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Crystallinity of Polyamide-6 Matrix in Glass Fibre/Polyamide-6 Composites Manufactured from Hybrid Yarns

Abstract
Glass fibre/polyamide-6 (GF/PA6) composites were manufactured from specially designed hybrid yarns. PA 6 multifilament and staple polyamide fibres were used as thermoplastic materials. The glass multifilament EC9 was used as a reinforcing material. The hybrid yarns were produced by three different spinning systems: friction spinning, ring twisting and pneumatic texturing. Each of these systems gives a different yarn structure and a different blending level of both the reinforcing and thermoplastic fibres: ***friction yarn*** *(a core of yarn from reinforcing multifilaments and a braid from staple thermoplastic multifilaments),* ***twisted yarn*** *(two multifilaments arranged parallel and twisted together),* ***pneumatic textured yarn*** *(connected over the length of the multifilaments by shifting the filament fibres of both components and forming loop structures in the yarn surface). Thus, the distance between the fibres in the composites produced under the same press conditions and the impregnation of fibres by molten polymer is different. Consequently, the crystalline structure of the polymeric matrix is also different, according to the structure of the yarn being used. A comparison of the crystallinity of PA 6 matrix in the composites studied was carried out by means of the Differential Scanning Calorimetry (DSC) and the Wide-Angle X-ray Scattering (WAXS) methods.*

Key words*: hybrid yarn, glass fibres/polyamide-6 composite, crystallinity, DSC, WAXS.*

Introduction

The increasing interest in using thermoplastic composites in research and industry has resulted in new, modified manufacturing processes. The economical factor and the possibility of designing the material properties are very important. The final properties of composites are determined by the properties of components and bond quality.

The application of hybrid textile fabrics, especially hybrid yarns, as hybrid prepreg products is an approbated method of composite production. The use of hybrid yarns allows the reduction of delamination [1]. The use of continuous reinforcing fibres arranged in parallel leads to the improvement of the composite tensile properties [2]. Moreover, a textile fabric, for example a knitting fabric, is a very good intermediate product for obtaining the sophisticated shapes of composites [3].

In this study, the GF/PA6 composites manufactured from specially designed hybrid yarns were investigated. The hybrid yarns were produced by three different spinning systems: friction spinning, ring twisting and pneumatic texturing [4]. Each of these systems yields a different yarn structure (a different level of blending of the reinforcing and thermoplastic fibres) ‑and different mechanical properties of the yarn.

Friction spinning gives a slightly mixed arrangement of both kinds of fibres (a core of yarn from reinforcing multifilaments and a braid from staple thermoplastic multifilaments), and good strength properties of the yarn.

Ring twisting gives the strength properties of yarn like friction spinning. Two multifilaments arranged in parallel and slightly twisted together form a loose structure with a slightly mixed arrangement of fibres.

The structure of pneumatic textured yarn obtained by means of a stream of compressed air has a greatly mixed arrangement of fibres. Multifilaments are connected over their length by shifting filament fibres and forming loop structures in the yarn surface.

In the hybrid yarn composites obtained, the matrix comes from a semi-crystalline polymer. The mechanical properties of the composites are influenced by, among other aspects, the crystalline structure of the matrix [5].

With the increasing degree of crystallinity, the tensile strength, the stiffness, the modulus of resilience, the hardness of the polymer increase and the impact strength decreases [6]. The formation of the crystalline structure of the matrix depends on the applied melting temperature, the time it is maintained at this temperature, and the rate of cooling [7]. The same conditions were used for each kind of composite. Different yarns were used.

In this work, the crystallinity of the polymer matrix was studied by DSC (Differential Scanning Calorimetry) and X-ray spectroscopy. The influence of the yarn structure, and consequently the arrangement of the fibres in the composite on the microstructure, was determined. The degree of crystallinity of the polyamide matrix in composites from hybrid yarns manufactured by three different systems were estimated.

Materials

Fibres

As thermoplastic materials, PA 6 multifilament and staple polyamide fibres produced by ZWCh "Stilon", Gorzów Wielkopolski (Poland), were used. The PA 6 fibres were coated with the DT2 antielectrostatic preparation. The glass multifilament EC9 produced by Krośnieńskie Huty Szkła (Poland) was used as reinforcing material. Glass fibres were coated with an aminosilane preparation with dispersion of poly-

Table 1. *Characteristics of test material.*

Fibre type	Linear density	Tensile strength	Var. coeff. of tensile strength	Elongation	Var. coeff. of elongation	Number of filaments	Linear density of elementary filament
	tex	cN/tex	%	%	%	-	dtex
EC9 glass multifilament	68	48.7	12.5	2.12	14.9	420	1.6
Polyamide-6 multifilament	26	41.2	3.9	39.0	4.35	192	1.3
Staple PA 6 fibres, 38 mm long	-	44.8	31.0	68.5	26.0	-	2.8

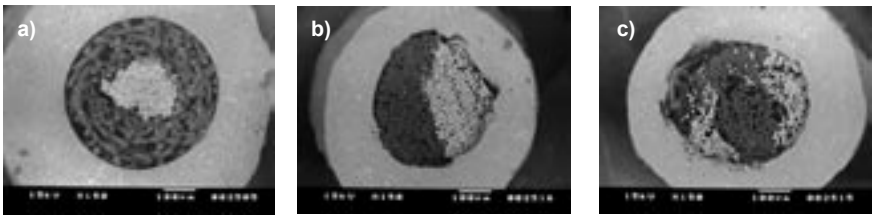

Figure 1. *A view (SEM) of cross-sections of yarns: a - friction yarn, b - twisted yarn, c - textured yarn; light points - glass fibres, dark points - PA 6 fibres.*

Figure 2. *A view (POM) of cross-sections of composites based on: a - friction yarn, b - twisted yarn, c - textured yarn.*

urethane resin. The characteristics of the materials used are given in Table 1.

Hybrid yarns

For comparative analysis, the following hybrid yarns were used:

- ***friction yarns, 135.0 tex*** (Figure 1a) - glass multifilament 68 tex + staple PA 6, mass content of glass - 51.4 %,
- ***twisted yarns, 115.0 tex*** (Figure 1b) - glass multifilament 68 tex + 2x PA 6 multifilament 26 tex, number of twists - 90 twists/m, mass content of glass - 56.6 %,
- ***pneumatic textured yarns, 119.0 tex*** (Figure 1c) - glass multifilament 68 tex + 2x PA 6 multifilament 26 tex, mass content of glass - 55.2 %.

Composites

The thermoplastic composites made from the hybrid yarns were investigated. The composites were obtained from parallel-arranged yarns on a hydraulic press with a water-cooling system and an additional system supplying nitrogen. The optimal thermal and pressure conditions, favourable for good wettability of reinforcing fibres with a melted polymer at low degradation of polyamide, were determined in a separate study [8]. The dried yarns wound onto a metallic plate were pressed under the following conditions:

- consolidation time 15 min, pressure during consolidation 0.012 MPa, temperature 260°C,
- cooling time 15 min, pressure during cooling 0.012 MPa, temperature decreasing to 20°C.

Composites of approximately 0.1 mm thickness were manufactured from an equal amount of yarn.

Methods

Optical microscopy

The arrangement of the glass fibres was observed by means of an Olympus BX51 optical microscope, equipped with a colour video camera/CCD-IRIS and a PC computer. The specimens, like the cross-sections of the composites, were specially prepared. The polyamide matrix was dyed a dark colour for higher contrast between matrix and fibres; the composite was then embedded in 7022 18500 Leica Historesin to form a specimen block holder. The thin cross-sections (within a range of 10-20 μm) of the composites were cut by using an HM 355 S rotary microtome with a sintered carbide knife for hard materials. The distances between neighbour fibres were manually estimated and measured by the Lucia program.

Differential Scanning Calorimetry

The Differential Scanning Calorimetry (DSC) investigations were carried out by a 2920 Modulated DSC (TA Instruments), under a nitrogen atmosphere, at temperatures from 20-260ºC. The calibration of the temperature and heat flow scales at the same heating rate was performed with In, Zn and Sn.

A determination of the crystalline forms of PA 6 was performed with identical conditions of the heating-cooling-heating process, at a temperature scanning speed of 10ºC/min within the temperature range 20-260ºC, and isothermal conditions for 5 minutes at 260ºC.

For the DSC studies of polyamide-6, small samples (about 6 mg) were prepared, and cut from composites in the perpendicular direction to the glass fibres. The samples were not dried before measurement. Additionally, PA 6 in hybrid yarns was analysed.

X-ray spectroscopy

The wide-angle X-ray scattering (WAXS) investigations were made with a TUR-M62 horizontal diffractometer with a HZG-3 goniometer, controlled by a PC computer. The X-ray diffraction pattern was recorded within an 2Θ angle range of 10-30° with a step of 0.04°/3s using Cu_α=1.5418 Å radiation. The deconvolution of peaks was performed by the method proposed by Hindeleh and Johnson [9], as improved and programmed by Rabiej [10]. The measurements were made for samples of 40×40 mm parallel to the direction of fibres. The degree of crystallinity (X_c) of polyamide-6 in hybrid yarn composites was determined by comparing the areas under crystalline peaks and the amorphous curve.

Results and Discussion

Arrangement of the glass fibres in composites

The cross-sections of the composites are shown in Figure 2. One can observe the shortest mean distance between glass fibres in the friction yarn composite (Figure 2a). Concentrations of fibres are visible. In the twisted and textured yarn composites, the distances between fibres are longer, but in the twisted yarn

Table 2. *The distances between fibres in the friction yarn composite.*

Sample no.	Mean, μm	Min, μm	Max, μm
1	2.21	0.20	11.14
2	2.79	0.29	14.97
3	2.64	0.20	9.52

Table 3. *The distances between fibres in the twisted yarn composite.*

Sample no.	Mean, μm	Min, μm	Max, μm
1	5.30	0.20	20.87
2	5.29	0.40	18.30
3	4.66	0.40	16.22

Table 4. *The distances between fibres in the textured yarn composite.*

Sample no.	Mean, μm	Min, μm	Max, μm
1	7.00	0.40	31.27
2	6.68	0.45	28.29
3	5.30	0.45	16.06

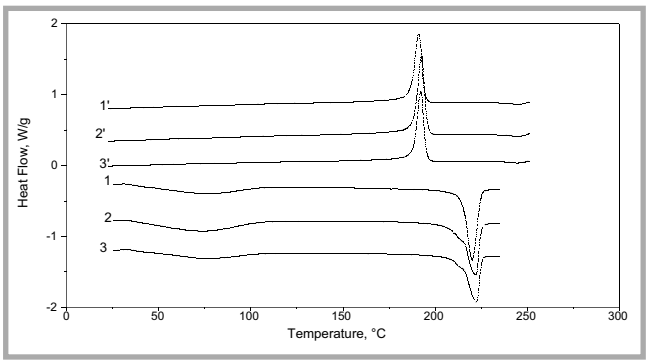

Figure 3. The DSC thermograms for PA6 in hybrid yarns - first heating and cooling: 1, 1' - friction yarn, 2, :2' - twisted yarn, 3, 3' - textured yarn.

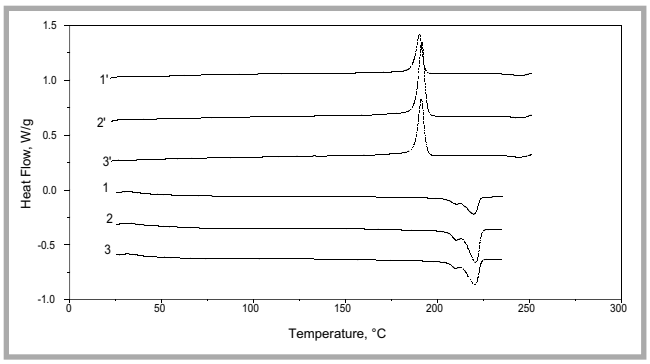

Figure 4. The DSC thermograms for PA6 in hybrid composites - first heating and cooling: 1, 1' - composite from friction yarn, 2, :2' - composite from twisted yarn, 3, 3' - composite from textured yarn.

composite (Figure 2b) the arrangement is more uniform, and almost every fibre is coated by a matrix. In the textured yarn composite (Figure 2c), glass fibres contact often, and the distances between them are diversified. The results are given in Tables 2-4.

DSC

Besides an amorphous phase, semi-crystalline polyamide-6 can exhibit three main crystalline forms, the stable monoclinic α form, the metastable pseudohexagonal β form, and the unstable monoclinic γ form [11]. The β and γ forms may reorganise into the α form during the DSC scan.

DSC investigations of polyamide-6 in hybrid GF/PA6 yarns and the composites produced from them have been reported earlier in detail in literature [12].

The DSC thermograms for PA 6 in hybrid yarns and composites exhibit different kinds of melting peaks. These peaks may be due to the presence of the different crystalline forms of PA 6, the recrystallisation during the DSC scan, the different order of crystal perfection or the different thickness of the lamellae. The higher perfection of the crystals and a higher content of fibrillar crystals lead to a higher-temperature endotherm [13].

In the first heating scan for PA 6 in yarns, one melting peak with a shoulder appearing at lower temperatures can be seen. A melting peak (T_m about 220°C), characteristic of the α form of PA 6, appears in each yarn, although of a different size. Moreover, for twisted and textured yarns, the γ form can tentatively be identified. The crystallisation peak extends from 200 to 130°C, and the peak temperature is about 192°C (Figure 3).

The second heating process, after the first cooling, reveals the existence of two main crystalline phases (α and γ) of polyamide in each hybrid yarn studied (see Table 5).

A small melting peak (probably indicating a recrystallisation phenomenon) is observed at a temperature close to the crystallisation temperature, around 194°C [14]. Recrystallisation phenomena were also observed for other polyamides [15].

The manufacture of the composite from glass/PA 6 yarn in the optimal conditions causes a decrease in the degree of crystallinity of polyamide.

In the first heating scan for PA 6 in composites, as in the second heating scan for polyamide in hybrid yarns, two characteristic endothermic peaks can be found. The melting peak around 220°C could be attributed to the melting of the α-crystalline form. The one around 215°C probably indicates the melting of the thermodynamically unstable γ-crystalline form. The different crystalline forms have to be confirmed by X-ray analyses. Moreover, around 185°C a small peak indicating a recrystallisation phenomenon can be observed (see Figure 4).

The crystallisation temperature for each kind of composite is similar to the crystallisation temperature obtained for the yarns studied (see Table 6), and there is also a tail at the lower temperatures (Figure 4).

In the second heating, one can observe a shift of the melting temperature of the γ form and the recrystallisation form to higher values. After crystalline transformation, a more perfect crystalline phase

Table 5. *The characteristics of crystal morphology of PA 6 in hybrid yarns and composites on the basis of the second heating scan (DSC).*

Sample	Wt.% of PA6	ΔH_f	Crystallinity	The ratio of enthalpies α/γ
	%	J/g_{PA6}	%	-
Friction yarn	48.6	77.9	34	0.54
Twisted yarn	43.4	94.5	41	0.52
Textured yarn	44.8	81.1	35	0.54
Composite from friction yarn	24.1	51.7	22	0.57
Composite from twisted yarn	30.5	68.8	30	0.72
Composite from textured yarn	31.7	53.1	23	0.63
Film from PA6 multifilament	-	77.7	34	0.53

Table 6. *The crystallisation enthalpies - the first cooling (DSC).*

Sample	ΔH_f	ΔH_f	T_c
	$J/g_{hybrid\ sample}$	J/g_{PA6}	°C
Friction yarn	38.5	79.2	191.0
Twisted yarn	41.9	97.4	192.6
Textured yarn	35.8	79.9	192.2
Friction composite	12.6	60.2	190.1
Twisted composite	21.0	75.1	191.4
Textured composite	16.6	52.3	191.1
Film from PA6 multifil.	-	81.1	193.3

(which results in a higher melting temperature) probably forms.

To determine the total crystallinity, the following expression was used [16-17]:

$$W_c = (\Delta H_f / \Delta H_{100}) \times 100\%$$

where:

ΔH_f - the measured enthalpy of the sample's melting,

ΔH_{100} - the enthalpy of a 100% pure crystalline sample's melting; 230 (±20) J/g is a commonly accepted average value for PA 6 [11,13,17].

The values of the degree of crystallinity of polyamide-6 in the samples studied are given in Table 5. The ratio of the enthalpies of α- and γ-crystalline forms was estimated on the basis of the DSC curve for the second heating process. The way in which the two crystalline forms' enthalpies separated is presented in Figure 5. This way, the only one possible in the computer program we used, is not correct but in each case is the same. This allows us to compare the ratio of the enthalpies of the α- and γ-crystalline forms for the samples studied.

The DSC investigations of polyamide in the different glass/PA6 hybrid yarn structures indicate that the ΔH_f value, which is correlated with the degree of crystallinity, is the highest for twisted yarn (see Tables 7 and 8). This yarn exhibits a scattered bond of component multifilaments twisted together.

A similar difference in the crystallinity of polyamide arises in the matrix of the composites manufactured from these yarns. The value of the degree of crystallinity of PA6 in the composite from twisted yarn W_c=30% decreases to W_c=22% for the composite from friction yarn. The decrease in the degree of crystallinity of polyamide in the composites is a result of their hybrid structure. After the wetting process of fibre bundles with molten polymer, their interface bonding arises. The presence in the polymer matrix of glass fibres contributes to an occurrence of smaller spherulite structures.

A higher γ phase volume content is observed in polyamide in each studied material in the second heating. This thermodynamically unstable γ phase leads to a lower density and stiffness in the composites than is noted in the α phase. The highest ratio of α/γ is estimated for the composite from twisted yarn.

Table 7. *The melting enthalpies - the first heating (DSC).*

Sample	ΔH_f J/g$_{hybrid\ sample}$	ΔH_f J/g$_{PA6}$	T_m °C
Friction yarn	43.7	89.6	220.0
Twisted yarn	38.6	88.8	221.9
Textured yarn	34.5	77.0	222.4
Friction composite	12.9	53.6	219.7
Twisted composite	17.8	63.5	220.7
Textured composite	19.5	61.4	220.2
Film from PA6 multifil.	-	73.5	221.5

Table 8. *The melting enthalpies - the second heating (DSC).*

Sample	ΔH_f J/g$_{hybrid\ sample}$	ΔH_f J/g$_{PA6}$	T_m °C
Friction yarn	37.9	77.9	220.4
Twisted yarn	41.0	94.5	220.7
Textured yarn	36.3	81.1	221.2
Friction composite	12.4	51.7	219.3
Twisted composite	21.0	68.8	220.4
Textured composite	16.8	53.1	220.2
Film from PA6 multifil.	-	77.7	220.7

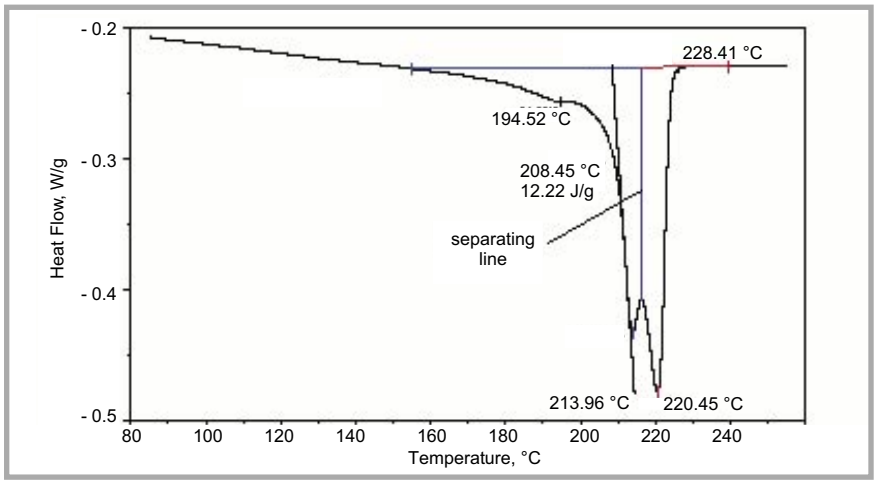

Figure 5. *The estimation of enthalpies of the different crystalline forms.*

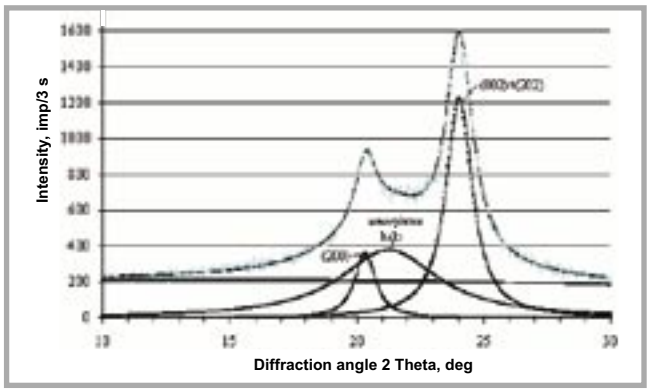

Figure 6. *The fitting of a theoretical diffraction curve to the experimental curve for the polyamide-6 film (upper curves) and the theoretical curve, resolved into diffraction peaks (lower curves).*

WAXS

The fitting of the theoretical diffraction curves for the experimental curves and the diffraction curves with a deconvolution of peaks for the composites studied is illustrated in Figure 7. The crystalline forms existing in the polyamide-6 examined were first determined for the polyamide-6 film obtained from the multifilament (Figure 6). The polyamide-6 in each composite only has a monoclinic α-crystalline form, which does not endorse

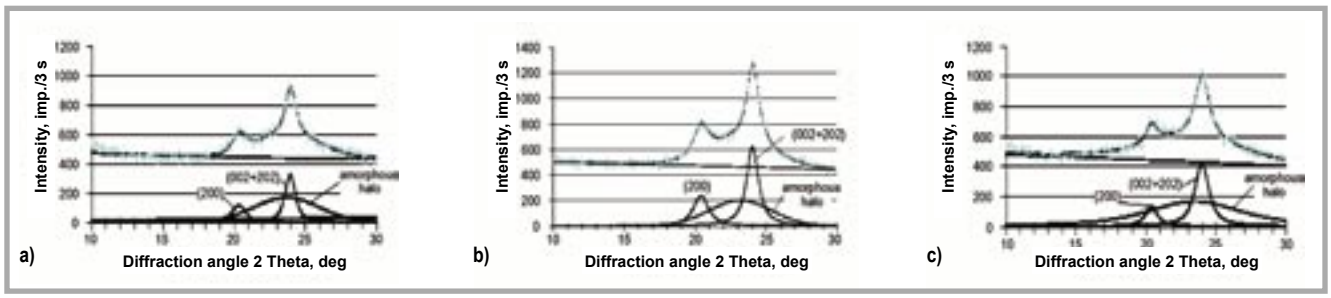

Figure 7. *The fitting of a theoretical diffraction curve to the experimental curve (upper curves) and the theoretical curve, resolved into diffraction peaks (lower curves) for the composites based on: a - friction yarn, b - twisted yarn, c - textured yarn.*

the DSC results. The diffraction curves exhibit two peaks characteristic for the α-form. One peak with (002+202) Miller indices at an angle 2θ=23.8° is sharp, and much larger than the second one, with (200) Miller indices at 2θ=20.2°. The peaks with maximum at 2θ=20.8-21.4° and 10.4-10.9°, which are typical of the γ form, are not observed [11,18].

In the DSC method, the detection of crystalline forms and their volume depends on the melting and crystallisation conditions, for example, the scanning rate. Sometimes, as we can find in literature [15], a γ-crystalline form only reveals itself at a high value of scanning rate. Moreover, the melting behaviour of polyamide during the second heating is different than during the first heating, because the sample's thermal history has changed. In our case, a γ-form is observed, and the volume of γ-form is higher during the second heating at 10°C/min than during the first heating at the same scanning rate. The presence of the melting peak determined by the DSC method, which is related to the γ-form crystals of PA 6, is not confirmed, as in [19], by the WAXS method. This is because of the different kind of parameters studied by these methods.

As can be seen in Table 9, the value of the degree of crystallinity of PA 6 in the polyamide-6 /glass fibres composite is lower than in a pure film manufactured from PA 6 multifilament. The mean

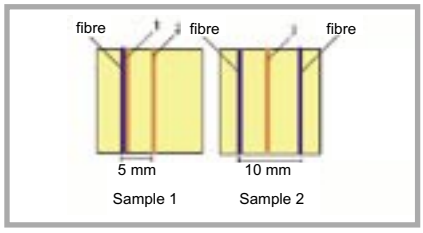

Figure 8. *Model composites: one glass fibre/PA 6 matrix (sample 1), two glass fibres/PA 6 matrix (sample 2); 1 - first place, 2 - second place, 3 - third place.*

dimensions of the PA 6 crystallites perpendicular to the planes with (200) and (002) + (202) Miller indices are shown in Table 10.

In the WAXS method, as in the DSC method (the first and the second heating), the polyamide-6 matrix of the twisted yarn composite shows the highest value of degree of crystallinity among the composites studied, and the matrix of the friction yarn composite shows the lowest value. The component fibres in the twisted yarn are slightly blended, and the yarn structure is loose. Consequently, the fibre arrangement in the composite is uniform and parallel. The use of the friction yarn with a closed structure (glass multifilament as a core, and staple PA 6 fibres as a braid) leads to a low degree of crystallinity of the composite matrix. A slightly higher degree of matrix crystallinity is found in the textured yarn composite. The component fibres in the textured yarn are blended more accurately.

The comparison of WAXS results with the first heating DSC results and with the second heating results shows certain differences, which follow from the differently determined physical parameters in the DSC and WAXS methods. Moreover, in the DSC method in the first heating and in the second heating, the thermal history of the sample is different. However, to compare the crystallinity of the composites studied, both methods can be used. The crystallinity determined in the first DSC heating for the matrix of the textured yarn composite and for the matrix of the twisted yarn composite is similar (Table 11). As in the second heating, these values change, and the relative differences between the crystallinity of the samples studied are similar to the WAXS results.

The different degree of crystallinity of the polyamide matrix GF/PA 6 composites results from the different arrangement of the glass fibres in the matrix. The first differences are visible in the crystallinity of the polyamide in the different hybrid yarns. To confirm the dependence of the crystallinity of the matrix upon the distance between fibres, some additional measurements were made.

The next stage of our studies was the analysis of the crystalline structure of PA6 as a function of the distance from the fibre.

The X-ray investigations of the dependence of the crystallinity of polyamide-6 on the distance from the fibre and the presence of another fibre were carried out. The following samples were investigated:

- sample 1, made from single glass fibre/polyamide matrix,
- sample 2, made from two glass fibres/polyamide matrix.

Glass multifilament was used as a glass fibre and polyamide-6 multifilament as a matrix. The samples were manufactured on a hydraulic press machine. For X-ray investigations, samples of 40×40 mm were cut. The measurements were made in parallel to the direction of the fibres.

The degree of crystallinity was measured at three different places (Figure 8):

- close to the fibre, i.e. up to 2 mm from fibre (for sample 1),
- 5 mm from the fibre (for sample 1),
- in the middle of the distance (10 mm) between two fibres (for sample 2).

The results of degree of crystallinity are given in Table 12.

The value of the degree of crystallinity of the polyamide near the fibre (41%) is much lower than at 5 mm distance from fibre. Moreover, the value of the crystallinity decreases due to the presence of another glass fibre in the vicinity. The

Table 9. *The degree of crystallinity of PA 6 in the composites from hybrid yarns, determined by the WAXS method.*

Composite	Crystallinity, %
From friction yarn	36
From twisted yarn	49
From textured yarn	39
Film from PA 6 multifilament	55

Table 10. *The mean dimensions of the PA 6 crystallites perpendicular to the planes with (200) and (002) + (202) Miller indices in the composites from hybrid yarns.*

Composite	α (200), Å	α (002)/(202), Å
From friction yarn	76.99	103.09
From twisted yarn	71.89	90.09
From textured yarn	99.51	73.56

Table 11. *The comparison of crystallinity measured by different methods.*

Composite	1st heating DSC, %	2nd heating DSC, %	WAXS, %
From friction yarn	23	22	36
From twisted yarn	27	30	49
From textured yarn	26	23	39

Table 12. *The degree of crystallinity of PA 6 in the fibre(s)/matrix composites.*

Measurement place	Crystallinity, %
Close to fibre	41
5 mm from fibre	55
In the middle of the distance (10 mm) between 2 fibres	47

degree of crystallinity at 5 mm from the fibre is higher (55%) than at the same distance but between two fibres (47%). One can thus conclude that in composites manufactured from hybrid GF/PA 6 yarns with different structure, the crystallinity is higher in bulk matrix than when close to the fibres. These investigations confirm that if the distances between fibres are longer, the crystallinity of the matrix is higher.

Summary

- The crystalline structure of polyamide-6 in the GF/PA 6 composites manufactured from specially designed hybrid yarns was studied by DSC and X-ray methods. The crystalline structure of the polymeric matrix is different according to the structure of the yarn used, and depends on the arrangement of the fibres in the matrix. The presence of glass fibres leads to a decrease in the crystallinity of PA 6. The degree of crystallinity of PA 6 increases as a function of distance from the glass fibre.
- For the materials studied, the polyamide in twisted yarn and in the composite from twisted yarn shows the highest values of crystallisation and the highest value of the melting enthalpies at the second heating. The degree of matrix crystallinity as determined by the DSC method is the highest in the twisted yarn composite, where glass fibres are distributed evenly and distances between fibres are relatively longer. The X-ray results confirm that.
- In the friction yarn composite, the degree of crystallinity of matrix is the lowest. The low level of glass fibres and matrix blending in the composite, and the presence of fibres not impregnated by polymer or impregnated only slightly, are influenced by the compact structure of the yarn and by the lack of blending of fibres in yarn. The distances between fibres are very short, which causes a decrease in the crystallinity of the matrix.
- The presence of the amorphous glass fibres as a reinforcing component in the composites studied causes a decrease in the percentage content of the crystalline phase of PA 6 in the composite. The degrees of crystallinity of PA 6 as determined for hybrid yarn composites are lower than for pure film obtained from PA 6 multifilament in the same conditions.
- In each method used, the way of determining crystallinity is different, and consequently the values of degree of crystallinity are different. But the order of these values for the composites studied is the same, irrespective of the method used.
- The DSC analysis indicates a presence of two main crystalline forms, α and γ. However, X-ray investigations indicate only a monoclinic α phase, beside an amorphous phase. The differences result from different measurement conditions and the different parameters determined in each method.
- Among the composites studied, the twisted yarn composite has the highest degree of matrix crystallinity, and the highest content of α phase in the matrix. This fact suggests that the mechanical properties of the twisted yarn composite, apart from impact strength, are the highest as well.

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