

Teresa Mikołajczyk  
Magdalena Olejnik

Technical University of Łódź,  
Faculty of Textile Engineering and Marketing  
Department of Man-Made Fibres  
ul. Żeromskiego 116, 90-924 Łódź, Poland  
E-mail: mikolter@mail.p.lodz.pl

# Influence of the Plastification Drawing Conditions and Distribution of Drawing Ratios on the Structure and Properties of Fibres Made of a Polyimidoamide Nanocomposite Containing Montmorillonite

## Abstract

*The influence of a single- and two-stage drawing process on the structure and properties of fibres made from a polyimidoamide (PIA) nano-composite containing montmorillonite (MMT) were investigated. A comparative property analysis of the fibres tested enabled the selection of appropriate technological conditions for the drawing process. We observed that with an increase in the temperature of the plastification bath, an increase in the drawing degree is possible, and this increase causes a rise in the tensile strength properties of the polyimidoamide nano-composite fibres. A decrease in the sorption properties at 65% RH and in the water retention value is connected with them. We noticed that by carrying out a two-stage drawing process, a slightly higher tensile strength of PIA nano-composite fibres can be obtained than by a single stage process. On the other hand, a single-stage drawing process is advantageous as there is an increase in the sorption properties at a moderate tenacity of about 12.5cN/tex. From a technological point of view, a one-stage drawing process can be recommended as a simpler way of realisation.*

**Key words:** polyimidoamide, nano-composites fibres, montmorillonite, nano-particles, tenacity, porosity, sorption properties, single-stage process, two-stage process.

## Introduction

The final properties of fibres wet formed from a spinning solution depend on the structure created at the solidification stage, and the deformation forced during drawing stage. Irrespective of the temperature and composition of the solidification bath, the as-spun-draw ratio is the basic parameter of the solidification stage, on which the possibility of obtaining a given deformation during drawing depends.

Conducting the drawing process in a plastification medium causes that the molecular mobility of the fibre matrix macromolecules has an essential influence on the proceeding of the drawing process and its efficiency.

We observed that in the case of polyimidoamides (PIA) with a structure of rigid macromolecules, modification of the polymer matrix, consisting in introducing flexible segments from diaminodiphenylmethane or diaminodipheny-

looxide into it, resulted in an increase in the susceptibility to deformation during drawing [1, 2]. As an effect of the appropriate selection of forming conditions, PIA fibres which unify increased porosity and sorption properties with a tenacity of about 15 cN/tex were obtained for further textile processing [2 - 4]. At the same time, their high thermal resistance was retained [5]. The above-mentioned features ensure the increased use-comfort of protective clothing manufactured from such fibres. Especially important is their application in clothing devoted to use by fire-brigades exposed to the first impact of heat and flame. Such clothing, which is characterised by barrier properties against the impact of heat stream and flame, caused by the chemical structure of the clothing fabric, at the same time will have an increased ability to transport moisture. This is connected with the porous structure created, which means increased sorption properties.

In order to achieve a further increase in the sorption properties of the fibres with a parallel, advantageous decrease in their ignitability, montmorillonite (MMT) was inserted into the fibre matrix [6, 7]. The presence of MMT in the fibre matrix causes an increase in the fibre porosity. During the impact of higher temperature, a layer of carbonised polymer is created on the surfaces of the packets, and layers

of the MMT carbonised polymer form a barrier which cuts the supply of oxygen and limits the oxidation of gaseous decomposition products [6]. Unfortunately, as a side-effect of this modification, the tenacity was slightly lowered, which was connected with the presence of a non-fibre-grade nanoadditive in the fibre matrix. An essential part was also played by the quantity of nanoparticles and the kind of their bond with the polymer. Furthermore, the dispersion of MMT packets in the fibre matrix were also of importance, as well as its intercalation or exfoliation, and the orientation of MMT packets and particular plates in relation to the fibre axis [7, 8]. It is clear that the above-mentioned structure peculiarities must have an essential influence on the tenacity level of fibres manufactured from a PIA nano-composite [8, 9]. We noticed that an increase in the tensile strength properties of PIA nano-composite fibres is connected with the partial exfoliation of the packets of MMT which were modified with a monododecane acid. This increase is visible when comparing it with the strength properties of PIA nano-composite fibres, which include MMT modified by octadecylamine, where exfoliation of the aluminosilicate packets does not occur.

In the case of fibres wet-spun from a solution, the structure formed at the solidification stage and the basic parameters of the

drawing process are decisive for the proceeding of deformation processes and the drawing efficiency, which we can consider as an increase in the general structural orientation of the fibre matrix and, at the same time, in the tenacity. This commonly takes place with an increase in the drawing ratio and speed, as well as with a decrease in the temperature of the process and an increase in the molecular mobility characteristic for drawing in a plastification bath. An increase in the drawing tension, in the majority of cases, also has an advantageous impact on the strength properties of the fibres. In contrast, with an increase in the temperature of the plastification bath, the drawing proceeds more easily. It is possible to achieve high drawing values, but the process is realised at lower drawing tensions. Finally this may lead to the obtaining of lower tensile strength properties of the fibres drawn [10].

Designing a real technological process, most often it is necessary to accept a compromise between conditions which guarantee that the drawing process runs easily and regularly and such which allow to obtain the assumed effect, which means an appropriate degree of orientation and, at the same time, a suitable tensile strength. It is already known that not only decisive for the value of the total drawing but also for the drawing ratio distribution are decisive for the fibre strength [10]. We determined earlier that for fibres with a flexible macromolecule structure, in order to increase the tenacity, drawing should be carried out as a multistage process, with a suitable drawing ratio distribution throughout the particular stages [10]. Furthermore, a deformation near the maximum should be applied at the last stage. In order to conduct the drawing process as described above, the solidification stage should be carried out in mild conditions, considered as a bath with an increased solvent content, at a low temperature, and at negative as-spun draw ratios. Thus, the solidification proceeds according to the diffusion mechanism, which is a necessary condition to obtain a finely porous structure with a small number of defects. In our research work we fulfilled all the above-mentioned conditions while solidifying the PIA nano-composite fibres [10, 12] as a part of our research work.

The value of the longitudinal velocity gradient, which changes over the post-spinneret zone (after the spinning solution stream leaves the spinning nozzle),

is decisive for the macromolecule orientation in the solidifying spinning solution. The kind of the MMT dispersion and its joining with the polymer matrix overlap in its action with the phenomena which occur at this stage of the spinning of the PIA nano-composite fibres.

We suppose that the chemical structure of the MMT's modifier, which is used in order to change its hydrophilic properties into organophilic properties [12], and on which depends the chance to create secondary bonds with the PIA macromolecules [13], is also of essential importance for the proceeding of the drawing process [7 - 10, 12].

All the above-mentioned factors together with the drawing process parameters are decisive for the proceeding of the drawing process and its efficiency. Undoubtedly, the presence of a non-fibre-grade na no-addition in the fibre matrix has a negative influence on the plastification drawing process efficiency of PIA nano-composite fibres, similar to conditions when the process is performed at a temperature significantly below the glass-transition temperature of the fibre matrix (283 °C). A lowering of this disadvantageous effect may be achieved by selecting drawing conditions appropriate for the PIA composite used, which means applying a two-stage drawing process with a suitable drawing ratio distribution, among others.

It should be mentioned that the drawing process of PIA nano-composite fibres was performed in conditions selected by us for the modified PIA matrix [2 - 4], which are different from the conditions for the matrix of Kermel fibres.

Investigations concerning the introduction of MMT into the modified PIA fibres were related mainly to the solidification stage. The value of deformation, which was possible to achieve at the drawing stage, depended on the variable value of the as-spoon-draw ratio. The effects of investigations realised in such a way, concerning differently modified MMT introduced in an amount of 1 - 5% into

the PIA fibre matrix are presented in our previous publications [12]. In order to further increase the tensile strength properties of the nano-composite fibres, an attempt was undertaken in this work to verify the parameters of the plastification drawing process earlier accepted by us.

The aim of our research was to determine the influence of the following factors on the drawing process efficiency:

- the solvent content in the plastification bath and its temperature, which influence the molecular mobility of the fibre matrix's macromolecules, and
- the drawing ratio distribution among the stages of the two-stage drawing process.

These determinations enabled the selection of a suitable technological variant of drawing, as well as the acceptance of the process parameters at which the PIA nano-composite fibres would be characterised by increased sorption properties and, at the same time, by tensile strength properties, which designated them for further textile processing.

## ■ Experimental

### Materials and methods

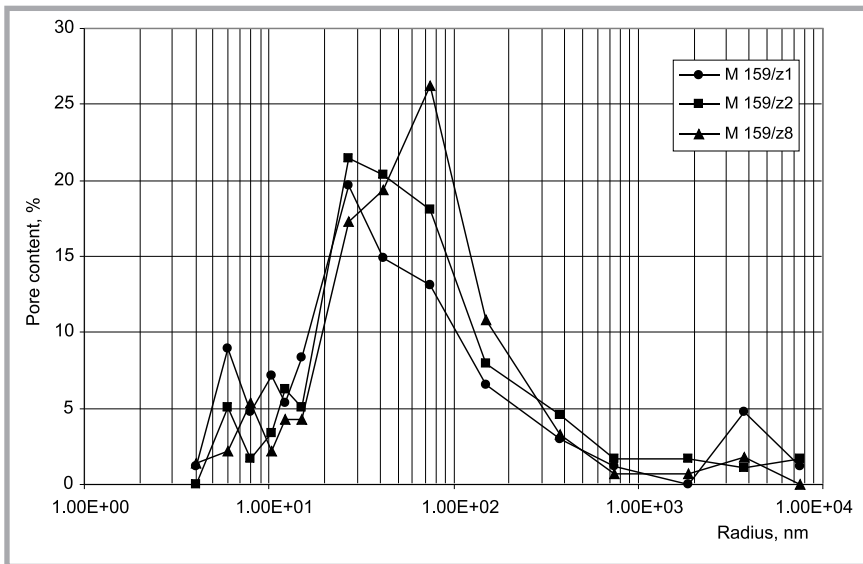
A PIA solution in N-methylpyrrolidone (NMP) (directly after synthesis) containing 3% of MMT (recalculated in relation to the polymer) modified by aminododecane acid was used for spinning the fibres. The MMT was subjected to ultrasound processing at a temperature of 20 °C over 60 minutes. The MMT modification was carried out at the Institute of Industrial Chemistry, Warsaw.

**Table 1** presents the characterisation of the spinning solution, including its rheological parameters. The characterisation of MMT is in a paper [9].

Fibres were wet spun from the spinning solution with the use of NMP as a solvent. A large-scale laboratory spinning-machine was used, of a structure which enabled the stabilisation of the technological parameters at levels accepted by us as well as continued monitoring of these pa-

**Table 1.** Characteristics of the spinning solution of polyimidoamide containing MMT in N-methylpyrrolidone.

Symbol of solution	Concentration, %	Intrinsic viscosity $[\eta]$ , dl/g	Apparent dynamic viscosity, Pa s	Rheological parameter, n	Rheological parameter, k
M - 159z	19.30	1.41	34.41	0.96	34.41



**Figure 1.** Dependence of pore percentage on pore radius of PIA nano-composite fibres after the single- and two-stage drawing processes.

rameters. 240-hole spinnerets with holes of a diameter of 0.08 mm were used.

The solidification process of the PIA nano-composite fibres was conducted at mild conditions, in a bath of the solvent (NMP) content, having increased up to 55%, at a relative low temperature of 15 °C, and at a negative value (-20%) of the as-spun drawing ratio. These conditions were advantageous for the solidification process in order to proceed according to the diffusion mechanism.

Tests of the influence of the plastification bath concentration on the structure and properties of the PIA nano-composite fibres were carried out for solvent contents in baths of 40%, 55%, and 65%. The plastification bath temperature was changed within the range of 50 to 70 °C.

The drawing process was performed as a two-stage process with different drawing ratios, in which the second stage was carried out in preheated steam at a temperature of 140 °C. According to a procedure developed previously [10], the drawing of the first stage was performed at suitable lowered deformations, whereas the drawing ratio of the second stage was near the maximum value.

Moisture absorption at 65% and 100% relative air humidity was determined by the desiccative method in accordance with Polish Standard PN-80/P-04635.

The water retention was measured by a method using centrifuge. Fibre samples

were immersed in distilled water containing a surface-active agent (Rokafenol Nx-3 in an amount of 0.1%) for 24 h, and then the absorbed water was centrifuged off for 10 min at an acceleration of 10,000 m/s<sup>2</sup>.

The fibre tenacity and elongation at break were measured in accordance with European Standard PN-EN-ISO-2062:1997, referring the breaking force to the fibre linear density in tex.

Fibre porosity was measured by means of a Carlo-Erba mercury porosimeter, which was linked to a computer system to register the numerical values of the parameters measured. The values measured included the total pore volume, the total internal surface, the volume of the capillary group with a defined radius and the percentage content. This method allows to determine the pore percentage content within given ranges in the total capillary size-range, from 5 nm to 7,500 nm.

The presence and distribution of the particular elements, especially those which come from the MMT content inserted into the spinning solution, on the fibre surface were estimated by the SEM+EDS X-ray microanalysis using a JSM 5400 unit with an EDX LINK IBIS analyser to record the dispersion energy of the characteristic X-ray radiation.

## ■ Discussion of the results

The single-stage drawing process was conducted in a plastification bath of op-

timal parameters determined on the basis of earlier investigations [9]

Conducting the drawing process in a plastification bath with the same solvent content as that of the solidification bath facilitates the proceeding of the deformation processes thanks to the increased molecular mobility, and enables a drawing ratio to be obtained at a level up to 119.5% (**Table 2**). Increasing the plastification bath temperature acts in the same direction, which is confirmed by the fact that the highest drawing degree of 119.5% was obtained at a temperature of 70 °C. Independently, drawing ratio values suitably lower than the maximum value were applied with the aim of obtaining a homogenous fibre stream, which also takes into account the proceeding of the two-stage drawing process applied in the second series of tests. The tenacity values of fibres manufactured according to the procedure above-described were within the range of 11.34 to 12.5cN/tex, and at the same time the fibres were characterised by high moisture sorption values within the range of 10.7 to 13% at 100% RH, whereas the water retention values were within the range of 45% to 79.5%. The moisture sorption values, which changed within the range of 6.02 – 5.58% at 65% RH, may be connected not only with the chemical structure of the fibre matrix but also with the use of different amounts of acid groups of the MMT's modifier (the aminododecane acid) in order to create secondary bonds with the PIA macromolecules. In the case of fibres drawn at a temperature of 50 °C, obtaining the lowest values of the drawing ratio and tenacity is accompanied by using a smaller amount of acid groups of the MMT modifier in order to create the secondary bonds. Thus, the chance to chemically bind the absorbed water is greater, and a sorption value of 6% is obtained at 65% RH. Such regularity is also valid for fibres drawn in a bath of lower solvent concentration, for example of 40% (**Table 3**).

Moisture sorption at 100% RH and the water retention value are connected not only with the total volume of pores but also with the kind of the porous structure created. The percentage shares of pores of the M-159z/1 sample as a function of the pore radius are presented in **Figure 1**. Irrespective of the two-modal character of the pore distribution curve, distinctive maxima are visible within the range of medium pores and at the beginning of the

**Table 2.** Conditions of the single drawing process of PIA nano-composite fibres and their sorption properties and tenacity; Coagulation conditions: Concentration of solvent (N-metylopyrrolidone) in coagulation bath: 55%, Temperatue of coagulation bath: 15°C, - As spun draw out ratio: -20%.

Sample symbol	Concentration of plastification bath, %	Temperature of plastification bath, °C	Total draw ratio, %	Total deformation	Moisture absoprtion at 65% RH, %	Moisture absoprtion at 100% RH, %	Water retention, %	Tenacity, cN/tex	Elongation, %
M-159z/5	55	50	91.2	1.529	6.02	10.75	79.42	11.34	10.950
M-159z/6	55	60	112.4	1.699	5.86	11.59	67.27	11.35	10.530
M-159z/1	55	70	119.5	1.755	5.58	13.09	45.53	12.46	11.860

**Table 3.** Conditions of the single drawing process of PIA nano-composite fibres and their sorption properties and tenacity; Coagulation conditions: Concentration of solvent (N-metylopyrrolidone) in coagulation bath: 55%, Temperatue of coagulation bath: 15°C, As spun draw out ratio: -20%.

Sample symbol	Concentration of plastification bath, %	Temperature of plastification bath, °C	Total draw ratio, %	Total deformation	Moisture absoprtion at 65% RH, %	Moisture absoprtion at 100% RH, %	Water retention, %	Tenacity, cN/tex	Elongation, %
M-159z/8	40	50	86.2	1.489	6.04	10.54	92.84	10.78	11.490
M-159z/9	40	60	95.6	1.565	5.72	13.04	79.89	11.04	11.220
M-159z/10	40	70	107.3	1.658	5.03	13.64	69.64	11.29	11.180
M-159z/7	65	70	131.5	1.851	5.58	11.34	49.06	11.79	10.52

**Table 4.** Character of the porous structure of PIA nanocomposite fibres after the single and two-stage drawing processes

Sample symbol		M 159/z1		M 159/z2		M 159/z8	
Dimensions of capillaries		Volume per-centage pore content, %	Total volume per-centage pore content for the subrange, %	Volume per-centage pore content, %	Total volume per-centage pore content for the subrange, %	Volume per-centage pore content, %	Total volume per-centage pore content for the subrange, %
	Radius, nm						
Very large	7500	1.19	5.95	1.69	4.51	0	2.52
	3750	4.76		1.13		1.8	
	1875	0		1.69		0.78	
Large	750	1.19	10.72	1.69	14.12	0.72	14.75
	375	2.98		4.52		3.24	
	150	6.55		7.91		10.79	
Medium	75	13.1	55.95	18.08	64.97	26.26	67.27
	41.7	14.88		20.34		19.42	
	26.8	19.64		21.47		17.27	
	15	8.33		5.08		4.32	
Small	12.3	5.36	27.38	6.21	16.37	4.32	15.48
	10.3	7.14		3.39		2.16	
	8	4.76		1.69		5.4	
	6	8.93		5.08		2.16	
	4	1.19		0		1.44	
<b>Total pore volume, cm<sup>3</sup>/g</b>		0.442		0.478		0.751	
<b>Internal surface, m<sup>2</sup>/g</b>		56.546		46.047		70.429	

range of great pores, and it should be emphasised that the water retention values at a level exceeding 45% are connected with these phenomena, which are caused by the ability of pores of such dimensions to absorb water and retain it even after the process of its mechanical removal is finalised. It should be emphasised here that in earlier works different fibre-grade materials depended not only on the volume of the pores tested and their internal surface but also on the percentage share of pores of different dimensions, which are responsible for these factors [9].

On the other hand, small pores, which are able to absorb water on the basis of the capillary condensation phenomenon, are decisive for the value of moisture sorption at 100% RH. With the percentage share of small pores at the level of 27.4% (Table 4) and the first, small developed

maximum on the pore distribution curve, the value of moisture retention at 100% RH, at a level of 13%, agrees with the percentage share of small pores at the level of 27.4% (Table 4) and with the first, small developed maximum on the pore distribution curve.

Considering the total volume of pores at the level of 0.44 cm<sup>3</sup>/g, and the internal surface exceeding 56 m<sup>2</sup>/g, the fibres obtained may be accepted as fibres of increased porosity. At the same time, their tenacity is at the level of 12.5 cN/tex.

As the efficiency of the fibre drawing process is dependent on the value of the drawing tension, and in turn its increase results, in the majority, in an increase in the tensile strength properties of the fibres, we decided to decrease the value of the solvent concentration in the plas-

tification bath down to 40%, thereby decreasing the molecular mobility of the fibre matrix's macromolecules. However, this resulted in a significant decrease in the susceptibility of the fibre matrix to deform and in a decrease in drawing within the range of 107.3 to 86.2% with dependence on the temperature (Table 3). Notwithstanding that the process was probably performed under the influence of higher drawing tensions, the tenacity of the fibres was significantly lowered – up to a level of 10.7 – 11.3 cN/tex. This proves that in the case of a fibre matrix with a rigid structure of the macromolecules, the value of the drawing degree, which is possible to obtain under the actual conditions, is the decisive factor for the tensile strength properties of the fibres. According to the regularity indicated by us for various fibre-grade matrices, the lowering in the tensile strength



**Table 5.** Spinning and drawing conditions, the structure and the properties of PIA nano-composite fibers after the single- and two-stage drawing processes; Coagulation conditions: Concentration of solvent (*N*-metylopyrrolidone) in coagulation bath: 55%, Temperatue of coagulation bath: 15°C, As spun draw out ratio: -20%

Sample symbol	Concentration of plastification bath, %	Temperature of plastification bath, °C	Draw ratio, %			Total deformation	Moisture absorption at 65% RH, %	Moisture absorption at 100% RH, %	Water retention, %	Tenacity, cN/tex	Elongation, %
			R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>						
M-159z/2	55	70	119,5	17,5	157,9	2,063	4,78	11,80	38,37	13,10	9,91
M-159z/3	55	70	97,6	30,8	158,5	2,068	4,86	10,54	39,28	12,73	8,86
M-159z/4	55	70	62,8	58,78	158,5	2,068	4,88	9,41	45,91	12,54	9,19

properties is accompanied by an increase in the sorption properties. Similarly, as when conducting the drawing in a bath with a solvent content of 55%, the lowering in the bath temperature was connected with an increase in the moisture sorption at 65% RH and in the water retention of the fibres, unfortunately, together with a decrease in the strength properties (**Table 2**). Fibres drawn in a plastification bath at a temperature of 50 °C with an NMP content of 40% (sample 159 z/8) are characterised by an especially high water retention value of 92.8%. Such a high retention value is connected with the total volume of pores, which is at a level of 0.75 cm<sup>3</sup>/g, with the internal surface of the pores, which is at a level of 70.43 m<sup>2</sup>/g, and with the percentage share of medium and great pores, which is at a level of 63% (**Table 4**). This is related to the distinctive broad maximum occurring on the pore distribution curve (**Figure 1**). At the same time, the two-modal character of the distribution curve is preserved, and only the first maximum of the small pore range is lowered. This explains the value of moisture sorption at

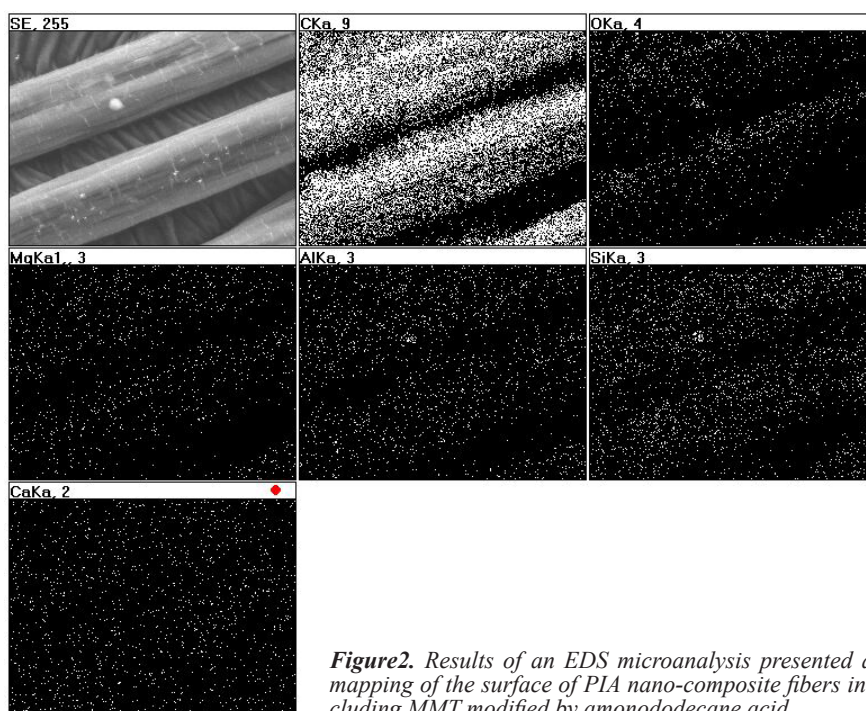
100% RH at a level of 10%. It is clearly visible that the decrease in the molecular mobility and related decrease in the drawing ratio value results in a porosity increase and increase in the sorption properties of the fibres.

Conducting the drawing process under conditions of greatly increased molecular mobility, which means in a bath of solvent concentration of 65%, higher than that of the solidification bath (55%), at a bath temperature of 70 °C, enables to obtain a deformation at a level of 131.5% (sample 159 z/7). But as the process is conducted under the influence of too small tensions, the fibre tenacity is lower by about 0.7 cN/tex compared with that of fibres drawn in a bath with an NMP content of 50% and similar temperature (**Table 3**). However, the sorption properties of both kinds of fibres are similar. Therefore it seems advisable to conduct the drawing process in a bath with a solvent content of 55%, at a temperature of 70 °C.

Carrying out the second drawing stage in a medium of preheated steam, at a tem-

perature of 140 °C, and conducting the process with an appropriate drawing ratio distribution, enabled a significantly higher total drawing, at a level of 158%, as well as a higher total deformation of 2.06 to be obtained (**Table 5**). Only in the case of applying a slightly decreased deformation at the first drawing stage and a drawing ratio near to the maximum value at the second stage (sample 159z/2) we obtained a higher fibre tenacity, by 0.64 cN/tex, in comparison with the single-stage process. On the other hand, conducting the drawing process at a lower drawing ratio value at the first stage and increasing the drawing ratio at the second stage, which means with a typical drawing ratio distribution, the fibre' tenacity was comparable with that obtained by the single-stage process, notwithstanding the differences in the values of the total drawing degree. A decrease in the deformation value at the first stage, and a lowering of the tensile strength properties and of the moisture sorption value at 100% RH were connected, as well as an increase in the retention value from 38% to 46%. A lower sorption value of 4.8% at 65% RH may suggest the use of a greater amount of acid groups of the MMT's modifier in order to create secondary bonds with the PIA macromolecules. Obtaining higher drawing degrees (in a two-stage drawing process) was advantageous for a better orientation of the macromolecules and MMT layers, as well as for the creation of a greater number of secondary bonds. Generally, with the arrangement of MMT packets and layers in the fibre forming matrix, the sorption properties as well as the tensile strength properties of the fibres manufactured are connected. In the case of using lower drawing degrees, the lower arrangement of these layers is accompanied by the obtainment of lower strength properties and higher sorption features.

From the comparison of the porous structures obtained by the two technological variants of one-stage and the two-stage drawing, it can be concluded that fibres drawn by the two-stage system are distin-



**Figure 2.** Results of an EDS microanalysis presented as a mapping of the surface of PIA nano-composite fibers including MMT modified by monododecane acid.

guished by a slightly higher total volume of pores, at lower by about 10.5 m<sup>2</sup>/g of the internal surface. Applying the second drawing stage does not change the character of the pore distribution function. The two-modal character of the curve remains (**Figure 1**), only the first maximum, positioned within the range of small pores, decreases which is connected with a slightly lower sorption values at 100% RH. An advantageous phenomenon is a decrease in the percentage share of pores with the greatest dimensions (within the range of up to 7,000 nm), which are structural defects decreasing the strength properties of the fibres.

The presence of characteristic elements, which come from MMT introduced into the fibre matrix, on the surface of the PIA fibres obtained by us, was estimated on the basis of a SEM + EDS X-ray microanalysis.

From the linear analysis and mapping, (which is presented in **Figures 2**), it can be stated that irrespective of the elements originating in the fibre matrix, such as oxygen, silicon and aluminium atoms, also atoms of magnesium and calcium, characteristic elements, which are included in the structure of MMT, are also visible on the fibre surface. On the basis of the mapping presented in **Figure 2** it can be concluded that on the fibre surface not only regions of even distribution of the elements originating in the inserted MMT exist. Also clearly visible are surface fragments with a greater concentration of the particular elements. These testify to the sporadic occurrence of the agglomeration phenomena, in spite of the application ultrasonic processing in order to disintegrate the MMT agglomerations.

By summarising our investigations we can state that the research tests carried out indicate that, contrary to our earlier assumptions, applying the two-stage drawing process did not lead to a significant increase in the tensile strength properties. This means that applying the single-stage drawing variant of PIA nano-composite fibre drawing is advantageous considering the simplicity of the technological solution and the possibility of obtaining increased retention values at a tenacity of 12.5 cN/tex. Conducting the process under conditions of increased molecular mobility (with the same solvent content in the plastification bath as in the solidification bath, and at an increased tempera-

ture) results in an increase in the drawing degree, whose value mainly influences the tensile strength properties of the fibres.

The fibres obtained as described above, with a tenacity of 12.5 cN/tex were used for manufacturing twisted multifilament yarn. This yarn was used for producing woven fabrics of plain weave with an area mass of 350 g/m, a tensile strength in warp direction of 873 N, and in weft direction of 892 N. In this case we were able to confirm the good capability of the PIA nano-composite fibres to produce flat textile products.

## Conclusions

1. Verification of the conditions of plastification drawing indicated that the process parameters selected for fibres from a modified matrix are also appropriate for PIA nano-composite fibres containing montmorillonite.
2. Considering the rigidity of the macromolecule of the matrix structure, the efficiency of the plastification drawing process of PIA nano-composite fibres mainly depends on the drawing degree value possible to obtain.
3. An increase in the molecular mobility as a result of an increase in the concentration and temperature of the plastification bath, as well as an increase in the drawing ratio are connected with the drawing process efficiency.
4. As the limit of increasing the macromolecule mobility can probably be accepted, a decrease in the drawing tension to such a level at which the increase in the drawing degree is not accompanied by a further increase in the fibre tensile strength properties.
5. We indicated that by carrying out a two-stage drawing process, a slightly higher tensile strength property of PIA nano-composite fibers containing MMT can be obtained than by a single stage process. From a technological point of view, one-stage drawing can be recommended as a simpler way of realisation, which assures the obtaining of a tenacity of 12.5 cN/tex.

## Acknowledgment

The investigation was financially supported by the Ministry of Science and Higher Education within the scope of research project Nr. 4708E08724.

## References

1. Mikołajczyk T, Janowska G, Urbaniak – Domagała W, Szczapińska M.; Multifunctional Thermostable Fibres from Modified Polyimidoamide. *Fibres&Textiles in Eastern Europe*. 2004; 12(1):27-31.
2. Mikołajczyk T.; Effect of As-spun Draw Ratio and Extent of Deformation During Fibre Drawing on the Structure and Properties of Fibres from a New Polyimidoamide Material. *Fibres&Textiles in Eastern Europe*. 2002; 10(1):52-55.
3. Mikołajczyk T, Skwarski T.; Effect of the Solvent Content in Solidifying and Plasticising Baths on the Properties of Polyimidoamide Fibres. *Fibres&Textiles in Eastern Europe*. 2002; 10(3):14-17.
4. Janowska G, Mikołajczyk T.; Thermal Properties of Fibres from a New Polymer of the Polyimidoamide Group. *J. Thermal Anal. Cal.* 2003; 71: 549 – 558.
5. Janowska G, Mikołajczyk T, Olejnik M.; Thermal Properties and Flammability of Fibres Made From Polyimidoamide Nanocomposite in press in *J. Thermal Anal. Cal.*
6. Janowska G, Mikołajczyk T, Olejnik M.; Effect of Montmorillonite Content and the Type of Its Modifier on the Thermal Properties and Flammability of Polyimidoamide Nanocomposite Fibres in press in *J. Thermal Anal. Cal.*
7. Mikołajczyk T, Rabiej S, Olejnik M, Urbaniak – Domagała W.; Strength Properties of Polyimidoamide Nanocomposite Fibres in Terms of Porous and Supermolecular Structures. *J. Appl. Polym. Sci.* 2007; 104: 339-344. DOI:10.1002/app.24357.
8. Mikołajczyk T, Olejnik M.; Effects of Spinning Conditions on Structure and Properties of Multifunctional Fibers of Polyimidoamide Nanocomposites. *J. Appl. Polym. Sci.* 2006; 100: 3323-3331. DOI: 10.1002/app.23211.
9. Mikołajczyk T, Olejnik M.; Analysis of the Structure and Properties of Polyimidoamide Nanocomposite Fibers Containing Various Modified Montmorillonite. *J. Appl. Polym. Sci.* 2006; 101:1103. DOI: 10.1002/app.24006.
10. Mikołajczyk T.; Modification of the Manufacturing Process of Polyacrylonitrile Fibres. *Scientific Bulletin of Technical University of Lodz, No 781. Scientific Theses Z 243, Łódź 1997.*
11. Sinha Ray S, Okamoto M.; Polymer/layered silicate nanocomposites: a review from preparation to processing. *Prog. Polym. Sci.* 2003; 28:1539-1641.
12. Mikołajczyk T, Rabiej S, Olejnik M.; „Analysis of the Effect of the Amount and Type of Montmorillonite on the Supermolecular Structure, Porosity and Properties of Polyimidoamide Fibres” *J. Appl. Polym. Sci.* 2007, 105 p. 1937.

Received 27.05.2008 Reviewed 19.11.2008