

# Comparison of Structure and Properties of Conventional and “High-Crystallinity” Isotactic Polypropylenes and Their Blends with Metallocene-Catalyzed Linear Low-Density Polyethylene. I. Relationships between Rheological Behavior and Thermal and Physical Properties

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Received 18 August 1999; accepted 24 January 2000

**ABSTRACT:** The present study was conducted to compare the structure and properties of conventional and so-called “high-crystallinity” (hcr) polypropylene (PP) and to establish characteristic features of the latter that are responsible for its superior thermal and mechanical performance. Moreover, structure–properties relationships of hcr PP blends with metallocene-catalyzed, linear low-density polyethylene (mLLDPE) were compared with those of conventional PP/mLLDPE blends. In Part 1, relationships between rheological behavior (viscosity and melt density) and thermal (transition temperatures and level of crystallinity) and mechanical properties (impact strength and Young’s modulus) were analyzed with reference to composition. The rheological and MDSC tests showed that both types of the blends were miscible at the processing temperatures, whereas immiscible in the solid state and in vicinity of the PP melting point. It was found that the improved mechanical properties and the extraordinary high crystallization temperature of hcr PP (and, correspondingly, hcr PP/mLLDPE blends) are not due to the assumed high level of crystallinity but due to alteration of internal structure of this polypropylene. © 2000 John Wiley & Sons, Inc. *J Appl Polym Sci* 77: 1591–1599, 2000

**Key words:** level of crystallinity, stiffness-toughness balance, polymer blends

## INTRODUCTION

“High crystallinity” (hcr), high stiffness, “enhanced” grades of polypropylene (PP) produced by

conventional catalysts are novel, developing materials with growing popularity. Chisso (Tokyo, Japan) was the first into the market. Nowadays, PCD Polymere (Austria), Solvay (Belgium), Amoco (USA), Borealis (USA), and DSM (Netherlands) produce a wide range of hcr PP with better (by up to 40% compare to conventional PP) stiffness and strength, thermal resistance, gloss, surface hardness, strain resistance, and water vapor transmission rate. Also, hcr PP was reported to have better flow performance than 20% talc-filled

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Contract grant sponsor: Royal Melbourne Institute of Technology and Cooperative Research Centre for Polymers (NK).

*Journal of Applied Polymer Science*, Vol. 77, 1591–1599 (2000)  
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**Table I Physical Characteristics of the Raw Materials**

Materials	Grade	MFI (g/10 min)	Density at 23°C (kg/m <sup>3</sup> )	Young's Modulus (MPa)	Yield Stress (MPa)	Impact Strength (J/m)	$T_m$ (°C)	$T_{cr}$ (°C)	$T_g$ (°C)
PP	K2XMOD	8.0	905.0	1815	38.5	28.5	169.1	139.0	—
PP	GWM 22	4.0	905.0	1500	35.5	24.5	166.3	122.5	—
mLLDPE	ENGAGE EG 8200	5.0	870.0	12.8	2.3	—	—	—	-52.1

conventional PP with comparable rigidity and impact resistance. Tests carried out by DSM (Sitard, Netherlands) indicated that for materials with any given melt flow index, the Charpy impact resistance of hcr PP at -40°C was several points above that of the conventional talc-filled grade.<sup>1</sup>

Common to all enhanced PPs is the use of the upgraded fourth version of the resin's catalysts. This fourth generation of Z-N catalysts was claimed to be superactive, yield high crystallinity, and permit precise comonomer insertion in copolymer.<sup>2</sup>

However, there is no published literature data on the internal structure of the pure hcr polypropylene nor on its blends. The present study was conducted to trace characteristics and morphological features of hcr PP, that are responsible for its superior thermal and mechanical performance. As a "reference," a grade of conventional isotactic PP was used. Also, the structure-property relationships of blends of hcr PP with metallocene-catalyzed linear low-density polyethylene (mLLDPE) were studied over a wide range of compositions and compared to those of blends of conventional PP and mLLDPE.

## Experimental

### Materials

In the current study two different types of isotactic polypropylene homopolymers were used: the so-called hcr ("high stiffness" or "enhanced") PP, K2XMOD, supplied by PCD Polymere (Austria) and a conventional grade of PP, GWM 22, supplied by ICI Australia.

A grade of metallocene-catalyzed ethylene-octene copolymer, ENGAGE EG 8200, referred to as mLLDPE, with a melt flow index (MFI) similar to the PPs was chosen as the potential impact modifier of the polypropylenes. It was

supplied by Dow Plastics (Dow Chemical Company).

The main physical characteristics of these materials are listed in Table I.

### Blends and Sample Preparation

Blends were prepared by extrusion and test samples by injection molding. The set up was identical to that described elsewhere.<sup>3</sup>

### Mechanical Characterization

Values of Young's modulus (MPa) from tensile testing were used to characterize stiffness of the blends. Testing of the samples was performed using an Instron 4467 tensile testing machine according to ASTM D 638-87b. The interval of a standard deviation was 40–80 MPa.

Values of impact strength (J/m) from Notched Izod Impact testing were chosen to characterize toughness of the PP/mLLDPE blends. Testing was carried out using a Davenport Izod impact tester according to ASTM D 256-93a. Because the absolute values of the standard deviation differed notably, the accuracy of the results was estimated as a percent of error in proportion to the absolute value of impact strength for an individual blend. For all the blends, the calculated error was 5–10%.

### Rheological Tests

MFI and melt density of the blends as well as pure materials were defined using a Ceast MFI tester. The tests were performed according to ASTM D 1238-90b with one exception: the test temperature was set at 220°C. This temperature was chosen earlier as the temperature of blends preparation (extrusion) and processing (injection moulding)<sup>4</sup>. The detailed description of selection of the rheological parameters are given in the same reference.

The influence of composition on melt behavior of the prepared blends was also studied at the processing range of shear rates, that is, at high shear rates, using a Capillary Haake Viscometer (Rheocord 90). Shear rate/viscosity data were collected and processed automatically. Data collection ("Measurement") was performed according to "Instruction Manual Capillary Software, Version 2.61," 1993, provided by the producer of the Rheocord 90 (Haake Company, Saddle Brook, NJ). All measurements were performed at 220°C.

### Modulated Differential Scanning Calorimetry

All tests were performed on pieces of injection moulded samples. A TA Instruments (New Castle, DE) modulated differential scanning calorimeter (MDSC) type 2920 was used to record the thermograms. The heating (cooling) rate was 2°C/min, the modulation amplitude was 0.212°C, and the modulation period was 40 s.

The level of crystallinity of pure PP was calculated from the enthalpy of crystallization as follows:

$$\begin{aligned} \% \text{ Crystallinity} &= \frac{\Delta H_{cr\text{-experiment}} \text{ (J/g)}}{\Delta H_{100\% \text{ crystallinity}} \text{ (J/g)}} \times 100 \\ &= \frac{\Delta H_{cr\text{-experiment}} \text{ (J/g)}}{209 \text{ (J/g)}} \times 100 \quad (1) \end{aligned}$$

where 209 J/g is the theoretical enthalpy of fusion of 100% crystalline PP.<sup>5</sup>

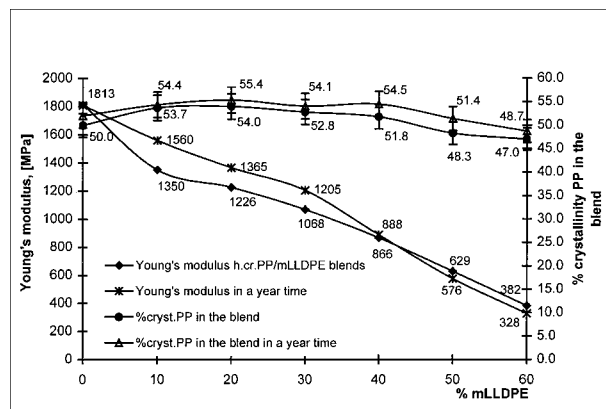
It was found that the studied mLLDPE grade was almost 100% amorphous and thus, it was not expected to contribute to the blend's crystallinity. In other words, total crystallinity of the PP/mLLDPE blends was "provided" only by PP. Hence, the level of crystallinity of PP in blends was calculated as follows:

% Crystallinity PP in the blend

$$= \frac{\Delta H_{cr(\text{blends})/\text{experiment}} \text{ [J/g]}}{209 \text{ [J/g]} \times X} \times 100 \quad (2)$$

where  $X$  is the nominal mass fraction PP in the blend.

The mean deviation of the glass transition temperature ( $T_g$ ), the melting temperature ( $T_m$ ), and the temperature of crystallization ( $T_{cr}$ ) of the blends was experimentally defined as 0.2°C. The



**Figure 1** Influence of composition on Young's modulus and the level of crystallinity PP in hcr PP/mLLDPE blends.

reproducibility of the level of crystallinity data was estimated as 5%.

## RESULTS AND DISCUSSION

### Correlation MDSC and Mechanical Test Data

In the present study, a series of MDSC tests were performed twice: a week after and a year after the sample preparation. This was carried to determine the blends' stability over time.

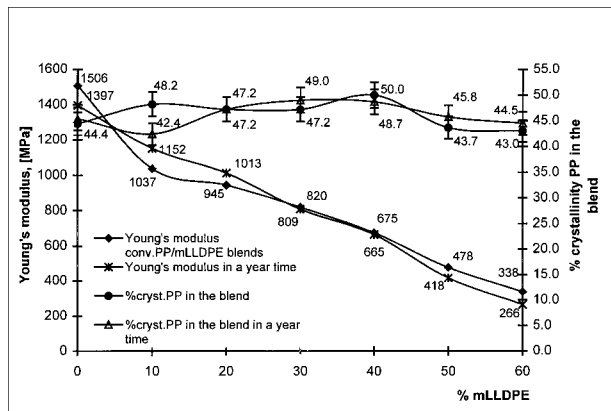
The  $T_g$ , the  $T_m$ , and the  $T_{cr}$  of the blends and the pure materials were characterized. Previous works<sup>6,7</sup> showed shifts in these temperatures in blends of iPP/LLDPE and some partial miscibility of the blends' components in the melt.

Also, a relationship between the level of crystallinity and physical properties of the blends was expected: several authors reported that a drop in the level of crystallinity of PP provided an increase in toughness (ductility) of its blends with other polyolefins.<sup>8–10</sup>

### Comparison of Level of Crystallinity: Mechanical Properties Relationships for Conventional and hcr PP and Their Blends

Results showing the relationships of composition, level of crystallinity of PP in the blend and mechanical properties are shown in Figures 1–4. They are as follows:

1. Values of Young's modulus of the hcr PP blends are higher by about 20–25% than those of the conventional PP blends. The same difference

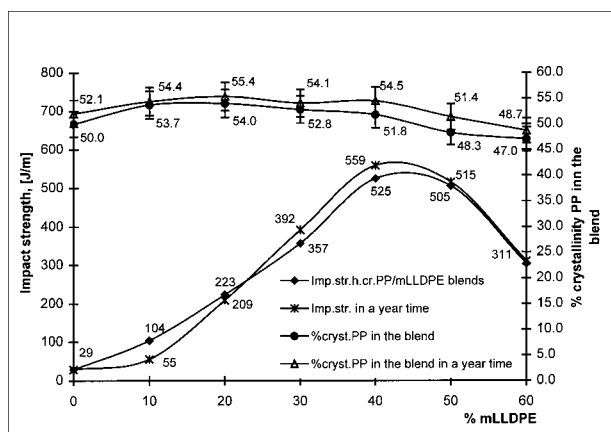


**Figure 2** Influence of composition on Young's modulus and the level of crystallinity PP in conventional PP/mLLDPE blends.

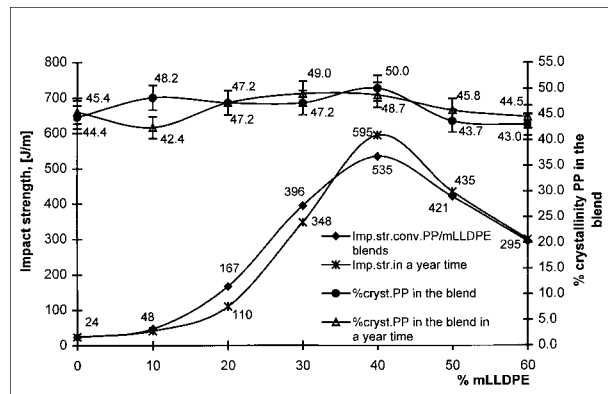
in modulus was established previously for the pure PPs (see Table I).

2. The level of crystallinity of the hcr PP and its blends is only about 5% higher than that of the conventional PP and conventional PP/mLLDPE blends.

3. The level of crystallinity of PP in the blends seems to be independent of composition for both of the hcr PP/mLLDPE and conventional PP/mLLDPE systems. The small variations in values of crystallinity are considered to be insignificant (Variations observed in the results are about 5–8% from the absolute value of the total heat flow. It was reported<sup>11</sup> that the best reproducibility of the total heat flow for MDSC is 3%. In Figs. 3–6, error bars on the Y-axes on the “level of



**Figure 3** Influence of composition on impact strength and the level of crystallinity PP in hcr PP/mLLDPE blends.



**Figure 4** Influence of composition on impact strength and the level of crystallinity PP in conventional PP/mLLDPE blends.

crystallinity” curves correspond to the 5% error, estimated for the present study.).

4. For both types of systems studied, the values of Young's modulus and impact strength showed no correlation with the level of crystallinity. It seems that it is not the higher level of crystallinity but something else that provides the improved mechanical properties of hcr PP and its blends. The authors think that this improvement might be due to the reinforcing effect of the nucleating agents present in hcr PP.<sup>12</sup>

**Characterization of Transition Temperatures of PPs and Their Blends**

*Glass transition temperature.* The blends with 40%, 50%, and 60% of mLLDPE phase and either type of PP exhibited the  $T_g$  of the pure mLLDPE (see Table I) at  $-52.1 \pm 0.2^\circ\text{C}$ , the position of which was little affected by the presence of PP at this range of mLLDPE. This suggests that the amorphous phase of the mLLDPE does not contain PP in these compositions at low temperatures. Immiscibility of PP/LLDPE as well as PP/mLLDPE was claimed by a number of authors.<sup>13–20</sup>

For the blends with a higher content of PP phase, it was not possible to define the  $T_g$  because of limitations of the MDSC method—it became undistinguishable.

Results obtained from determination of the influence of composition on the melting and crystallization temperatures are shown in Tables II and III and Figures 5 and 6. According to the definition used in DSC techniques, “onset” is the intersection of the slope of the melting/crystallization

**Table II Influence of Composition on the Melting and Crystallization Temperatures of hcr PP/mLLDPE Blends**

mLLDPE (%)	$T_m$ (°C)	$T_m^*$ (°C)	$T_m$ (onset) (°C)	$T_m^*$ (onset) (°C)	$T_{cr}$ (°C)	$T_{cr}^*$ (°C)	$T_{cr}$ (onset) (°C)	$T_{cr}^*$ (onset) (°C)
0	169.1	170.2	161.0	158.3	139.0	139.2	142.0	142.1
10	168.8	170.0	160.8	159.0	139.0	139.4	141.8	142.4
20	169.0	169.9	160.3	158.4	138.8	139.3	141.7	142.2
30	168.8	169.8	160.5	158.7	138.6	139.0	141.5	142.1
40	169.0	169.8	160.5	160.7	138.5	138.9	141.5	141.9
50	169.0	169.6	161.0	161.0	138.2	138.6	141.2	141.5
60	169.1	169.5	160.9	161.0	138.0	138.5	140.5	141.0

peak with the base line. Overall, both time and composition affect the transition temperatures of both PPs in slightly different ways, discussed below. The major difference in the thermal behavior of hcr PP and conventional PP is that the  $T_{cr}$  of pure hcr PP is very high compared with that of conventional PP. The tendency remains the same for their blends with the same grade of mLLDPE.

#### Changes over Time for hcr PP/mLLDPE Blends

It seems that  $T_m$  increased and  $T_m$ (onset) decreased in proportion to the content of hcr PP in the composition. These changes imply that an original melting peak has transformed to a broader one with time. Moreover, the increase in melting temperatures, which is inversely proportional to the mLLDPE content and which is not accompanied by a detectable increase in the crystallinity, indicates that some recrystallization or reorganization occurred in the blends over the 1-year period.

As expected, no change in the crystallization temperatures was recorded for these blends: in a

MDSC run, crystallization takes place after melting, which erases any transformations the phases underwent because of aging.

#### Changes over Time for conventional PP/mLLDPE Blends

As for the hcr PP/mLLDPE blends, the  $T_{cr}/T_{cr}$ (onset) did not change over time for conventional PP/mLLDPE blends because of the reason discussed above.

Both the  $T_m$  and  $T_m$ (onset) increased over time in proportion to the content of PP in the composition. This indicates that although further recrystallization of the PP phase took place over time, as in the hcr PP/mLLDPE blends, the conventional PP phase did not recrystallize to the thicker lamellae as was the case for the former.

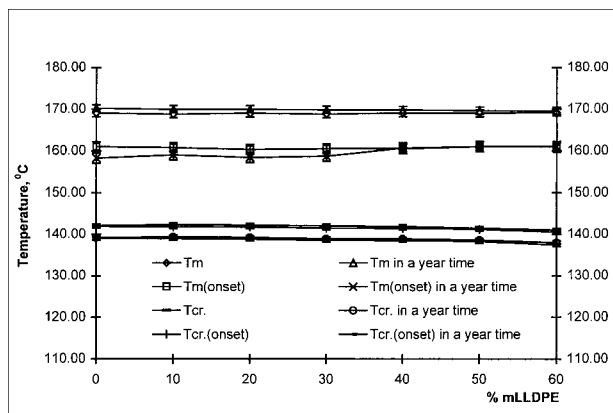
#### Changes with Compositions for hcr PP/mLLDPE Blends

It appears that the  $T_m/T_m$ (onset) and the  $T_{cr}/T_{cr}$ (onset) values for pure hcr PP were only

**Table III Influence of Composition on the Melting and Crystallization Temperatures of Conventional PP/mLLDPE Blends**

mLLDPE (%)	$T_m$ (°C)	$T_m^*$ (°C)	$T_m$ (onset) (°C)	$T_m^*$ (onset) (°C)	$T_{cr}$ (°C)	$T_{cr}^*$ (°C)	$T_{cr}$ (onset) (°C)	$T_{cr}^*$ (onset) (°C)
0	166.3	167.4	158.3	159.3	122.5	122.8	127.5	126.5
10	166.3	167.1	158.4	159.1	124.1	124.5	127.6	128.0
20	166.0	166.9	158.0	159.4	124.7	125.4	128.1	128.9
30	165.8	166.5	158.2	159.5	125.8	125.1	128.9	128.3
40	165.8	166.0	157.4	157.6	122.7	122.7	126.4	126.3
50	166.0	166.0	158.0	158.2	123.0	123.3	126.5	126.5
60	165.6	165.7	156.6	157.1	122.4	122.7	126.5	126.4

$T_m/T_{cr}$  and  $T_m^*/T_{cr}^*$  are the melting/crystallization temperatures of "fresh" and "aged" for a year samples, respectively; and the  $T_m$ (onset)/ $T_{cr}$ (onset) and the  $T_m^*$ (onset)/ $T_{cr}^*$ (onset) are the onsets of these temperatures.



**Figure 5** Influence of composition on melting and crystallization temperatures of hcr PP/mLLDPE blends.

slightly depressed by the presence of mLLDPE. These changes might suggest very limited solubility of hcr PP in mLLDPE. It is rather unusual that  $T_{cr}(\text{onset})$ , which is a measure of nucleation rate, shows no dependence on compositions. We might explain this by the following assumption: the crystallization of the hcr PP is nucleated [ $T_{cr}(\text{onset}) > 140^{\circ}\text{C}$ ] and, therefore, it does not follow the homogeneous nucleating scheme.

#### Changes with Compositions for conventional PP/mLLDPE Blends

As for the hcr PP systems, the  $T_m/T_m(\text{onset})$  temperatures for conventional PP/mLLDPE blends decreased slightly with increasing mLLDPE content.

In contrast to the hcr PP blends, there was a pronounced increase in the  $T_{cr}/T_{cr}(\text{onset})$  for the blends with a lower mLLDPE content, up to 30% in the composition. It appears to be an interface effect as with an increase of concentration of the dispersed phase the interface (nucleating area) would become larger, leading to the higher crystallization temperatures. For compositions with a higher content of mLLDPE (i.e., with 40%, 50%, and 60%), values of  $T_{cr}/T_{cr}(\text{onset})$  were similar to those of the pure PP. This indicates that the main structural type has changed from a dispersed matrix to a co-continuous type with an increasing concentration of the second phase in these systems.<sup>12</sup>

From the MDSC results, we can conclude that blends of hcr or conventional PP and mLLDPE with 40%, 50%, and 60% of the latter, appear to be immiscible in the solid state and at temperatures

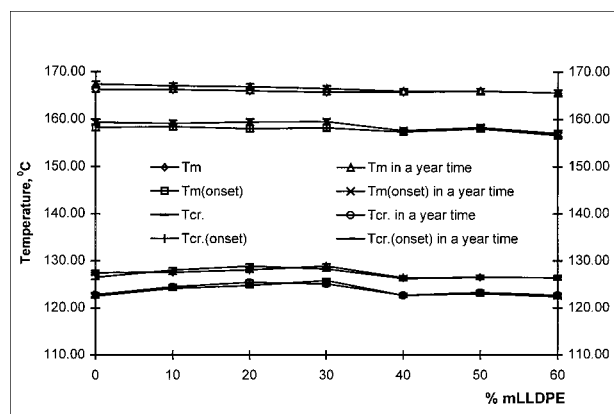
equal and below the melting temperature of PP (165–170°C). However, it has to be emphasized that the apparent immiscibility detected and claimed in the solid state for these compositions is unrelated to immiscibility of these blends at higher temperatures. Blends with a lower content of mLLDPE, especially conventional PP—based blends, might be considered to be partially miscible, although further verification of this hypothesis is required.

#### Rheological Study of Conventional and hcr PP and Their Blends

The values of MFI and melt density of the blends as well as pure materials are presented in Tables IV and V.

Overall, data from Tables IV and V show that (1) MFI of blends increased with increasing mLLDPE content in nearly an additive manner, which is as expected; (2) melt density was independent of composition across the whole concentration range; and (3) values of melt density for hcr PP/mLLDPE and conventional PP/mLLDPE blends were almost equal to the melt density of the pure polypropylene and did not depend on the type of PP.

That the values of blends melt density are unchanged across the whole composition range is unexpected: MDSC data showed apparent absence of miscibility in compositions with higher content of mLLDPE in the solid state and, also, in the vicinity of PPs' melting point. However, if the blends' components were immiscible in the melt at the temperature 220°C as well, values of melt densities for the blends would be additive. Un-



**Figure 6** Influence of composition on melting and crystallization temperatures of conventional PP/mLLDPE blends.

**Table IV Melt Flow Index and Melt Density Data for hcr PP/mLLDPE Blends**

Composition (PP/ mLLDPE)	100/0	90/10	80/20	70/30	60/40	50/50	40/60	0/100
MFI (g/10 min)	6.95	7.05 (7.27)	7.28 (7.59)	7.67 (7.92)	7.91 (8.24)	8.24 (8.56)	8.65 (8.88)	10.17
Melt density (g/cm <sup>3</sup> )	0.75	0.74	0.74	0.74	0.75	0.74	0.75	0.80

The values of MFI given in parentheses represent numbers calculated according to the additive rule using the weight fractions of the blends' components.

changed melt density data indicate that the blends were miscible or partially miscible in the molten state, at the temperature of the MFI test conducted. In this case, miscibility of the systems would not be detected by MDSC because this technique does not provide any information about the blends' state above melting of the PPs.

It was recommended<sup>21</sup> to conduct rheological measurements of immiscible polymer blends under conditions (temperature, flow field type, shear rate, and residence time) approximating as closely as possible the conditions anticipated during processing. Hence, further rheological study was conducted using a Capillary Haake Viscometer because it is closer to processing conditions for PP, PE, and their blends in industry in terms of shear rate.

Experimental viscosity data at 220°C from capillary rheometry are presented in Figures 7 and 8.

Both types of blends as well as their constituents demonstrate shear thinning behavior. Technically this means that for a given force or pressure more material can be made to flow or the energy can be reduced to sustain a given flow rate. Shear thinning behavior (pseudoplasticity) is common for polymers and can be explained by orientating, disentangling, and stretching effects of flow on long-chain polymer macromolecules.<sup>21</sup>

Further analysis was carried out to investigate the effect of composition on viscosity of the blends studied (Figs. 9, 10). Solid lines in the figures represent the additive rule (eq. 3):

$$\log(\eta_{\text{blend}}) = \sum w_i \log \eta_i \quad (3)$$

where  $\eta_{\text{blend}}$  and  $\eta_i$  are viscosities of a blend and its components  $i$ , respectively, and  $w_i$  is the weight fraction of component  $i$ .

The additive blends' viscosity values were calculated using experimental viscosity data for pure components at three shear rates (100, 1000, and 10,000 1/s). These calculated values of the blends' viscosities were compared with measured experimental data.

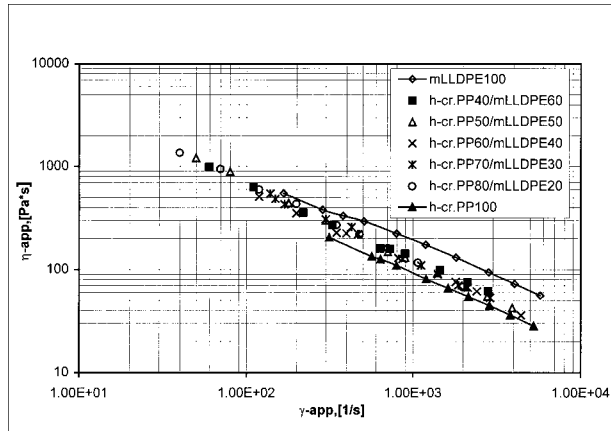
Overall, the following conclusions were made from capillary rheological test results under high shear rates (Figs. 7–10): (1) for hcr PP/mLLDPE and conventional PP/mLLDPE blends, experimental values of viscosity do not depend on composition and, correspondingly, do not follow the rule of mixture at the shear rates of the experiment; (2) the slopes of the blends' viscosity curves are different from the slope of viscosity curves of their components: (a) at relatively low shear rates (about 100 1/s) the viscosities of the blends are close to the viscosity of mLLDPE and (b) at higher share rates, from about 1000 1/s, the blends' viscosities are very close to the viscosity of pure PP.

The difference in slopes of the viscosity curves for pure materials and their blends shows, as for melt density, that the blends do not have the same liquid structure as either of their components. The authors suggest that the absence of additivity for values of the blends' viscosities at

**Table V Melt Flow Index and Melt Density Data for Conventional PP/mLLDPE Blends**

Composition (PP/ mLLDPE)	100/0	90/10	80/20	70/30	60/40	50/50	40/60	0/100
MFI (g/10 min)	5.00	5.24 (5.52)	5.90 (6.03)	6.42 (6.55)	6.88 (7.07)	7.48 (7.59)	7.90 (8.10)	10.17
Melt density (g/cm <sup>3</sup> )	0.74	0.72	0.73	0.75	0.74	0.75	0.74	0.80

The values of MFI given in parentheses represent numbers calculated according to the additive rule using the weight fractions of the blends' components.



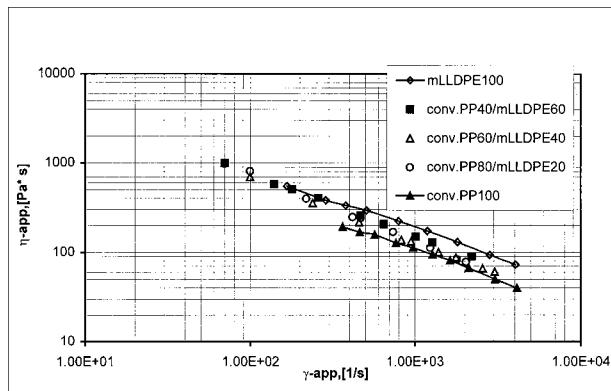
**Figure 7** Influence of composition on viscosity of hcr PP/mLLDPE blends at high shear rates.

high shear rates is due to the presence of miscibility in the systems under the conditions of the experiment conducted.

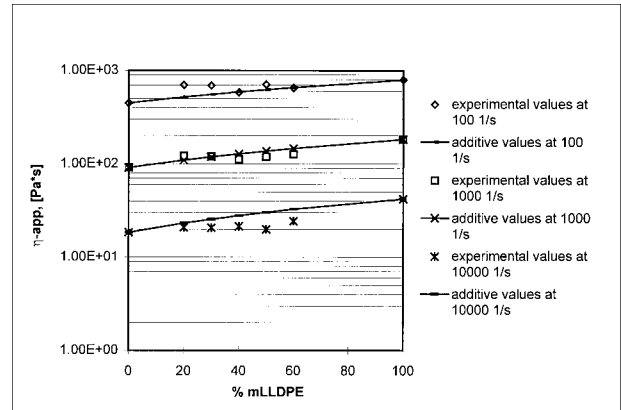
**CONCLUSIONS**

1. From rheological (nonadditive melt density and viscosity data) and MDSC (transition temperatures data) tests it seems that the blends' components in both types of systems appear to be miscible at the temperatures and shear rates used for processing, and then, the phases separate upon cooling, being immiscible in the solid state and in vicinity of the PP melting point.

2. Both types of blends in this study demonstrate linear reduction in stiffness and remarkable nonlinear increase in toughness. The hcr PP blends provide a significant increase in a stiff-



**Figure 8** Influence of composition on viscosity of conventional PP/mLLDPE blends at high shear rates.

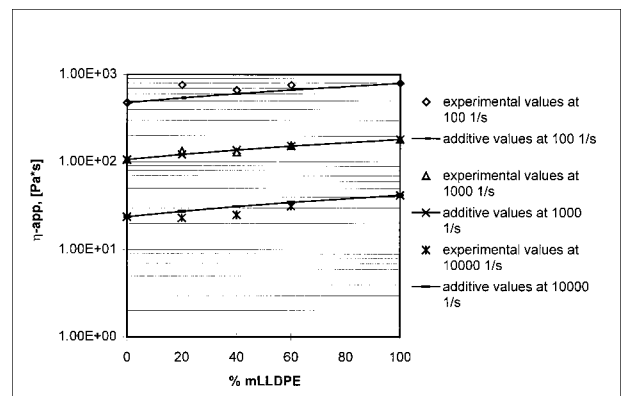


**Figure 9** Comparison of experimental and additive values of  $\eta_{app}$  at high shear rates, hcr PP/mLLDPE blends.

ness/toughness ratio for all blend levels compared to conventional PP because of the originally higher value of modulus.

3. Improved mechanical properties and an extraordinary high crystallization temperature of hcr PP (and, correspondingly, hcr PP/mLLDPE blends) are not due to the assumed high level of crystallinity but to alteration of internal structure of this polypropylene. This alteration is probably achieved by incorporation of nucleating agents in the composition. A further morphological study is being currently undertaken to clarify this conclusion (Part 2).<sup>12</sup>

The authors thank the Polymer Technology Centre within RMIT for provision of industrial processing and testing equipment.



**Figure 10** Comparison of experimental and additive values of  $\eta_{app}$  at high shear rates, conventional PP/mLLDPE blends.



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