Eulalia Klata,
Izabella Krucińska,
Bogusław Więcek*

Technical University of Łódź
Department of Textile Metrology
ul. Żeromskiego 116, 90-543 Łódź, Poland

***Technical University of Łódź**
Institute of Electronics
ul. Wólczańska 223, 90-924 Łódź, Poland

Investigations of Thermal Conduction in GF/PA 6 Composites with a Thermovision Camera

Abstract
The main aim of this paper is the development of a thermovisual method for determining the influence of the distance between reinforcing fibres in thermoplastic composites on the thermal conductivity, and indirectly on the crystallinity of the matrix. The investigations were completed using model composite samples made from glass fibres and a polyamide matrix. Studies showed that the thermal conductivity of the polyamide matrix in a GF/PA6 composite is dependent on the geometry of the distribution of reinforcing fibres in the polyamide matrix. The shorter the distance between the fibres, the lower the thermal conductivity of the polyamide matrix. Hypothetically, this could mean that the degree of matrix crystallinity could be lower if the fibres in the composite are spaced closely, and higher if the fibres are placed farther from each other.

Key words: *GF/PA6 composite, degree of crystallinity, thermal conductivity, thermovision method.*

Introduction

Thermography is a science which enables the illustration of temperature distribution on the surface of the body tested, by means of a non-contact method based on measuring the energy of infrared radiation. The thermovisual method is based on the observation and recording of the radiation distribution, the transformation of IR-radiation into visible light, and obtaining the results in the form of colour thermograms. In this way we obtain a visible image of invisible radiation. The colour of visible light is related to the determined power values of the radiation emitted by the body observed. As the power of radiation depends on the body temperature, the warmer places are presented on the visible image in a colour different than the colour of the cooler places, which enables the observation of the actual temperature. At present, the thermovision method is widely applied for the identification of areas with differentiated temperature distribution. For example, the identification of such areas enables the location of inflammation centres on living organisms, the localisation of thermally damaged areas in buildings or machine elements, and revealing defects in composite materials.

In the work presented in this article, the thermovisual method was used to estimate the heat stream permeation in composite materials as a function of the distribution of the distances between reinforcing fibres. The method proposed might have many different practical applications, but in this work it was used only for identifying thermal conductivity changes in the polymer matrix in dependence on the geometrical distribution of the reinforcing fibres.

The strength of unidirectional stretched composite materials depends, according to the formula of blends, on the strength of the matrix and of the reinforcing fibres [9] in accordance with the following dependency:

$$\sigma_{tw} = \sigma_w V_w + \sigma_o(1-V_w) \qquad (1)$$

where:
σ_{tw} - the tensile strength of the composite material,
σ_w - the tensile strength of the reinforcing fibres,
σ_o - the tensile strength of the matrix,
V_w - the volumetric contents (dimensionless fractional value) of the reinforcing fibres,
$V_o=(1-V_w)$ - the volumetric contents (dimensionless fractional value) of the matrix.

According to the literature [10,11], the strength properties of the matrix depend on its crystalline structure, whose development is influenced by the thermodynamic conditions existing during the crystallisation process. The mutual fibre arrangement in a molten polymer during composite formation determines the heat exchange in the system, and consequently creates conditions of matrix crystallisation [12]. On the other hand, it is known that the matrix crystallinity determines not only the mechanical properties but also the thermal conductivity. In literature much evidence is given which proves the proportional relationship between the thermal conductivity and the degree of crystallinity of the polymer [13]. The main aim of this paper is to develop a thermovisual method for determining the influence of the distance between reinforcing fibres on the thermal conductivity, and indirectly on the crystallinity, of the matrix of composites.

Model Composite Sample

The composite samples were manufactured on the basis of polyamide fibres as

Table 1. *Basic materials for composite manufacturing (*data from literature [14], **data from literature [15]).*

Material	Linear density, tex	Number of filaments -	Linear density of elementary filament, dtex	Filament diameter, µm	*Coefficient of thermal conductivity k at 20°, W/m·K	**Specific heat, cal/(g·K)
Polyamide 6 multifilament	26	192	1.3	12	0.21 - 0.22	0.330 - 0.430
EC9 glass multifilament	68	420	1.6	9	0.037	0.157 - 0.190

raw material for the thermoplastic matrix and glass fibres as reinforcing material. Both kinds of fibres were used in the form of multifilaments. The PA6 fibres, produced by the ZWCh Stilon Company (Poland), were coated with DT2 anti-electrostatic preparation, whereas the glass fibres were produced by the Krośnieńskie Huty Szkła (Glass-Works of Krosno) Company (Poland); these were coated with an aminosilane preparation prepared as a dispersion of polyurethane resin. This preparation was chosen as the agent which is most pro-adhesive to polyamide. The properties of fibres are listed in Table 1.

Polyamide and glass multifilaments were wound on a metallic plate covered with teflon foil, and then were subjected to a process of consolidation. In the process of winding, the glass multifilaments were arranged in parallel, at different distances apart. The spaces between the reinforcing multifilaments were filled with thermoplastic multifilaments.

The approximately 0.1 mm thick composites were made with the use of a hydraulic press with water cooling system and an additional system supplying nitrogen under the conditions described in [16]. The displacements of glass multifilaments in the polyamide matrix of a model composite are presented in Figure 1. The prepared composite samples were subjected to measurements by the thermovisual method. The measurements were completed along the lines indicated in Figure 1, as No 1 to No 9.

Method of Investigation

The temperature distribution at different places of the composite samples in the heating process was estimated by the thermovisual method using the Inframetrics 760 camera with a microscope objective. This is one of the non-contact and non-destructive testing methods.

The heat propagation in composite samples observed is visible as an increase in material temperature on a multicoloured thermogram. Each shade of the thermogram corresponds to the appropriate temperature, according to a given scale. The surface of the heated object emits infrared radiation to the detector placed in the camera equipped with the microscope objective. The camera is installed perpendicularly over the object. The infrared radiation is transformed by a detector into an electrical signal, and then further transformed into a visible radiation signal. Finally we receive the coloured image of temperature distribution in the tested sample with the use of a personal computer. The camera used works as a thermovision scanner, which scans horizontally at a frequency of 4 kHz and vertically at a frequency of 60 Hz. This scanning system enables us to record the temperature of objects in which thermal processes proceed very quickly. For maximum sensitivity and high resolution, a detector with cadmium-mercuric telluride (HgCdTe) as its active material was used; it was cooled to a temperature of 77 K by means of a thermo-electric refrigerating device. The resolution of the thermovision camera was 0.1°C, and the scanning time ranged within 0.05-0.06 s/frame (about 17-20 frames per second).

For the thermovisual investigation, the composite sample with dimensions of 3 cm × 3 cm × 0.1 mm was located be-

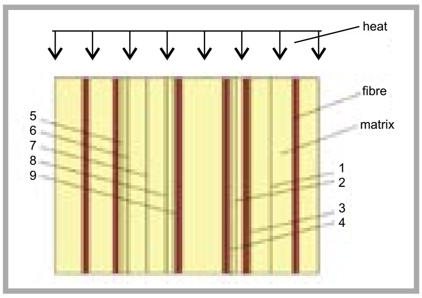

Figure 1. *The scheme of the tested sample with successive temperature measurements.*

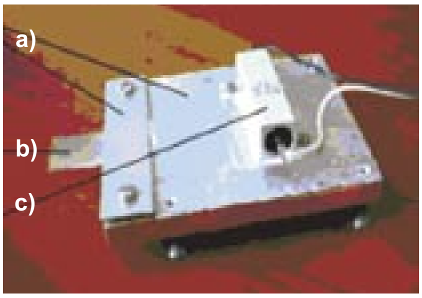

Figure 2. *The thermal clamping device, with a composite sample for thermovision measurement.*

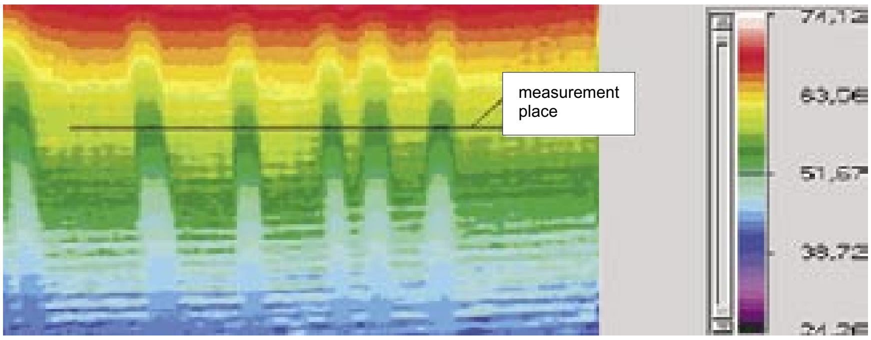

Figure 3. *The image of heat propagation in the GF/PA6 composite during the heating process.* ***Remark:*** *This figure is presented in color in the internet - edition of the journal (www.fibtex.lodz.pl)*

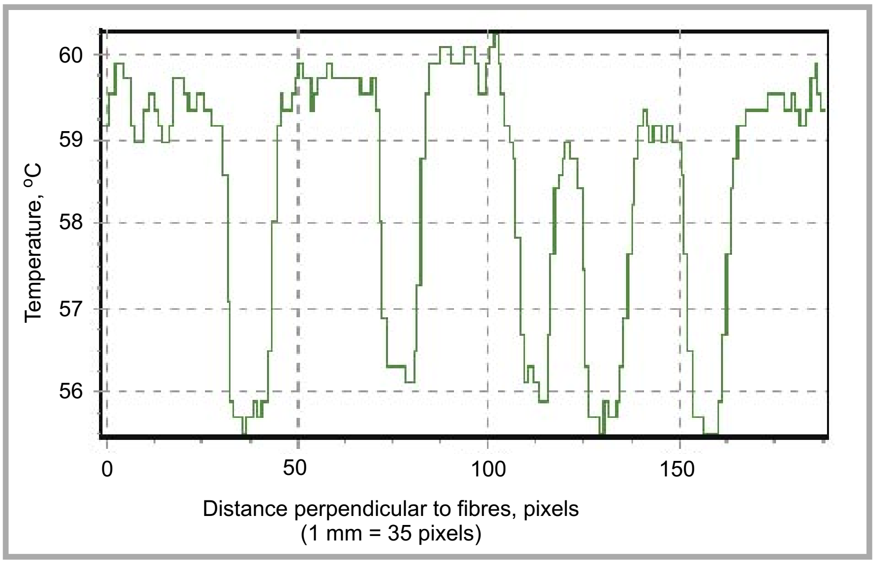

Figure 4. *The temperature distribution in the GF/PA6 composite during the heating process, determined perpendicularly to fibres.*

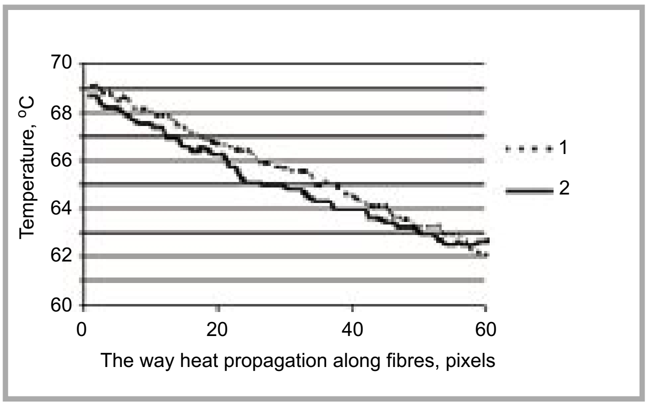

Figure 5. Distribution of polyamide temperature measured along the line placed in the middle between reinforcing fibres during the heating process; for arrangement see Figure 1 (1 mm - 35 pixels); 1 - at the mid-point of the greater distance between two fibres, 2 - at the mid-point of the smaller distance between two fibres

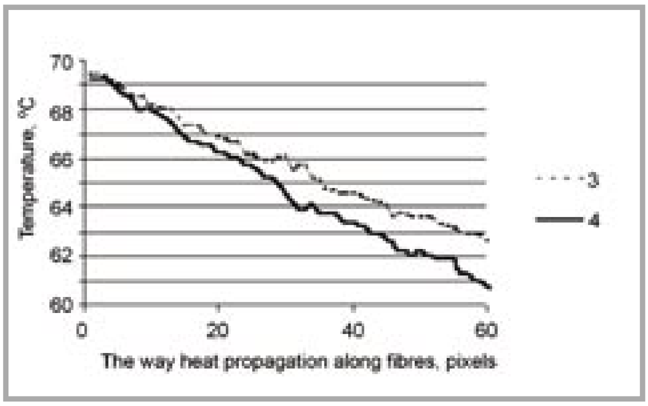

Figure 6. Distribution of polyamide temperature along glass fibres during the heating process; for arrangement see Figure 1 (1 mm - 35 pixels); 3 - close to fibre when the distance between two fibres is greater, 4 - close to fibre when the distance between two fibres is smaller.

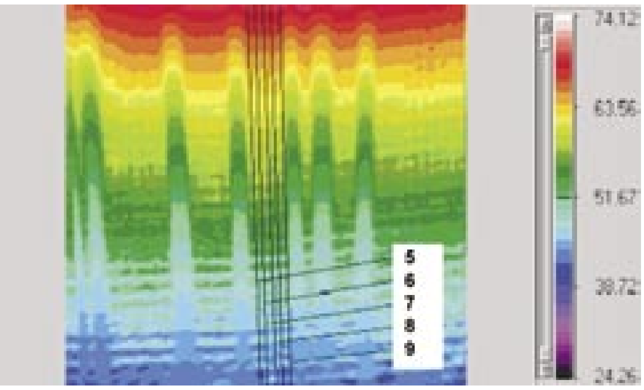

Figure 7. The image of heat propagation in the GF/PA6 composite during the heating process (successive measurement places) are indicated by numbers. ***Remark:*** *This figure, as well as Figure 9 is presented in color in the internet - edition of the journal (www.fibtex.lodz.pl)*

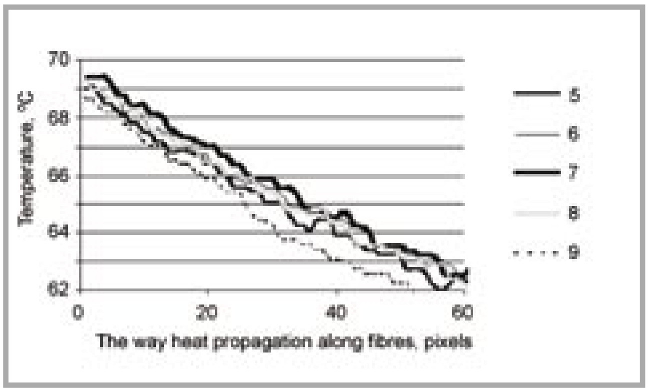

Figure 8. Distribution of polyamide temperature along glass fibres during the heating process, measurements at different distances between two fibres; for arrangement see Figure 1 (1 mm - 35 pixels); 5 - the first place, close to the first fibre, 6 - the second place, farther from the first fibre, 7 - the third place, at the mid-point of the distance between fibres, 8 - the fourth place, closer to the next fibre, 9 - the fifth place, very close to the next fibre.

Figure 9. The image of heat propagation in the GF/PA6 composite during the cooling process.

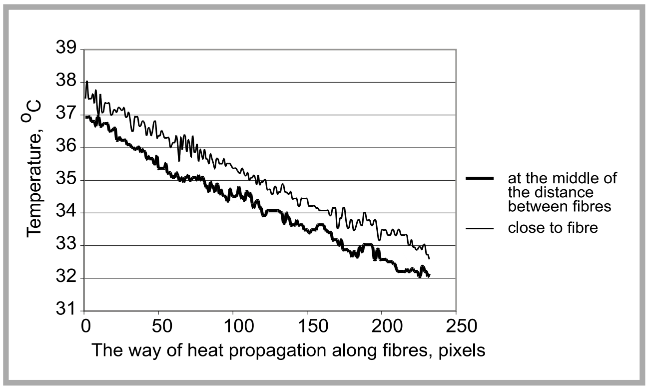

Figure 10. Distribution of polyamide temperature along glass fibres during the cooling process (1 mm - 35 pixels).

tween two aluminium plates, as shown in Figure 2. The lower plate acts as the base and the upper one is the sample holder. A resistor with a radiator was attached to the base plate. The resistor changes the electrical energy delivered to heat, and the radiator uniformly diffuses heat in the testing sample.

The ambient temperature during measurement was 23°C, the block temperature was managed within the range of 87-88°C, the emission coefficient 0.98, and the power dissipated 22W. During the heating process, heat comes from the resistor and is propagated in the sample along the fibres, from the aluminium plates to the end of the sample. The applied block temperature eliminated the possibility of the structure of the composite matrix being thermally damaged.

In order to show the dependence of the thermal conductivity and the hypothetical crystalline structure of the polymer matrix on the distance from the glass fibres, and on the distance between neighbouring fibres, the temperature distribution was measured at several places, as illustrated in Figure 1. In order to investigate the influence of the distance between the reinforcing fibres on the thermal conductivity of the matrix, the values of temperature at places No 1-4 were determined. The measurements were taken at places No 2 and No 4 for a shorter distance between reinforcing fibres, and at No 1 and No 3 for a longer distance. Places No 5-7 were chosen to elucidate the dependence of the thermal conductivity of the polyamide matrix on the distance from a glass fibre.

Results of Experiment

The image of heat propagation in the sample during the heating process is shown in Figure 3. The visible perpendicular zones with considerably lower temperature indicate the existence of glass fibres. During the same heating time, polyamide 6 achieves a higher temperature than glass, because the thermal conductivity of polyamide is higher and the heating proceeds more rapidly, as shown in Figure 4.

The diagram presented in Figure 4 shows the dependence of thermal conductivity of the polyamide matrix on the glass fibre arrangement. When the fibres are close together, the temperature of the polyamide matrix between them during the heating process is lower than in the case when the fibres are placed at a greater distance from each other. It is connected with the lower thermal conductivity of the matrix build between closely-spaced reinforcing fibres. The curves of temperature distribution along the fibres presented in Figures 5 and 6 confirm that conclusion.

The diagrams presented in Figures 5 and 6 indicate the temperature diversification of the preheated polyamide matrix in relation to the different distances of reinforcing fibres in the composite. Moreover, the temperature of the matrix is lower in the vicinity of the fibres than in the middle of the fibres, as shown in Figures 7 and 8. Based on these results, it is possible to draw the conclusion that the presence of fibres interferes with the supermolecular structure of the matrix build during the consolidation process, which results in a differentiation of the thermal conductivity of composites. Hypothetically, the matrix formed closer to the fibre is characterised by a lower degree of crystallinity than at the matrix placed at the mid-point of the distance between two fibres.

During the cooling process, the reverse situation occurs. Glass fibres, which are characterised by lower thermal conductivity, lose heat more slowly than polyamide, and exhibit a higher temperature, as shown in Figure 9. At the mid-point of the distance between fibres, in polyamide, the pre-cooling proceeds most quickly, as presented in Figure 10.

Conclusions

Our studies have shown that the thermal conductivity of the polyamide matrix in GF/PA6 composites depends on the geometry of distribution of reinforcing fibres in the polyamide matrix. The shorter the distance between fibres, the lower the thermal conductivity of the polyamide matrix. Hypothetically, this could mean that the degree of crystallinity of the matrix could be lower when fibres in the composite are spaced closely together, and higher when the fibres are placed farther from each other. The observed phenomena can be explained in the following way.

During the manufacturing process of composites, the reinforcing fibres, which are characterised by a lower thermal conductivity than the conductivity of the matrix, can absorb and accumulate heat during the polymer melting process; then, during the cooling process, the heat realised in the vicinity of reinforcing fibres takes a longer time to propagate than in places lying far from the reinforcing fibres. This results in the fibre surface keeping a high temperature for longer than the polymer does. In this case, during matrix crystallisation the nucleation ability of the fibre is reduced. A crystalline structure starts forming in the bulk matrix, and consequently the degree of crystallinity is different depending on the place: far from fibre, close to fibre, and in the interphase zone (Figures 7 and 8). Basing on the results of the thermovisual investigation carried out, we can state that the thermal conductivity of the polyamide-6 matrix is higher at a farther distance from the glass fibre, and if the distance between the glass fibres is larger. On the basis of the proportional relation between thermal conductivity and the crystallinity of polyamide-6, we can state that the crystallinity of the polyamide-6 matrix increases as a function of the distance from glass fibre and as a function of the distance between glass fibres.

These relationships obtained by means of the thermovision camera confirm the results known from earlier studies of the crystallinity of a polyamide-6 matrix in model GF/PA6 composites, and in composites from hybrid yarns which have been carried out by the DSC and WAXS methods [17-18].

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References

1. *Michalak M., Więcek B., Krucińska I., ‘Investigation of nonwoven layer systems by means of a new thermovision method (in Polish)’, Proceedings of the 6th Domestic Conference ‘Thermography and Thermometry’ Infrared - Ustroń-Jaszowiec, Poland, 343-347, 2004.*
2. *Busse G., Zweschper T., Riegert G., Dillenz A., Advanced dynamic thermography for industry-related applications, Proceedings of the 6th Domestic Conference ‘Thermography and Thermometry’ Infrared - Ustroń-Jaszowiec, Poland, 2004, pp. 61-71.*
3. *Michalak M., Krucińska I., Więcek B., Application of Thermography for slow and fast varying processes in textile research, Quantitative InfraRed Thermography 5 QIRT’2000 - Proceedings of Eurotherm Seminar n° 64, edited by Balageas D., Beaudoin J.-L., Busse G., Carlomagno C.M., Reims, France, 2000, pp. 283-288.*
4. *Michalak M., Stasiak M., Thermal phenomena in spinning as a new source of information of the process (in Polish), Przegląd Włókienniczy, 7 (1991), pp. 189-190.*
5. *Jung A., Więcek B., Zwolenik S., Żuber J., Kalicki B., Kłosowicz S.J., Usefulness of thermovision in venous system pathology, Quantitative InfraRed Thermography 5 QIRT’2000 - Proceedings of Eurotherm Seminar n° 64, edited by Balageas D.,*

Beaudoin J.-L., Busse G., Carlomagno C.M., Reims, France, 2000, pp. 393-398.
6. *Büscher K.A., Wiggenhauser H., Wild W., Amplitude sensitive modulation-thermography the new way of moisture measurement in building materials, Quantitative InfraRed Thermography 4 QIRT'98 - Proceedings of Eurotherm Seminar n° 60, edited by Balageas D., Busse G., Carlomagno C.M., Łódź, Poland, 1998, pp. 184-190.*
7. *Vavilov V.P., Almond D.P., Busse G., Grinzato E., Krapez J.-C., Maldague X., Marinetti S., Peng W., Shirayev V., Wu D., Infrared thermographic detection and characterisation of impact damage in carbon fibre composites: results of the round robin test, Quantitative InfraRed Thermography 4 QIRT'98 - Proceedings of Eurotherm Seminar n° 60, edited by Balageas D., Busse G., Carlomagno C.M., Łódź, Poland, 1998, pp. 43-52.*
8. *Krucińska I., Michalak M., The thermovision method in strength investigation of textile composite materials (in Polish), Techniczne Wyroby Włókiennicze, 2001, 3, pp. 65-70.*
9. *Królikowski W., Tworzywa wzmocnione i włókna wzmacniające, Wydawnictwa Naukowo-Techniczne, Warszawa, 1988.*
10. *Cartledge H.C.Y., Baillie C.A., Studies of microstructural and mechanical properties of Nylon/Glass composite, Journal of Materials Science, 1999, 34, pp. 5113-5126.*
11. *Cartledge H.C.Y., Baillie C., Mai Y-W., Friction and wear mechanisms of a thermoplastic composite GF/PA6 subjected to different thermal histories, Wear 1996, 194, pp. 178-184.*
12. *Borysiak S., The structure and properties of polypropylene composites with cellulose fibres (in Polish), Ph.D. Thesis, Technical University of Poznań, Institute of Technology and Chemical Engineering, Polymer Department, Poznań, 2000.*
13. *Albrecht W., Poliamidy, Wydawnictwo Naukowo-Techniczne, Warszawa, 1964.*
14. *Urbańczyk G., Fizyka włókna - Zagadnienie wybrane, Edition PŁ, Łódź, 1991.*
15. *Urbańczyk G., Fizyka włókna - Własności fizyczne włókna, Wydawnictwo Naukowo-Techniczne, Warszawa, 1974.*
16. *Krucińska I., Mikołajczyk T., Klata E., Studies on Selecting Conditions of Manufacturing of Composites Glass/PA6 from Hybrid Textile Products, Fibres & Textiles in Eastern Europe, 2000, 8, pp. 38-41.*
17. *Klata E., Van de Velde K., Krucińska I., DSC investigations of polyamide 6 in hybrid GF/PA6 yarns and composites, Polymer Testing, 2003, 22, pp. 929-937.*
18. *Klata E., Borysiak S., Van de Velde K., Garbarczyk J., Krucińska I., Crystallinity of polyamide-6 matrix in glass fibre/polyamide-6 composites manufactured from hybrid yarns, Fibres & Textiles in Eastern Europe, 2004, 12, pp. 64-69.*

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