

Optoelectronic System for Control of Nanofiber Materials Production Using Wire Electrospinning Process

DARINA JASIKOVA¹, DANIEL HANCIL¹, MICHAL KOTEK¹, RADIM KRENEK², VACLAV KOPECKY¹

¹Institute for Nanomaterials Advanced Technology and Innovation
Technical University of Liberec
Bendlova 1407/7, 461 17 Liberec
CZECH REPUBLIC

²Elmarco, s. r. o.
Svarovska, 460 10 Liberec
CZECH REPUBLIC

darina.jasikova@tul.cz <http://www.cxi.tul.cz>

Abstract: - Here we come up with the novel idea of production quality management that will be based on the correlation of data on-line system with inputs of materials and spinning conditions and output information of nano fibrous layer. This paper demonstrates application of optical monitoring on Nanospider™, technology and has wider impact on understanding the process on wire with moving carriage than fiber diameter relation only, unlike the stationary needle process. Here we have come out from this empirical knowledge and we used computer visualization for process analyzing. The basic idea was the detection of the stable parts and the raising jets of fibers. We also designed the illuminating system according to further image analysis presented as signal in data series. We analyzed multi-level data using graph theory. Using this progress we can express the behavior of quantum of jets over time into one graph. These parameters are explained and correlated to the setup of industrial application.

Key-Words: - electrospinning, two phase flow, visualization, measurement, image analysis, signal processing

1 Introduction

The electrospinning process of producing nonwoven textiles from nanofibers found its place in many branches, such as filtration, wound healing, affinity membrane, protective clothing, energy generation, enzyme immobilization, drug delivery, scaffolds in tissue engineering, or biomedical applications. [1,2] The methods for industrial nanofibers processing could be divided into two main categories: needle electrospinning or needless one - electrospinning from free surface (splashing electrospinning, rotating drum - solid cylindrical electrode, rotating spiral wire electrode, disc collector, and sharp pin inside the rotating collector, knife-edged electrodes, and single wire electrode). [3-5]

Last mentioned method, electrospinning from single wire electrode, is world well known as the Nanospider™ technology [6]. This technique raised under evolution of rotating drum technique in order to decrease user contact with solvents and to increase the effect of the process. This process is based on stationary, charged single wire electrode. The solution is applied to the electrode using an applicator head, so called carriage with reservoir.

The fibers are collected on substrate between the spinning and the opposite grounded plate electrode. As spinning wire electrode (SE) is covered with a polymeric solution, it is immediately divided into separate droplet and spinning jets start from them. The behavior of the jet depends on the properties of polymer solution (viscosity, conductivity, surface tension) as well as the applied voltage and surrounding effecting character of electric field. [7, 8]

From the view of fluid mechanics, the process of electrostatic spinning could be classified as two phase flow. The liquid phase (polymer solution) is disintegrated into fibers due the force of electric field. The strong electric field is acting in two mechanism: influencing the charge of the liquid, and as the outer force acting due the electric wind that effecting further fibers motion toward the grounded electrode – collector. The simplified process is well physically described as electro hydrodynamics. [9, 10] These works are extremely valuable because they provide insights into the mechanism of fluid disintegration in an electric field and the idea of distributing into drops or fibers. [11] The disintegration of the liquid to the fibers occur two

kinds of instabilities: bending and break-up. The prevailing instability is dependent on the chemical and physical properties of the solution and also led to different kind of fibers, their diameter and surface morphology. Nowadays point of view is focused to the explanation of fluid discretization directly on the charged electrode. There is an assumption of process control outgoing of the knowledge that primary distribution is influencing further product of fiber layer.

We can also use knowledge of the field of experimental fluid mechanics describing the process itself. The methods and techniques used for visualization and description of flows and fluid motions can be implemented into this part of research.

The aim of the project is to develop optoelectronic system for on-line quality control of production of nanofiber materials using an electrospinning from wires, known as Nanospider™. So far the parameters (voltage, carriage frequency and distance between SE and the substrate etc.) of the processes were set experimentally, strongly depending on the skills and know-how of the staff and based on empiric knowledge, without deeper understanding about behavior of liquid on the SE. The quality assurance of the raising fibrous layer is evaluated in post-process, usually using microscope, permeability, porometry, etc. This is very time consuming process and the backward reaction is slow. The need for the automation progress in this area is obvious.

Visualization of the electrospinning process was broadly investigated by Reneker and Yarin, whose experiments were focused on understanding the process of raising nanofibres from the Taylor cone on a needle. This work was extended by Gevelber et al. that pointed the relation between the geometry of spinning jet (Taylor cone), process parameters (humidity, rate of evaporation), and product parameters (fiber diameter). [12, 13]

This paper demonstrates application of optical monitoring on Nanospider™, technology and has wider impact on understanding the process on wire with moving carriage than fiber diameter relation only, unlike the stationary needle process. It concerns to statistical behavior of the process with other unknown parameters, like number droplets and spinning jets per unit length of wire, direction of jets, droplet size and geometry, droplet lifetime, stability and way of consumption, wire vibrations etc.

Here we come up with the novel idea of production quality management that is based on the correlation between data on-line system with inputs

of materials and spinning conditions and output information of nanofibrous layer.

2 Experimental Setup

The system of quality management is based on optical measurements and visualization of electric process. Optical system helps to resolve basic parameters and increase objectivity of process description that is unique by small objects (droplets, jets, nanofibres) on long distance (safety distance of high voltage) and fast dynamic process.

2.1 Laboratory Device

The concept of the laboratory device for electrostatic spinning was based on original commercially used system Nanospider™. This system uses the electrostatic field between the grounded upper plate and the charged spinning wire.

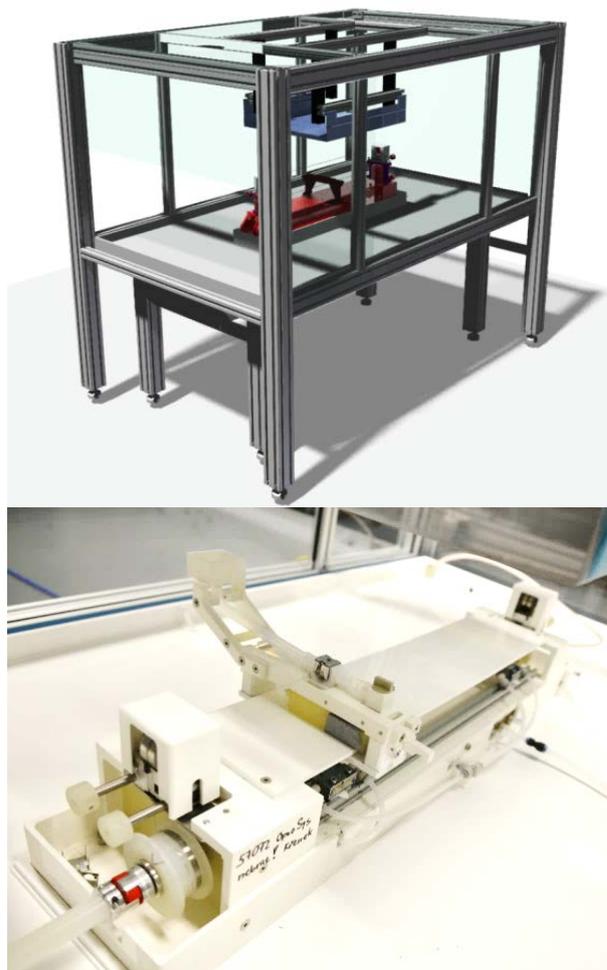


Fig. 1 Laboratory device based on industrial machine for production of nanofibers layers. There is design of the machine in the first picture and the carriage with reservoir in the second one.

The polymer solution is applied to the wire using a linearly moving carriage. Unlike the industrially produced machine, the laboratory device is optically accessible from all sides. At very beginning of the design phase of optoelectronic system, the final configuration of the visualization setup was still not considered. The construction of the experimental device has been optimized with respect to safety and functional characteristics. Variable voltage setting, the distances between electrodes and the ventilation system are enabled.

2.2 Visualization technique

The setup of monitoring software was setup for proceedings of the spinning process. The resulting fabric is influenced by a wide range of parameters, not all of them can be measured objectively. The optical system for visualization of electrospinning process is based on lightening unit, chamber with optical access, optical lens system, camera, and software for image analysis.

The visualization of polymer jets is strongly effected of the lighting scenes. When placing the illumination in the opposite wall of the chamber under consideration (with respect to the geometry of the machine), we tested these configurations and their combinations using two basic setups. The reflective method was based on arrangement that the camera is in off to axis of the light beam, angle α is varying according to the reflection index of polymer solution. The second illuminating method was based on the shadow method. Here we set the camera directly in the axis of light beam, so the angle is identical with axis of the camera, α is 180° .

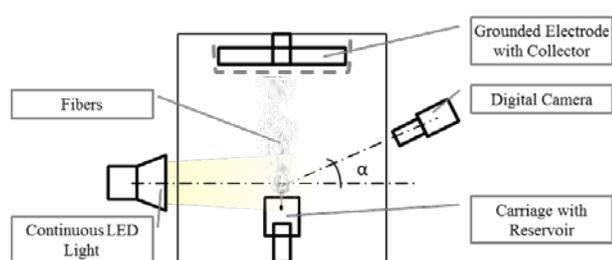


Fig. 2 Experimental setup of visualization method.

The reflective method may be implemented in a wide range of angles α . Minimum angle axis arises from the requirement that the light shines directly into the camera. Considering the diameter and divergence of the light, the distance between the light and the objective lens (whose aperture is also necessary to count) there can be set minimum angle between 5° and 10° . Maximum angle is the ratio of the size of the viewing window and the length of the

chamber. The sight glass is the limiting element due to the requirements for electromagnetic shielding and the size of chamber length that is given by a safe distance (due to a high electric field). We were able to achieve an angle of 25° (at a scanning center of the machine with the deflection of light in the horizontal plane) in the tested configurations. Calculation and testing, however, confirms that the highest reflection is achieved at small angles. Thus, we sought to reduce the divergence of the beam and the diameter of the beam size close to the size of the scene. This we achieved through a simple lens light-guide.

Shadow method displays objects in backlit, thus highlighting the contours of the object (fluid on the spinning electrode). This method required light with very homogeneous diffuser, which affects the quality of the background image analysis. This was also proved and confirmed in tests. Diffuser itself absorbs huge amount of light. These increase requirements of the luminous flux.



Fig. 3 Example of reflective method (inversed image) using collimated light - with good visibility flexural instability, but with significant spot reflections on drops on the spinning electrode (wire). Contour and shape of liquid are difficult to see.

Tests showed the strengths and weaknesses of both methods. Reflexive method reflects light very well to stable part of the Taylor cone, jet, and emerging fiber for bending instability. The spatial resolution of images is higher than when using the shadow method. A major deficiency of reflection method is large amount of overexposed sites (due to reflection at spherical drops) and the object size (diameter of the jet) that is very dependent on the level of illumination (angle of a luminous flux). Furthermore, following precise angles is a complication for the production and quality, and moreover restricts monitoring to extreme positions.

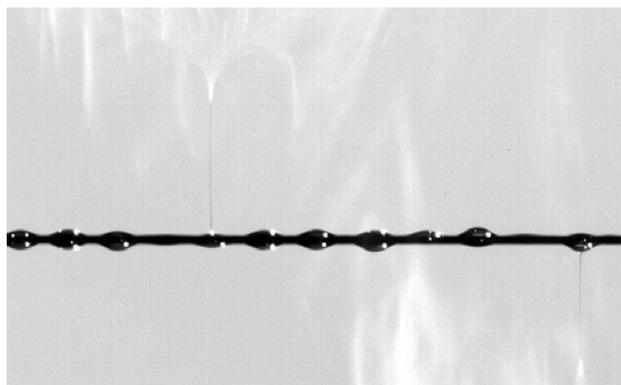


Fig. 4 Result of combined reflective and shadow method (without image inversion). There can be seen bending instability and contours, but still with a strong reflection point.

Reducing of light divergence led to excessively increase of image contrast. On the other hand, higher divergence deteriorated quality background (Fig. 6). Quality deteriorated with higher intensity of electric field. The very high intensity is used in industrial applications to reach high yield of fibers. The scene rendering, or changes in brightness between the individual phases of the process, were not satisfactory.

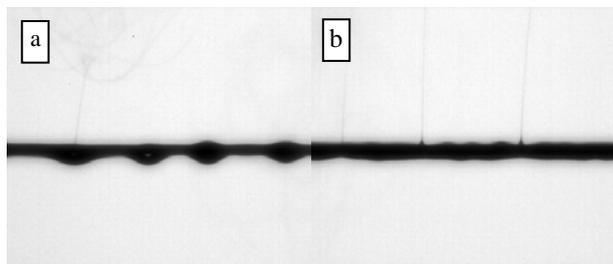


Fig. 5 Comparison of electric process (a) PAN (polyacrylonitrile solution of low viscosity), and (b) PUR (polyurethane solution of high viscosity).

The difference between monitoring using the shadow method (the image is not inverted), and diffuse light is seen in figure 4 and 5. The diffuse light illumination is more sensitive to fluid volume close to wire than the bending instability above. The jets appear to be thinner in comparison to reflection method illumination.

Shadow method visually translates the subject in a dark color (shade) against light background as a vertical plane cut. It is relatively easy way to obtain high quality image for further analysis in a construction (mechanical) and optical terms. However, we encountered the problem of small jets diameter rising from Taylor cones in the industry commonly used polymers compared to previous studies with a limited number of "polymer demonstration". This makes the further image

analysis more difficult. The relation between average jet and fiber diameter requires greater magnification. Here we also reach the optical limits, not only caused by hardware (camera, lens system), but also physical limits based on light divergence and light scattering.

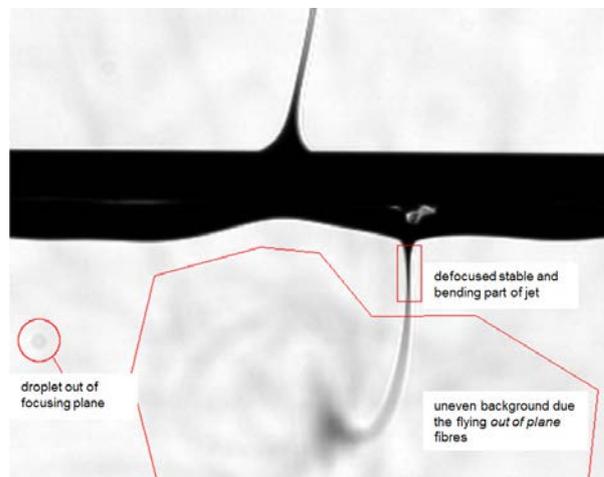


Fig. 6 Shadowgraph method - enlarged area in the vicinity of jet and image impurities around.

However, the process behavior can be described by the changes in shape of the liquid that is formed to stable jets. The great advantage of the shadow method is the luminance uniformity. We obtained image that can be faithfully followed by application of most detection algorithms.

Both methods require high resolution and fast phenomena high illumination due to the large distances. The influence of light heating is inconsiderable. High light output carries an energy conversion to the temperature increase on fibers. This increase evaporation rate of the solution and the final fiber diameter is decrease. There was required cold light - suitable highly bright LEDs that are recently undergoing great development. Here we used LED chips operating lumen up to 6000 lm. Multi-chip lighting provided about 3 times more light than a reported one chip (in test conditions). The divergence of commercial lighting was solved only at the level of conventional reflector. We used 1-chip unite set with improved and designed lens, thus we have a sufficient illumination (> 100 thousands of Lux), and low light divergence to illuminate the scene. We ultimately used informal method, and we are also optimized diffuser (material, thickness) according to the process demands.

We tested several configurations of illumination system. The most suitable one was chosen depending on the reflectivity of light by multiple jets, light absorption, surface texture, and the

background. Here we were dealing with the light color – its spectrum and wavelength. We tried to use red light to achieve the highest contrast, but finally, this way was abandoned.

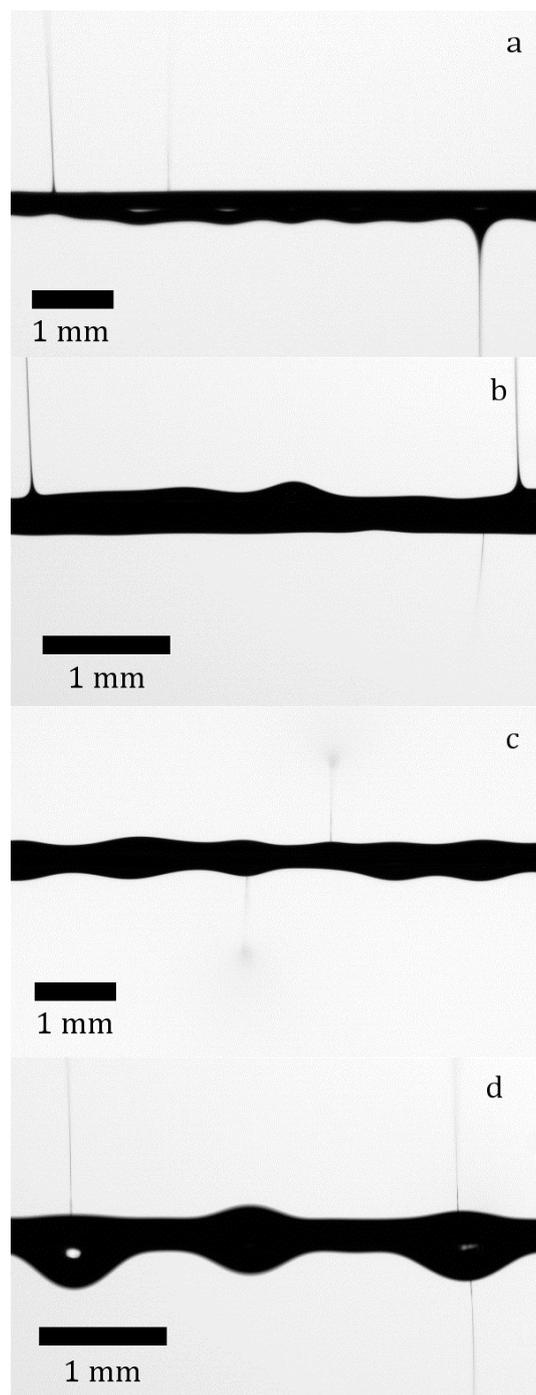


Fig. 7 Shadowgraph method - examples of PUR (a, b) and PA6 (c, d) are displayed at 11 $\mu\text{m}/\text{px}$ (a, c) and 7 $\mu\text{m}/\text{px}$ (b, d).

We also tested various light sources: high output of sulphuric lamp, powerful focused white light illuminator, diffuse red light, and 532nm laser sheet lightening. We finally chose simple cold white LED

light source that is suitable for further industrial system. The flat matrix LED illuminator was fitted with diffuse filter. We set the principle of rear lighting. This setup use lighting towards the camera. The major advantage of this setup is the bright contouring of the object and enables to achieve high contrast of the picture.

The analysis of records at reduced working distance provided several conclusions. It is not confirmed that the thickness of the stable part of jets was measurable. Even if the resolution is 7 $\mu\text{m}/\text{px}$, the jet is more pronounced than at 11 $\mu\text{m}/\text{px}$, but it is still not sufficient amount of pixel for reliable analysis. Although brightness could be used, thickness estimates would be unreliable. This is in contradiction with Gevelber et al. [12] The difference is minimum in two aspects. The Gevelber publication is based on needle electrospinning, where the initial drop drop is several times greater than that of free-electrospinning. In the Gevelber publication, a high molecular weight polymer (ethylene oxide) (PEO) solution was used for spinning. This solution produces thicker fibers than polymers used for most industrial applications (such as PA6, PVDF, etc.)

It was confirmed that higher resolution leads to better defined shape of the liquid interface. Although the shape recognition algorithms are not yet fully developed, this method obviously refines the calculation of the shape change based on the change of roughness. Here we can come out of directly analytical tools for searching the match between the characteristic shape (solitary jet, Plateau-Rayleigh instability, bending instability) and its mathematical functions. Furthermore, in thin-walled thin-film situations, the thickness change estimate is more than 50%.

2.3 Image Processing

The analysis of electrospinning production is based on direct correlation between the process itself and the produced nanofibers layer. There exist some significant features of the process related to uniform/uneven layer, instabilities or thickening of fibers. Here we have come out from this empirical knowledge and we used computer visualization for process analyzing. The basic idea was the detection of the stable parts and the raising jets of fibers.

There was extremely important appropriate selection of the visualization method. The fundamental dilemma was between images that have been photographically perfect but further computer processing and analysis was impossible. In the image analysis the quality of lighting is as important

as the picture quality (resolution, depth of field, sensitivity, and noise factor).

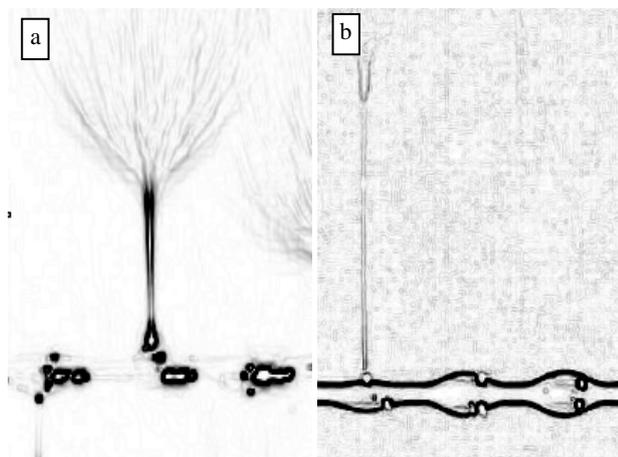


Fig. 8 Method of edge detection is applied to (a) the reflection method (a), (b) the combined illuminating method.

Figures 8 and 9 show examples of image processing that are using the edge detection in three configurations of illumination. The system was set with sensed inspection lens and camera with 1" chip of sensor pixel size 5.5 microns. Dark lines contours describe fluid distribution of solution at wire electrode at any given time. Sequencing of these images brings temporal characteristics of geometric parameters, which are tied with procedural storyline on the electrode.

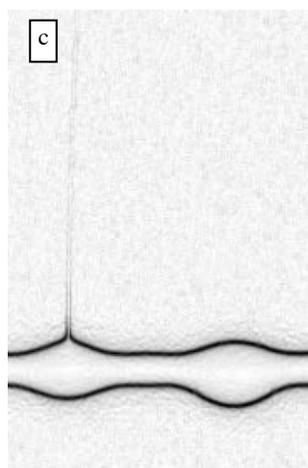


Fig. 9 Shadow method provides minimum of artifacts when analyzing the shape of the liquid (formation of Taylor cones) on wire electrode.

We used reflector Constellation 120 for the illumination and process capturing with high speed camera SpeedSense from DantecDynamics with resolution (1200x800) px for laboratory purpose. We run the light in the continuous regime. The process of electrostatic spinning was visualized by

the camera running at the frequency of 500 Hz with exposition of 1500 us for the statistical valuation. We also used frequency of 5 kHz and the exposition 190 us for capturing very start of the process. The recorded data were image analyzed using own designed algorithms.

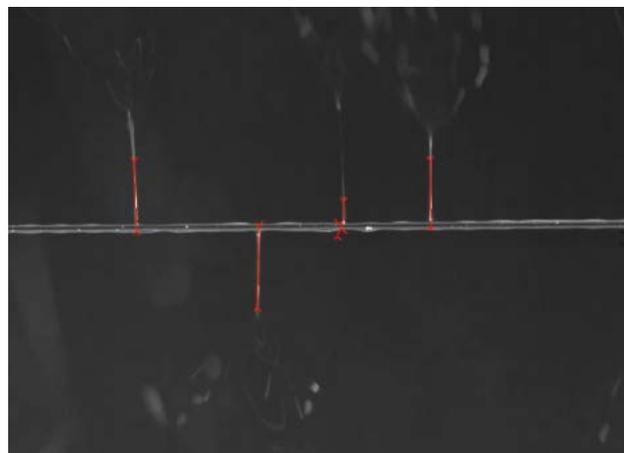


Fig. 10 Records of the spinning process and the software identification of stable part of jets.

Image data from this method provide us an overview on the course of the spinning process. The record of size (50x150 mm) was scanning over whole wire length during spinning PVA 10%. Recorded movies were loaded with LabVIEW software and additional algorithms programmed in C++ for further automatic processing. The figure 10 shows the course of the analysis of stable Taylor cones. Here it is automatically identified each cone, and it is determined by its length and each cone lifespan. These parameters play an important role in determining the quality of the resulting product. The identification of stable jets (colored red) is seen in Fig. 10.

The software automatically works with individual image data files. This release adjusted some parameters of image analysis depending on the initial conditions. It consists of several modules individually programmed in a modular language LabVIEW.

Another important function of data analysis gathered by the monitoring system is the image correlation process and microscopic images of the resulting product. This feature should be included to online process control as future help guide, and identification of undesirable phenomena. This analysis is focused on the entire around the wire. This function is used for some of the horizontal levels (profiles). Analysis of the entire image area would require complex algorithms and longer processing time.

The records on selected position were analyzed in horizontal lines - 1 mm below the wire and 1 mm above the string. In these lines is a profile evaluated image brightness and plotting. Each process is thus described by a set of profile graphs. The advantage of this interpretation against images is the fact that this data can be easy to precisely quantify and compare.

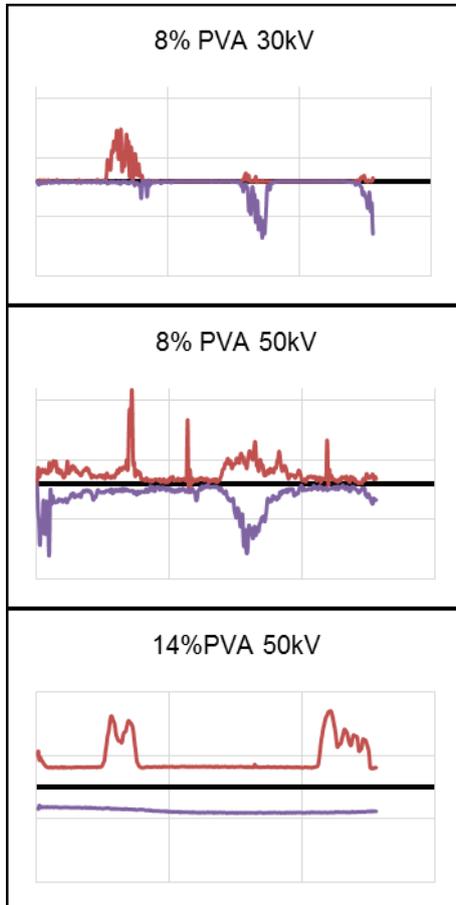


Fig. 11 Results of brightness analysis 2mm above and below the wire – marked with solid line. There is seen jets statistical behavior under different voltage and for various polymer concentrations.

We were not satisfied with simple image analysis that provided object detection or recognition. There is need of time development of process identification in industry. Commonly, the data interpretation that has more than three components is difficult. Here, we were dealing with two space axis, time and image information (brightness, contrast, and gamma). Image processing over time led to interesting results. The time record was assembled into one image following changes in brightness. Figure 12 shows the typical pattern of time-lapse record of electrospinning process. This pattern can be called as a map. The map determines the thickness of the polymer over, or under the SE.

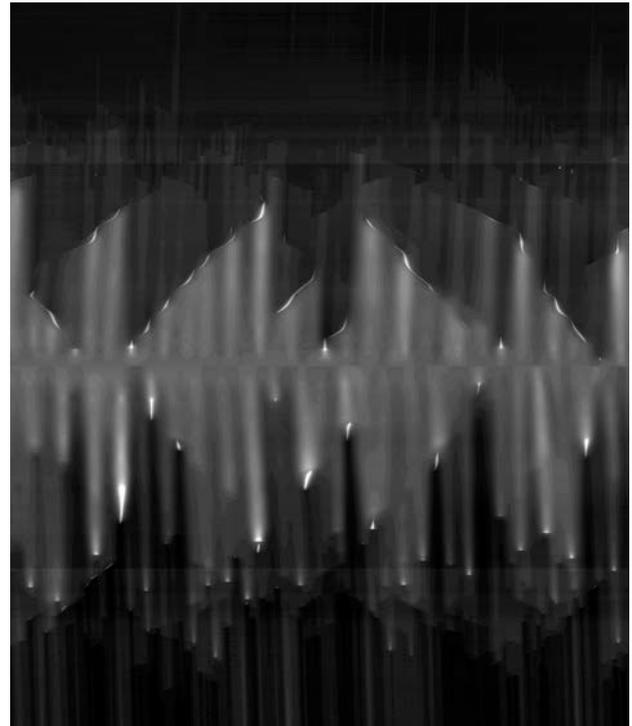


Fig. 12 An example of time-lapse analysis for further graph theory processing.

It is relative to the wire thickness. The x axis in figure 12 responds to position over SE. The sum of brightness correlates with the stable jet part. This map predicated 3D behavior of jet in principle. The maximal brightness relates to 100% of polymer solution over the picture and also fully load SE. The value will be 0 percent, if there is no polymer solution on SE. The fully load SE situation is only theoretically assumption. The maximal value of brightness according to stabile lightning is 200 (the maximum intensity of 255). Each column in the map represents one time record of situation over SE. All records fold together gives two values of start and end of electrostatic process.

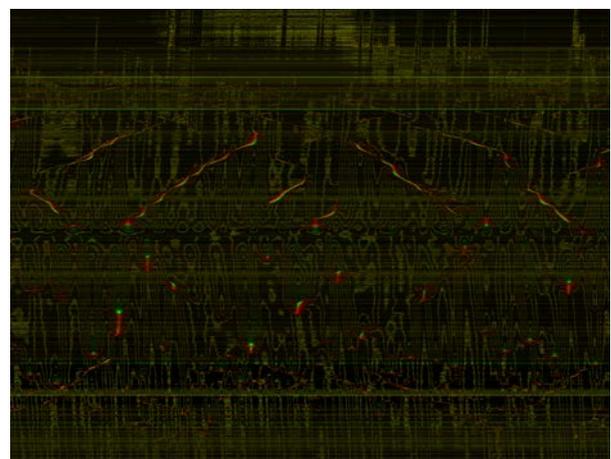


Fig. 13 The map of differences.

The map of differences is created by subtracting of two consecutive images or lines from the previous map (Fig. 13). The green color indicates positive values (filling volume is increasing), red is negative part of process. The intensity of the colors (not so visible) is then proportionally directly proportional to the magnitude of scattered difference.

Better contrast is obtained when figure 14 is transformed to binary image and the map of jet is represented as white lines.

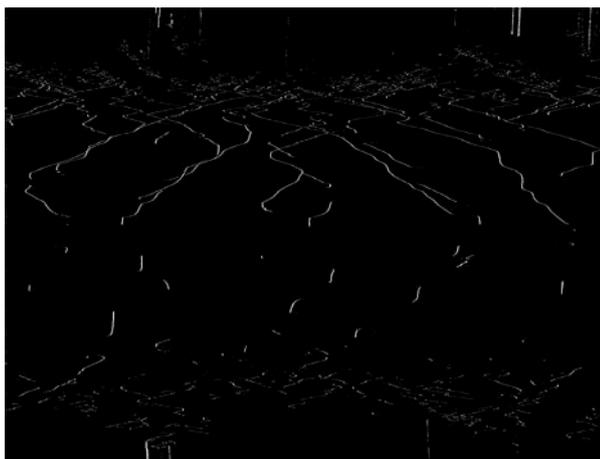


Fig. 14 The map of jets over SE.

These maps contribute to the novel idea in analysis of non-stationary, time-dependent process. So far, there is difficult to express the behavior of quantum of jets over time into one graph. Especially, when each jet live with its life. The jets differ in position, life spun, geometry etc. We can read stable jet as the lines are linear. The example seen in figure 14 shows the movement of jets along the wire in both directions. The jets are not stationary, but they travel toward the places with free polymer reservoir as the initial place is exhausted. We can also track the clear situation above and below SE. The lines above SE are denser and more dynamic then below. Another interesting point is the repeatability in jets behavior and movement. This takes effect in fiber orientation on grounded electrode and substrate. We can reveal weak places or empty space on SE, its damage or impurity.

Numerically, the filling over SE can be expressed as standard deviation. The lower number in figure 15 means uniform polymer solution deposition. We can also read a number of cones over SE as the s.d. increases.

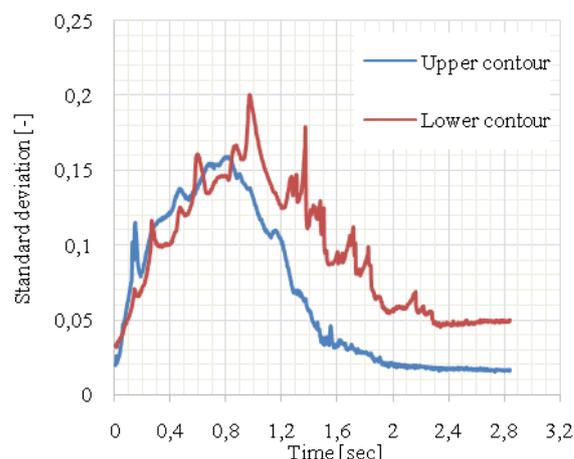


Fig. 15 Standard deviation of amount of solution over SE

3 Results and Discussion

The electrospinning process is very unstable, if the parameters (voltage, temperature, ventilation, solution characteristics) are not corresponding to each other. The process can be well tuned and in that case the stable jets can be determined. We tried to determine the image view into the signal that can be further described with function. There can be seen that the electrospinning process behave differently above and below the wire. The aim of balanced process is the high yield of fibers that means all stable part should occurs in the position toward the collector in ideal case.

Comparing signal from two investigated profiles there is difference between stable (top) and unstable (bottom) part during the process (Fig. 11). The graph takes form of narrow peaks luminance profile for stable course of the process. Instabilities or agglomerates of fibers in the luminance profile of these phenomena exhibit significantly. The profile plot fold wider and take up a significant part of the profile in this case. There can be also seen overlap. All these phenomena are identifiable directly form profiles and can be further analyzed with signal processing functions.

The yield of electrospinning process, the fibers properties, and layer uniformity are primarily determined by several limiting conditions of the key parameters (solution viscosity, humidity and air temperature, air flow and air saturation vapor of the solvent), the local values (at a given position on the spinning electrode). Not all parameter can be determined by classical methods. There are parameters: manifest of secondary parameters such as electric field strength, frequency of solution

recovery on the electrode, solution flow applicator head etc. There was given large number of input characteristics, the main objective of monitoring the optical recognition of the current state solution and traceable process and its relationship to the product and process parameters. Examples of detectable parameters of the analysis of the records of the polymer layer on the electrode wire processed in the following graph in figure 10.

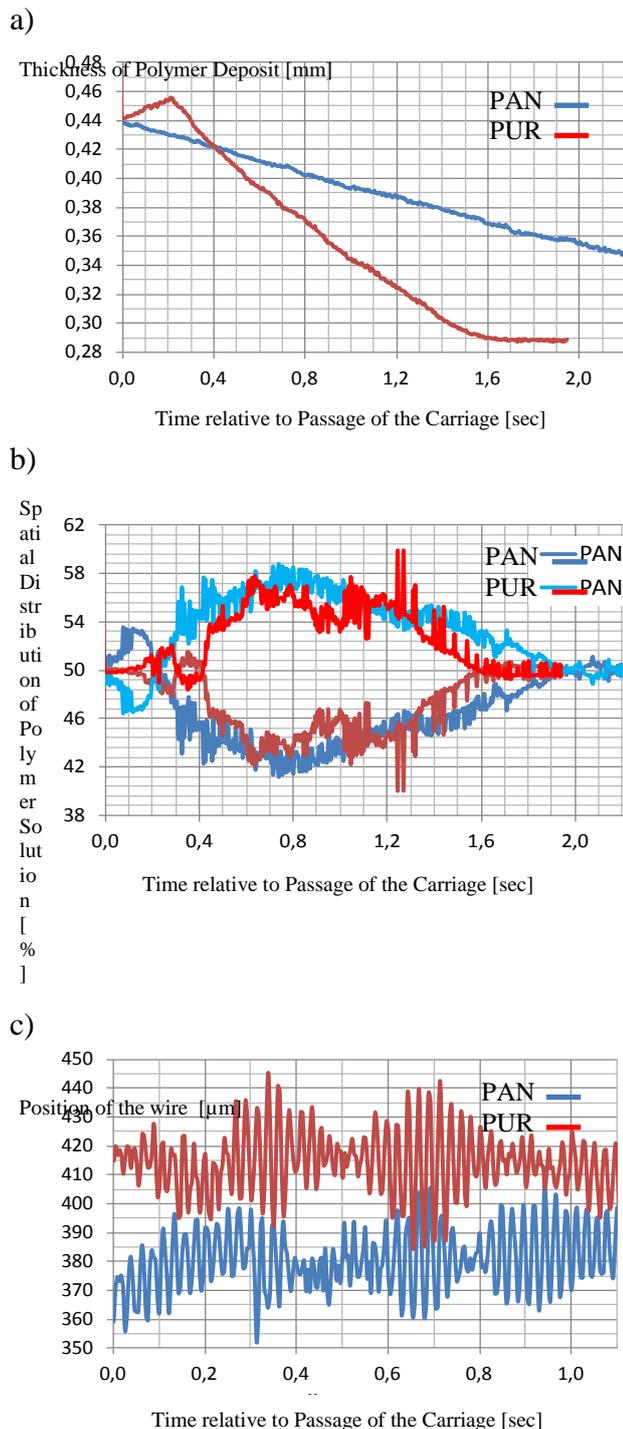


Fig. 16 Analysis of polymer solution on wire electrode (a). Evaporation of the solvent (B), percentage distribution of the solution above and below the electrode(C), vertical movement of the wire (vibration).

The basic parameter is the presence of a solution on the wire electrode. Actual amounts can be assessed based on the average thickness of the coating solution on the wire and presented over the unit length of the wire, the distance between the upper and lower contour - edge (Fig. 16 c). Electrospinning must always take place with the solution at the electrode. We can obtain the maximum required for recovery solution and the associated frequency of movement of the application head (Fig. 16 a) based on this characteristic. If lower frequency of carriage head is set, it has a major impact on performance or reduction, and the quality of nanofiber layer (higher fiber diameter distribution, instabilities). For viscous solution fluid movements are slower and therefore takes longer time to observed initiation of the process and formation of jets (extraction liquid) than outweighs consumption (Fig. 16 a) shows peak at 0.22 sec for PUR polymer solution).

The spatial distribution of the liquid is varying in time. Cumulatively, it can be expressed as the thickness of the coating above and below the wire (Fig. 10 b). Solution of low viscosity (PAN) very quickly responds to the electric field than outweigh the gravitational force (0.2 sec). The solution of high viscosity (PUR) exhibits reacting time as 0.1 sec. It is not independent to the fact that up to 0.4 sec with alternate states prevailing above and below the wire than outweighs the influence of gravitational forces. There is compensation of the spatial distribution of the lifetime of the process in both cases. There can be observed random oscillation curves related to the shape change of the liquid formation to stable jets.

The wire vibrations are also observable and important part of electrospinning process. The chart of wire electrode vibration can be seen in Fig. 16 c). The current wire position and deflection can be determined based on the position of the liquid stable sections (minimum, or maximum). Observed vibrations cease after 1.5 sec. This time is dependent on the carriage head speed, i.e. independently on the life time and amplitude from 10 to 50 microns, which is about 5%-25% of the diameter of the wire electrode. Vibration is one of the main causes of defects (drops) in nanofibres layers. The quantitative relationship and determination will be

subject to further studies and technology development.

Within a very short period of testing different polymer solutions we achieved significant benefits of optical monitoring. The lifespan of the single fibers jet was measured with the optical according to set conditions. There was observed very short lifetime for some kinds of PVDF polymer (0.7 sec), or conversely very long life for PA6 (about 7 seconds), which is one of the most stable systems.

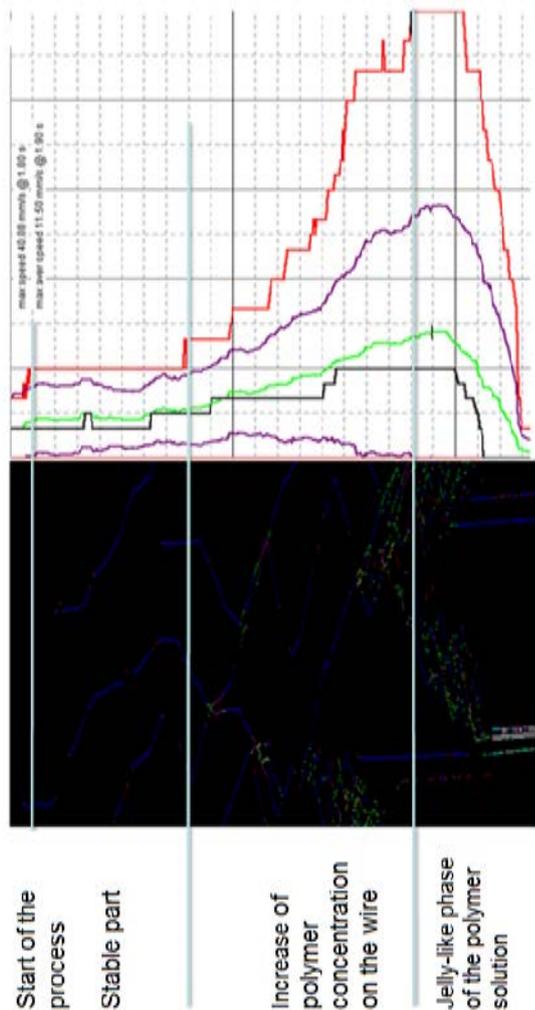


Fig. 17 Jet motion analysis using graph theory. The map is a horizontally displayed position on the string and a vertical (top-down) time scale. On the right, the velocity of jets movement over time is analyzed by different methods.

There cannot be described the polymer jet behavior with simple quotation just based on the variable constants according to these experiments. There has to be provided complex system analysis and signal processing that can be generalized with quotation but with variables set to individual polymeric solution. These data can be obtained from the signal processing as frequency characteristic.

These data are suggestive to increase the frequency of recovery solution (the set of flow allows 1.0 s - 1.5 s, according to the width of the machine) and for adjusting the formulation (composition of the solution, process control) to extend the life. The acquired data specifying the knowledge gained long-term subjective observation.

Furthermore, there was set the correlation between viscosity and changes in the form of liquid on the wire electrode. Obviously solutions of low viscosity are readily converted into droplets, whereby the curvature of the liquid improves the stability of jets, if droplets are not too large. Very stable position of jets (exhibits minimal movement and deviations) we see again on PA6, and PVDF. Unstable are viscous polymer solution such as PUR.

Microanalysis of shape of jets is currently limited with resolution of the inspection system, because of low magnification. With the development of analytical system we expect algorithmic improvements also focused on the emergence of jets from a larger volume drops. The volume changes would be linked with yield and product characteristics of the process, and fiber diameter in theory.

4 Conclusion

Here were shown visualizations of an experimental spinning device. There have been selected polymer solutions to observe changes with increasing excitation voltage on the wire. The scene lighting used LED light source, providing high light intensity and negligible thermal influence in two regimes of light setup.

The visualizations have to be analyzed using image processing to identify and subsequently quantified. There are more characters that require further analysis: density and spacing of the cones with spouted solution geometry, cones themselves, the length of the steady region, shape of branching and subsequent splitting.

The illumination using white light enables to observe the light scattering on the jets and separation part. This diffracted light and colors could be used for fiber diameter detection. The analysis of single jet, angles of whipping or branching will be the next step in research. For all these observations mathematical description would be created, so that different parameters could be stored in datasets.

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