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Rheological Behaviour of Polypropylene/Boehmite Composites, Structure and Properties of Hybrid Fibres

Abstract

In this paper, the effect of the re-melting and kneading of polypropylene (PP)/Boehmite composites in a twin-screw extruder on their rheological properties and spinning as well as the effect of the uniaxial deformation of composite fibres on their thermal properties, orientation and mechanical properties are presented. The composite fibres were prepared by the melt spinning of polymer composites based on PP and commercial Boehmites of Disperal mark. The rheological properties of the polymer composites were evaluated using an extruder-capillary rheoviscosimeter. The thermal properties of the PP/Boehmite fibres were investigated by DSC analysis. DSC measurements were carried out using the conventional method (CM) and the constant length method (CLM) in which a constant length of the fibres during measurement was assured. The melting temperature and melting enthalpy of the PP and PP/Boehmite composite fibres increased proportionally with a drawing ratio. These thermal parameters were correlated with orientation and basic mechanical properties, tenacity and Young's modulus of fibres. The tensile strength and Young's modulus of the PP/Boehmite fibres increased proportionally with a higher melting temperature and melting enthalpy obtained by the DSC CLM method.

Keywords: polypropylene, composites, nanofiller, thermal properties, mechanical properties.

Introduction

Recently, many polymer composites have been prepared using an inorganic filler with dimensions on a nanometer scale to improve some properties of the original polymers, such as mechanical, thermal stability, electrical and magnetic properties, among others [1 - 3].

Boehmites are hydrophilic inorganic aluminium oxides based on γ -AlO(OH). They have characteristic hydroxyl groups and their aspect ratio can vary depending on the method of preparation [4]. Boehmites can be obtained as highly pure and highly dispersible colloidal powders in water with primary particle on a nanometer scale [5]. With regard to hydrophilic nature, the Boehmites can also be used in latex and water solutions of polymers [6]. After a suitable surface treatment they are dispersed in organic solvents and become interesting inorganic nanofillers for polymers [7].

In a polymer matrix e.g. in polyamide 6, the Boehmite particles form well defined nanocomposites [7, 8]. They can form a liquid crystalline structure, although the critical concentration for nematic order in polymers is higher than in aqueous solutions [9]. Boehmites in a polymer matrix contribute to their mechanical properties being higher, namely, [6, 10], hydrophilicity [6, 10, 11] and porosity [12]. They can also change their UV barrier properties and photostability [11].

Several papers have reported the positive effect of alumina inorganic nanoparticles

on the wear and friction of the polymers. The addition of inorganic nanoparticles to polyetherether ketone led to the formation of a cohesive and tenacious transfer film on the steel counterface during wear and reduction of the wear rate and coefficient of friction [13]. A similar effect of the alumina nanoparticles was observed on the wear and friction behaviour of polyphenylene sulphide [14] and poly(ethylene) terephthalate [15].

Anisotropic polymer composites, such as fibres and oriented films are peculiar in terms of their structure and properties in comparison with isotropic polymers.

In this paper, the effect of the re-melting and kneading of PP/Boehmite composites in a twin-screw extruder on their rheological and spinning properties as well as the effect of the uniaxial deformation of the composite fibres on their thermal properties, orientation and mechanical properties are studied. Commercial Boehmites, Disperal marks, were used for preparation of the PP composite fibres by melt spinning. The rheological properties of PP/Boehmite composites were evaluated using an extruder-capillary rheoviscosimeter. The thermal properties of the PP/Boehmite fibres were investigated by DSC analysis. Measurements were carried out using two methods, a conventional method (CM) and a constant length method (CLM). The rheological properties of the composites as well as thermal parameters of the fibres were correlated with their basic mechanical properties, such as tenacity and Young's modulus.

Experimental

Material used

Polymers

PPHF 500R (PP500R), MFR 25 g/10 min, flakes and PP Moplen HP561N (PP561N), MFR 11 g/10 min, both produced by BASELL, Polyolefins, Italy.

Boehmite

- Disperal 40 (D40), Al₂O₃ content: 82.5 wt%, surface area 105 m²/g,
- Disperal 60 (D60), Al₂O₃ content: 83.3 wt%, surface area 97 m²/g,
- Disperal modified with undecylenic acid (DUN), Al₂O₃ content: 77.4 wt%, surface area 106 m²/g and Disperal modified by 3-aminopropyltriethoxiloxane (DAM), Al₂O₃ content: 73.1 wt%, specific surface 257 m²/g,

all produced by SASOL, Germany.

Preparation of PP/Disperal composite fibres

Conventional masterbatch technology was used for the preparation of PP/Disperal nanocomposite fibres. The method consists of two steps:

1. Preparation of concentrated dispersion of Disperal particles in PP (masterbatch): The PP500R (flakes) and Disperal were mixed in a high r.p.m mixer for 3 minutes. The powder mixture was melted and kneaded using twin-screw corotating extruders ϕ 28 mm (and ϕ 16 mm). The temperature of the extruded melt was 229 °C (and 220 °C).
2. Melt mixing of fibre-grade polypropylene with the masterbatch in a simple one-screw extruder and spinning of the nanocomposite fibres. The PP500R/Disperal masterbatch was dried at 80 °C for 8 hours. The mixture of PP561N chips and PP500R/Disperal masterbatch was melted and spun using an extruder ϕ 16 mm and spinneret with 11 orifices, d = 0.5 mm. The spinning temperature was 280 °C. The fibres were drawn using a laboratory drawing machine at 120 °C.

Methods used

Rheological properties of the PP/Boehmite composites

The rheological properties of the PP and PP composites were measured using a Götffert N 6967 capillary extrusion meter with extruder ϕ = 20 mm with capillary length 30 mm and diameter 2 mm at 250 °C. Both Newton's and Oswald de Waele's laws were used for the evaluation of basic rheological parameters:

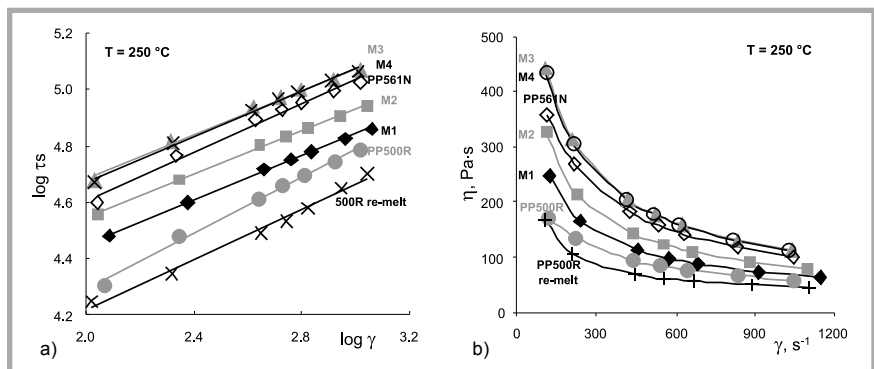


Figure 1. Dependences of the log shear stress τ_s on log shear rate $\dot{\gamma}$ (a) and viscosity η on shear rate $\dot{\gamma}$ (b) for PP composites (masterbatches) at 250 °C; M1 – 10 wt% masterbatch of D40 in PP500R, M2 – re-melted masterbatch M1, M3 – diluted and re-melted masterbatch M2 for final 2.5 wt% of filler by adding of the PP561N, M4 – diluted and re-melted masterbatch M3 for final 1.0 wt% of filler by adding of the PP561N.

apparent viscosity $\eta = \tau/\dot{\gamma}$ and power law exponent n ($\tau = k \cdot \dot{\gamma}^n$), which characterises the non-Newtonian behaviour of the polymer melt, where τ - shear stress, $\dot{\gamma}$ - shear rate, η - apparent viscosity, n - power law exponent, k - coefficient.

DSC analysis

The measurements were performed using a Perkin Elmer DSC 7 in the temperature range of 30 - 220 °C. The standard heating rate was 10 °C/min. The measurements were carried out on cut fibres with a length of 1-2 mm using the method CM and on the fibres with the method CML. Constant length was achieved by winding the fibres on a small spool and they were subsequently placed in a DSC pan. The melting peak temperature T_m and melting enthalpy ΔH_m were evaluated.

Mechanical properties of the fibres

An Instron (Type 3343) instrument was used for the measurement of the mechanical properties of the fibres (according to ISO 2062:1993). Coefficients of variation of tenacity and elongation at break, evaluated from 15 measurements, were used as a measure of the internal (structural) non-uniformity of fibres. The initial length of the fibres was 25 cm and the time of deformation was about 20 sec.

Orientation of fibres

The average factor of orientation of fibres (f_α) was evaluated by sonic velocity measurements using a Dynamic Modulus Tester-PPM-5R. The factor of orientation was calculated according to the equation:

$$f_\alpha = 1 - C_n^2/C^2 \quad (1)$$

where f_α - average factor of orientation, C - sonic velocity of oriented sample and

C_n - sonic velocity of standard isotropic fibre.

Results and discussion

Rheological properties of the PP/D40 masterbatch and tensile properties of the composite fibres

The apparent viscosity of the PP500R/D40 masterbatch (M1) is about 20-30 % higher in comparison with the viscosity of the neat PP500R (Figure 1, Table 1).

Table 1. Power law exponent n and viscosity η for PP and for PP/D40 masterbatch at 250 °C; M1 – 10 wt% masterbatch of D40 in PP500R, M2 – re-melted masterbatch M1, M3 – diluted and re-melted masterbatch M2 for final 2.5 wt% of filler by adding of the PP561N, M4 – diluted and re-melted masterbatch M3 for final 1.0 wt% of filler by adding of the PP561N.

Sample	250 °C	
	n	$\eta, \text{Pa}\cdot\text{s}$ $\dot{\gamma} = 300 \text{ s}^{-1}$
PP500R	0.50	112
PP500R re-melted	0.44	89
PP561N	0.38	223
M1	0.39	145
M2	0.39	178
M3	0.39	245
M4	0.40	243

Table 2. Effect of drawing and annealing on elongation at break of the PP561N/D40 composite fibres F1 - F4, prepared using masterbatches M1 - M4. Fibres were drawn for drawing ratio $\lambda 3$.

Fibres	E, %		
	undrawn	drawn	annealed
PP561N	332	34.9	25.4
F1	334	33.7	24.3
F2	341	36.4	26.5
F3	343	35.3	25.1
F4	418	48.6	33.8

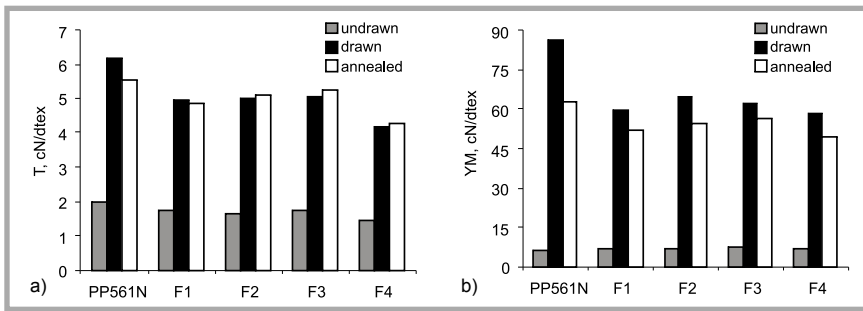


Figure 2. Effect of drawing and annealing on tenacity T (a) and Young's modulus YM (b) of the PP561N/D40 composite fibres F1 - F4 (drawing ratio $\lambda 3$), prepared using masterbatches M1 - M4; M1 - 10 wt% masterbatch of D40 in PP500R, M2 - re-melted masterbatch M1, M3 - diluted and re-melted masterbatch M2 for final 2.5 wt% of filler by adding of the PP561N, M4 - diluted and re-melted masterbatch M3 for final 1.0 wt% of filler by adding of the PP561N.

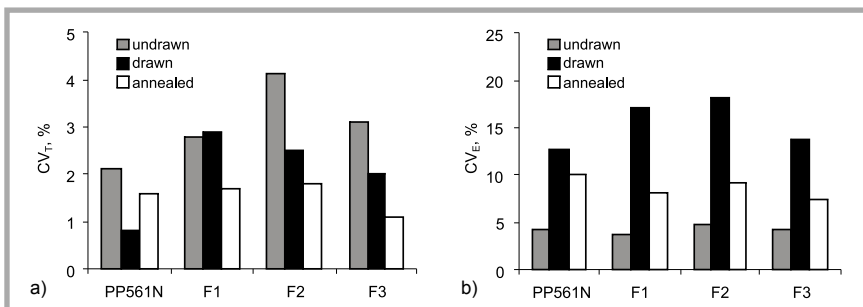


Figure 3. Effect of drawing and annealing on coefficient of variation of tenacity CV_T (a) and elongation at break CV_E (b) of PP561N/D40 composite fibres F1 - F3, prepared using masterbatches M1 - M3.

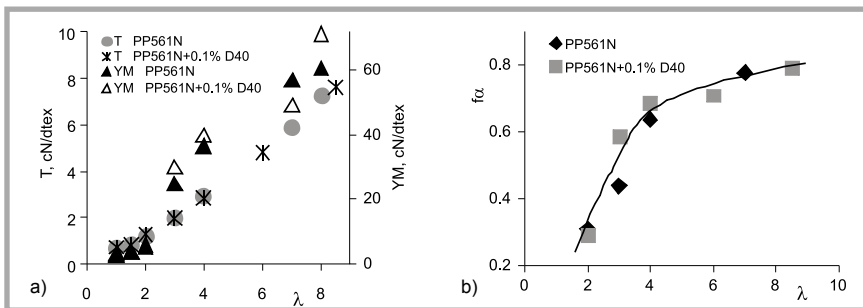


Figure 4. The effect of drawing ratio λ on tenacity T and Young's modulus YM (a) and on average factor of orientation f_a (b) of the PP561N/D40 composite fibres.

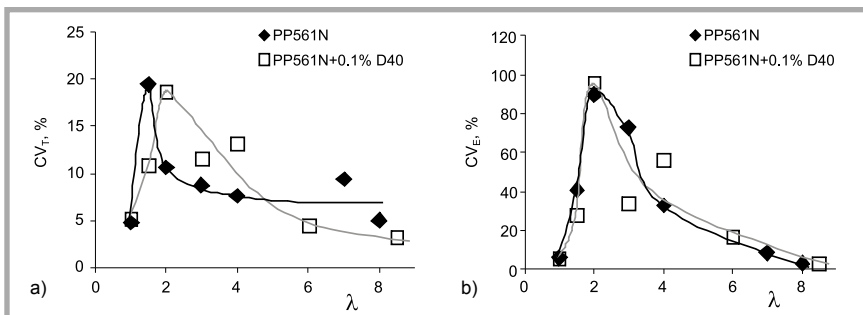


Figure 5. The effect of drawing ratio λ on coefficients of variation of tenacity CV_T (a) and elongation at break CV_E (b) of the PP561N/D40 composite fibres.

Re-melting of the PP500R without a nanofiller in the twin-screw extruder led to a decrease in the PP500R viscosity by about 10% but re-melting of the master-

batch M1 increases its viscosity by about 15%. Likewise, the viscosity of the M3 and M4 masterbatch is clearly higher than the viscosity of the PP561N, in spite of the

low concentration of D40 in the PP (2.5 and 1.0 wt%) and expected degradation of the masterbatch by repeated melting and kneading. An increase in the viscosity of the masterbatch during repeated kneading using the twin-screw extruder could be explained only as a result of the higher degree of disintegration of the D40 particles in the PP matrix. The power law exponent, which characterises a deviation from Newtonian flow, decreased from 0.5 (PP500R) to 0.4 for the M1 masterbatch and did not change with the processing conditions and composition of the M2 - M4 masterbatches. The particles with lower size had a tendency to agglomerate due to their higher specific surface.

The masterbatches M1 - M4 were used for the spinning of PP561N composite fibres, marked as F1 - F4, containing a constant 1.0 wt% of D40 nanofiller. The fibres were drawn at a drawing ratio of $\lambda = 3$ and annealed at 100 °C at a constant length. The Tenacity and Young's modulus of the F1 - F4 composite fibres decrease compared with PP561N fibres, then slightly increase gradually with repeated processing of the masterbatch from M1 to M3 (Figure 2.a, b). The lower tensile properties of the F4 fibres in comparison with the F1 - F3 ones result from the higher elongation at break of all undrawn, drawn and annealed fibres (Table 2). Annealing of the PP561N composite fibres led to a decrease in Young's modulus and tenacity for the neat PP fibres. The decrease in both tenacity and modulus for composite fibres during annealing is reduced gradually with repeated melting of the masterbatch in the twin-screw extruder (Figure 2.b). The Tenacity of the annealed composite fibres exhibited even higher values with repeated melting of the masterbatch (Figure 2.a), which means that the nanofiller retards the relaxation of the macromolecular chains at higher (annealing) temperature. However, processes with a short relaxation time in the amorphous phase were carried out during annealing, which resulted in a decrease in the Young's modulus of the annealed fibres (Figure 2.b).

The variation coefficients of tenacity and elongation for drawn and mainly for annealed fibres have a tendency to decrease with repeated melting of masterbatch (Figure 3). However, their absolute values are relatively high compared to unmodified PP561N fibres. Other trials revealed that higher non-uniformity of

the composite fibres exists in the fibres of middle drawing ratio and with a relative high concentration (1.0 wt%) of the nanofiller (Figure 5).

In any case, repeated mixing and kneading of the PP/Disperal boehmite masterbatch (composites) before spinning plays a positive role from the point of view of the tensile properties of the composite annealed fibres. Based on these results a new series of PP/Disperal composite fibres were prepared and studies of their orientation and mechanical properties are presented in the next chapters.

Effect of the uniaxial deformation on the orientation and mechanical properties of PP/D40 composite fibres

The tensile properties of the composite fibres are strongly affected by both spinning and drawing conditions as well as by the orientation of the structure elements. Many internal and external variables are involved in these processes. The effects of drawing ratio and content of the nanofiller on the tensile and thermal properties of PP561N/D40 composite fibres were investigated in this experimental work. Results, given in Figure 4.a, reveal that the tenacity and Young's modulus of the fibres gradually increases with a higher drawing ratio. The value of maximum drawing ratio for the PP fibres containing 1.0 wt% of D40 was higher than for the unmodified fibres (Figure 4.a). At the same time, the average orientation of fibres exhibits the maximum values (Figure 4.b). The coefficients of variation of the tenacity and elongation at break, which can be considered as a measure of the structural non-uniformity of the fibres, pass through the expressive maximum at a middle drawing ratio and decrease to a minimum for the maximum drawing ratio (Figure 5.a, b). Boehmite D40 facilitates the deformation and orientation of the fibres and contributes to the higher tensile properties of the fibres.

On the basis of these results a series of PP/Disperal fibres were prepared and drawn out at the maximum drawing ratio. The concentration of the nanofiller was within 0.02 - 3.0 wt%. The results obtained indicate that the tenacity and Young's modulus of the fibres increase with a higher content of nanofiller, pass through the maximum and above 0.3 wt% decrease gradually with a higher concentration of boehmites (Figure 6.a, b). The maximum tenacity and modulus of the

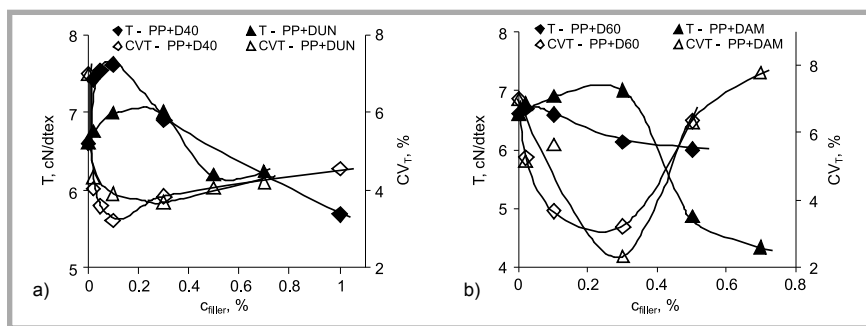


Figure 6. Dependence of tenacity T of PP/Disperal composite fibres and their coefficients of variation CV_T on concentration of Disperal nanofiller for PP/D40 and PP/DUN (a) as well as PP/D60 and PP/DAM (b).

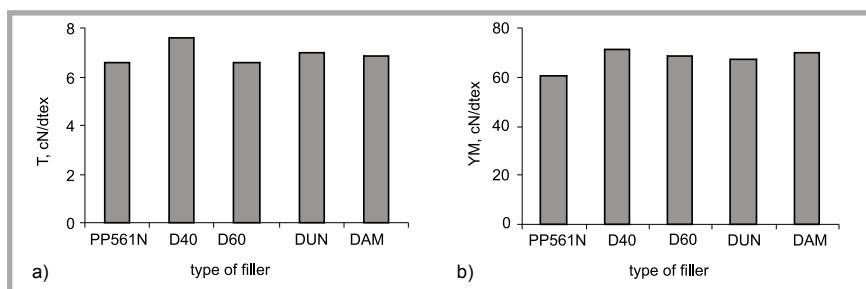


Figure 7. Tenacity T (a) and Young's modulus YM (b) of PP/Boehmite composite fibres containing 0.1 wt% for series of Disperals in dependence on the type of filler for maximum drawing ratio λ_{max} .

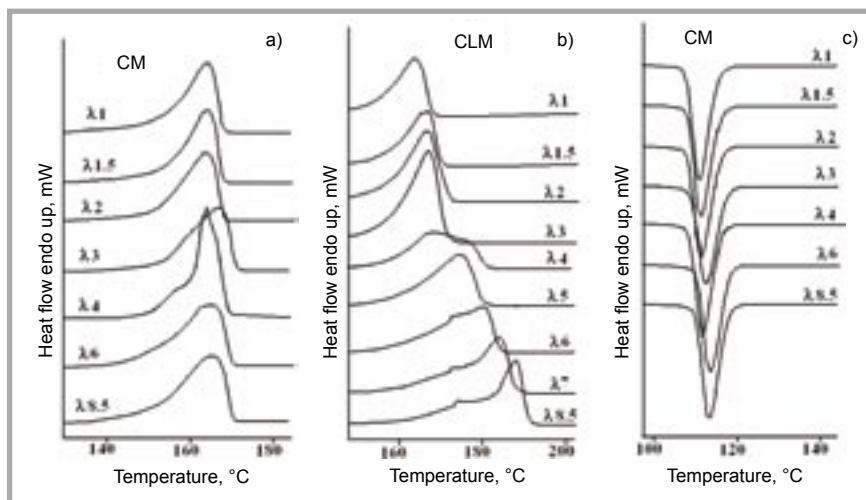


Figure 8. DSC melting (a, b) and crystallisation (c) thermograms of the PP561N/D40 (0.1%) nanocomposite fibres in dependence on drawing ratio obtained by CM and CLM method.

PP composite fibres were obtained at low concentration up to 0.2 wt% for all kinds of Disperals. Modified samples of the Disperal such as DUN and DAM did not provide clearly improved both the spinning or mechanical properties of the fibres in comparison with D40 or D60 nanofiller (Figures 6 and 7). These results indicate a contribution of the nanofiller to the absolute values of the tensile properties of the fibres, mainly for the maximum drawing ratio. This contribution is considered as an effect of the nanofiller to suppress the melt

elasticity in spinning line and facilitate the deformation and orientation of the fibres. The higher structure the homogeneity of the fibres, the lower the coefficient of variation of tenacity CV_T is (Figure 6).

Thermal properties of the PP/Disperal composite fibres, and correlation with mechanical properties

The CM and CLM methods for DSC analysis were used for evaluation of the basic thermal parameters, melting temperature and melting enthalpy of the

Table 3. Melting temperature T_m and melting enthalpy ΔH_m of the PP561N and PP561N/D40 (0.1%) composite fibres measured by CM and CLM method.

drawing ratio λ	PP561N (CM)			PP561N/D40 (CM)			PP561N/D40 CLM	
	T_{m1} , °C	T_{m2} , °C	ΔH_m , J/g	T_{m1} , °C	T_{m2} , °C	ΔH_m , J/g	T_{m1} , °C	ΔH_m , J/g
1.0	-	162.1	86.9	-	164.0	90.1	163.8	84.6
1.5	-	163.0	88.1	-	164.0	91.5	166.8	89.7
2.0	-	162.6	92.9	-	163.8	97.2	166.7	96.8
3.0	157.9	163.3	98.2	159.7	166.8	97.7	167.0	100.1
4.0	154.5	163.6	102.5	156.6	163.8	104.3	168.2	104.5
5.0	-	-	-	-	-	-	174.5	104.8
6.0	-	-	-	-	165.0	106.9	180.2	107.5
7.0	160.4	165.0	106.3	-	-	-	184.0	113.2
8.5	-	-	-	-	165.0	113.2	188.0	114.4

Table 4. Melting temperature T_m and melting enthalpy ΔH_m of the PP561N/Boehmite composite fibres drawn on maximum drawing ratio λ_{max} in dependence on concentration of nanofiller; measured by CLM method. For PP500R fibre was $T_m=181.2$ °C, $\Delta H_m=112.9$ J/g and for PP561N fibre was $T_m=186.2$ °C and $\Delta H_m=106.4$ J/g; *conc.=1.0% D40.

$C_{Dispersal}$, %	D40		D60		DUN		DAM	
	T_m , °C	ΔH_m , J/g	T_m , °C	ΔH_m , J/g	T_m , °C	ΔH_m , J/g	T_m , °C	ΔH_m , J/g
0.02	186.7	110.2	188.5	109.8	187.7	112.0	186.7	111.7
0.10	188.0	114.4	187.3	112.1	188.0	107.9	186.0	111.7
0.30	187.3	111.6	187.2	112.6	185.7	113.0	187.0	109.9
0.50	-	-	182.8	111.1	184.2	112.4	182.7	107.5
0.70	182.7*	106.5*	-	-	186.0	113.8	181.5	110.8
3.00	178.8	102.1	181.8	103.4	183.7	100.4	164.5	56.4

PP/Dispersal composite fibres. A significantly higher melting (peak) temperature at practically the same melting enthalpy was obtained by the DSC CLM methods in comparison with the CM method for oriented fibres above a certain drawing ratio (Figure 8, Table 3). Both methods result in thermograms with a single melting peak for low and high draw-

ing ratio, and double melting peaks for middle drawing ratio (Table 3). For the CM method a change in the crystalline structure was observed for a drawing ratio within 3 to 5 and a structure with a lower melting temperature is developed. This structure is not stable and disappears at higher deformation of the fibres (Table 3). For the CLM method the

change in the crystalline structure of the fibres, characterized by the double endotherm, was found only for a drawing ratio of 4. Above this value, the melting temperature of the fibres markedly increases and crystalline structure is characterized by a single peak at a temperature above 180 °C. The difference between thermograms obtained by the CM and CLM methods can be explained by a change in the crystalline structure of the polymer before melting, which is similar to the crystallisation mechanism from the melt under spinneret at high spinning speed. Axial tension contributes to the orientation of the macromolecule chains and shifts the crystallization temperature to higher values (closer to spinneret).

The melting temperature of the PP/Dispersal composite fibres in the function of the concentration of the nanofiller passes through the maximum within the concentration of 0.1 - 0.3 wt% and decreases due to the higher content in the PP matrix (Figure 9, Table 4). In any case, the highest melting temperatures correspond to the highest tenacity (Figure 6).

Correlations of the thermal analysis results and the average orientation of the fibres reveal that the melting temperature changes radically above an orientation factor $f(\alpha)$ of about 0.7. The linear dependency of the $f(\alpha)$ versus T_m , for the CM method has a higher slope in comparison to the CLM method (Fig-

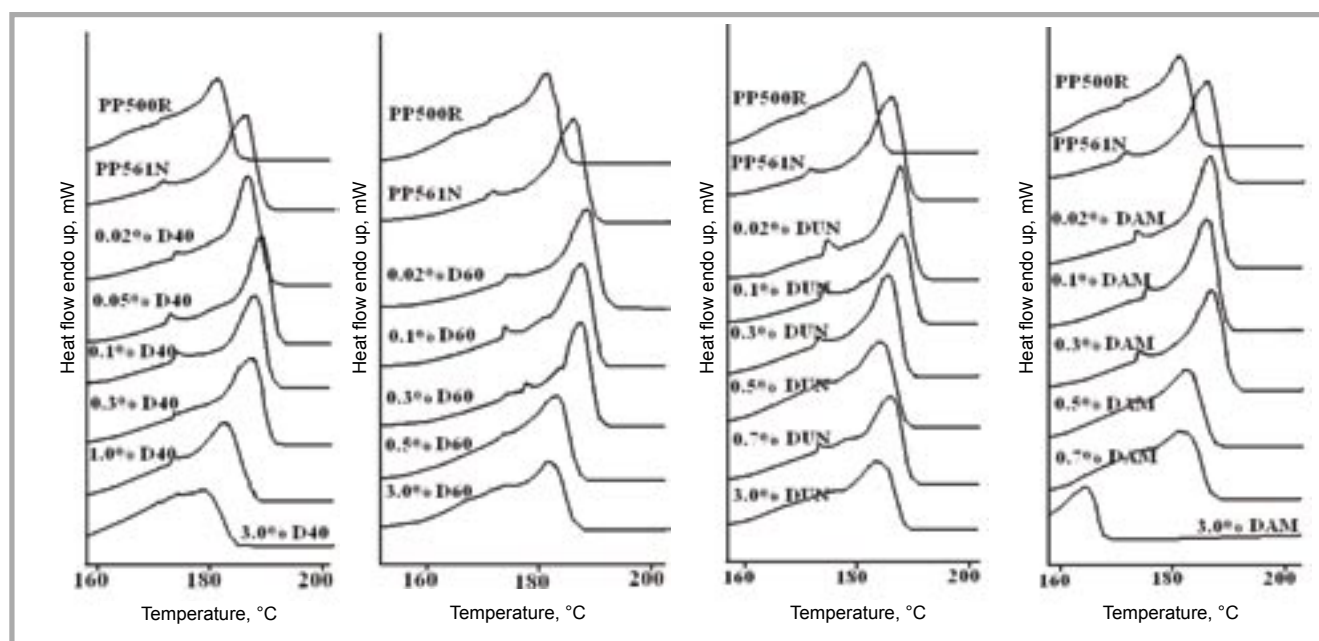


Figure 9. DSC thermograms of the PP561N/Boehmite composite fibres in dependence on type of Dispersal, obtained by CLM method.

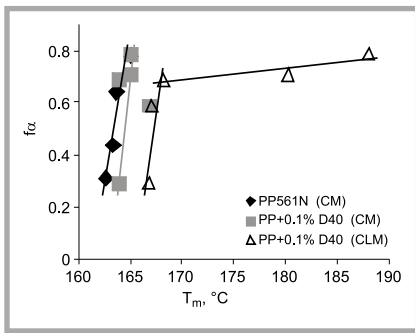


Figure 10. The correlation between the average factor of orientation f_a and melting temperature T_m of the PP/D40 (0.1%) composite fibres for various drawing ratio.

ure 10). Similar correlation was found for the tenacity versus T_m (CLM). The change in the melting temperature for low orientation of fibres is negligible, but above $f(\alpha) \approx 0.7$, and the tenacity increases exponentially as T_m increases (CLM), (Figure 11.a). By contrast, the tenacity of the composite fibres increases gradually with the melting enthalpy in the whole deformation and orientation scale (Figure 11.b) Figure 12 shows the tenacity in the function of the melting temperature (graph a) and tenacity in the function of the melting enthalpy (graph b) of the oriented PP/Disperal

composite fibres for the DSC CLM method. The orientation factor, $f(\alpha)$, depends linearly on the melting temperature and enthalpy for values higher than 0.7 (CLM). However, T_m is a more sensitive parameter for the oriented structure of fibres than $f(\alpha)$, (Figure 10).

Conclusions

On the basis of the experimental results the following conclusions can be drawn:

- Re-melting of the PP/Disperal masterbatch has a positive impact on the spinning and mechanical properties of the composite fibres.
- The minimum values of coefficients of variation of tenacity and elongation at break were found for the lowest and highest drawing ratio of fibres.
- The highest tenacity of the PP/Disperal composite fibres was obtained for the PP/D40 at 0.1 wt% of the nanofiller. At the same time, these fibres provide the highest structure uniformity.
- The DSC CLM method is suitable for evaluation of the structure of oriented PP composite fibres. The melting tem-

perature is a sensitive parameter for the characterisation of the orientation and mechanical properties of highly oriented fibres.

- For the CLM method, the linear dependencies of the tenacity of PP/Disperal composite fibres versus their melting temperature and melting enthalpy were found.

Acknowledgement

The authors would like to thank Mr. Angelo Ferraro and Mr. Giuseppe Ferrara of BASELL for supplying polypropylene and Mr. Olaf Torino of SASOL Company for supplying Boehmites organophilic and unmodified. This work is supported by the EU. FP6 Project: NMP3-CT-2005-516972 "NANOHYBRID".

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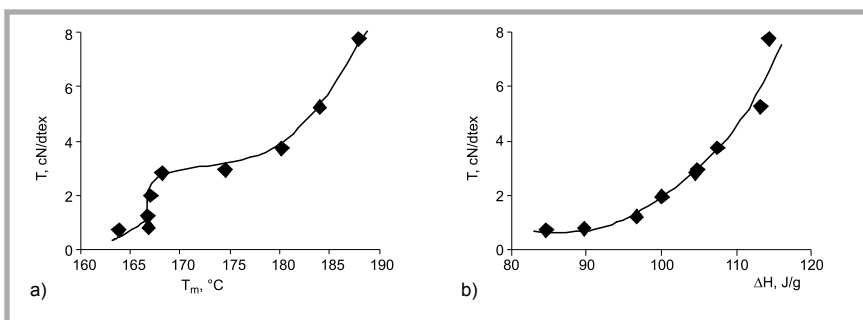


Figure 11. The correlation between the tenacity T and melting temperature T_m (a) as well as the tenacity and melting enthalpy ΔH_m (b) (measured by CLM method) of the PP/D40 (0.1%) composite fibres drawn for various drawing ratio.

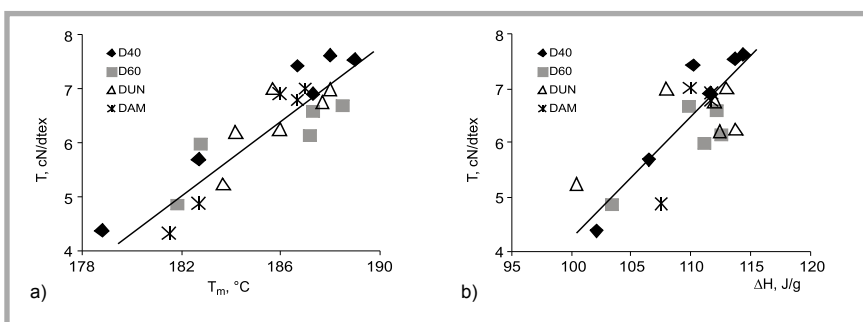


Figure 12. The correlation between the tenacity T and melting temperature T_m (a) as well as tenacity and melting enthalpy ΔH_m (b) of the PP/Boehmite composite fibres drawn for maximum drawing ratio λ_{max} .

Received 15.11.2007 Reviewed 15.01.2008