 6 \times 50$

Material		$T_{ m cyl.}$	(°C)		T _{mould} (°C)	р _{іпј.} (%)		p _{hold.} (%)	1		$t_{ m hold.}~({ m s})$		t _{cool.} (s)
LCP	280	285	290	290	62	30	30	20	10	3	2	2	30
PET	290	295	295	290	40	20	7	5	3	5	10	15	25
LCP-PET	290	295	295	290	40	10	7	5	3	5	10	15	20
PP	200	210	210	220	43	40	40	25	10	5	10	15	15
LCP-PP	280	285	290	290	49	40	40	25	10	3	3	3	40
PPS	310	320	325	325	133	30	30	20	10	2	2	2	40
LCP-PPS	310	320	325	325	133	30	30	20	10	2	2	2	40

 $^{\rm a}\,p_{\rm inj.}$ and $p_{\rm hold.}$ are expressed as a percentage of the maximal pressure (2020 bar).

mm. For PPS and its blends a pendulum of 10 kpcm was used, while for all other materials a pendulum of 40 kpcm was used.

Heat deflection temperature (HDT) was determined according to ISO 75 using a Zwick 4204 testing machine. The measurements were performed by method A using a load of 1.82 N/mm² and raising the temperature by 2°C/min. The dimensions of the test bars were $4 \times 6 \times 112$ mm.

The differential scanning calorimetry (DSC) measurements of the extruded samples were made by a Perkin-Elmer DSC-7. The heating and cooling rates were 20° C/min.

The linear thermal expansion coefficients of the 17-mm-long specimens were determined using a TMA 40 thermomechanical analyzator. The temperature range was -80 to $+70^{\circ}$ C at a rate of $10-15^{\circ}$ C/min in a nitrogen atmosphere.

The morphology of the fractured surfaces of the extrudates and injection-moulded specimens were examined using a JEOL JXA-840A scanning electron microscope (SEM). The samples were fractured at liquid nitrogen temperature. The fractured surfaces were coated with a 15-nm gold layer for microscopy purposes.

The dynamic mechanical thermal analysis (DMTA) properties of the samples were measured with a DMTA Mk II thermoanalyzator using the "single" method. The experimental length was 19 mm, the temperature range was -50 to +200°C at a rate of 3°C/min with a frequency of 1 Hz. Nitrogen was used as the inert gas.

RESULTS AND DISCUSSION

Mechanical Properties

Table III shows the tensile and impact properties for the injection-moulded blends. The results of elastic moduli, E and storage moduli E' for the injection-moulded blends are also plotted against LCP content in Figure 1(a)-(c).

These results show that the tensile strength and elastic modulus of a thermoplastic matrix generally increased when LCP was added to the matrix, while elongation at break and Charpy impact strength decreased significantly. This means that LCP addition made the matrices studied stiffer but more brittle.

The elastic modulus and tensile strength of injection moulded LCP-PET blends increased almost linearly with increasing LCP content. At low LCP

Table IIIMechanical Properties of theInjection-Moulded Blends

Material	Tensile Strength (MPa)	Elastic Modulus (MPa)	Elongation (%)	Charpy Impact Strength (kJ/m ²)
LCP	123	7588	3.3	64
PET/0	61	2631	294	a
PET/10	60	2717	3.3	21
PET/20	74	4051	2.4	11
PET/30	84	5502	2.0	8.9
PET/50	108	6698	2.5	13
PP/0	34	1805	588	(61) ^b
PP/5	32	2072	13.2	30
PP/10	34	2063	9.6	23
PP/20	39	2438	4.8	14
PP/30	44	2937	3.6	11
PP/50	51	4730	2.5	7.6
PPS/0	82	3697	2.7	8.0
PPS/5	84	4035	2.9	12
PPS/10	87	4040	3.4	24
PPS/20	73	4512	2.2	15
PPS/30	69	4952	1.7	14
PPS /50	100	6447	2.1	13

* Not broken.

^b Only some of the samples broken.

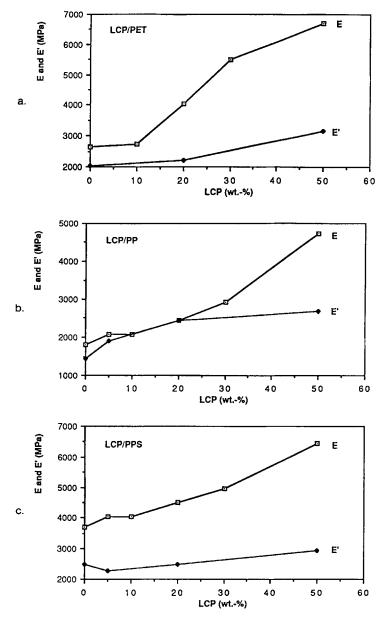


Figure 1 Elastic (E) and storage moduli (E') for the injection moulded blends: (a) LCP-PET, (b) LCP-PP, and (c) LCP-PPS blends.

contents the results followed those predicted by the so-called rule of mixture (Ref. 36, p. 455). At LCP contents higher than 20 wt % the obtained values even slightly exceeded the predicted ones. For a 30/70 LCP-PET blend the elastic modulus was more than twice the value of the pure matrix. This increase in stiffness can be related to the good orientation and fiber formation of the LCP phases in the PET matrix, which can be seen in the SEM micrographs in Figure 3.

Figure 1 shows a good correlation between the curves of elastic and storage moduli. The level of

PET crystallinity also increased with increasing LCP content. LCP was thought to act as a nucleating agent for PET, something which has also been suggested by Joseph et al.^{10,20}

Injection-moulded blends of para-hydroxybenzoic (PHB) acid and PET based LCPs with PET have been studied by Joseph et al.^{10,20} and Brostow et al.⁸ The former found that the bending modulus of PET increased with increasing LCP content, being as much as three times the value of pure PET for a 50/ 50 blend. Our results are virtually similar, although a different type of LCP was used. Brostow et al.⁸ have compared the mechanical properties of PHB-PET copolymers with those of PET and PHB-PET blends containing LCPs at the same PHB content. An island model was developed by them to explain their results. The blends were not much weaker than the corresponding copolymers. Partial miscibility can exist in those blends due to the similarities in the structure of LCP and PET. Extruded or melt spun blends of LCPs and PET have been studied by various researchers.^{19,21,22} The results are in qualitative agreement with our own. However, in those methods the orientability of LCPs can be utilized easier and better mechanical properties are achieved.

The tensile strength and elastic modulus obtained for pure LCP were slightly smaller than the corresponding values given by the producer (165 and 9700 MPa). This can be due to the differences in processing conditions or mould geometry, both crucial for the resultant properties of LCP mouldings. It is important to point out that the injection moulding conditions used here were not optimized for every individual blend composition, but similar conditions were used for each blend type.

LCP-PP and LCP-PPS blends showed rather similar trends in mechanical properties as LCP-PET blends. Elastic moduli increased in both blends quite steeply with increasing LCP content and almost according to the rule of mixture. PP lost its high impact strength at higher LCP contents, which also happened to PET. In contrast, the addition of LCP slightly improved the impact properties of PPS. The maximal impact strength for LCP-PPS blends was found at 10 wt % LCP content. However, the impact properties of pure PPS were poor if compared to PP or PET, and the improvement was thus obvious.

Ramanathan et al.³¹ have extruded films made of PPS and LCP blends. They noticed that Vectra, which was thought to be ideally suited for blending with PPS from thermal and rheological considerations, reacted with PPS during extrusion. The blends exhibited a foam structure, and no elongation of the LCP phases was found. The other LCPs of the PHB-PET type formed fibers and enhanced the physical properties of PPS. In our studies no reaction was found between Vectra and PPS, but a fibrous blend morphology and mechanical reinforcement was obtained.

Thermal Properties

The heat deflection temperatures (HDT/A) of the injection-moulded blends are shown in Table IV. LCP increased the heat deflection temperature of

Table IVHDT/A (°C) of theInjection-Moulded Blends

		LCP Fract	ion (wt %)	
Matrix	0	10	30	50
PET	62	62	70	79
PP	52	55	78	110
PPS	108	111	119	147

all the three matrices studied. The most significant effect was found in the LCP-PP blends. For pure LCP a HDT of 154°C was measured.

The DSC curves of the blends are shown in Figure 2, while the DSC data and the crystallinities of the matrices calculated thereof are presented in Table V. The crystallinity of the PET matrix was found to increase, when LCP was added. One possible explanation that was proposed, also by Joseph et al., ¹⁰ could be that LCP can act as a nucleating agent for the crystallization of PET. In the case of PP and PPS this kind of effect was not found.

Crystallinity values of moulded polymers can be calculated according to Khanna³⁷ from the equation

$$W_{c} = \left(1 - \frac{\Delta \log E'(T_{g} - T_{s})}{2, 46}\right) 100$$
 (1)

where $\Delta \log E' =$ change in the storage modulus of the sample between T_g and T_s

 $T_g =$ glass transition temperature

 $T_s =$ glass to rubber transition temperature

The calculated crystallinities of the injectionmoulded blends are shown in Table VI. Because of the limitations in the applicability of the method to crystallizable polymers and possible crystallinity changes during the experiment,³⁷ the values in Table VI should not be taken as absolute values of crystallinity. Besides, unlike for pure polymers, the values for the blends are only calculatory.

In any case, the calculated DMTA crystallinity describes at least approximately the degree of crystallinity in the injection-moulded blends at different blend compositions, and the results can be correlated to mechanical properties of the moulded specimens.

The calculated crystallinities of all blends increased with increasing LCP content. In LCP-PET blends the increase was, however, most significant, ranging from nearly amorphous pure PET to 71%crystalline 50/50 blend. The calculated crystallinities of LCP-PP and LCP-PPS blends did not change so dramatically, but the level was higher due to higher crystallinities of PP and PPS matrices. The increase in the calculated crystallinities of the blends can be correlated to their physical properties, especially to the increase in stiffness (Table III). The exceptionally high values of calculated crystallinity of LCP-PET blends could again be due to the nucleating effect of LCP for PET.¹⁰

The linear thermal expansion coefficients for LCP and the blends are shown in Table VII. The

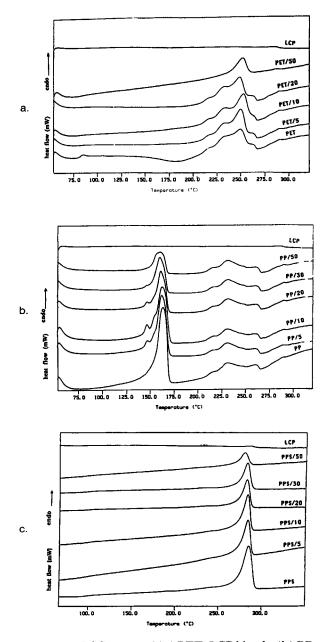


Figure 2 DSC curves of (a) PET-LCP blends, (b) PP-LCP blends, and (c) PPS-LCP blends.

Table V	Melting Temperature, the Heat of
Melting,	and the Crystallinity of Extruded
PET-, P	P–, and PPS–Matrices with Different
LCP Con	tents

	T_m	ΔH	W_{c}
Matrix/LCP %	(°C)	(J/g)	(%)
PET	252	81	58
PET/5	252	76	55
PET/10	250	82	60
PET/20	251	84	61
PET/30	251	92	66
PET/50	249	117	85
PP	163	91	56
PP/5	162	91	56
PP/10	162	113	69
PP/20	161	95	58
PP/30	160	87	54
PP/50	160	83	51
PPS	285	31	_
PPS/5	285	27	_
PPS/10	285	31	
PPS/20	284	28	_
PPS/30	285	28	
PPS/50	283	20	

measurements were made in the flow direction of the injection-moulded specimens, from -80 to +70 °C. Pure LCP exhibited a very low positive thermal expansion coefficient and caused also a clear reduction in the thermal expansion behavior of the blends.

Table VI Crystallinities of Injection-Moulded PET-, PP-, and PPS-LCP Blends Calculated using Eq. (1)

		T_{g}	T_s	W _c
Matrix/LCP %	$\Delta \log E'$	(°Č)	(°C)	(%)
LCP	0.33	74	127	87
PET	2.39	60	95	3
PET/5	2.42	60	100	2
PET/20	1.67	67	99	32
PET/50	0.71	70	95	71
PP	1.11	-5	120	55
PP/5	1.19	-7	124	52
PP/20	1.03	-4	136	58
PP/50	0.86	1	148	65
PPS	0.85	83	148	65
PPS/5	0.79	89	142	68
PPS/20	0.65	90	137	74
PPS/50	0.46	82	135	81

Matrix/LCP %	$lpha (10^{-5} \ { m K}^{-1})$
LCP	0.1
PET	5.0
PET/10	4.1
DET/20	3.0
PET/30 PP	5.8
PP/10	4.5
PP/30	3.4
PPS/	3.6
PPS/10	3.3
PPS/30	3.1

Table VIILinear Thermal ExpansionCoefficients of PET-, PP-, andPPS-LCP Blends

Morphology

As the polymers are not miscible, the adhesion between the two separate phases affects the properties of the blend. LCP does not normally mix well with thermoplastics and thus the blends consisted of two separate phases. The LCP phase appeared in the matrix in the form of small spheres or fibers depending on the LCP content and the orientation during processing.^{8,38-41}

Mechanical and dynamic mechanical properties increased when fiber formation increased (Fig. 1).

The fractured surfaces of the extruded specimens are shown in Figures 3, 4, and 5. At an LCP content of 5% there was no orientation in LCP-PET and LCP-PP blends, and the LCP phases occurred as small spheres. In PPS matrix, however, LCP formed slightly deformed ellipsoidal domains also at 5% content. Mechanical properties decreased at an LCP content of 5–10% because LCP plasticized the matrix.²³ When LCP content reached 20%, this resulted in many fibrils orientated in the direction of the flow, more in PET-LCP blends than in the two other blend types.

The diameters of the LCP domains in LCP-PET and LCP-PPS blends at lower LCP contents were about 1-2 μ m. In PP matrix LCP formed significantly greater domains at all compositions.

CONCLUSIONS

A polyester-type totally aromatic thermotropic main-chain LCP was melt blended with various thermoplastics and the blends were injection moulded. LCP was found to act as a reinforcement in the matrix polymers and to improve their dimensional and thermal stability.

The linear thermal expansion coefficients of all matrices reduced and the heat deflection temperatures increased by LCP addition.

The stiffness of the thermoplastics increased with increasing LCP content. But at the same time the blends became more brittle than the matrices. This brittleness was assumed to be due to the poor in-

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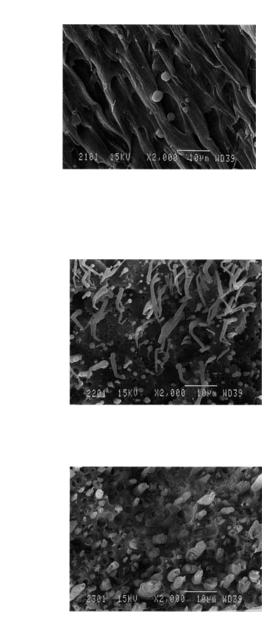
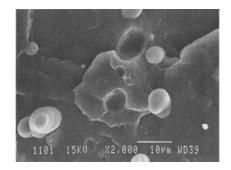
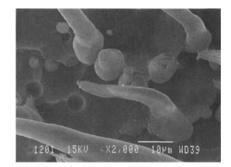


Figure 3 SEM images of the fractured surfaces of extruded samples of PET-LCP blends: (a) PET/5% LCP, (b) PET/20% LCP, and (c) PET/50% LCP.

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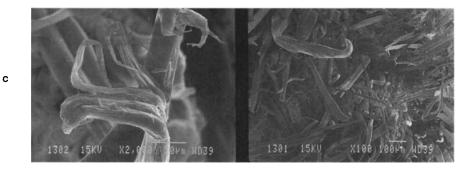


Figure 4 SEM images of the fractured surfaces of extruded samples of PP-LCP blends: (a) PP/5% LCP, (b) PP/20% LCP, and (c) PP/50% LCP.

terfacial adhesion between the LCP and the matrix polymers.

The blends are two phase systems with properties dependent on miscibility, interfacial adhesion between the blend components, and orientation of the LCP phases.

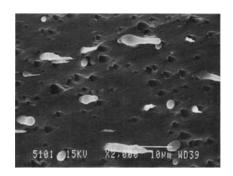
The orientation of the LCP phases has a considerable effect on the growth of the crystallinity of the matrix and the blend; and, together with the LCP amount, it affects the mechanical properties of the blends.

The toughness of the blends could be improved by using small amounts of suitable compatibilizers in order to improve the interfacial adhesion between the blend components. In this work no compatibilizers were used, but the subject is being studied.

Nomenclature

DMTA	Dynamic mechanical thermal analysis
DSC	Differential scanning calorimetry

- LCP
- Liquid crystalline polymer



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Figure 5 SEM images of the fractured surfaces of extruded samples of PPS-LCP blends: (a) PPS/5% LCP, (b) PPS/20% LCP, and (c) PPS/50% LCP.

PA	Polyamide
PBT	Poly(butylene terephtalate)
PC	Polycarbonate
PEEK	Poly(ether-ether ketone)
PEI	Poly(ether imide)
PES	Poly(ether sulphone)
PET	Polyethylene terephtalate
PHB	Para-hydroxy benzoic acid
PP	Polypropylene

PPS	Polyphenylene sulfide
PSU	Polysulphone
DITO	

- PVC Poly(vinyl chloride)
- SEM Scanning electron microscopy

Symbols

- E Elastic modulus (MPa)
- *E'* Storage modulus (MPa)
- ΔH Heat of melting (J/g)
- HDT Heat deflection temperature (°C)
- P_{hold} Holding pressure (of the maximal pressure 2020 bar)
- $P_{inj.}$ Injection pressure (of the maximal pressure 2020 bar)
- t Time (h)
- $t_{\rm cool}$ Cooling time (s)
- $t_{\rm hold}$ Holding time (s)
- T Temperature (°C)
- $T_{\rm cyl.}$ Cylinder temperature (°C)
- T_g Glass transition temperature (°C)
- T_m Melting temperature (°C)
- T_{mould} Mould temperature (°C)
- T_s Glass to rubber transition temperature (°C)
- W_c Crystallinity (%)
- $\begin{array}{ll} \alpha & \text{Linear thermal expansion coefficient (1/K)} \\ \end{array}$

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Received November 21, 1990 Accepted April 24, 1991