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Monitoring System for the Formation Process Line of PAN Composite Fibres

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Abstract

The aim and scope of this work included the design and practical implementation of a digital monitoring system for the polyacrylonitrile (PAN) fibre spinning process line used for the creation of different PAN based fibres doped with silver (Ag), polyaniline (PANI), carbon nanotubes (CNT) and 2,3,5-triphenyltetrazolium chloride (TTC). After the collecting and processing of process parameters, including bath temperatures and the rotational speed of the feeding-receiving points, available in the form of digital data, they were compared with the appearance of fibres obtained (their surface structure and cross-section shape) and with the results of the fibre specific strength (WtP). Archiving of speed and temperature measurement data allowed to create a database combining the process parameters with the parameters of the fibres obtained. Online monitoring of the parameters enabled programmable change of the speed and temperature in important parts of the process in order to develop appropriate production profiles.

Key words: digital monitoring system, speed and temperature measurement, on-line monitoring, fibre spinning process line, composite polyacrylonitrile fibres.

ber of cheap and small devices which can communicate with each other. It is possible to easily install such devices at critical points of the technological process in order to control and monitor the quality and capture emergency states. A particularly important issue is monitoring in the continuous and direct mode (on-line) [1]. This is particularly important in monitoring high-speed processes, where error or change in process parameters can cause severe economic consequences. The results obtained during the monitoring of industrial processes can also be used to develop other important scientific disciplines. They allow, for example, to create a knowledge base consisting of relevant process data, leading to the development of so-called expert systems that give the opportunity to draw in and design new process variants. They also contribute to the development of model-simulation studies enabling the transfer of the entire process to the computer simulation environment [2-3]. Data collected during the monitoring process can be used to validate the computer model of the system. The subject of monitoring is intensively developed by supervising various technological processes in the textile industry [1,4]. For example, spinning, knitting, weaving, etc. processes require a precise series of successive operations. The continuous monitoring and control process is able to ensure a high quality of the final product. It also provides information in the form of reports and graphical presentations about the process performance, repairs required and machine maintenance as well as about emergency and alarm states. Monitoring also contributes

to the optimisation of various production processes in textile technologies [5-7].

This article describes the implemented solution of a simple on-line monitoring system for the production of synthetic fibres. The system performs the function of continuous monitoring of the technological process parameters. With its help, a knowledge base of the process parameters is also built, which in the future can be used to model the technological line for the production of fibres.

In the technology of fibre production by the method of wet solution, three basic stages of the manufacturing process can be distinguished:

- the formation of fibre from a filter-cleaned, deaerated spinning solution
- elongation of the fibres
- finishing treatment.

Formation of fibre from a filter-cleaned, deaerated spinning solution

A solution of polyacrylonitrile in dimethylformamide (DMF) with a dynamic viscosity in the range of 20-30 Pas is extruded through the holes of the spinning nozzles into a coagulation bath at a suitable temperature in the range of 20 to 30 °C. Just after the contact of the stream of the spinning solution with the bath, the fibres consolidate and the solvent molecules from the fibre stream are washed out into a solidifying bath. The composition of this bath is essential for the quality of the fibres produced [8]. When the coagulation bath is only water, poor quality fibres are obtained. It was

Introduction

The subject of the monitoring of technological or production process parameters is a very important research issue, intensively developed in the last decades. It is closely related to the widespread use and easy implementation of various measurement methods using digital and microprocessor techniques. Modern programmable systems provide great freedom in the design of measuring devices that are characterised by repeatability, stability, easy implementation of special functions and, most importantly, the possibility of software reconfiguration to adapt to new needs. From the production monitoring point of view, the digital technique makes it possible to make a large num-

proved in [8] that optimal fibre properties are achieved when the share of solvent (DMF) in the solidifying bath is about 70-80%.

Elongation of the fibres

Depending on the number of holes in the spinning nozzle, a band consisting of several hundred monofilament fibres can be formed, which is subjected to multistage elongation between the feeding-receiving points. The elongation value depends on the rotational speed of the subsequent feeding-receiving points. The elongation process is always carried out in a plasticising bath which is composed of a 50-60% solution of DMF in water at a temperature ranging from 40 °C to 80 °C, and then in lost water vapour (at temperatures from 100 °C to 135 °C). The value of the total elongation fluctuates within quite wide limits of ranges from 200% to 1000%. During these processes, the structure of the fibre is ordered, improving the mechanical properties of fibres and, above all, their mechanical strength.

Finishing treatment

During the finishing treatment, the fibres were rinsed in a scrubber on oblique shafts to remove residual solvent. Then they were dried by blowing air at ambient temperature.

Experimental

Polyacrylonitrile (PAN) and PAN fibres doped with silver (Ag) [9], polyaniline (PANI) [10], carbon nanotubes (CNT) [11] and 2,3,5-triphenyltetrazolium chloride (TTC) [12] were formed by the wet solution method. The polymer was dissolved in dimethylformamide (DMF) to obtain a specific viscosity solution. When planning to modify such a spinning solution with admixtures, it was necessary to ensure that the dopants did not change the rheological properties of the spinning fluids. A too high viscosity value caused that the solution could not form fibres, and thus it could not be “squeezed” through the holes of the spinning nozzle or, on the contrary, when the viscosity of the solution obtained was too small, the spinning fluid could not be adequately dosed for the coagulation bath (solidifying). By introducing dopants [12-14] or by directing the synthesis of nanoparticles directly in the spinning solution [15-17], changes are observed not only in the rheological parameters of the solution but also in the parameters of

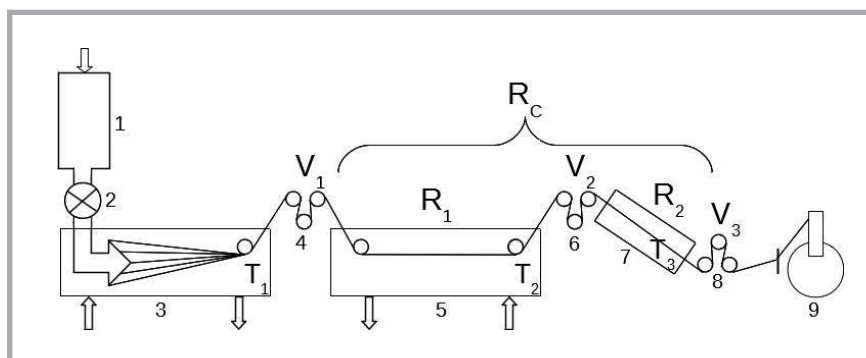


Figure 1. Schematic diagram of a stand for forming chemical fibres using the wet processing method: 1 – tank with polymer solution, 2 – dosing pump, 3 – solidifying bath (coagulation), 4, 6 & 8 – feeding-receiving points, 5 – plasticising bath, 7 – evaporator, 9 – coil with multifilament (fibres), R_1 – elongation in plasticising bath, R_2 – elongation in steam, R_C – total elongation, T_1 – solidifying bath temperature, T_2 – plasticising bath temperature, T_3 – temperature in evaporator, V_1 – speed of point 4, V_2 – speed of point 6, V_3 – speed of point 8.

the forming process, and consequently in the properties of fibres formed from such solutions. For this reason, each fibre modification is a complex process [18]. Several authors before investigated the influence of fibre formation conditions on the properties of PAN and polypropylene fibres and tried to find their optimal values for different applications [19-20]. Also, the influences of separate parameters such as the temperature of the solidifying (coagulation) bath [21-22], the as-spun draw ratio [23-25] and plasticising drawing conditions [26] were investigated as important factors in the fibre formation process. Therefore, it is important to have knowledge of the exact values of the process parameters (mainly solution temperatures and fibre transport speed) to determine the correctness and repeatability of the technological process of obtaining polyacrylonitrile and composite fibres based on PAN solutions.

Forming fibres using the “wet forming” method

In the studies presented in the article, PAN and PAN based composite fibres were obtained using the laboratory devices illustrated in the diagram (**Figure 1**).

The spinning solutions were fed from the reservoir (position 1 in **Figure 1**) to the gear pump (item 2 in **Figure 1**) with a capacity of 0.6 cm³/rot., at a pressure of 3 atm. Fibres were formed using a 500 hole nozzle with a 0.08 mm hole diameter. After pressing the polymer solution through the filler, it was subjected to two successive baths – solidifying and plasticising. In the solidifying bath (60% DMF solution in water) at 20 °C and in the plasticizing one (50% DMF

solution in water) at a temperature of 70 °C, intensive physical processes of removing the solvent and impurities from the fibres formed took place. Both baths were in special tanks (positions 3 & 5 in **Fig. 1**) in continuous circulation, where the solidifying bath was fed concurrently to the circulation of the solidified fibre, while the plasticising bath was supplied countercurrently (as illustrated by the arrows in **Figure 1**). The bath concentrations were kept constant by adjusting the composition changes in the intermediate tanks, to which an appropriate amount of the solidifying agent was dosed. The exchange rate of each bath was about 25x/h. The fibres formed and elongated were wound on coils (item 9 in **Figure 1**).

Description of the construction of the monitoring system

The process of spinning fibres using the “wet processing” method is a complex physico-chemical process and is characterised by several subsequently occurring operations. These operations should be characterised by the constancy or assumed variability of parameters over time. The parameters are associated, on the one hand, with the occurring chemical reactions, but on the other with specific mechanical conditions of the spinning process. The main parameters of the spinning process are the temperatures of the solidifying and plasticising baths as well as the values of the multi-stage fibre elongation (R_1 & R_2). Temperature values can be measured directly in the solidifying and plasticising bath solutions, and in the column used to generate steam. Fibre elongation measurement was carried out indirectly, measuring the rotational speed at individual feeding-receiving points

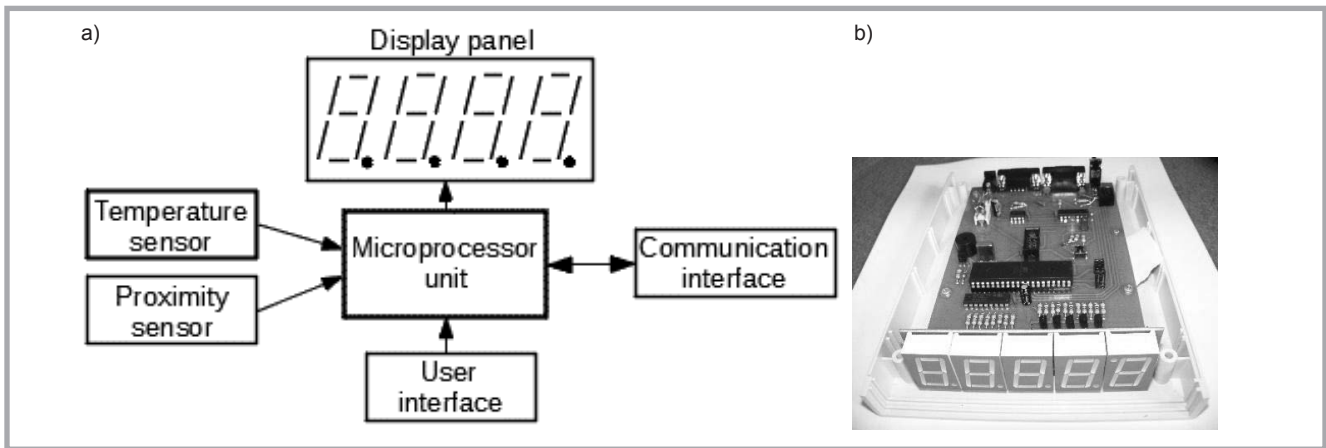


Figure 2. a) Block diagram of the measurement module and b) module prototype.

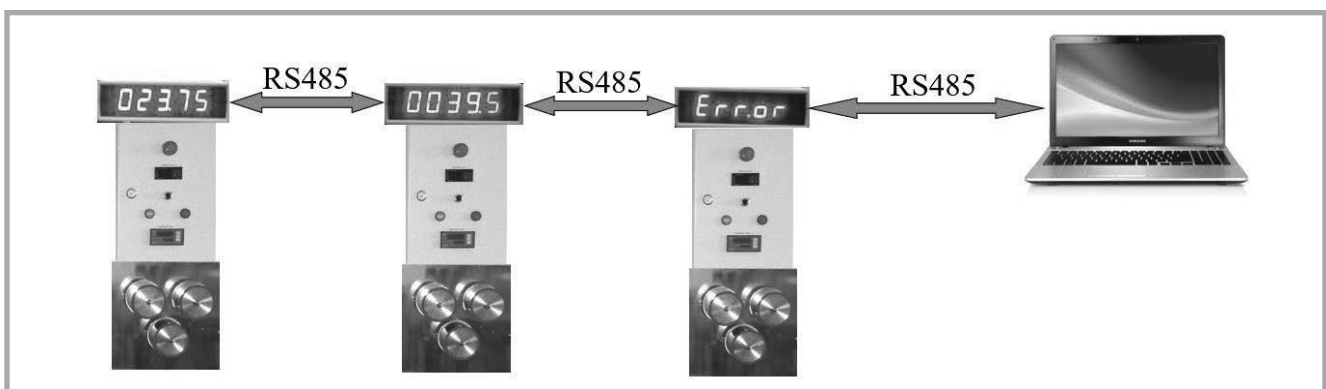


Figure 3. Block diagram of the temperature and velocity measurement system, consisting of three measurement modules connected using an RS485 industrial network.

(items 4,6 & 8 in **Figure 1**). Further processing of rotational speed data is carried out in the offline mode, which leads to results showing the variability of fibre elongation over time at different stages of the spinning process. In the schematic diagram shown in **Figure 1**, several similar, repeating elements can be distinguished. Each elongation step (R_1 , R_2 in **Figure 1**) corresponds to a specific chemical fibre forming operation. The first elongation of fibres (R_1 **Figure 1**) takes place in the plastifying bath and the second during the fibre passing through the evaporator (R_2 **Figure 1**). The temperature of each chemical treatment process should be measured (T_1 , T_2 & T_3 in **Figure 1**) as well as each rotational speed value at the feeding-receiving points (4, 6 & 8 – **Figure 1**). Due to the fact that the number of chemical treatment points at which the temperature should be monitored is equal to the number of feeding-receiving points and that individual chemical treatment baths alternate with the feeding-receiving points, a modular design of the monitoring system was proposed. One monitoring module allows to display the current process temperature in one bath

or evaporator and to show the current rotational speed at one feeding-receiving point. Each monitoring module also has the option of sending monitored data to a data collecting device or PC computer for archiving and further processing “offline”.

Figure 2.a shows a block diagram of the measurement module and **Figure 2.b** – a prototype of the module tested. It consists of several important elements: a microprocessor unit, a temperature sensor, a proximity sensor, a display panel, a user interface and a system that transmits measurement data. The most important element of the module is the microprocessor system, built with the use of a Microchip – Atmega162 microcontroller [27]. This system reads information from the measuring sensors, displays measurement results on a seven-segment display, reacts to user’s commands and allows to send information about measurements to the master device. As a temperature sensor, a thermocouple with a conditioning circuit was used. The rotational speed sensor is an inductive proximity sensor that generates a rectangular voltage wave

with a frequency proportional to the rotational speed of the feeding-receiving point. The RS485 industrial standard was used as the communication interface, which enables serial data transmission over relatively large distances [28]. It uses only two wires for communication purposes.

Figure 3 shows a block diagram of the whole measuring system, consisting of three measuring modules. This diagram presents real photographs of three feeding-receiving points equipped with three measuring modules. Measurement modules have the ability to independently measure speed and temperature at any point. They are connected to each other and to the master computer using an industrial network the RS485 standard. This network allows the transmission of digital rotational speed and temperature data at regular intervals for archiving and offline processing.

It was important to have knowledge of the exact values of process parameters to determine the correctness and repeatability of the technological process dur-

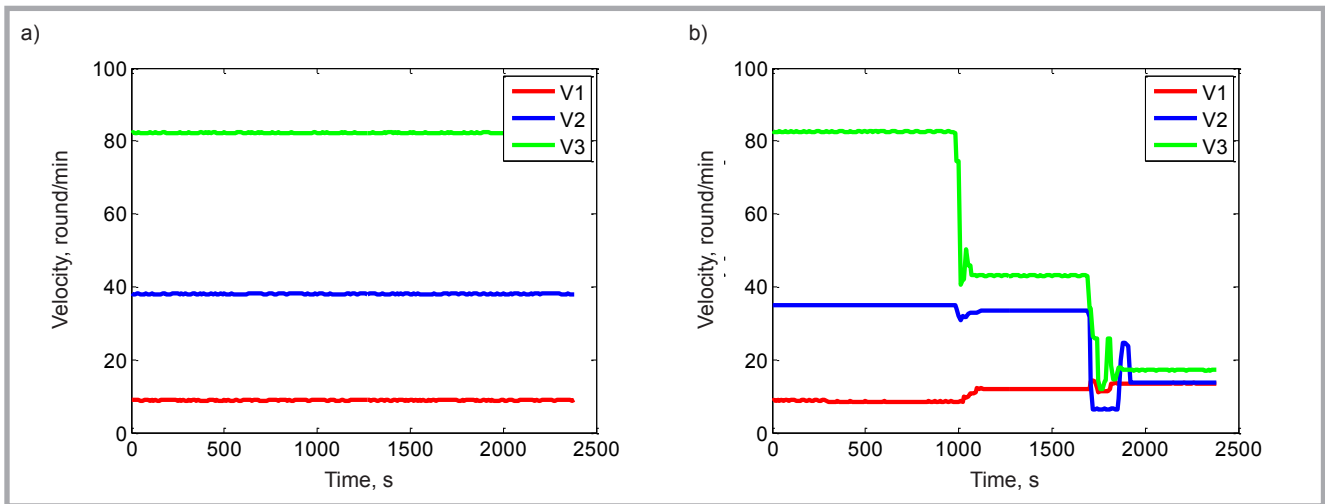


Figure 4. Graphs showing process data of the rotational speed at the three feeding-receiving points.

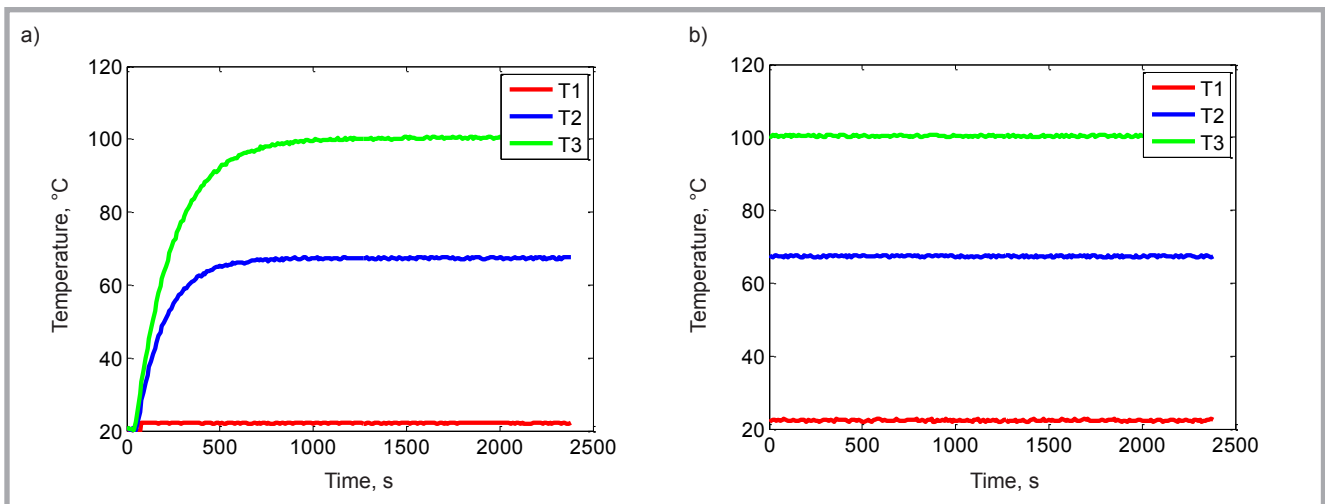


Figure 5. Graphs showing temperature process data in the two baths (T1, T2) and in the evaporator (T3).

ing the spinning of polyacrylonitrile and composite fibres. Therefore, during the measurements, measurement data packages were sent from all the devices every 10sec. An equally important issue during the wet-spinning process is to provide appropriate process parameters during the start-up of the process line. At this stage of the process, it is also very important to monitor the process parameters of the technological line. The correct start-up of the line consists of two elements: ensuring adequate temperatures in the solidifying and plastifying baths, an appropriate steam temperature in the steam generating column, and suitable corresponding velocity values at the feeding-receiving points. Each of these processes is characterised by a different time constant that causes the line to reach a steady state. Determination of the speed value is related to the time constant, in seconds; therefore, it can be said that it occurs almost

instantaneously. The temperature of the bath and the steam in the evaporator have a much longer setting time. These times are of the order of minutes or tens of minutes and must be taken into account during the start-up process.

Figure 4 shows the speed courses at the three feeding-receiving points in various operational states. **Figure 4.a** shows the steady state of the line, and **Figure 4.b** presents an example of programmed speed change. The curves present the transient states, in seconds, needed to achieve a new, stable speed value.

Figure 5 presents the changes in temperature values at three characteristic points of the line: in the solidifying bath (T₁), plastifying bath (T₂), and column for the production of water vapour (T₃). The curves shown in **Figure 5.a** present the process of heating and stabilisation

of temperature values, presenting that the bath and steam heating processes are characterised by relatively long time constants of the order of minutes and tens of minutes. **Figure 5.b** shows the steady state of the temperature monitoring points. The continuous process of parameter monitoring allows to perform a controlled start-up of the line so as to achieve and maintain the set values of speed and temperatures.

Processing of measurement data

The specificity of the measurement at the feeding-receiving point provides discrete values of the rotational speed of the point in rpm. Characteristic parameters of the fibres produced are the quantities brought to the length of the fibre, i.e. the linear parameters. In the first stage, it is necessary to transform measurements of the rotational speeds of the feeding-receiving points into corresponding linear veloci-

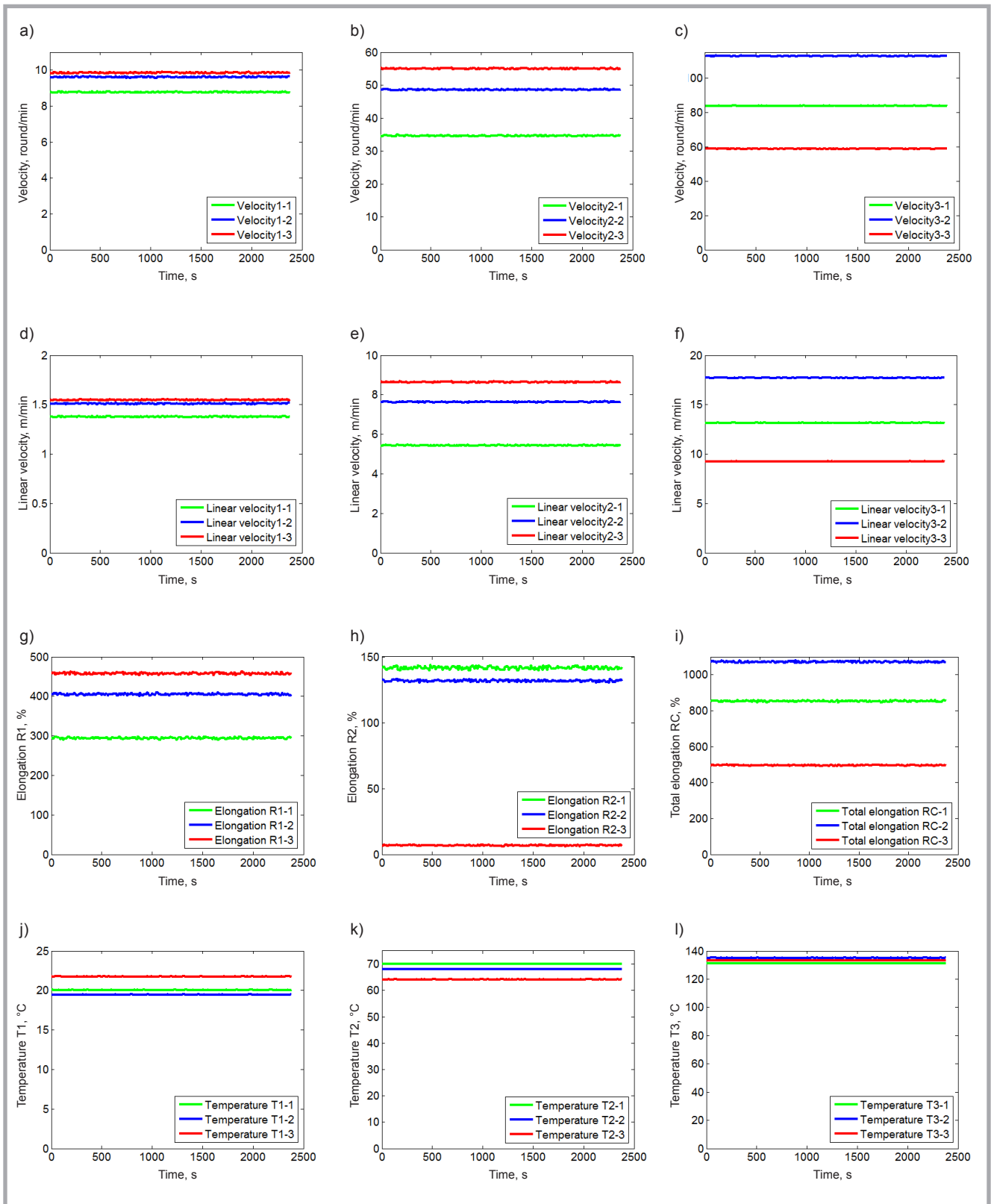


Figure 6. Plots showing the courses of process data of rotational speeds (a, b & c), linear velocities (d, e & f), elongations (g, h & i) and temperatures (j, k & l) at characteristic points of the process line in the operation.

Table 1. Average values of rotational speeds and temperatures for different spinning conditions of the same PAN solution.

Lp.	Sample	V ₁ , m/min	V ₂ , m/min	V ₃ , m/min	R ₁ , %	R ₂ , %	R _c , %	T ₁ , °C	T ₂ , °C	T ₃ , °C
1	PAN <i>Figure 7.a</i>	1.54	8.61	9.22	468.2	8.4	497.4	22.2	64.1	131
2	PAN <i>Figure 7.b</i>	1.51	7.63	17.67	406.7	137.6	1070.2	19.4	69.3	130
3	PAN <i>Figure 7.c</i>	1.37	5.44	13.11	298.2	142.7	856.9	20	70	133

ties, expressed in m/min. The characteristic value for performing this conversion is the diameter of the shaft at the feeding-receiving point. This transformation occurs according to the formula:

$$V_L = \pi dn \quad (1)$$

Where, d – shaft diameter in meters, n – shaft rotation speed in revolutions per minute.

Another characteristic parameter associated with fibre production using the “wet-processing” method is the degree of fibre elongation, defined as the ratio of the fibre length after the elongation process to the initial length:

$$R = \frac{\Delta l}{l_0} 100 \quad (2)$$

Where, Δl – increase in fibre length, l_0 – initial length of fibre.

Since the measurements of the linear velocity of the feeding-receiving points take place at fixed time intervals, it is possible to use the measurement of the elongation to measure the difference in linear velocity at two neighboring feeding-receiving points:

$$R_n = \frac{V_{L_{n+1}} - V_{L_n}}{V_{L_n}} 100 \quad (3)$$

Where, $V_{L_{n+1}}$ – linear velocity of the next point, V_{L_n} – linear velocity of the previous point.

The amount of the total elongation is equal to the sum of elongations at individual stages, which can be expressed in % following the formula:

$$R_c = \left[\left(\frac{R_1}{100} + 1 \right) \left(\frac{R_2}{100} + 1 \right) - 1 \right] 100\% \quad (4)$$

Where, R_1 – size of the plasticising elongation, %, R_2 – amount of elongation in the pair, %.

Results

Results of measuring data acquisition

First, it was necessary to determine process data for the production of undoped polyacrylonitrile fibres. For this purpose, during the spinning process, the settings at the feeding-receiving points were changed (Table 1) in a way that enabled the smooth acquisition of three fibre bundles on the coils (Figure 1 item 9) and so that a ribbon composed of 500 fibres did not break. Therefore, it was possible to

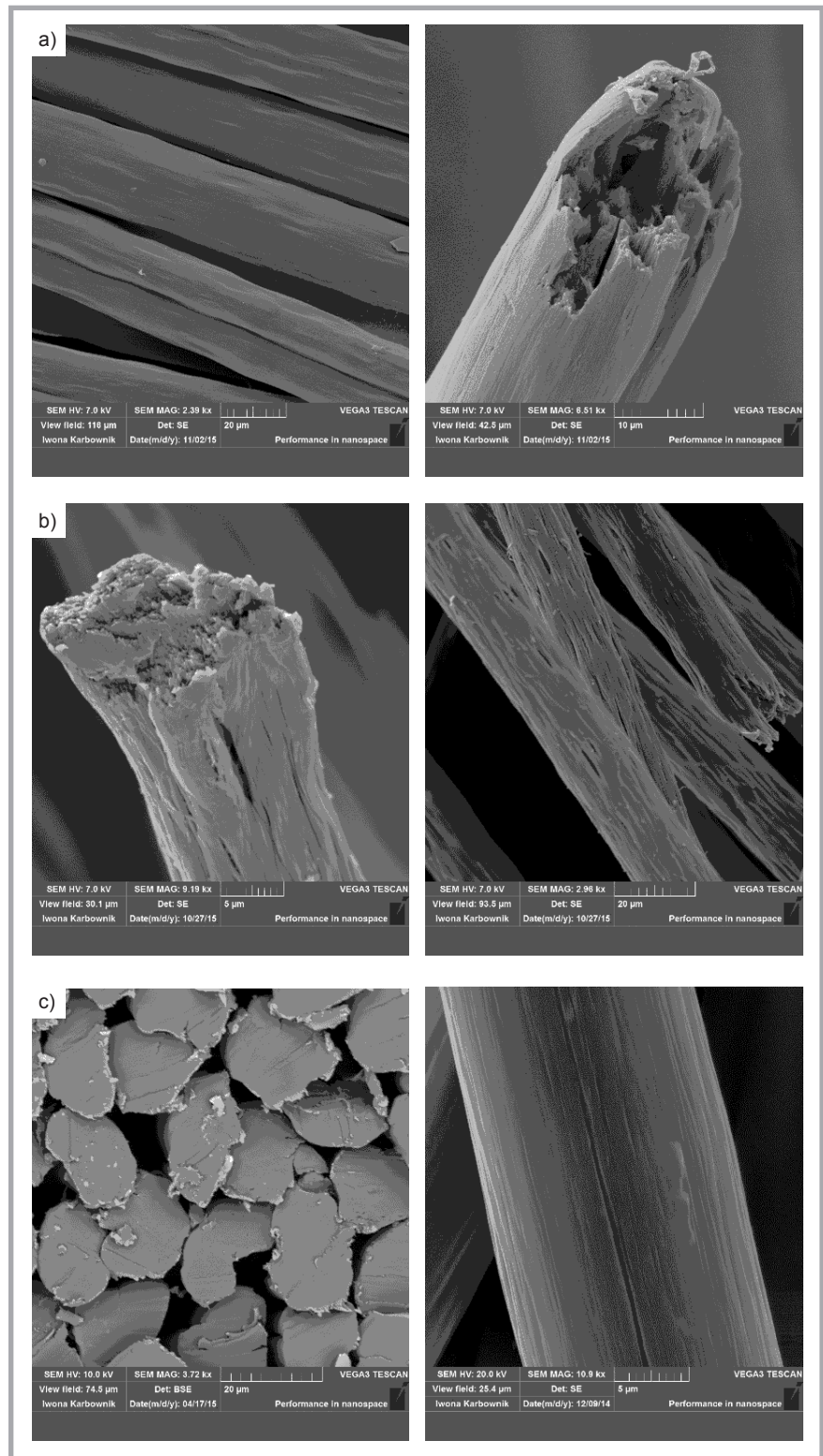


Figure 7. Polyacrylonitrile fibres: a) unelongated in steam of specific strength $WiP = 11.43$ cN/tex, b) subjected to too much tension at all stages of the spinning process with $WiP = 23.20$ cN/tex, c) at correct process parameters with $WiP = 38.51$ cN/tex.

compare the influence of various thermal conditions and the given elongation of fibres on their appearance and physical parameters. The averaged results of measurements of rotational speeds and temperatures collected during the spinning of the samples are given in Table 1. It can

be seen that as a result of different rotational speeds at individual points, these samples differ in their total elongation. The results of measurements of rotational speeds, linear speeds and temperatures recorded during the spinning of these fibres are illustrated in Figure 6. For com-

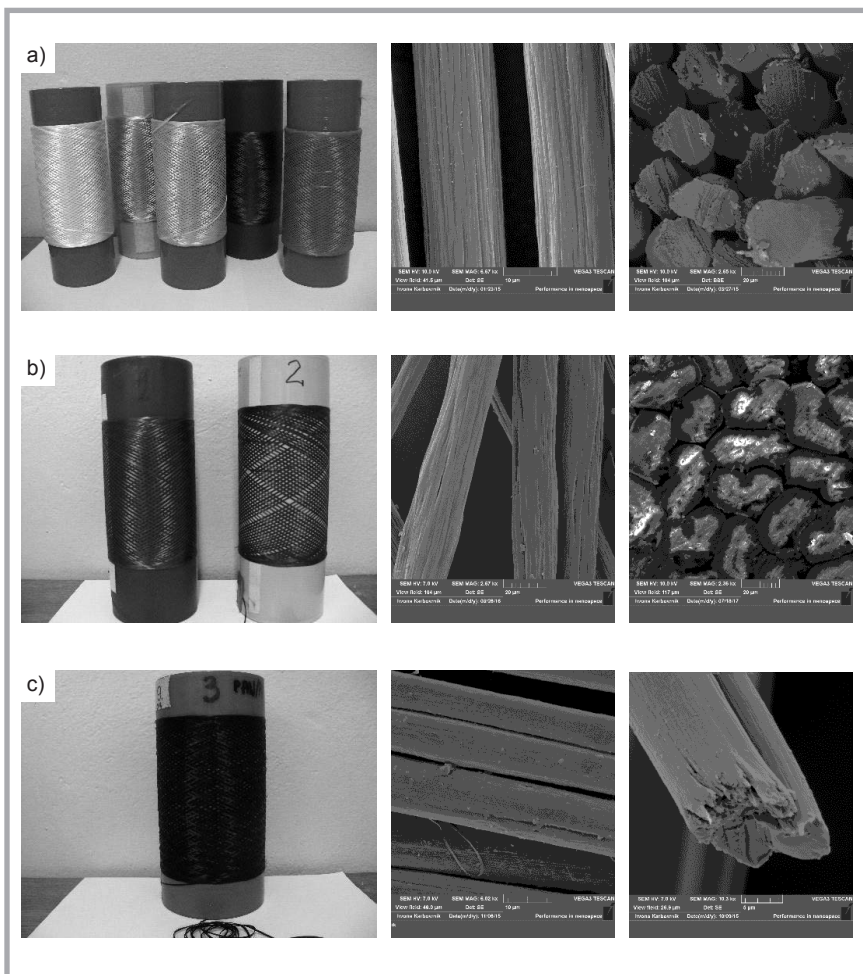


Figure 8. Polyacrylonitrile fibres: a) doped with silver nanoparticles (PAN + Ag), b) doped with polyaniline (PAN + PANI), c) doped with carbon nanotubes (PAN + CNT).

parison purposes, various parameters of the spinning process for one type of fibre were used to check how they affect the physical parameters, appearance and quality of these fibres.

Figure 6 shows instantaneous process data of rotational speeds and temperatures for the samples from **Table 1** at characteristic points of the line. The remaining waveforms (linear velocities and elongations) are calculated on the basis of formulas (3 & 4). As can be seen from the graphs shown in **Figure 6**, the PAN fibre spinning process was characterised by the assumed stability of the rotational speed and temperature parameters in important elements of the processing line. The constant values of rotational speeds (**Figure 6.a, 6.b** and **6.c**) throughout the spinning process implies a constant value of linear velocities in various sections of the process line (**Figure 6.d, 6.e** and **6.f**). The linear velocities, in turn, are the basis for calculating the elongations in individual sections of the line (**Figure 6.g** and **6.h**) and the total elongation (**Figure 6.i**).

As can be seen, the waveforms of these quantities are also characterised by constancy over time. The slight ripples or changes in values visible on the waveforms are the result of noise and measurement inaccuracies. The temperature waveforms shown in **Figure 6.j, 6.k** and **6.l** are also characterised by constancy of their value during the whole spinning process, indicating the correct stabilisation of bath temperatures and the steam generating column.

Methods of analysis and properties of fibres obtained

The spinning solutions were subjected to rheological tests and the fibres obtained to mechanical and structural tests. Rheological measurements of the spinning solutions were obtained with a Rheoviscometer, Anton Paar, ReolabQC.

A PC controlled low-load machine was used for testing mechanical properties of the fibres formed (Zwick Z2.5/TN1S, Germany) according to the PN-EN ISO 2062:2010 standard [29]. The threads

were measured in order to examine changes occurring in the structure of the fibres before and after the composite structure had been prepared.

The fibres samples' morphology was analysed under a TESCAN VEGA3–EasyProbe (TESCAN Brno, s.r.o., Czech Republic) scanning electron microscope equipped with VEGA TG software, which was also used for morphological analysis of the doped polyacrylonitrile fibres (high vacuum mode (SE); accelerating voltage: 7÷20 kV). Before the measurements, the samples were sputtered with Au-Pd (Cressington Sputter Coater 108 auto, UK) for 120s.

PAN fibres (**Figure 7**) and composite fibres were obtained, in which the polymer matrix was polyacrylonitrile, and the addition was silver nanoparticles (**Figure 8.a**), polyaniline (**Figure 8.b**) or carbon nanotubes (**Figure 8.c**), and 2,3,5-triphenyltetrazolium chloride TTC (**Figure 9**). Depending on the additives used, the fibres could differ in colour (**Figure 8**).

When planning the modification of the spinning solution to obtain composite fibres, it was necessary to ensure that the properties characteristic for a given type of fibre did not degrade. It was found that this requirement was met with respect to the polyacrylonitrile matrix using nanoparticles obtained during the preparation of spinning solutions. The additions used during the modification of the PAN fibres did not lead to a change in the rheology of spinning solutions (**Table 2**), which would have prevented them from being forced through the spinneret's holes. The diameter, morphology and structure of the composite fibres obtained determine their physicochemical properties.

Table 2 shows the linear density of undoped fibre (PAN) and composite PAN fibres doped with silver (PAN + 1% Ag, PAN + 3% Ag), with carbon nanotubes (PAN + 1% CNT), and with polyaniline (PAN + 5% PANI), as well as rheological coefficients of the spinning solutions from which these fibres were obtained, elongation values in the plastifying bath and steam, the total elongation, and the specific strength of fibres.

The basic properties of each spinning solution that demonstrate its quality are the rheological properties. By modifying the composition of these solutions in

a predefined way, it is necessary to determine the effect of modifications on these properties. When analysing the rheological properties of spinning solutions, the following are analysed:

- n – characteristic flow index [–].
- k – consistency coefficient [$\text{Pa} \cdot \text{s}^n$].

These two basic rheological parameters of each spinning solution were determined experimentally. **Table 2** presents the values of exponents n (flow index) and consistency coefficients k of spinning solutions that contain admixtures and of those that do not.

The value of the n -flow index depends on how the polymer macromolecules in motion are placed and form a system of mutually sliding layers. The linear polymer chains in the spinning solution (with sufficiently high concentration) can be largely entangled. Thus, the determined value $n < 1$ indicates that all spinning solutions obtained are shear thinned liquids, which is desirable for technological reasons. The flow rates of solutions containing additives are slightly lower, meaning that additive molecules affect the orientation of polyacrylonitrile macromolecules. The lowest value of the n coefficient was shown for 5% of the PANI supplement. This effect on the n -value may be due to the difference in the molecular structure of both polymers. As is known, the melt flow index depends on several factors, e.g. the average molecular weight of the polymer and its degree of polydispersity, the degree of branching of the macromolecules, and the content of additional components (especially fillers) and plasticisers.

In the system under study, both polymers are linear, but PAN macromolecules form entangled structures, and PANI macromolecules are largely rigid due to the presence of quinone rings in them. This parameter, in addition to the temperature, pressure and concentration, has a significant impact on the value of the consistency coefficient k . Considering the technological requirements, the coefficient of consistency k of the spinning solutions should be greater than $20 \text{ Pa} \cdot \text{s}^n$. In comparison to the changes in the exponent value, the presence of polyaniline particles and the relatively high proportion of nanosilver in the spinning solution (3%) caused a significant change in the consistency ratio. However, all spinning solutions were characterised by good over time stability. There was no agglomeration of nanoparticles or change in the rheological parameters of the solutions for 72 h.

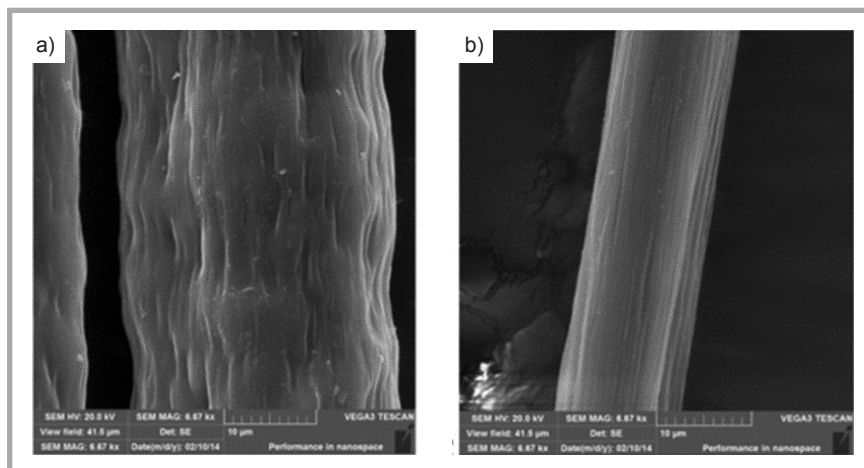


Figure 9. Morphology of 2,3 & 5-triphenyltetrazolium chloride TTC doped fibres: a) unelongated fibres ($m_1 = 302.77 \text{ tex}$, $\text{WtP} = 11.31 \text{ cN/tex}$), b) fibres elongated in steam ($m_1 = 71.97 \text{ tex}$, $\text{WtP} = 6.53 \text{ cN/tex}$) [12].

Table 2. Linear density, rheological coefficients, elongation values and specific strength of fibres. **Note:** m_1 – linear mass of fibres; $m_1 = m/l \cdot 1000 \text{ [g/m} \cdot 1000 = \text{tex]}$, m – average sample weight, l – length of sample; n – power exponent in dependence on the way in which the polymer macromolecules are arranged; k – coefficient of consistency in dependence on the temperature, pressure, concentration, and average molecular weight of the polymer; R_1 , R_2 , R_c – elongations: in the plastifying bath, steam and the total, respectively; WtP – specific strength of fibres.

No.	Sample name/ addition and its % share in polymer	m_1 , tex	Rheological coefficients of spinning solutions		R_1 , %	R_2 , %	R_c , %	WtP, cN/tex
			n	k , Ns^n/m^2				
1	PAN	95.59	0.9585	23.18	298.5	140.8	859.6	38.51
2	PAN+1%Ag	89.84	0.9550	25.73	299.0	140.2	858.4	41.17
3	PAN+3%Ag	95.55	0.8674	55.74	298.5	141.2	861.2	39.50
4	PAN+1%CNT	107.77	0.8349	22.29	266.6	78.1	552.0	22.47
5	PAN+5%PANI	100.03	0.6983	53.65	264.1	82.2	557.9	18.39

Undoped and composite polyacrylonitrile fibres were produced in the form of a continuous ribbon in which there were 500 monofilament fibres. The thickness of the tape was $90 \pm 110 \text{ tex}$, which was closely related to the control and stabilisation of technological parameters. Also, the cross-section of the fibres (bean-shaped, renal or irregularly oval) and the appearance of their surface were dependent on these parameters. On the surface of the fibres, furrows along their longitudinal axis were visible, as well as numerous cracks, sometimes even distinct ones (**Figure 7.a, 7.b**). It was important to obtain fibres with the best strength parameters, which was reflected in the appearance of their surfaces and cross-sections. PAN fibres, illustrated in **Figure 7.c**, had the best strength (**Table 2** item 1), and the appearance of their surface and cross-sections was considered appropriate. The study attempted to obtain composite fibres similar in both appearance and in the strength values of those fibres.

For TTC doped fibres, slightly different technological conditions were used because the temperature could not exceed $40 \text{ }^\circ\text{C}$ at all stages of fibre production. Thus, the fibres could not be subjected to a bath at $70 \text{ }^\circ\text{C}$ or be exposed to superheated steam. The picture in **Figure 9.a** illustrates such a fibre with a linear mass $m_1 = 302 \text{ tex}$, whose total stretch was equal to that obtained in the plastifying bath ($R_1 = R_c = \text{approx. } 200\%$). Whereas, the fibre illustrated in **Figure 9.b** was subjected to elongation in steam ($R_c = \text{approx. } 1000\%$), which resulted in partial decomposition of the TTC compound; but the appearance of the fibre surface improved significantly. Its linear mass was $m_1 = 71 \text{ tex}$, but the strength of the fibre obtained was significantly worse than the version not subjected to elongation in steam. The share of dopant (TTC) caused changes in the structure of the entire volume of fibre. In fibres that were elongated in steam, the proportion of the

For TTC doped fibres, slightly different technological conditions were used because the temperature could not exceed $40 \text{ }^\circ\text{C}$ at all stages of fibre production. Thus, the fibres could not be subjected to a bath at $70 \text{ }^\circ\text{C}$ or be exposed to superheated steam. The picture in **Figure 9.a** illustrates such a fibre with a linear mass $m_1 = 302 \text{ tex}$, whose total stretch was equal to that obtained in the plastifying bath ($R_1 = R_c = \text{approx. } 200\%$). Whereas, the fibre illustrated in **Figure 9.b** was subjected to elongation in steam ($R_c = \text{approx. } 1000\%$), which resulted in partial decomposition of the TTC compound; but the appearance of the fibre surface improved significantly. Its linear mass was $m_1 = 71 \text{ tex}$, but the strength of the fibre obtained was significantly worse than the version not subjected to elongation in steam. The share of dopant (TTC) caused changes in the structure of the entire volume of fibre. In fibres that were elongated in steam, the proportion of the

crystalline phase increased by more than 20% compared those not exposed to this type of elongation; and TTC partially decomposed, which caused that these fibres had twice less specific strength [12].

The wet forming process of composite polyacrylonitrile fibres depends, among other things, on the structure of the polymer used to make the spinning solution (quantitative proportion of different types of mers or polymer particles of different mass) as well as on the type of admixture and method of its introduction into the spinning solution. The conditions of the fibre forming process are very important, i.e. conditions for the setting, shaping the stretching stages, thermal stabilisation and drying. The diameter, morphology and fibre structure determine the physicochemical properties of fibres obtained by this method. These, in turn, depend on the type of polymer and parameters of their forming process.

Conclusions

To sum up, it can be stated that the system proposed for monitoring a technological line for the production of synthetic fibres is an effective solution enabling full control of the spinning process with simultaneous archiving of data.

In particular, this system allows:

- execution of the spinning process in repeatable conditions,
- programmable change of speed and temperature parameters in order to develop appropriate production profiles,
- quick response of the operator to compensate for any changes or differences in velocity or temperature that occur during the spinning of fibres,
- easy transition from the fibre's linear velocity to the momentary strain that is responsible for the fibre strength (Table 2),
- protection against fibre breakage (while maintaining maximum stretch) by on-line monitoring of rotational and linear velocities at the feeding-receiving points,
- archiving of measurement data in order to create a real process database for later reproduction and to create a simulation model.

The acquisition of measurement data performed during fibre spinning was crucial to obtain the fibres desired. As a consequence of a properly conducted technological process, fibres with a certain

appearance and breaking strength were obtained. Parameters influencing the morphology and structure of PAN composite fibres can be divided into groups according to:

- the type of polymer used and the admixture for the spinning solution,
- parameters of the spinning solution obtained,
- the process of fibre formation and its parameters,
- parameters of the fibre processing environment.

By modifying any of the parameters, it affects the kinetics of the entire forming process as well as physicochemical properties of the fibres. By introducing unusual dopants or by synthesising particles in the spinning solution, changes are observed not only in its rheological parameters but also in the kinetics of the forming process and, consequently, the properties of the fibres. Therefore, it is necessary to archive speed and temperature measurement data, which allows the creation of a database combining process parameters with the parameters of the fibres obtained. The database allows interpolation or extrapolation of the parameter space to obtain new composite fibre production profiles of the same composition but with different stretching and thermal processing values. The monitoring system also makes it possible to apply similar technological parameters (speed and temperature) to different fibre material compositions in order to check the influence of the dopants used on the physico-chemical properties of the fibres obtained.

References

1. Lewandowski S. Monitorowanie produkcji i jej jakości jednym z celów procesu przędzenia. *Przegląd Włókienniczy – WOS* 2007; 2: 58-60.
2. Jackowska-Strumiłło L, Cyniak D, Czekański J, Jackowski T. Neural Model of the Spinning Process Dedicated to Predicting Properties of Cotton-Polyester Blended Yarns on the Basis of the Characteristics of Feeding Streams. *FIBRES & TEXTILES in Eastern Europe* 2008; 16, 1(66): 28-36.
3. Bilińska L, Żyła R, Smółka K, Gmurek M, Ledakowicz S. Modeling of Ozonation of C.I. Reactive Black 5 Through a Kinetic Approach. *FIBRES & TEXTILES in Eastern Europe* 2017; 25, 5(125): 54-60. DOI: 10.5604/01.3001.0010.4628.
4. Petruyte S. Influence of Technological Parameters on the Periodical Effects of

Fancy Yarns. *FIBRES & TEXTILES in Eastern Europe* 2008; 16, 3(68): 25-29.

5. Hasani H, Tabatabaei SA. Optimizing Spinning Variables to Reduce the Hairiness of Rotor Yarns Produced from Waste Fibers Collected from the Ginning Process. *FIBRES & TEXTILES in Eastern Europe* 2011, 19, 3(86): 21-25.
6. Mikołajczyk Z. Optimisation of the Knitting Process on Warp-Knitting Machines in the Aspect of the Properties of Modified Threads and the Vibration Frequency of the Feeding System. *FIBRES & TEXTILES in Eastern Europe* 2011, 19, 6(89): 75-79.
7. Eldeeb M, Demir A. Optimising the Production Process of Rieter Air Jet Spun Yarns and a Model for Prediction of their Strength. *FIBRES & TEXTILES in Eastern Europe* 2018; 26, 1(127): 36-41. DOI: 10.5604/01.3001.0010.7794.
8. Urbańczyk G. *Nauka o włóknie cz.II*, Lodz University of Technology Press, 1978 (in polish).
9. Karbownik I, Rac O, Fiedot M, Suchorska-Woźniak P, Teterycz H. In Situ Preparation of Silver – Polyacrylonitrile Nanocomposite Fibres. *European Polymer Journal* 2015; 69: 385-395.
10. Karbownik I, Rac-Rumijowska O, Fiedot-Toboła M, Rybicki T, Teterycz H. The Preparation and Characterization of Polyacrylonitrile-Polyaniline (PAN/PANI). *Fibers Materials* 2019; 12, 664.
11. Rac O, Karbownik I, Fiedot M, Rybicki T, Karpińska A. Composite Fibres Doped with Carbon Nanoparticles for use in Textronics. *IMAPS, 2016; 25-28 September, Wałbrzych*.
12. Kozicki M, Szaśiadek E, Karbownik I, Maniukiewicz W. Doped Polyacrylonitrile Fibres as UV Radiation Sensors. *Sens. and Actuators B* 2015; 213, 234-243.
13. Mikołajczyk T, Boguń M, Szparaga G. Influence of the Type of Montmorillonite and the Conditions of Fibre Formation from a Polyacrylonitrile Nanocomposite on the Fibre Properties. *FIBRES & TEXTILES in Eastern Europe* 2007; 15, 3(62): 25-31.
14. Rahatekar SS, Rasheed A, Jain R, Zammarano M, Koziol KK, et al, Solution Spinning of Cellulose Carbon Nanotube Composites using Room Temperature Ionic Liquids. *Polym.* 2009; 50: 457-458.
15. Karbownik I, Rybicki T, Karpińska A, Teterycz H. PAN Fibers with Nanoprecipitated Ag and Au for Use in Textronics. *Mater. Sci.-Pol.* 2016; 34: 564-570.
16. Karbownik I, Teterycz H, Rybicki T, Kulpiński P, et al. Sposób wytwarzania włókien poliakrylonitrylowych o właściwościach antybakteryjnych, antygrzybiczych lub/i elektroprowadzących. Patent PL 229181.
17. Karbownik I, Fiedot M, Rac O, Rybicki T, Suchorska-Woźniak P, Teterycz H. Effect of Doping Polyacrylonitrile Fibers on their Structural and Mechanical Properties. *Polym.* 2015; 75: 97-108.
18. Karbownik I. Modyfikacja włókien z zastosowaniem wybranych technik i nano-

technologii, Lodz University of Technology Monographs, 2016 (in polish)

19. Mikołajczyk T, Boguń M, Kurzak A, Wójcik M, Nowicka K. Influence of Forming Conditions on the Tensile Strength Properties of PAN Fibres Containing a Ferromagnetic Nanoaddition. *FIBRES & TEXTILES in Eastern Europe* 2007, 15, 3(62): 19-24.
20. Broda J, Przybyło S, Lewandowski S. Selection of Optimal Formation Parameters of Polyporpylene Fibrillated Fibres Designed for Concrete Reinforcement. *FIBRES & TEXTILES in Eastern Europe* 2012; 20, 6A(95): 69-74.
21. Mikołajczyk T, Szparaga G, Rabiej S, Frączek-Szczypta A. Influence of Formation Conditions on the Structure and Properties of Nanocomposite PAN Fibres Containing Silver and Hydroxyapatite Nanoadditives. *FIBRES & TEXTILES in Eastern Europe* 2010; 18, 5(82): 16-23.
22. Boguń M, Mikołajczyk T. Influence of Coagulation Bath Temperature on the Porous Structure and Strength Properties of PAN Fibres Including Montmorillonite. *FIBRES & TEXTILES in Eastern Europe* 2006; 14, 3(57): 19-22.
23. Mikołajczyk T, Szparaga G, Rabiej S, Frączek-Szczypta A. Influence of Formation Conditions on the Structure and Properties of Nanocomposite PAN Fibres Containing Silver and Hydroxyapatite Nanoadditives. *FIBRES & TEXTILES in Eastern Europe* 2010, 18, 5(82): 16-23.
24. Boguń M, Mikołajczyk T. Influence of Coagulation Bath Temperature on the Porous Structure and Strength Properties of PAN Fibres Including Montmorillonite. *FIBRES & TEXTILES in Eastern Europe* 2006; 14, 3(57): 19-22.
25. Mikołajczyk T, Szparaga G. Influence of Fibre Formation Conditions on the Properties of Nanocomposite PAN Fibres Containing Nanosilver. *FIBRES & TEXTILES in Eastern Europe* 2009; 17, 4(75): 30-36.
26. Mikołajczyk T, Olejnik M. 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. *FIBRES & TEXTILES in Eastern Europe* 2009; 17, 1(72): 20-25.
27. ATmega162(V)-Complete Datasheet – 8-bit AVR Microcontroller with 16K Bytes In-System Programmable Flash, ATmega162, ATmega162V (pdf document).
28. The RS-485 Design Guide – Texas Instruments Application Report, 2008, revised 2016, (slla272c.pdf, pdf document).
29. PN-EN ISO 2062:2010 Textiles-Yarns from packages – Determination of single-end breaking force and elongation at break using constant rate of extension (CRE) tester.

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